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Operative Dentistry
Indiana University School of Dentistry, Room S411
1121 West Michigan Street, Indianapolis, IN 46202-5186
Phone 317-350-4371, Fax: 317-852-3162
<http://www.jopdent.org>

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Ultrathin CAD-CAM Ceramic Occlusal Veneers and Anterior Bilaminar Veneers for the Treatment of Moderate Dental Biocorrosion: A 1.5-Year Follow-Up

TH Resende • KR Reis • LH Schlichting • P Magne

Clinical Relevance

Ultrathin bonded posterior occlusal veneers represent a conservative alternative to traditional onlays and complete coverage crowns for the treatment of moderate and severe erosive lesions.

SUMMARY

Dental biocorrosion can produce a devastating impact on oral health. The restorative phase of the treatment should not cause additional

Tayane H Resende, MS, Analytical Laboratory of Restorative Biomaterials - LABiom-R, School of Dentistry, Federal Fluminense University, Niterói, RJ, Brazil

Kátia R Reis, DDS, MS, PhD, Department of Prosthodontics and Dental Materials, School of Dentistry, Federal University of Rio de Janeiro, Rio de Janeiro, RJ, Brazil

*Luís H Schlichting, DDS, MS, PhD, Department of General Dentistry, School of Dental Medicine, East Carolina University, Greenville, NC 27834, USA

Pascal Magne, DMD, PhD, The Don & Sybil Harrington Professor of Esthetic Dentistry, Department of Restorative Sciences, Ostrow School of Dentistry, University of Southern California, Los Angeles, CA 90089, USA

*Corresponding author: East Carolina University, General Dentistry, 1851 MacGregor Downs Rd., Mail Stop #701, Greenville, NC 27834, USA; e-mail: luishschlichting@gmail.com

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damage of the remaining sound tooth structure. Ultrathin occlusal veneers are a conservative alternative to traditional onlays and complete crowns for the treatment of severe biocorrosive lesions. This strategy is explained in the present case report through a full-mouth rehabilitation of a patient with moderate biocorrosion. Maxillary anterior teeth were restored using the bilaminar technique (lingual direct composite veneers with labial ceramic veneers) and posterior teeth using ultrathin CAD-CAM ceramic occlusal veneers. The technical aspects required for the implementation of this new restorative design are presented with a special emphasis on the control of tooth preparation based on diagnostic wax-up, provisionalization, and the use of CAD-CAM technology.

INTRODUCTION

The dental enamel shell is designed to withstand a lifetime,¹ as witnessed in many patients in their 80s

and 90s who can still enjoy their teeth despite obvious signs of wear and cracking. However, the enamel thickness is reduced by dental biocorrosion. By definition, the term “erosion” is a physical mechanism, making the term “corrosion” more appropriate for dental tissues. Likewise, the term “biocorrosion” differs from “corrosion” because the prefix “bio-” designates chemical, biochemical, and electrochemical actions on enamel and dentin. Therefore, biocorrosion comprises endogenous and exogenous acidic and proteolytic chemical degradation of enamel and dentin as well the electrochemical piezoelectric action of the collagen in the dentin.² Modern lifestyles have led many to poor eating habits and dysfunctional behaviors that could explain the increase in prevalence of erosive lesions as well as their severity.³ When experienced by young individuals, the tissue loss in moderate and severe cases of dental biocorrosion becomes problematic, resulting in significant biological (sensitivity and pulpal damage), esthetic, and functional consequences in addition to the complications due to the occlusal changes (with or without loss of the vertical dimension).⁴

Ultrathin occlusal veneers have been advocated as a conservative alternative to “re-enamelize” the occlusal surface compared to traditional overlays or full coverage crowns.^{5,6} The intrinsic strength of materials like lithium disilicate–reinforced glass ceramics and high-performance composite resins,⁷⁻¹² combined with immediate dentin sealing,¹³⁻¹⁵ allows for thinner designs. It has been observed that patients get motivated and encouraged when during a treatment planning presentation they learn that minimum or no additional reduction of tooth structure will be needed.

This article illustrates the application of these concepts in a full-mouth rehabilitation of a patient affected by moderate biocorrosion. In contrast with a previous article¹⁶ where ultrathin CAD-CAM composite resin occlusal veneers were utilized (Lava Ultimate, 3M ESPE, St Paul, MN, USA), this case used a ceramic (e.max CAD, Ivoclar Vivadent AG, Schaan, Liechtenstein).

CLINICAL TECHNIQUE REPORT

A 34-year-old man presented to the Department of Prosthodontics and Dental Materials at the Federal University of Rio de Janeiro in 2015 requesting treatment for dentin sensitivity and dental wear. Intraoral evaluation revealed generalized dental wear, including dentin exposure and clinical evidence of dental biocorrosion.³ Smooth and concave

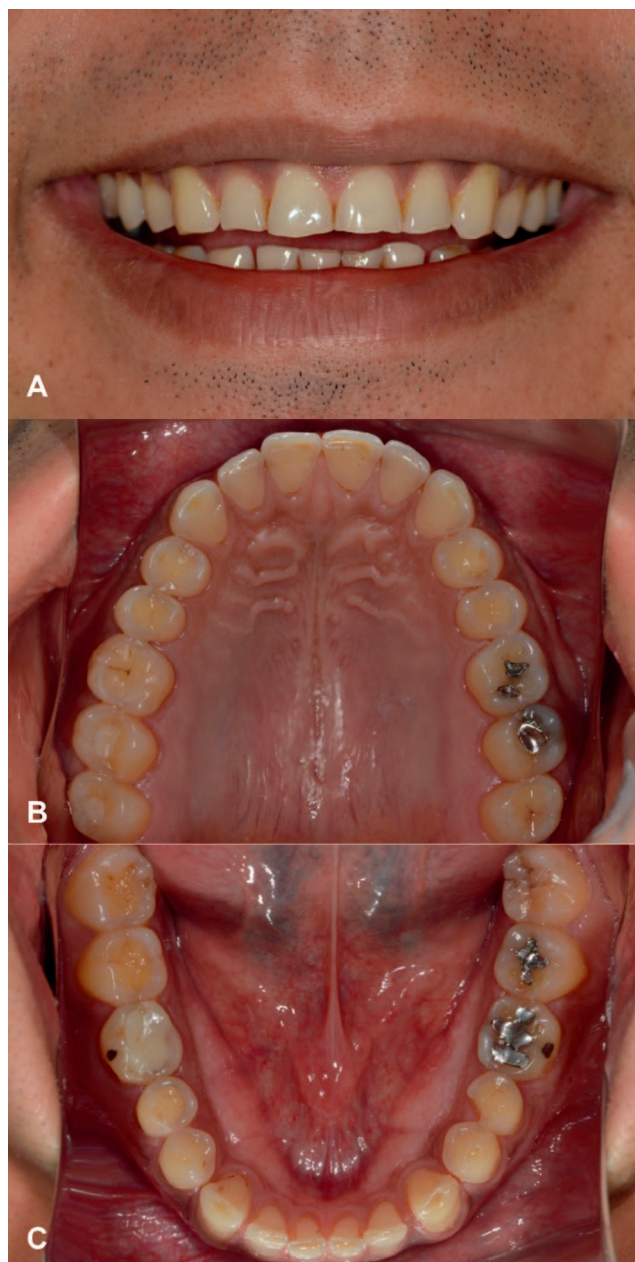


Figure 1. Preoperative views of a 28-year-old male patient with moderate to severe dental biocorrosion and wear. (A): “Visual tension” can be perceived during smile due to biocorrosion/wear signs. (B-C): Intraoral occlusal views display severe loss of enamel with higher degree of destruction in the upper premolars and lower first and second lower molars.

lesions become apparent on facial surfaces as well as cupped cusp tips, thinned or inexistent enamel covering the occlusal perimeter, and restorations standing above the level of the adjacent tooth structures (Figure 1A through 1C).

Regarding the etiology, the hypothesis of intrinsic origin associated with gastroesophageal reflux dis-

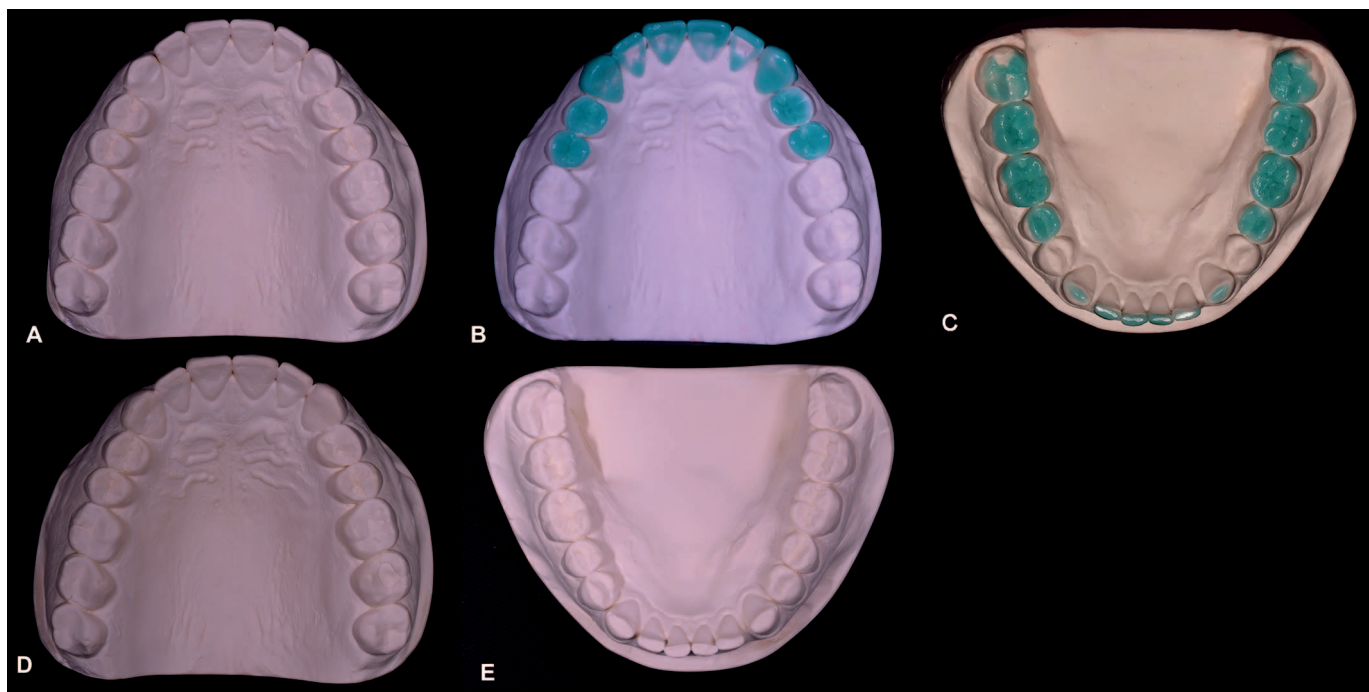


Figure 2. (A): Scannable stone replica of waxed cast with unaltered canines as repositioning reference. (B-C): Complete waxing (including canines, mandibular incisal edges and mandibular third molars). (D-E): Scannable stone replica of the complete waxing were useful for anterior restorations (mock-up, selective preparations, provisionals and enabled the use of the biogeneric copy tool).

ease could not be confirmed because of the dental wear pattern and patient medical history. However, the frequent consumption of acidic beverages (yerba mate tea with lemon) may explain the severity in this case. Furthermore, the presence of wear facets on the functional surfaces, such as the palatal and incisal edges of the maxillary and mandibular incisors, suggests the combination of biocorrosion and attrition disorder. The patient was immediately advised to control the intake of acid drinks and to use an occlusal guard to protect the teeth from mechanical wear.

The restorative treatment was set in two stages: ultrathin CAD-CAM ceramic occlusal veneers on selected posterior teeth and bilaminar veneers (direct composite resin on palatal and ceramic on labial) on maxillary anterior teeth.^{17,18} Diagnostic casts (Pearl White GC Fujirock EP, GC America, Alsip, IL, USA) were obtained from complete arch polyvinyl siloxane impressions (Virtual, Ivoclar Vivadent). The bite was recorded at maximum intercuspal position (MIP), and casts were mounted on a semiadjustable articulator. Centric relation (CR) was initially considered; however, MIP position was utilized instead because the patient already had a retrognathic profile, which would be aggravated if

CR was chosen. The vertical dimension of occlusion (VDO) was arbitrarily augmented by 1 mm at the incisal to allow space for the restoration on the palatal aspect of the maxillary anterior teeth and to minimize the amount of preparation required for the ultrathin occlusal veneers.¹⁶ A complete mouth additive waxing was ordered (GEO Classic Mint-Opaque Wax, Renfert GmbH, Hilzingen, Germany) and carried out in two steps by the dental technician. First, all teeth planned to be restored (maxillary arch: anteriors and premolars; mandibular arch: second premolars, first and second molars) were additively waxed, except for the canines. Both waxed models were then duplicated (Elite Double 32 Fast, Zhermack, Badia Polesine, Italy) and poured with scannable stone (Snow Rock 3D Scan, Dk Mungyo Corp, Hanrim-myun Gimhae-si, Gyeongsangnam-do, South Korea) (Figure 2A). This step provided technical accuracy for intraoral repositioning of silicone guides for mock-up fabrication and provisional restorations and primarily enabled the use of the biogeneric copy in the software (CEREC inLab v4.0.2, Sirona Dental Systems GmbH, Bensheim, Germany) to generate the definitive restorations. Untouched canines and rearmost molars were strategic for the correlation between the waxed models and the preparations. Then the technician

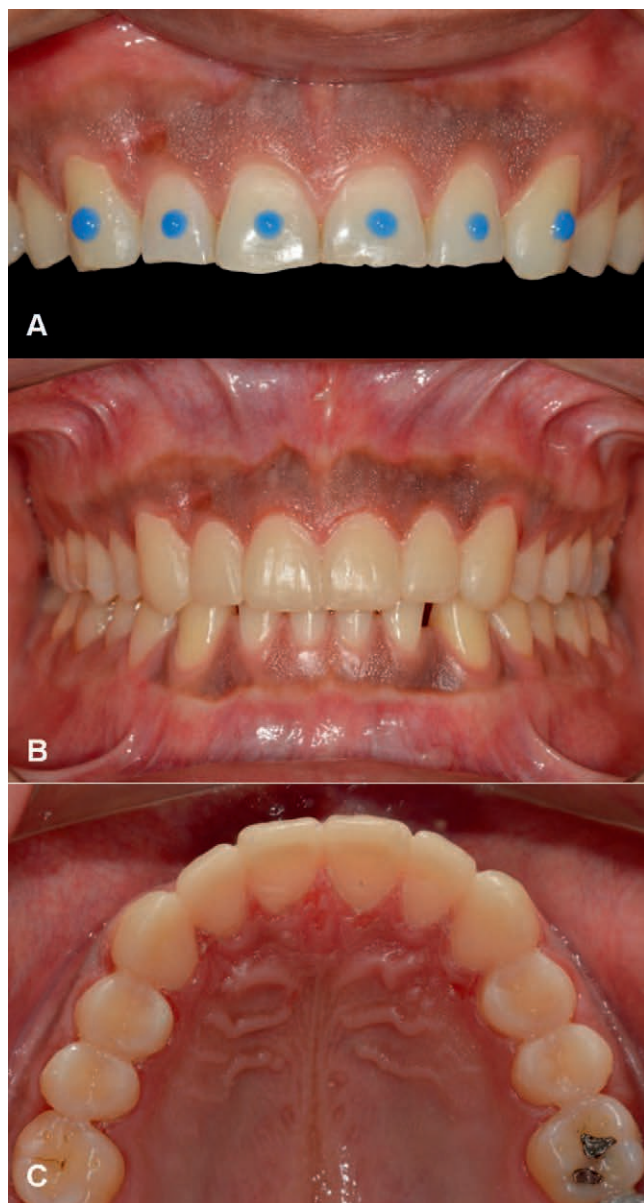


Figure 3. (A): Spot etching on unprepared enamel previous to mock-up fabrication. (B): Maxillary and mandibular mock-up (vertical dimension of occlusion increased by 1 mm). (C) Occlusal view of the posterior splinted provisional acrylic restorations.

completed the waxing of the canines, lower incisal edges, and lower third molars (Figure 2B,C). The waxed models were duplicated and poured again (Figure 2D,E).

At the next appointment, mock-up restorations were fabricated intraorally for esthetic and VDO evaluation. Multispot etching on unprepared enamel was carried out to increase the retention of provisionals in poly(methyl methacrylate) resin (Dencôr Lay, Clássico, São Paulo, Brazil) (Figure 3A). The

influence of the new VDO (+1 mm) in facial appearance as well as muscular comfort was evaluated for 15 days with splinted bonded provisional acrylic restorations (Figure 3B,C).¹⁹ On the patient's approval, the provisional restorations were used as a reduction guide for the preparations with tapered diamond rotary instruments (product code 850.31.023 FG; Brasseler, USA). Depth cuts were marked with a pencil. The remaining acrylic was removed with an instrument, and preparation was completed by reducing between the guide grooves and erasing the pencil marks (Figure 4A,B). The average occlusal clearance was 0.4 to 0.6 mm (central groove) and 1.0 to 1.3 mm (cusp tips), generating minimal space for ultrathin occlusal veneers. After preparation, a rubber dam was placed, and areas of exposed dentin were refreshed with a coarse diamond bur at a maximum of 1500 rpm. Immediate dentin sealing (IDS) was achieved using a three-step etch-and-rinse dentin bonding agent (OptiBond FL, Kerr Corp, Orange, CA, USA) (Figure 4C)¹³⁻¹⁵ according to the manufacturer's instructions. IDS was carried out without air thinning of the adhesive and with an additional light polymerization at 1000 mW/cm² (Valo, Ultradent Products Inc, South Jordan, UT, USA) for 10 seconds under an air barrier (K-Y Jelly, Johnson & Johnson, New Brunswick, NJ, USA) to reduce the oxygen inhibition layer. Excess adhesive resin and irregularities were then removed from the surrounding enamel by using a round diamond rotary cutting instrument (801.31.023 FG, Brasseler, USA) or tapered burs (850.31.023 FG, Brasseler, USA) at 1500 rpm (Figure 4D). Among the most reliable contemporary systems, OptiBond FL is especially indicated for the application of IDS because of its ability to form a consistent and uniform layer (about 80 μ m when placed over a slightly convex dentin surface) and its cohesiveness with the final luting composite.

The four posterior sextants were prepared and restored over a period of 15 days (two per week; maxillary arch first). In this regard, the additive stone replicas (without the canine waxing) were scanned first for correlation (Figure 5A) (Cerec AC Bluecam, Sirona Dental Systems) and then the preparations (Figure 5B). A preview of the restorative design was proposed after the correlation of the models in the software (InLab v4.0.2, Sirona Dental Systems) (Figure 5C). Restorations were made entirely in-house with the assistance of the design tools of the software (InLab v4.0.2) set in Biogeneric Copy. The design was proposed by correlating the

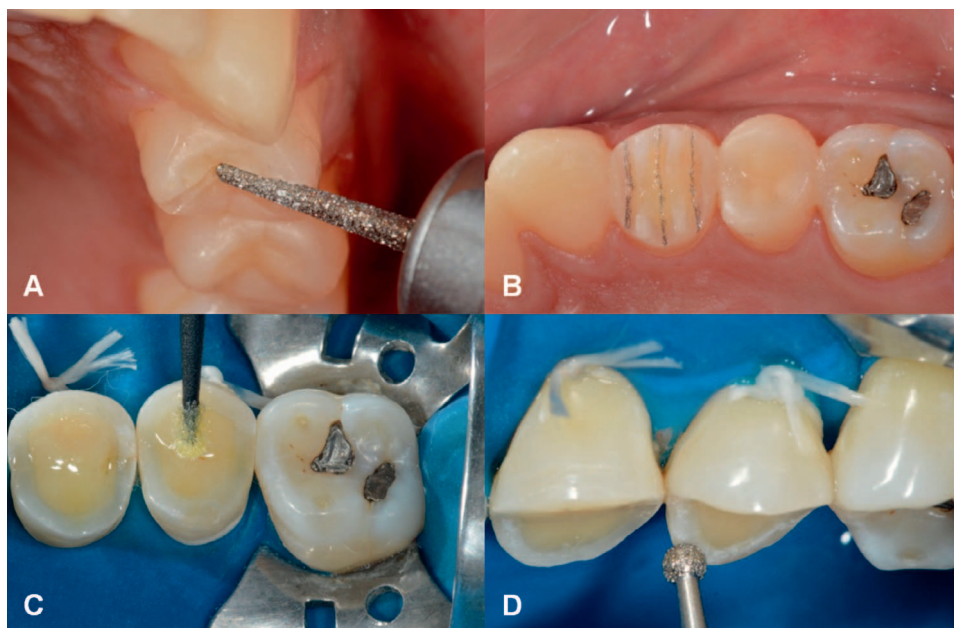


Figure 4. (A): Preparation driven by provisional restorations. (B): Depth cuts are marked with pencil, remaining acrylic is removed, and preparation is completed when pencil marks are erased. (C): Immediate dentin sealing with a three-step etch-and-rinse dentin bonding agent (Optibond FL, NOT AIR-THINNED). (D): Removal of excess adhesive from instrumented enamel.

preparations with the anatomy of the additive waxing. Minimum editing was needed to obtain precise margins and ideal contact strength. The ultrathin occlusal veneers were milled from lithium disilicate glass-ceramic blocks (A2 High Translucency e.max CAD). The sprue was preferably located at the nonworking cusps: lingual (inferior arch) and buccal (superior arch). In the same appointment, the restorations were visually inspected to detect eventual cracks and marginal chipping and, while in the precrystallized stage, tried in for occlusal contacts and general fit (Figure 6A).

Due to the nonretentive geometry of the preparation, provisionals were fabricated with some redundancy with respect to retention measures. The surrounding occlusal enamel was spot etched with 37.5% phosphoric acid (Ultra-Etch, Ultradent Products Inc, South Jordan, UT, USA) (Figure 6B) followed by coating protection of the previously sealed dentin surfaces with a water-soluble separating agent (Pro-V Coat, Bisco Dental Products, Schaumburg, IL, USA) (Figure 6C) in order to prevent potential resin bonding to the provisional poly(methyl methacrylate) resin (Dencôr Lay). The polyvinyl siloxane indexes (Platinum 85, Zhermack) were then loaded with the acrylic resin (Dencôr Lay) and precisely repositioned (Figure 6D). Provisionals were not retrieved, and excesses on the embrasures were purposely left in order to provide extra retention. Mandibular third molars were provisionalized during all restorative phases (although not

planned for restoration) in order to avoid early supraeruption, which could compromise the process of correlation. Restorations were crystallized (Programat P300, Ivoclar Vivadent) and characterized (stains mahogany, copper, and white IPS e.max CAD) and finally glazed.

At the following appointment, the ultrathin occlusal veneers were luted without the need for anesthesia. Provisionals were removed, and the preparations were inspected for any remaining adhesive resin/acrylic resin. The conditioning of the restorations included 10% hydrofluoric acid etching (Dentsply) for 20 seconds. After rinsing for 20 seconds, the restorations were subjected to postetching cleaning using 37.5% phosphoric acid (Ultra-Etch) under motion for one minute, followed by air-water spray cleaning for 30 seconds. After air-drying, intaglio surfaces were silanated (Monobond Plus, Ivoclar Vivadent) and heat dried at 68°C for around five minutes (Calset, AdDent Inc, Danbury, CT, USA).

Under rubber dam, tooth preparations were airborne-particle abraded with 50 μ m aluminum oxide at 0.2 MPa and etched for 30 seconds with 37.5% phosphoric acid (Ultra-Etch), rinsed, and dried. Both fitting surfaces, restoration and tooth, were coated with adhesive resin (Optibond FL, Bottle No. 2) and left unpolymerized until the preheated luting material (Z100, 3M ESPE) (preheated at 68°C with Calset, Adent) was applied to the tooth.²⁰ The restorations were seated (the entire sextant) followed by careful elimination of gross

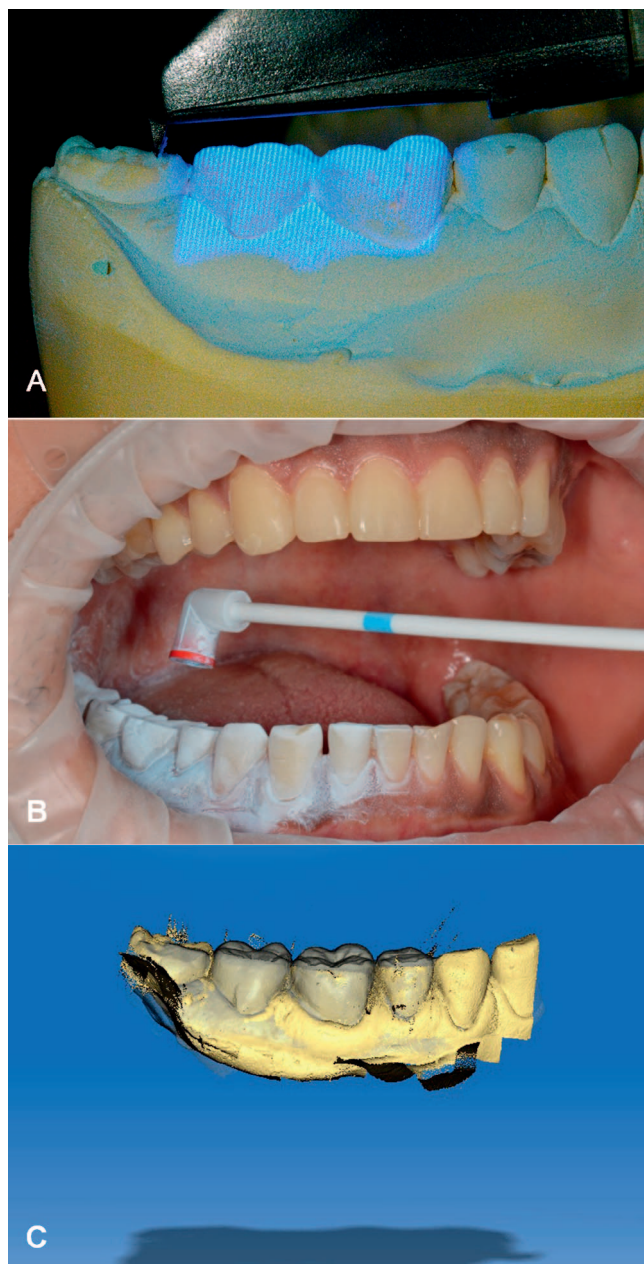


Figure 5. (A): Scanning of wax-up replica. (B): Powdering of prepared teeth. (C): Preview of the restorative design after the correlation of the models. Note the excellent overlap between the wax-up and prepared teeth.

excess composite resin and initial light polymerization.²¹ Each surface was exposed at an irradiance of 1000 mW/cm² (Valo, Ultradent) for 60 seconds (20 seconds per surface repeated three times). All margins were then covered with an air barrier (K-Y Jelly,) and light polymerized for 10 seconds. Occlusal adjustments were minor due to the adjustments in the precrystallized stage (Figure 7). The

margins were finished and polished at the following appointment first with silicon-impregnated rubber polishers (Jiffy, Ultradent) and, in case of minor horizontal discrepancy, with diamond ceramic polishers (W16Dg, W16Dmf, and W16D, EVE Diapol, EVE Ernst Vetter GmbH, Pforzheim, Germany). At this clinical stage, after MIP stabilization, the mandibular third molar provisionals were removed, allowing supraeruption and intercuspation of these teeth according to the Dahl principle.¹⁷

The anterior maxillary sextant was restored in the most conservative way: according to the bilaminar approach.¹⁸ The palatal surface of the six teeth was rebuilt with direct composite under rubber dam isolation (including clamp #212). Preparation was limited to the refreshing of the exposed dentin with a diamond bur at low speed (1500 rpm). After the application of a total-etch dental adhesive (Optibond FL), the marginal ridges, cingulum, and palatal fossa were recovered with freehand composite resin application (Empress Direct, Ivoclar Vivadent, Figure 8A). If ceramic had been chosen, additional preparation of the palatal surface would have been needed. In the following two appointments, anterior teeth were prepared (Figure 8B), and monolithic stained CAD-CAM lithium disilicate–reinforced glass ceramic (e.max CAD) was fabricated (Figure 8C) and luted (Variolink Veneer, Ivoclar Vivadent). Finally, the incisal edges of the mandibular anterior teeth were restored with direct composite resin (IPS Empress Direct).

DISCUSSION

It is notable how well patients presenting with moderate to severe loss of tooth structure due to biocorrosion react when minimally invasive reconstructive approaches are proposed to them. Both biological and economic costs are significantly lower compared to traditional and more aggressive options, making the decision-making process easier and the acceptance rate higher. The diagnostic wax-up and mock-up play a key role in this matter, helping both patient and clinician assess the physical aspects of the proposal as well as in the following technical steps of the work flow. Despite the fact that digital design along with the possibility of 3D printing is already available, the wax-up was delegated to a skilled technician and performed in the traditional additive way.

The option of using the mock-up for a period of time (in the present case, two weeks) provides a good level of predictability, mainly when the VDO is augmented. Based on the study of Abduo,¹⁹ the

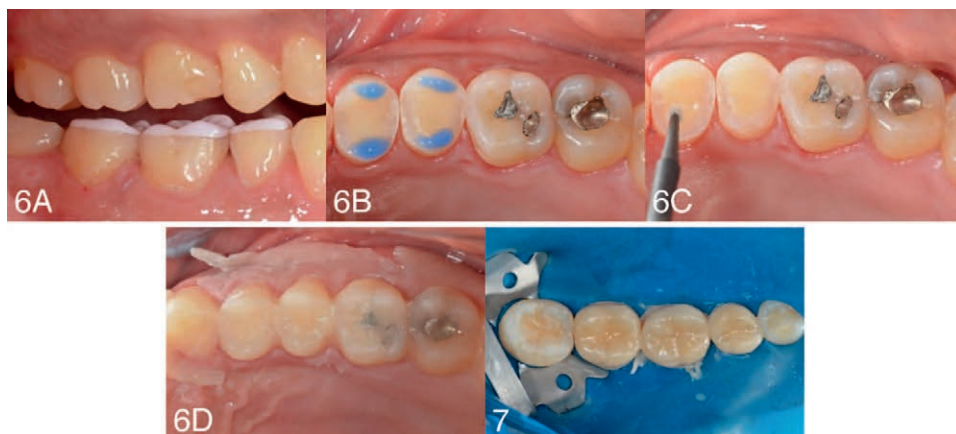


Figure 6. (A): Try-in of ultrathin occlusal veneers in blue phase. (B): Spot etching of prepared enamel with 37.5% phosphoric acid provides extra retention for provisional restorations. (C): Separating agent (Pro V Coat) for selective retention of provisional. (D): Provisional splinted restoration still showing the excesses to be removed.

Figure 7. Luting completed in mandibular right posterior sextant.

option of using the mock-up for two weeks provided a good level of acceptance with the VDO augmented in only 1 mm. Considering the two additional weeks to deliver the occlusal veneers, the total time of provisionalization was one month. Acrylic resin is the material of choice for provisionals because of its good fracture resistance compared to the bis-acryl resins, which is critical for ultrathin restorations. Additionally, one of its drawbacks (the high polymerization shrinkage) can be an advantage since it will help to keep the provisional locked. Having the confirmation of occlusal comfort, the same mock-up is used as a reference guide for an accurate preparation.

Regarding the bonding strategy, Optibond FL was the first choice for immediate dentin sealing. Its filler content allows for a more consistent and uniform layer compared to unfilled systems. This feature also decreases the chances of dentin exposure during cleaning and roughening procedures at the delivery appointment.¹⁵ It was also used along with the direct composite because of the intrinsic advantages of a fourth-generation system and also in order to simplify the process using one system for all purposes.

The choice of a preheated traditional light-polymerizing composite resin as a luting agent is supported by clinical²²⁻²⁴ and laboratory evidence²⁵ as well as by practical advantages: unlimited working time (compensating for the difficulty of positioning the occlusal veneers because of the lack of insertion path), less formation of interfacial voids because of the low viscosity,²⁶ ideal consistency for seating and excess removal, and better mechanical properties because of the higher filler content and higher degree of conversion.^{1,20} In addition, preheated composite reached a high degree of conversion

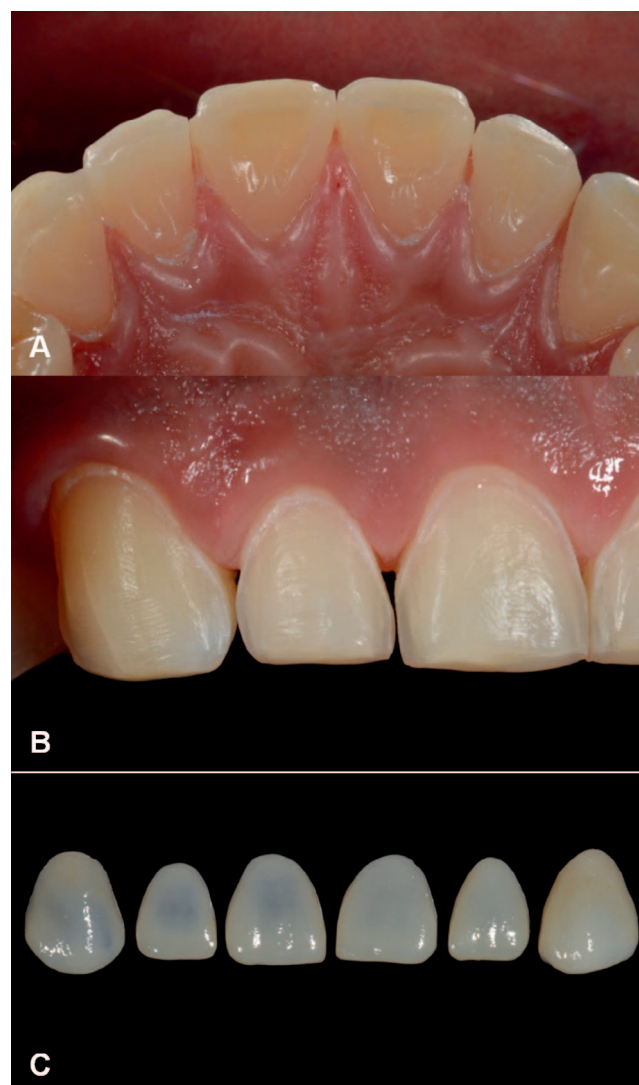


Figure 8. (A): Palatal direct veneers (freehand composite, Empress Direct). (B): Facial veneer preparations. (C): Lithium disilicate veneers after staining/glazing.

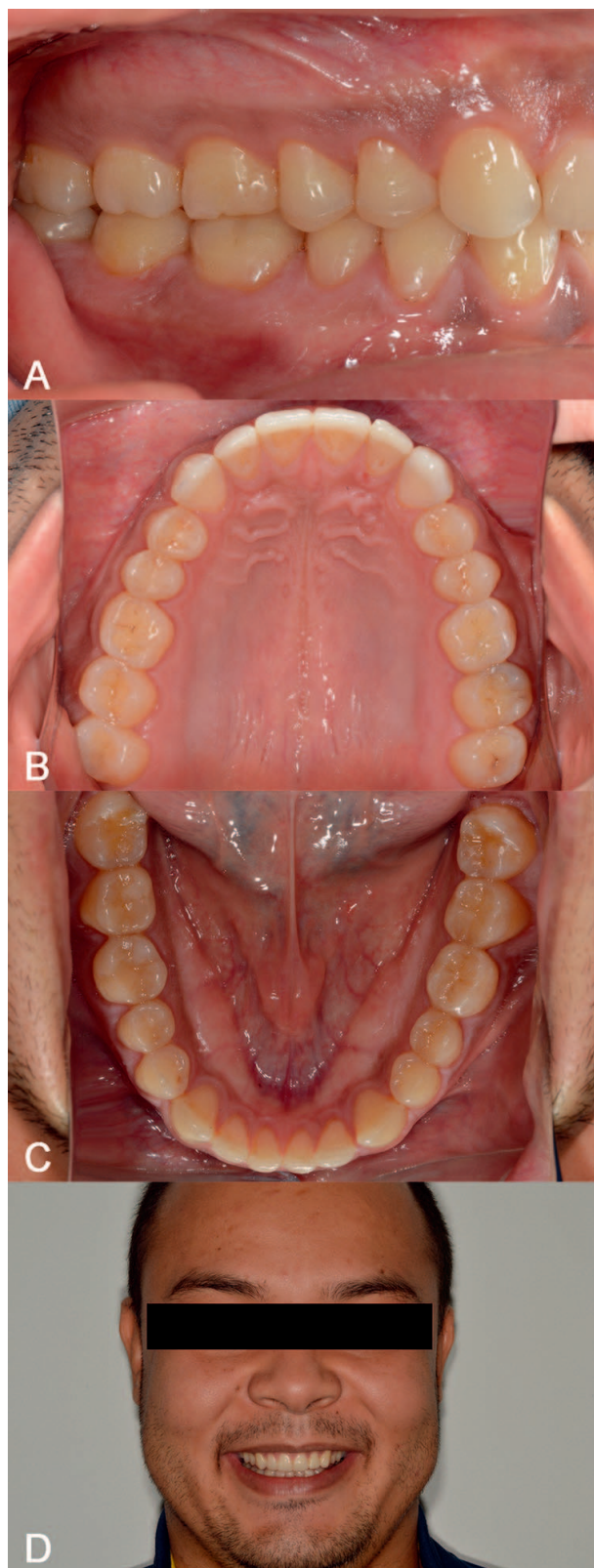


Figure 9. Right (A) sextant in occlusion. Occlusal views of the completed restorations (B,C). Patient's smile after treatment conclusion (D).

using low radiant exposures, between 50% to 70%, compared to composite polymerized at room temperature.²⁷

When investigating the material to fabricate ultrathin occlusal veneers, both ceramic and composites were able to meet the requirements for mechanics, function, and esthetics under an *in vitro* accelerated fatigue study⁵ that was validated by numeric simulation.⁶ However, CAD-CAM composite resins may provide better fracture resistance under extreme load conditions.⁵ In these patients, it is highly recommended to use an occlusal splint regardless of the restorative material. Further research is required to assess the clinical performance of those novel-design ultrathin occlusal veneers. For this reason, the case presented here is part of a comprehensive clinical trial ongoing at the Dental School of the Federal University of Rio de Janeiro. Patients affected by moderate to severe biocorrosion are being randomly treated with this novel design using two different CAD-CAM materials (composite resin and ceramic).

Clinical Follow-Up

The intraoral aspects of the patient can be observed before treatment (Figure 1A through 1C), after treatment completion (Figure 9A through 9E), and after 1.5 years of treatment (Figure 10A through 10D).

CONCLUSIONS

This case report demonstrates the feasibility of restoring the dental sequelae of moderate biocorrosion with ultrathin CAD-CAM ultrathin occlusal veneers and anterior bilaminar veneers. The treatment was successful due to the conservation of the dental tissue, esthetics, and effectiveness to restore dental wear caused by biocorrosion confirmed by the reevaluation after 1.5 years of clinical service, although it has been observed that addressing the etiologic factor along with the use of an occlusal splint (when associated to attrition) is of paramount importance.

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Figure 10. 1.5-year follow-up. (A): Restorations well integrated in the smile. (B) Palatal view of the anterior teeth. Note the subtle blending between composite - ceramic. (C-D): Lower posterior teeth (no signs of cracks or chipping in the restorations).

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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 Universidade Federal do Rio de Janeiro.

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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Home Dental Bleaching Monitored With Microelectronic Sensors to Record the Wearing Times of an Acetate Tray/Bleaching Product

CC Pavani • D Sundfeld • TC Schott • APM Bertoz • R Bigliazzi • RH Sundfeld

Clinical Relevance

Measuring/quantifying the wearing time of carbamide peroxide in acetate trays will better guide professionals to promptly identify and overcome problems of cooperation resulting from the inappropriate use of bleaching products and provide a more effective bleaching treatment.

SUMMARY

The present case study quantifies the clinical wearing time of an acetate tray/bleaching product during home dental bleaching. The patient was instructed to use a 10% carbamide peroxide gel for 6-8 hours each day for 20 days. The wearing time of the tray was measured by a microsensor from TheraMon microelectronic system (Sales Agency Gschladt, Hargelsberg, Austria) completely embedded in the tray so that the clinician would be informed as to the

precise time the tray was placed in the mouth. Evaluation of the daily printout during dental bleaching demonstrated that the patient did not correctly follow the clinician's instructions as to the wearing time of the tray. Recording the wearing time of dental bleaching agents can be beneficial for both dental professional and patient to obtain a more effective treatment according to the patient's needs and expectations.

INTRODUCTION

The carbamide peroxide bleaching product, poured in custom acetate trays during home dental bleach-

Caio Cesar Pavani, MDS student, Department of Restorative Dentistry, Araçatuba Dental School, São Paulo State University–UNESP, Araçatuba, SP, Brazil

Daniel Sundfeld, DDS, MDS, PhD, professor, Department of Restorative Dentistry and Prosthodontics, Ingá University Center–UNINGÁ, Maringá, PR, Brazil

Timm Cornelius Schott, DMD, professor, Department of Orthodontics and Orofacial Orthopedics, Eberhard Karls University, Tübingen, Germany

André Pinheiro de Magalhães Bertoz, DDS, MDS, PhD, professor, Department of Pediatric and Social Dentistry, Araçatuba Dental School, São Paulo State University–UNESP, Araçatuba, SP, Brazil

Renato Bigliazzi, DDS, MDS, PhD, professor, Department of Pediatric and Social Dentistry, Araçatuba Dental School, São Paulo State University–UNESP, Araçatuba, SP, Brazil

*Renato Herman Sundfeld, DDS, MDS, PhD, professor, Department of Restorative Dentistry, Araçatuba Dental School, São Paulo State University–UNESP, Araçatuba, SP, Brazil

*Corresponding author: Araçatuba Dental School, 1193 José Bonifácio St, Araçatuba, SP, Brazil; e-mail: sundfeld@foa.unesp.br

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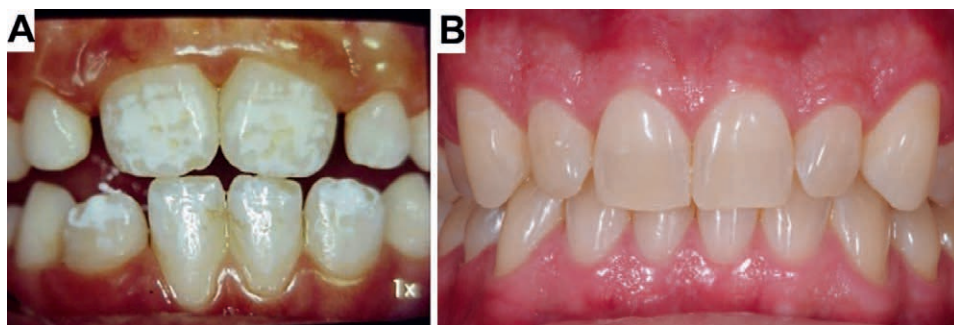


Figure 1. (A) In 1989, a 9-year-old boy showing white enamel stains of hard texture located on his maxillary and mandibular teeth removed by the enamel microabrasion technique. (B) Clinical condition before starting bleaching treatment.

ing, provides a significant margin of clinical success and good patient acceptability due to its easy handling; however, this bleaching treatment should be supervised by a dental professional.¹⁻⁷ This remarkable technique, introduced by Haywood and Heymann in 1989,⁶ requires the patient to use the bleaching product nightly for 6 to 8 hours for 6 weeks.⁶ However, patient cooperation, such as wearing time, will certainly influence the success of dental bleaching.

Recently, an electronic microsensor, TheraMon (TheraMon Microelectronic System; Sales Agency Gschladt, Hargelsberg, Austria. <http://www.english.thera-mon.com/the-product/index.html>),⁸⁻¹⁵ was developed to measure the wearing time of removable orthodontic appliances, verifying that patients do not always follow the clinician's instructions, thereby jeopardizing the final quality of the orthodontic treatment. Therefore, to provide a more effective and safe bleaching process, measuring the wearing time of the carbamide peroxide-containing trays will better guide professionals in promptly identifying and addressing concerns of cooperation. Based on the capability of the TheraMon electronic device,⁸⁻¹⁵ the aim of the present case study was to assess the level of commitment of a home dental bleaching patient by embedding a microsensor in the custom tray.

CASE REPORT

In 1989, a 9-year-old boy presented to the Clinic of Dentistry of Araçatuba School of Dentistry (São Paulo State University–UNESP) showing white enamel stains of a hard texture on the upper and lower teeth (Figure 1A). The enamel stains located on the maxillary central incisors and mandibular teeth were removed by the enamel microabrasion technique,^{1,16} except for the stains located on the lower right central incisor, which presented a deep white stain that needed to be restored with resin composite. In 2016, the patient aimed to bleach his teeth (Figure 1B). During clinical examination, the

patient did not reveal any contraindication to the treatment. Also, the patient reported no smoking habits and no history of a dental bleaching treatment. The bleaching was performed using 10% carbamide peroxide gel (Opalescence, Ultradent Products Inc, South Jordan, UT, USA). As this case report is not a clinical investigation, no institutional review board was necessary.

To begin the treatment, maxillary and mandibular alginate impressions were taken, soft vinyl mouth trays were fabricated, and an electronic microsensor, TheraMon (TheraMon), was embedded in the labial region of the acetate tray in order to evaluate the wearing time of the trays (Figure 2). The trays and four bleaching gel tubes were given to the patient, who was instructed to place a small drop of the bleaching gel into each tooth section of the tray, from first molar to first molar, and advised to use the tray at night for 6 to 8 hours per day (h/d), during sleep. All tubes of the bleaching gel were used during the bleaching treatment, which accomplished the patient's expectations. The size and appearance of the embedded TheraMon did not affect patient comfort during bleaching (Figure 3).

The microsensor measures the temperature of the mouth with an accuracy of $\pm 0.1^{\circ}\text{C}$, and the temperature is recorded every 15 minutes. The collected data were transferred using a wireless connection between the functional unit and a reading station (Figure 4) coupled to a USB connection. TheraMon software is able to read and interpret data, providing a daily diagram of the wearing time of the tray at each treatment consultation interval. The wearing time of the trays was considered the dental bleaching time, as the patient was instructed to always pour the bleaching gel when the tray was positioned on both dental arches.

Figures 5 and 6 indicate that, during the 22 days of the bleaching period, corresponding from the beginning to the end of the treatment, the mean wearing time of the trays were 6.6 and 6.58 h/d for the upper



Figure 2. Microsensor TheraMon embedded in the labial region of the acetate custom tray.

Figure 3. Maxillary and mandibular acetate trays containing the microsensor TheraMon placed on teeth (A and B).

and lower arches, respectively. It was observed that the patient used the trays containing the bleaching gel for a shorter period of time than instructed on the first, second, fifth, and ninth day of treatment. In addition, an additional time of 1 h/d was recorded on the eighth and 21st day of treatment, in both arches. It was also possible to point out that even when guiding the patient about the wearing time per day, there were variations in the wearing time during treatment, ranging from 2.25 to 9.25 h/d. This result demonstrated that the patient did not correctly follow the recommendations for the daily wearing time of the bleaching product on both dental arches. However,



Figure 4. Microsensor TheraMon read-out station.

er, during treatment, the patient used the trays containing bleaching product in both arches at the same time he had been instructed.

Dental color assessment was performed before and after the bleaching treatment using a digital spectrophotometer (Vita Easyshade Advance, Vita Zahnfabrik, Bad Säckingen, Germany) (Figure 7). The junction of the middle and cervical third of teeth 6 to 11 and 22 to 27 was used for color determination; therefore, an opening on the labial surface, which served as a guide for placing the Easyshade tip before and after bleaching, was made on each tooth position of the tray. According to the data obtained from the Easyshade analysis, the color of the teeth before and after bleaching was determined following the classic Vita shade guide parameter (Tables 1 and 2). Figure 8 depicts the final result of the bleaching treatment. No gingival irritation or sensitivity was observed, and the patient reported satisfaction with the bleaching treatment.

Fig 5

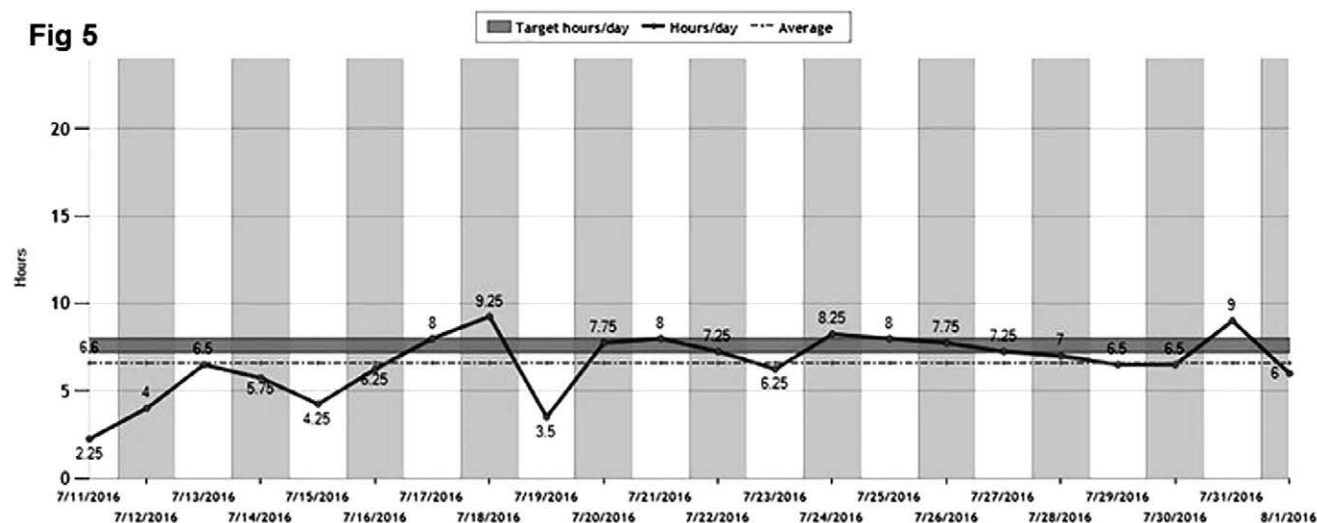


Fig 6

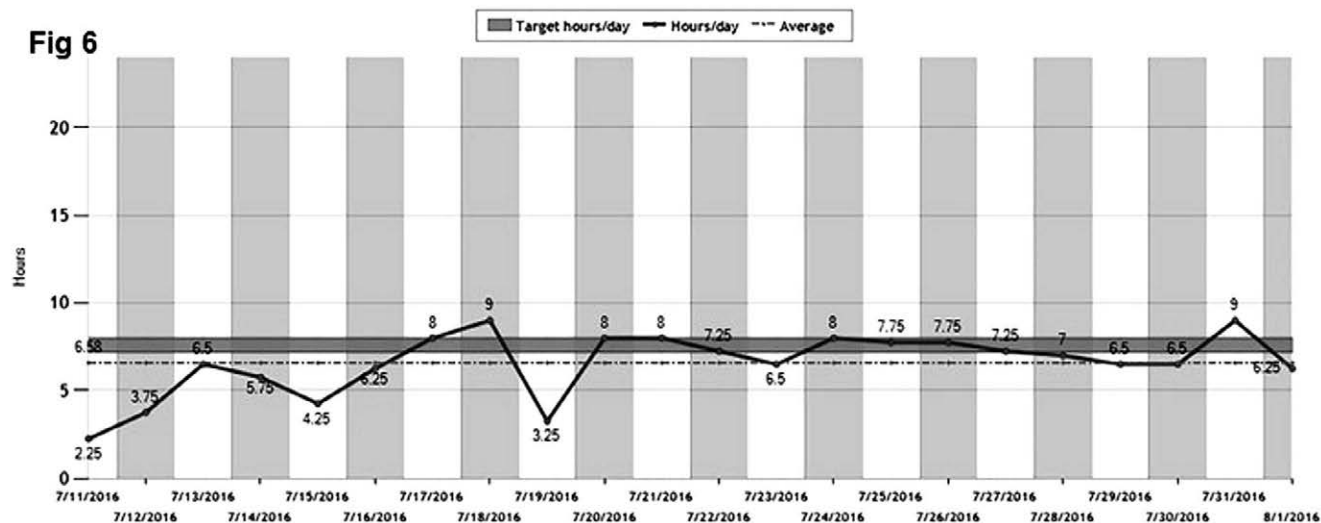


Figure 5. Diagram evaluating daily tooth bleaching time (time recommended: 6-8 h/d) in the upper arch measured by the TheraMon system microsensor.

Figure 6. Diagram evaluating daily tooth bleaching time (time recommended: 6-8 h/d) in the lower arch measured by the TheraMon system microsensor.

DISCUSSION

The present clinical case report demonstrated the precise wearing times of acetate trays measured by an electronic microsensor device to evaluate the patient's compliance during bleaching treatment.

A significant factor that must be raised for obtaining bleaching effectiveness is related to the degree of patient commitment, especially about the daily wearing time of the trays containing the bleaching product. Several wearing times of dental bleaching products have been reported, from 2 to 8 h/d;^{6,7,17} without assuming, however, whether the patient actually used the bleaching product for the

time recommended by the professional. Since the bleaching product containing carbamide peroxide is applied to the teeth by the patients themselves, the wearing time of the trays is under their control. Previous orthodontic studies⁸⁻¹⁵ have demonstrated the use of the electronic microsensor (TheraMon), which identifies the temperature changes within the oral cavity, making it possible to assess the wearing time of removable orthodontic appliances.^{11,12} Based on the described assessments, the aim of the present study was to record the daily wearing time of the carbamide peroxide gel during dental bleaching using the microsensor.

Table 1: Color Analysis of Teeth Submitted to Dental Bleaching Following the Classical Vita Shade Guide^a and Its Correlation With the Score Corresponding to Value Arrangement^b

Tooth No	Initial Color/Value	Final Color/Value
6	B3/11	A1/2
7	A2/5	A1/2
8	A1/2	B1/1
9	A1/2	B1/1
10	B2/3	B1/1
11	A2/5	A1/2
22	A2/5	B1/1
23	A2/5	A1/2
24	A1/2	B2/3
25	A1/2	B1/1
26	B2/3	B1/1
27	A2/5	A1/2

^a Obtained from the Easyshade spectrophotometer assessment (Vita Easyshade Advance, Vita Zahnfabrik, Bad Säckingen, Germany).
^b Vita Classical, Vita Zahnfabrik, Bad Säckingen, Germany.

The TheraMon is able to monitor the usage time of the trays by recording the temperature of the surrounding area every 15 minutes. Temperatures between 32°C and 36°C count as wear times. The sensitivity of the temperature module makes it extremely difficult to record false data, since the temperature of the trays being used correctly in the oral cavity will move slightly up and down, especially during sleep when body temperature falls at night and increases in the morning. To alert the practice to the “suspect” use time, the software may highlight any abnormal lack of temperature fluctuation or “atypical” temperature change in the oral cavity, with slightly suspect periods of time indicated in yellow and periods strongly suspect in red.^{9,10}

It was found that the wearing times of the trays can be recorded during the bleaching treatment (Figures 5 and 6). This technology permits precise measurement and recording of the daily wearing times of the trays by the patients and also provides additional data of mean times by the software at any time. It was found that the patient had not followed the instructions about the daily wearing time (6-8 h/d) of the bleaching product. During the 22 days of treatment, the mean daily wearing time of the trays was 6.6 (from 2.5 to 9.25 h/d) and 6.58 h/d (from 2.5 to 9 h/d) for the upper

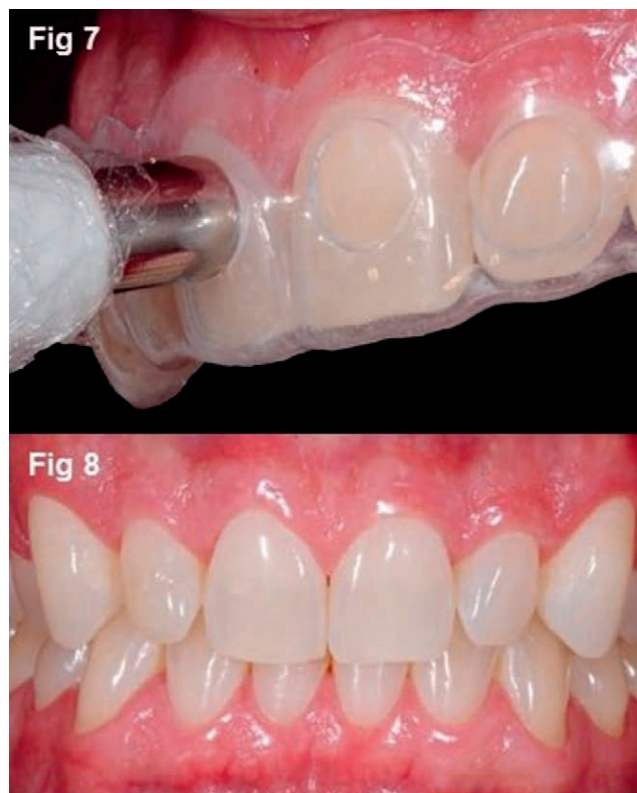


Figure 7. Dental color evaluation using Easyshade spectrophotometer.

Figure 8. Twenty-seven years after removal of white stains on the maxillary and mandibular teeth and 8 months after dental bleaching.

and lower arches, respectively. Schott and Ludwig in 2014¹³ assessed the wearing time of orthodontic appliances and observed that the patients compensated for the fewer hours used in one day by using it for more hours than recommended on succeeding days. It is noteworthy that the patient consented to embedding the microsensor in the trays and knew that monitoring would be accomplished during treatment.

The mean wearing times found in this study allowed us to suppose that if the patient had used the trays with gel for the recommended time per day, a satisfactory bleaching could have been obtained in a shorter time.^{7,17} We noted that the patient appeared to be anxious and usually did not sleep quietly and for a predetermined time, as was reported during anamnesis. This condition may have influenced the adequate wearing time of the trays recommended by the clinician. No tooth sensitivity was reported during or after treatment.

Table 2: Vita Classical Shade Guide Arranged by Value Scale^a

Shade	B1	A1	B2	D2	A2	C1	C2	D4	A3	D3	B3	A3,5	B4	C3	A4	C4
Value	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16

^a Vita Classical, Vita Zahnfabrik, Bad Säckingen, Germany.

The present case study agrees with Schäfer and others¹⁴ who quantified the wearing times of a removable appliance during active orthodontic treatment using the TheraMon, confirming that patients contribute to the success of treatment. It is also necessary to modify the usual treatment plan to avoid treatment failures. Therefore, using the microsensor can be beneficial to both clinicians and patients to obtain more effective treatment according to the patient's needs and expectations. Likewise, it has been mentioned that this commercial sensor, besides being inexpensive (about \$30 in the United States and €28.5 in Europe), can be inserted into the removable appliance by a dental technician as part of routine practice.¹⁴ Use of the TheraMon device will enable researchers to more accurately measure patients' wearing time and may help in identifying optimal times necessary for satisfactory tooth whitening. Further, clinical studies with shorter bleaching times are essential for future research using the TheraMon device to determine the ideal bleaching time.

CONCLUSION

Based on this clinical case report, it can be concluded that the wearing time of the carbamide peroxide bleaching product can be monitored during bleaching treatment by the microsensor TheraMon embedded in the custom acetate tray.

Regulatory Statement

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

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, or company presented in this article.

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Effectiveness of In-office Hydrogen Peroxide With Two Different Protocols: A Two-center Randomized Clinical Trial

IEB Martins • S Onofre • N Franco • LM Martins • A Montenegro
LA Arana-Gordillo • A Reis • AD Loguercio • LM da Silva

Clinical Relevance

When an alkaline and desensitizing-containing in-office gel is used, it is recommended to be applied for one 40-minute application without gel refreshing.

SUMMARY

Objectives: The aim of this study was to compare the bleaching efficacy and tooth sensitivity (TS) of a 38% hydrogen peroxide bleaching agent used for in-office bleaching, applied under different time protocols: a 40-minute application or two 20-minute applications.

Methods and Materials: Forty-four patients from Brazil and Colombia, with right superior canines darker than C2, were selected for this

multicenter, single-blind, randomized trial. The teeth were bleached in two sessions, with a one-week interval between them, in a split-mouth design. The bleaching agent was applied in two 20-minute (2×20) applications or one 40-minute (1×40) application in each session according to the manufacturer's instructions. The color changes were evaluated by using subjective (Vita Classical and Vita Bleachedguide) and objective (Easysshade Spectrophotometer) methods at baseline and 30 days after the second session. Tooth sensitivity was recorded up to 48 hours with a 0-10 visual analog scale. Also, the pH values during

Izabelly EB Martins, DDS, MS, Department of Restorative Dentistry, School of Dentistry, Federal University of Amazonas, Manaus, Brazil

Stefania Onofre, DDS, School of Dentistry, University of Santiago de Cali, Cali, Colombia

Natalia Franco, DDS, School of Dentistry, University of Santiago de Cali, Cali, Colombia

Leandro de Moura Martins, DDS, Ms, PhD, Prosthetics, Bauru Dental School, Bauru, Brazil

Andres Montenegro, DDS, MS, Ponta Grossa State University, Ponta Grossa, Brazil

Luis Alfonso Arana-Gordillo, DDS, MS, PhD, Department of Restorative Dentistry, School of Dentistry, University of Santiago de Cali, Cali, Colombia

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

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

Luciana Mendonça da Silva, DDS, PhD, Federal University of Amazonas, Manaus, Brazil

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

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the application of bleaching were recorded. Color change in shade guide units and ΔE were analyzed by using the Student *t*-test ($\alpha=0.05$). The absolute risk and intensity of TS were evaluated with the McNemar test, the Wilcoxon signed-rank test, and the Friedman test, respectively ($\alpha=0.05$).

Results: Significant whitening was observed in both groups after 30 days of clinical evaluation. The use of a 40-minute application did not significantly influence the absolute risk of TS (68%, 95% confidence interval [CI] = 53-80) as well as the intensity of TS compared with the acid bleaching gel (absolute risk of 82%, 95% CI = 68-91). The pH values did not differ significantly between groups and at the different assessment periods ($p=0.42$).

Conclusion: The use of a 40-minute in-office bleaching agent gel application produced the same whitening degree and TS that the two 20-minute bleaching agent applications did. The former preferably should be applied because one 40-minute application does not require gel refreshing.

INTRODUCTION

Dental treatment patterns have changed due to patients' interest in dental esthetics, and more emphasis has been placed on having a beautiful smile.¹ When dealing with discolored teeth, dental bleaching should be the first choice for treatment, as it is an effective and conservative approach² when compared with other treatment modalities, such as veneers, crowns, or composite bonding.^{3,4} Carbamide peroxide at 10% is most used as a standard product for at-home bleaching.⁴ However, high concentrations of hydrogen peroxide (HP), ranging from 25% to 40%, have been used in office to achieve faster bleaching effectiveness.^{5,6}

In-office bleaching with high concentration HP provides visible results after a single session, thus increasing the acceptance of the in-office protocol.^{7,8} Additionally, it is a treatment alternative for patients who do not feel comfortable using a bleaching tray at home and who require faster results. It may also be employed in some patients who require close attention due to the presence of tissue recession or nonrestored cervical lesions.³

The wide range of in-office bleaching products in the dental market makes it difficult to choose one. Considerable variation exists in the products' active

concentrations of HP,^{5,6} additives, pH variations,⁹⁻¹¹ and application modes.

With regard to application modes, the results from clinical studies that compared the bleaching efficacy of a single 45-minute application and three 15-minute applications are not in agreement in terms of color change and risk and intensity of tooth sensitivity (TS).^{7,12-14} For instance, Reis and others⁷ concluded that a single application of a 35% HP gel for 45 minutes decreased bleaching efficacy and slightly increased overall patient sensitivity compared with three 15-minute applications. On the other hand, Kossatz and others¹⁴ generated the opposite findings. The authors concluded that subjects who received three 15-minute applications each had more TS than did those who received a single 45-minute application each. The variations in compositions, mainly pH and additives, among the different brands of HP gels and the decreased pH stability of some products may explain such results.^{7,12-15}

Therefore, conducting randomized clinical trials that investigate the effect of a prolonged application time on the efficacy, risk, and intensity of TS for other bleaching gel brands is still required. Therefore, the aim of this single-blind, randomized controlled study was to evaluate the color change, risk, and intensity of TS of an in-office bleaching gel applied in a single 40-minute application or two 20-minute applications. We also investigated the *in vivo* pH stability of the gel for both application modes.

METHODS

Protocol Registration

This study was registered at the Brazilian registry of clinical trials under the identification number REBEC:RBR-3h6n6c.

Study Designs, Settings, and Locations of Data Collection

This was a randomized, single-blind (evaluators), split-mouth and equivalent multicenter trial with an equal allocation rate between groups. This study took place within the dental clinics of the UFAM and USC from September 2015 to February 2016.

Recruitment

The participants who took part in this study were those who sought dental treatment at the dental clinics at the universities. At this time, the participants were examined in a dental chair after dental

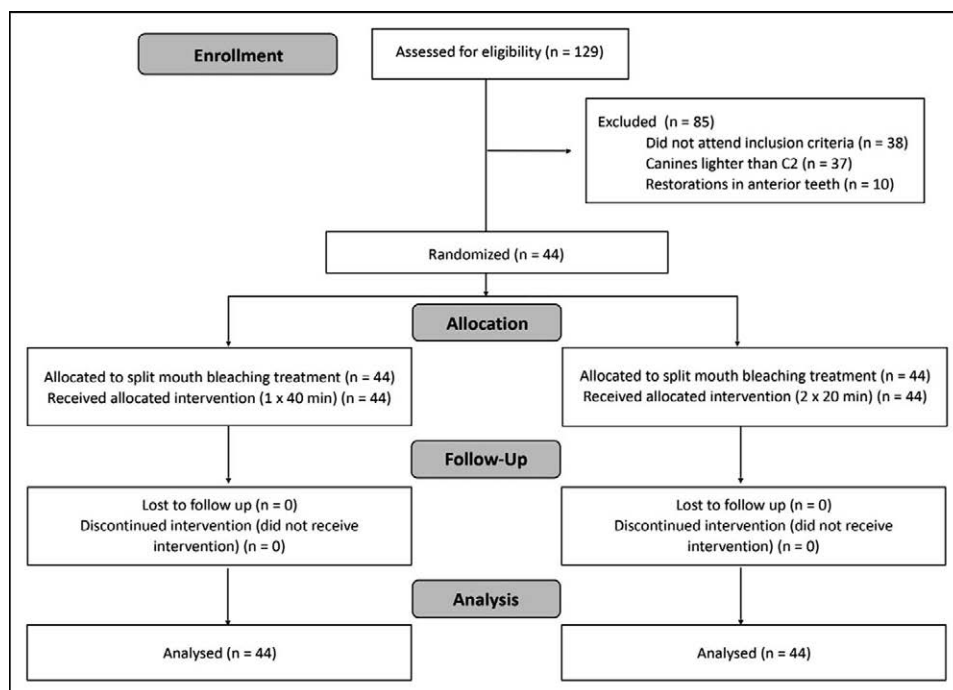


Figure 1. Flow diagram of the clinical trial, including detailed information on the participants.

prophylaxis with pumice and water to check whether they met the study's eligibility criteria.

Eligibility Criteria

To be included in this study, participants had to be age 18 years or older and be in good general and oral health. Participants had to have canines that were at least shade C2 or darker during assessments with the value-oriented shade guide (VITA classical, VITA Lumin, VITA Zahnfabrik, Bad Sackingen, Germany), and all anterior teeth without restorations. Participants with anterior teeth presenting noncarious cervical lesions, full crowns, or veneers; gingival recession; endodontically treated teeth; internal tooth discoloration; moderate to severe fluorosis; or spontaneous tooth pain were not included in the study. Additionally, pregnant or lactating women were also excluded from this study.

Sample-Size Calculation

The sample-size calculation was done for the color change, the primary outcome, measured with the spectrophotometer (ΔE). If no difference was found between the standard and experimental treatments, then 44 patients were required to be 90% sure that the limits of a two-sided 90% confidence interval would exclude a difference in means of three units in the ΔE values. Each center recruited 22 participants for the study (Figure 1).

Random Sequence, Allocation Concealment, and Blinding

A third operator, not involved in the research protocol, conducted the randomization procedure by using computer-generated tables. We used blocked randomization (block sizes of two and four) with an equal allocation ratio (www.sealedenvelope.com). The randomization sequence was placed in sequentially numbered, opaque, and sealed envelopes.

Once the participant was eligible for the procedure and completed all baseline assessments, the side of the patient's arch where treatment was applied first was decided by tossing a coin. At this time, the operator could open the envelope to reveal the group where the experiment would be conducted first. Neither the participant nor the operator knew the group allocation before this stage. The other treatment was applied on the patient's second side.

Participants and operators could not be blinded to the study groups, as they could easily identify the side in which the material had been refreshed once. However, the evaluator who performed the color assessments and the statistician was blinded to the treatments.

Study Intervention

The gingival tissue was isolated with a light-cured resin dam (Opal Dam, Ultradent Products Inc, South Jordan, UT, USA). The 38% HP gel Opalescence

Table 1: Products, Composition, and Application Regimens	
Products and Composition	Application Regimen for Both Groups
Opalescence Boost Gel: 38% Hydrogen peroxide, 20% water and desensitizing agents (3% potassium nitrate and 1.1% fluoride)	1. Dry teeth and apply Opal Dam to dental arch slightly overlapping enamel (building the barrier 4-6 mm high and 1.5-2.0 mm thick) and interproximal spaces. 2. Light cure Opal Dam for 20 s per arch using a scanning motion. Carefully check the resin cure with an instrument. 3. Attach both syringes before mixing. Press in the plunger of the red syringe, pushing all the contents into the clear syringe. Forcefully press the small clear stem completely into the larger clear stem. Then press the clear plunger completely into the red syringe. To activate, press the chemical from the red syringe into the clear syringe with thumbs. Reverse action and mix a minimum of 25 times on each side. 4. Press all mixed gel into the red syringe. Separate the two syringes and attach the Micro 20ga FX tip onto the red syringe. 5. Apply a 0.5-1.0 mm thick layer of Opalescence Boost to the labial surface of the tooth and slightly onto the incisal/occlusal surfaces. Group 2 × 20 min^a 5.1 Leave gel on for 20 min. 5.2 Suction off using a surgical aspirator tip. Do not use water. 5.3 Repeat gel application for more 20 min (40 min total). Group 1 × 40 min Leave gel on for 40 minutes undisturbed 6. After the last application, suction all the gel off, then wash and apply suction. 7. Remove Gingival Barrier by lifting it from one end.
^a According to the manufacturer's indications.	

Boost (Ultradent Products Inc) was applied in a single 40-minute application or in two 20-minute applications in all maxillary incisors, canines, and premolars of the same patient following the description of each group (Table 1).

After seven days, this procedure was repeated using the same protocol. All participants were instructed to brush their teeth at least three times a day using a fluoridated toothpaste (Colgate, Colgate-Palmolive, São Paulo, Brazil).

Color Evaluation

Color was recorded before the bleaching procedure, seven days later, and 30 days after the end of the bleaching treatment. For this purpose, we used an objective (Easyshade spectrophotometer, Vident, Brea, CA, USA) and a subjective instrument (value-oriented shade guide VITA classical). Color evaluation was completed in a room under artificial lightning conditions without interference from outside light. For both devices, color was checked at the middle third of the canine.

For calibration, the color assessments of 10 patients were conducted in both study centers three times with an interval of three days between times to check the intra- and interagreement. The same person, who visited both centers, performed the calibration process and coordinated the administration of the study. The operator was considered calibrated only when he could get a weighted kappa of 80% in two consecutive readings of the same teeth in 10 different patients.

For the objective shade evaluation, an impression of the maxillary arch with high-putty silicon paste (Clonage, Nova DFL, Rio de Janeiro, Brazil) was taken, and a window on the labial surface of the silicon guide was created by using a metal device with a 6-mm radius to standardize the area for color evaluation with the spectrophotometer. Color was determined using the parameters of the digital spectrophotometer, on which the following values were indicated: L*, a*, and b*, where L* represented luminosity (the value from 0 [black] to 100 [white]), and where a* and b* represented the color along the red-green axis and the color along the yellow-blue axis, respectively.

The difference between baseline and each recall period (ΔE^*) was calculated by using the following formula: $\Delta E^* = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2}$. For the subjective evaluation, the 16 tabs of the shade guide (VITA classical, VITA Zahnfabrik) were arranged from lightest to darkest as follows: B1, A1, B2, D2, A2, C1, C2, D4, A3, D3, B3, A3.5, B4, C3, A4, C4. Color changes were calculated from the beginning of the active phase through to the individual recall times by calculating the change in the number of shade guide units ($\Delta SGUs$), which occurred toward the lighter end of the value-oriented list of shade tabs.

Two examiners at each center, blinded to the allocation assignment, scheduled these patients for bleaching and evaluated their teeth against the shade guide at the different time assessments. In

the event of disagreements between the examiners during shade evaluation, a consensus was reached.

Tooth Sensitivity Evaluation

Patients were asked to record their perceptions of TS during the first and second bleaching sessions using a 0-10 visual analog scale (VAS) in three different assessment periods: during treatment and up to 1 hour after bleaching, from 1 hour to 24 hours after bleaching, and from 24 hours to 48 hours after bleaching.

This VAS scale is a 10-cm horizontal line with scores of 0 and 10 at their ends, where 0 = no sensitivity and 10 = severe sensitivity. The patient had to mark the intensity of the TS with a vertical line across the horizontal line of the scale. Then, the distance in millimeters from the zero ends was measured with the aid of a millimeter ruler.

If the patient scored zero (no sensitivity) during all time assessments from both bleaching sessions, this patient was insensitive to the bleaching protocol. In all other circumstances, the patients were sensitive to the bleaching procedure. This dichotomization allowed us to calculate the absolute risk of TS, or the percentage of patients who reported TS at least once during treatment. As two bleaching sessions were performed, the worst VAS score obtained was considered for statistical purposes.

In vivo pH Analysis

A pH meter with a 6-mm circular and flat surface pH electrode (Extech pH100: ExStik pH Meter; Extech Instruments, Nashua, NH, USA) was positioned directly on the middle tooth surfaces of canines, lateral incisors, and central incisors in both of the patient's arches and held in position until the pH was stabilized on the screen. As the pH electrode is very sensitive, it was possible to make three measurements for each tooth. For both application protocols (two 20-minute applications or a single 40-minute application), pH was registered every 20 minutes.

Statistical Analysis

The analysis followed the intention-to-treat protocol and involved all of the participants who were randomly assigned. The statistician was also blinded to the study groups. The color change (Δ SGU and Δ E) between the baseline versus 30 days was calculated for each group. The Δ E and Δ SGU data from both groups were subjected to a paired Student *t*-test.

Table 2: Baseline Characteristics of the Participants Included in This Clinical Trial

Characteristics	2 × 20 Min	1 × 40 Min
Age (mean ± SD, y)	27.5 ± 6.2	26.1 ± 5.5
Baseline color (mean ± SD, SGUs)	12.3 ± 2.3	11.7 ± 2.1
Abbreviations: SD = standard deviation; SGU = shade guide units Vita Classical.		

The absolute risk of TS of both groups was compared using the McNemar test ($\alpha=0.05$). The relative risk and the confidence interval for the effect size were calculated. The comparison of the TS intensity of the two groups at two different assessment points was performed using the Wilcoxon signed-rank test. Comparisons between times within each group were performed using the Friedman test. Data from the pH values were compared using a two-way repeated measures analysis of variance.

In all statistical tests, the significance level was 5%. We performed all analyses by using the software SigmaPlot version 11.0 (Systat Software, San Jose, CA, USA).

RESULTS

A total of 129 participants were examined; 44 participants were selected (Figure 1). Patients were excluded mainly due to the presence of anterior restorations and color (shades lighter than C2). The mean age (years) of the participants and the baseline SGUs are described in Table 2. One can observe comparable data among treatment groups. None of the patients discontinued the intervention. No medication and/or desensitizer were necessary to prescribe/apply in the participants from this study for the relief of bleaching-induced TS. We did not observe statistical differences between the study centers for any of the outcomes ($p>0.05$; data not shown). Therefore, the data from these centers were merged for statistical evaluation.

Color Change

After 30 days, whitening of approximately 9 to 10 SGUs and a variation of approximately eight units in Δ E were detected for the 2×20 and 1×40 groups (Table 3). No statistically significant difference was observed between the study groups.

Tooth Sensitivity

The absolute risks of TS were 82% and 68%, respectively, with no significant difference observed

Table 3: Color Change in Shade Guide Units (Δ SGUs for Vita Classical) and Δ E (Means $\pm$ Standard Deviations) Between Baseline Versus 30 Days After Bleaching for the Two Treatment Groups*			
Color Evaluation Tools	2 $\times$ 20 Min	1 $\times$ 40 Min	p-Value*
Δ SGU	9.8 $\pm$ 2.8	10.4 $\pm$ 2.6	0.68
Δ E	8.9 $\pm$ 3.2	8.2 $\pm$ 3.0	0.12
* Paired Student t-test.			

between them ($p=0.27$; Table 4). The TS intensity of both bleaching protocols was statistically similar ($p>0.42$). The overall TS intensity at the different assessment points (Table 5) showed that most of the TS complaints occurred within the first 24 hours after bleaching.

pH Analysis

The pH values did not differ significantly between groups and at the different assessment periods ($p=0.42$; Table 6).

DISCUSSION

The results of this study indicated that both in-office bleaching protocols showed significant whitening after two bleaching sessions. The application of a 38% HP gel for 40 minutes, without being refreshed, maintained the bleaching efficacy compared with the two 20-minute applications that the manufacturer recommended.

HP is usually applied and left undisturbed on the tooth surface for up to 15 to 20 minutes, and then manufacturers recommend gel removal and reapplication twice or even five times according to the bleaching gel brand. This recommendation is probably based on the assumption that after 15 to 20 minutes, no HP will be available for the oxidization of the organic content of dentin.

Table 4: <i>Comparison of the Number of Patients Who Experienced Tooth Sensitivity at Least Once During the Bleaching Regimen in Both Groups Along With Absolute Risk and Risk Ratio*</i>				
Treatments	Number of Participants With Tooth Sensitivity		Absolute risk* (95% Confidence Interval)	Risk Ratio (95% Confidence Interval)
	Yes	No		
2 × 20 min	36	08	82 (68-91)	0.82 (0.63-1.08)
1 × 40 min	30	14	68 (53-80)	
* McNemar test (p=0.27).				

Table 5: Tooth Sensitivity Intensity (Means $\pm$ Standard Deviations) at the Different Assessment Points for Both Study Groups			
Time Assessments	Visual Analog Scale		p-Value*
	2 $\times$ 20 Min	1 $\times$ 40 Min	
Up to 1 h	1.3 $\pm$ 1.9 A**	1.0 $\pm$ 1.8 a***	0.54
1 h to 24 h	1.1 $\pm$ 2.1 A	0.9 $\pm$ 1.8 a	0.72
24 h to 48 h	0.3 $\pm$ 0.9 B	0.3 $\pm$ 1.0 b	0.91
* Wilcoxon signed rank test ($p=0.42$). ** Friedman test ($p=0.01$); *** Friedman test ($p=0.03$). Within each column, means identified with the same letters are statistically similar.			

HP-based products for at-home bleaching release most of their active HP within 60 to 75 minutes.^{16,17} After application of 3% HP gel for 20 minutes in a bleaching tray, authors showed that 50% of the HP was still available.¹⁶ Similar findings were observed in another study that reported that around 60% of the active ingredient was still available after 20 minutes for a 7.5% HP gel.¹⁷

Although we lack data about the degradation kinetics of a 35% HP gel, one may assume that the degradation kinetics are not dependent on the concentration, with similar findings occurring for highly concentrated HP products. Bringing this information to the present study, we hypothesized that in the first 20 minutes of application, only 50% of the 38% HP gel would degrade or be consumed by oxidative reactions, leaving 50% of active HP for the next 20 minutes. This may explain the similar whitening efficacy of both groups in the present study.

The protocol of not replenishing the gel during in-office whitening was first introduced by Kwon and others¹⁸; similar findings were observed in other studies that evaluated a single application time versus multiple application times in the same clinical appointment^{7,14} and seemed to gather enough evidence to support the findings that the

Table 6: pH Variation (Means $\pm$ Standard Deviations) at the Different Assessment Points for Both Study Groups and the Statistical comparison*		
Time Assessments	pH Variation	
	1 $\times$ 40 Min	2 $\times$ 20 Min
Baseline	7.4 $\pm$ 0.3	7.5 $\pm$ 0.2
After 20 min	7.5 $\pm$ 0.6	7.5 $\pm$ 0.6
After 40 min	7.7 $\pm$ 0.4	7.8 $\pm$ 0.3
* Two-way repeated measures analysis of variance test ($p=0.42$).		

bleaching gel does not need to be removed and reapplied within 40 to 50 minutes. This single application approach is of clinical interest, as it results in a shorter amount of chair time associated with a reduced cost of in-office bleaching because less material is spent per patient. Furthermore, it could reduce the risk of occasional soft tissue burns because the material would be handled only once.

When it comes to the whitening effectiveness, the bleaching product also showed significant color improvement; this was also true for other studies that used highly concentrated HP products for two clinical sessions.^{7,14,19-21} A change of approximately 10 SGUs and 8.5 units of ΔE were observed after two bleaching sessions, which is within the range reported by other investigators.

In terms of absolute risk and intensity of TS, no significant difference was observed between the study groups. Some earlier studies hypothesized that the risk and intensity of TS were higher in a single gel application, likely due to the reduction of the gel during bleaching.^{7,14} The current study evaluated the pH variations of the bleaching gel during in-office bleaching. In the present study, we observed a stable pH throughout the bleaching process, which may be the key factor for the similarity between the groups.

Indeed, alkaline pHs also seemed to be associated with a reduced risk and intensity of TS. In a previous study,¹⁵ the authors evaluated two in-office bleaching gels, with one of them being the same as that used in the present study (Opalescence Boost, Ultradent Products) and the other a product with acidic features (Pola Office, SDI, Victoria, Australia). A lower risk of TS for Opalescence Boost was observed compared with Pola Office. A very important difference between these two products was that the initial pH may prevent or minimize the passage of HP to the pulp chamber, consequently diminishing the pulp damage and TS. An *in vitro* study by Mena-Serrano and others²² showed reduced HP penetration into the pulp chamber for bleaching gels with alkaline pH. Also, it is worth mentioning that the effect of rheological properties of the gel could affect the HP penetration, and consequently TS.^{23,24} Opalescence Boost is a viscous gel while Pola office is mixed with HP liquid and a powder with inherent differences in viscosity.

Other studies that compared two bleaching gels from the same manufacturer also demonstrated a significantly lower risk and intensity of TS for the product with the highest pH.^{14,25} Obviously, other

studies with different bleaching gel brands should be conducted, and variations in pH should be correlated with the risk and intensity of TS to detect if these two variables are indeed associated.

From a theoretical point of view, keeping the pH slightly alkaline and stable also brings advantages in terms of bleaching effectiveness. It is known that the HP dissociation constant (pKa) is around 11.5,²⁶ meaning that the closer the pH is to the pKa, the higher the HP dissociation rate. Indeed, it was experimentally detected that the HP dissociation rate was 2.7 times higher in a pH of 9 than in an acidic solution (pH = 4.4). However, this higher whitening efficacy was not demonstrated in studies that evaluated bleaching gels with different pHs.^{14,25}

Another factor that may reduce bleaching-induced TS is the presence of additives in the bleaching gel, such as potassium nitrate, sodium fluoride, or amorphous calcium phosphate.²⁷ Opalescence Boost contains two desensitizing agents (3% potassium nitrate and 1.1% fluoride, according to the manufacturer's information). However, so far, no clinical studies have compared the risk and intensity of TS of in-office bleaching gels with and without desensitizers. This comparison was done for at-home bleaching gels, and controversial results were observed. Some authors reported a reduction of bleaching-induced TS when desensitizing-containing gels were used,^{15,17,28-32} whereas others reported no significant difference between desensitizing-containing and desensitizing-free at-home bleaching.^{33,34}

Unfortunately, conflicting results have been published in the literature in terms of TS when in-office bleaching gels are applied.³⁵ The high number of bleaching gels and protocols evaluated in different randomized clinical trials^{7,15,21,36} makes it difficult for clinicians to reach a clear conclusion regarding which protocol presents an increased risk and intensity of TS induced by bleaching.^{35,37} Future studies need to be done to evaluate the application protocols for different bleaching brands.

In summary, we can conclude that a simpler protocol (40-minute application) produces the same bleaching effectiveness with no increase in the risk and intensity of TS compared with two 20-minute applications.

CONCLUSIONS

The use of one 40-minute in-office bleaching agent gel produced the same whitening degree and TS

associated with two 20-minute applications. The former preferably should be applied because one 40-minute application does not require gel refreshing.

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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 Federal University of Amazonas and Santiago de Cali University. The approval codes for this study were 1.178.283 and 441.014.019, respectively.

Conflict of Interest

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

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Clinical Follow-up of a Fissure Sealant Placed Using Different Adhesive Protocols: A 24-month Split-mouth Study

AR Yazici • E Ozturk Bayazit • ZB Kutuk
G Ozgunaltay • E Ergin • A Berber

Clinical Relevance

The use of self-etch adhesive alone prior to fissure sealant application might not be a good choice for a predictable clinical performance.

SUMMARY

The purpose of this study was to evaluate the retention rates of a fissure sealant placed using different adhesive protocols over 24 months. Twenty-four subjects with no restorations or caries received fissure sealants (Clinpro Sealant, 3M ESPE) placed using different

adhesive protocols. A total of 292 sealants were placed as follows by two previously calibrated dentists using a table of random numbers (n=73): group I, acid-etch/without adhesive; group II, with a self-etch adhesive (Adper Easy Bond, 3M ESPE); group III, with an etch-and-rinse adhesive (Adper Single Bond 2, 3M ESPE); group IV, with acid + self-etch adhesive (Adper Easy Bond). Two other calibrated examiners independently evaluated the sealants at baseline and at six-, 12-, 18-, and 24-month recalls. Each sealant was evaluated in terms of caries formation being present or absent and retention using the following criteria: 1 = total retention, 2 = partial loss, and 3 = total loss. Pearson's χ^2 test was used to evaluate differences in retention rates among the sealants for each evaluation period. At the end of 24 months, total retention rates were 57.5%, 27.4%, 84.9%, and 76.7% in the acid-etch, self-etch adhesive, etch-and-rinse adhesive, and acid + self-etch adhesive groups, respectively. Although there were no statistically significant differences between the retention rates among the adhesive protocols at 6 months ($p=0.684$), significant differences were observed at the 12-, 18-, and 24-month evalua-

*A. Rüya Yazici, DDS, PhD, Department of Restorative Dentistry, Hacettepe University, School of Dentistry, Ankara, Turkey

Elif Ozturk Bayazit, DDS, PhD, Department of Restorative Dentistry, Hacettepe University, School of Dentistry, Ankara, Turkey

Zeynep Bilge Kutuk, DDS, PhD, Department of Restorative Dentistry, Hacettepe University, School of Dentistry, Ankara, Turkey

Gul Ozgunaltay, DDS, PhD, Department of Restorative Dentistry, School of Dentistry, Hacettepe University, Ankara, Turkey

Esra Ergin, DDS, PhD, Department of Restorative Dentistry, Hacettepe University, School of Dentistry, Ankara, Turkey

Asli Berber, DDS, PhD, Mamak Oral and Tooth Health Center, Ankara, Turkey

*Corresponding author: Sihhiye, Ankara 06100, Turkey; e-mail: ruyay@hacettepe.edu.tr

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tions. At 24 months, the lowest retention rates were observed in the self-etch group ($p < 0.05$). No caries development was observed in any of the groups. The retention rate of sealants placed using self-etch adhesive was poor compared with the other groups.

INTRODUCTION

Dental caries is a multifactorial disease that affects most populations throughout the world, and it is still the primary cause of oral pain and tooth loss. The occlusal surfaces of posterior teeth are the most vulnerable sites for dental caries due to their anatomy.¹ Dental sealants were introduced in the 1960s to protect pits and fissures on the occlusal tooth surfaces from dental caries.² Pit-and-fissure sealant therapy is a procedure that places a liquid material into the pits and fissures of teeth that are susceptible to caries, mainly the occlusal tooth surfaces.³ The hardened material forms a strong micromechanical bond to etched tooth enamel and acts as a barrier that prevents the accumulation of caries-producing bacteria and the onset of dental caries.⁴ Resin-based fissure sealants are the best choice and the most common preventive applications against caries progression in everyday clinical practice.^{5,6} It has been reported that loss of sealant is directly related to subsequent caries development.⁷ If the sealant is fully retained, then recurrent caries or progression of caries beneath the restoration is negligible.²

It is generally accepted that the efficacy of sealants in caries prevention depends on long-term retention of sealant.⁸ Traditionally, fissure sealants are placed after cleansing and phosphoric acid etching of the fissure enamel. Pit-and-fissure sealants require enamel acid etching to allow material infiltration into porosities, which yields mechanical strength and clinical retention.^{9,10} Phosphoric acid etching at a 30%-40% concentration is still the most common strategy prior to fissure sealant placement to obtain maximum retention because it removes contaminants, creates an irregular microporous enamel surface that is infiltrated by the resin-based fissure sealant material, and increases the surface free energy of enamel.^{11,12}

To enhance the longevity of pit-and-fissure sealants, several materials and techniques have been developed, including the use of adhesive systems under sealants. However, there is still debate regarding this use. Although some studies have shown improved results when an intermediate bonding layer was applied between enamel and

sealant, which increases retention,¹³⁻¹⁵ some have reported no difference in terms of retention and caries formation.^{14,16,17}

Modern adhesives combine conventional conditioning, priming, and bonding steps (etch-and-rinse adhesives) together into one or two bottles (self-etch adhesives) to simplify adhesive protocols. However, it is still unclear whether modern self-etch adhesives have bonding capacity equal to that of the conventional etch-and-rinse systems.¹⁸ Therefore, clinical trials are necessary to evaluate the long-term adhesive performance of self-etch adhesives compared with the conventional etch-and-rinse ones.

Researchers are still attempting to ascertain whether techniques for use in modifying the application of sealant therapy can improve the retention and effectiveness of sealants. Furthermore, there is still no consensus in the literature on which type of adhesive to use, probably due to the lack of comparative data regarding the use of self-etch and etch-and-rinse adhesives before resin-based fissure sealant application. Although many studies have evaluated the retention rates of fissure sealants, only a limited number of long-term clinical studies have been performed evaluating the new materials and comparing the conventional and current adhesive systems.^{15,19-21}

Therefore, the primary objective in this clinical study was to evaluate the sealant retention rate and caries-prevention efficiency with or without an adhesive system. The second objective was to compare fissure sealant retention rate applied with etch-and-rinse adhesive versus a self-etching adhesive system over a 24-month period. The null hypotheses were as follows: 1) the sealants placed without any adhesive (acid-etch) would have lower retention rates than the sealants placed with any of the adhesive protocols, 2) the retention rates of fissure sealants bonded with etch-and-rinse adhesive would be better than those of sealants placed using self-etch adhesives, and 3) there would be no difference between the retention rates of fissure sealants bonded with the self-etch adhesive either with or without acid etch.

METHODS AND MATERIALS

The protocol and consent form of this study were reviewed and approved by the Human Ethics Committee of Hacettepe University (protocol HEK 10/13-9). The clinical trial registration number is NCT02998814. A written informed consent for

involvement in the study was obtained from all the participants.

A total of 24 subjects (19 females and five males) who were seeking routine dental care at the restorative dentistry clinics at Hacettepe University School of Dentistry were selected. Patients who participated in the current study had good general and oral health and hygiene. They had no detectable caries, bruxism, malocclusion, previously placed restorations, or sealants on the fissures and no allergies to resins, and they were willing to return for follow-up examinations. The mean age of the patients was 21 years, ranging from 20 to 23 years.

Bitewing radiographs were taken, and intraoral examinations were performed. The plaque and debris were removed with slurry of pumice applied with a bristle brush in a slow-speed handpiece. Each tooth was isolated with standard cotton rolls and suction to avoid saliva contamination. Using a table of random numbers, two previously calibrated dentists (EOB and EE) placed a total of 292 sealants on the permanent premolars and molars using different adhesive protocols (group I, acid-etch/without adhesive; group II, one-step self-etch adhesive [SE]; group III, etch-and-rinse adhesive [ER]; group IV, one-step self-etch adhesive after acid-etching [acid + SE]). Because of the split-mouth study design, each selected patient had at least four molar or premolar teeth to receive fissure sealants. Finally, 73 sealants were included for each adhesive protocol group. The adhesive protocols were performed as follows. Group I (acid-etch/without adhesive): the occlusal surface (including all of the pits and fissures) was etched with a 37% phosphoric acid (Total Etch, Ivoclar Vivadent, Schaan, Lichtenstein) for 30 seconds. Then the surface was gently rinsed with water for 30 seconds and air-dried until an opaque appearance was obtained on the enamel. Group II (self-etch): one drop of SE adhesive, Adper Easy Bond (3M ESPE, St Paul, MN, USA), was applied on the previously cleaned occlusal surface with the help of a disposable application brush for 20 seconds. The adhesive was gently dried by air for five seconds and cured with a light-emitting-diode (LED) curing unit (Radii Plus SDI, Victoria, Australia) for 10 seconds prior to sealant application. Group III (etch-and-rinse): the etching protocol of the surfaces was performed in the same way as in group I. The etch-and-rinse adhesive Adper Single Bond 2 (3M ESPE) was applied on the etched enamel surface in two consecutive coats using a fully saturated applicator, gently air thinned for five seconds to evaporate solvents, and cured with the same LED

unit for 10 seconds. Group IV (acid + self-etch): following the same etching protocol as in group I, the same self-etch adhesive, Adper Easy Bond, was applied by the technique described for group II.

After completion of the adhesive application, a low-viscosity fluoride containing resin-based sealant (Clinpro Sealant, 3M ESPE) was applied and gently teased through the fissure with the tip of a periodontal probe to prevent voids and air entrapment. Then the applied fissure sealants were polymerized using a LED unit (1500 mW/cm²; Radii Plus SDI). The complete coverage and retention of the sealant were checked with an explorer. The occlusion was checked with articulation paper. Finishing and polishing were performed using fine-grit diamond burs (Diatech, Swiss Dental, Heerbrugg, Switzerland) and rubber cups (Edenta AG, Au, St.Gallen, Switzerland). All of the materials were used according to the manufacturers' instructions (Table 1).

Two other calibrated examiners (ARY and GO), who were unaware of which adhesive protocol had been used, independently evaluated the sealants at baseline and at six-, 12-, 18-, and 24-month recalls. At the beginning of the study, κ values were calculated to test intra- and interexaminer reproducibility. The κ values were high (0.95) and showed powerful intra- and interexaminer agreement.

All examinations were carried out using a mouth mirror and explorer, using the visual and tactile method. Sealant retention was recorded according to the following criteria²¹: 1, total retention (TR); 2, partial loss (PL); 3, total loss (TL). Each sealant was evaluated in terms of presence or absence of caries formation. Loss of enamel translucency along the margins, softness at the base of exposed fissures, and defects along the margins with discoloration were denoted as dental caries.²²

In the present study, when partial/total loss of sealant was observed, it was repaired/replaced, and those teeth were subsequently excluded from the study.

Statistical analysis was carried out using IBM SPSS for Windows (Version 22.0, SPSS Inc, Chicago, IL, USA). Pearson's χ^2 test was used to evaluate differences in the retention rates of the sealants used with different adhesive protocols for each evaluation period at a 5% level of significance. The total retention rates between baseline and all recall times for each group were compared using Cochran Q test and McNemar's test with Bonferroni correction ($p < 0.001$). Additionally, Kaplan-Meier survival

Table 1: <i>Materials Used in This Study</i>				
Brand Names	Types	Manufacturer	Compositions	Batch No.
Clinpro Sealant	Low-viscosity fluoride containing resin based sealant	3M ESPE	Bis-GMA, TEGDMA, ethyl 4-(dimethylamino) benzoate, diphenyliodonium, hexafluorophosphate, DL-camphorquinone, butylated hydroxytoluene, dichlorodimethylsilane reaction product with silica, tetrabutylammonium tetrafluoroborate, titanium dioxide, rose bengal sodium	12637
Adper Easy Bond	Self-etch adhesive		HEMA, Bis-GMA, methacrylated phosphoric esters, 1,6 hexanediol dimethacrylate, methacrylate functionalized polyalkenoic acid (Vitrebond Copolymer), finely dispersed bonded silica filler with 7-nm primary particle size, ethanol, water, initiators based on camphorquinone, stabilizers	385180
Adper Single Bond 2	Etch-and-rinse adhesive		Bis-GMA, HEMA, dimethacrylates, ethanol, water, a novel photoinitiator system and a methacrylate functional copolymer of polyacrylic and polyitaconic acids	9XN
Abbreviations: Bis-GMA, bisphenol A diglycidylmethacrylate; HEMA, 2-hydroxyethyl methacrylate; TEGDMA, triethylene glycol dimethacrylate.				

analysis and the log-rank (Mantel-Cox) test were used to estimate the probability of adhesive protocols' success ($p<0.001$).

RESULTS

A total of 292 teeth were sealed in 24 patients in the present study. All patients attended all visits, and the recall rate was 100% for all evaluation periods. A flow diagram of the patients is presented in Figure 1.

The distribution of sealant retention rates in each evaluation period is given in Table 2. The sealant retention rate, along with total retention and partial and total loss of sealants, is depicted in Figure 2. At the six-month recall, only two fissure sealants from the self-etch group were completely lost, and no difference was observed among the groups ($p=0.684$). At the 12-month recall, of the sealants placed with the etch-and-rinse protocol, the complete

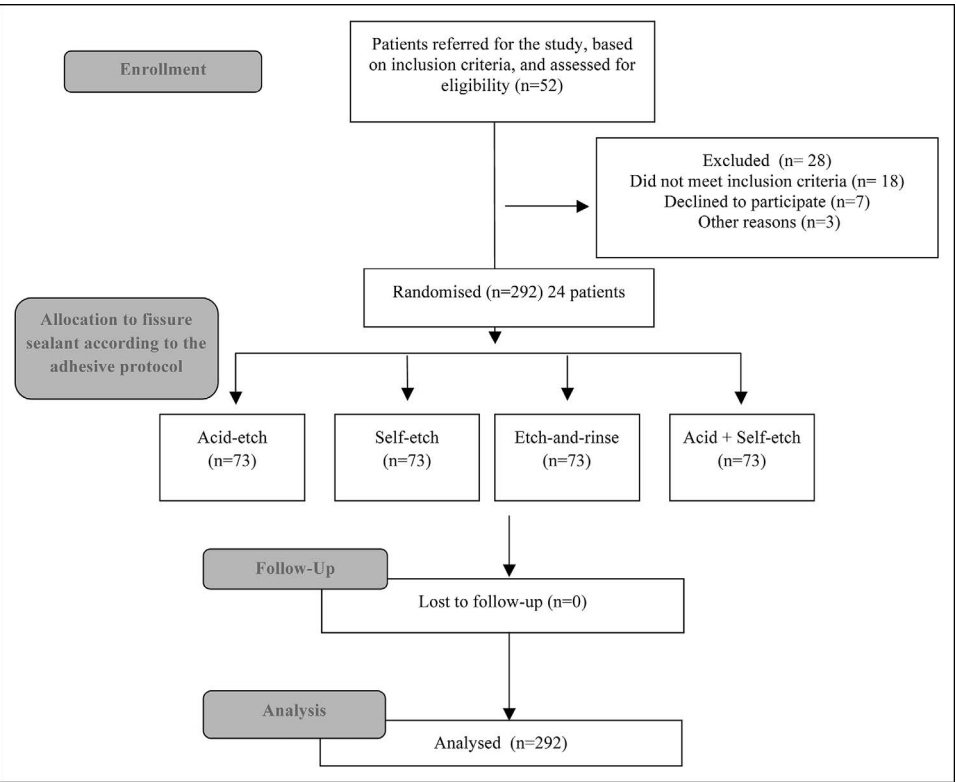


Figure 1. *Flow diagram of patient recruitment.*

Groups	Baseline, n (%)			6 Months, n (%)			12 Months, n (%)			18 Months, n (%)			24 Months, n (%)		
	TR	PL	TL	TR	PL	TL	TR	PL	TL	TR	PL	TL	TR	PL	TL
Group I (acid-etch)	73 (100)	0	0	70 (95.9)	3 (5.5)	0	62* (84.9)	9 (12.3)	2 (2.7)	46* (63)	8 (11)	19 (26)	42* (57.5)	9 (12.3)	22 (30.1)
Group II (self-etch)	73 (100)	0	0	70 (95.9)	1 (1.4)	2 (2.7)	60* (82.2)	5 (6.8)	8 (11)	26* (35.6)	7 (9.6)	40 (54.8)	20* (27.4)	8 (11)	45 (61.6)
Group III (etch-and-rinse)	73 (100)	0	0	72 (98.6)	1 (1.4)	0	71 (97.3)	2 (2.7)	0	68 (93.2)	3 (4.1)	2 (2.7)	62* (84.9)	6 (8.2)	5 (6.8)
Group IV (acid + self-etch)	73 (100)	0	0	70 (95.9)	3 (4.1)	0	69 (94.5)	4 (5.5)	0	57* (78.1)	11 (15.1)	5 (6.8)	56* (76.7)	10 (13.7)	7 (9.6)

Abbreviations: PL, partial retention; TL, total loss; TR, total retention.
* Significant difference in comparison with baseline according to Cochran Q test for total retention rates ($p < 0.05$).

retention rate was 97.3%, which was not significantly different from that of the acid + self-etch group, with a retention rate of 94.5% ($p > 0.05$). No significant difference was observed between the acid-etch, self-etch, and acid + self-etch groups ($p > 0.05$).

At the 18-month recall, the lowest retention rate (35.6%) was observed in sealants that were placed with self-etch adhesive, and it was significantly different from the other groups ($p < 0.05$). Although the retention rate of the etch-and-rinse group (93.2%) was significantly higher than that of the acid-etch (63%) and self-etch groups (35.6%) ($p < 0.05$), no difference was observed with the acid + self-etch group ($p > 0.05$).

At the 24-month recall, the best retention rates were for the etch-and-rinse adhesive and acid + self-etch groups (84.9% and 76.7%, respectively). There was no statistically significant difference between these groups ($p > 0.05$). With a retention rate of 57.5%, the acid group showed statistically similar results with the acid + self-etch group ($p > 0.05$). In the self-etch group, only 20 teeth remained fully sealed over the 24-month follow-up period, and the retention rate of this group (27.4%) was significantly lower than that of the other tested groups ($p < 0.05$).

Differences between the baseline and each recall time within each group are displayed in Table 2. There were no statistically significant differences between the baseline and six-month recall for all

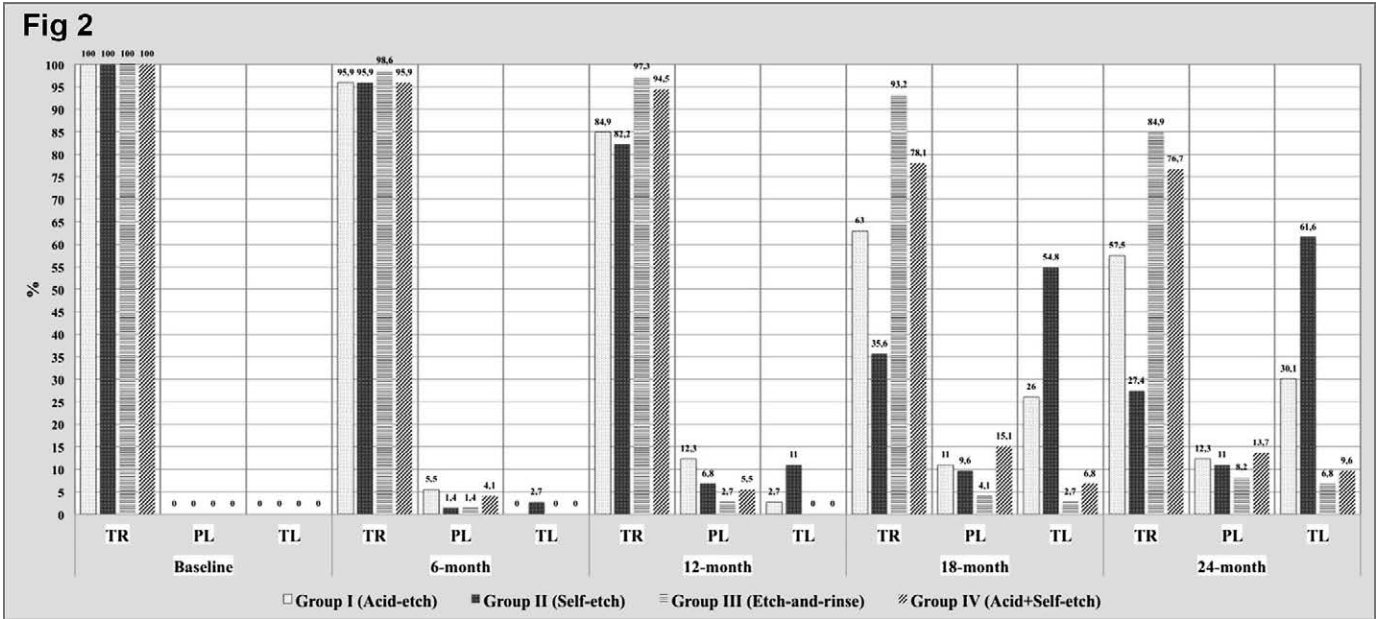


Figure 2. Sealant retention rates at baseline and 6, 12, 18, and 24 months.

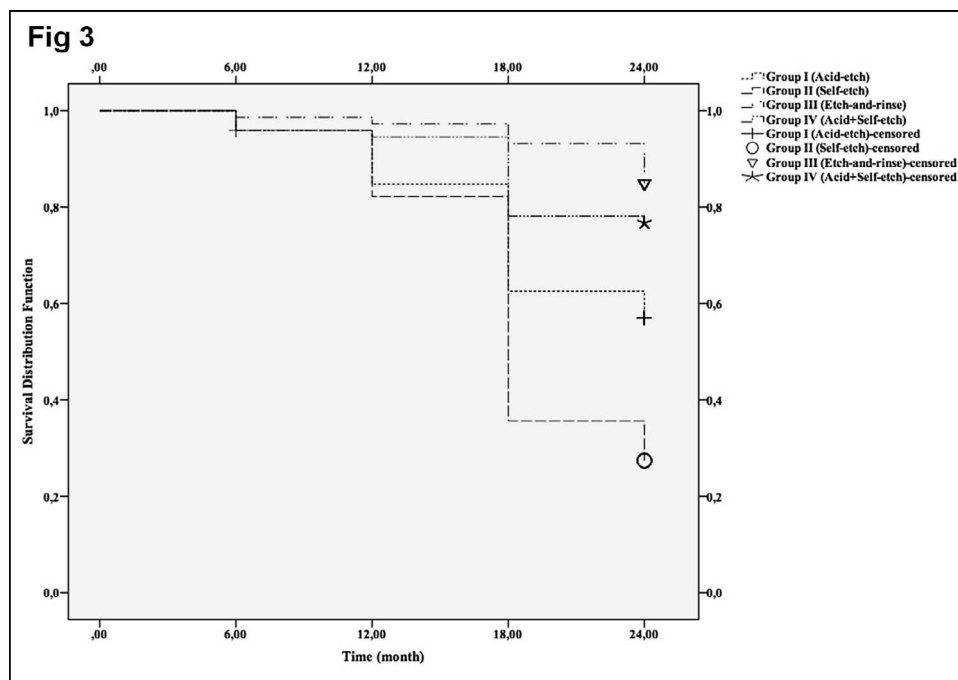


Figure 3. Cumulative survival analysis of the adhesive protocols.

groups ($p > 0.001$). For groups I (acid-etch) and II (self-etch), significant differences were observed between the baseline and each recall time. There were statistically significant differences between baseline and the 24-month recall in group III (etch-and-rinse) and between baseline and the 18-month recall and between baseline and the 24-month recall in group IV (acid + self-etch).

Figure 3 shows the retention possibilities. The Kaplan-Meier survival analysis and log-rank test revealed that significant differences were found between the tested adhesive protocols ($p < 0.001$), and it was observed that the longer the period of time, the higher the failure rate when considering the self-etch group.

Caries were not observed on any of the sealed teeth throughout the 24-month period of follow-up.

DISCUSSION

In the present study, the clinical success of a resin-based fissure sealant placed with conventional acid-etching, self-etch adhesive, etch-and-rinse, and acid + self-etch adhesive applications was compared. There are still some controversies about the use of adhesive prior to the placement of fissure sealants, as retention has a great influence on the success of sealants.

Nazar and others¹⁶ found no differences in teeth sealed with primer and bond compared with teeth sealed without primer and bond in enhancing

sealant retention and preventing caries. They recommended applying fissure sealants without using an adhesive, as this method is more cost-effective and less time-consuming. Similar findings were also reported by Khare and others¹⁴ and Boksman and others.¹⁷ In contrast, some studies favor the use of adhesives before sealant placement.^{22,23} In one of them, it was reported that the use of adhesive prior to fissure sealants significantly improved the success rate of sealants over sealant-alone groups.²³ Feigal and others²² also mentioned the importance of using a bonding agent layer between sealant and saliva-contaminated enamel for adequate bond strength and improvement of resin sealants' retention. These observations have been confirmed by Sakkas and others.²⁴ It has been assumed that the advantage of using an adhesive prior to the application of a sealant is based on the moisture-chasing effect of hydrophilic primers and increased flexibility of the combined adhesive-resin complex. This feature is important especially where saliva contamination takes place.

In the present study, at 24-month recall, because the sealants placed without any adhesive (acid-etch) had lower retention rates than the sealants placed with etch-and-rinse adhesive but higher than with self-etch adhesive, the first tested null hypothesis should be partly accepted. In other words, even without the use of an adhesive system, acid-etching yielded better results than did the sole application of self-etch adhesive. Thereby, we can assume that the

benefit of using an adhesive beneath the sealant differs according to the type of adhesive used.

However, we just tested one type of self-etch adhesive. The results might vary if different brands of self-etch with different compositions and pH are used. In a previous study, the clinical performance of sealants placed with the self-etch adhesive Adper Prompt L-Pop and conventional phosphoric acid-etching was compared,²⁵ and it was concluded that replacing acid-etching with self-etch adhesive did not compromise sealant retention in primary teeth after one year. However, we cannot directly compare our results with those of that study, as ours was only a short-term clinical evaluation. Even in our study, at 12-month follow-up we did not find significant differences between the acid-etch, self-etch, and acid + self-etch groups. Furthermore, in contrast to ours, primary teeth were evaluated in that study.

With the ease of application, reduced operation time, and nonsensitive technique, the placement of sealants with the use of self-etch adhesives has become a hot topic in recent years. Especially for children at younger ages, self-etch adhesive might be preferable due to the elimination of rinsing, cooperation difficulties, and field isolation. Although many studies have been published that compare etch-and-rinse and self-etch adhesives for the placement of fissure sealants, no consensus has been reached.^{15,21,26-28}

In our study, the placement of sealants with the use of self-etch adhesives did not appear to be practical due to the high retention loss of sealants observed. In other words, there might be a problem in terms of retention, because 45 sealants were completely lost in the self-etch group. As sealants placed with etch-and-rinse adhesive showed better performance than those placed with self-etch adhesive, the second hypothesis tested should be accepted. Therefore, it might be speculated that the use of self-etch adhesives prior to application of sealant still presents a challenge, and this finding might be related to the inappropriate surface pattern obtained for sealant infiltration and tag formation.

Similar results were also obtained in a study conducted by Sakkas and others.²⁴ In their 36-month clinical study, they reported that fissure sealants placed with the use of fourth- and fifth-generation adhesive systems (three- and two-step etch-and-rinse adhesives) yielded better performance than a sixth-generation adhesive (one-step self-etch) in terms of retention rate and caries development. Even using an acid-etch technique without an

adhesive provided better results than did the one-step self-etch adhesive, which is in agreement with our observations. The low retention rate of self-etch adhesive bonded sealants was explained by the uncured acid monomers from the oxygen-inhibited layer of cured adhesive. Another reason was the self-etch adhesive's inability to dissolve remaining organic debris entrapped in fissures. In another study, the effect of a self-etching primer (Xeno III, Dentsply, De Trey, Konstanz, Germany) and an etch-and-rinse adhesive on the clinical performance of fissure sealant was compared over a six-month period.²⁹ They found that sealant retention was poorer with the use of self-etch.

Yazici and others²⁰ also applied sealants in patients with a mean age of 20 years. They reported that the use of an etch-and-rinse adhesive prior to the placement of sealants gave better retention than did the use of a self-etch adhesive. Even in their 48-month clinical evaluation study, they obtained the same results.²¹ They attributed these results to the insufficient etching capacity of the self-etch adhesive on intact prismless enamel surfaces. The self-etch adhesive used in that study was a mild adhesive with a pH of 1.4. In the present study, even though the pH of the used adhesive system (Adper Easy Bond) was stronger (0.7-1.0), higher failure rates were again observed in the self-etch group. The less pronounced microretentive etching pattern obtained with even more acidic self-etch adhesives might be the reason. On the other hand, contrary to our findings, in a study that compared two adhesive systems, Adper Single Bond (an etch-and-rinse adhesive system) and Clearfil SE (a two-step, self-etch adhesive, Kuraray, Tokyo, Japan), on sealant retention in newly erupted teeth, no difference was observed between the adhesive systems tested in terms of the success of the sealants under isolated and contaminated conditions.²³ The opposite result obtained might be related to the age and composition of the teeth. In our study, the sealants were applied on fully erupted and highly mineralized teeth, which are quite different in terms of mineralization and isolation from the teeth in the mentioned study.

In the present study, when comparing the self-etch and acid + self-etch groups, poorer results were obtained in the self-etch group at the end of 24 months, and therefore the third hypothesis should be rejected. It is not surprising that self-etch with prior acid-etching showed better results, as separate enamel etch is generally recommended for self-etch adhesive when used on unprepared enamel. It is known that phosphoric acid used to superficially

demineralize the hydroxyapatite of enamel increases the surface area available for bonding. In a recent study, an improvement in sealant retention rates was observed when self-etch adhesives were used with prior acid-etching.¹⁵ Many *in vitro* findings confirm this statement even though most of them were obtained from prepared teeth.^{27,30} In one study, the same self-etch adhesive (Adper Easy Bond) that we used in our study was evaluated, and pre-etching enamel with phosphoric acid improved the bond strength significantly.³⁰

In the present study, the complete retention rates were 84.9% and 76.7% for the etch-and-rinse adhesive and acid + self-etch groups, respectively, and no difference was observed between these two groups. Our results are in line with those given by Erbas Unverdi and others,¹⁵ who reported similar retention rates in their self-etch group with prior acid etching and etch-and-rinse group.

There are two recently published reviews regarding the use of adhesives and the type of adhesive for improving the success of fissure sealants.^{31,32} In one of them, the clinical studies that compared the retention rate of sealants placed on occlusal surfaces following the use of self-etch adhesive systems with traditional acid etching with or without the application of an adhesive system were analyzed.³¹ Regardless of the use of adhesive systems, it was reported that occlusal sealants applied with self-etch systems showed lower retention over time than sealants applied in the conventional approach. In the other one, Bagherian and others³² systematically reviewed the literature to evaluate fissure sealant retention with and without the use of an adhesive system and to compare fissure sealant retention using etch-and-rinse adhesive systems versus self-etch adhesives. They concluded that the use of adhesive systems beneath fissure sealants can increase the retention of fissure sealants and etch-and-rinse systems were preferable.

There are also some *in vitro* studies that evaluated the efficacy of the use of different adhesives on the bond strength of sealants.^{27,28} In an *in vitro* study, the effect of phosphoric acid and a self-etch adhesive on the bond strength of a sealant to unground primary and permanent enamel was compared.²⁷ No significant difference was noted in bond strengths between the phosphoric acid-etch and the self-etch adhesive groups nor between the one-week and one-year results. They concluded that the self-etch adhesive could be an attractive alternative to the acid-etch technique for sealant retention, especially in young children. On the other hand, another study

reported that using either self-etch or etch-and-rinse adhesive provided bond strength values higher than those obtained with acid-etch only without adhesive.²⁸ Similar bond strength values were achieved with self-etch and etch-and-rinse adhesive. However, these studies were conducted on ground enamel, whereas sealants in clinics are generally applied without any fissure preparation and self-etch adhesive might have been less effective on unground enamel and yielded poorer results. Both of the studies mentioned above were conducted under *in vitro* conditions, and therefore these findings cannot be directly extrapolated to clinical situations because all clinical factors such as field isolation and patient factors are omitted.

In the present study, isolation was accomplished using cotton rolls and a saliva ejector. In most studies conducted in older patients, similar to ours, cotton rolls were used for isolation.^{19-21,33} Proper isolation is generally hard to achieve in pediatric patients, and therefore the risk of contamination is higher compared with in adults. As this study was conducted in teenagers with a high ability of contamination control, we used cotton roll isolation, which is more convenient in terms of patient acceptance and placement time. Moreover, in a three-year clinical study, no difference was noted between the retention rates of sealants placed under rubber dam isolation or not.³⁴

No caries development was observed in teeth that had sealant at any evaluation period in the present study. This could also be related to the regular attendance of patients to their recall appointments and their high motivation for dental care. Moreover, the lack of any restoration or carious lesion up to that age is another sign of the good oral hygiene habits of the participants. In a systematic review, it was reported that children and adolescents who received sealants in sound occlusal surfaces or noncavitated pit-and-fissure carious lesions in their primary or permanent molars (compared with a control without sealants) experienced a 76% reduction in the risk of developing new carious lesions after two years of follow-up.³⁵

This is just a short-term clinical study. Thus, our findings need to be confirmed by further long-term studies with different self-etch adhesives.

CONCLUSION

Within the limitations of the current clinical study, it can be concluded that resin-based fissure sealant, in conjunction with etch-and-rinse adhesive, showed

better retention rates than did self-etch adhesive over a 24-month period. However, no significant differences were observed between the retention rates of fissure sealants bonded with etch-and-rinse adhesive and acid + self-etch adhesive. The self-etch adhesive system did not provide a sufficient retention rate compared to other adhesive protocols. Additional long-term clinical investigations are necessary to confirm these results.

Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the Hacettepe University. The approval code for this study is HEK 10/13-9.

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

Self-etching Primers vs Acid Conditioning: Impact on Bond Strength Between Ceramics and Resin Cement

JPM Tribst • LC Anami • M Özcan • MA Bottino • RM Melo • GSFA Saavedra

Clinical Relevance

A conditioning protocol of dental ceramics with a less hazardous material and a reduction of clinical steps, making the resin cement bonding procedure safer and easier, is an alternative to hydrofluoric acid conditioning.

SUMMARY

This study tested whether a self-etching surface agent and the conventional hydrofluoric acid (HF) would provide the same bonding capacity between resin cement and feldspathic (Fd) and lithium disilicate (Ld) ceramics. Ceramic blocks were cut with a low-speed diamond saw with water cooling (Isomet 1000, Buehler, Lake Bluff, IL, USA) into 20 blocks of $5 \times 7 \times 4$ mm, which were ground flat in a polishing machine (EcoMet/AutoMet 250, Buehler) under water cooling. The blocks were

randomly divided into eight groups (n=5), according to ceramic type (Ld or Fd), surface conditioning (HF + Monobond Plus or Etch and Prime), and aging by thermocycling (TC or absence-baseline). After 24 hours in 37°C distilled water, blocks were embedded into acrylic resin and 1-mm² cross-section beams composed of ceramic/cement/composite were obtained. The microtensile test was performed in a universal testing machine (DL-1000, EMIC, São José dos Campos, Brazil; 0.5 mm.min⁻¹, 50 kgf load cell). Bond strength (MPa) was calcu-

João Paulo Mendes Tribst, DDS, MSc, PhD student, Department of Dental Materials and Prosthodontics, São Paulo State University (Unesp), Institute of Science and Technology, São José dos Campos, São Paulo, Brazil

Lilian Costa Anami, DDS, MSc, PhD, professor, Department of Dentistry, Santo Amaro University, São Paulo, São Paulo, Brazil

Mutlu Özcan, DDS, MSc, PhD, professor, University of Zurich, Dental Materials Unit, Zurich, Switzerland

Marco Antonio Bottino, DDS, MSc, PhD, professor, Department of Dental Materials and Prosthodontics, São Paulo State University (Unesp), Institute of Science and Technology, São José dos Campos, São Paulo, Brazil

*Renata Marques de Melo, DDS, MSc, PhD, researcher III, Department of Dental Materials and Prosthodontics, São Paulo State University (Unesp), Institute of Science and Technology, São José dos Campos, São Paulo, Brazil

Guilherme de Siqueira Ferreira Anzaloni Saavedra, DDS, PhD, professor, Department of Dental Materials and Prosthodontics, São Paulo State University (Unesp), Institute of Science and Technology, São José dos Campos, São Paulo, Brazil

*Corresponding author: Av. Engenheiro Francisco José Longo, 77, SP 12245000, Brazil; e-mail: renata.marinho@fosjc.unesp.br

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lated by dividing the load at failure (in N) by the bonded area (mm^2). The fractured specimens were examined under stereomicroscopy, and one representative sample of each group was randomly selected before the cementation and was further used for analysis using scanning electron microscopy (SEM) and energy-dispersive x-ray spectroscopy (EDS). The self-etching agent showed the highest bond strength for Fd (24.66 ± 4.5) and Ld (24.73 ± 6.9) ceramics and a decrease in surface wettability. SEM and EDS showed the presence of similar components in the tested materials with different topographies for both. Therefore, the self-etching primer was able to deliver even higher bonding than HF+silane to a resin cement.

INTRODUCTION

The use of feldspathic (Fd) porcelain as an indirect restorative material is common in modern dentistry because of its esthetic and functional capacities.¹ Hydrofluoric acid (HF) etching is often used in preparing the material, but bonding in regions that require large masticatory effort can result in failures.² Lithium disilicate (Ld)-based ceramics represent attractive alternatives because of their esthetic and resistance properties; these materials are also amenable to HF conditioning. Thus, these ceramics are used in various indications, such as onlays, inlays, veneers, and even fixed partial dentures as far posterior as premolars.²⁻⁴

Working with acid-sensitive ceramics and their component materials requires a comprehensive understanding of their response to HF exposure, including concentration and time, to properly condition the material surface for adhesion to resin cement.⁵⁻⁷

Etching of ceramic with HF selectively dissolves the glassy phase and makes the surface porous to allow resin cement penetration.²⁻⁵ This porous surface includes hydroxyl groups that have great interaction with silane-coupling agents.⁸

Self-etching materials, which could replace the current standard treatment of HF and silanization, would offer reduced occupational hazards (i.e., occupational and patient exposure to potential risks and biological damage from acid contact with living tissue),⁹ as well as a decrease in clinical steps. Monobond Etch and Prime (Ivoclar Vivadent AG, Schaan, Liechtenstein), composed of ammonium polyfluoride, trimethoxypropyl methacrylate, alco-

hols, and water, is one such self-conditioning silane that, according to the manufacturer, replaces HF etching without compromising bond strength.

According to the manufacturer, this material allows for superficial etching of the ceramic restoration through ammonium polyfluoride and silanization with trimethoxypropyl methacrylate. Moreover, according to the manufacturer, the roughened surface is less pronounced than the surface formed after conditioning with HF but allows for adequate adhesion of the restoration.

Beyond the evaluation of the initial adhesion, the maintenance of the bond after continued use is paramount for significant adoption. When adhesive interfaces are submitted to aging, they tend to degrade and drastically compromise the bonding between resin cement and glass ceramic.¹⁰

In this study, the bond strength between Ld and Fd ceramics to a resin cement via HF etching followed by silanization or self-etching silanes was evaluated in the absence or presence of aging by thermocycling. The hypothesis was that the simplified self-etching agent would provide bond strengths as high and durable as HF for both ceramics.

METHODS AND MATERIALS

Specimen Preparation

The materials used in this study, as well as the respective manufacturers, compositions, and batch numbers, are described in Table 1.

Ld and Fd ceramic blocks were cut with a low-speed diamond saw with water cooling (Isomet 1000, Buehler, Lake Bluff, IL, USA) into 20 ($5 \times 7 \times 4$ mm) blocks of each ceramic type. The surfaces of the blocks were ground flat with decreasing granulation SIC paper (600, 800, and 1200 grit) using a polishing machine (EcoMet/AutoMet 250, Buehler) under water cooling.

Ld ceramic blocks were crystallized following the instructions of the manufacturers. The blocks were randomly divided into eight groups ($n=5$), according to ceramic type (Ld or Fd), silanization technique (Plus or EP), and the presence of aging by thermocycling (TC or absence/baseline).

Prior to the conventional silanization technique using Monobond Plus (Plus), "LdPlus" and "LdPlusTC" blocks were etched with 10% HF (Condacporcelana, FGM, Joinville, Brazil) for 20 seconds, rinsed with tap water for the same time, and dried with an oil-free air jet; "FdPlus" and "FdPlusTC" blocks were etched with 10% HF (Condacporcelana,

Table 1: Materials, Trademarks, Manufacturers, Composition, and Batch Numbers of Materials Used in the Present Study			
Material	Trademark	Manufacturer	Composition
Lithium dissilicate ceramic (Ld)	Emax CAD	Ivoclar Vivadent, AG, Schaan, Liechtenstein	SiO, Li ₂ O, K ₂ O, MgO, Al ₂ O ₃ , P ₂ O ₅
Feldspathic ceramic (Fd)	VITA Mark II	Vitablocs Mark II, Vita Zahnfabrik, Bad Säckingen, Germany	Al ₂ O ₃ , Na ₂ O, K ₂ O, TiO ₂ , SiO ₂ , CaO
Silane (Plus)	Monobond Plus	Ivoclar Vivadent, AG, Schaan, Liechtenstein	Alcohol solution of silane methacrylate, phosphoric acid methacrylate, sulphide methacrylate
Self-etching silane (EP)	Monobond Etch and Prime	Ivoclar Vivadent, AG, Schaan, Liechtenstein	Ammonium polyfluoride, trimethoxypropyl methacrylate, alcohols, water
Resin Cement	Variolink II	Ivoclar Vivadent, AG, Schaan, Liechtenstein	BisGMA, UDMA, TEGDMA, DMA, bariumsulfate, Ba-Al-F-glass, silica benzoperoxy glycerol.
Composite	IPS Empress Direct	Ivoclar Vivadent, AG, Schaan, Liechtenstein	Dimethacrylates, barium glass, ytterbium trifluoride, mixed oxide, silicon dioxide copolymer

FGM) for 60 seconds, rinsed, and dried. Monobond Plus was then applied on the surface, and the time for volatilization of the solvent was observed before cementing.

Ceramic blocks in the Ld subgroup were not acid etched and received a single-step surface treatment using Monobond Etch and Prime (EP). Blocks from LdEP, LdEPTC, FdEP, and FdEPTC groups received an active application of EP for 20 seconds, followed by 30 seconds of setting. The samples were then washed with running tap water and dried with an oil-free air jet. The protocols used herein are those indicated by the manufacturers. The materials have different microstructures requiring different etching times. However, the groups treated with EP have the same time because this material is indicated to be used with this same protocol no matter the ceramic.

A silicone matrix with a 5 × 7 × 4 mm cavity was prepared. With it, composite blocks (IPS Empress) were prepared using the layering technique (maximum 1-mm-thick layers). The freshly polymerized composite blocks were cemented onto ceramic blocks using a resin cement (Variolink II). A 750-g weight was used to keep blocks in position during the removal of excess cement, using a microbrush and photoactivation for 40 seconds on each surface.

Microtensile Bond Strength Test

After 24 hours of storage in 37°C distilled water, the blocks were immersed into acrylic resin, and 1-mm² cross-section beams composed of ceramic/cement/composite were obtained by means of a cutting machine (Isomet 1000, Buehler) under constant

cooling. The external beams of each block were demarcated and excluded.

Baseline beams were immediately tested, while beams from the TC subgroups were used to analyze the effect of aging on the bond strength resulting from each silanization technique on different ceramic materials.¹¹ TC samples were subjected to 5000 thermal cycles in water at temperatures of 5 and 55°C, with 30 seconds of immersion and 5 seconds of transition (521-6D, Ethik Technology, Vargem Grande, São Paulo, Brazil).

The dimensions of the specimens were measured with a digital caliper, and the specimens were glued to the testing device (OG01, Odeme, Lucerne, Brazil) with cyanoacrylate (Superbonder, Loctite, Lucerne, SC, Brazil). Microtensile bond strength (μTBS) testing was performed on at least 15 beams from each block in a universal testing machine (DL-1000, EMIC, São José dos Campos, Brazil; 0.5 mm.min⁻¹, 50 kgf load cell), and the bond strength (MPa) was calculated by dividing the load at failure (N) by the adhesive area (mm²). For the pretest failures, the lowest load at failure (N) for the group was assigned.

Failure Analysis

The fractured specimens were examined by stereo-microscopy (Stereo Discovery V20, Zeiss, Göttingen, Germany), and the failure modes were classified as cohesive failure of composite resin (COHES res), adhesive failure between composite resin/resin cement (ADHES cim/res), a mixed adhesive/cohesive failure (MIXED), adhesive failure between ceramic/

resin cement (ADHES cer/cim), or cohesive failure of the ceramic (COHES cer).

Goniometry

Ld and Fd blocks of $3 \times 3 \times 2$ mm were used for the wettability evaluation ($n=3$). The samples were prepared by Plus or EP treatment as described above. One additional sample of each ceramic was only polished and used as a control surface.

The wettability was analyzed by the sessile drop technique using an optical tensiometer (TL 1000, Theta Lite Attention, Lichfield, Staffordshire, UK). First, a syringe (No. 1001 Gastight Syringes, 1 mL, Hamilton, Reno, NV, USA) deposited a drop of distilled water on the sample surface with a mean volume of $8.3 \mu\text{L}$. After 20 seconds of drop settling,¹² a series of 60 images per second was recorded by the equipment for 20 seconds. OneAttention (Biolin Scientific, Lichfield, Staffordshire, UK) software was used for calculation of the contact angle mean value from the images obtained in 10 different areas for each sample.

Scanning Electron Microscopy

One representative sample from each group was randomly selected before the cementation and was further used for analysis using scanning electron microscopy (SEM; Inspect S50, FEI, Czech Republic). The specimens were sputter coated with gold for 180 seconds at 40 mA, creating a 30-nm-thick layer and examined under different standard SEM magnifications operated at 20 KV using secondary electron detection by a single operator. One sample from each group was irradiated using x-ray fluorescence (EDX) spectroscopy.

Data Analysis

Data were tabulated and the assumptions of normality were confirmed. Blocks were the experimental units for statistical analysis of the bond strength results, and cohesive failures were removed from statistical analysis. μTBS results were statistically

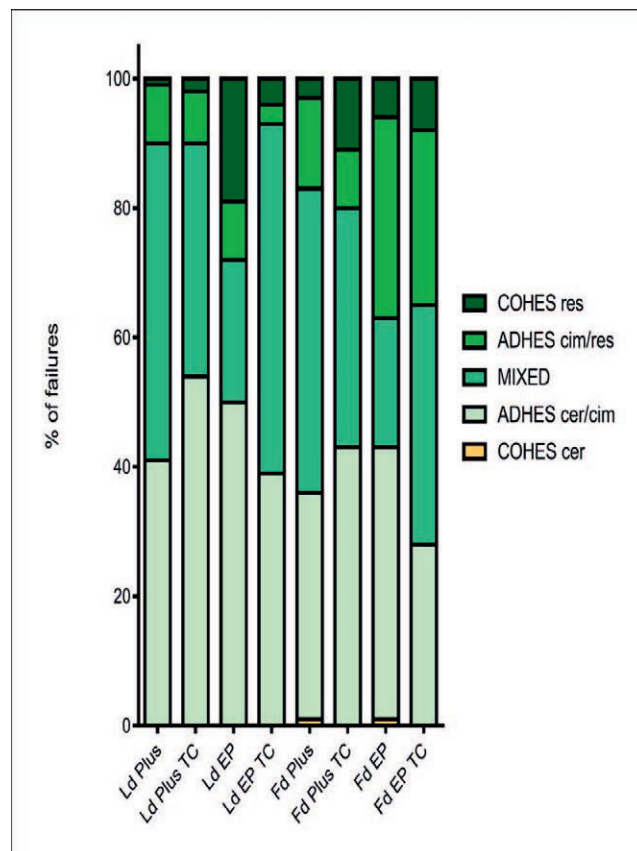


Figure 1. Column graph of the analysis of different failure profiles between the groups.

analyzed by three-way analysis of variance (ANOVA; ceramic $\times$ silanization technique $\times$ aging), and differences were detected by Tukey test (both $\alpha=0.05$). Contact angle values were analyzed by two-way ANOVA (ceramic $\times$ silanization technique) and Tukey test (both $\alpha=0.05$). EDX and SEM data were qualitatively analyzed.

RESULTS

The mean values of μTBS ranged between 16.05 and 24.73 MPa (Table 2). ANOVA showed that the “aging” factor significantly influenced the bond strength results ($p<0.0001$); thermocycling signifi-

Table 2: Means (in MPa) and SD of the μTBS Test^a

	Lithium Disilicate		Feldspathic	
	Plus	EP	Plus	EP
Baseline	22.60 $\pm$ 3.0 ^A	24.73 $\pm$ 6.9 ^A	21.35 $\pm$ 2.5 ^A	24.66 $\pm$ 4.5 ^A
TC	16.05 $\pm$ 4.0 ^B	16.08 $\pm$ 5.4 ^B	18.26 $\pm$ 3.3 ^B	19.53 $\pm$ 2.3 ^B

^a Same capital letters indicate absence of significant differences.

Table 3: Mean (in °) and SD of the Goniometry Test		
	Ceramic	
	Lithium Disilicate	Feldspathic
Polished samples	27.28 ± 6.17 ^A	31.56 ± 4.51 ^A
Plus	19.23 ± 4.13 ^B	12.47 ± 3.41 ^B
EP	89.31 ± 9.30 ^C	88.19 ± 4.07 ^C

^a Same capital letters indicate absence of significant differences, α=5%.

cantly reduced the bonding between ceramics and resin cement.

Mixed failures (association of adhesive and cohesive failures) were present in all groups, being predominant only for LdEPTC. For the other groups, adhesive failures were mostly seen, with more failures in the interface between ceramic and resin cement than composite resin and resin cement. Cohesive failures of composite resin were present in all groups, and cohesive failures of ceramic were present only for Fd ceramic without thermocycling regardless of surface treatment (Fd Plus and Fd EP), presenting the lowest frequency of occurrence in these groups compared with the other types of failures. All failures are summarized in Figure 1.

The wettability results showed a statistically significant difference between groups, with a decrease in the surface wettability caused by the EP surface agent (Table 3).

Figure 2 presents the representative micrographs of tested samples. Polished samples of Ld and Fd showed smooth and homogeneous surfaces without any porosity as a result of polishing. The etched surfaces showed changes in the surface microstructure with the presence of irregularities including numerous microporosities, grooves, and striations as a result of the dissolution of the glassy phase. The Ld etched for 20 seconds predominantly showed a glassy phase and small, isolated pores, while the Fd specimens showed large cavitations. EDX spectroscopy showed the presence of similar components in the tested materials. Ld-based ceramic showed peaks attributed to aluminum, silicon, phosphorus, potassium, and lightweight elements that could not be resolved in the analysis. The Fd samples were composed of sodium, aluminum, silicon, and potassium. The samples treated with Plus also presented sulfur.

DISCUSSION

The hypothesis that the simplified self-etching agent would provide bond strengths as high and durable as HF for both ceramics was accepted. The use of a

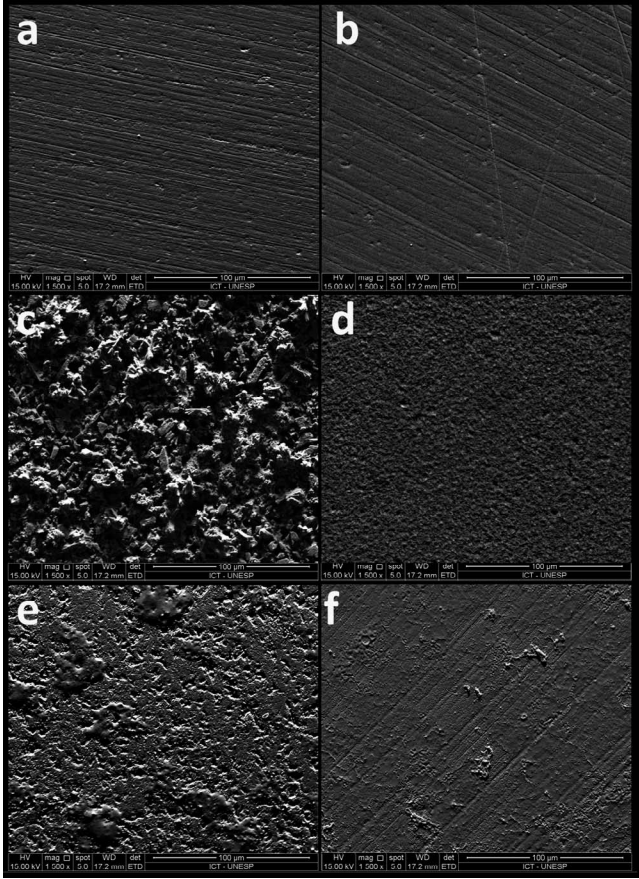


Figure 2. Representative micrographs. 1500×. Polished samples of (a) lithium disilicate and (b) feldspathic ceramics. (c) LdPlus. (d) FdPlus. (e) LdEP. (f) FdEP.

chemical agent capable of performing as both surface etchant and adhesive between the inorganic matrix of the ceramic and the organic matrix of the resin cement is attractive to dentists because of the streamlined bonding process. This simplification of clinical steps during the surface preparation of both the tooth and substrate was obtained with self-etching dental adhesives,¹³ self-adhesive resin cements,¹⁴ and, more recently, with self-etching primers for glass ceramics.

The use of self-conditioning primers in this study resulted in similar bonding strengths as those of the conventional acid-etched group. Because glass ceramics are acid sensitive, there was no need to significantly change the surface treatment protocol.^{3,14,15} The results revealed that self-conditioning primers do not present disadvantages related to bond integrity while reducing clinical steps and minimizing risks related to HF etching.⁹ The protocol of self-etching primers also appears to be

more predictable than others tested in the literature, such as mechanical conditioning, which damages the material surface with and without silica coating,^{15,16} and silane heating before its use.¹⁷

Kimmich and Stappert¹⁸ mentioned some approaches for repair of fractured ceramic restorations in the oral environment. Among the options were repair with resin or with the ceramic fragment. To do that, the restoration must be properly prepared in the intraoral environment, which includes surface conditioning with HF to achieve adequate adhesive strength. However, the authors emphasized the need for total control of the technique because of the toxicity of HF. Intraoral conditioning with EP has not yet been reported in the literature, and its effects on tissues have yet to be studied, but this technique requires fewer clinical steps and is possibly not as harmful as HF.

In this study, both ceramics presented similar contact angles and wettability for HF-treated surfaces. This indicates that the removal of the glass matrix and exposure of chemical ligands was similar in both tested ceramics; any overtreatment would increase the number of pores and consequently influence the surface wettability.^{6,19} These modifications can be observed in SEM micrographs (Figure 2c, d). Fd and Ld displayed decreased wettability after surface treatment with the self-etching silane, suggesting that the resulting microstructural changes (Figure 2e, f) possibly led to the formation of a layer containing debris, making the surface less susceptible to wetting.^{6,20}

Topographic change is essential to the maintenance of adhesive strength, as an increased number of pores of various sizes and widths allows for better micromechanical bonding area.¹⁹ This increase in surface roughness also affects the wettability of the ceramic surface for the application of silane-coupling agents and resin composites.^{6,21} An adequately porous surface is vital for the durable cementation of both Ld⁶ and Fd ceramic^{4,7} indirect restorations. In addition to roughness, other factors that contribute to the effectiveness of bonding between the materials are clean surfaces, suitable wetting, and the use of low-viscosity adhesives and cements.²⁰ Unfortunately, the presence of fluoride in EP materials decreases the wettability of the substrate,²⁰ but this does not appear to have affected the ceramics in this study.

Ramakrishnaiah and others⁶ demonstrated that wettability is directly proportional to surface

irregularities, so that increasing the HF action time on a ceramic surface causes increased wettability of the ceramic. Despite that, the authors counterindicated a prolonged conditioning of the ceramic surface, because although wettability increases, it does not lead to increased bond strength.²² On the other hand, self-etching silane cannot dissolve the glassy phase as profoundly as HF (Figure 2), but silane will spread more because of active application. This justifies a higher contact angle in the EP groups, although bonding was efficiently attained after the silane was actively applied on the ceramic surface. Moreover, fewer surface defects that could weaken the ceramic as a result of overetching were created when using a self-etching silane.

Aging by thermocycling had a negative effect on bond strength in this study. Samples in groups without cycling had an average bond strength of 25.2 MPa, while the aged groups showed lower mean values (18 MPa). Most samples withstood the thermal cycles, and the low percentage of predominantly adhesive failure between the resin cement and ceramics showed that surface treatment in a single step ensures a stable bond, similar to the conventional treatment of ceramic and cement for both glass ceramics used.²³ Moreover, the EDX analysis did not show the presence of fluoride, likely because of the small concentration and the low atomic weight of that element. Thermocycling instead of long-term water storage was chosen because both protocols show similar behaviors with regard to bond degradation.²⁴

Microtensile testing of composite-ceramic bonding to evaluate ceramic processing minimizes the effects of dental substrate variation originating from different diameters of exposed dentinal tubules.^{6,18,25} Most beams showed adhesive failure between resin cement and ceramics, which is corroborated with studies that analyzed conventional silanization.^{6,25} Regardless of the material present at the adhesive interface, all specimens were vulnerable to temperature changes in the water, resulting in a significant decrease in bond strength.

Through SEM, it was observed that the Monobond EP caused morphological changes compared with the control groups for both materials, producing an increase in surface roughness as well as the formation of a discrete residual layer.

For the Monobond Plus group, there was an even more remarkable morphological change: the use of HF resulted in a significantly rougher surface, as

previously described in the literature.^{6,18,25} However, this morphological difference changed only the wettability and did not affect the bond strength.

CONCLUSIONS

Considering the positive results obtained for the surface treatments and the potential risks associated with HF procedures, it is suggested that the self-etching system is ideal for maintaining adhesive bonds to glass ceramics. Further studies are needed to explore the time required for conditioning of ceramics due to the observed structural changes caused by the self-etching primer.

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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Influence of Thermal Stress on Simulated Localized and Generalized Wear of Nanofilled Resin Composites

A Tsujimoto • WW Barkmeier • T Takamizawa • MA Latta • M Miyazaki

Clinical Relevance

When making treatment decisions, clinicians should consider the impact on nanofilled resin composite wear characteristics of degradation by occlusal and thermal stresses.

SUMMARY

Objective: This study investigated the influence of thermal stress on the simulated localized and generalized wear of nanofilled resin composites.

Methods: Six nanofilled resin composites were evaluated and then subjected to a wear challenge of 400,000 cycles in a Leinfelder-Suzuki (Alabama) wear simulation device after 24

hours of water storage (24-hour group) and 24 hours of water storage and 10,000 thermal cycles (TC group). Simulated localized wear was generated using a stainless-steel ball bearing, and simulated generalized wear was generated using a flat-ended stainless-steel cylinder. Wear testing was accomplished in a water slurry of polymethyl methacrylate beads. Simulated localized and generalized wear was determined using a noncontact profilometer (Proscan 2100) in conjunction with Proscan and AnSur 3D software.

Results: Wear was significantly different ($p < 0.05$) among the resin composites for both simulated localized and generalized wear of either the 24-hour group or the TC group. The simulated localized wear of the TC group was significantly greater than that of the 24-hour group; however, the simulated generalized wear of most of the resin composites of the TC group was not significantly different from that of the 24-hour group.

Conclusion: The simulated localized and generalized wear of nanofilled resin composites is material dependent. The simulated localized wear of nanofilled resin composites appears to

*Akimasa Tsujimoto, DDS, PhD, Operative Dentistry, Nihon University School of Dentistry, Tokyo, Japan; General Dentistry, Creighton University School of Dentistry, Omaha, NE, USA

Wayne W Barkmeier, DDS, MS, General Dentistry, Creighton University School of Dentistry, Omaha, NE, USA

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

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

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

*Corresponding author: 1-8-13, Kandasurugadai, Chiyodaku, Tokyo, 101-8310, Japan; e-mail: tsujimoto.akimasa@nihon-u.ac.jp

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be influenced by thermal stress, whereas this effect is not as apparent in simulated generalized wear testing.

INTRODUCTION

Considerable improvements have been made over the past several years in resin composite materials with the modification of resin matrix formulations and filler characteristics.¹ More recently, improvements in resin composite technology have led to the introduction of nanofilled resin composites for clinical use.² Such resin composites have been reported to exhibit good mechanical properties,³ improved surface characteristics,⁴ better gloss retention,⁵ and reduced polymerization shrinkage.⁶

However, controlled clinical studies on posterior restorations using nanofilled resin composites and microhybrid resin composites revealed no significant differences in clinical performance.^{7,8} Wear properties of nanofilled resin composites remain a clinical concern, particularly in extensive posterior restorations and treatment of patients with abnormal occlusal behavior, such as clenching and/or bruxing.⁹

In clinical situations, occlusal stress is transmitted to resin composite restorations through rigid and brittle fillers into the more flexible resin matrix during both function and parafunction activities.¹⁰ Stress concentrations at the filler–resin matrix interface can result in filler dislodgement and exposure of the resin matrix, leading to wear.¹¹ Such stress concentrations can also be generated by cyclic temperature changes.¹² During cyclic temperature changes, differences in thermal expansion coefficients between fillers and the resin matrix in resin composites may lead to interfacial stresses in restorations.¹³

A thermal cycling test is the process of subjecting a specimen to cyclic temperature changes through water immersion to mimic intraoral thermal changes.¹⁴ Investigations in a previous study contended that 10,000 thermal cycles correspond to one year of clinical function of restorations. This estimate was based on the hypothesis that such cycles might occur 20 to 50 times per day.¹⁵ A previous study reported that the flexural properties of nanohybrid resin composites significantly decreased after thermal cycling protocols (15,000, 30,000, and 45,000 cycles).¹³ Another study reported that the effect of thermal cycling was material dependent in microhybrid resin composites.¹⁶ However, the influence of the combination of occlusal and thermal stress on

nanofilled resin composites is still unclear. Evaluation of the interactive effects between occlusal and thermal stress on wear properties of resin composites is important for assessing their clinical performance¹⁷ because these two types of stresses are different and may cause more wear in combination than they do individually. In addition, previous studies showed good agreement between simulated wear and clinical wear.^{18,19}

Nanofilled resin composites incorporating a larger amount of nanosized filler particles in a more homogeneous distribution in the resin matrix have a larger interface area between fillers and a resin matrix than do conventional resin composites.¹⁷ Thus, the total interfacial stress between filler and resin matrix generated by intraoral conditions in nanofilled resin composite has been estimated to be greater than that in conventional resin composites.¹⁸ This may lead to unpredicted clinical performance of nanofilled resin composite restorations, especially in extensive posterior restorations and patients with abnormal occlusal habits, such as clenching or bruxing. Therefore, evaluation of the wear resistance of nanofilled resin composites after thermal cycling test may provide valuable information about the longevity of clinical restorations.

This study aimed to investigate the influence of thermal stress on simulated localized and generalized wear of nanofilled resin composites. The null hypotheses to be tested were 1) that no significant differences exist in simulated localized and generalized wear of different nanofilled resin composites and 2) that simulated localized and generalized wear of nanofilled resin composites are not influenced by thermal stress.

METHODS AND MATERIALS

Study Materials

Six nanofilled resin composites were evaluated in this study: 1) Filtek Supreme Ultra Universal Restorative (FS; 3M ESPE, St Paul, MN, USA), 2) MI Gracefil (MG; GC, Tokyo, Japan), 3) TPH Spectra high viscosity (TS; DENTSPLY Caulk, Milford, DE, USA), 4) Clearfil Majesty ES-II (CM; Kuraray Noritake Dental, Tokyo, Japan), 5) Estelite Σ Quick (EQ; Tokuyama Dental, Tokyo, Japan), and 6) Beautifil II (B2; Shofu, Kyoto, Japan). The nanofilled resin composites used in this study are listed in Table 1 with their associated lot numbers, components, and the percentage of filler loading provided by the manufacturers.

Table 1: *Nanofilled Resin Composites*

Nanofilled Resin Composite (Code, Shade)	Resin Matrix Composition	Inorganic Filler (Content)	Manufacturer (Lot No.)
Filtek Supreme Ultra Universal Restorative (FS, A2)	Bis-GMA, UDMA, TEGDMA, Bis-EMA, PEGDMA	Nonagglomerated/nonaggregated silica filler, nonagglomerated/nonaggregated zirconia filler, aggregated zirconia/silica cluster filler (78.5wt%, 63.3vol%)	3M ESPE, St Paul, MN, USA (N697117)
MI Gracefil (MG, A2)	Bis-EMA, UDMA	Silica glass, strontium glass, fluoro-alumino-silicate glass (77.0 wt%, 65.0 vol%)	GC, Tokyo, Japan (1407281)
TPH Spectra High Viscosity (TS, A2)	Urethane modified Bis-GMA, TEGDMA	Silanated barium-alumino-borosilicate glass, silanated barium boron fluoro alumino silicate glass (77.2wt%, 57.0vol%)	DENTSPLY Caulk, Milford, DE, USA (150317)
Clearfil Majesty ES-II (CM, A2)	Bis-GMA	Silanated barium glass filler, (78.0wt%, 66.0vol%)	Kuraray Noritake Dental, Tokyo, Japan (A90026)
Estelite Σ Quick (EQ, A2)	Bis-GMA, TEGDMA	Silica zirconia filler (82.0wt%, 71vol%)	Tokuyama Dental, Tokyo, Japan (13394)
Beautifil II (B2, A2)	Bis-GMA, TEGDMA	Fluoro boro alumino silicate glass (83.3wt%, 68.6vol%)	SHOFU, Kyoto, Japan (011592)
Abbreviations: Bis-GMA, bisphenol A glycidyl methacrylate; UDMA, urethane dimethacrylate; Bis-EMA, ethoxylated bisphenol A dimethacrylate; TEGDMA, triethylene glycol dimethacrylate; Bis-MEPP, bisphenol A ethoxylate dimethacrylate.			

Specimen Preparation

Forty wear specimens of each of the six resin composites were prepared for simulated localized wear (occlusal contact area [OCA] wear), and 40 wear specimens of each material were prepared for generalized wear (contact-free area [CFA] wear). Custom stainless-steel fixtures were machined for localized wear testing with a cylindrical cavity 6.5 mm in diameter and 4.0 mm in depth. Stainless-steel fixtures for generalized wear testing were machined with a cylindrical cavity 4.5 mm in diameter and 4.0 mm in depth. Two increments of the resin composites (approximately 2.0 mm in thickness) were placed in the cylindrical-shape cavities of the localized and generalized fixtures, and each increment was cured for 40 seconds using a quartz-tungsten halogen unit (Spectrum 800, DENTSPLY Caulk) set at 600 mW/cm². The specimens for localized and generalized wear testing were randomly allocated into two groups (n=20 per group): 1) specimens stored in 37°C distilled water for 24 hours (24-hour group) and 2) specimens stored in 37°C distilled water for 24 hours and thermal cycled for 10,000 cycles (TC group). After 24 hours of water storage, all the composite surfaces were wet polished flat to a 4000-grit surface using a sequence of silicon carbide papers (Struers, Cleveland, OH, USA). For the TC group, thermal cycling was conducted using a custom-built apparatus, and each cycle consisted of water bath incubation for 30 seconds between water baths of 5°C and 55°C with a transfer time of five seconds for 10,000 cycles.

Wear Simulation

A Leinfelder-Suzuki (Alabama) device was used for wear simulation. The simulator has a plastic water bath, and the custom wear fixtures were mounted inside the four-station bath. A brass cylinder was then placed around each fixture in the bath to serve as a reservoir for the abrasive media (ie, a water slurry of unplasticized polymethyl methacrylate [PMMA] with an average particle size of 44 μ m). The media was placed inside the brass cylinders to cover the surface of the resin composite in the custom fixtures. The water slurry of PMMA inside the brass cylinders was approximately 6.0 mm in thickness over the surface of the resin composite.

Two different wear antagonists were used in this study. For the localized (OCA) wear simulation, a stainless-steel ball bearing (2.387-mm radius) was mounted inside a collet assembly. The antagonist for the generalized (CFA) wear simulation was a stainless-steel cylinder (6.5 mm in diameter) with a flat-end stylus tip. The antagonist tips were mounted on spring-loaded pistons to deliver the wear challenges. During the application of the load, the antagonists rotated approximately 30 degrees as the maximum force was reached (maximum load of 78.5 N at a rate of 2 Hz) and then counterrotated back to the original starting position as the load was relaxed to complete the cycle. Each set of specimens was exposed to 400,000 cycles in the wear simulator.

Wear Measurements

Prior to wear simulation, each resin composite specimen was profiled using a Proscan 2100 noncontact optical profilometer (Scantron Industrial Products, Taunton, UK) with Proscan software. These profiles provided the pretest digitized contours (20 specimens for each of the six resin composite materials for both localized and generalized wear testing).

After the 400,000 wear cycles, the specimens were ultrasonically cleaned (L&R T-14B solid-state ultrasonic cleaner, L&R Manufacturing Company, South Orange, NJ, USA) in distilled water for three minutes and then profiled again using the Proscan 2100 unit. The X, Y, and Z coordinates of the before and after scans were exported from the Proscan software to another computer for analysis using AnSur 3D software (Minnesota Dental Research Center for Biomaterials and Biomechanics, University of Minnesota, Minneapolis, MN, USA).

Facet depth and volume loss measurements were determined from differences between the before and after data sets. A computerized fit was completed using the before and after data sets in AnSur 3D, and volume loss (VL; mm^3) was then determined for both localized and generalized wear simulation for each of the six resin composites. The maximum depth of the wear facet (MXD; μm) was also determined for the localized wear specimens, and the mean depth (MD; μm) of the wear facet was determined for the generalized wear specimens.

Scanning Electron Microscopic Observations of Resin Composite Surfaces

Ultrastructural observations were conducted on the polished surfaces of the nanofilled resin composites after argon-ion etching. Three specimens per group were observed using field-emission scanning electron microscopy (SEM; ERA 8800FE, Elionix, Tokyo, Japan).

The surfaces of the nanofilled resin composites were wet polished with a sequence of SiC papers (#600, #1200, and #4000 grit) using a grinder-polisher (Ecomet 4, Buehler). The surfaces were then polished with abrasive discs (Fuji Star Type DDC, Sankyo-Rikagaku, Saitama, Japan) followed by a series of diamond pastes down to 0.25- μm particle size (DP-Paste, Struers, Ballerup, Denmark) to bring the surfaces to a high gloss. SEM specimens of the polished surfaces were dehydrated by immersion in aqueous *tert*-butanol solutions with ascend-

ing concentrations (50% for 20 minutes, 75% for 20 minutes, 95% for 20 minutes, and 100% for two hours) and were then transferred to a critical-point dryer (Model ID-3, Elionix, Tokyo, Japan) for 30 minutes. These polished surfaces were etched for 30 seconds using an argon ion-beam (EIS-200ER, Elionix, Tokyo, Japan) directed perpendicular to the surface at an accelerating voltage of 1.0 kV and an ion current density of 0.4 mA/cm^2 to enhance the visibility of the filler particles. Surfaces were coated with a thin film of gold in a vacuum evaporator (Quick Coater SC-701, Sanyu Electron, Tokyo, Japan). SEM observations were carried out using an operating voltage of 10 kV.

SEM Observations of Wear Facets

Ultrastructural observations were carried out on representative wear facets of the nanofilled resin composites of the 24-hour group and the TC group after 400,000 cycles for both localized and generalized wear simulation using SEM (TM3000 tabletop microscope, Hitachi-High Technologies, Tokyo, Japan). After the wear analysis, representative specimens were coated with a thin film of gold-palladium in a vacuum evaporator (Emitech SC7620 Mini Sputter Coater, Quorum Technologies, Ashford, UK). The SEM observations were carried out using an operating voltage of 15 kV.

Statistical Analysis

The VL for localized and generalized wear, MXD for localized wear, and MD for generalized wear of the nanofilled resin composites were analyzed using a commercial statistical software package (SPSS Statistics Base, International Business Machines, Armonk, NY, USA). A two-way analysis of variance (ANOVA) and a Tukey *post hoc* test were used for data analysis of each data set, with a significance level of $\alpha = 0.05$.

RESULTS

Localized Wear Simulation

The two-way ANOVA of VL and MXD of wear facets showed a significant effect ($p < 0.05$) for the factors of material type and thermal cycling. No significant interaction ($p > 0.05$) was found between these factors for either VL or MXD.

The results for the simulated localized wear of nanofilled resin composites are shown in Tables 2 and 3. The VL and MXD of wear facets in both the 24-hour group and the TC group were significantly different ($p < 0.05$) depending on the material, and

Table 2: Results for Simulated Localized Wear of Resin Composites^a

Material	Volume Loss (VL, mm ³)		Maximum Depth of Wear Facets (MXD, μm)	
	24 h	10,000 TCs	24 h	10,000 TCs
FS	0.034 (0.012) aA	0.048 (0.009) aB	110.6 (13.5) aA	139.4 (14.5) aA
EQ	0.040 (0.015) aA	0.065 (0.017) abB	125.6 (12.2) abA	155.7 (11.1) bB
MG	0.045(0.012) abA	0.058 (0.010) abB	138.8 (18.0) bA	152.6 (10.7) bA
BII	0.057 (0.018) bA	0.084 (0.017) cB	144.9 (15.7) bcA	178.9 (12.6) cA
TS	0.058 (0.020) bA	0.076 (0.016) bcB	148.4 (15.5) bcA	165.8 (10.5) cA
CM	0.059 (0.021) bA	0.089 (0.025) cB	151.5 (14.7) cA	179.5 (12.8) cA

Abbreviations: BII, Beautifil II; CM, Clearfil Majesty ES-II; EQ, Estelite Σ Quick; FS, Filtek Supreme Ultra Universal Restorative; MG, MI Gracefil; TS, TPH Spectra High Viscosity.
^a Values in parentheses are standard deviations (n=20). Same small letter in same vertical column indicates no significant difference (p>0.05). Same capital letter within individual rows indicates no significant difference (p>0.05).

the VL and MXD of wear facets of the TC group were significantly greater ($p<0.05$) than those of the 24-hour group.

Generalized Wear Simulation

The two-way ANOVA of VL and MD of wear facets showed a significant effect ($p<0.05$) for the factor of material type. No significant effect ($p>0.05$) was found for the factor of thermal cycling or for the interaction of material type and thermal cycling for either VL or MD.

The results for the simulated generalized wear of nanofilled resin composites are presented in Tables 4 and 5. The VL and MD of wear facets for both the 24-hour group and the TC group showed significant differences ($p<0.05$) among materials when comparing the wear within each group. However, the VL and MD of wear facets of the TC group were, in general, not significantly different ($p>0.05$) from those of the 24-hour group. The exception was EQ, for which both VL and MD were significantly greater ($p<0.05$) for the TC group than for the 24-hour group.

SEM Observations of Resin Composite Surfaces

Representative SEM images of polished nanofilled resin composite surfaces with argon-ion etching are shown in Figure 1. The argon-ion etching revealed clear differences in filler particle size, shape, and distribution. Resin composites exhibited a wide variety of filler particle sizes and shapes.

SEM images of polished FS surfaces with argon-ion etching showed a wide size range (1 to 8 μm) of irregular particles and small irregular particles. SEM images of polished MG surfaces with argon-ion etching showed a wide size range (1 to 2 μm) of irregular particles and small irregular particles. SEM images of polished EQ surfaces with argon-ion etching showed uniform small spherical particles. SEM images of polished CM surfaces with argon-ion etching showed a wide size range (1 to 15 μm) of irregular particles and small irregular particles. SEM images of polished TS surfaces with argon-ion etching showed a wide size range (1 to 4 μm) of irregular particles and small irregular particles, and filler particle plucking was clearly observed. SEM images of polished B2 surfaces with argon-ion etching

Table 3: Results for Simulated Generalized Wear of Resin Composites^a

Material	Volume Loss (VL, mm ³)		Mean Facet Depth (MD, μm)	
	24 h	10,000 TCs	24 h	10,000 TCs
FS	0.284 (0.082) aA	0.296 (0.095) aA	21.6 (5.9) aA	24.5 (8.5) aA
EQ	0.269 (0.075) aA	0.432 (0.115) bB	24.6 (5.7) aA	38.6 (10.4) bB
MG	0.346 (0.066) abA	0.391 (0.082) abA	31.7 (6.1) bA	34.6 (7.7) bA
BII	0.402 (0.117) bcA	0.412 (0.107) abA	31.5 (8.9) bA	33.9 (9.6) bA
TS	0.520 (0.094) cA	0.501 (0.080) bcA	38.5 (6.5) bcA	37.4 (6.5) bA
CM	0.553 (0.153) cA	0.589 (0.141) cA	40.2 (7.3) cA	44.5 (9.8) bcA

Abbreviations: BII, Beautifil II; CM, Clearfil Majesty ES-II; EQ, Estelite Σ Quick; FS, Filtek Supreme Ultra Universal Restorative; MG, MI Gracefil; TS, TPH Spectra High Viscosity.
^a Values in parentheses are standard deviations (n=20). Same small letter in same vertical column indicates no significant difference (p>0.05). Same capital letter within individual rows indicates no significant difference (p>0.05).

Table 4: Simulated Localized Wear of Resin Composites -Volume Loss (VL, mm³)

Material	24h group	TC group
FS	0.034 (0.012) ^{a,A}	0.048 (0.009) ^{a,B}
EQ	0.040 (0.015) ^{a,A}	0.065 (0.017) ^{a,b,B}
MG	0.045 (0.012) ^{a,b,A}	0.058 (0.010) ^{a,b,B}
B2	0.057 (0.018) ^{b,A}	0.084 (0.017) ^{c,B}
TS	0.058 (0.020) ^{b,A}	0.076 (0.016) ^{b,c,B}
CM	0.059 (0.021) ^{b,A}	0.089 (0.025) ^{c,B}

Values in parenthesis are standard deviations (n = 20). Same small letter in same vertical column indicates no significant difference ($p > 0.05$). Same capital letter within individual rows indicates no significant difference ($p > 0.05$).

showed a wide size range (1 to 10 μm) of irregular particles and small irregular particles.

SEM Observations of Wear Facets

Representative SEM images of wear facets after simulated localized wear of the 24-hour group and the TC group are shown in Figure 2. Low-magnification SEM images show that the localized wear facets of the nanofilled resin composites of the TC group were larger than those of the 24-hour group, and high-magnification SEM images showed that wear facets of the TC group exhibit more filler particle plucking or cracking than do those of the 24-hour group.

Representative SEM images of wear facets of the 24-hour group and the TC group after simulated generalized wear are shown in Figure 3. SEM images of the generalized wear facets of the nanofilled resin composites, except those of EQ, did not show any clear difference between the wear of the 24-hour group and the TC group. The generalized wear facets of EQ revealed severe cracking and damage for the TC group compared to the 24-hour group.

DISCUSSION

Typically, the wear resistance of restorative materials is evaluated by laboratory methods that involve an electric device that repeatedly makes contact with the material being tested using an antagonist object, such as a stylus.¹⁷ In 2001, the International Organization for Standardization (ISO) published a technical specification, "Wear by Two- and/or Three-Body Contact," describing eight laboratory methods.²⁰ One of the methods identified by the ISO publication was the Alabama method, which was developed at the University of Alabama.²¹ Barkmeier and others²² have developed two distinct types of wear simulation using the Alabama wear simulation

Table 5: Simulated Localized Wear of Resin Composites Maximum depth of wear facets (MXD, μm)

Material	24h group	TC group
FS	110.6 (13.5) ^{a,A}	139.4 (14.5) ^{a,A}
EQ	125.6 (12.2) ^{a,b,A}	155.7 (11.1) ^{b,B}
MG	138.8 (18.0) ^{b,A}	152.6 (10.7) ^{b,A}
B2	144.9 (15.7) ^{b,c,A}	178.9 (12.6) ^{c,A}
TS	148.4 (15.5) ^{b,c,A}	165.8 (10.5) ^{c,A}
CM	151.5 (14.7) ^{c,A}	179.5 (12.8) ^{c,A}

Values in parenthesis are standard deviations (n = 20). Same small letter in same vertical column indicates no significant difference ($p > 0.05$). Same capital letter within individual rows indicates no significant difference ($p > 0.05$).

device. One of these is simulated localized wear (the type of wear represented by direct tooth to material contact), and the other is simulated generalized wear (the type of wear generated by a food bolus during mastication). Barkmeier and others²² also reported test methods for determining simulated localized and generalized wear with the Alabama machine using a Proscan 2100 noncontact profilometer in conjunction with AnSur 3D software for wear analysis. In the present study, measurements of simulated wear of resin composites were conducted according to these methods.

Nanofilled resin composites incorporating a larger amount of nanosized filler particles in a more homogeneous distribution in the resin matrix have a larger interface area between fillers and a resin matrix than do microhybrid resin composites.²³ Thus, the total interfacial stress between filler and resin matrix generated by intraoral conditions in nanofilled resin composite has been estimated to be greater than that in microhybrid resin composites.²⁴ As some but not all microhybrid resin composites showed reduced wear resistance after thermal cycling,¹⁶ it is important to confirm the effect of such treatment on nanofilled resin composites.

The simulated localized and generalized wear of resin composites of the 24-hour group was found to be significantly different depending on the material.

Bayne and others²⁵ have reported that the mean filler particle size affects the simulated wear of resin composites and that resin composites that include smaller filler particles exhibit less simulated wear. The results of the present study are consistent with this observation to a certain extent. EQ, which contains smaller spherical filler particles (less than 1.0 μm), exhibited less simulated wear in the 24-hour group, whereas the wear of CM (1 to 15 μm), which includes a wider size range of larger filler particles,

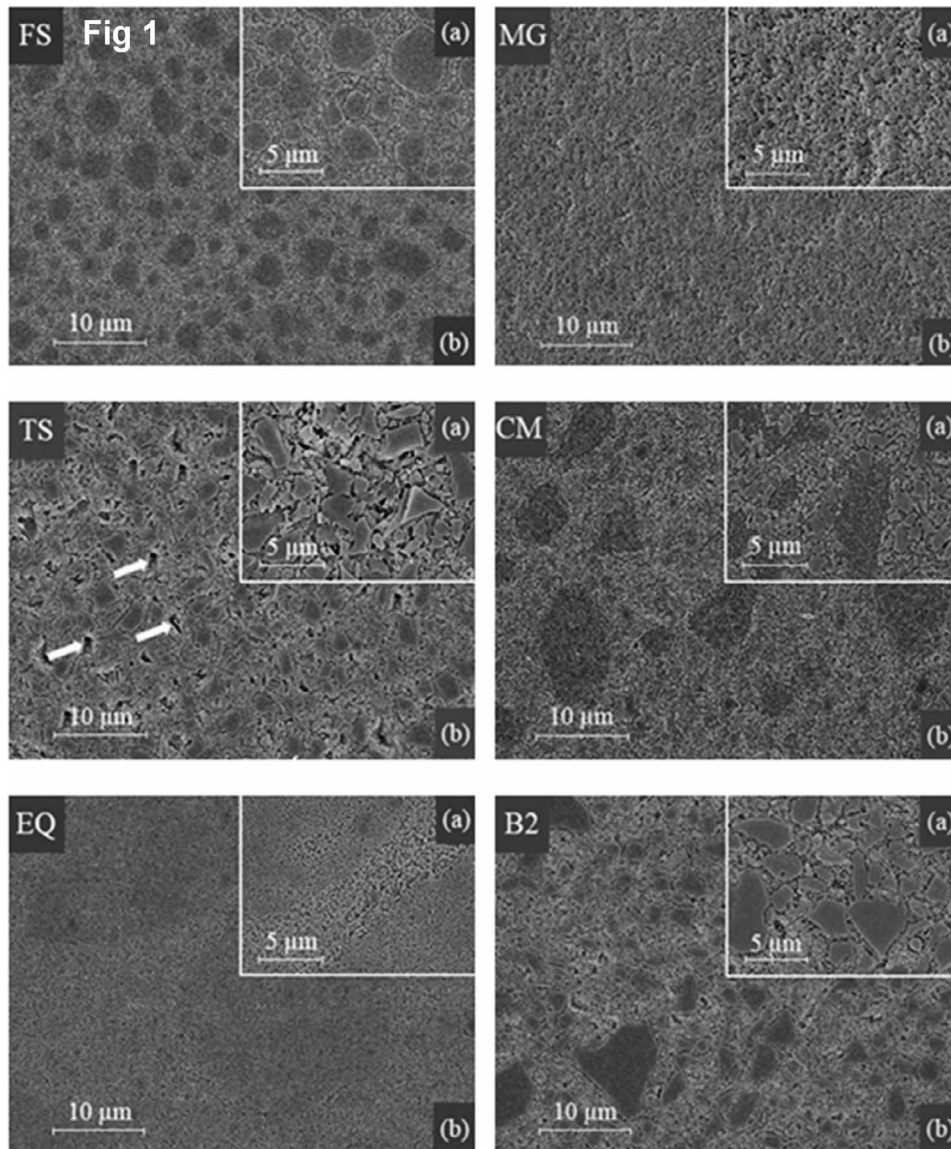


Figure 1. Representative scanning electron microscopic (SEM) images of the polished resin composite surfaces at 10,000 $\times$ magnification (a) and 2500 $\times$ magnification (b). FS: wide size range (1 to 8 μm) of irregular-shaped particles and small irregular-shaped particles; MG: wide size range (1 to 2 μm) of irregular-shaped particles (black arrow) and small irregular-shaped particles (white arrow); TS: wide size range (1 to 4 μm) of irregular-shaped particles and small irregular-shaped particles; CM: wide size range (1 to 15 μm) of irregular-shaped particles and small irregular-shaped particles; EQ: uniform small spherical particles; B2: wide size range (1 to 10 μm) of irregular-shaped particles and small irregular-shaped particles. Size and distribution of fillers of nanofilled resin composites differed depending on the material. Filler particle plucking was notably observed in TS (white arrows).

was greater regardless of the testing method. The difference in mean filler particle size between EQ and CM was clearly visible in SEM observations of their polished and argon-ion-etched surfaces. The difference in wear between these materials has been hypothesized to result from the smaller interparticle spacing between the small filler particles. The smaller filler particles are more closely packed; thus, the resin matrix between them is protected from further wear.²⁶ By contrast, when larger filler particles are dislodged from the resin composite surfaces, a void is produced, and the underlying resin matrix is exposed to the wear challenges.²⁷ In addition, the dislodged particles may act as a further abrasive to the resin composite surface, resulting in increased wear.²⁸

On the other hand, TS, which includes relatively small filler particles (1 to 4 μm), exhibited significantly greater simulated localized and generalized wear than MG (1 to 2 μm) and SUFS (1 to 8 μm) and was similar to CM (1 to 15 μm) in both the localized and the generalized wear simulation and to B2 (1 to 10 μm) in localized wear simulation. Therefore, in this study, mean filler size did not directly influence the simulated localized and generalized wear of resin composites. As simulated wear is affected by many factors, it is not surprising that the association with a single factor is relatively weak. For instance, in SEM observations, TS clearly exhibited filler particle plucking not only in the worn surfaces of wear simulation specimens but also on polished surfaces after argon-ion etching. Although wear simulation

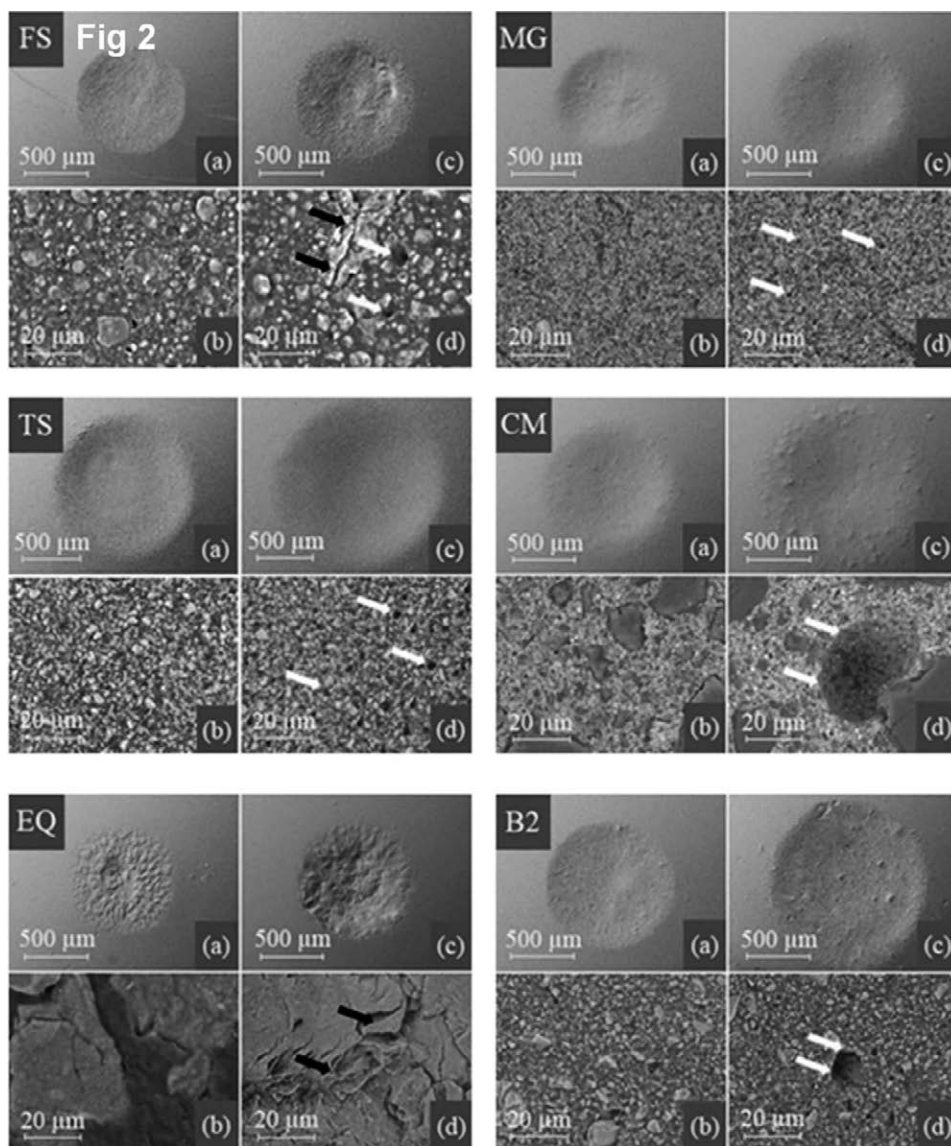


Figure 2. Representative scanning electron microscopic (SEM) images of the localized wear facets of nanofilled resin composites after 24 hours of water storage, as viewed at 100 $\times$ magnification (a) and 2500 $\times$ magnification (b) and after 24 hours of water storage and thermal cycling at 100 $\times$ magnification (c) and 2500 $\times$ magnification (d). These low-magnification SEM images show that the localized wear facets of nanofilled resin composites after thermal cycling were larger than those after 24 hours of water storage. These high-magnification images show that wear facets after thermal cycling exhibit more filler particle plucking or cracking than those of 24 hours of water storage.

specimens of other resin composites also showed filler particle plucking, filler particle plucking was not clearly observed after argon-ion etching of the polished surfaces for any tested resin composites apart from TS. One possibility is that weaker interfacial bonding between the filler and the resin matrix, which is associated with the surface treatment of the fillers, may be responsible for TS showing greater simulated wear. In addition, filler load has been reported to play a particularly important role in the wear resistance of resin composites, and a higher filler load has been reported to reduce the level of wear.²⁹ The values for filler load provided by the manufacturers vary greatly among the tested materials (Table 1). This may be another reason why the mean filler size did

not directly determine the simulated wear of resin composites.

Although the simulated localized wear of tested resin composites in the TC group was significantly greater than that of the resin composites in the 24-hour group, the simulated generalized wear of resin composites of the 24-hour group and the TC group surprisingly did not show a significant difference, except in the case of ES. SEM observations of the worn surfaces of the localized wear simulation specimens in the TC group tended to indicate more cracking and filler particle plucking than was observed for corresponding specimens in the 24-hour group, whereas the generalized wear simulation specimens did not show any clear difference between wear in the 24-hour group and the TC group. In

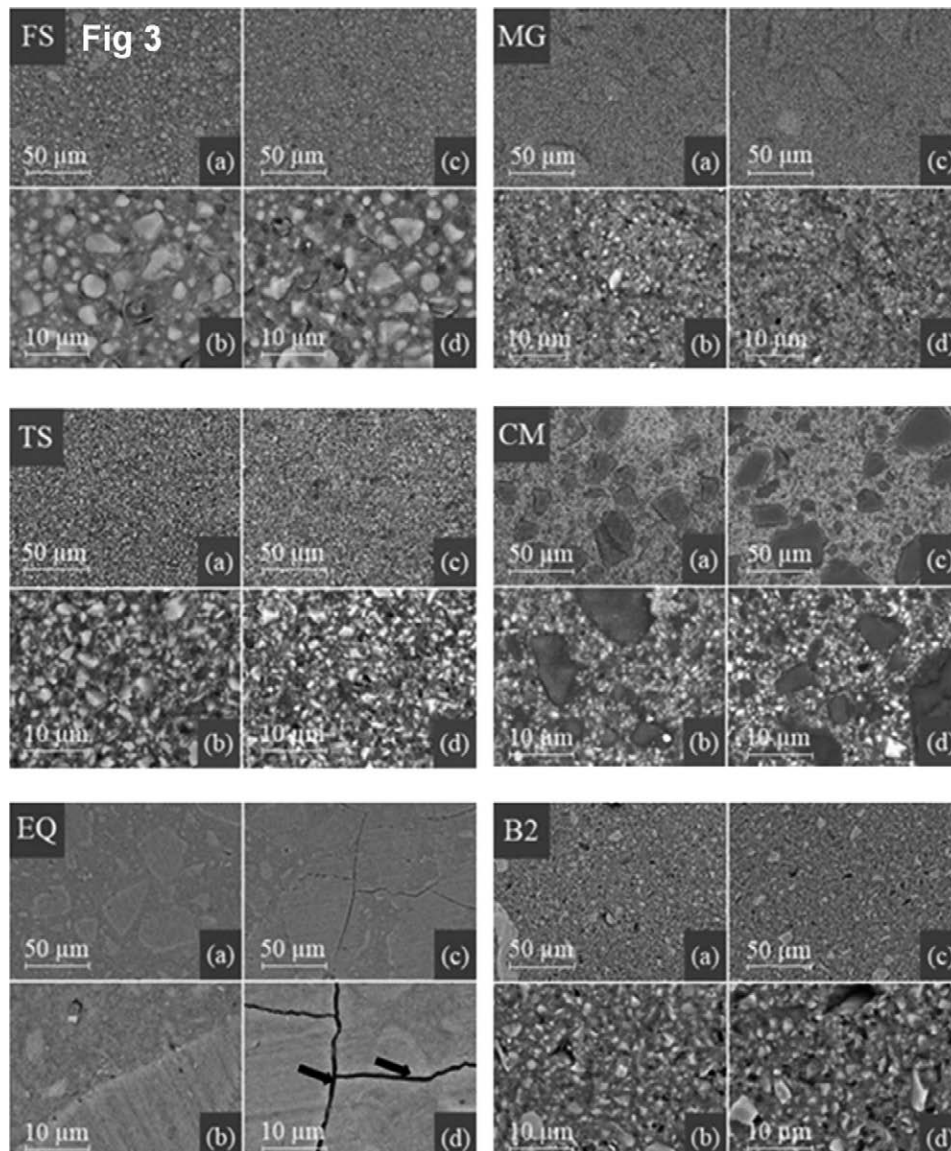


Figure 3. Representative scanning electron microscopic (SEM) images of the generalized wear facets of nanofilled resin composites after 24 hours of water storage, as viewed at 1000 $\times$ magnification (a) and 5000 $\times$ magnification (b) and after 24 hours of water storage and thermal cycling at 1000 $\times$ magnification (c) and 5000 $\times$ magnification (d). SEM images of the generalized wear facets of nanofilled resin composites, except EQ, did not show any clear difference between wear after 24 hours of water storage and after thermal cycling. SEM images of the generalized wear facets of EQ after thermal cycling revealed severe cracking and damage compared to worn surfaces after 24 hours of water storage.

addition to the thermal stresses introduced by differential thermal expansion between the filler and the matrix, immersion in water causes water penetration into the resin matrix of the resin composite, resulting in softening of the polymer matrix.³⁰ However, long-term thermal cycling may positively affect the resin composites and improve their mechanical properties with the resultant postcuring effects of time and heat.³¹ The material degradation associated with the cyclic temperature changes and immersion in water of resin composites may be mitigated by the postcuring process. However, localized wear simulation of restorative materials appears to be much more aggressive than generalized wear simulation. Simulated localized wear facets may be more than twice as deep as the

generalized facet depth because of accelerating filler plucking and the loss of matrix resin.³² Therefore, material degradation of both the filler-resin matrix interface and the resin matrix itself caused by localized stress appeared to more directly influence the results of simulated localized wear of the resin composites in the TC group when compared with the results of simulated generalized wear.

The one exception in the aforementioned experiments was EQ, in which wear in the TC group was significantly greater than that of the 24-hour group regardless of the testing method. Similarly, SEM observations of the worn surface of wear simulation specimens of EQ in the TC group showed severe cracking and damage when compared with the 24-hour group. One possible explanation is that the

increase in surface area of particles that accompanies the reduction in filler size in EQ results in a material more prone to water uptake, which might negatively affect mechanical properties as previously reported.³ In addition, in connection with the larger surface area of filler particles, silanated fillers are susceptible to hydrolytic degradation,⁶ which might adversely influence the filler-resin matrix interfaces of EQ over time.

On the basis of the results of this study, the first null hypothesis that no significant differences exist between simulated localized and generalized wear of nanofilled resin composites is rejected. The second null hypothesis was rejected for the simulated localized wear of nanofilled resin composites but is not rejected for the generalized wear of most nanofilled resin composites.

Overall, the simulated localized and generalized wear of the resin composites tested in this study appeared to be material dependent. Furthermore, the simulated localized wear of the resin composites was influenced by the thermal stress associated with thermal cycling, although such an influence was not as apparent in generalized wear simulation. However, several testing methodologies for laboratory simulation of wear are currently available, and these other methods may lead to substantially different results.³³ The results from other wear testing methods should therefore be carefully considered when attempting to discern the wear susceptibility of resin composites. Further *in vitro* wear tests combined with thermal cycling are important as initial evaluation methods of resin composites, especially for new products, to get an estimate of their clinical performance.

CONCLUSION

The results of this study indicate that the simulated localized and generalized wear of nanofilled resin composites is different, depending on the material. In addition, the simulated localized wear of nanofilled resin composites was influenced by thermal cycling, but the simulated generalized wear of most nanofilled resin composites was not. It appears that thermal cycling has potentially detrimental consequences for nanofilled resin composite restorations, particularly in occlusal contact areas. While this does not mean that such composites cannot be used in high-stress-bearing areas, it is important for clinicians to bear in the mind the risk of wear and to routinely check the restorations to determine whether repair is necessary.

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 Nihon University School of Dentistry.

Conflict of Interest

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

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Effect of Different Adhesive Systems Used for Immediate Dentin Sealing on Bond Strength of a Self-Adhesive Resin Cement to Dentin

RC Ferreira-Filho • C Ely • RC Amaral • JA Rodrigues • J-F Roulet • A Cassoni • AF Reis

Clinical Relevance

Immediate dentin sealing (IDS) has been suggested in order to reduce postoperative sensitivity. Although IDS tends to enhance initial bond strength values, IDS does not prevent a decrease in bond strength values after three months of storage in water.

SUMMARY

Objective: The purpose of this study was to investigate the immediate and three-month water storage behavior of adhesives when used for immediate dentin sealing (IDS).

Ramiro Chavasco Ferreira-Filho, MS, Department of Operative Dentistry, Guarulhos University, Guarulhos, Brazil

Caroline Ely, DDS, MS, PhD, Department of Operative Dentistry, Guarulhos University, Guarulhos, Brazil

Roberto C Amaral, DDS, MS, Dental Materials and Restorative Dentistry, University of Oeste of Santa Catarina, Joacaba, Brazil

José Augusto Rodrigues, DDS, MS, PhD, Department of Restorative Dentistry, Guarulhos University, Guarulhos, Brazil

Jean-Francois Roulet, DDS, PhD, College of Dentistry, University of Florida, Gainesville, FL, USA

Alessandra Cassoni, PhD, Department of Restorative Dentistry, Guarulhos University, Guarulhos, Brazil

*Andre F Reis, DDS, MS, PhD, Department of Operative Dentistry, Guarulhos University, Guarulhos, Brazil

*Corresponding author: Praça Tereza Cristina, 229, Guarulhos, Sao Paulo 07023-070, Brazil; e-mail: reisandre@yahoo.com

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Methods and Materials: Four adhesive systems were used to perform IDS: a one-step self-etch (Xeno V), a two-step self-etch (Clearfil SE Bond), a two-step etch-and-rinse (XP Bond), and a three-step etch-and-rinse (Optibond FL). For the control group, IDS was not performed. The self-adhesive resin cement RelyX Unicem was used for the luting procedures. After seven days of water storage, specimens (n=6) were sectioned into beams (n=5) with an approximately 1-mm² cross-sectional area. Half of the specimens were tested in tension after seven days of water storage at 37°C, while the other half was stored for three months prior to testing in tension using a universal testing machine (1 mm/min). The failure pattern was determined using a stereomicroscope and scanning electron microscopy. Microtensile bond strength (μ TBS) data were statistically analyzed by two-way analysis of variance and Tukey *post hoc* test ($\alpha=0.05$).

Results: After seven days, the control group presented the lowest μ TBS but did not differ from XP Bond and Clearfil SE Bond. After three months, there was no μ TBS difference between the IDS groups and the control.

Conclusions: After seven days of water storage, the groups with IDS presented higher μ TBS values than the control group, although XP Bond and Clearfil SE Bond did not present significant differences. However, after three months of storage in water, IDS groups did not differ significantly from control group, which did not receive IDS.

INTRODUCTION

The multistep adhesive cementation technique is considered complex and sensitive.^{1,2} Dentin hypersensitivity is a common symptom reported by patients after cementation procedures and can be associated with several factors, such as overheating and desiccation during tooth preparation, bacterial infiltration, and the fluid movement through dentinal tubules.³

Self-adhesive luting agents do not require any pretreatment of the tooth surface and were developed in an attempt to simplify bonding procedures and reduce the shortcomings of the conventional multistep adhesive cementation technique.^{4,5} Their application on smear layer-covered substrates keep dentin permeability at very low levels,⁶ contributing to reduced postoperative sensitivity and lower susceptibility to moisture degradation.⁷

Immediate dentin sealing (IDS) has been suggested to reduce postoperative sensitivity and bacterial infiltration while contributing to improved bond strength of indirect restorative procedures.^{3,8-10} According to previous reports, patients treated with the IDS technique experienced improved comfort during the provisional restoration stage.^{8,11}

In this technique, the dentin is hybridized using either a two-step self-etching or a three-step etch-and-rinse adhesive system (hydrophobic resin covering the primer layer) immediately after preparation and before impression taking, contributing to a reduction of dentin permeability.¹²⁻¹⁴ Simplified etch-and-rinse and self-etching adhesive systems are widely available for clinicians; however, their efficacy in the IDS technique has not been reported so far.

The null hypotheses evaluated in this study were 1) that there is no difference in the bond strength produced by the different adhesives in the IDS technique and 2) that storage in water for three months does not affect the bond strength.

METHODS AND MATERIALS

Four adhesive systems were used in this study: two etch-and-rinse: the three-step Optibond FL (Kerr

Corporation, Orange, CA, USA) and the two-step XP Bond (Dentsply De Trey, Konstanz, Germany); two self-etching: the two-step Clearfil SE Bond (Kuraray Medical, Okayama, Japan) and the one-step Xeno V (Dentsply De Trey). RelyX Unicem (3M ESPE, St Paul, MN, USA) was used as the self-adhesive resin luting agent. Materials, compositions, and directions for use are shown in Table 1.

Teeth were randomly assigned to five experimental groups: four according to the adhesive system used for IDS and one control group (without IDS).

Tooth Preparation

Sixty caries-free third molars were used after the protocol was approved by the review board. After disinfection and removal of soft tissues, flat middle-depth coronal dentin surfaces were exposed using 600-grit SiC paper (3M of Brazil Ltd, Sumare, Brazil) under running water to create a standardized smear layer. Teeth had their roots removed 2 mm below the cemento–enamel junction using a diamond saw (Isomet, Buehler, Lake Bluff, IL, USA). Teeth assigned to the control group were then stored in water for seven days before cementation procedures. All other groups received immediate dentin sealing as described below.

IDS

The four adhesive systems evaluated in this study were applied according to the manufacturers' instructions to perform IDS. After initial light curing (Radii Cal Plus, SDI, Victoria, Australia) for 10 seconds (1500 mW/cm²), the adhesive layer was covered by a layer of glycerin gel (KY gel, Johnson & Johnson do Brasil, Sao Paulo, Brazil) and light polymerized for 20 seconds to avoid the oxygen inhibition layer. After removing the gel using copious irrigation, the teeth were stored in water at 37°C for seven days before luting procedures.

Luting Procedures for Microtensile Bond Strength

Four-millimeter-thick composite resin discs with a 12-mm diameter were prepared by layering 2-mm-thick increments of a microhybrid composite resin (Filtek Z250, shade A1; 3M ESPE) into a silicone mold. Each increment was light activated for 40 seconds using a Radii Cal Plus (1500 mW/mm²) light curing unit. One side of the composite resin discs was abraded with 600-grit SiC paper under water cooling to create a flat surface with standardized roughness for cementation. The composite surface

Table 1: Classification, Manufacturer, Lot Number, and Composition of Adhesive Systems, Luting Agent, and Composite Resin Used in This Study

Type	Manufacturer (Lot Number)	Composition
Self-adhesive resin cement	RelyX Unicem 3M ESPE (366321)	Base paste (white): methacrylate monomers containing phosphoric acid groups, methacrylate monomers, silanated fillers, initiator components, stabilizers Catalyst paste (yellow): methacrylate monomers, alkaline (basic) fillers, initiators components, stabilizers, pigments
One-step self-etching adhesive	Xeno V Dentsply De Trey (0906000336)	Bifunctional acrylate, acidic acrylate, functionalized phosphoric acid ester, acrylic acid, water, tertiary butanol, initiator, stabilizer
Two-step self-etching adhesive	Clearfil SE Bond Kuraray Medical (primer, 01065A; bond, 01585A)	Primer: 10-MDP, HEMA, DMA, catalyst, water Bonding: 10-MDP, HEMA, DMA, Bis-GMA, filler, catalyst
Two-step etch-and-rinse adhesive	XP Bond Dentsply De Trey (1011000654)	34% phosphoric acid conditioner gel Adhesive: TCB resin, PENTA, UDMA, TEGDMA, HEMA, stabilizer, EDAB, CQ, functionalized amorphous silica, t-butanol
Three-step etch-and-rinse adhesive	Optibond FL Kerr Corp (primer: 3462545; bond: 3586282)	37.5% phosphoric acid conditioner gel Primer: HEMA, GPDM, MMEP, ethanol Bonding: HEMA, MPS, 2-hydroxy-1,3-propanediyl bismethacrylate, disodium hexafluorosilicate
Composite Resin	Filtek Z250 3M ESPE (N2580)	UDMA, Bis-EMA, Bis-GMA, silica/zirconia filler (average particle size of 0.6 μm)

Abbreviations: Bis-EMA, ethoxylated bisphenol A dimethacrylate; Bis-GMA, bisphenol-glycidyl methacrylate; CQ, camphorquinone; DMA, dimethacrylate; EDAB, ethyl-4-dimethylamino benzoate; GPDM, glycerol phosphate dimethacrylate; HEMA, 2-hydroxyethyl methacrylate; MMEP, mon-2-methacryloxyethyl phthalate; MPS, 3-methacryloxypropyltrimethoxysilane; PENTA, phosphonated penta-acrylate ester; TCB resin, tetracarboxylic acid modified dimethacrylate; TEGDMA, triethylene glycol dimethacrylate; 10-MDP, 10-methacryloyloxydecyl dihydrogen phosphate.

was airborne-particle abraded with 50- μm aluminum oxide particles for 10 seconds. Before the luting procedures were performed, the composite resin discs were ultrasonically cleaned in distilled water for 10 minutes, rinsed with running water, air-dried, and silanated (RelyX Ceramic Primer, 3M ESPE).

After silanization, the luting procedures were performed according to the manufacturers' instructions. The composite resin discs were pressed on the cement using proper digital pressure, which was sustained until light curing was performed from the buccal and lingual sides. The cementation procedures were randomly processed. Specimens were light activated for 40 seconds from the buccal, lingual, and occlusal directions. Bonded specimens were stored in distilled water at 37°C for seven days.

Microtensile Bond Strength Evaluation

Seven days after the cementation procedures, the restored teeth were serially sectioned perpendicular to the adhesive-tooth interface into slabs and the slabs into beams ($n=5$) with a cross-sectional bonded area of approximately 1 mm^2 using a diamond saw (Isomet 1000, Buehler Ltd). Ten beams were selected

for testing for each tooth. Half of the beams were fixed to the grips of a universal testing machine (EZ Test, Shimadzu Corp, Kyoto, Japan) using a cyanoacrylate adhesive (Loctite Super Bonder Gel, Henkel, Düsseldorf, Germany) and tested in tension at a crosshead speed of 1 mm/min until fracture occurred. The other half was stored in water at 37°C for three months prior to microtensile testing. Maximum tensile load was divided by the specimen cross-sectional area to obtain results in units of stress (MPa). Mean values were calculated for each tooth at each testing time.

Failure modes were determined by examining the fractured specimens using a stereomicroscope and classified into the following types: CD (cohesive in dentin), AD (adhesive between resin cement and dentin—sealed or unsealed), MI (mixed), ADR (adhesive between indirect restoration and resin cement), and CR (cohesive in composite resin).

Bond strength values were submitted to two-way analysis of variance, considering the factors "dentin sealing" and "water storage time" (5×2) and Tukey *post hoc* test ($\alpha=0.05$) (SAS for Windows version 8, SAS Institute Inc, Cary, NC, USA).

Table 2: Mean Bond Strength Values in MPa (Standard Deviation) for the Different Materials Applied to Dentin With Immediate Dentin Sealing and Without (Control Group)^a

	7 Days	3 Months
Xeno V	48.0 (14.1) Aa	18.6 (9.8) Ab
Clearfil SE Bond	33.0 (8.4) ABa	25.4 (3.9) Aa
XP Bond	30.8 (14.0) ABa	21.9 (2.5) Aa
OptiBond FL	45.1 (6.0) Aa	28.3 (9.2) Ab
RelyX Unicem (control)	22.8 (7.7) Ba	23.3 (17.3) Aa

^a Means followed by different uppercase letters (columns) and lowercase letters (rows) are significantly different by Tukey test at 5% confidence level.

RESULTS

Microtensile Bond Strength

Mean microtensile bond strength (μ TBS) values are presented in Table 2. Two-way analysis of variance revealed a significant difference for the factor “dentin sealing” ($p=0.02380$) and for the factor “water storage time” ($p=0.00024$). The interaction

between factors was also statistically significant ($p=0.03771$).

After seven days, among the four groups subjected to IDS, the μ TBS showed no significant differences between adhesive systems. However, Xeno V and Optibond FL presented significantly higher values when compared to the control group (no IDS). Clearfil SE Bond and XP Bond showed intermediate values and did not differ significantly from the control group.

The evaluation after three months of water storage demonstrated no significant differences among groups. However, when compared with seven days of storage, there was a significant reduction in bond strength values for the single-step self-etching system Xeno V and for the three-step etch-and-rinse system Optibond FL.

Failure mode analysis (Figure 1) showed a prevalence of adhesive failures between the resin cement and dentin. After three months, there was a slight

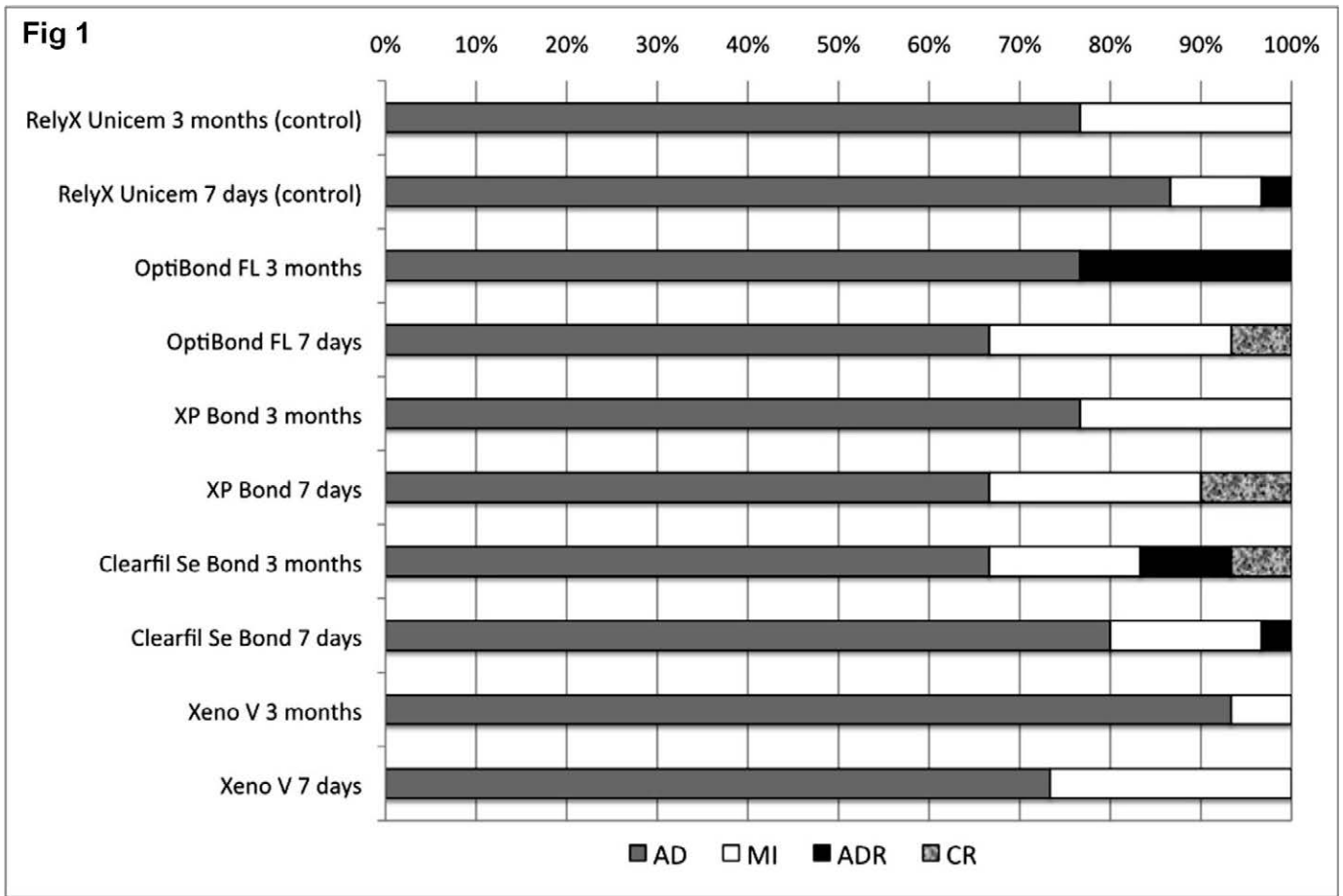


Figure 1. Distribution of failure modes within groups. CD, cohesive in dentin; AD, adhesive between resin cement and dentin (sealed or unsealed); MI, mixed; ADR, adhesive between indirect restoration and resin cement; CR, cohesive in composite resin.

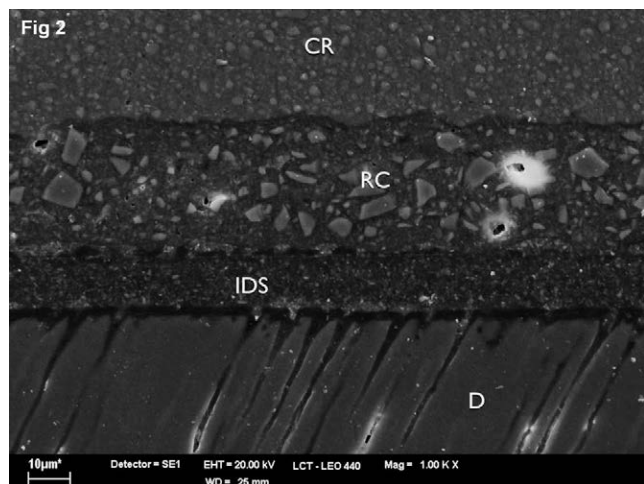


Figure 2. Representative SEM image of the interface when IDS was performed with Optibond FL adhesive system. D: dentin; IDS: immediate dentin sealing; RC: resin cement; CR: indirect composite resin.

increase in the amount of adhesive failures for Xeno V, XP Bond, and Optibond FL. No cohesive failures in dentin were observed.

DISCUSSION

The IDS technique can contribute to stability and reduction in permeability of the adhesive interface in indirect restorations.^{12,14} This technique consists of dentin hybridization with an adhesive system (Figure 2) after preparation and contributes to increased bond strength and reduced dentin sensitivity during the provisional phase.^{8,9}

Adhesive systems that have a hydrophobic resin coating the primer layer play an important role in reducing dentin permeability.¹² On the other hand, simplified adhesive systems, which have the hydrophilic primer and the hydrophobic resin in the same bottle, have been increasingly used because of their user-friendly characteristics. Single-step self-etching adhesives are considered the most susceptible to degradation when stored in water due to the increased hydrophilicity of the interface.⁴ Thus, in this study, the first hypothesis was validated since there was no significant difference when simplified adhesive systems were compared with the three-step etch-and-rinse and the two-step self-etch adhesives.

Although XP Bond and Clearfil SE Bond did not demonstrate a significant difference from the control after seven days, all groups in which IDS was performed presented higher μ TBS values when compared to the control group. This is probably due to the etch-and-rinse and self-etch adhesives having a

better demineralization capacity and hybrid layer formation than the self-adhesive resin cement.¹⁵ The hybrid layer produced by the self-etching adhesive systems used in this study was approximately 0.5 μ m, while for the etch-and-rinse, it was around 5 μ m.^{5,15-18} Moreover, recent studies demonstrated that the demineralization depth produced by RelyX Unicem is approximately 200 nm,¹⁵ and the initial bond strength is higher for multistep resin cements that are associated with some type of adhesive system.⁵

The second hypothesis was partially rejected because two of the materials used for IDS were not able to eliminate the interface degradation effects produced by storage in water for three months. IDS performed with Xeno V and Optibond FL had a significant μ TBS reduction after three months of water storage. In addition, there was no significant difference between the IDS groups and the control after three months. A previous study showed a μ TBS increase when IDS was performed with Clearfil SE Bond and used RelyX Unicem as a luting agent in the absence of simulated pulpal pressure.¹⁴ These data corroborate the μ TBS stability observed after three months of water storage in the present study when IDS was performed using Clearfil SE Bond.

In this study, specimens were sectioned into beams with a 1-mm² cross-sectional area and stored in water for a period of three months. Despite the relatively short storage time, the beam shape accelerates the interface degradation.¹⁹ Interestingly, the μ TBS of the control group did not decrease after water storage. Reports show that, even after one year of storage in water, there was no μ TBS reduction for this material. It is believed that the change of the self-adhesive cement from hydrophilic to hydrophobic improves the interface stability.¹⁵ After three months, IDS with Clearfil SE Bond and XP Bond demonstrated no reduction in μ TBS values, remaining stable during the study period.

As previously indicated, the IDS technique is recommended in order to increase μ TBS and reduce dentin sensitivity during the provisional phase.^{8,9} According to the limitations of this study, the one-step self-etching adhesive (Xeno V) and conventional three-step adhesive (Optibond FL) provided significantly higher μ TBS values after seven days when compared to the control group. Although after three months this performance was not maintained, there was no difference when compared to the control group. Therefore, a decisive factor for clinically indicating this technique would be its ability to reduce dentin hypersensitivity.

The failure pattern SEM images showed that the failure normally occurred between the resin cement and the sealing, which suggests that the dentin would still remain sealed even if interface debonding occurred. According to Magne and others,^{20,21} the adhesion of the sealed dentin to resin cement may occur due to the presence of residual free radicals, van der Waals-type interactions, and micromechanical retention.

CONCLUSIONS

Based on the findings and limitations of this study, we conclude the following:

- The μ TBS values produced by the different materials used for IDS were not significantly different from each other.
- After seven days of water storage, the μ TBS of groups sealed with XP Bond and Clearfil SE Bond did not differ significantly from the control group.
- Storage in water for three months produced a significant reduction in μ TBS for groups in which IDS was performed with Xeno V and Optibond FL.
- After three months of water storage, the μ TBS values for groups with IDS were not significantly different from the control group (without IDS).

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 SISNEP. The approval code for this study is 94/2008.

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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Stability of the Light Output, Oral Cavity Tip Accessibility in Posterior Region and Emission Spectrum of Light-Curing Units

CB André • G Nima • M Sebold • M Giannini • RB Price

Clinical Relevance

This study provides practitioners with information on the differences in spectral peak, irradiance, radiant exposure, output stability, mouth accessibility, and tip size for a variety of light-curing units, which can define the choice of proper equipment for clinical use.

SUMMARY

Objectives: This study evaluated the light output from six light-emitting diode dental curing lights after 25 consecutive light exposures without recharging the battery, tip accessibility in the posterior region, and light beam spread from light-curing units.

*Carolina Bosso André, DDS, MS, PhD, State University of Campinas, Piracicaba Dental School, Restorative Dentistry, Piracicaba, Brazil

Gabriel Nima, DDS, MS, PhD student, State University of Campinas, Piracicaba Dental School, Dental Materials, Piracicaba, Brazil

Maicon Sebold, DDS, MS, PhD student, State University of Campinas, Piracicaba Dental School, Restorative Dentistry, Piracicaba, Brazil

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

Richard B. Price, BDS, DDS, MS, PhD, FDS RCS (Edin), FRCD(C), Dalhousie University, Dental Clinical Sciences, Halifax, Nova Scotia, Canada

*Corresponding author: Av. Limeira 901, Bairro Areião, Piracicaba, 13414-903, Brazil; e-mail: carolina.bosso@gmail.com

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Methods: Irradiance, spectral peak, and radiant exposure were measured with the battery fully charged (Bluephase Style, ESPE Cordless, Elipar S10, Demi Ultra, Valo Cordless, and Radium-Cal) and monitored for 25 light exposures (each lasting 10 seconds). The tip diameter was measured to identify the beam size and the ability of the six light-curing units to irradiate all areas of the lower second molar in the standard output setting.

Results: Four curing lights delivered a single peak wavelength from 454 to 462 nm, and two (Bluephase Style and Valo Cordless) delivered multiple emission peaks (at 410 and 458 nm and 400, 450, and 460 nm, respectively). The irradiance and radiant exposure always decreased after 25 exposures by 2% to 8%, depending on the light unit; however, only ESPE Cordless, Valo Cordless, and Radium-Cal presented a statistical difference between the first and the last exposure. The tip diameter ranged from 6.77 mm to 9.40 mm. The Radium-Cal delivered the lowest radiant exposure and irradiance. This light was also unable to access all the teeth with the tip parallel to the occlusal surface of the tooth.

Conclusion: Not all of the blue-emitting lights deliver the same emission spectra, and some curing lights delivered a lower irradiance (as much as 8% lower) after the 25th exposure.

INTRODUCTION

To overcome some of the problems associated with quartz-tungsten-halogen lights,^{1,2} dental light-emitting diode (LED) curing lights were developed. According to Leprince and others,³ LED curing lights offer the best option because they have a narrower emission spectrum with peak output close to the camphorquinone absorption peak of 470 nm and can be battery operated.

To achieve an adequate amount of polymerization of the resin material, practitioners should use curing lights that deliver the correct wavelengths to each resin composite.^{4,5} Camphorquinone is the most commonly used photoinitiator in dental resins.⁶ However, alternative photoinitiators with different peak absorptions have been added to the composition of some resin-based composites (RBCs) and resin cements to improve their mechanical behavior, depth of cure, and final color.^{3,7} To deliver wavelengths of light that will activate more than one photoinitiator, some LED units deliver a light output that has two or more wavelength peaks, instead of a single emission peak (conventional LEDs).^{2,8}

Another concern regarding dental light curing in the mouth is whether or not sufficient radiant exposure (sometimes incorrectly described as energy density) has been delivered to cure the resin composite.⁴ The radiant exposure (J/cm^2) is the product of light irradiance (mW/cm^2) multiplied by the exposure time.^{4,9} Depending on the brand and shade of the RBC, the radiant exposure required to adequately polymerize the RBC can range from 6 to 24 J/cm^2 for a 2-mm increment.^{9,10} A handheld dental radiometer usually measures only the irradiance delivered at the tip end of the light-curing unit and not the radiant exposure received by the restoration.⁹ The single irradiance value uses a method described in ISO 10650 standard (calculated from the quotient of the radiant power and light tip optical area) that is inversely proportional to the tip area¹¹; in other words, small changes in the light-curing tip diameter will have a large effect on the irradiance.¹² Consequently, if the same radiant power is delivered, changing the tip diameter from 10 to 7 mm halves the tip area and doubles the irradiance.

LED lights consume low power and can be battery operated.^{3,11} However, the light output should

remain stable during all activations, with no decrease as the battery discharges, and the equipment should indicate when to recharge the battery.¹¹ According to Pereira and others,¹³ low battery levels of LED curing lights can influence some properties of composite resins, but is not well known if the irradiance will decrease after a clinically relevant number of light exposures.

Besides the technical achievements of light-curing units, the exterior design of the tip and the light beam size are also important factors for efficient resin polymerization. The external design of the tip should allow the tip to reach posterior regions both as close as possible and parallel to the restored tooth.^{4,14,15} An angle, a space between the light-curing tip and the tooth to be restored, any operator error, or movement of the light tip reduces the amount of light and energy delivered to the resin composite restoration.⁹

The objective of this study was to evaluate the light output from six contemporary LED dental curing lights after 25 light exposures each lasting 10 seconds without recharging the battery. In addition, the depth of penetration and accessibility in the mouth were analyzed. The null hypotheses were that 1) the 25 repetitions would not deliver the same irradiance for all six lights, 2) the single peak lights tested would not have the same spectral peak, and 3) the multiple peak (Polywave) lights would not have the same spectral peaks.

METHODS AND MATERIALS

Light-Curing Units on the MARC-Patient Simulator System

The six light-curing units were chosen to represent a wide variety of contemporary LED curing lights (Table 1). Both the exterior and the interior tip diameters were measured using digital calipers (Mitutoyo Co., Kanagawa, Japan). An image was obtained of the tip of each light curing unit (Nikon D5100, Nikon Corp, Tokyo, Japan), and a second image was obtained with a ruler to add the scale at the final image (combining all tip lights in one figure at the same scale). All lights were attached to a rigid stand with the light tip parallel to the MARC patient simulator (MARC-PS, BlueLight Analytics Inc, Halifax, NS, Canada) anterior sensor, shown in Figure 1. The 4-mm-diameter light sensor used in the MARC-PS was located between two maxillary central incisors. The manufacturers of the LED curing lights, serial numbers, and the classification are described in Table 1.

Table 1: Manufacturer, Serial Number, Light-Emitting Diode (LED) Classification, and Power Source of Light-Curing Units				
Light-Curing Unit	Manufacturer	Serial Number	LED Classification	Power Resource
Bluephase Style	Ivoclar Vivadent AG, Schaan, Liechtenstein	1100000611	Multiple peak	Rechargeable battery (Lipo, 3.7 V 600 mAh)
ESPE cordless	3M ESPE, Sumaré, SP, Brazil	0879857109	Single peak	Rechargeable battery (Li-ion, 3.7 V 2200 mAh)
Elipar S10	3M ESPE, St Paul, MN, USA	939112007892	Single peak	Rechargeable battery (Li-ion, 3.7 V 2300 mAh)
Demi Ultra	Kerr Corporation, Orange, CA, USA	786019408	Single peak	U-40 Ultracapacitor
Valo Cordless	Ultradent Products Inc, South Jordan, UT, USA	C26561	Multiple peak	Rechargeable battery (Life PO4, 3.2 V 750 mAh)
Radii-Cal	SDI Limited, Victoria, Australia	4-18944	Single peak	Rechargeable battery (Li-ion, 7.4 V 1550 mAh).

Determination of Irradiance, Total Energy, and Changes in Irradiance After 25 Exposures

The MARC-PS uses a fiber-optic spectroradiometer (USB 4000, Ocean Optics, Dunedin, FL, USA) to measure the irradiance, the radiant exposure, and the emission spectrum received by simulated cavity preparations in a dental mannequin head. After the lights had been set up the MARC-PS, they were activated for 25 consecutive light exposures each lasting 10 seconds with a fully charged battery or ultracapacitor. The lights were then recharged at the end of the 25th exposure, and the procedure was repeated two more times (n=3, determined by the low standard deviation), always fully charging the battery after the 25th exposure.

All lights were randomly tested. Since Radii-Cal has no 10-second exposure time, a portable digital timer (Herweg, Timbó, SC, Brazil) was used to stop the exposure as close as possible to 10 seconds. The number of exposures was chosen based on the maximum number of exposures that the ultracapacitor of Demi Ultra could deliver in one charge. This study also simulated consecutive light exposures that could be delivered in cases where six metal-free crowns are cemented and where four 10-second exposures on each tooth surface (buccal, occlusal, lingual, mesial, and distal) are recommended, giving a total of 24 exposures. Moreover, 25 exposures of 10 seconds are easily reached by practitioners in dental offices in several procedures, such as multiple direct resin composite restorations. The first and the last exposures (25th) obtained for all light-curing units were tested for the normality of data (Kolmogorov-Smirnov) and compared by paired two-tailed *t* test ($\alpha=0.05$).

Determination of the Incidence Angle and Depth of Penetration

A transparent plastic box was filled with red-stained water (rhodamine B, Sigma-Aldrich, St. Louis, MO,

USA). All light-curing units were fixed to a retort stand, turned on at same time, with the tip inside the inked water. An image was obtained of the light dispersion with a camera. A second image was obtained with a ruler beside the light tip to add the scale to the final image. Although the refractive index of stained water is different from the refractive index of the air and the composites this methodology was performed to visually observe the differences in the light beam spread from the lights.

Tip Accessibility in Posterior Region

The maximum mouth opening was fixed to 35 mm at the incisors in a dental mannequin (Marília Dental Mannequin, Marília, SP, Brazil), to represent an average mouth opening.¹⁶ The mannequin and the camera (Nikon D5100, Nikon Corp, Tokyo, Japan) were fixed in the same position (and distance) for all the images. The ability to reach the lower second molar with the tip parallel to the occlusal surface of this tooth was determined, and for those lights unable to fulfill this parameter, a second picture was

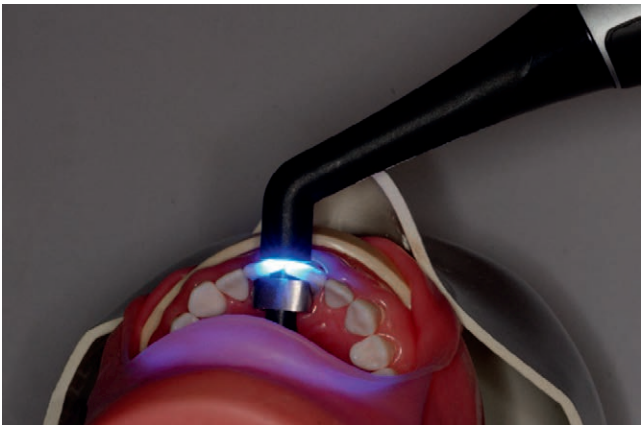


Figure 1. Light-curing tip parallel to the MARC-PS anterior sensor.

Table 2: External and Internal Tip Diameter (mm) of Light-Curing Units

	Bluephase Style	ESPE Cordless	Elipar S10	Demi Ultra	Valo Cordless	Radii-Cal
External	9.73	7.96	9.79	10.57	13.10	11.65
Internal	8.62	6.77	8.81	7.83	9.40	6.92

taken with the tip parallel to the most distant tooth possible.

RESULTS

Light-Curing Units and Tip Size

The external and internal dimensions of the light tip and the characteristics of the light-curing tips are showed in Table 2 and Figure 2, respectively. The small tip diameters, found for ESPE Cordless and Radii-Cal, may not cover an entire cavity preparation in a molar class II that is greater than 7 mm in mesiodistal width; thus, overlapping exposures are necessary to cover all of the resin composite restoration. Figures 2A to 2C show that the light source is not always close to the tip. In contrast, the LED emitters can be seen at the tip ends of the Demi Ultra, Valo Cordless, and Radii-Cal (Figures 2D, 2E, and 2F, respectively).

Determination of Irradiance, Total Energy, and Changes in Irradiance After 25 Exposures

The mean irradiance, radiant exposure (first and 25th), and wavelength emission peaks are shown in Table 3. After collecting the data using the MARC-

PS, the results were plotted with GraphPad Prism 6 (GraphPad Software, Inc., La Jolla, CA, USA). Figure 3 shows the wavelength of single peaks (Figure 3A) and multiple peak lights (Figure 3B), and the irradiance for all six lights (Figure 3C). The single peak lights delivered different spectral peak and irradiance. Multiple peak lights also had different spectral peaks and irradiance. The Demi Ultra delivered a pulsed light output (Figure 3C) and the mean irradiance was determined by the average of the high and low pulse values. Radii-Cal did not emit a stable irradiance compared with the other lights tested, as shown by the irregularities in the irradiance curve (irradiance/time).

After 25 exposures, the data were combined and are reported in Figure 4. All six lights delivered a relatively stable irradiance for the 25 exposures (Figure 4A,B). The difference in irradiance between the first and the last exposure is shown in Figure 4C and D. Depending on the light-curing unit, there was a small decrease ranging from 1.9% to 8.4% in the irradiance for all six lights; however, for the ESPE Cordless, Valo Cordless, and Radii-Cal there was a statistical difference between the first and the last exposure ($p=0.0086$, $p=0.0103$, and $p=0.0118$, re-



Figure 2. LED light-curing tips. (A): Bluephase Style. (B): ESPE Cordless. (C): Elipar S10. (D): Demi Ultra. (E): Valo Cordless. (F): Radii-Cal. Note the different tip diameters (all tips imaged to the same mm scale).

Table 3: Mean (Standard Deviation) of Irradiance and Radiant Exposure, and Spectral Emission Peak of Light-Curing Units^a

	Mean Irradiance, mW/cm ²	First Radiant Exposure, J/cm ²	25th Radiant Exposure, J/cm ²	Mean Decrease, %	Emission Peak, nm
Bluephase Style	1036.1 (31.6)	11.2 (0.4)a	10.5 (0.1)a	6.1	410
					458
ESPE Cordless	1579.6 (74.5)	15.9 (0.7)a	14.5 (0.5)b	8.4	460
Elipar S10	1840.7 (46.2)	18.9 (0.5)a	18.5 (0.4)a	1.9	458
Demi Ultra	1607.9 (5.3)	16.3 (0.2)a	15.8 (0.5)a	3.3	462
Valo Cordless	1474.2 (35.8)	15.1 (0.5)a	14.6 (0.3)b	3.2	400
					450
					460
Radii-Cal	839.6 (61.2)	8.5 (0.7) a	7.9 (0.9)b	7.2	454

^a Different letters show significant difference between the first and the 25th exposures (p<0.05).

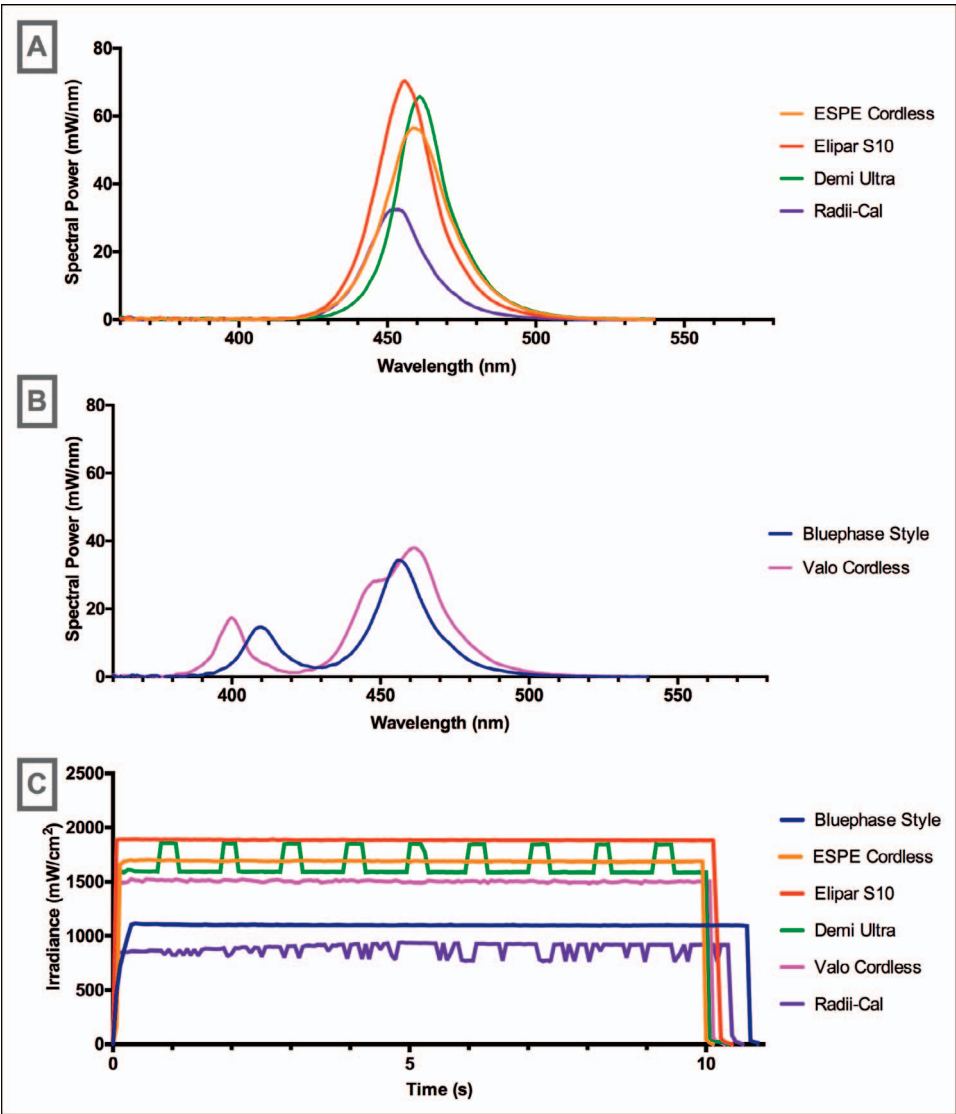


Figure 3. Spectral radiant power output (mW/nm) and irradiance (mW/cm²) from six curing-light units. (A): Emission spectrum from single peak lights. (B): Emission spectrum from multiple peak lights. (C): Irradiance delivered at the tip of all six lights.

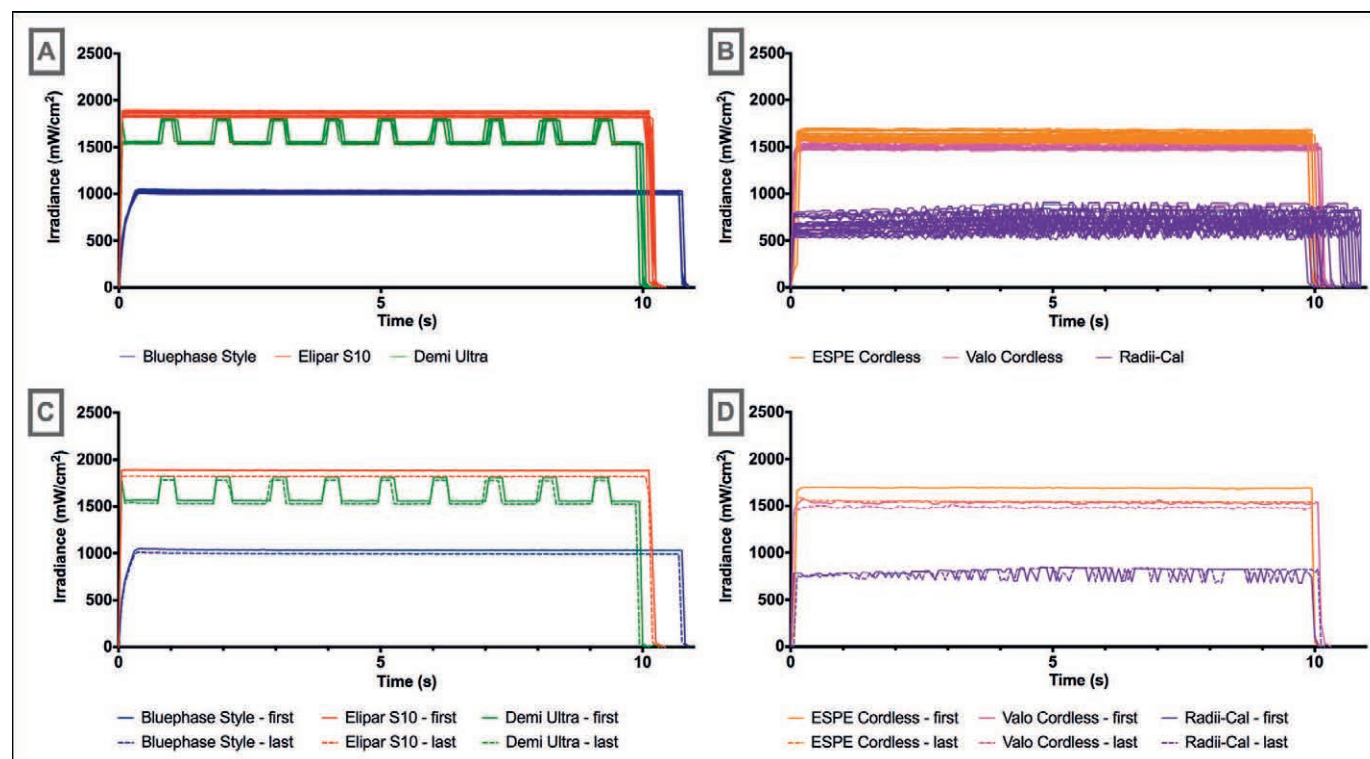


Figure 4. Irradiance at the light tip of the curing-light units. (A, B): Twenty-five exposures of 10 seconds. (C, D): The first (solid lines) and the last exposure (dotted lines) of each light.

spectively). The ESPE Cordless showed the largest decrease (8.4%) when the last exposure was numerically compared to the first exposure, whereas there was a 3.2% decrease for the Valo Cordless light.

Radii-Cal delivered the lowest irradiance and radiant exposure numerically compared with the other LED light-curing units tested. For this light-curing unit, a 10-second exposure was obtained manually with the help of a portable digital timer, so the time of each exposure was not exactly the same (Figure 4B). However, using the average irradiance delivered, the radiant exposure that would be delivered in 10 seconds was calculated.

Determination of Light Beam Spread

An image of the light beam spread through the red water of all six curing light units is shown in Figure 5. Light-curing units with higher radiant exposure (Table 3) delivered a deeper brighter area (*) compared with Radii-Cal (Figure 5F). The brighter regions may be related to the irradiance received and the amount of energy delivered from the light to a resin composite. When compared on the same scale, the light-curing units from A to E showed a brighter area that was 7-mm to 10-mm length and light-curing unit F (Radii-Cal) only 3 mm. Likewise, the

depth of light penetration in the red water ranged from 26 mm to 30 mm (Figure 5A to E) and Radii-Cal was only 21 mm. In addition, it is possible to observe differences in the light dispersion and the region where the restoration would be irradiated.

Tip Accessibility in the Posterior Region

The ability to align the light tip parallel to the tooth is shown in Figure 6. The Valo Cordless and Bluephase Style (Figure 6A, F) were able to reach the second molar with the tip parallel to the tooth (occlusal exposure). At the 35 mm interincisal opening, the Radii-Cal was unable to reach any posterior tooth with the light tip parallel to the occlusal surface (Figure 6E). Figures 6A to 6F show the most posterior location where each tip can reach, yet still keep the light tip parallel to the occlusal surface. Figures 6G to 6J show the inclination of the light tip that would occur when polymerizing an occlusal restoration in the second molar region at an interincisal opening of 35 mm.

DISCUSSION

Light-cured resin composites have become the material of choice for direct restorations and are widely used in dental practice.¹⁷ In 2012, it was

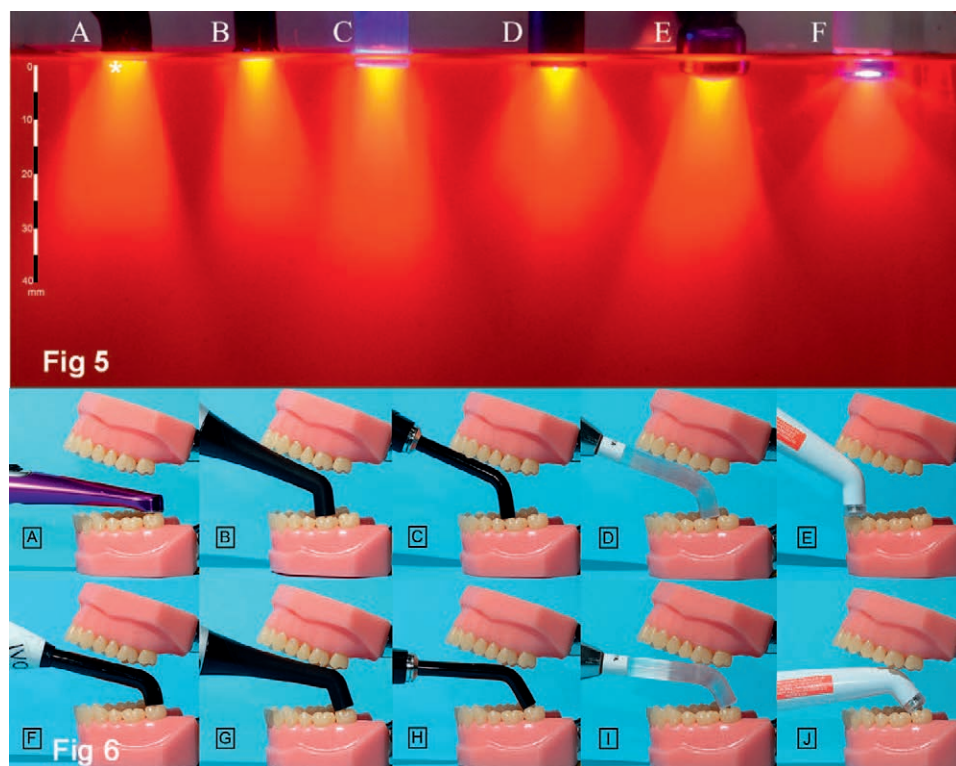


Figure 5. Beam spread of curing-light units. (A): Bluephase Style. (B): ESPE Cordless. (C): Elipar S10. (D): Demi Ultra. (E): Valo Cordless. (F): Radium-Cal. *Brighter area, ie, the irradiance delivered to a restoration.

Figure 6. Ability of six light-curing units to reach the mandibular second molar. (A): Valo Cordless. (B, G): Demi Ultra. (C, H): ESPE Cordless. (D, I): Elipar S10. (E, J): Radium-Cal. (F): Bluephase Style. (A–F): Accessibility limit. (G–J): Angulation of the light tip required to access the second molar.

stated that more than 260 million direct RBC dental restorations are placed annually worldwide,¹⁸ and this number will only increase as the use of dental amalgam is phased down and becomes less frequent.¹⁹ For many years, halogen light-curing units were used; however, LED curing units have now become the most popular curing lights. The irradiance delivered by a light-curing unit is calculated from the radiant power incident on a surface of known area, and unless a sufficient number of photons are received at the appropriate wavelengths, the polymerization of the RBC can be inadequate.²⁰ In this study, the irradiance decreased slightly after 25 exposures of 10 seconds each, accepting the first null hypothesis that the 25 repetitions would not deliver the same irradiance for all six lights.

All the lights tested delivered a very stable irradiance during each exposure, suggesting that the battery charge had minimal influence on irradiance after 25 exposures of 10 seconds. However, the Radium-Cal did not show a homogeneous irradiance for all exposures and presented the lowest irradiance and radiant exposure numerically compared with the other curing lights tested. Higher temperatures generally reduce the output from LED emitters and have a negative influence on both the reliability and durability of the LED chip. The increase in temper-

ature can also change the LED peak wavelength.²¹ For all lights, the decrease in irradiance and energy was not large; however, it is suggested to recharge the battery often and let the light cool down between multiple uses. Allowing the cooling of LED lights may minimize the small changes in irradiance and increase the durability of the light. Demi Ultra uses an ultracapacitor as an energy source, and the advantage of this is the reduction in the time required to fully charge the unit. In this study, it took only about 40 seconds to fully recharge the Demi Ultra.

Resin photopolymerization can be affected by differences in the spectral emission of light-curing units.^{4,5,22} Different spectral emission peaks were found for single peak lights, accepting the second null hypothesis stating that all single peak lights would not have the same emission peak. Also, the multiple peaks lights had different spectral emission peaks, accepting the third null hypothesis for the same reason. Camphorquinone is the most commonly used photoinitiator in dental polymers.^{6,23} The maximum absorbance peak of camphorquinone is at 470 nm, but is activated in a range from 400 to 500 nm,²⁴ and it is also activated by light of wavelengths below 320 nm.²⁵ All of the single peak lights and multiple peak lights tested in this study deliver a spectral emission that is compatible with the

camphorquinone absorbance range. The Radii-Cal delivered the lowest spectral power numerically compared to the other single peak lights tested.

Alternative photoinitiators, such as PPD, Lucerin TPO, BAPO (Irgacure 819), and Ivocerin, are also found in the wide range of dental polymers available on the market.^{6,26} TPO is very sensitive to light below 420 nm,²⁶ PPD below 460 nm, and BAPO below 440 nm.²⁴ Ivocerin is a photoinitiator under patent protection, and according to the manufacturer (Ivoclar Vivadent, Schaan, Liechtenstein), it is very sensitive to light below 450 nm, with an absorbance peak close to 410 nm. Because of their relatively narrow emission spectra, all four single peak lights tested in this study cannot activate the TPO initiator, that is used in Tetric EvoCeram (Ivoclar Vivadent) and Vit-l-escence (Ultradent Products Inc, South Jordan, UT, USA) composites.²⁷ However, the multiple peak lights, such as Valo and Bluephase Style should be able to activate the alternative photoinitiators.

Resin composite materials require different radiant exposure levels,²⁸ and the practitioner should be aware of the radiant exposure or the exposure time and the irradiance recommended by the manufacturer of each material. The shade of the composite, thickness of the increment, and distance from the light source should be taken into account regarding the required exposure time and the radiant exposure required to adequately polymerize the resin.⁹ However, the practitioner should not arbitrarily increase the time of exposure, but instead they should follow the exposure time/irradiance according to the resin composite manufacturer's recommendations for each resin shade. Otherwise, the practitioner may deliver too much energy and cause an unacceptable temperature increase in the pulpal or soft tissues.^{9,29,30}

Besides the technical guidance of light-curing units described in this study, to achieve optimal polymerization, dentists should (1) take in account the size and external design of the tip, (2) place the light directly on the composite restoration at 90° perpendicular to the surface, and (3) almost in contact with restorative material.³¹ According to Konerding and others, a tilted light tip will reduce energy delivery and may impair the polymerization.^{14,31} Excessive tip inclination will decrease the radiant exposure received by the resin. The Valo Cordless and Bluephase Style were able to reach the second molar occlusal with the tip parallel to the occlusal surface of the tooth. Figure 5 represents an easy method for practitioners to understand how the light beam spreads from light-curing

units and by examining the brighter area and depth of penetration, may be used to compare curing lights. The light with least irradiance produced the least bright area and the lowest depth of penetration compared to the other lights. Another concern regarding the curing light is the tip diameter. The practitioner should be aware that the internal diameter from where the light is emitted should cover the full width of a large restoration.⁵ Lights such as the ESPE Cordless and Radii-Cal may require multiple exposures to cover the full width of a molar class II cavity preparation. Otherwise, at the proximal box, inadequate polymerization may cause premature failure of class II composite resins at the gingival margin.³²

Excessive light tip inclination can decrease the radiant exposure received by the resin, and the percentage of decrease is dependent on the light-curing unit. However, even lights with high irradiance values will experience a significant decrease in radiant exposure as the tilt angle increases.³¹ In addition to correctly positioning the light tip over the RBC, it is important that the practitioner watch what he or she is doing when light curing through a blue-blocking filter to protect their eyes.^{15,33}

CONCLUSION

Within the limitations of the current study, the following conclusions can be made:

1. Not all light-curing units deliver the same quality (wavelength) and quantity (radiant exposure) of blue light in 10 s. Practitioners should match the light-curing unit with the wavelengths required by the photoinitiators used in the restorative materials they are using.
2. Practitioners should be aware that after several exposures in a row (25 times), the radiant exposure may decrease between 2% and 8%.
3. The light-curing unit should be designed to allow the light tip to be brought parallel to and as close as possible so that the light is delivered at 90° perpendicular to the surface of the restoration.

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Conflict of Interest

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

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Effect of Surface Sealants and Polishing Time on Composite Surface Roughness and Microhardness

VC Ruschel • VS Bona • LN Baratieri • HP Maia

Clinical Relevance

Application of surface sealants do not improve the surface smoothness and microhardness of the composite resin.

SUMMARY

The objective of this study was to evaluate the effect of surface sealants and polishing delay time on a nanohybrid resin composite roughness and microhardness. Eighty disc specimens were made with a nanohybrid resin (Esthet-X HD, Dentsply). The specimens were divided into two groups (n=40) according to polishing time: immediate, after 10 minutes; delayed, after 48 hours. Each group was sub-

divided into four groups (n=10), according to the surface treatment: CG, control-rubber points (Jiffy Polishers, Ultradent); PP, rubber points + surface sealant (PermaSeal, Ultradent); PF, rubber points + surface sealant (Fortify, Bisco); PB, rubber points + surface sealant (BisCover, Bisco). Surface roughness (R_a) and microhardness (50 g/15 seconds) were measured. Surface morphology was analyzed by scanning electron microscopy and atomic force microscopy. The data were analyzed statistically using one-way analysis of variance and the Games-Howell *post hoc* test ($\alpha=0.05$). PermaSeal roughness (G2) in the delayed polishing group was significantly higher ($p=0.00$) than that of the other groups. No difference was observed among the groups between immediate and delayed polishing ($p=1.00$), except for PermaSeal ($p=0.00$). Moreover, PermaSeal showed the lowest microhardness values ($p=0.00$) for immediate polishing. Microhardness was higher at delayed polishing for all the surface treatments ($p=0.00$) except Fortify ($p=0.73$). Surface smoothness similar to polishing with rubber points was achieved when surface sealants were used, except for PermaSeal surface sealant, which

Vanessa Carla Ruschel, DDS, MS, PhD, Department of Dentistry, Center of Health Sciences, Federal University of Santa Catarina, Florianópolis, Brazil

*Vitor Schweigert Bona, DDS, MS, PhD Student, Department of Dentistry, Center of Health Sciences, Federal University of Santa Catarina, Florianópolis, Brazil

Luiz Narciso Baratieri, DDS, MS, PhD, Department of Dentistry, Center of Health Sciences, Federal University of Santa Catarina, Florianópolis, Brazil

Hamilton Pires Maia, DDS, MS, PhD, Department of Dentistry, Center of Health Sciences, Federal University of Santa Catarina, Florianópolis, Brazil

*Corresponding author: Sala 138, Campus Universitário Trindade, Florianópolis SC 88040-970, Brazil; e-mail: vsbona@gmail.com

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resulted in a less smooth resin composite surface. However, surface sealant application did not significantly improve composite resin microhardness.

INTRODUCTION

The finalization of a composite resin restoration on either anterior or posterior teeth implies applying finishing and polishing steps.¹ This procedure is essential to maintaining marginal integrity,² esthetics,³ longevity of the restorations,⁴ oral health,^{5,6} comfort of the patient,^{2,7,8} and reduced pigmentation and wear.⁹ Therefore, negligence during execution of the restoration may result in exposure of restoration margins, leading to microleakage,^{10,11} caries recurrence, greater susceptibility to surface staining,¹ decreased wear resistance of the material,¹² and greater accumulation of plaque, leading to gingival irritation¹³ and thus contributing to greater patient discomfort.^{14,15}

The technique of finishing and polishing a restoration seeks to maintain a proper anatomy by removing small excesses and thus obtain a smooth surface that provides adequate light reflection.^{2,16-18} These procedures can be achieved with rubber points, diamond burs, sandpaper discs and strips, and polishing pastes^{2,19-22} and usually consist of three to four steps.²³⁻²⁷ Restorations carried out carefully achieve a result that requires minimal finishing and that obtains smooth and contiguous edges of the tooth structure.² For a restoration to be imperceptible, the surface should be similar to enamel in terms of brightness and texture. These characteristics are directly related to the surface roughness of the restorative material.²⁸⁻³²

In addition, the mechanical properties of the composite, such as microhardness and flexural strength, are essential for the material to resist masticatory forces and have greater longevity. Microhardness is directly related to the depth of cure of the restorative material.^{33,34} The lower the microhardness of a resin, the more susceptible the material is to forming grooves and surface defects, which can lower the resistance and cause premature failure of the restoration.³⁵⁻³⁸

To this end, surface sealants were developed to improve the final appearance of the restoration.³⁹ These sealants consist of fluid resins with little or no filler, which are applied to the surface of composite resins or glass ionomers after the finishing and polishing step.⁴⁰⁻⁴² Their application fills irregularities, making the restoration smoother and brighter

and promoting improved marginal sealing and consequent reduced microleakage.^{41,43-45} Surface sealant may be applied on all composite restorations, including temporary acrylic resin restorations.^{39,46}

Another important issue is the optimal time for performing the polishing of composite restorations. There is still controversy in the literature. It is known that immediate polishing can compromise the initial marginal sealing of the restoration; however, marginal sealing may become better with higher hygroscopic expansion of the material in contact with the oral fluid.¹⁰ This probably compensates for damage caused by immediate polishing.^{5,11} In addition, some authors have shown that higher surface smoothness, higher microhardness,²³ and lowest microleakage⁵ can be achieved when the polishing is performed immediately after photoactivation.

Therefore, this study aimed at determining the effect of three different surface sealants and polishing delay time on the surface roughness and microhardness of a nanohybrid resin. The null hypothesis tested was that the application of the sealing surface 1) would not alter the surface roughness of the composite, 2) would not alter the composite microhardness, and 3) the polishing period would not influence the composite surface roughness or microhardness.

METHODS AND MATERIALS

One polishing system and three different surface sealants for resin composite were used in this study. The polishing composite system consisted of rubber polishers for abrasive composite polishing (Jiffy Composite Adjusters and Polishers, Ultradent, South Jordan, UT, USA). The polishers are flame-shaped and available in three grain sizes. The surface sealants evaluated were PermaSeal (Ultradent), Fortify (Bisco Inc, Schaumburg, IL, USA), and BisCover (Bisco Inc). A nanohybrid composite was used (Esthet-X HD, Dentsply Caulk, Milford, DE, USA). The composition of the materials used is described according to the manufacturer's information (Table 1). Light-emitting-diode-based photocuring (Translux Blue, Heraeus Kulzer, Hanau, Germany) was used with a light intensity of 876 mW/cm².

A total of 80 composite discs (Esthet-X HD, Dentsply), 12 mm in diameter and 2.5 mm in height, were fabricated in a custom metallic matrix. The specimens were made by inserting two increments of composite into a metal matrix. A glass slide (1.1 mm) and polyester strip were positioned at both ends of

Table 1: <i>Materials and instruments used.</i>			
Commercial Name	Composition	Application Mode	Manufacturer
Jiffy Polishers	Bowls, discs, or abrasive rubber butts, impregnated with silicon carbide particles and aluminum oxide	In descending order of abrasiveness, rinsing between the point of change	Ultradent Inc
PermaSeal	Bisphenol A glycidyl ether dimethacrylate, triethylene glycol dimethacrylate, initiators	Initial polishing, 37% phosphoric acid application for 20 seconds, wash, dry, spread the product on the surface, soft air jets, and polymerization for 20 seconds	Ultradent Inc
Fortify	Urethane dimethacrylate, bisphenol A dimethacrylate, ethoxylated	Initial polishing, 37% phosphoric acid application for 20 seconds, wash, dry, apply the product using rubbing movements, soft jets of air, and polymerize for 10 seconds	Bisco Inc
BisCover	Bisphenol A ethoxylate dimethacrylate, acrylate urethane ester, polyethylene glycol acrylate	Polishing, 37% phosphoric acid application for 15 seconds, wash, dry, apply the product in one direction, once, without rubbing, wait 15 seconds, and polymerize for 30 seconds	Bisco Inc
Esthet-X HD	Bisphenol A glycidyl methacrylate (Bis-GMA), bisphenol A ethoxylate (Bis-EMA), and triethylene glycol dimethacrylate (TEGDMA), camphorquinone, photoinitiator, stabilizer, pigments and glass fluoroborosilicate barium; particles from 0.04 to 2.5 μm , with a mean of 0.6-0.8 μm	Increments up to 2 mm and light cure for 20 seconds, finishing with diamond butts and polishing with cups, discs, or candle flames	Dentsply

the matrix and pressed digitally to promote the flow of excess material and to obtain a flat surface. The curing was performed according to the manufacturer’s instructions, and the specimens were kept dry and at room temperature. In addition, the surface of all specimens was polished with a metal device (DP-10, Panambra, São Paulo, SP, Brazil), using SiC #1200 sandpaper under constant irrigation for 10 seconds, to ultimately obtain a flat stable surface.

The specimens were randomly divided into two groups (n=40), according to the time elapsing from preparation to polishing: immediate polishing (10 minutes after preparation of specimens) or delayed polishing (48 hours after preparation of the specimens). Each group was subdivided into four groups (n=10), according to the surface treatment used: CG, control group with polishing with abrasive sequential rubber points of decreasing grain (Jiffy polishers, Ultradent); PP, rubber points + surface sealant (PermaSeal, Ultradent); PF, rubber points + surface sealant (Fortify, Bisco Inc); PB, rubber points + surface sealant (BisCover, Bisco Inc). The group consisting only of polishing with rubber points was used as the control for both immediate and delayed polishing periods. The specimens in the delayed polishing group were stored in distilled water at 37°C for 48 hours before applying the surface treatment.

A new polishing instrument was used after polishing three specimens for all groups. The polishing time

for each instrument was standardized at 30 seconds; the pressure on the surface of the composite was applied intermittently and determined by the operator. After each application of the polishing instrument, the surface of the specimens was cleaned with an air/water spray for 15 seconds. Surface sealants were applied to the surface of the specimens according to the manufacturers’ recommendations, which advocate prior polishing. The surface roughness was measured for all 10 specimens of each group, using a Portable Digital Surface Roughness Tester (PR-100, Instrutherm, São Paulo, SP, Brazil), according to the Japan Industrial Standard (B 0601, 1994), to obtain the arithmetic average roughness parameter (R_a). Three consecutive measurements were made at different regions of each specimen, turning the specimen 90° for each measurement. The device was operated with a 0.8-mm cutoff, a 0.1-mm/s read speed, and a 4-mm measuring path. The mean values obtained were subjected to statistical analysis. The Vickers microhardness (VHN) was measured on all 10 specimens of each group, using a microdurometer (HMV 2 version 1.23, Shimadzu, Japan). Four indentations were performed on each sample, with a 50 gm load for 15 seconds, measured according to each quadrant of the specimen. The average individual microhardness value for each specimen was used for statistical analysis.

The analysis of surface morphology was performed on one specimen for each surface treatment, only for

Table 2: Description and comparison of R_a and VHN of the evaluated groups

Surface Treatment	Polishing Time Point	Roughness	Microhardness
Polishing (control)	Immediate	0.34 (0.10)A,B	52.30 (2.87)b,c
	Delayed	0.33 (0.06)A,B	69.75 (4.57)d
PermaSeal	Immediate	0.30 (0.05)A	33.01 (7.83)a
	Delayed	0.61 (0.08)C	57.03 (9.46)b,c
Fortify	Immediate	0.38 (0.10)A,B	49.39 (3.14)b
	Delayed	0.40 (0.12)A,B	53.88 (6.12)b,c
BisCover	Immediate	0.42 (0.05)B	49.15 (5.44)b
	Delayed	0.31 (0.08)A,B	58.59 (6.65)c

Similar capital (R_a) and lowercase letter (VHN) at the same column are not statistically significant ($p>0.05$).

the delayed polishing period, by scanning electron microscopy (SEM) and atomic force microscopy (AFM).⁴⁷ SEM observation was enabled by coating each sample with gold-palladium alloy, and the morphologic examination was performed with 500× magnification. For AFM analysis, the specimens were dried and observed under the microscope to obtain images relating to a surface sample area of 15 μ m.

The R_a and VHN data were analyzed by one-way analysis of variance (ANOVA) and *post hoc* Games-Howell. All analyses were conducted at a significance level of 5% ($p\leq0.05$), using SPSS statistical software (version 20.0 for Mac, SPSS Inc, Armonk, North Castle, New York, USA).

RESULTS

ANOVA showed statistically significant differences between groups with regard to R_a . The PermaSeal R_a values for delayed polishing were significantly higher compared with all the other groups ($p=0.00$). There was no difference between immediate and delayed polishing for all the groups ($p=1.00$), except PermaSeal ($p=0.00$; Table 2; Figure 1).

Regarding the VHN test, ANOVA showed statistically significant differences between groups. The PermaSeal group showed significantly lower values for immediate polishing, a result statistically different from the other groups ($p=0.00$). The highest microhardness values were obtained when only polishing points (control) and delayed polishing were performed, a result statistically different from the other groups ($p=0.00$). Moreover, the microhardness was lower at immediate polishing for all the surface treatments ($p=0.00$) except Fortify, which showed no

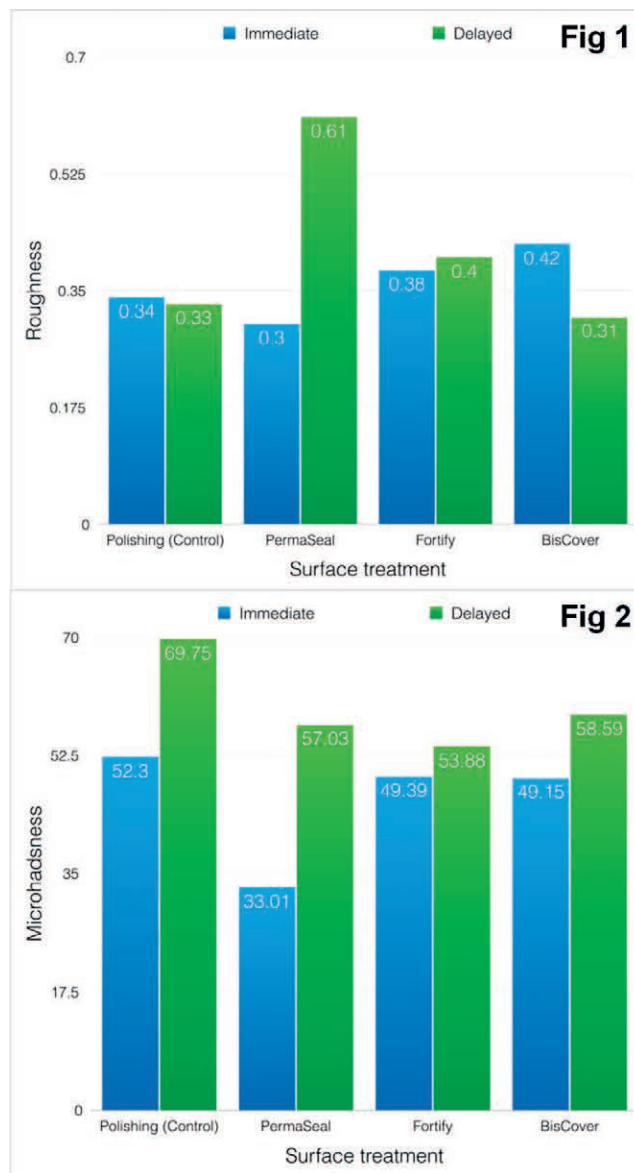


Figure 1. Graphical representation in the form of vertical bars of the arithmetic average R_a of the evaluated groups.

Figure 2. Graphical representation in the form of vertical bars of the arithmetic averages of VHN of the evaluated groups.

difference between immediate and delayed polishing ($p=0.73$; Table 2; Figure 2).

In the surface morphology analysis by SEM, ripples/waves were observed on the surface of PermaSeal (Figure 3B) and Fortify (Figure 3C), but more frequently on the surface of PermaSeal. BisCover and Polishing points (control) had fewer surface irregularities. Observing the surface morphology using AFM, the most irregular surfaces were observed for Fortify (Figure 4C) and BisCover (Figure 4D).

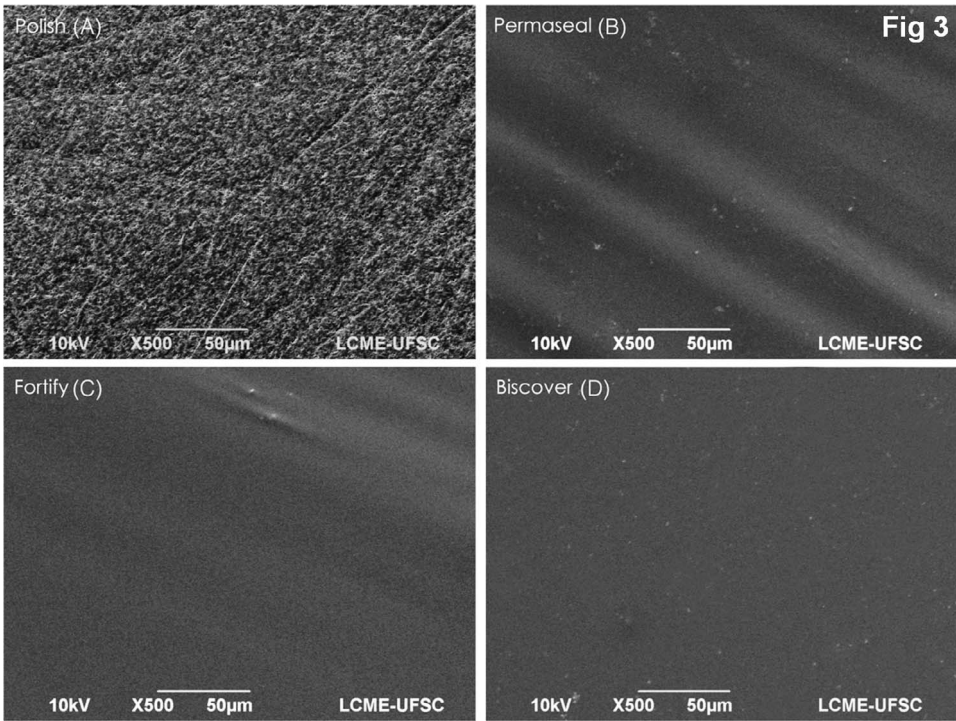


Figure 3. SEM observation of delayed polishing and application of sealants (500× magnification): A, polish; B, Permaseal; C, Fortify; D, Biscover.

DISCUSSION

According to the results of this study, the null hypotheses were partially rejected, because there was some difference among the sealants tested in relation to surface roughness, microhardness, and polishing time.

In this study, the control group showed no statistical difference between immediate and de-

layed polishing regarding surface roughness, similar to previous studies.^{21,22} However, microhardness was higher for delayed polishing for this same group, which is in line with other studies that showed increased microhardness for delayed polishing.^{24,33} Despite the controversial results in regard to time point, there is consensus among the studies that the influence of immediate or delayed polishing on the physical and mechanical properties of the composite

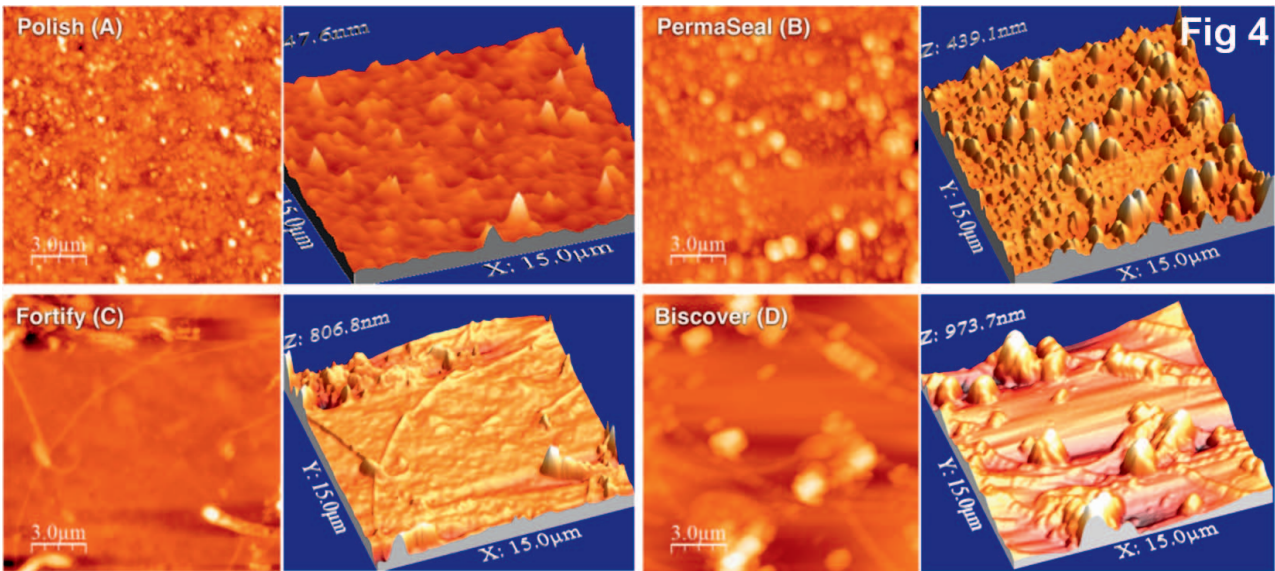


Figure 4. AFM images of delayed polishing and application of sealants (3.0-µm magnification): A, polish; B, Permaseal; C, Fortify; D, Biscover.

is materials and time dependent.^{23,24,33} Nonetheless, there are limitations to comparing the results of previous studies with this study, due to variations in the materials and methods used.

Some similar studies demonstrate that the application of sealants results in lower roughness values (R_a) compared with when only polishing is performed.^{25,39} These results differ from those found in this study, in which there were no significant roughness-related differences between polishing and application of surface sealants. This difference between the studies can be attributed to the fact that, in this study, the sealants were applied to prepolished composite, unlike the studies cited.

Despite the use of the same technique of finishing and polishing, different composites have different patterns of surface smoothness.^{23,26} This occurs because, during the procedure, the organic matrix wears away more easily than the filler particles, which remain exposed on the surface and are more prone to becoming detached from the material.^{23,26,27} The loss of these particles results in surface defects in the material, making it more irregular,²³ as seen in Figures 3A and 4A. Because the sealants are highly fluid^{40,41} and the materials are applied in a thin layer on the composite, the sealants probably penetrated the surface defects of the composite resin, thus maintaining the microtopography of the previously existing surface. This probable occurrence may explain the statistical similarity of most sealants polished only with rubber points.

In this study, when delayed polishing was performed and PermaSeal was used, a rougher surface was obtained, differing from the other groups, where the intragroup results were similar. When PermaSeal is rubbed vigorously on the surface of the composite with the applicator tip provided by the manufacturer (Black FX Micro Tip), a higher incorporation of air bubbles can occur in the sealant.⁴² The presence of bubbles and application of air jets prior to curing can create surface irregularities, which can be seen in Figure 3B. The presence of these factors may have contributed to increasing the roughness observed in this group, which differed from the others at the same polishing time point. The same surface topography can be observed in Fortify, but with lower intensity (Figure 3C). This material is also applied vigorously to the surface of the composite resin and requires an air jet prior to curing. In addition, the two SEM images taken for Fortify differ from the images obtained for BisCover (Figure 3D), which is applied in a single layer without friction and without the application of

an air jet. The SEM images differed from the AFM images, probably because the AFM image provided a view of a nanometric area of the sample⁴⁷ and was therefore not representative of the whole surface morphology.

The surface roughness obtained in all the groups was higher than the surface roughness limit for the adhesion of bacteria, which is 0.2 μm .⁸ This situation may promote the accumulation of plaque, which predisposes the restorations to decay and/or causes gum inflammation. However, with the exception of PermaSeal at the delayed polishing point, the values in the other groups were less than 0.5 μm , which corresponds to the threshold of human perception of composite resin restoration roughness.⁶

It was observed that delayed polishing resulted in a surface with greater microhardness compared with immediate polishing, even with the application of the sealant, as also reported in other studies.^{18,24} That is because the monomers continue to be converted into polymers after the initial polymerization, resulting in increased microhardness with the passage of time.³³

It should be stressed that PermaSeal had the lowest microhardness value for the immediate polishing period, statistically different from the other groups. Similar results were observed in another study, in which PermaSeal had the lowest microhardness values after 10-minute polymerization, compared with BisCover.⁴⁰ However, after delayed polishing, PermaSeal had greater microhardness, statistically similar to that of the other sealants. Thus, it can be assumed that immediate polishing impairs microhardness more when PermaSeal is used.

Despite the limitations of this *in vitro* study, sealants can be considered a viable alternative to increase the surface smoothness of composite resin. This being the case, further clinical research is needed to endorse the results of the present study.

CONCLUSIONS

Despite the limitations of this study, the following conclusions were made: 1) it is possible to obtain surface roughness similar to polishing with rubber points when surface sealants are used, except in the case of PermaSeal; 2) application of a surface sealant does not significantly improve the microhardness of the composite resin; and 3) the microhardness values were higher for the delayed polishing period, except for Fortify. In contrast, the polishing delay time did

not influence the surface roughness, regardless of the surface treatment applied.

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Conflict of Interest

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

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Efficacy of Modern Light Curing Units in Polymerizing Peripheral Zones in Simulated Large Bulk-fill Resin-composite Fillings

N Ilie • BI Luca

Clinical Relevance

Bulk-fill restorations with resin composites are increasing in popularity, but sufficiency of curing in depth and in peripheral zones of large fillings is still questioned. Modern curing units, displaying more homogeneous light beam profiles, claim enhanced homogeneity of curing, also in large fillings.

SUMMARY

The variation in micro-hardness (HV) within simulated large cavities (10×6 mm) filled in one increment with three bulk-fill resin-based composites (BF-RBC) was assessed by means of a universal hardness device. Modern blue and violet-blue light curing units (LCUs) were applied in three different positions, by rotating the LCU in 120° steps. The exposure distance was 3 mm. One center and two peripheral (4-mm apart from the center) HV line profiles were measured in 0.5-mm steps at 24 hours postpolymerization to calculate the depth of cure (DOC). Incident light, irradiance, and spectral distribution were recorded. A

multivariate analysis (general linear model) assessed the effect of the varied parameters as well as their interaction terms on HV and DOC. The effect of LCU rotation was not significant ($p=0.109$). The DOC varied between 3.46 mm and 5.50 mm and was more strongly influenced by the BF-RBC ($p<0.001$, $\eta_p^2=0.774$), followed by the width of specimen ($p<0.001$, $\eta_p^2=0.554$), while the influence of the LCU was very low ($p<0.06$, $\eta_p^2=0.070$). Whether a BF-RBC filling is cured as well in the periphery as in the center depends more on the material than on the curing unit used.

INTRODUCTION

Bulk-fill resin-based composites (BF-RBC) have gained increased interest and acceptance as restorative materials in the daily clinical routine. It is recognized that a bulk-filling technique allows for both reduced chair time and a decrease in the risk of inducing defects or contaminants between layers when compared with an incremental placement technique.¹ Whether a bulk-filling technique reduces

*Nicoleta Ilie, PhD, University Hospital, Ludwig-Maximilians-University, Munich, Germany

Department of Operative Dentistry and Periodontology

Bianca I Luca, postgraduate student, University Hospital, Ludwig-Maximilians-University, Munich, Germany

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

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shrinkage stress development in an RBC restoration is still controversial.²⁻⁴

The large variation in mechanical properties identified in BF-RBCs⁵ determined their classification in low-filled (flowable) and high-filled (sculptable) BF-RBCs. Restorations with flowable BF-RBCs require an occlusal covering layer of a high-viscosity, sculptable RBC. Despite concerns raised by the very low modulus of elasticity of flowable BF-RBCs,⁵ the described restoration model is successful, as evidenced in a five-year clinical follow-up study.⁶ In contrast, sculptable BF-RBCs may be used in bulk-fill restorations without capping and are characterized by good mechanical properties that are comparable or even higher than values measured in conventional (cured in 2-mm increments), micro-hybrid RBCs.⁵ Long-term clinical studies assessing their longevity are missing so far.

Opponents of using BF-RBCs impugn the sufficiency of curing in deep layers⁷ or in large fillings. *In vitro* studies identified that BF-RBCs are sufficiently cured in a 4-mm increment by using light curing units (LCU) with moderate irradiances (~ 1000 mW/cm²) and exposure times of ~ 20 seconds that correspond to a radiant exposure of ~ 20 J/cm².^{8,9} Interestingly, these curing conditions are comparable to radiant exposure values identified in conventional RBCs for sufficient polymerization (21-24 J/cm²).^{10,11} Variances in radiant exposure are, nonetheless, concerning because there can be grave clinical implications if too little, or conversely too much, radiant exposure is delivered to a BF-RBC restoration. Insufficient polymerization was identified to affect negatively several properties in conventional RBCs, including wear,¹² quality of restoration margins,¹³ bond strength to tooth structure,¹⁴ depth of cure,¹⁴ mechanical properties,^{15,16} degree of conversion, or amount of eluted substances from polymerized specimens.¹⁷ In contrast, increasing the radiant exposure is clinically not a guarantor for adequate curing, owing to the increased risks of thermal damage to the soft tissues and pulp.^{18,19}

The impact of LCUs on BF-RBC properties is assessed *in vitro* preponderantly by using narrow specimen sizes or considering specimen regions that correspond to ideal curing conditions, such as regions close and perpendicular to the center area of the light guide. From a clinical point of view, a sufficient polymerization in peripheral zones of large fillings is of great relevance. There is evidence that several contemporary LCUs have different light output characteristics, and both blue and polywave light-emitting diode (LED) LCUs may provide a

highly inhomogeneous spectral emission and radiant exitance beam profile.²⁰ This aspect is directly reflected in an uneven resin polymerization, manifested in poor physical properties in RBCs.²¹ Particularly when using polywave LED LCUs, the impact of the location of different LED types on the polymerization quality and homogeneity is questioned. This potential negative effect might even be intensified in deeper and peripheral regions of a BF-RBC restoration, considering that the light reaching deeper RBC layers is dependent on the thickness of the layer as well as on the wavelength of light.²² It has been shown that merely 24%-44% of the incident blue light and 9%-14% of the incident violet light is transmitted through 2-mm BF-RBC increments. These values are yet further reduced in 4-mm increments (9%-24% and 3%-9%, respectively).²²

Therefore, the following null hypotheses were tested: 1) the LCU type, 2) the placement of the LCU (by rotation in 120°), 3) the location of measurement (center – peripheral or surface – depth), and 4) the BF-RBC have no effect on hardness (HV) and depth of cure (DOC).

METHODS AND MATERIALS

The effect of two blue and one violet-blue LED LCUs (Table 1) on the variation of hardness within simulated large (10-mm) cavities restored with three bulk-fill RBCs (Table 2) was assessed by means of a universal hardness device. The BF-RBCs were polymerized as recommended by the manufacturer (Table 2). Incident light, irradiance, and spectral distribution were recorded for each LCU.

Characterization of the LCUs

The irradiance and spectral distribution of the used LCUs were measured on a laboratory-grade NIST-referenced USB4000 Spectrometer (Managing Accurate Resin Curing [MARC] System, Bluelight Analytics Inc, Halifax, Canada). The distance between sensor and light guide tip was set at 3 mm to reproduce the exposure distance used for curing the analyzed BF-RBC specimens. The recorded irradiance corresponds thus to the irradiance received by the RBC specimen. The miniature fiber-optic spectrometer uses a 3648-element Toshiba linear CCD array detector and high-speed electronics. The spectrometer has been spectroradiometrically calibrated with Ocean Optics' NIST-traceable light source (300-1050 nm). The system uses a CC3-UV Cosine Corrector to collect radiation over a 180° field of view, thus mitigating the effects of optical interference associated with light collection sam-

Table 1: Characteristics of the Analyzed Light Curing Units

LCU	Bluephase Style (With Updated Light Guide Tip)	Elipar Elipar DeepCure	Demi Ultra
Manufacturer	Ivoclar Vivadent	3M ESPE	Kerr
Series No.	1110002669	939133000012	787016098
Wavelength range, nm	385-515m	430-480	450-470

pling geometry. The detector of the spectrometer had a diameter of 4 mm. Consequently, irradiance reaching this area was considered. Irradiance at a wavelength range of 360 to 540 nm was individually collected at a rate of 16 records/s on five occasions. The sensor was triggered at 20 mW.

Hardness Profiles

To evaluate the variation in micro-hardness as a function of LCU and material, 6-mm-high disc-shaped specimens with a diameter of 10 mm were prepared by applying the BF-RBCs in one increment in metal molds ($n=6$ for each material and LCU). The material was light cured in one step according to the manufacturers' recommendation, as indicated in Table 2. The curing unit was placed by means of a mechanical arm at 3-mm distance²³ from the specimen's surface, simulating a clinically relevant exposure condition, and it was applied in three different positions, by rotating the LCU in 120° steps. The position of the blue and violet LEDs in the violet-blue LCU Bluephase Style is indicated in Figure 1 for each rotation in relation to the specimen's surface. The analyzed blue LED LCUs contained similar LEDs that were distributed symmetrically. However, the blue LCUs were also rotated in a similar way as the violet-blue LCU to allow for equivalent measurement conditions.

Specimens were stored after curing in 37 °C distilled water for 24 hours, sectioned prior to testing in the middle along the z -axis as indicated in Figure 1 with a slow-speed diamond saw (Isomet low-speed saw, Buehler, Germany) under water cooling, then ground with SiC paper until grit 4000 and polished

with a diamond suspension (mean grain size: 1 μ m). Measurements were made with an automatic micro-hardness indenter (Fischerscope H100C, Fischer, Sindelfingen, Germany) along the z -axis in 500- μ m steps, starting at 0.5 mm under the surface that has been exposed to the curing light. Hardness profiles were measured through the middle as well as at two peripheral positions, located 4 mm apart from the middle of the specimens. These profiles are indicated as 1 mm (peripheral left), 5 mm (center), and 9 mm (peripheral right).

The test procedure was carried out force controlled, while the test load increased for 20 seconds and decreased for 20 seconds with constant speed between 0.4 mN and 500 mN. Load and penetration depth of the indenter were continuously measured during the load-unload hysteresis. Universal hardness is defined as the test force divided by the apparent area of indentation under applied test force. From a multiplicity of measurements stored in a database supplied by the manufacturer, a conversion factor between Universal hardness and Vickers hardness (HV) was calculated and input into the software.

The DOC, usually acknowledged as the thickness of an RBC that is adequately cured or rather as the depth where HV equals the surface value multiplied by an arbitrary ratio, usually 0.8²⁴ (= HV-80%), was also calculated. Therefore, for each sample, HV in the depth was compared within each material to the 0.5-mm subsurface value (mean value of all specimens) and noted when it became less than 80%. This depth is defined as DOC.

Table 2: Characteristics of the Analyzed Bulk-Fill Resin-Based Composites

RBCs	Filtek Bulk-Fill, FBF	Tetric Evo Ceram Bulk Fill, TEVOBF	SonicFill, SF
Manufacturer	3M ESPE	Ivoclar Vivadent	Kerr
Lot No.	N637888	T14294	5338297
Exposure time, s	20	10	20
Shade	A3	IVA	A3
Filler wt%	76.5	76-77	83.5
Filler Vol. %	58.4	53-54	n/a
Light-initiator system	Camphorquinone/amine	Camphorquinone/amine, germanium-based, acyl phosphine oxide	Camphorquinone/amine

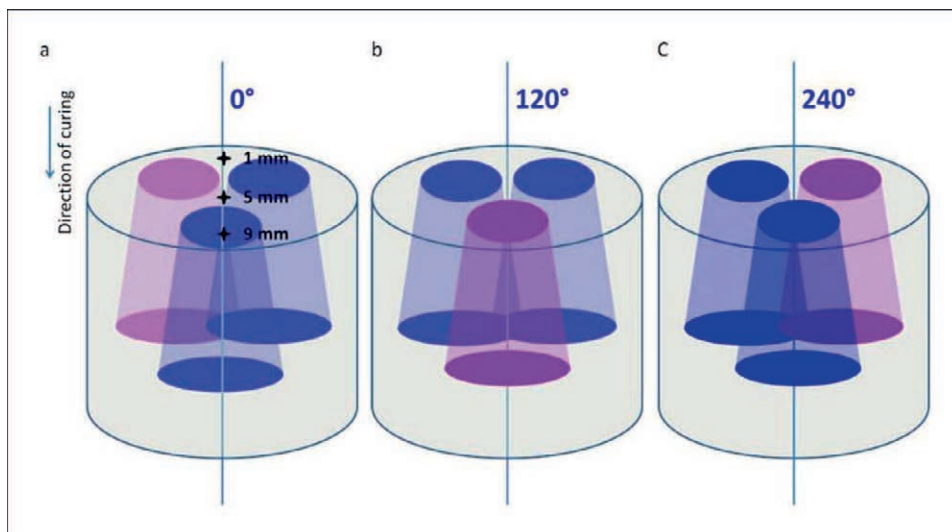


Figure 1. Projection of the light-emitting diodes (Bluephase Style) on specimens' surface (diameter = 10 mm, depth = 6 mm) during curing. The vertical line indicates the section plane; degree (0° , 120° , and 240°) indicates the rotation of the curing unit. The right half of the specimen was used for micro-hardness measurements. Three hardness line profiles were evaluated in depth in the section plane through the middle (5 mm) as well as at 4-mm left (1 mm from the specimen's margin = peripheral left) and right (9 mm from the specimen's margin = peripheral right) from the middle of the specimens.

Statistical Analyses

A Shapiro-Wilk test verified the normal distribution of the data. A multivariate analysis (general linear model) assessed the effect of the parameters LCU (three different LCUs), LCU-rotation ($3 \times 120^\circ$), RBC (three different BF-RBCs), depth (0.5 to 5.5 mm), and width (peripheral and center) as well as their interaction terms on HV and DOC. The partial eta-squared statistic reported the practical significance of each term, based on the ratio of the variation attributed by the effect. Larger values of partial eta-squared indicate a greater amount of variation accounted for by the model, which figure up to a maximum of 1.0. In addition, a one-way analysis of variance (ANOVA) with post hoc Tukey HSD test was used. In all statistical tests, p values < 0.05 were considered statistically significant when using SPSS Inc (version 23.0, Chicago, IL, USA).

RESULTS

The incident irradiance was identified as 1786.0 ± 56.9 mW/cm² for Elipar DeepCure, 1550.8 ± 50.3 mW/cm² for Demi Ultra, and 1184.6 ± 3.9 mW/cm² for Bluephase Style (Fig. 2a). Elipar DeepCure and Demi Ultra were identified as blue-LED LCUs, with a peak maximum located at 449 nm and 460 nm, respectively. Bluephase Style is a violet-blue LED-LCU, with two distinct peaks at 410 nm (violet) and 456 nm (blue; Figure 2b).

The effect of the parameter LCU rotation on HV was not significant ($p=0.109$) in any of the analyzed LCUs. Therefore, data of the three rotations of an LCU were pooled together. No significant difference was identified in HV for the positions peripheral-left and peripheral-right ($p=0.615$), while the difference

between peripheral-left and center ($p<0.001$) as well as peripheral-right and center ($p<0.001$) was significant. Consequently, the peripheral-left and peripheral-right data were merged as well. All other analyzed parameters, LCU, RBC, depth, and width (defined now as either peripheral or center), exerted a significant ($p<0.001$) effect on HV. The highest influence on HV was exerted by the parameter RBC ($p<0.001$; partial eta squared $\eta_p^2=0.805$), followed by the parameter depth ($p<0.001$; partial eta squared $\eta_p^2=0.788$; Figure 3). As a result, statistically similar HV values were identified in a depth of 0.5 mm for SonicFill (SF) and Filtek Bulk-Fill (FBF; $p=0.145$) that were significantly higher compared with values measured in Tetric Evo Ceram Bulk Fill (TEVOBF; $p<0.001$; Figure 4a). At a depth of 4 mm, SF and TEVOBF showed statistically similar HV values ($p=0.06$) that were lower compared with FBF ($p<0.001$; Figure 4b). The effect of width on HV ($p<0.001$; partial eta squared $\eta_p^2=0.292$; Figure 5a-c) was consistently lower and the effect of LCU was still significant but very low ($p<0.001$; partial eta squared $\eta_p^2=0.057$).

The DOC varied among 3.46 mm (SF, peripheral, LCU Bluephase Style) and 5.50 mm (FBF, center, all LCUs; Figure 6). The highest influence on DOC was exerted by the parameter RBC ($p<0.001$, $\eta_p^2=0.774$), followed by width ($p<0.001$, $\eta_p^2=0.554$), while the influence of the LCU was extremely low ($p<0.06$, $\eta_p^2=0.070$). The DOC measured peripheral or center was similar in FBF ($p=0.124$). DOC measured peripheral, compared with the DOC measured center, was at 1.12 ± 0.11 mm lower in TEVOBF ($p<0.001$) and at 1.04 ± 0.11 mm lower ($p<0.001$) in SF.

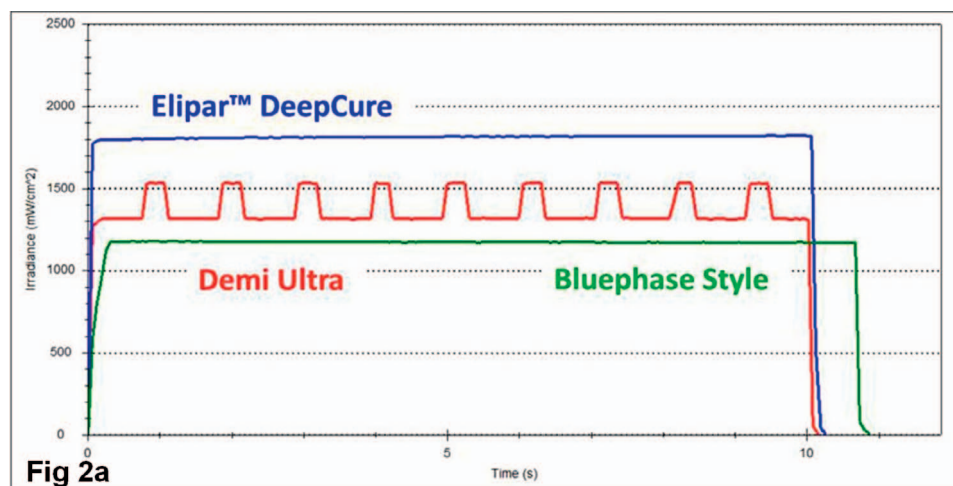
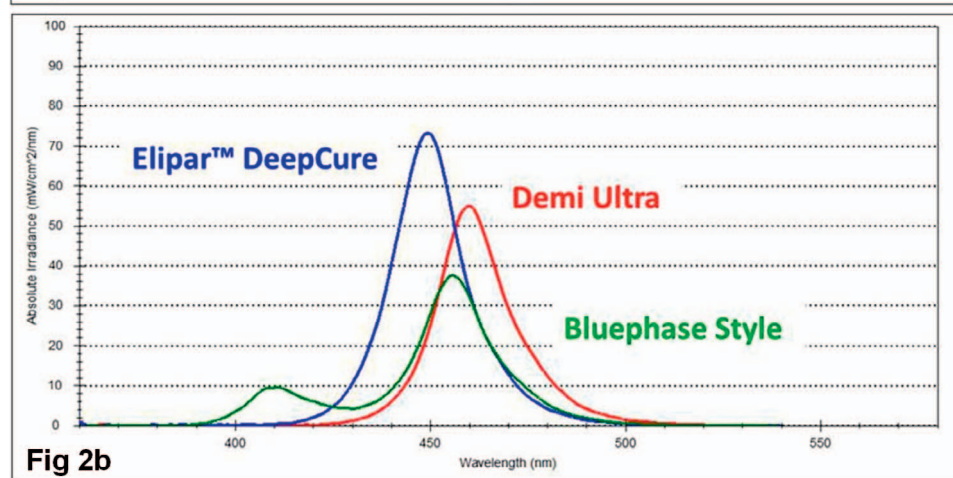


Figure 2. Characteristics of the LCUs collected at 3-mm distance from the spectrophotometer sensor. (a): LCU irradiance. (b): Emission light spectrum.



FBF reached the significantly highest DOC of 5.4 ± 0.2 mm ($p < 0.001$), while the DOC in TEVOBF (4.1 ± 0.7 mm) and SF (3.9 ± 0.6 mm) was statistically similar ($p = 0.168$). As for the LCUs, a statistically significant difference in the induced DOC was recorded only between Elipar DeepCure (4.6 ± 0.8 mm) and Demi Ultra (4.3 ± 0.9 mm; $p < 0.001$), while Bluephase Style induced a DOC of 4.5 ± 0.89 mm that was statistically similar to Elipar DeepCure ($p = 0.202$) and Demi Ultra ($p = 0.089$).

DISCUSSION

The fundamental approach of this study was to identify the efficacy of modern blue and violet-blue LED LCUs in curing adequately peripheral zones in large BF-RBC specimens. This attempt was founded in the notably heterogeneous light distribution of contemporary LCUs²⁵ that might produce irregular polymerization and inhomogeneity in mechanical properties within a RBC filling.²¹ Consequently, much effort has been currently invested to develop LCUs displaying homogeneous light beam profiles.

A recently launched LED LCU, Elipar Deep Cure-S, evidencing homogeneous light beam profile along a light guide²⁵ with a diameter of 10 mm, was used as a positive reference in the present study. The emission spectrum of Elipar Deep Cure-S indicates that the peak maximum in the blue wavelength range is shifted to a lower wavelength (449 nm) when compared with the other analyzed LCUs (460 nm and 456 nm; Figure 2b). The particularity of the blue-LED LCU Demi Ultra is seen in positioning the LEDs at the tip. This allows avoiding energy loss vs LCUs with a bent light guide and also to reduce light collimation. Two alternating levels of irradiances were identified in Demi Ultra: a base level, set at 1300 mW/cm^2 and maintained for 0.75 seconds followed by a higher irradiance level (1550 mW/cm^2) for the subsequent 0.25 seconds (Figure 2b). This switch in irradiance is repeated for each second of the curing cycle and is intended to reduce heat buildup. Apart from blue LED LCUs, a violet-blue LCU, Bluephase Style, has also been analyzed. This LCU is characterized by a lower irradiance com-

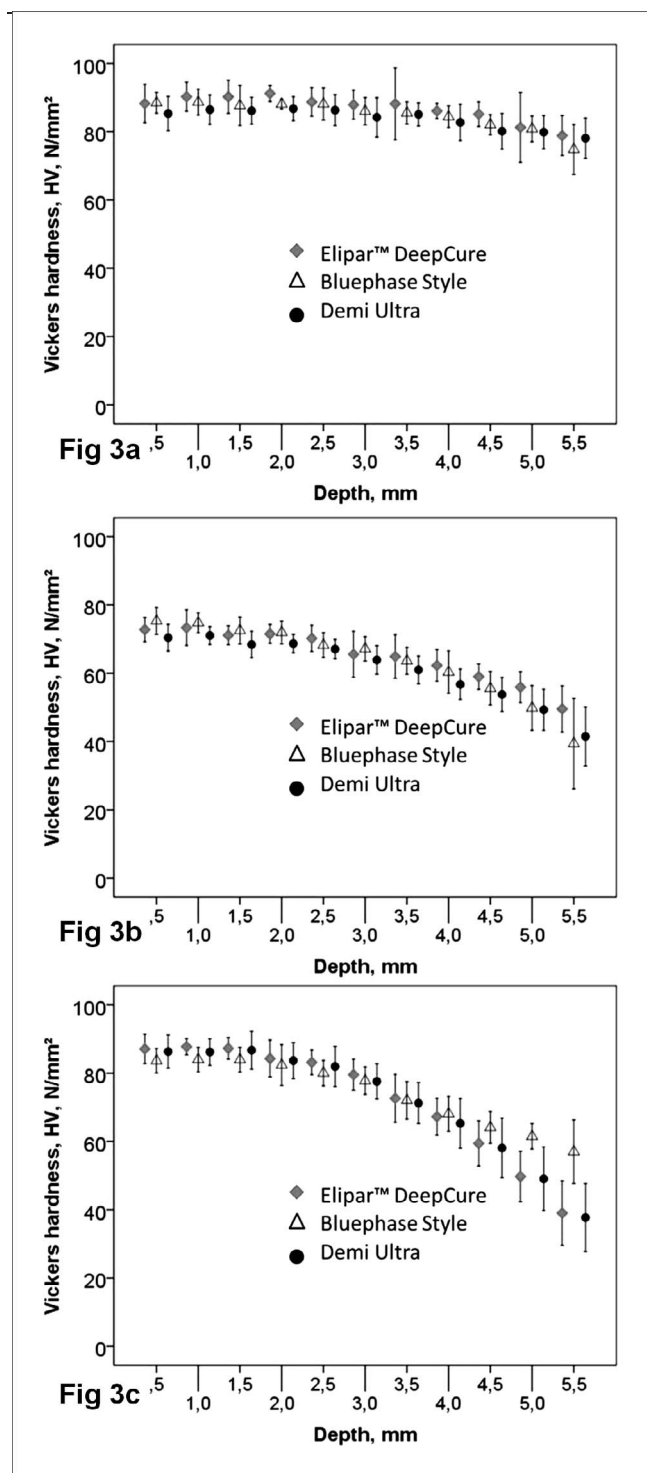


Figure 3. Mean and standard deviation of the Vickers hardness as function of depth and LCU for the bulk-fill composites. (a): FBF. (b): TEVOBF. (c): SF. Compared with values measured at a depth of 0.5 mm, one-way ANOVA ($p < 0.05$) identified in FBF a significant decrease in HV starting with a depth of 4.5 mm (Bluephase Style) or 5 mm (Elipar DeepCure and Demi Ultra). The corresponding depths were 2.5 mm (Bluephase Style) and 3 mm (Demi Ultra and Elipar DeepCure) in TEVOBF and 3 mm in SF (all LCUs). FBF: A significant difference among LCUs was identified only at 1-, 1.5-, 4-, and 4.5-mm

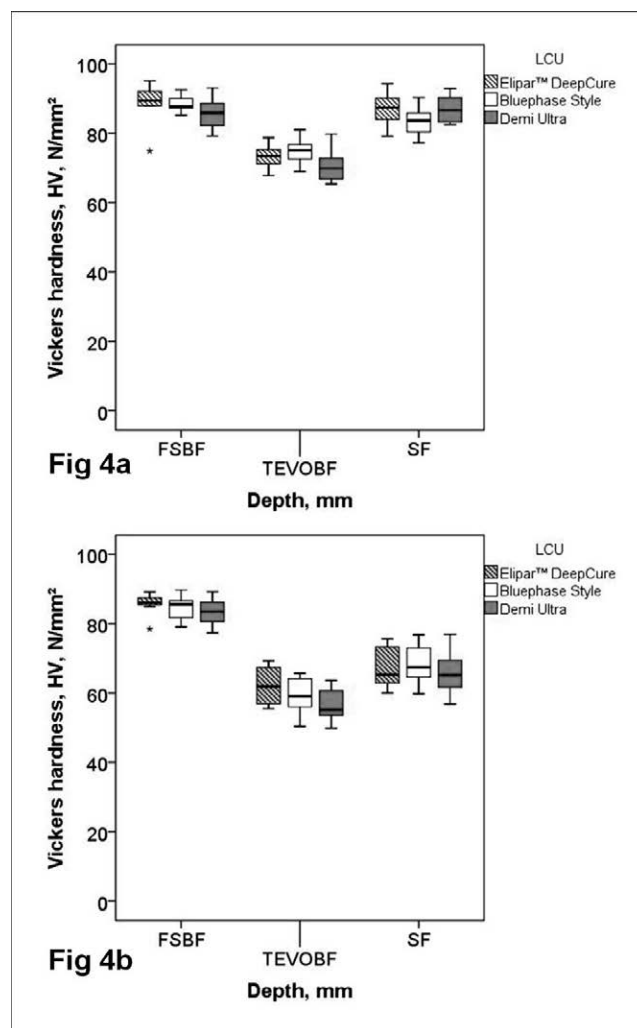


Figure 4. Vickers hardness as a function of material and LCU at depths of (a) 0.5 mm and (b) 4 mm.

depths (Elipar DeepCure induced significantly higher HV values compared with Demi Ultra, while Bluephase Style performed comparably to both LCUs). TEVOBF: Bluephase Style induced significantly higher HV values than Demi Ultra up to a depth of 2 mm, while Elipar DeepCure performed comparably to both LCUs. Subsequently, up to a depth of 3.5 mm, all three LCUs had statistically similar performance. Following that, Elipar DeepCure performed significantly better compared with Demi Ultra at 4 and 4.5 mm, while Bluephase Style performed comparably to both LCUs. At depths up to 5.5 mm, Elipar DeepCure performed significantly better compared with both other LCUs. SF: Elipar DeepCure induced significantly higher HV values than Bluephase Style up to a depth of 1 mm, while Demi Ultra performed comparably to both LCUs. Subsequently, up to a depth of 4 mm, all three LCUs had a statistically similar performance. At 4.5 mm, Bluephase Style performed significantly better than Demi Ultra, while Elipar DeepCure performed comparably to both LCUs. Bluephase Style then performed significantly better compared with the other LCUs.

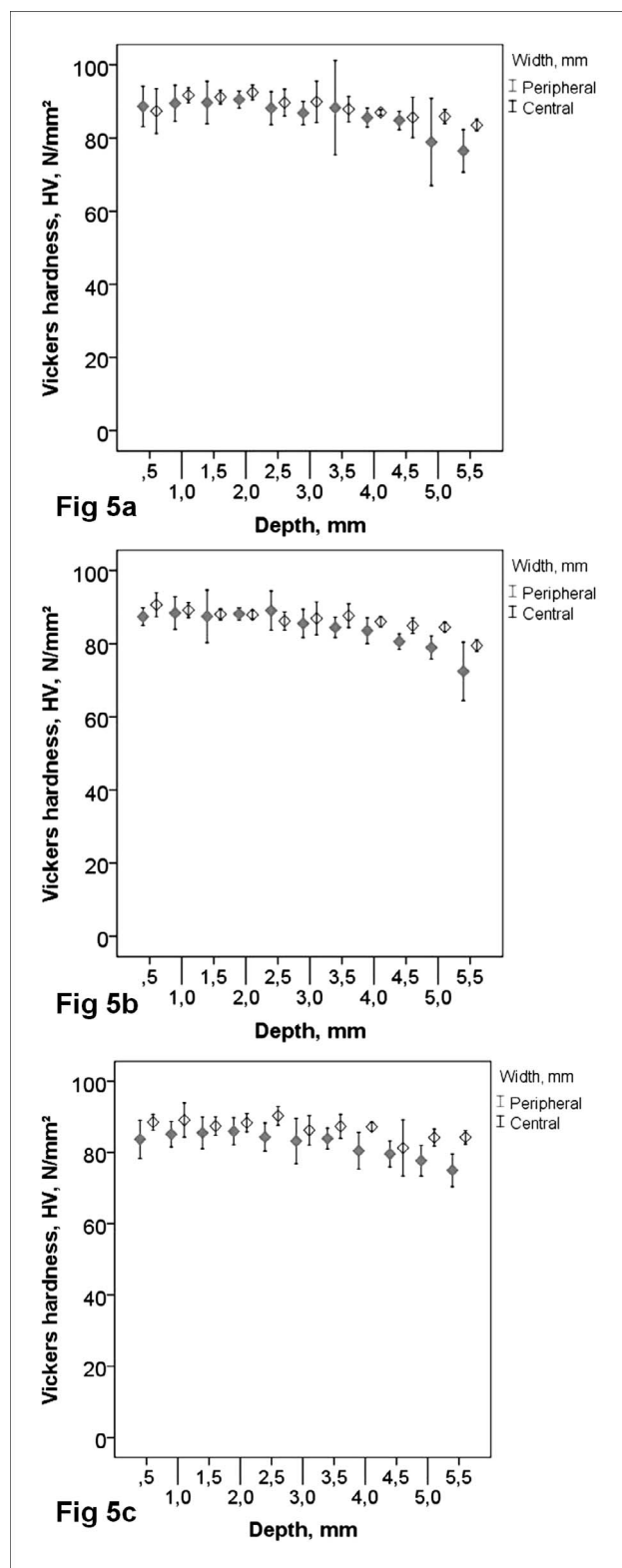


Figure 5. Mean and standard deviation of the Vickers hardness as a function of depth and width (center and peripheral position) after curing with the LCUs. (a): Elipar DeepCure. (b): Bluephase Style. (c): Demi Ultra, exemplified for the bulk-fill composite FBF.

pared with the above described LCUs, but the emission spectrum covers a wider wavelength range (390-500 nm) that allows initiating camphorquinone (CQ) and also alternative photo-initiators. This is particularly important when curing TEVOBF, for it contains, apart from a CQ/amine system that is present in FBF and SF, also two Norrish Type 1 initiators, namely, a dibenzoyl germanium derivative, bis-(4-methoxybenzoyl)diethylgermane), and an acyl phosphine oxide (APO).

Germanium-based photo-initiators for dental materials are characterized by lower absorption maxima ($\lambda_{\max} = 411$ nm or $\lambda_{\max} = 418$ nm) when compared with CQ ($\lambda_{\max} = 468$ nm).²⁶ They match accordingly well the emission spectrum (Figure 2b) of the violet-blue LCU Bluephase Style. The absorption maxima of germanium-based photo-initiators (411 nm, 418 nm) lie outside the emission spectra of blue LED LCUs (Figure 2b). However, their absorption spectra start in the violet wavelength range and extend up to 455 nm in the blue wavelength range, matching in large parts the emission spectra of blue LED LCUs.

It seems that the wider emission spectrum of Bluephase Style that initiates CQ, APO, and the germanium-based photo-initiator compensates for its lower irradiance, since it induced in TEVOBF statistically similar DOC values as Elipar DeepCure and Demi Ultra. Comparing both analyzed blue LED LCUs, it become evident that the emission spectrum of Elipar DeepCure starts at lower wavelength (410 nm) compared with Demi Ultra (425 nm), thus providing a better matching with the absorption spectrum of the germanium-based photo-initiator. Both effects—a slightly higher irradiance (Figure 2a) and a better spectra match with the germanium-based photo-initiator—may contribute to a slight but significant improvement in mechanical properties in TEVOBF when using Elipar DeepCure vs Demi Ultra.

Apart from spectra-matching considerations, the reactivity of the germanium-based photo-initiator was identified to be much higher compared with CQ, a fact attributed to the larger molar extinction coefficient ($\epsilon\lambda$).²⁶ A large $\epsilon\lambda$ indicates a high probability of light absorption at a certain wavelength, leading to large quantum yields of the initiating species and improved degree of conversion. For germanium-based initiators, a significantly stronger absorption at their maximum was identified ($\epsilon = 1460$ dm²mol⁻¹ and $\epsilon = 5470$ dm²mol⁻¹) in comparison to CQ ($\epsilon = 380$ dm²mol⁻¹).²⁶

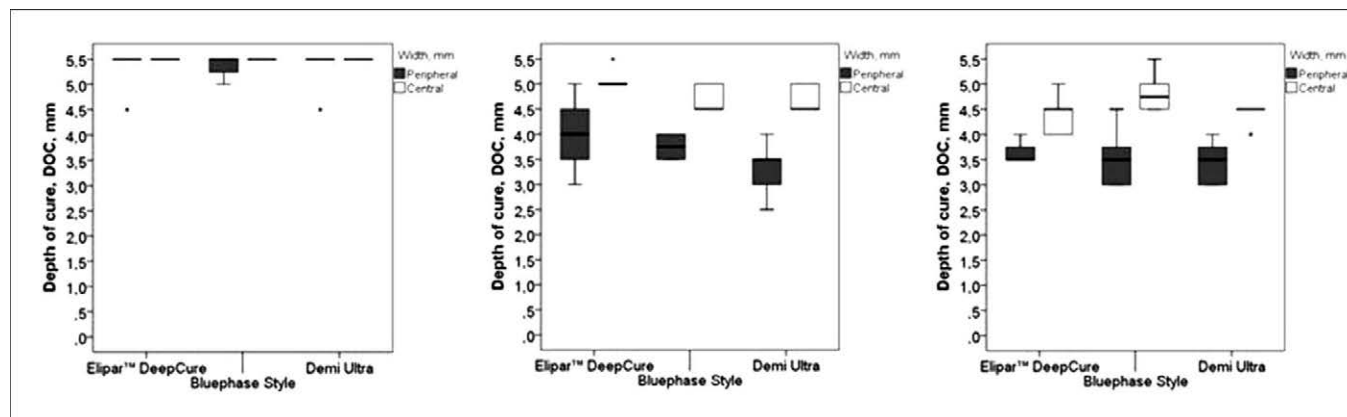


Figure 6. Depth of cure as a function of material and LCU. (a): FBF. (b): TEVOBF. (c): SF.

As for the APO initiator that is also contained in TEVOBF, it can be activated only by the violet-blue LCU Bluephase Style.²⁷ Similar to the germanium-based photo-initiators, APO is more efficient than CQ, since it undergoes homolytic α -cleavage of the C-P bond and generates two free radical species, that is, $[-(O)=C\cdot]$ and $[\cdot P(=O)<]$, both capable of initiating polymerization.²⁷ Albeit very efficient, APO is activated at shorter wavelengths compared with CQ, while the attenuation of light in depth is known to follow an exponential decay (Lambert's Law)²⁸ and is wavelength dependent. Hence, the violet light that is characterized by shorter wavelengths will be attenuated faster in depth than the blue light.²² The contribution of APO to the polymerization process of TEVOBF in deeper layers must therefore be considered as very low.

When comparing the quality of curing in center vs peripheral areas, differences in HV are low and started to be perceptible only at depths larger than 2.5 mm. It was also found in the present study that this difference is material dependent. Interestingly, the DOC measured for FBF was neither dependent from the LCU nor from the width (center-peripheral), attesting to a sufficiency of curing in peripheral areas under all analyzed curing conditions. As for TEVOBF, both LCU ($\eta_p^2=0.28$) and width ($\eta_p^2=0.672$), but not their interaction product, exerted an effect of DOC. Related to Elipar DeepCure, the DOC induced by Bluephase Style in TEVOBF was about 0.338 ± 0.141 mm lower, while the DOC induced by Demi Ultra was about 0.583 ± 0.137 mm lower. The peripheral areas cured with Demi Ultra reached in TEVOBF a mean DOC value that was lower than the 4-mm limit stipulated as a minimum for adequate curing in bulk-fill resin

composites. It must, however, be emphasized that TEVOBF specimens were cured for only 10 seconds compared with 20 seconds as done in all other materials and thus received half of the radiant exposure applied on the other materials. It becomes obvious that exposure duration of 10 seconds cannot be generalized for curing this material, since one of three analyzed LCUs induced peripheral DOC values lower than 4 mm. An exposure time of 10 seconds might become insufficient at peripheral regions of large fillings. As for SF, only the width showed an influence ($\eta_p^2=0.658$) on DOC, but not the LCU, while all measured peripheral DOC values were lower than 4 mm. In contrast, values measured in the center region exceeded this limit. This fact must be related to the higher filler content in SF, which generates a less translucent material in which light is more strongly attenuated. For more homogeneous properties, it is suggested to cure this material longer as recommended by the manufacturer, thus as long as 20 seconds.

The DOC was defined in the present study as the depth at which HV equals 80% of the HV value measured at a subsurface of 0.5 mm. While this method is well established, there might be reticence in accepting a 20% decrease in mechanical properties as an adequate curing. Besides, the used LCUs were applied in this study design perpendicular to the specimen's surface, at an exposure distance of 3 mm that simulates clinically relevant curing conditions for curing BF-RBCs fillings. All deviations from these curing conditions owing to a particular clinical situation, such as increased exposure distance or variances from perpendicularity when applying the LCU on the filling, must result in an enhanced exposure time.

CONCLUSIONS

The analyzed LCUs showed less to no difference in their effect on the measured properties within one BF-RBC. The placement of the LCU by rotation in 120° steps as well as the peripheral positions measured 4-mm left and right apart from the center showed no effect on the analyzed properties in any material.

Whether a BF-RBC filling is cured peripherally as well as in the center depends more on the material than on the curing unit used.

Acknowledgements

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Conflict of Interest

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

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Effect of Baicalein on Matrix Metalloproteinases and Durability of Resin-Dentin Bonding

J Li • B Chen • N Hong • S Wu • Y Li

Clinical Relevance

Baicalein used as a preconditioner in an etch-and-rinse adhesive system has potential value in clinical bonding procedures because it can effectively improve resin-dentin bonding durability and reduce interface nanoleakage *in vitro*.

SUMMARY

Objective: In an attempt to increase resin-dentin bonding quality, this study used baicalein as a preconditioner in an etch-and-rinse adhesive system to evaluate its effect on matrix metalloproteinases (MMPs) and adhesive durability.

†Jiayan Li, BDS, postgraduate, Department of Prosthodontics, Guanghua School of Stomatology, Hospital of Stomatology, Sun Yat-sen University; Guangdong Provincial Key Laboratory of Stomatology, Guangzhou, China

†Boli Chen, BDS, postgraduate, Department of Prosthodontics, Guanghua School of Stomatology, Hospital of Stomatology, Sun Yat-sen University; Guangdong Provincial Key Laboratory of Stomatology, Guangzhou, China

Nanrui Hong, BDS, postgraduate, Department of Prosthodontics, Guanghua School of Stomatology, Hospital of Stomatology, Sun Yat-sen University; Guangdong Provincial Key Laboratory of Stomatology, Guangzhou, China

*Shuyi Wu, DDS, PhD, Department of Prosthodontics, Guanghua School of Stomatology, Hospital of Stomatology, Sun Yat-sen University; Guangdong Provincial Key Laboratory of Stomatology, Guangzhou, China

*Yan Li, DDS, PhD, Department of Prosthodontics, Guanghua School of Stomatology, Hospital of Stomatology, Sun Yat-sen University; Guangdong Provincial Key Laboratory of Stomatology, Guangzhou, China

*Corresponding authors: No. 56 Lingyuan West Road, Guangzhou, Guangdong, China, 510055; e-mail: shuyiwu@21cn.com (S Wu), dliyan2004@126.com (Y Li)

†These authors contributed equally to the work.

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le in as a preconditioner in an etch-and-rinse adhesive to evaluate its effect on matrix metalloproteinases (MMPs) and adhesive durability.

Methods: As a MMP inhibitor and potential collagen cross-linking agent, baicalein was used as a preconditioner in an etch-and-rinse adhesive system. The degree of conversion was evaluated by Fourier-transform infrared spectroscopy. EnzChek gelatinase/collagenase assay kits were then used to detect the MMP inhibitory effect of different concentrations of baicalein (0.1, 0.5, 2.5, and 5.0 µg/mL) on dentin powders. During *in vitro* bonding procedures, flat dentin surfaces on sound third molars were preconditioned with 2.5 µg/mL baicalein after being acid-etched; this step was followed by continuation of adhesive processes and build-up of resin composite. After resin-dentin stick preparation, bonding strength, failure mode, and interface nanoleakage were respectively evaluated via microtensile testing, stereomicroscopy, and field emission scanning electron microscopy either immediately or after storage in artificial saliva for three or six months. Data were analyzed by two-way analysis of variance and Tukey test ($\alpha=0.05$).

Results: Baicalein at a concentration of 0-5.0 µg/mL did not influence the conversion of

adhesives. However, it inhibited the activities of dentin-bond gelatinase and collagenase, especially at a concentration of 2.5 µg/mL, while effectively increasing microtensile bonding strength and decreasing nanoleakage *in vitro*, both immediately and after aging.

Conclusions: Baicalein used as preconditioner in an etch-and-rinse adhesive system has an anti-MMP function and effectively improves resin-dentin bonding durability *in vitro*, which has potential value in clinical bonding procedures.

INTRODUCTION

Although innovation in adhesive techniques has occurred since the “hybrid layer” was proposed,¹ the problem that exposed collagen fibrils deep inside the hybrid layer are vulnerable to hydrolysis or enzymolysis remains unsolved.²⁻⁴ The discrepancy between the depth of acid-etching and resin infiltration in etch-and-rinse adhesive system leads to further catastrophic failure of the bonding interface, which significantly influences the durability of resin-dentin adhesive.

Studies⁵⁻¹⁶ have found that the use of matrix metalloproteinase (MMP) inhibitors or cross-linkers is effective in protecting the dentin collagen matrix in the hybrid layer. MMP inhibitors such as chlorhexidine can terminate collagen degradation caused by host-derived endogenous MMPs,⁵⁻⁷ including gelatinases (MMP-2 and MMP-9) and collagenases (MMP-1 and MMP-8).² Furthermore, cross-linking agents stabilize collagen fibrils and interfere with the identification and complexation of protease.⁸ Some cross-linking agents, such as glutaraldehyde,^{9,10} proanthocyanidins,¹¹⁻¹⁴ and genipin,^{15,16} can markedly increase immediate or aging dentin bonding strength. However, because of cytotoxicity,^{17,18} the use of chemical cross-linkers is restricted to laboratory experiments, while natural agents have a greater potential for clinical usage as a result of their biosafety and cost-effectiveness.

Baicalein (BAI) is one of the major flavonoids in *Scutellaria baicalensis*. Recently, an increasing number of studies have demonstrated that BAI can inhibit the activity and expression of MMP-2 and MMP-9 via PI3K/AKT,^{19,20} p38-MAPK-NF-κB,^{21,22} and MAPK-ERK1/2^{23,24} signal pathways in various tumor cells. The effect of BAI on these anti-MMPs may offer more plausible reasons for its use in dentin adhesives. Moreover, BAI and proanthocyanidins are natural plant polyphenols that share a similar molecular structure with phenolichydroxyl function-

al groups,^{25,26} suggesting they share comparable cross-linking properties. A previous study²⁷ showed that BAI could improve enzymatic resistance of the dentin collagen matrix better than could proanthocyanidins and quercetin at a concentration of 50 g/L. Therefore, we speculated that because of its potential MMP inhibition and cross-linking abilities, BAI is capable of stabilizing collagen fibrils and protecting the integrity of the hybrid layer in dentin bonding.

The purpose of this study was to evaluate the influence of BAI on MMPs and resin-dentin adhesive. The null hypothesis tested was that BAI used as a preconditioner in an etch-and-rinse adhesive system does not stabilize resin-dentin interfaces or increase bonding durability.

METHODS AND MATERIALS

Degree of Conversion (DC) Measurement

A flow chart of this study is shown as Figure 1. The resin monomer conversion of the preconditioner-adhesive complex was determined using Fourier-transform infrared spectroscopy (FTIR), as previously reported,^{28,29} with a slight modification. BAI (Sigma-Aldrich, St Louis, MO, USA) was dissolved in 1% dimethyl sulfoxide (DMSO) to achieve the required concentrations (0.1, 0.5, 2.5, and 5.0 µg/mL) as a preconditioner. Then 5 µL of preconditioner was smeared upon clean 2.0 × 2.0-cm² polypropylene membranes. After gentle drying, 5 µL of AdperTM Single Bond 2 (3M ESPE, St Paul, MN, USA) adhesive was applied, and another cleaned polypropylene membrane was covered (n=5). DMSO at 1% in distilled water was used as a solvent control group. Absorption spectra (before and 20 seconds, 60 seconds, five minutes, 10 minutes, and 30 minutes after curing) were then observed with a FTIR spectrometer (Nicolet 6700, Thermo Scientific, Waltham, MA, USA). Each specimen was cured for 20 seconds at 0.5-cm distance with a light-curing unit (EliparTM S10 LED, 3M ESPE). The DC of unreacted double carbon bonds was calculated from the ratio of absorbance intensities of aliphatic C=C (peak at 1638.6 cm⁻¹) and aromatic C=C (peak at 1609.4 cm⁻¹) before and after curing using the following formula:

$$DC(\%) = \left(1 - \frac{Abs(C=C)_{After}/Abs(C\cdots C)_{After}}{Abs(C=C)_{Before}/Abs(C\cdots C)_{Before}} \right) \times 100\%.$$

Tooth Collection

Caries-free human third molars of patients aged 18-39 years were collected with informed consent under

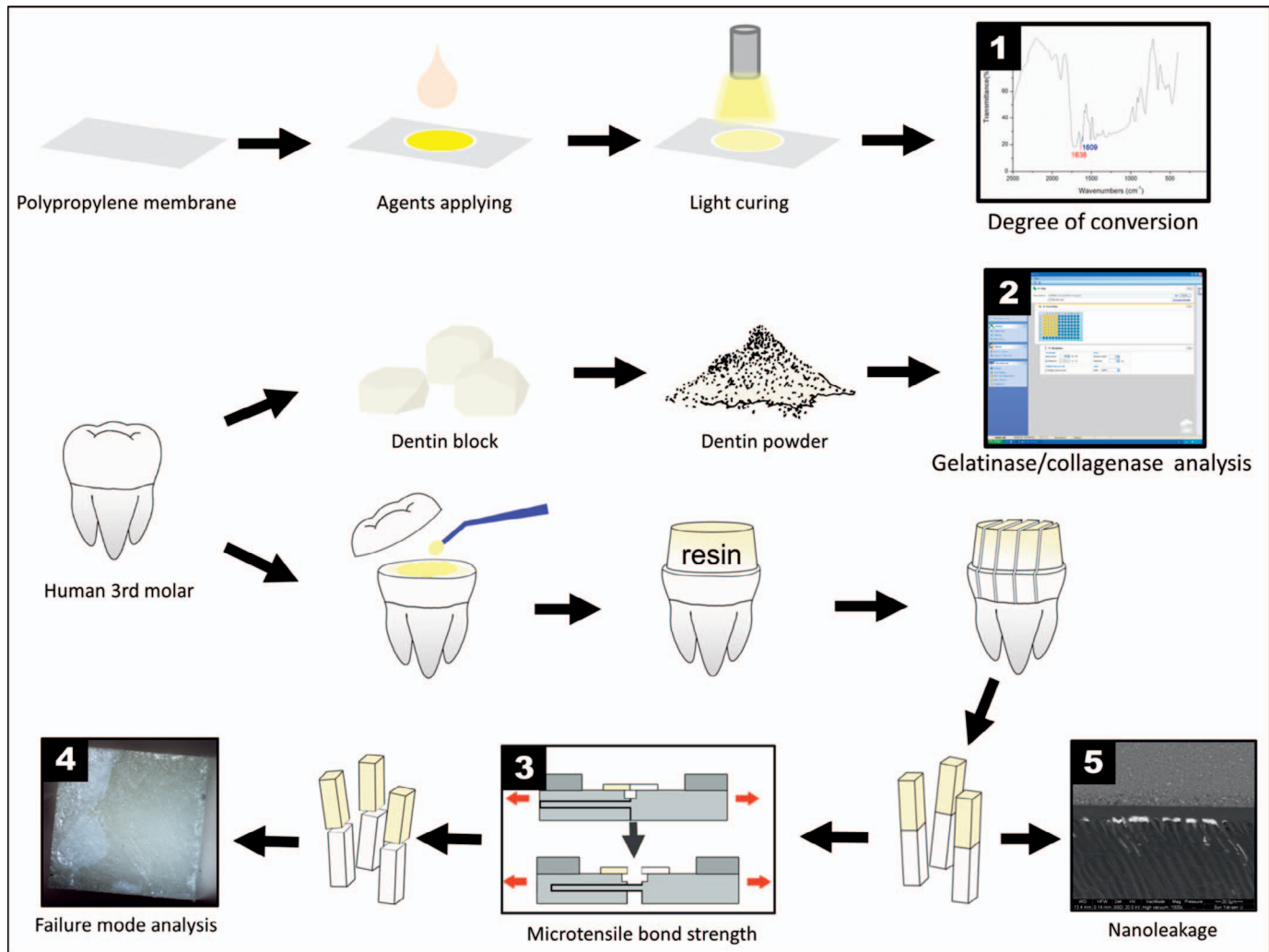


Figure 1. Flow chart of this study. (1) Degree of conversion measurement obtained using FTIR. (2) Gelatinase and collagenase analysis obtained using EnzChek assay kits. (3) Bonding strength evaluation using microtensile tester. (4) Failure mode analysis using stereomicroscope. (5) Nanoleakage evaluation by FE-SEM.

a protocol approved by the relevant ethics committee (approval code ERC-2014-23). Teeth were cleaned and stored in 0.5% chloramine-T solution at 4°C and were used within two weeks.

Gelatinolytic and Collagenolytic Activity Evaluation

The gelatinolytic and collagenolytic activities of dentin were measured by means of EnzChek gelatinase/collagenase assay kits (E-12055, Molecular Probes, Eugene, OR, USA), basically as previously reported.³⁰ Following this step, teeth were sectioned at the level of the cemento-enamel junction and enamel and pulp tissues were removed. After verifying that only the dentin remained using a stereomicroscope (M205A, Leica, Solms, Germany), the dentin blocks were dehydrated in anhydrous

acetone at 4°C, then frozen in liquid nitrogen; we then continued triturating and sieving through multiple screens to obtain a fine powder smaller than 80 µm in size. The dentin powders were then demineralized with 35% phosphoric acid for 15 seconds and rinsed with distilled water.

After drying, the demineralized dentin powders were equally assigned to seven groups, as follows: preconditioned with BAI (0.1, 0.5, 2.5, and 5.0 µg/mL BAI), 2% chlorhexidine, 1% DMSO, or distilled water for 120 seconds, respectively. Adper™ Single Bond 2 was applied for one minute, and the reaction was stopped by acetone; this step was followed by rinsing and dehydration. Eighty milligrams of dentin powders and 20 µL of 100 µg/mL gelatin/collagen substrates were added to 96-well plates and incubated at 37°C (n=5). Fluorescent cleavage products were

recorded under a microplate reader (Infinite 200 PRO, Tecan, Männedorf, Switzerland) with ~ 495 -nm absorption maxima and ~ 515 -nm fluorescence emission maxima at one hour, 24 hours, 48 hours, and 72 hours. The relative fluorescence unit (RFU) of each well was corrected by subtracting the background fluorescence value of the non-enzyme control. Tests were repeated twice and the average taken.

Microtensile Bond Strength (μ TBS) Evaluation

The occlusal enamel and superficial dentin of teeth were removed perpendicular to the long axis of each tooth. After verification using a stereomicroscope, a standardized smear layer was created on the exposed middle coronal dentin with 600-grit silicon carbide paper. The teeth were then equally and randomly assigned to four treatment groups: exposed dentin surfaces were preconditioned with 2.5 μ g/mL BAI, 5% glutaraldehyde, 1% DMSO, and distilled water for 120 seconds for each group after etching with 35% phosphoric acid for 15 seconds. Adper™ Single Bond 2 adhesive was applied and cured and then the coronal height was reconstructed with 4-5-mm thick layers of resin composite (Filtek Z250, 3M ESPE). After storing in distilled water at 37°C for 24 hours, teeth were sectioned perpendicular to the long axis of the adhesive surfaces with cutting equipment (Accotom-50, Struers, Rodovre, Denmark) to create 0.9 mm $\times$ 0.9 mm $\times$ 8 mm resin-dentin sticks as nontrimming specimens for the microtensile test.³¹ Each tooth was cut into 12 sticks. These sticks were then randomly divided into three subgroups; one group was tested immediately and the others were stored in artificial saliva at 37°C for three and six months as aging groups. The artificial saliva was changed weekly.

The adhesive surface area (S) of each resin-dentin stick was calculated with the help of a digital caliper (with an accuracy of 0.01 mm) and then microtensile testing was performed at a loading speed of 1 mm/min (T-6102K, BISCO, Schaumburg, IL, USA). The maximum loading force (F) was recorded and the μ TBS was calculated using the following formula: μ TBS (MPa) = F/S .

Failure Mode Analysis

After microtensile testing, all specimens were collected and observed with a stereomicroscope at 100 $\times$ magnification. Failure modes were classified as follows:^{32,33} 1) adhesive failure: failure occurred at the adhesive agent or in the layers between the adhesive agent and the dentin or resin; 2) cohesive

failure: failure occurred at the inner parts of the dentin or resin; and 3) mixed failure: mixture of adhesive and cohesive failures.

Interfacial Nanoleakage Testing

Three sticks of each tooth remained for interfacial nanoleakage evaluation,³⁴ including the immediate group and the three-month and six-month storage groups. Ammoniacal silver nitrate was prepared according to a protocol previously described.³⁵ The sticks were coated with two layers of nail varnish applied up to 1 mm thick on the bonded interfaces and placed in the 50 mass% ammoniacal AgNO₃ in darkness for 24 hours. They were then rinsed thoroughly in distilled water three times and immersed in photodeveloping solution for eight hours under a fluorescent light. All of the sticks were wet-polished with silicon carbide paper to remove the nail varnish and were then polished down with 800-, 1500-, 3000-, and 5000-grit silicon carbide paper. The sticks were then carbon coated and analyzed with a field emission scanning electron microscope (FE-SEM; Quanta 400F, FEI, Hillsboro, OR, USA) operated in the backscattered electron mode. Three discontinuous fields of vision at the central area of each sample's hybrid layer were photographed. The percentage of the total hybrid layer length with nanoleakage was calculated with the Image J program (ImageJ 1.48v, National Institutes of Health, Bethesda, MD, USA).

Statistical Analysis

In the bonding evaluation, the tooth was considered the statistical unit. The μ TBS (MPa) and interfacial nanoleakage (%) from the same experimental unit were averaged for statistical purposes at each storage time. After observing normality and homogeneity of variances, data were subjected to a two-way analysis of variance, and multiple comparisons were performed with the Tukey test at $\alpha = 0.05$ using SPSS 22.0 (IBM Corp, Chicago, IL, USA).

RESULTS

Degree of Conversion

The DC results are shown in Figure 2. DC increased dramatically in all groups initially, but the rate of increase decreased and tended to stabilize with time. No significant differences were found among different pretreatments groups at different time points ($p > 0.05$).

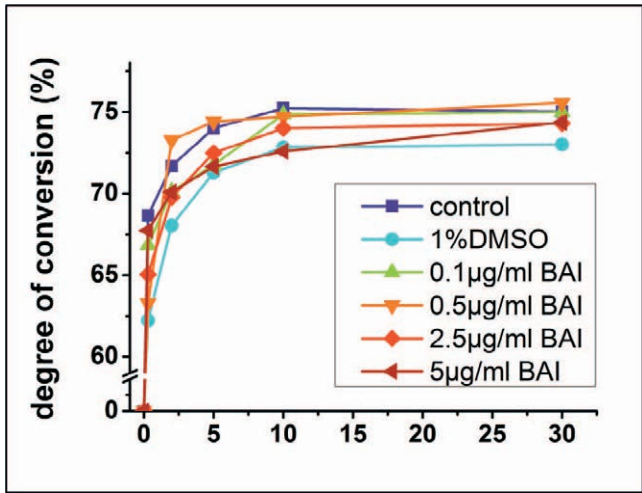


Figure 2. Degrees of conversion of different groups had no statistical differences at different time points ($p>0.05$).

Gelatinolytic/Collagenolytic Activity

Results of the EnzChek assay kit for gelatinolytic activities are shown in Table 1. The 2.5 µg/mL BAI and 2% chlorhexidine groups had significantly lower RFU values than other groups at all time points ($p<0.05$), but there were no significant differences between them ($p>0.05$). However, the 5.0 µg/mL and 0.1 µg/mL BAI groups showed no significant differences with the control group after being incubated for ≥ 48 hours ($p>0.05$). The 1% DMSO group and control group had the highest RFU values at all time points, with no significant difference between them ($p>0.05$). Similarly, collagenolytic activities increased in all groups over time (Table 2). The 2.5 µg/mL and 0.5 µg/mL BAI and 2% chlorhexidine groups showed an equally strong inhibitory effect on collagenase ($p<0.05$), while the 5.0 µg/mL and 0.1 µg/mL BAI groups had an attenuating effect, but the effect was still better than that associated with the control group ($p<0.05$). However, in contrast to gelatinase testing, significant differences between the 1% DMSO and control groups in collagenase testing were recorded at 24 hours and 48 hours ($p<0.05$).

Microtensile Bonding Strength

The results for µTBS are shown in Table 3 (premature failures were not included in the statistical analysis). In both the immediate and aging groups, the µTBS values of both the 5% glutaraldehyde and 2.5 µg/mL BAI groups were significantly higher than that of the control group ($p<0.05$). Although the 2.5 µg/mL BAI group had slightly lower values than did the 5% glutaraldehyde group, there were no significant differences between groups ($p>0.05$). The µTBS values of the 1% DMSO group were significantly lower than those of the 2.5 µg/mL BAI and 5% glutaraldehyde groups ($p<0.05$) but higher than that of the control group ($p<0.05$), with statistical differences.

Failure Mode Analysis

The results of the failure mode analysis are shown in Figure 3. Mixed and adhesive failures were the most common in each group. With the aging processing, adhesive failures increased, especially in the control and 1% DMSO groups, with growth rates of 27% and 17%, respectively. However, the increase was not significant in the 5% glutaraldehyde and 2.5 µg/mL BAI groups, with mixed failure still being the predominant failure mode after six months of aging.

Nanoleakage

The nanoleakage results are shown in Table 4, and representative images under FE-SEM are presented in Figure 4. The nanoleakage percentage of the 2.5 µg/mL BAI group was significantly lower than that in the control group, especially before aging ($p<0.05$). The 5% glutaraldehyde group acted as a positive control group and had a low nanoleakage percentage, and there was no significant difference between the result before and after aging ($p>0.05$). Although the 1% DMSO group had a relatively lower nanoleakage result than the control group ($p<0.05$), it was still higher than that in the 2.5 µg/mL BAI group ($p<0.05$).

Table 1: Relative Fluorescence Intensity (Means and Standard Deviations) of Dentin-bond Gelatinase ^a							
	Control	2% CHX	1% DMSO	0.1 µg/mL BAI	0.5 µg/mL BAI	2.5 µg/mL BAI	5.0 µg/mL BAI
1h	27.94 ± 6.33 A	3.29 ± 2.60 B	22.16 ± 1.71 AC	26.16 ± 2.32 A	17.18 ± 4.09 CD	2.75 ± 2.89 B	14.89 ± 1.34 D
24h	54.69 ± 6.53 A	14.26 ± 2.42 B	53.55 ± 3.61 A	38.37 ± 4.99 CD	28.23 ± 5.64 C	15.62 ± 6.29 B	44.64 ± 4.88 AD
48h	59.11 ± 6.12 A	23.71 ± 2.90 B	58.30 ± 3.43 A	54.09 ± 8.63 A	37.87 ± 4.37 C	25.75 ± 4.23 B	48.86 ± 5.00 A
72h	71.96 ± 10.40 A	39.93 ± 3.87 BC	71.96 ± 7.80 A	64.88 ± 7.50 AD	51.39 ± 6.09 CD	33.05 ± 3.58 B	60.85 ± 5.48 AD
Abbreviations: BAI, baicalein; CHX, chlorhexidine; DMSO, dimethyl sulfoxide.							
^a Different uppercase letters indicate significant differences between treatment groups at the same time point ($p<0.05$). Control = distilled water.							

Table 2: Relative Fluorescence Intensity (Means and Standard Deviations) of Dentin-bond Collagenase^a

	Control	2% CHX	1% DMSO	0.1 µg/mL BAI	0.5 µg/mL BAI	2.5 µg/mL BAI	5.0 µg/mL BAI
1h	31.42 ± 9.01 A	1.60 ± 1.49 B	22.79 ± 7.52 AC	20.61 ± 3.59 CD	3.55 ± 2.21 B	2.95 ± 2.52 B	10.81 ± 2.10 BD
24h	49.64 ± 6.09 A	13.00 ± 5.48 B	37.75 ± 5.39 C	36.56 ± 4.34 C	23.01 ± 4.08 BD	18.59 ± 5.74 B	30.24 ± 4.90 CD
48h	59.95 ± 3.38 A	24.48 ± 5.87 B	48.24 ± 7.54 C	46.30 ± 4.05 CD	29.94 ± 1.25 BE	27.31 ± 4.84 BE	37.12 ± 5.40 DE
72h	69.17 ± 4.65 A	36.59 ± 4.40 B	61.41 ± 6.00 AC	54.85 ± 5.38 C	40.72 ± 3.55 B	35.45 ± 3.69 B	51.57 ± 7.41 C

Abbreviations: BAI, baicalein; CHX, chlorhexidine; DMSO, dimethyl sulfoxide.
^a Different uppercase letters indicate significant differences between treatment groups at the same time point ($p < 0.05$). Control = distilled water.

DISCUSSION

This study evaluated the influence of BAI on MMPs and a resin-dentin adhesive. Acting as the connecting media between the dentin and adhesive resin, the hybrid layer plays an important role in resin-dentin adhesives. The complete infiltration and curing of the adhesive agent in the demineralized dentin matrix are extremely important for quality hybrid layer formation. However, the prerequisite for use of BAI as a MMP inhibitor and cross-linking agent in the bonding procedure to improve adhesive strength is that it does not alter the inherent curing characteristic of the adhesive. In this study, the results from FTIR showed that BAI had no significant influence on the curing behavior of adhesive within a concentration range of 0-5.0 µg/mL, indicating that it could be used as a preconditioner in follow-up experiments.

The present results of EnzChek gelatinase/collagenase assay kits showed that BAI has a direct and remarkable inhibitory effect on the total activity of dentin-bond gelatinase/collagenase, especially at concentrations of 0.5 µg/mL and 2.5 µg/mL. Previous studies^{36,37} have determined that the C6 and C7 hydroxyl groups of BAI have powerful metal-chelate characteristics and can chelate transition metals such as Zn^{2+} . In consideration of MMPs as a family of Zn^{2+}/Ca^{2+} -dependent enzymes, the competitive reaction of metal ions might be the main inhibitory mechanism of BAI on MMPs. Moreover, cross-linked collagen has a higher resistance to collagenase digestion,³⁸⁻⁴¹ indicating that there might be a strong relationship between cross-linking and MMP

inhibition. Prompted by the bonding mode of BAI and human serum albumin,⁴² BAI can act as a potential cross-linker since its hydroxyl groups can form hydrogen bonds with the amide carbonyl of proteins. Furthermore, it has been reported that cross-linkers can cross-link not only proteins but also proteases. This may directly interfere with the molecular mobility of proteases or inactivate C-terminal telopeptidases, maintaining telopeptides' ability and sterically blocking collagenase binding to the critical peptide bond.^{43,44} Therefore, we speculated that the MMP inhibitory mechanism of BAI in endogenous dentin-bond MMPs might be as follows. First, BAI could compete with the active center of the enzyme through a metal-chelating effect, grabbing metal ions such as Zn^{2+} to suppress the activity of MMPs. Second, BAI might cross-link and alter the three-dimensional structure or molecular mobility of MMPs, resulting in the loss of the collagen enzymolysis ability of the latter. Third, BAI might cross-link with dentin collagen fibers through hydrogen bonds, changing or covering the recognition sites of MMPs in collagen to interfere with enzymatic coordination and complexation, thereby protecting noncoated collagen from degradation.

However, the inhibitory effect of BAI on gelatinase/collagenase was not a simple positive correlation, and a concentration limitation of BAI still existed. The increasing concentration of BAI might result in a competition between BAI molecules themselves for the bonding sites of protease, leading to the attenuation of the inhibitory effect. Another reason might be that the high concentration of

Table 3: Microtensile Bond Strengths (MPa, Means and Standard Deviations) of Different Treatment Groups at Different Aging Time Points^a

	Control	1% DMSO	5% GD	2.5 µg/mL BAI
Immediate	41.89 ± 5.18 Aa	49.32 ± 5.03 Ba	58.86 ± 4.29 Ca	58.32 ± 3.95 Ca
3 mo	34.46 ± 6.22 Ab	43.59 ± 5.8 2Bb	56.10 ± 5.8 9Ca	53.07 ± 3.97 Cb
6 mo	26.82 ± 5.30 Ac	40.73 ± 4.91 Bb	51.86 ± 6.42 Cb	52.43 ± 5.43 Cb

Abbreviations: BAI, baicalein; DMSO, dimethyl sulfoxide; GD, glutaraldehyde.

^a Different uppercase letters indicate significant differences between different treatment groups. Different lowercase letters indicate statistically significant differences between different aging time points ($p < 0.05$). Control = distilled water.

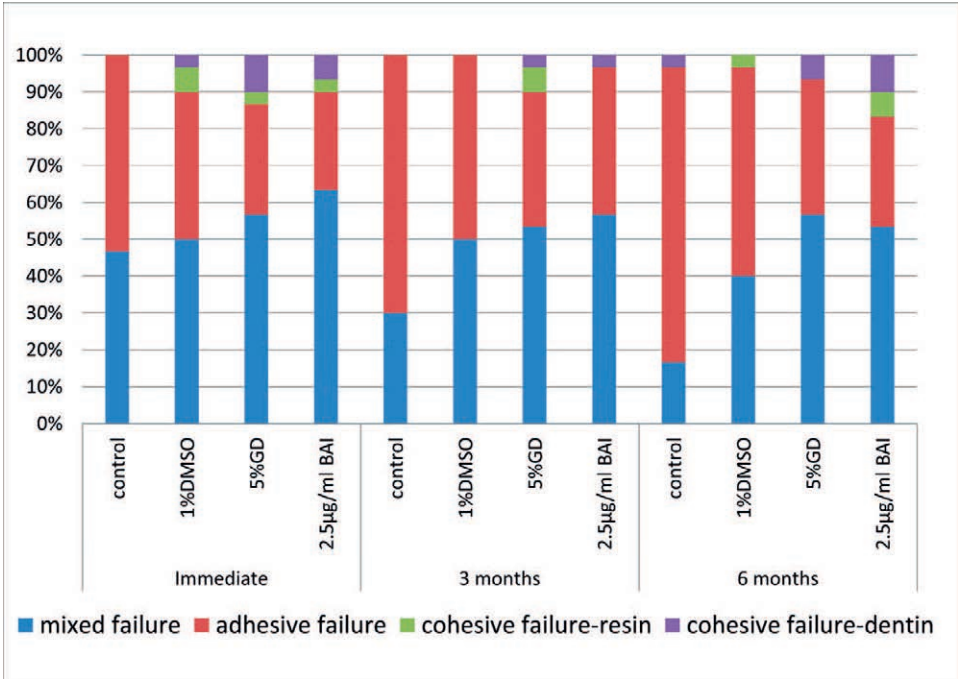


Figure 3. Failure modes for each group immediately or after aging. Adhesive failures increased remarkably in the blank groups after aging, but mixed failure was still the predominant failure mode in the 2.5 µg/mL baicalein group after six months of aging.

solutes increased the viscosity, which obstructed the infiltration of BAI into the dentin-collagen matrix and sequentially affected the combination with dentin-bond MMPs deep inside. In addition, the optimum concentration effects of BAI on gelatinase and collagenase were slightly different. This might be explained by the content or structure differences between these two types of proteases. However, the results of EnzChek gelatinase/collagenase assay kits only demonstrated that BAI could inhibit the total activities of dentin-bond gelatinase/collagenase; the inhibitory effect on definite MMP subtypes is still unclear; further research is, therefore, required.

Synthesizing the DC results and gelatinase/collagenase activities, we chose to apply 2.5 µg/mL BAI in an etch-and-rinse adhesive system to investigate its influence on bonding strength. According to the results of microtensile bond strength and nano-leakage testing, the 2.5 µg/mL BAI group had a significantly higher microtensile bond strength, less

adhesive failure, and lower nanoleakage compared with the control and 1% DMSO groups, both immediately and after artificial saliva storage. This demonstrated the enhancement of resin-dentin bonding strength by BAI. Therefore, the null hypothesis of this study was rejected. We speculated that the BAI mechanism responsible for the enhancement of resin-dentin adhesive might be the coefficient of its MMP inhibitory effect and protein cross-linking property. Haslam⁴⁵ demonstrated that the amphiphilic structure of natural plants' polyphenol could help them cross-link with proteins. The principal driving forces toward complexation were "hydrophobic effects" from aromatic nuclei groups, and then enhancement by "hydrogen bonding" of phenolic hydroxyl groups. BAI has fewer phenolic hydroxyl groups and stronger hydrophobic performance compared with proanthocyanidins, implying that BAI might react with dentin collagen via hydrophobic bonding, mainly, similar to the bonding

Table 4: Percentage of Nanoleakage (%; Means and Standard Deviations) of Different Treatment Groups at Different Aging Time Points ^a				
	Control	1% DMSO	5% GD	2.5 µg/mL BAI
Immediate	10.69 ± 3.12 Aa	7.26 ± 2.48 Ba	8.16 ± 3.11 ABa	3.38 ± 1.26 Ca
3 mo	19.72 ± 3.59 Ab	11.49 ± 3.52 Bb	8.10 ± 2.9 7BCa	7.33 ± 3.32 Cb
6 mo	34.96 ± 6.87 Ac	19.41 ± 6.10 Bb	9.62 ± 3.59 Ca	10.72 ± 4.71 Cb

Abbreviations: BAI, baicalein; DMSO, dimethyl sulfoxide; GD, glutaraldehyde.
^a Different uppercase letters indicate significant differences between different treatment groups. Different lowercase letters indicate statistically significant differences between different aging time points (p<0.05). Control = distilled water.

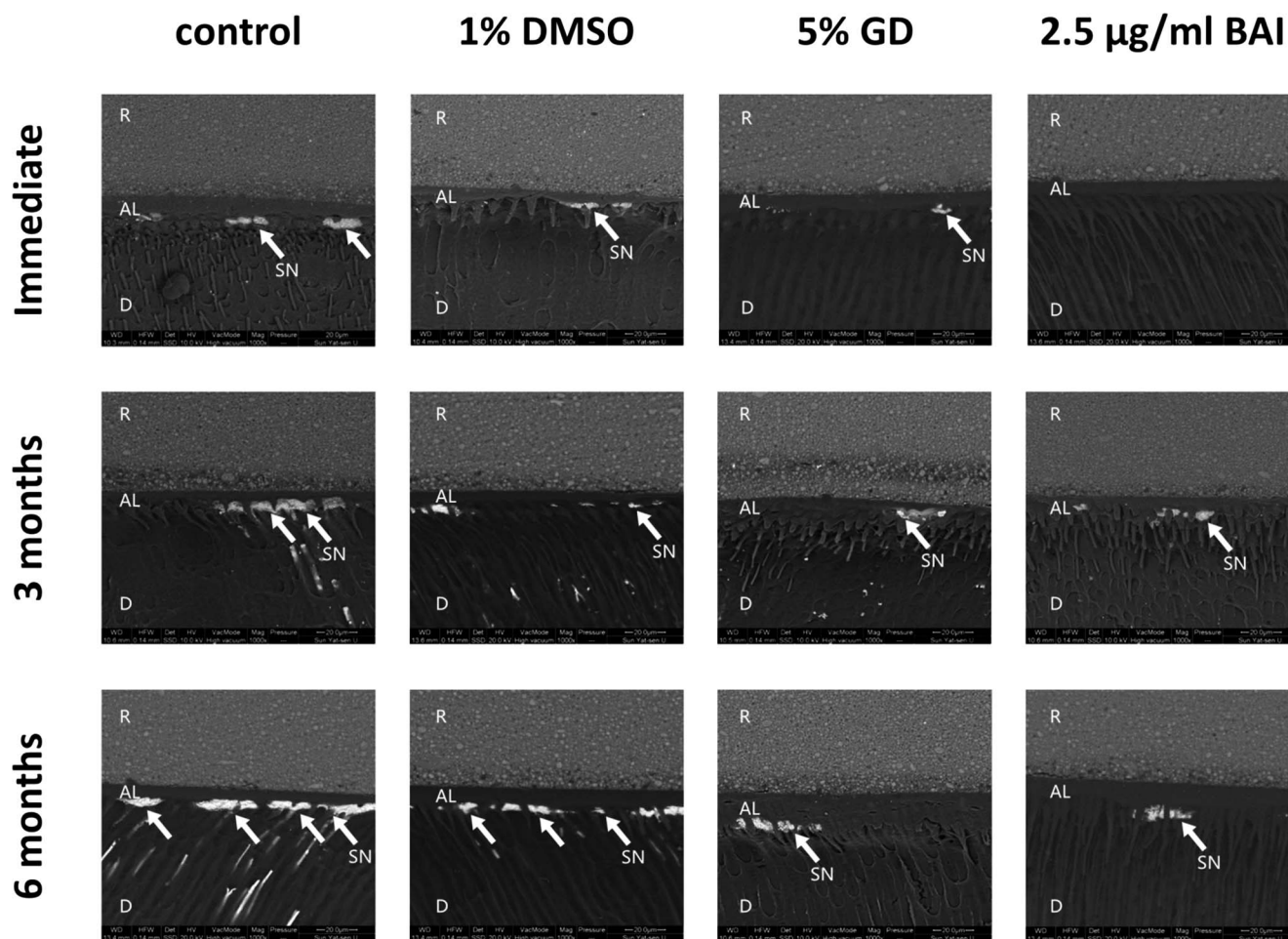


Figure 4. Nanoleakage images of different groups immediately or after aging under SEM (1000 $\times$) showed that the baicalein group had a significantly lower nanoleakage percentage than the control group at each time point. ($p < 0.05$). R, resin; AL, adhesive layer; D, dentin; SN, silver nitrate.

mode of BAI and human serum albumin.⁴² Consequently, BAI might improve the mechanical properties of dentin collagen by forming hydrogen bonds with the latter to increase the cross-linking and prevent the collagen fibrils network from extending and slipping. Furthermore, according to the cross-linking mechanism of proanthocyanidins,^{46,47} the stronger hydrophobic performance of BAI might decrease water absorption and the swelling of collagen, thereby minimizing sensitivity to hydrolysis and protecting collagen from degradation.

In addition, DMSO had a certain effect on bond strength improvement in the present study. The findings of other studies⁴⁸⁻⁵⁰ also reached an agreement about the enhancement of DMSO on dentin adhesives. It is worth mentioning that the μ TBS and nanoleakage of the 1% DMSO group at all time points were significantly superior to those of the control group, but they were inferior to those of the 2.5 μ g/mL BAI group, indicating that 1% DMSO has

a synergistic effect on bonding improvement with BAI. The effect of DMSO might be explained as follows. First, the amphiphilic character of DMSO could help dissolve hydrophobic monomers,⁵¹ including BAI, for improved diffusion and collagen encapsulation. Secondly, DMSO has the ability to break down water's self-associative tendency,⁵² establishing a stable hydrophobic microenvironment to enhance cross-linkage between BAI and collagen. In addition to its other abilities, such as collagen moisturization and hydrogen-bond modification,⁴⁸ and its influence on the surface wetting behavior of resins,⁵³ DMSO was capable of synergistically improving bonding quality with BAI in the resin-dentin adhesive procedure.

A limitation of this study is that it only investigated the effect of BAI on the Adper™ Single Bond 2 etch-and-rinse adhesive system with a certain preconditioning time (120 seconds). The potential difference in findings regarding other adhesive

systems and treatment periods is still unclear. Further experiments should focus on the influence and mechanism of BAI on definite subtypes of MMPs while using BAI in other adhesive systems for clinical exploration.

CONCLUSIONS

BAI used as a preconditioner in an etch-and-rinse adhesive system could effectively improve resin-dentin bonding durability and decrease interface nanoleakage under the premise that it has no significant influence on the curing behavior of the adhesive agent. The effect of BAI in dentin adhesion might be related to its anti-MMP function and cross-linking property, which enhance the collagen matrix and stabilize the hybrid layer synergistically with DMSO during the bonding process.

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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 Ethics Review Committee, Guanghua School of Stomatology, Hospital of Stomatology, Institute of Stomatological Research, Sun Yat-sen University. The approval code for this study is ERC-[2014]-23.

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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Effects of Polishing Bur Application Force and Reuse on Sintered Zirconia Surface Topography

NG Fischer • A Tsujimoto • AG Baruth

Clinical Relevance

Polishing application force and the reuse of polishing burs on CAD/CAM zirconia should be considered in light of possible effects on surface topography.

SUMMARY

Objective: Limited information is available on how to polish and finish zirconia surfaces following computer-aided design/computer-aided manufacturing (CAD/CAM), specifically, how differing application forces and reuse of zirconia polishing systems affect zirconia topography.

Purpose: To determine the effect of differing, clinically relevant, polishing application forces and multiple usages of polishing burs on the surface topography of CAD/CAM zirconia.

Methods: One hundred twenty 220-grit carbide finished zirconia disks were sintered according to manufacturer's directions and divided into two groups for the study of two coarse polishing bur types. Each group was divided into subgroups for polishing (15,000 rpm) at 15

seconds for 1.0 N, 4.5 N, or 11 N of force using a purpose-built fixture. Subgroups were further divided to study the effects of polishing for the first, fifth, 15th, and 30th bur use, simulating clinical procedures. Unpolished surfaces served as a control group. Surfaces were imaged with noncontact optical profilometry (OP) and atomic force microscopy (AFM) to measure average roughness values (Ra). Polishing burs were optically examined for wear. Scanning electron microscopy (SEM) was performed on burs and zirconia surfaces. One-way ANOVA with post hoc Tukey HSD (honest significant difference) tests ($\alpha=0.05$) were used for statistical analyses.

Results: AFM and OP Ra values of all polished surfaces were significantly lower than those of the unpolished control. Different polishing forces and bur reuse showed no significant differences in AFM Ra. However, significant differences in OP Ra were found due to differing application forces and bur reuse between the first and subsequent uses. SEM and optical micrographs revealed notable bur wear, increasing with increasing reuse. SEM and AFM micrographs clearly showed polished, periodic zirconia surfaces. Nanoscale topography, as analyzed with kurtosis and average groove

Nicholas G Fischer, BS, Creighton University, Department of Physics, Omaha, NE, USA

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

*Andrew G Baruth, PhD, Creighton University, Department of Physics, Omaha, NE, USA

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

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depth, was found dependent on the specific polishing bur type.

Conclusions: These *in vitro* results suggest changes in OP Ra due to bur reuse and polishing application force. Within the parameters of this study, the resultant topography of zirconia polishing is force-dependent and the reuse of coarse polishing burs is possible without statistically significant differences in Ra values after initial use. Nanoscale and microscale topography were shown to depend on specific polishing bur type.

INTRODUCTION

Zirconia's use in computer-aided design/computer-aided machining (CAD/CAM) systems has facilitated the increased use of zirconia in dentistry,¹ as CAD/CAM represents one of the fastest growing technologies in dentistry.² Favorable mechanical properties of zirconia include color stability, plaque resistance, hardness, wear resistance, translucency, chemical inertness, and low thermal conductivity.^{3,4} Due to variability in CAM, practitioner preferences, and specificities of each clinical case, zirconia processed via CAD/CAM requires clinician finishing and polishing.⁵ While clinicians undertake polishing for a variety of reasons, many relate to the resultant topography of the surface.

The topography of zirconia is important to consider, as surface roughness (Ra) can increase enamel abrasion and bacterial retention.⁶⁻⁹ Furthermore, Ra has been correlated with mechanical properties such as flexure strength¹⁰ and low-temperature degradation.¹¹ Esthetically, Ra is correlated with gloss,¹² a notable advantage of zirconia over more traditional materials such as titanium. Polishing zirconia has been shown to affect its topography, particularly Ra.^{8,13-16} As polishing is a manual skill, the force applied varies from individual to individual.¹⁷ In polishing hybrid composites, Ra values have been shown to be modulated from force differences as small as 2 N.¹² Previous research^{18,19} has shown greater than 2 N differences in force applied by clinicians in a variety of situations, including caries probing and crown cementation.

Zirconia polishing forces previously investigated have ranged from 1 N⁸ to 2 N^{20,21} to 10 N.²² However, to the authors' knowledge, no work has been done on the force dependence of polishing zirconia materials. Some lines of work^{5,16} have standardized polishing force with trained operators, a potential source of variability, as many have

pointed out the need for standardization in polishing experiments.^{12,21,22} Notably, Jung and others²³ have shown differences in Ra produced by a single user polishing resin composites to be as large as 100%. Thus, an important consideration for examining resultant surface characteristics is the standardization of application forces.

Similar to cutting burs, reuse of polishers and finishers is subject to user preference and depends on the manufacturer, if recommendations even exist.²⁴ Some work²⁵ has been done on bur deterioration used in CAM systems, but a paucity of information exists for reuse of diamond burs for polishing zirconia. Other recent work²⁶ has focused on the reuse of milling burs, but not those used in polishing.

This research combines the techniques of atomic force microscopy (AFM), with growing interest in dentistry for its nanoscale resolution, and optical profilometry (OP), a time-tested technique in dental research on the microscale. The null hypotheses were that different polishing forces will not result in differing Ra roughness values and that each reuse of polishers will not affect surface topography. The imaging is supplemented with scanning electron microscopy (SEM) and optical photography to access further size scales. This allows for greater insight into the behavior of surface topography at two lateral resolutions and the transition between the two.

Both scales are important to consider, as polishing operates on both, and both scales contribute to the clinical outcomes of polished surfaces. Combined, AFM and OP allow for waviness and roughness of zirconia surfaces to be separated and so commented on. This technique follows the recommendations of Wennerberg and others.²⁷ This, alongside SEM, is the proper use of the three most common dental materials imaging techniques. This research is a first step toward evaluating and standardizing zirconia polishing protocols.

METHODS AND MATERIALS

Specimen Fabrication

One hundred twenty Very High Translucency (ZirkonZahn, Gais, Italy) specimens ($\phi=5$ mm, 2 mm thick; Table 1), were prepared from green zirconia, which follows a biaxial press of zirconia particles into a mold, where plastic deformation of the particles leads to confinement and solidification into a monolithic block. As received ($\phi=95$ mm, 15 mm thick), zirconia monolithic disks were sectioned into

Table 1. Materials Used in This Study			
Brand Name (Lot)	Type	Manufacturer	Recommendations
Very High Translucency (95-18)	Zirconia, for use in CAD/CAM systems	Zirkonzahn, Gais, Italy	Final hold temperature of 1480°C for 2 h
Zilmaster Coarse Polish (0658)	Bullet-shaped, diamond-embedded polisher	Shofu, Kyoto, Japan	Dry polish up to 20,000 rpm
Coarse Polish (Z&LC6C)	Flat-ended, cylinder-shaped, diamond-embedded polisher	Supér, Germany	Dry polish up to 20,000 rpm

2-mm sheets with a low-speed diamond wet saw (Model C, Pistorius Machine Co, Hicksville, NY, USA), flat ground with silicon carbide sandpaper, (220-grit; 3M, St Paul, MN, USA) and machined into disks using a vertical milling unit (Trak K2 SX, Southwestern Industries, Rancho Dominguez, CA, USA). Samples were desiccated at 78°C overnight in a drying oven (Precision 658 Compact Oven, Thermo Fisher, Waltham, MA, USA) and then sintered in a box furnace (Lindberg/Blue M 1700°C Tube Furnace, Thermo Fisher) with the associated control unit (Lindberg/Blue MCC59246PCOMC-1, Thermo Fisher) following manufacturer’s directions, including a final hold temperature of 1480°C for 2 hours.

Specimen Polishing and Finishing

Specimens were randomly divided into two groups: group A: Coarse Polish Zilmaster (Shofu, Kyoto, Japan) and group B: Coarse Polish Z&LC6C (Supér, Germany; Table 1). As laid out in Figure 1, each

group, A and B, was divided into subgroups for dry polishing with 1.0 N, 4.5 N, or 11 N of force. Finally, subgroups were divided further, being polished at the first, fifth, 15th, or 30th bur use, simulating clinical procedures. Between each evaluated use, burs were used at the associated force on an additional unpolished control specimen. This culminated in 12 unique specimen types for each group, A and B, with 24 specimen types (plus unpolished control) in total, and 120 total specimens investigated (n=5 per specimen type).

Polishing was accomplished with a custom polishing fixture, (Figure 2). A rotary tool (Dremel 4000, Dremel Corp, Racine, WI, USA) was mounted in a prefabricated mounting unit (Dremel 220) to polish at 15,000 rpm, as recommended by the manufacturer and previous work²⁰ for 15 seconds. A stroboscope (Strobotac Type 1531-A, General Radio Co, Boston, MA, USA) verified rotation frequency. Specimens were placed in a spring-loaded chuck (McMaster-

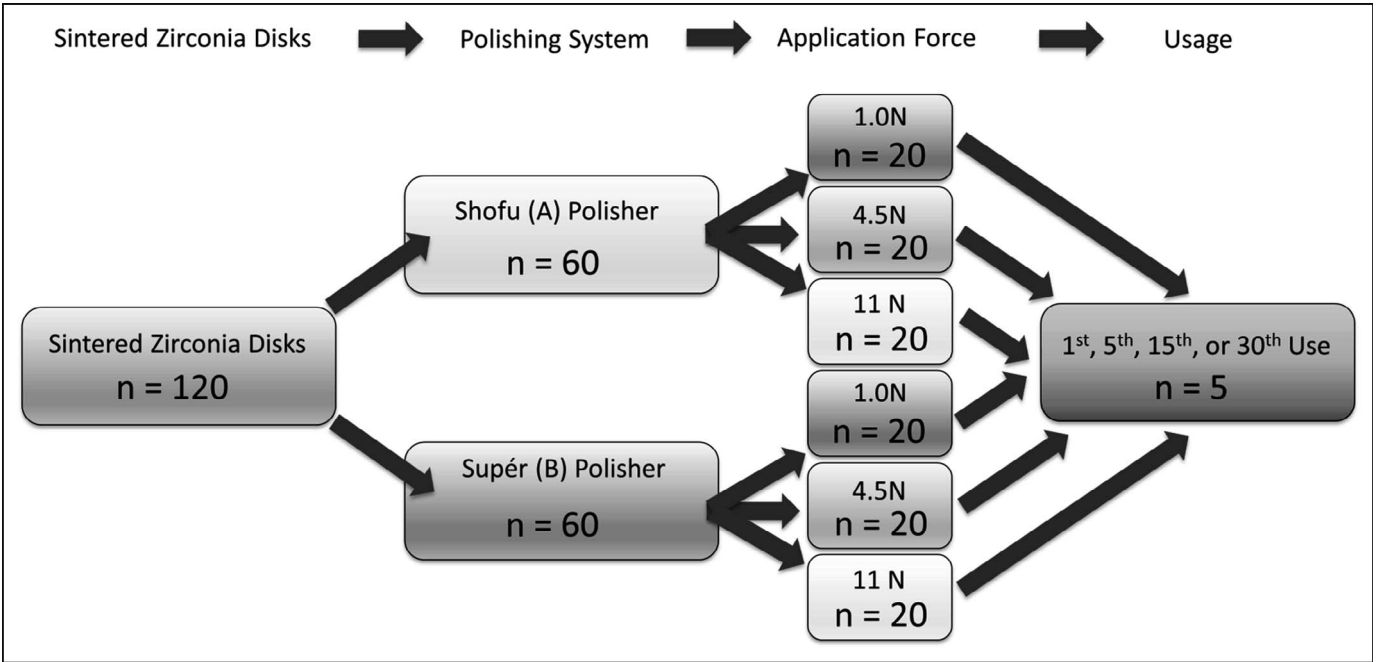


Figure 1. Schematic flow chart of specimen preparation for each study group, including sample size n.

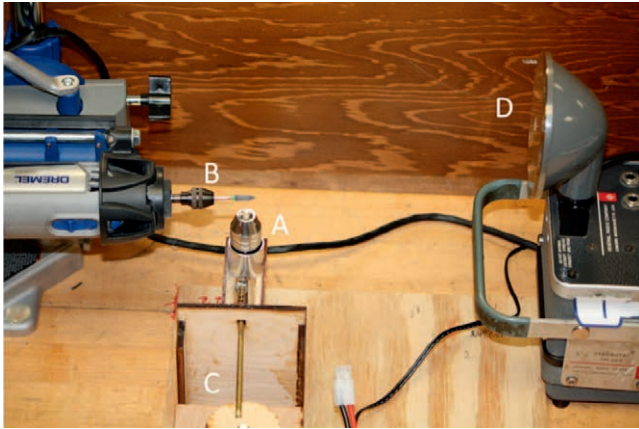


Figure 2. Image of polishing fixture. Interchangeable spring-loaded chuck (A), variable speed rotary tool (B), and lateral motion actuator (C), not used in the present study, allows for quick, independent control of polishing parameters. A stroboscope (D) allows for rotation frequency monitoring.

Carr, Elmhurst, IL, USA), calibrated to apply either 1.0 N, 4.5 N, or 11 N force via interchangeable, calibrated springs (McMaster-Carr) perpendicular to the polishing burs. To isolate the role of force and reuse, the polishing apparatus applied no lateral movement. Following polishing, specimens were sonicated in acetone for 5 minutes to clear any polishing debris before imaging.

Atomic Force Microscopy

AFM measurements were performed in a mechanical and acoustic isolation chamber on an Agilent 5420 SPM/AFM (Agilent Technologies, Santa Clara, CA, USA) in ambient ($22 \pm 2^\circ\text{C}$, $40\% \pm 20\%$ relative humidity) conditions. Three independent areas ($30 \times 30 \mu\text{m}$) were measured per specimen. Each of the three areas was measured in the center of the polished region, as determined with optical microscopy, for consistency. Images were obtained in a constant force mode with silicon nitride cantilevers (spring constant of 0.2 N/m and tip radius of $\leq 10 \text{ nm}$) (BudgetSensors, Sofia, Bulgaria) at a 4.0 Hz scan rate of 512 lines per image. The AFM micrographs ($30 \times 30 \mu\text{m}$) were analyzed with Gwyddion software (Central European Institute of Technology, Brno, Czech Republic) to extract surface parameters following row alignment by median matching and plane-fit subtraction. Zirconia surface roughness was quantified in terms of Ra, the average of the absolute values of the height deviations from the mean, recorded within a consistent sampling area. Ra shows the average of a set of individual measurements of a surface's peaks and valleys, which depends on the size of the area evaluated. In addition, lateral pitch, average groove

depth, and kurtosis were measured, wherein kurtosis is the fourth moment of the absolute values of the height deviations from the mean, recorded within a consistent sampling area. For kurtosis, a higher value indicates a variance that is the result of infrequent, extreme deviations (as opposed to frequent, modest deviations).

Optical Profilometry

OP measurements were performed on a Proscan 2100 noncontact optical profilometer (Scantron Industrial Products Ltd, Taunton, UK). Proscan software was used for analyses. Each specimen was imaged four times at an operational scan rate of 300 Hz, a cut-off length of 0.25 mm, and a sampling length of $0.30 \times 3.0 \text{ mm}$, following recommended ISO 4288 standards.²⁸ For the present study, shorter cut-offs revealed no differences in resultant Ra.

Scanning Electron Microscopy

SEM (TM3000 Tabletop Microscope, Hitachi-High Technologies, Tokyo, Japan) was performed at an acceleration voltage of 15.0 kV at a magnification of $10,000\times$ for representative zirconia surfaces and $50\times$ to $2500\times$ for polishing burs. Resultant micrographs were analyzed for grain size with ImageJ 1.50i (National Institutes of Health, Bethesda, MD, USA) using the intercept method.

Optical Imaging

Polishing burs were photographed with a Canon Rebel XS DSLR digital camera (Canon USA Inc, Lake Success, NY, USA) with a $5\times$ optical zoom in a custom-built, 6-light source studio to avoid shadowing.

Data Analyses

Mean Ra values were compared with a one-way ANOVA followed by a Tukey HSD (honest significant difference) post hoc test. GraphPad Prism 7.0a (GraphPad Software, San Diego, CA, USA) was used for calculations. A p value of <0.05 was considered statistically significant. Standard deviation is reported, where appropriate.

RESULTS

Zirconia Nanoscale Topography, AFM, and SEM

Upon qualitative visual inspection of AFM micrographs, a polished, periodic surface was achieved in every polishing group, shown for group A in Figure 3 and group B in Figure 4. Control samples revealed a densely populated network of individual zirconia

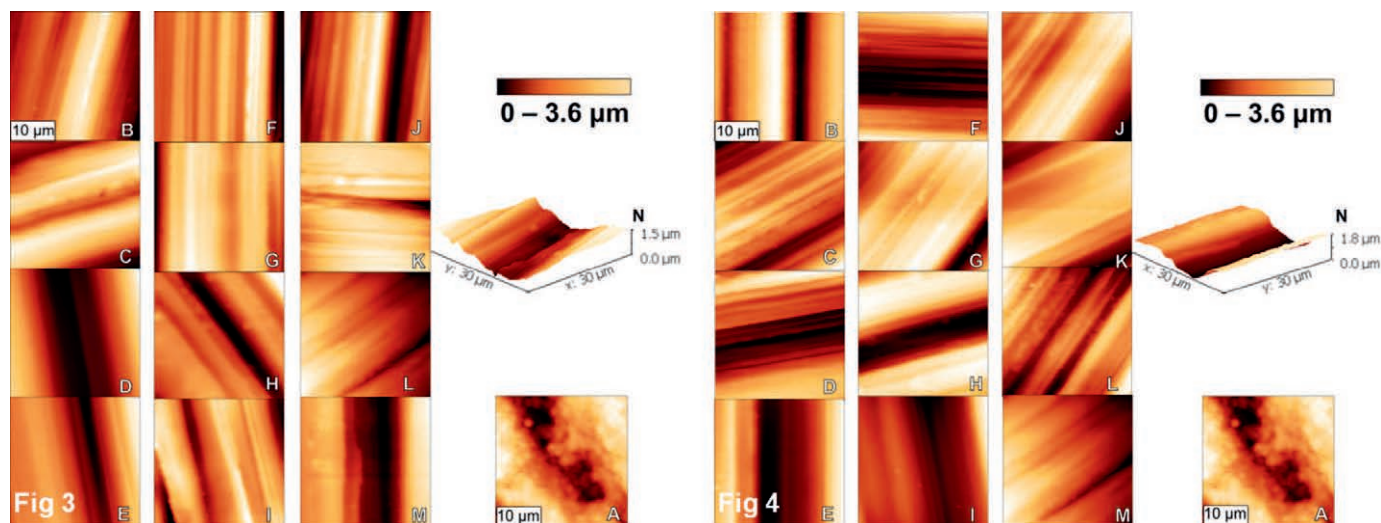


Figure 3. Representative plan view of topographic atomic force micrographs ($30 \times 30 \mu\text{m}$) of group A polished zirconia surfaces for various application forces and polishing bur use. Color scale represents height, as indicated by the color bar. (a) Control, 0.0 N—0th use. (b) 1.0 N—first use. (c) 1.0 N—fifth use. (d) 1.0 N—15th use. (e) 1.0 N—30th use. (f) 4.5 N—first use. (g) 4.5 N—fifth use. (h) 4.5 N—15th use. (i) 4.5 N—30th use. (j) 11 N—first use. (k) 11 N—fifth use. (l) 11 N—15th use. (m) 11 N—30th use. (n) 3-dimensional rendering of 11 N—30th use to highlight nanoscale topography. Figure 4. Representative plan view topographic atomic force micrographs ($30 \times 30 \mu\text{m}$) of group B polished zirconia surfaces for various application forces and polishing bur use. Color scale represents height, as indicated by color bar. (a) Control, 0.0 N—0th use. (b) 1.0 N—first use. (c) 1.0 N—fifth use. (d) 1.0 N—15th use. (e) 1.0 N—30th use. (f) 4.5 N—first use. (g) 4.5 N—fifth use. (h) 4.5 N—15th use. (i) 4.5 N—30th use. (j) 11 N—first use. (k) 11 N—fifth use. (l) 11 N—15th use. (m) 11 N—30th use. (n) 3-dimensional rendering of 11 N—30th use to highlight nanoscale topography.

crystallites with a grain size of $0.359 \pm 0.115 \mu\text{m}$. The results of the influence of force and reuse of group A polishing burs on zirconia's AFM Ra values are shown in Figure 5, and the results of group B polishing burs on AFM Ra are shown in Figure 6. Ra values across all polished zirconia specimens ranged from $0.169 \pm 0.043 \mu\text{m}$ to $0.274 \pm 0.111 \mu\text{m}$ with a control surface Ra of $0.448 \pm 0.093 \mu\text{m}$.

ANOVA analysis revealed a statistically lower Ra ($p < 0.05$, $F = 5.435$) for both group A and B over the control. However, no statistical differences ($p > 0.05$) were found between experimental subgroups within groups A or B. The changes in Ra strongly depended on the overall topography observed in AFM between groups A and B, so kurtosis and average groove depth were subsequently evaluated across all pol-

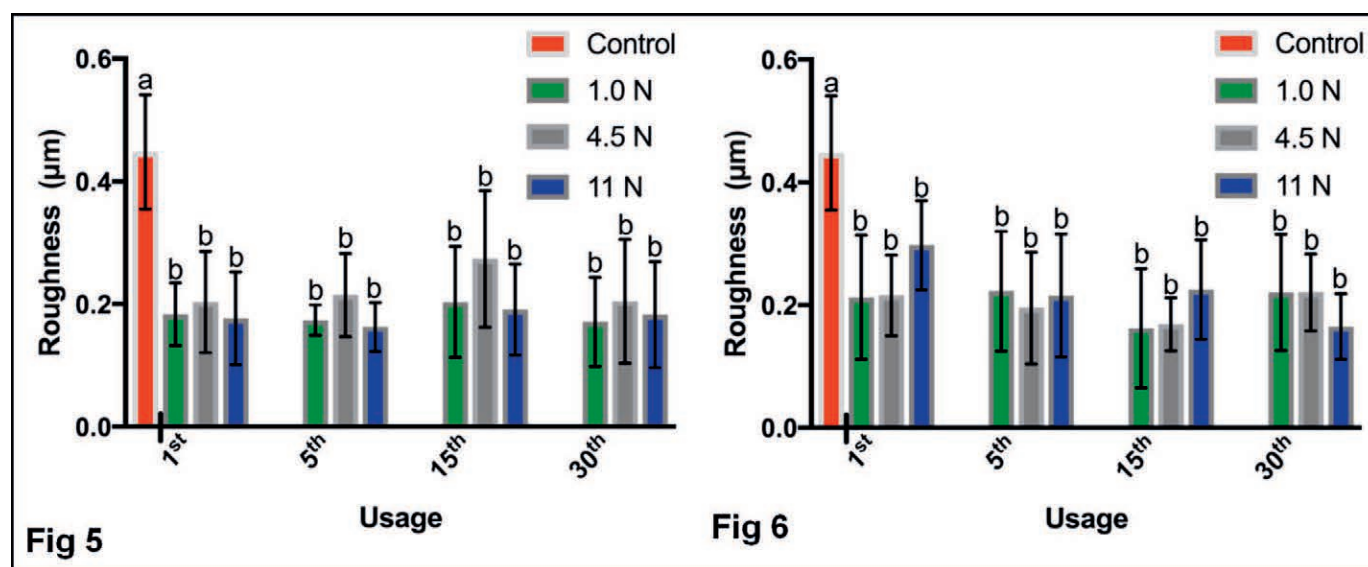


Figure 5. Atomic force microscopy Ra roughness for group A. Same letter indicates no significant difference ($p > 0.05$).

Figure 6. Atomic force microscopy Ra roughness for group B. Same letter indicates no significant difference ($p > 0.05$).

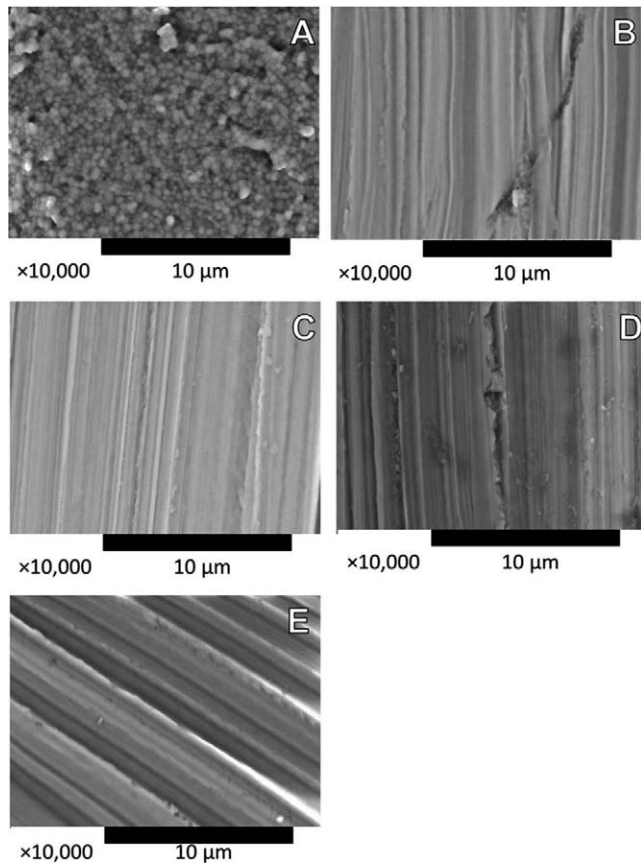


Figure 7. SEM micrographs of sintered zirconia surfaces at 10,000 $\times$. (a) Control: 0.0 N—0th use. (b) group A: 1.0 N—first use. (c) group A: 11 N—30th use. (d) group B: 1.0 N—first use. (e) group B: 11 N—30th use.

ished samples within groups A or B, with summative calculations. For group A, kurtosis was -0.342 ± 0.384 and average groove depth was $0.625 \pm 0.507 \mu\text{m}$. For group B, kurtosis was -0.633 ± 0.257 and average groove depth was $1.06 \pm 0.693 \mu\text{m}$. A negative value for kurtosis indicates that the height distribution has lighter tails and a flatter peak than the normal distribution. That, in combination with average groove depths, revealed an average surface topography for group A that had more frequent, smaller ($0.588 \pm 0.152 \mu\text{m}$ lateral pitch, as measured with SEM) grooves that overlaid the larger groove ($1.1512 \pm 0.502 \mu\text{m}$ lateral pitch, per SEM) features present from polishing. Conversely, group B had similar larger groove features ($1.719 \pm 0.502 \mu\text{m}$ lateral pitch per SEM), but these features were smoother and lacked the smaller grooves.

To further envision the microstructure compared with the nanostructure, Figure 7 shows selected SEM micrographs of the zirconia surfaces. As with AFM analysis, the control consisted of individually

visible zirconia crystals. After polishing in both groups A and B, as with AFM analysis, a periodic, polished surface was achieved.

Zirconia Microscale Topography, OP

To quantify the microscopic surface topography of zirconia, OP was employed. The results of the influence of force and reuse of group A polishing burs on OP Ra are shown in Figure 8, and the results of group B polishing burs on OP Ra are shown Figure 9. The ANOVA analysis revealed a statistically significant difference ($p < 0.05$, $F = 16.213$) between group A, including all subgroups, and the control. The ANOVA analysis also revealed a statistically significant difference ($p < 0.05$, $F = 36.77$) between group B, including all subgroups, and the control. In both groups A and B, the Ra of the control was statistically greater ($p < 0.05$) than that of all other subgroups within that particular group.

Considering bur use and applied force in group A (Figure 8), there were no statistically significant differences ($p > 0.05$) between the first, fifth, 15th, or 30th use for any force applied. Considering bur use and applied force in group B (Figure 9) on first use, Ra was shown to significantly decrease ($p < 0.05$) with increasing application force. Furthermore, for each use value in group B, Ra values from the first bur use were significantly greater ($p < 0.05$) than those of their counterparts at the fifth, 15th, and 30th use. However, there were no statistical differences ($p > 0.05$) between subgroups at the fifth, 15th, or 30th usage for any force applied.

Bur Optical Photography and SEM

Diamond-containing polishing burs for groups A and B are shown in Figure 10 for the as-received condition and following the 30th polishing at 11 N (ie, the two extremes of wear). Optically, wear is seen after the 30th polishing. Group A burs were composed of a compacted solid, with continued usage removing the crystalline material at an optically visible rate beginning with the first use. In contrast, group B burs, composed of diamond crystallites embedded in a polymeric substrate, did not show any visible signs of losing crystalline material with subsequent uses. Reassuringly, SEM analysis revealed that, indeed, zirconia debris was substantially present after the 30th polishing for both groups A and B. After polishing, group A polishing burs were found to have a crystallite size of $144 \pm 35.2 \mu\text{m}$ and group B burs a size of $75.5 \pm 14.4 \mu\text{m}$. This compares favorably with the control crystallite size of $160 \pm$

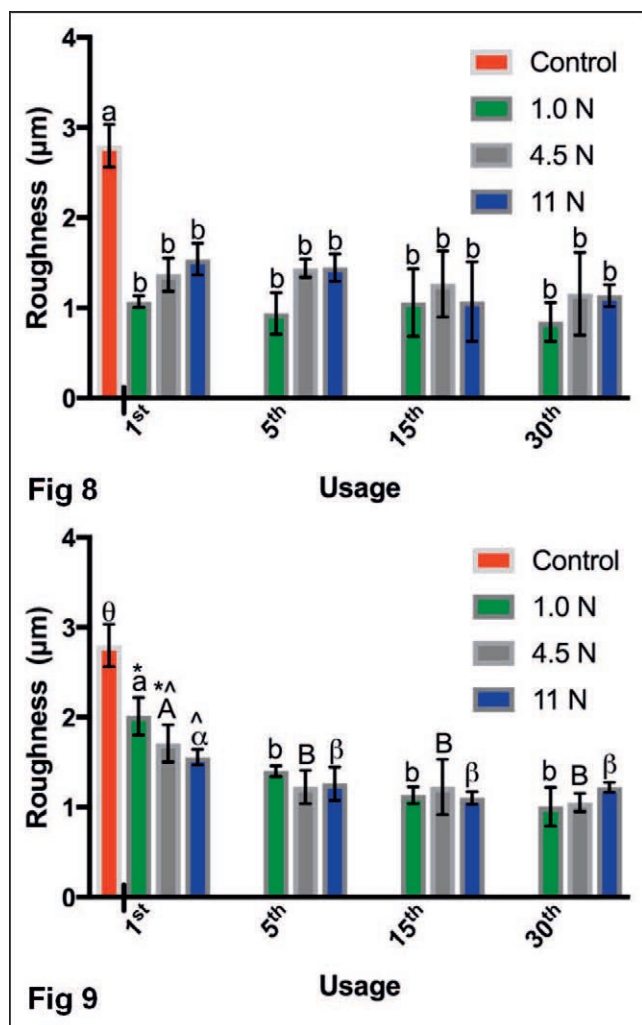


Figure 8. Optical profilometry Ra roughness for group A. Same letter indicates no significant difference ($p > 0.05$).

Figure 9. Optical profilometry Ra roughness for group B. Same lower case letter, upper case letter, Greek letter, or symbol ($^{\wedge}$ or *) indicates no significant difference ($p > 0.05$). Additionally, all subgroups with B, b, or β are not statistically significantly different from each other ($p > 0.05$). Theta (θ) is statistically significantly greater than all other groups ($p < 0.05$).

34.1 μm for group A and $72.9 \pm 14.5 \mu\text{m}$ for group B. This strongly suggests limited reduction in crystallite size due to polishing wear, indicating minimal crystallite fracture at clinically relevant application forces.

DISCUSSION

This study evaluated the influence of reusing polishing burs, as well as the role of differing polishing application forces, on the quantitative topography of sintered CAD/CAM zirconia disks. Polishing, ranging in force from 1.0 N to 11 N, coupled with the reuse of the polishing burs up to 30

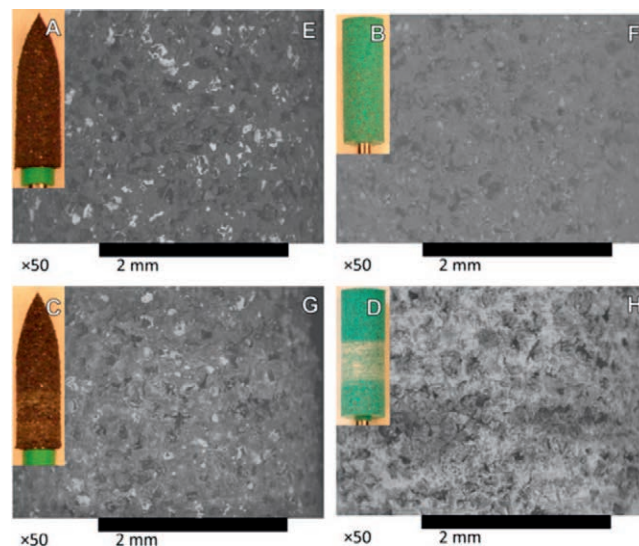


Figure 10. Optical images (a-d) and SEM micrographs (e-h) ($\times 50$) of polishing burs. (a) Unused group A bur. (b) Unused group B bur. (c) Group A: 11 N—30th Use. (d) Group B: 11 N—30th use. (e) Unused group A bur. (f) Unused group B bur. (g) Group A: 11 N—30th use. (h) Group B: 11 N—30th use.

times, produced a polished surface with an accompanying significant decrease in OP- and AFM-measured Ra values compared with the sintered zirconia control. Focusing on group B, the first use of the polishing bur consistently retained a higher OP Ra value than subsequent uses of that same bur. Furthermore, during this first use, the force was shown to significantly modulate the resultant OP Ra, in which Ra values decreased with increasing force. Therefore, under clinically plausible conditions, reuse of burs and the application force can modify the resultant topography as quantified by OP Ra. As such, the null hypotheses can be partially rejected.

This study considered both nanoscale and microscale topography with AFM/SEM and OP, respectively. While Ra continues to be an effective evaluation of surface topography, it is limited in describing complex topographies. This includes polished, sintered zirconia with the microscale polishing bur crystallites. For example, although having similar Ra values, evaluation of height variations of the polished zirconia surface reveals different nanostructure topographies between groups A and B. Most notably, the kurtosis and groove depth measurements were disparate between groups A and B, while retaining similar Ra values. In group A, this Ra roughness was a result of more frequent, smaller grooves, while group B revealed an Ra roughness that was a result of wider, deeper grooves.

These size scales may have clinical relevance when considering cellular attachment. Recent use of CAD/CAM zirconia in the manufacture of healing abutments requires consideration of surface properties, such as roughness and topography, for cellular adherence. Work from Mustafa and others²⁹ has shown that polished surfaces promote initial cellular attachment of human gingival fibroblasts (HGFs), important in the formation of peri-implantitis-preventing mucosal seals. Other work³⁰ has shown that polished zirconia increases HGF proliferation and cellular spreading, key factors in the formation of a sufficient mucosal seal. Additionally, polished surfaces have been shown to retain plaque at a lower rate, an important consideration in preventing pocket formation around an implant.^{6,7} On the other hand, insufficient roughness can increase probing depth and bleeding⁷ and do little to decrease plaque retention.³¹

Fine adjustment and polishing after CAD/CAM is inevitable. Scurria and others³² have shown that restorative glazing after machining should be preceded by polishing. Rough surfaces associated with a lack of polishing can increase the wear rate of occlusal enamel, an outcome that is against the very concept of conservative dentistry.^{9,33,34} Esthetically, polishing is employed to increase gloss to levels comparable with natural enamel.^{12,20} Polishing is even important in the prevention of patient discomfort from discernably rough surfaces.³⁵

Clinically, polishing, such as that investigated herein, would be preceded by CAM diamond milling, which can vary between units. In addition, the coarse polishing investigated in this study can be followed by additional polishing steps, commonly including a medium and fine grit. Different milling burs can produce topographically disparate zirconia surfaces.³⁶ Work by Corazza and others²⁵ has shown that even the reuse of diamond milling burs can significantly affect zirconia topography. Reuse is important in the light of patient savings and environmental consciousness. This milled zirconia is the substrate subjected to polishing. Importantly, this milling process may affect subsequent polishing and thus the surface topography of zirconia. In short, this is a potential variable to consider when polishing and a potential area of future work.

Relatively little work has been completed on prosthetic surfaces with AFM compared with OP.¹² For this reason, it is important to note that while AFM and OP both produce Ra values, these values should not necessarily be directly compared with each other, as shown by this work and others.³⁷

Bollen and others⁷ noteworthy and well-cited roughness threshold of 0.20 μm for both bacterial and tissue adhesion is frequently discussed^{8,9,11,15,16,21} with little consideration of the lateral resolution used to obtain the specific Ra value. Lateral resolution is critical when comparing Ra values, as it directly affects such values.²⁷

We have taken great care in considering these various Ra values at disparate lateral size scales and have included other metrics for topography quantification, including kurtosis and average groove depth. We further emphasize that AFM typically measures surface topography with a lateral resolution of 5-10 nm while OP is closer to 1-5 μm . Therefore, rather than being compared with each other, these Ra values are considered complimentary. Indeed, Ra values from OP are more strongly indicative of surface waviness (for example, microscopic groove patterns) whereas AFM reveals underlying nanoscale roughness (for example, nanoscale roughness superimposed on the microscopic groove pattern). While these different lateral resolutions are the reason that Ra values should not be directly compared across techniques, disparate lateral resolutions can show novel insight when combined.

Care should be taken in interpreting the study results. While the polishing parameters of application force and reuse have been shown to significantly affect topography, other factors such as rotational speed, zirconia sintering time and crystallite size, water lubrication, and operator variability may also affect outcomes. Chavali and others²⁰ have shown that a maximum temperature of 40.9°C is produced during dry polishing of zirconia for a variety of conditions. Reassuringly, work by Denry and others³⁸ has shown that a reverse transformation from the monoclinic to the tetragonal phase requires temperatures over 350°C. Therefore, temperature differences associated with dry vs lubricated may not be drivers of phase transformation and the associated mechanical property changes in CAD/CAM zirconia. However, as phase transformation results from a stress-induced martensitic phase transition,³⁹ future work explicitly examining differences in dry versus wet polishing of sintered zirconia is warranted, as both are currently being recommended by manufacturers. Notably for this study, the presented results show a disparity between two brands. This suggests that brand-specific recommendations that are ultimately utilized in different clinical manifestations are warranted to guide clinicians in polishing CAD/CAM zirconia. While differentiating application

forces of 1 N vs 2 N in a clinical setting may be challenging, the authors' experience shows that 1.0 N and 11.0 N are easily distinguished. This highlights the importance of recommendations that are not unduly burdensome to clinicians. Furthermore, we isolated force by removing polishing strokes, a common clinical procedure, as strokes may induce the potential confounder of additional torque. Thus, the interaction between strokes, force, and reuse merits further investigation.

CONCLUSIONS

The results of this *in vitro* study suggest, for the first time, that the reuse of polishing burs for two distinct systems is possible without any significant changes in the quantitative surface topography of zirconia. On initial use, the application force of the polishing burs can potentially modulate the resultant topography. However, this effect is lost following the reuse of the burs. Critically, this study combines microscopy techniques, including OP, AFM, and SEM, to highlight unique insight into the resultant zirconia surface topography. The product-dependent results reveal the presence of unique nanostructured topographies, even with similar Ra roughness values, which may have clinically relevant outcomes when considering cellular attachment and occlusal wear. The disparate results from two manufacturers show the need for further research and recommendations from manufacturers when considering polishing and finishing of CAD/CAM-prepared, sintered zirconia.

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Conflict of Interest

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

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**Effect of Resin-modified Glass Ionomer Cement
Dispensing/Mixing Methods on Mechanical Properties**

TA Sulaiman • AA Abdulmajeed • A Altitnchi • SN Ahmed • TE Donovan

Clinical Relevance: Automix resin-modified glass ionomer cements (RMGIs) may be easier and more efficient than a hand mix version of the same RMGI, but they may not have the same favorable mechanical properties, which could potentially affect the longevity of the cement.

doi: <https://doi.org/10.2341/17-166-L>

Application of the Self- Assembling Peptide P11-4 for Prevention of Acidic Erosion

S Suda • T Takamizawa • F Takahashi • A Tsujimoto • S Akiba • Y Nagura • H Kurokawa • M Miyazaki

Clinical Relevance: The use of peptide P11-4 may be considered for preventing enamel acid erosion.

doi: <https://doi.org/10.2341/17-175-L>

Nanofilled Resin Composite Properties and Clinical Performance: A Review

H Alzraikat • MF Burrow • GA Maghaireh • NA Taha

Clinical Relevance: Nanocomposites have been found to exhibit properties and clinical performance comparable to those of several hybrid composites but better than microfilled composites. However, there is no long-term evidence yet to show a superior performance that justifies their use in stress-bearing areas.

doi: <https://doi.org/10.2341/17-208-T>

**Do Nanofilled/Nanohybrid Composites Allow for Better Clinical Performance of Direct Restorations
Than Traditional Microhybrid Composites? A Systematic Review**

D Angerame • M De Biasi

Clinical Relevance: The effectiveness of direct restorations performed with nanofilled/nanohybrid composites was similar to that obtainable with traditional microhybrid composites. The weight of the available evidence supports the free choice in the clinical setting between these two classes of restorative materials.

doi: <https://doi.org/10.2341/17-212-L>

**At-home Bleaching With 10% vs More Concentrated Carbamide Peroxide Gels:
A Systematic Review and Meta-analysis**

JL de Geus • LM Wambier • TF Boing • AD Loguercio • A Reis

Clinical Relevance: Based on the results obtained in this study, similar results are obtained in terms of bleaching effectiveness with different concentrations of carbamide peroxide, but 10% carbamide peroxide produced lower risk and intensity of tooth sensitivity.

doi: <http://dx.doi.org/10.2341/17-222-L>

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Effect of Resin-modified Glass Ionomer Cement Dispensing/Mixing Methods on Mechanical Properties

TA Sulaiman • AA Abdulmajeed • A Altitinch • SN Ahmed • TE Donovan

Clinical Relevance

Automix resin-modified glass ionomer cements (RMGIs) may be easier and more efficient than a hand mix version of the same RMGI, but they may not have the same favorable mechanical properties, which could potentially affect the longevity of the cement.

SUMMARY

Statement of the Problem: Resin-modified glass ionomer cements (RMGIs) are often used for luting indirect restorations. Hand-mixing traditional cements demands significant time and may be technique sensitive. Efforts have been made by manufacturers to introduce the same cement using different dispensing/mixing methods. It is not known what effects these changes may have on the mechanical properties of the dental cement.

*Taiseer A Sulaiman (Taiseer), BDS, PhD, University of North Carolina, Department of Operative Dentistry, Chapel Hill, NC, USA

Awab A Abdulmajeed, DDS, University of North Carolina, Department of Operative Dentistry, Chapel Hill, NC, USA

Ali Altitinch, BDS, MSc, University of North Carolina, Department of Operative Dentistry, Chapel Hill, NC, USA

Sumitha N Ahmed, DDS, MS, University of North Carolina, Department of Operative Dentistry, Chapel Hill, NC, USA

Terrence E Donovan, DDS, University of North Carolina, Department of Operative Dentistry, Chapel Hill, NC, USA

*Corresponding author: 5405H Koury Oral Health Science Building, Chapel Hill, NC 27599, USA; e-mail: sulaiman@unc.edu

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Purpose: The purpose of this study was to evaluate the mechanical properties (diametral tensile strength [DTS], compressive strength [CS], and fracture toughness [FT]) of RMGIs with different dispensing/mixing systems.

Methods and Materials: The RMGI specimens (n=14)—RelyX Luting (hand mix), RelyX Luting Plus (clicker-hand mix), RelyX Luting Plus (automix) (3M ESPE), GC Fuji PLUS (capsule-automix), and GC FujiCEM 2 (automix) (GC)—were prepared for each mechanical test and examined after thermocycling (n=7/subgroup) for 20,000 cycles to the following: DTS, CS (ISO 9917-1) and FT (ISO standard 6872; Single-edge V-notched beam method). Specimens were mounted and loaded with a universal testing machine until failure occurred. Two-/one-way analysis of variance followed by Tukey honestly significantly different post hoc test was used to analyze data for statistical significance ($p<0.05$).

Results: The interaction effect of both dispensing/mixing method and thermocycling was significant only for the CS test of the GC group ($p<0.05$). The different dispensing/mixing methods had no effect on the DTS of the tested cements. The CS of GC Fuji PLUS was significantly higher than that of the automix version

($p < 0.05$). The FT decreased significantly when switching from RelyX (hand mix) to RelyX Luting Plus (clicker-hand mix) and to RelyX Luting Plus (automix) ($p < 0.05$). Except in the case of the DTS of the GC group and the CS of GC Fuji PLUS, thermocycling had a significant effect reducing the mechanical properties of the RMGI cements ($p < 0.05$).

Conclusions: Introducing alternative dispensing/mixing methods for mixing RMGIs to reduce time and technique sensitivity may affect mechanical properties and is brand dependent.

INTRODUCTION

Dental cements have been used to retain indirect restorations. The success and longevity of these restorations are highly dependent on the durability of the cements. Zinc phosphate cement was the traditional cement in use since 1879.¹ Zinc phosphate cement was provided as a powder and liquid that required a special mixing technique to achieve optimum success.² Over the past 40 years, other cements have been introduced with variable degrees of success. These cements are polycarboxylate cement, glass ionomer and resin-modified glass ionomer cements (RMGIs), and, finally, resin-based cement in its self-adhesive or adhesive versions.³⁻⁵

Extensive research has been conducted over the years, and most of the newer versions of these cements lack proper clinical evaluation. *In vitro* tests provide useful information concerning the material itself; however, caution should be taken before concluding any clinical significance from these studies, as many of these tests do not relate directly to the potential clinical performance of the cement. In regard to the retentive function of cements, it has been concluded^{6,7} that several variables interact with each other, such as the resistance and retention form of the preparation, surface characteristics of the tooth and the intaglio surface of the restoration, and, finally, variables pertaining to the cement itself.

Dental cements are provided with different dispensing/mixing methods. Some are hand mixed from a powder-liquid formulation, while others are supplied as a paste-paste consistency that is either hand mixed (in a "clicker" device) or equipped with an automixing tip that dispenses and mixes the cement. Clinicians likely assume that cements of the same brand with two different forms of mixing method have the same properties. However, for the cements

to be used with different dispensing/mixing devices, the basic formulations must be modified physically and chemically. These modifications may affect the resultant mechanical properties of the cements.

Johnson and others⁸ made the remarkable discovery that the powder-liquid version of some RMGI cements was more retentive than the paste-paste counterpart. Retention ability of the cements was the primary focus in their study. No evaluation of specific mechanical properties was done in that study. It is therefore important to further investigate the possible consequences of different dispensing/mixing methods on the diametral tensile strength (DTS), compressive strength (CS), and fracture toughness of RMGI cements. The research hypothesis was that changing the dispensing method of the cement has no effect on its mechanical properties.

METHODS AND MATERIALS

The RMGI cements used for this study and their dispensing methods are listed in Table 1. With regard to the DTS, 14 cylindrical-shaped specimens (6.0 ± 0.1 mm in diameter and 3.0 ± 0.1 mm in height) of each RMGI were prepared at room temperature ($23^\circ\text{C} \pm 1^\circ\text{C}$), according to specification No. 27 of the American National Standards Institute/American Dental Association,⁹ slightly overfilling the stainless-steel molds that rested on a glass slide with the mixed cement; two transparent ethylene films were used to confine the cement on either side of the mold to minimize oxygen exposure from the atmosphere. A second glass slide was placed on top of the mold and pressed by slight finger pressure to allow extrusion of excess cement. After each RMGI cement setting time, the specimens were then carefully removed from the molds; any specimen with an obvious void was excluded from testing, and a new specimen was made. A No. 15 blade was used to remove excess cement from the edges of the specimen; an 800-grit silicon-carbide abrasive paper (MicroCut™, Buehler, Lake Bluff, IL, USA) was used for final smoothing. The final dimensions of each specimen were calculated by taking the mean of two measurements at right angles to each other, made to an accuracy of 0.01 mm using a digital micrometer (Digimatic Micrometer, Mitutoyo Corporation, Aurora, IL, USA). Specimens were then stored in water at 37°C for 24 hours to achieve maximum maturity and were then mounted diametrically, and the DTS was calculated for the nonthermocycled specimens ($n=7$) and after thermocycling ($n=7$) (20,000 cycles at 5°C - 55°C and a 15-second dwell time; TC-8, SD Mechatronik,

Table 1: Resin-modified Glass Ionomer Cements Used for the Study and Their Dispensing/Mixing Method				
Resin-modified Glass Ionomer	Manufacturer	Dispensing Method	Composition	Lot No.
RelyX Luting	3M ESPE	Powder:liquid Hand mix	Powder: fluoroaluminosilicate glass, potassium persulfate, ascorbic acid, opacifying agent Liquid: methacrylated polycarboxylic acid, water, HEMA, tartaric acid	N768842
RelyX Luting Plus	3M ESPE	Paste:paste clicker–Hand mix	Paste A: fluoroaluminosilicate glass, proprietary reducing agent, HEMA, water, opacifying agent Paste B: methacrylated polycarboxylic acid, Bis-GMA, HEMA, water, potassium persulfate, zirconia silica filler	N748797
RelyX Luting Plus Automix	3M ESPE	Paste:paste automix	Paste A: radiopaque fluoroaluminosilicate glass, opacifying agent, HEMA, water, dispersion aid, reducing agent Paste B: nonreactive zirconia silica filler, methacrylated polycarboxylic acid, HEMA, resin monomers, water, potassium persulfate, photoinitiator	N769817
GC Fuji PLUS	GC Corporation	Powder:liquid Capsule-automix	Powder: fluoroaluminosilicate glass, initiator, pigment Liquid: 4-META, phosphoric acid ester monomer, water, UDMA, dimethacrylate, silica powder, initiator, stabilizer 65%-70 %wt	1602101
GC FujiCEM 2	GC Corporation	Paste:paste automix	Paste A: fluoroaluminosilicate glass, initiator, UDMA, dimethacrylate, pigment, silicon dioxide, inhibitor Paste B: silicon dioxide, UDMA, dimethacrylate, initiator, inhibitor	1602082
Abbreviations: Bis-GMA, bisphenol A diglycidyl ether dimethacrylate; 4-META, 4-methacryloxyethyl trimellitate anhydride; HEMA, hydroxyethyl methacrylate; UDMA, urethane dimethacrylate.				

Feldkirchen-Westerham, Germany). The maximum force (N) at failure was recorded using a universal testing machine (Instron 4411, Instron, Norwood, MA, USA) at a cross-head speed of 1.0 mm/min, according to the following formula:

$$DTS = 2F/\pi dt,$$

where *F* indicates maximum force applied in newtons; *d* indicates the diameter of the specimen; and *t* indicates the thickness of the specimen.

For the compressive strength test, 14 cylindrical-shaped specimens (4.0±0.1 mm in diameter and 6.0±0.1 mm in height) (ISO standard 9917-1)¹⁰ for each RMGI were prepared at room temperature (23°C±1°C) and mixed according to the manufacturers' instructions, and specimen preparation was handled similarly to that of the DTS test previously explained.

Specimens were then stored in water at 37°C for 24 hours. CS was calculated for half of the specimens before thermocycling, and the other half were thermocycled for 20,000 cycles at 5°C-55°C with a 15-second dwell time prior to CS calculation. Measurement of the force (N) at which failure occurred was recorded using a universal testing machine at a

cross-head speed of 1.0 mm/min. CS was calculated according to the following formula:

$$CS = 4P/\pi d^2,$$

where *P* indicates maximum force applied (in newtons) and *d* indicates the measured diameter of the specimen (in millimeters).

Concerning the FT test, the single edge V-notched beam method, according to ISO standard 6872, was used to measure the FT.¹¹ Beam-shaped specimens (n=14/group) measuring 21.0 ± 0.1 mm in length and with a rectangular cross section of 4.0 ± 0.2 mm in thickness and 3.0 ± 0.2 mm in depth, as seen in Figure 1A and B, were prepared for each RMGI cement at room temperature (23°C±1°C) and mixed according to the manufacturer's instructions. A notch depth of approximately 0.5 mm was machined into the bar specimen using a 300-µm-thick diamond blade. Diamond polishing paste (3.5 µm, Kent Supplies, Quebec, Canada) was placed into the notch tip, and a razor blade was inserted into the notch along with light force (5-10 N) that was applied using gentle back-and-forth motion and as straight as possible.

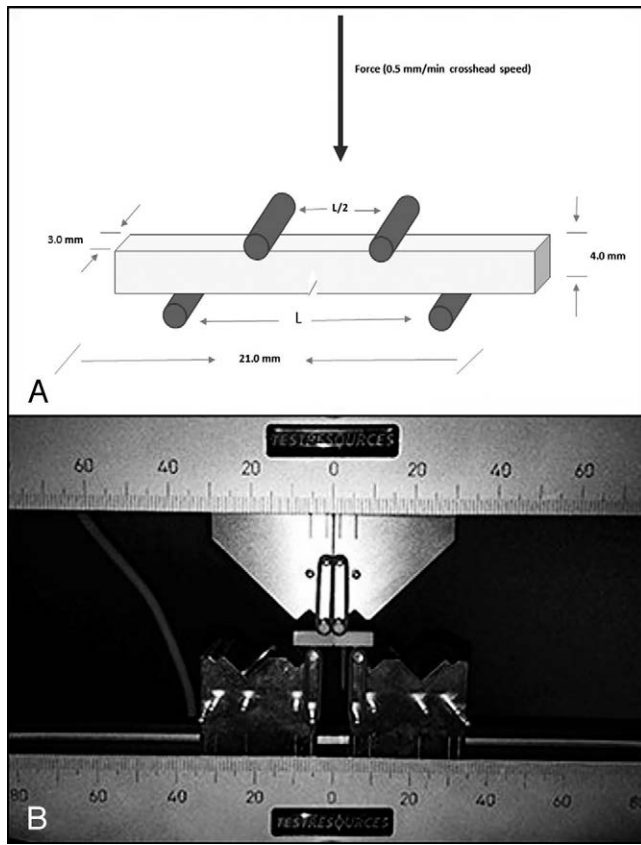


Figure 1. (A) Schematic illustration of fracture toughness specimen. L is the distance between the rollers in the four-point bending fixture. (B) Specimen placed on four-point bending fixture.

A calibrated light microscope was used to examine both ends of the V-notch and to measure the depths of the V-notches with magnification power of $>50\times$ to three significant figures. The width (b) and thickness (w) of each specimen were recorded using a digital micrometer capable of measurements to $\pm 1\text{-}\mu\text{m}$ accuracy (QuantuMike Micrometer, Mitutoyo Corporation). The specimens were then cleaned with acetone in an ultrasonic bath and were then dried by heating to 110°C for 10 minutes. The drying process time was modified from the original ISO standard 6872 of one hour intended for ceramic materials to avoid damage to the RMGI specimens. In addition to drying the specimens after ultrasonic cleaning, this heating process likely aided the polymerization reaction in reaching its maximum potential. Specimens were then immersed in water at 37°C for 24 hours prior to testing. The FT test was carried out before ($n=7$) and after thermocycling for 20,000 cycles at 5°C - 55°C with a 15-second dwell time ($n=7$), using a four-point bending fixture. The V-notch was placed facing down (tensile side). The specimens were loaded on a universal testing

machine with a crosshead speed of 0.5 mm/min . FT K_{Ic} ($\text{MPa}\times\text{m}^{1/2}$) was calculated using the following equation:

$$K_{Ic} = \frac{F}{b\sqrt{w}} \cdot \frac{S1 - S2}{w} \cdot \frac{3\sqrt{\alpha}}{2(1 - \alpha)^{1.5}} \cdot Y$$

$$Y = 1.9887 - 1.326\alpha - \frac{(3.49 - .68\alpha + 1.35\alpha^2)\alpha(1 - \alpha)}{(1 + \alpha)^2}$$

where K_{Ic} = fracture toughness; α = relative notch depth (m); Y = stress intensity shape factor; F = fracture load (MN); b = specimen width (m); w = specimen thickness (m); $S1$ = support span (m); and $S2$ = inner four-point span (m).

Two- and one-way analyses of variance (ANOVAs) were conducted for each RMGI cement group with the main variables of “hand mix” and “automix,” depending on the method of dispensing. The post hoc multiple-comparison Tukey test was used for pairwise comparisons between the group means. A significance level of $\alpha = 0.05$ was used for all tests. All statistical analysis was performed with Statistical Package for Social Sciences (SPSS version 24, SPSS Inc, Armonk, NY, USA).

RESULTS

The combined effect of both independent parameters (dispensing/mixing method and thermocycling) was significant only for the CS of the GC group ($p < 0.05$) according to two-way ANOVA.

According to one-way ANOVA, when comparing the effect of different dispensing/mixing methods on the mechanical properties tested (Table 2), there were no statistically significant differences in DTS within the RelyX and GC groups. The mean CS of GC Fuji Plus (capsule-automix) was significantly greater than that of GC FujiCem 2 (automix) ($p < 0.05$). The mean FT of RelyX Luting (hand mix, $0.50\text{ MPa}\times\text{m}^{1/2}$) was significantly greater than that of RelyX Luting Plus (clicker-hand mix, $0.36\text{ MPa}\times\text{m}^{1/2}$), which was also significantly greater than that of RelyX Luting Plus (automix, $0.24\text{ MPa}\times\text{m}^{1/2}$). There was no significant difference in FT between GC Fuji Plus (capsule-automix) and GC FujiCem 2 (automix). The percentage of change in mechanical properties according to the dispensing/mixing method can be seen in Table 3.

When determining the effect of the thermocycling on mechanical properties (Table 2), there was a significant reduction DTS of the RelyX cements after

Table 2: Mean and Standard Deviation (SD) of Mechanical Properties of Resin-modified Glass Ionomer (RMGI) Cements Before and After Thermocycling (TC)^a

Resin-based Cements		Diametral Tensile Strength, MPa (Mean±SD)	Compressive Strength, MPa (Mean±SD)	Fracture Toughness, MPa × m ^{1/2} (Mean±SD)
RelyX Luting (hand mix)	Before TC	20.93 ± 2.99*	104.24 ± 12.98*	0.80 ± 0.12*
	After TC	13.51 ± 1.91	65.07 ± 5.49	0.50 ± 0.06 _A
RelyX Luting Plus (clicker–hand mix)	Before TC	21.85 ± 4.17*	108.14 ± 6.27*	0.67 ± 0.11*
	After TC	11.79 ± 3.24	61.68 ± 5.11	0.36 ± 0.04 _B
RelyX Luting Plus (automix)	Before TC	26.44 ± 5.45*	114.37 ± 6.16*	0.55 ± 0.02*
	After TC	18.55 ± 9.84	68.24 ± 8.86	0.24 ± 0.01 _C
GC Fuji CEM Plus (capsule-automix)	Before TC	18.08 ± 4.10	91.81 ± 10.63	0.63 ± 0.07*
	After TC	12.61 ± 2.97	81.08 ± 7.75 _A	0.29 ± 0.05
GC FujiCEM 2 (automix)	Before TC	14.91 ± 3.46	85.22 ± 11.04*	0.50 ± 0.09*
	After TC	14.55 ± 3.11	46.86 ± 4.01 _B	0.28 ± 0.07

^a * Indicates statistical difference $p < 0.05$ between the same RMGI cement brand before and after TC. Different letters indicate statistical difference within the same cement group after TC; $p < 0.05$.

thermocycling ($p < 0.05$), with no significant effect on the DTS of the GC cements. The CS of all RMGI cements reduced significantly ($p < 0.05$) after thermocycling, except in the case of GC Fuji Plus. FT values decreased significantly for all RMGI cements after thermocycling ($p < 0.05$).

DISCUSSION

Based on the results of this *in vitro* study, it is evident that changing the dispensing/mixing method of the cement may translate into unfavorable effects on its mechanical properties. However, as in many other *in vitro* studies, caution should be taken before concluding any clinical relevance related to these changes. How this change in mechanical properties can affect the clinical longevity of the retention ability of RMGI cements is yet to be determined.

The research hypothesis for this study was that there was no effect of changing the dispensing/mixing method of the RMGI on the resultant mechanical properties. This hypothesis was rejected.

Changing the dispensing method altered at least one or more of the mechanical properties of each cement significantly. It is not exactly clear what modifications to the composition of the RMGI cements were made by the manufacturers to accommodate for the change in dispensing/mixing method. Further research should be done to examine in detail the changes in the cements' composition and their correlation to the changes in mechanical properties.

In the study of Johnson and others,⁸ the automix (paste:paste) RMGI cement was less retentive than the hand-mix (powder:liquid) counterpart. They acknowledged the fact that the two versions of the same cement are not the same and that the manufacturer modified the chemical composition of the cements. They speculate that the manufacturer may have formulated a new aqueous base paste to allow the automix reaction. Furthermore, the traditional chemical initiators are sensitive to water, and in order to control setting times and stability of the automix version of the cement, new initiators were

Table 3: Percentage Change in Mechanical Properties When Changing from Hand Mix Dispensing/Mixing Method of Each Resin-modified Glass Ionomer Cement Type

	Diametral Tensile Strength, MPa	Compressive Strength, MPa	Fracture Toughness, MPa × m ^{1/2}
RelyX Luting (hand mix)	↓ 12.7%	↓ 5.2%	↓ 28%
RelyX Luting Plus (clicker)			
RelyX Luting (hand mix)	↑ 37.3%	↑ 4.8%	↓ 52%
RelyX Luting Plus (automix)			
RelyX Luting Plus (clicker)	↑ 57.3%	↑ 10.6%	↓ 33.3%
RelyX Luting Plus (automix)			
GC Fuji PLUS (capsule- automix)	↑ 15.3%	↓ 42.2%	↓ 3.4%
GC FujiCEM 2 (automix)			

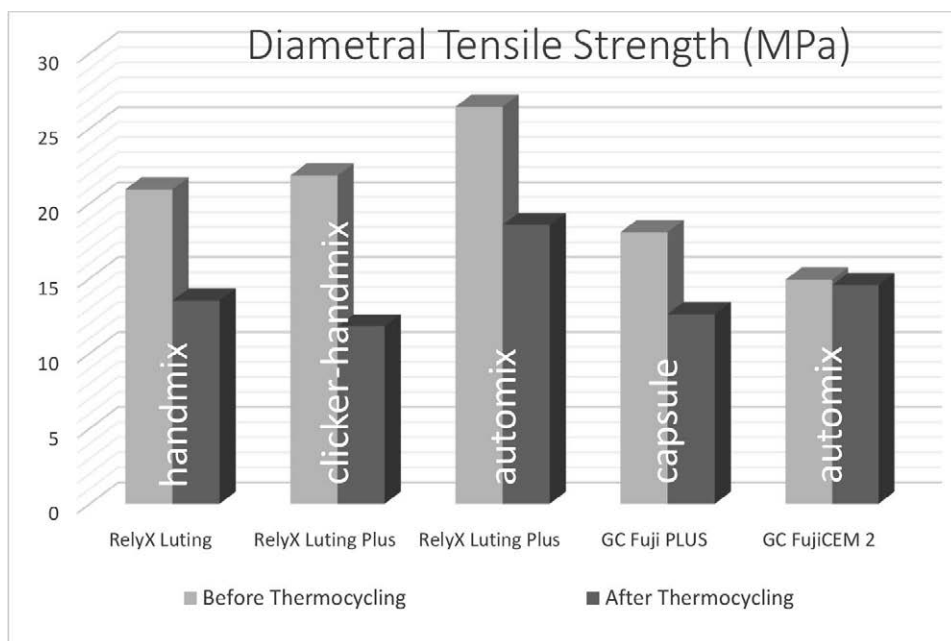


Figure 2. Mean diametral tensile strength of resin-modified glass ionomer cements before and after thermocycling.

developed. Therefore, this study investigated the changes in mechanical properties that resulted between the different versions of the same cement. This information may be important for the profession, as most clinicians likely assume equivalence in the performance of the same cement brand with different dispensing/mixing methods.

Based on the results of this study, there was no significant effect in DTS values of the RMGI cements with different dispensing methods (Figure 2), despite

the 37.3% increase in DTS value from the hand-mix (powder:liquid) to the automix (paste:paste) version of the RelyX RMGI cements. The CS values showed a significant effect with one type of RMGI cement (GC Fuji PLUS), with a 42.2% drop in its CS value noted when changing to the automix version (Figure 3). The FT value of the RelyX Luting (hand mix) dropped around 28% compared to the (clicker-hand mix) version and further dropped to 52% when changing to the automix version (Figure 4). Clearly, there is no equivalence in mechanical properties for

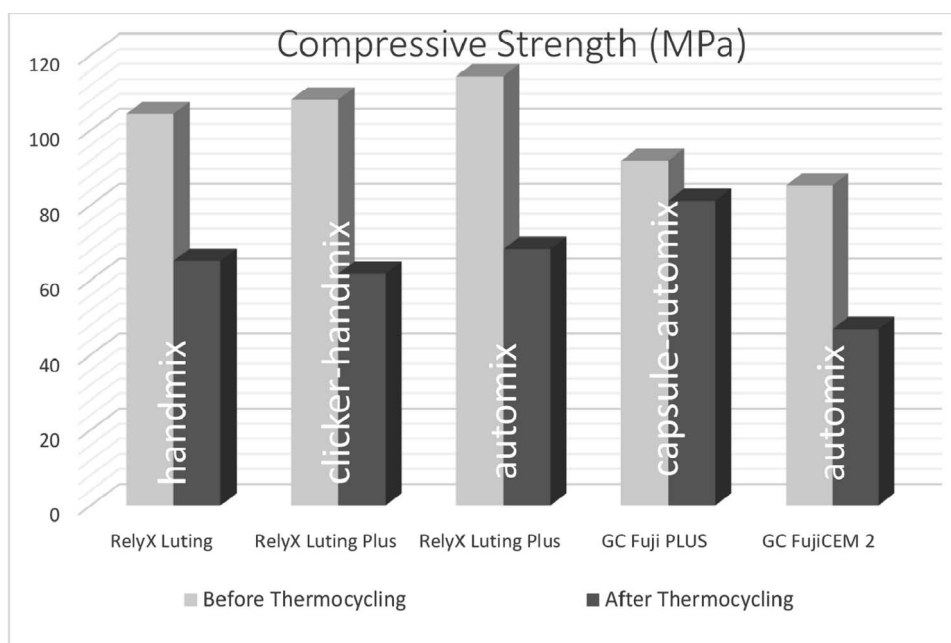


Figure 3. Mean compressive strength of resin-modified glass ionomer cements before and after thermocycling.

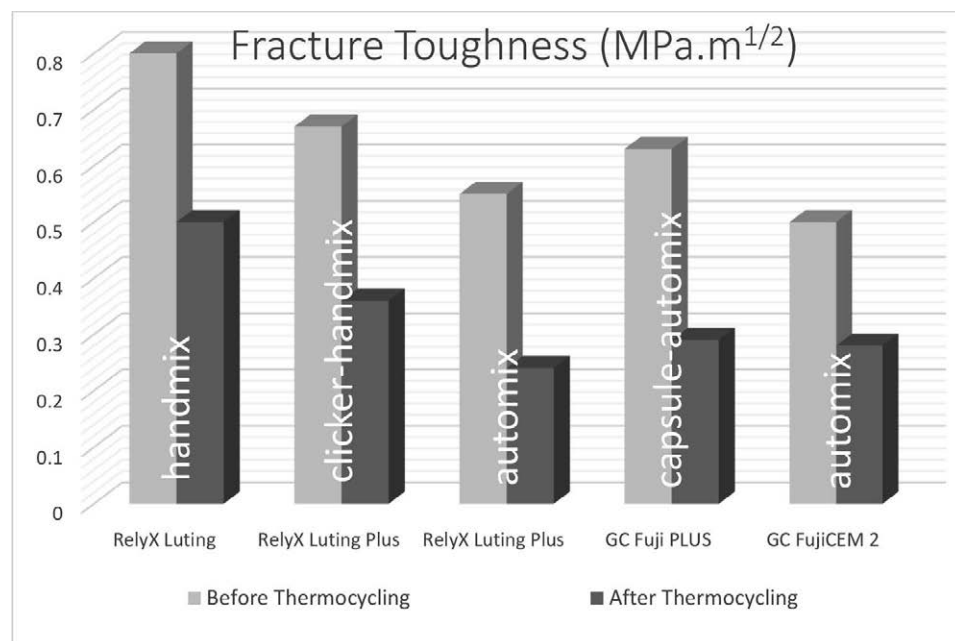


Figure 4. Mean fracture toughness of resin-modified glass ionomer cements before and after thermocycling.

the same cement with different dispensing/mixing methods.

Thermocycling (artificially aging) the RMGI cements added to this study in that it allowed us to simulate the physiological aging experienced by the cements in the oral environment. It has been reported¹² that TC for 10,000 cycles at 5°C-55°C with a dwell time of 15 seconds may approximately correspond to one year of clinical function. In this study, a period of 20,000 cycles was adopted to further age the cements to a two-year clinical function. Indeed, TC reduced the mechanical properties of all RMGI cements evaluated in this study, some more significantly than others.

It is very important to translate these findings with caution when searching for clinical recommendations; as one cement brand's mechanical properties reduce in value when switching from a powder-liquid mix to a paste-paste mix, other properties may be enhanced. The bottom line is that "easier may not always be better."

CONCLUSIONS

Within the limitations of this study, the following may be concluded:

1. The mechanical properties of different brands of RMGI cements are not the same, and certain properties may change when switching the dispensing/mixing method.

2. The mechanical properties of all RMGI cements are affected by thermocycling. This process may have an effect on the durability and function of the cement.
3. Clinical relevance from this study should be interpreted with caution. It is not clear how changing the dispensing/mixing method of the same cement brand affects the cements' durability, but the differences in mechanical properties are quite interesting.

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Conflict of Interest

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

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Application of the Self-Assembling Peptide P11-4 for Prevention of Acidic Erosion

S Suda • T Takamizawa • F Takahashi • A Tsujimoto • S Akiba
Y Nagura • H Kurokawa • M Miyazaki

Clinical Relevance

The use of peptide P11-4 may be considered for preventing enamel acid erosion.

SUMMARY

The aim of this study was to use ultrasonography to evaluate the effect of the self-assembling peptide P11-4 on acid erosion prevention. Curodont Repair (CR), which includes peptide P11-4, was used. Rectangular prisms of bovine enamel (4×1×1 mm) were immersed in pure orange juice for a period of 5 minutes

Syunichi Suda, DDS, Operative Dentistry, Nihon University School of Dentistry, Tokyo, Japan

Fuminori Takahashi, DDS, PhD, Operative Dentistry, Nihon University School of Dentistry, Tokyo, Japan

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

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

Shunsuke Akiba, DDS, Operative Dentistry, Nihon University School of Dentistry, Tokyo, Japan

Yuko Nagura, DDS, Operative Dentistry, Nihon University School of Dentistry, Tokyo, Japan

Hiroyasu Kurokawa, DDS, PhD, Operative Dentistry, Nihon University School of Dentistry, Tokyo, Japan

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

*Corresponding author: 1-8-13, Kanda-Surugadai, Chiyoda-Ku, Tokyo, 101-8310 Japan; e-mail: takamizawa.toshiki@nihon-u.ac.jp

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six times per day for 28 days. These samples were divided into four groups of six specimens each and treated differently for an additional period of 28 days: 1) baseline group specimens were stored in artificial saliva; 2) CR group specimens were exposed to curodont without acid challenge; 3) NCRA (no curodont+acid challenge) specimens were treated with orange juice without curodont exposure; and 4) CRA (CR+acid challenge) specimens were treated with curodont before treatment with orange juice. The propagation time of longitudinal ultrasonic velocity (UV) was measured. Ultrastructural observation of each tested enamel surface was carried out using field-emission scanning electron microscopy (SEM). The UV data were analyzed using two-way analysis of variance with time and treatment as confounding factors. Post hoc pairwise tests among groups were performed using the Tukey honestly significant difference test. The average UV in intact bovine enamel for the baseline group ranged from 4,483 to 4,549 m/s and did not vary significantly within the test period. The average ultrasonic velocity (UV) in all samples decreased after the initial erosion. The UV in NCRA decreased further over time. Increased UVs were found for CR and CRA. For CR and

CRA, there was no significant difference in UV at the end of the experiment from the initial value before erosion. In the results of SEM observation, the CR and CRA groups had similar morphologic features in that etching patterns were not clearly due to precipitation between the enamel rods. From the results of this *in vitro* study, it might be concluded that applying enamel matrix derivatives and self-assembling peptides on erosive lesions can improve remineralization.

INTRODUCTION

Tooth wear is becoming increasingly significant due to the wide availability and frequent consumption of acidic soft drinks, sports drinks, and fruit juices.¹ Erosive tooth wear involves a chemical process in which the inorganic phase of a tooth is demineralized, thereby allowing for surface dissolution of hard dental tissues. It is known that acidic food and drinks that contain citric, malic, phosphoric, and other acids can soften hard dental tissues.² In clinical situations, the initial sign of enamel erosion is the appearance of a smooth glazed surface. Moreover, reduced hardness may induce further enamel loss with the appearance of shallow concavities, rounding of the cusps, and eruption of restorations that seem to rise above the adjacent tooth level.

Prevention of the erosive process can be influenced by many factors, including tooth exposure to fluoride and the use of remineralization agents and protective materials. Many strategies have been developed to this end, including the use of materials containing fluoride compounds, casein phosphopeptide-amorphous calcium phosphate (CPP-ACP), and resin-based materials.³⁻⁸ In particular, although fluoride products are extensively recommended and widely used in oral health, fluoride formulations alone provide only limited protection against erosive tooth wear.⁹ There are few nonoperative treatments for remineralization or repair of erosive lesions at present.¹⁰

According to the principles of modern dentistry, optimal strategies should be favored for the treatment of early erosive lesions.¹¹ An early erosive lesion in this sense is one in which there has been no loss of bulk in the enamel, but demineralization has started, so that the enamel is softer. In clinical situations, pure early erosive lesions are very unusual, as some loss of bulk is almost inevitable, but they still represent a condition that is present, and which can be treated independently of bulk

restorations. Amelogenin use to control calcium and phosphate crystallization resulted in the growth of nano-sized rod-like apatite crystals.¹² A new product containing the peptide P11-4 was recently introduced to enhance remineralization and inhibit demineralization of the tooth substrate.¹³ Peptide P11-4 self-assembles into a three-dimensional (3D) scaffold on the surface of the tooth, where it promotes hydroxyapatite precipitation while inhibiting diffusive mineral loss. Therefore, due to its mineralization effect, this material is potentially useful not only for early carious lesions but also for enamel defects caused by erosion and tooth wear.¹⁴ A previous report indicated that the application of peptide P11-4 had effectively prevented demineralization and enhanced remineralization of enamel, which is considered an effective microinvasive approach for preventing enamel demineralization.¹⁵ The self-assembling peptide P11-4 is designed to form fibrils at a low pH and to be monomeric in solutions with higher pH, and dissolved Ca^{2+} is attracted by the negative charged surfaces and phosphorylated serine residues in the peptide. The presence of peptide P11-4 might permit a rapid return to normal mineral concentrations and allow ions to be reused in crystal growth, in addition to controlling the orientation and elongation of the hydroxyapatite crystals.¹⁶ As demineralization is an equilibrium process, an elevated local concentration of Ca^{2+} can be expected to inhibit it. However, these potential mechanisms have yet to be directly confirmed.

Ultrasonic imaging is a useful technique that shows considerable potential as a noninvasive diagnostic and research tool.¹⁷ This modality can be used to detect carious lesions and to measure the elastic modulus of the tooth substrate.¹⁸ The enamel substrate is mainly composed of hydroxyapatite, and differences in ultrasonic velocity (UV) are related to variations in the degree of mineralization, as UV increases proportionally with the volumetric concentration of minerals.¹⁹ UV has also been shown to be an index of remineralization and demineralization because of this correlation to the mineral content of the tooth substrate.²⁰ The level of mineralization of dental enamel is closely linked to its stiffness and/or elastic response.²¹ When remineralization of enamel occurs, mineral concentration and UV will increase.²² In this experiment, ultrasonic measurements were used to determine the density of internal structure, and thus degree of mineralization, of the enamel and not to measure any loss of enamel bulk.

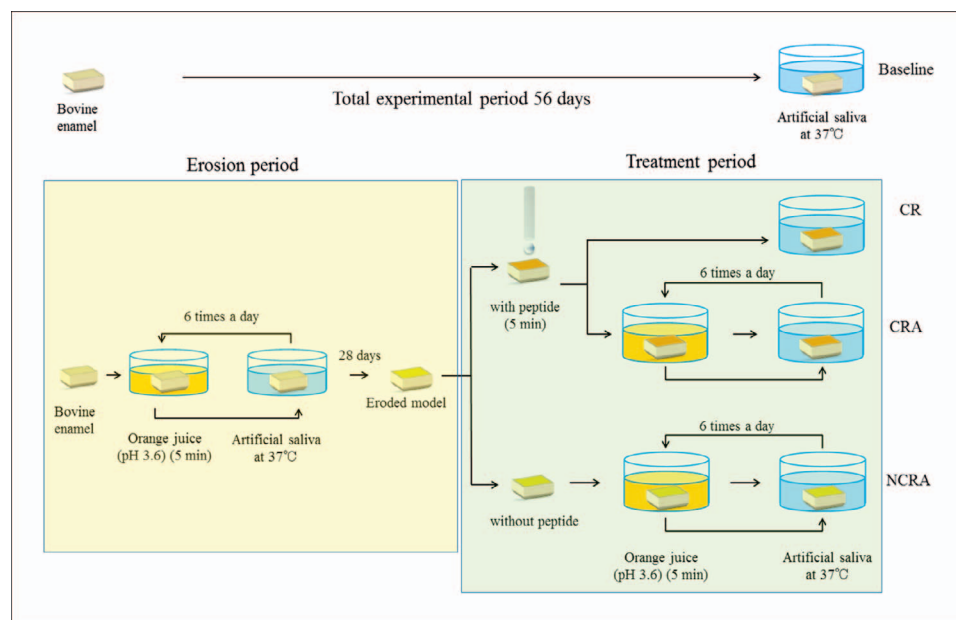


Figure 1. Experimental treatment steps of the enamel specimens.

Considering the potential of the self-assembly peptide P11-4 to enhance enamel remineralization, the purpose of this study was to use ultrasonic measurements to evaluate the preventive effects of peptide P11-4 on acidic erosion of enamel substrates. The null hypothesis was that use of the self-assembling peptide P11-4 would not result in a protective effect against enamel erosion.

METHODS AND MATERIALS

Specimen Preparation

A total of 26 freshly extracted bovine incisors without cracks or erosive lesions were cleaned and stored in physiological saline. The teeth were sliced into 1 mm sections in different directions with a low-speed diamond saw (Buehler–Illinois Tool Works Inc., Lake Bluff, IL, USA). Each enamel slab was carefully shaped into a rectangular form (4×4×1 mm) using a super-fine diamond finishing point (ISO #021; Shofu Inc, Kyoto, Japan) attached to an air-turbine (Twin-Power Turbine; J. Morita Mfg Corp, Kyoto, Japan). Specimen surfaces were successively ground using wet silicon carbide paper with grit sizes of #600, #1200, and #2000. The thickness and size of the specimens were measured using a dial gauge micrometer (CPM15-25DM; Mitsutoyo, Tokyo, Japan). The resource equation method was used for sample-size calculation²³ and the samples were divided into four groups of six samples each. Two eroded samples were used for scanning electron microscopy (SEM) observations and the remaining 24 were divided into three groups of six specimens each.

Experimental Protocol

To create a model of eroded enamel, the prepared samples were immersed in pure orange juice (pH 3.6) for 5 minutes six times per day for 28 days. For the first group, Curodont Repair (CR group), the eroded specimens were treated with peptide P11-4 and stored in artificial saliva (pH 7.0, 14.4 mM NaCl, 16.1 mM KCl, 0.3 mM MgCl₂·6 H₂O, 2.0 mM K₂HPO₄, 1.0 mM CaCl₂·2 H₂O, and 0.10 g% sodium carboxymethyl cellulose) without acid challenge for a further 28 days. For the second group (curodont + acid challenge; CRA group), the eroded specimens were treated with peptide P11-4 and then with orange juice in the same way for a further 28 days. For the CR and CRA groups, treatment with peptide P11-4 was only carried out once, after the initial erosion period finished (28 days) and before the treatment period began (the further 28 days). In the last group (no curodont + acid challenge; NCRA group), the eroded specimens were not treated with peptide P11-4 and then were treated in the same way as the CRA group. Baseline group specimens were stored in artificial saliva for the experimental period (erosion period + treatment period, 56 days). (Figure 1).

Ultrasonic Measurement

Ultrasonic velocity (UV) was measured using a pulser/receiver (Model 5900PR; Panametrics, Inc, Waltham, MA, USA), a transducer for longitudinal waves (V112; Panametrics, Inc), and an oscilloscope (Wave RunnerLT584; Teledyne LeCroy, Chestnut

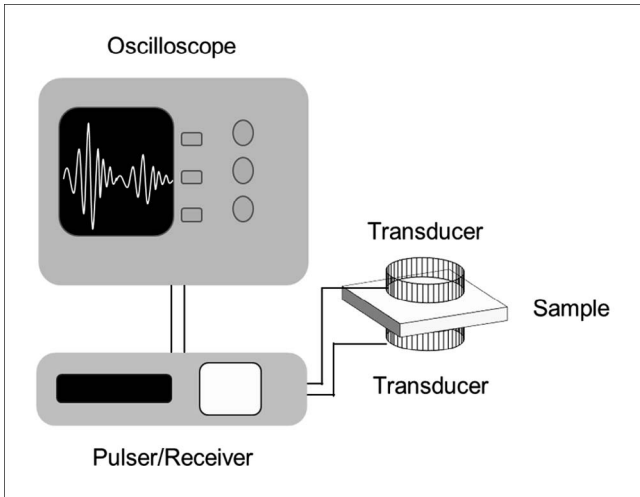


Figure 2. Set-up of the ultrasonic device to detect changes in enamel remineralization and demineralization.

Ridge, NY, USA). The equipment was initially calibrated using a standard procedure with 304 stainless steel calibration blocks (2211M; Panametrics, Inc). The transducer was oriented perpendicular to the contact surface of each specimen to obtain an echo signal (Figure 2). Ultrasonic waves were propagated from the transducer to the tooth, transmitted through the tooth, and then detected by the receiver on the opposite side. The transducers were connected to the pulser-receiver in through-transmission mode, and 16 μ J pulses were applied at a pulse repetition frequency of 500 Hz. The signal was captured using an oscilloscope. Heat generation was not observed from the transducers during measurement, and the system was calibrated following standard procedures before each specimen was measured. Each measurement was made at 23°C $\pm$

1°C and 50% $\pm$ 5% relative humidity. Measurements were taken before the test and on days 1, 7, 14, 21, and 28 during both the preparatory erosion period and the treatment period. In addition, every sample thickness was measured using a dial gauge micrometer (CPM15-25DM, Mitsutoyo) before each set of UV measurements. Measurements were made before peptide application on day 28 of the preparation period and then on day 1 of the test period, which was the day after peptide application. The propagation velocity of ultrasound (UV) was calculated from sample thickness and transmission time.

SEM Observations

Ultrastructural observation of enamel surfaces was carried out using field-emission SEM. Specimens were first dehydrated in ascending concentrations of tert-butanol (50% for 20 minutes, 75% for 20 minutes, 95% for 20 minutes, and 100% for 2 hours) and then transferred to a critical-point dryer for 30 minutes. The surfaces were then coated with a thin gold film in a vacuum evaporator (Quick Coater Type SC-701; Sanyu Denshi Inc, Tokyo, Japan) and observed by SEM (ERA 8800FE; Elionix Ltd, Tokyo, Japan) at an accelerating voltage of 10 kV.

Statistical Analysis

The UV data were analyzed using two-way analysis of variance with time and treatment as confounding factors. Time was treated as a repeated measure. Post hoc pairwise tests among groups were performed using the Tukey honestly significant difference test. The level of significance was set at 0.05, and all calculations were performed using SigmaStat software (Systat Software, Inc, San Jose, CA, USA).

Table 1: Mean (Standard Deviation) Ultrasonic Velocities (m/s) of Bovine Enamel Specimens by Treatment (n=6) ^a											
Group	Erosion Period (Days)					Treatment (Days)					
	0	7	14	21	28	Peptide P ₁₁₋₄	0	7	14	21	28
Baseline	4549 (90) aA	4538 (90) aA	4584 (61) aA	4547 (52.4) aA	4547 (44.2) aA	Not applied (without acid challenge)	4483 (61.1) aA	4485 (97.9) aA	4538 (88.6) aA	4520 (95.8) aA	4536 (87.1) aA
CR	4545 (90) aA	4308 (90) bB	4184 (61) cB	4147 (52) cB	4047 (44) dB	Applied (without acid challenge)	4083 (61) dC	4285 (98) bcBCCD	4338 (89) bB	4320 (96) bC	4336 (87) bC
CRA	4555 (53) aA	4341 (54) bB	4222 (90) bcB	4138 (35) cB	4092 (42) cB	Applied (with acid challenge)	4167 (22) cB	4341 (64) bB	4362 (71) bB	4416 (77) abB	4415 (62) abB
NCRA	4539 (54) aA	4231 (67) bB	4155 (78) cC	4113 (81) cC	4069 (41) dB	No applied (with acid challenge)	4088 (42) cC	4080 (26) cC	4062 (20) dC	4032 (10) dcD	3968 (11) cD
Abbreviations: CR, curodont repair; CRA, curodont + acid challenge; NCRA, no curodont + acid challenge.											
^a Within groups, means with the same lowercase letter are not significantly different (Dunnett test, p>0.05). Between groups at the same storage times, means with the same uppercase letter are not significantly different (Tukey-Kramer post hoc test, p>0.05).											

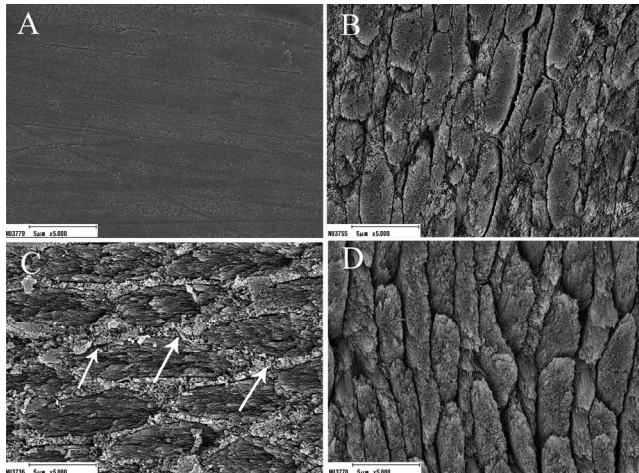


Figure 3. Representative SEM images of the enamel surface of each specimen. SEM observations revealed differences in morphologic features among storage conditions. Demineralization of the enamel surfaces was more pronounced in the NCRA group, whereas the enamel surfaces of the CRA groups exhibited unclear etching patterns due to precipitation between the enamel rods. (A): Untreated enamel ground with #2,000 SiC paper ($\times 5,000$). (B): After initial treatment with pure orange juice (pH 3.6) for 5 minutes six times per day for 28 days ($\times 5,000$). (C): Representative CRA sample at 28 days ($\times 5,000$). Arrows indicate precipitation between the enamel rods. (D): Representative NCRA sample at 28 days ($\times 5,000$).

RESULTS

Ultrasonic Measurement

Changes in the average UVs of each group are shown in Table 1. During the erosion period, the UVs in all groups decreased with increased erosion period. Because the differences between storage periods were greater than expected, multiple comparisons were performed on the data after factoring in the effects of storage conditions. The average UV in intact bovine enamel for the baseline group ranged from 4,483 to 4,549 m/s and did not vary significantly within the test period. The UVs in the NCRA group continued to decrease over time and were significantly lower than those of the other groups after 7 days. On the other hand, the velocities of the CR and CRA groups increased. In addition, there were no significant differences in UV after 28 days from the initial value before erosion between the CR and CRA groups. The UV changes in the CR and CRA groups are thought to be due to the influence of the peptide.

SEM Observations

Representative SEM images of specimens are shown in Figure 3. After initial treatment, the smear layer on the surface dissolved and etching patterns were observed. For the NCRA group, etching patterns and enamel rods were clearly observed. On the other

hand, the CR and CRA groups had similar morphologic features in that etching patterns were not clearly due to precipitation between the enamel rods (indicated by arrows).

DISCUSSION

This study assessed the potential of the self-assembling peptide P11-4 to prevent acidic erosion. Although we acknowledge that bovine and human enamel are not identical due to genetic, environmental, and dietary differences,²⁴ the samples in this study were prepared from bovine lower incisors as bovine teeth are easier to obtain in large quantities, are in better general condition, and have a more uniform composition when extracted for study purposes than human teeth.²⁵ Another reason for the use of bovine teeth is the larger size, which allows preparation of more samples from the same tooth, resulting in fewer differences between bovine and human enamel, although the mineral distribution characteristics of the resulting lesions were almost indistinguishable.²⁶ Since sonic velocity data were only compared within the same sample and only interpreted within the different groups of the present study, the advantages of using bovine teeth might outbalance the disadvantages of not using human teeth.

Demineralization processes in enamel from erosive tooth wear are difficult to detect by visual inspection at early stages of formation. If the development of such demineralization processes can be identified and evaluated at an early stage, reparative approaches can be applied to halt the erosion process or even repair the affected tooth structure. Current diagnostic techniques are relatively ineffective for quantifying early enamel erosion, which is an important parameter when determining the severity of demineralization and assessing the efficacy of preventive measures.

Most techniques to quantify the elastic modulus of teeth involve destructive contact probing, which renders repetitive measurements unreliable.²⁷ In this regard, a nondestructive technique with the potential for localized evaluation of changes to the mechanical properties of enamel is desirable. Sonic velocity related to the mineral content of the cavity of an enamel lesion has been reported.²⁸ Since the mineralization content of enamel is reported to be closely linked to its stiffness and elasticity,²⁹ during demineralization of the tooth surface, the mineral content of the enamel and the specific sonic velocity should decrease in response to changes in enamel stiffness. The sonic velocity increases with the

volumetric concentration of mineral components and, thus, is a suitable index of the degree of mineralization. Therefore, we were inspired to use an ultrasonic device to detect enamel structure altered by acid erosion. When viewing the plane of the tooth surface, the enamel prisms are at different angles to the direction of acoustic images.³⁰ In this study, the enamel specimens were obtained from the labial surfaces of bovine teeth and the orientation of the specimens was precisely determined to avoid this kind of negative effect on sonic velocity.

The present study supports the forward-looking objective to manage acidic erosive lesions noninvasively by biomimetic remineralization.³¹ The self-assembling peptide P11-4 is a rationally designed small molecule that undergoes hierarchical self-assembly into fibrillar scaffolds in response to specific environmental triggers.³² The peptide undergoes one-dimensional self-assembly, forming micrometer-long nanotapes, preceding the formation of fibrils and edge-to-edge fibers.³³ The design criteria for peptides enabling self-assembly are well understood and have led to the development of a class of self-assembling peptides with a number of candidates showing potential for hard tissue regeneration.³⁴ The surface of fibers of the self-assembling peptide P11-4 has been shown to support hydroxyapatite formation and the remineralization of early carious lesions in an *in vitro* pH-cycling model, resulting in surface remineralization with formation of needle-shaped crystals¹⁵ and increased microhardness after remineralization of subsurface lesions.²⁷ The penetration depth of the P11-4 peptide into subsurface lesions was addressed by confocal microscopy using a mixture of the P11-4 peptide and a fluorescent-labeled fusion peptide.³⁵

The results of this study showed no significant difference in UV between the start of the treatment period and the end of the treatment period in CR and CRA groups. SEM images revealed that after initial treatment, the smear layer on the surface dissolved and etching patterns were observed. For the NCRA group, etching patterns and enamel rods were clearly observed. On the other hand, the CR and CRA groups had similar morphologic appearances in that etching patterns were not clearly due to precipitation between the enamel rods. This precipitation might be related to the formation of the 3D scaffold structure, which, in turn, may play an important role in retaining various dissolved ions as a reservoir.³⁶ That is, the surface is negatively charged and attracts Ca^{2+} and other related ions, which may initiate crystal formation and control the orientation and elongation of the

hydroxyapatite crystals. In the results of this *in vitro* study, it might be concluded that the application of enamel matrix derivatives and self-assembling peptides on erosive lesions can prevent the progress of erosion and encourage remineralization, although further studies are needed to verify this approach under *in vivo* conditions.

CONCLUSION

Within the limitations of this *in vitro* study, application of the self-assembling peptide P11-4 may be able to prevent the progress of erosion from acid challenge and even to promote remineralization.

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Regulatory Statement

This study was conducted in accordance with all the provisions of the local oversight committee guidelines and policies of the Nihon University School of Dentistry.

Conflict of Interest

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

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Nanofilled Resin Composite Properties and Clinical Performance: A Review

H Alzraikat • MF Burrow • GA Maghaireh • NA Taha

Clinical Relevance

Nanocomposites have been found to exhibit properties and clinical performance comparable to those of several hybrid composites but better than microfilled composites. However, there is no long-term evidence yet to show a superior performance that justifies their use in stress-bearing areas.

SUMMARY

The aim of this review was to compile recent evidence related to nanofilled resin composite materials regarding the properties and clinical performance. Special attention was given to mechanical properties, such as strength, hardness, abrasive wear, water sorption, and solubility. The clinical performance of nanocomposite materials compared with hybrid resin composites was also addressed in terms of retention and success rates, marginal adapta-

tion, color match, and surface roughness. A search of English peer-reviewed dental literature (2003-2017) from PubMed and MEDLINE databases was conducted using the terms “nanocomposites” or “nanofilled resin composite” and “clinical evaluation.” The list was screened, and 82 papers that were relevant to the objectives of this work were included in the review. Mechanical properties of nanocomposites are generally comparable to those of hybrid composites but higher than microfilled composites. Nanocomposites presented lower abrasive wear than hybrids but higher sorption values. Their clinical performance was comparable to that of hybrid composites.

*Hanan Alzraikat, PhD, Department of Conservative Dentistry, Jordan University of Science and Technology, Irbid, Jordan

Michael F Burrow, EBM, BDS, MDS, PhD, MEd, MRACD-S(Pros), FRACDS, Faculty of Dentistry, University of Hong Kong, Prince Philip Dental Hospital, Hong Kong

Ghada A Maghaireh, BDS, MS, ABOD, Department of Conservative Dentistry, Jordan University of Science and Technology, Irbid, Jordan

Nessrin A Taha, DCD Endo, Department of Conservative Dentistry, Jordan University of Science and Technology, Irbid, Jordan

*Corresponding author: PO Box 3030, Irbid 22110, Jordan; e-mail: hjsa@just.edu.jo

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INTRODUCTION

Resin composite materials are increasingly used in modern dentistry due to several desirable qualities, such as esthetic appearance and good physical and mechanical properties.¹ The availability of numerous commercial products makes resin composite suitable for use in several clinical applications, including as restorative materials, cavity liners, core buildups, and luting cements, to name a few.²⁻⁴ Previous research has addressed several shortcomings of composites, such as polymerization shrink-

age, strength, and wear resistance.⁵⁻⁹ A wide range of resin composites is available for anterior and posterior restorations. This implies a wide range of organic and inorganic constituents that will influence their clinical handling and performance.¹ The main composition of resin composites consists of an organic polymeric matrix, inorganic fillers, and a silane coupling agent that links the first two components together.¹⁰ Mechanical properties and esthetic appearance of resin composites have been shown to be influenced by their composition and microstructure.^{11,12} Despite the improvements in various properties over recent years, major changes to their composition have involved mainly the fillers rather than the monomer systems, which were originally developed by Bowen¹³ in 1962.

The original resin matrix monomer system was based on the formula presented by Foster and Walker¹⁴ consisting mainly of (Bis-GMA: 2,2-bis[4-(2-hydroxy-3-methacryloxypropoxy) phenyl] propane) and later urethane dimethacrylate. Several approaches were suggested to modify the monomer component to create resin composites with no or minimal shrinkage on polymerization and improved wear resistance. One of the modifications in the monomer system was the use of ring opening monomers. These monomers resulted in the development of resin composites with decreased polymerization shrinkage (ie, silorane-based resin composites).¹⁵ Organically modified ceramics (ormocers) were introduced to overcome problems of polymerization shrinkage associated with conventional methacrylate-based resin composites. Ormocers contain inorganic-organic copolymers in addition to inorganic filler particles.¹⁶ Ormocers have shown lower wear rates compared to other composites¹⁷ and similar shrinkage to hybrid composites despite their lower filler content.¹⁸

It is well documented that mechanical properties of resin composites are significantly influenced by the filler particle morphology (shape), size range, and volume content.^{11,19,20} The increasing demand for esthetic dentistry has led to the development of resin composites used for direct restorations. These composites have demonstrated improved clinical performance both physically and esthetically.²¹ Traditional composites have been classified based on filler size. The classification divided the composites into macrofilled, microfilled, hybrid, and microhybrid materials. The introduction of nanometer-sized particles has been one of the latest developments in the field and is thought to offer superior

esthetics and polishability in addition to excellent wear resistance and strength.^{21,22}

In his review of resin composites, Ferracane² described the chronological development of resin composites outlining their classification according to the filler particle size as follows: macrofill (10 to 50 μm), microfill (40 to 50 nm), and hybrid (10 to 50 μm +40 nm). Hybrid composites were further distinguished as "midifill resin composites" with an average particle size slightly greater than 1 μm and a portion of the 40 nm fillers. Further refinement of the filler particles resulted in what is known as microhybrids (0.6 to 1 μm and 40 nm). Finally, nanofilled resin composites (1 to 100 nm) and nanohybrid that is a combination of microhybrid and nanofilled-size particles were introduced.

The growing interest in nanotechnology and its use in resin composites was based on the desire to utilize the ability of nanosized particles to alter the structure of the composite. This in turn may improve mechanical, chemical, and optical properties and develop a resin composite that can perform optimally in all parts of the mouth.^{22,23} Consequently, Mitra and others²² introduced novel nanofillers and then utilized various methacrylate resins and curing technologies to develop nanocomposites. This nanocomposite was subsequently marketed as the Filtek range of restorative materials (3M ESPE, St Paul, MN, USA).

Two classes of resin composites that include nanoscale filler particles in their composition have been introduced, namely, nanofilled and nanohybrid resin composites. While nanofilled composites use nanosized particles throughout the resin matrix,²² nanohybrids include a mixture of nanosized and conventional filler particles.²⁴ It has been previously suggested that the size of the fillers observed for the nanohybrid composites could be a reason to not refer to them as nanostructured materials. This suggestion was based on the fact that microhybrid composites may contain a mixture of similar nanosized particles in combination with larger filler particles.¹ Nanosized fillers can be categorized as either isolated discrete particles, with dimensions of around 5 to 100 nm, or fused aggregates of primary nanoparticles, where the cluster size may exceed 100 nm.²⁵ It has been proposed that finer particles when incorporated into resin composite will lead to less interparticle space, which will provide more protection to the more vulnerable, softer resin matrix. This in turn will result in reduced "plucking" of filler particles from the material surface.^{26,27}

Previous research has focused on testing various properties of resin composites to evaluate their performance in both laboratory and clinical studies. A large number of these studies aimed at comparing nanocomposites with hybrid and microfilled resin composites.²⁸ Therefore, the objective of the present work was to review laboratory studies that were undertaken on the so-called nanocomposites to examine strength, fracture toughness, surface hardness, abrasive wear, water sorption, and solubility. In addition, a review of the clinical performance of dental nanocomposites was undertaken. Three major categories of resin composites, namely, nanocomposites, hybrid, and microfilled composites, were compared for the sake of offering a clear distinction between their performance and properties. A search of English peer-reviewed literature (2003-2017) from PubMed and MEDLINE databases was conducted using the terms “nanocomposites” or “nanofilled resin composite” and “clinical evaluation.” The list was screened, and 82 papers that were relevant to the objectives of this work were included.

STRENGTH

The main aim of incorporating nanofillers into resin composites (ie, nanocomposites) is to create materials that can be used to restore both anterior and posterior teeth with a high initial polish and gloss. In addition, they should exhibit mechanical strength suitable for use in high-stress-bearing areas.²² Table 1 summarizes the main resin composites mentioned in this literature with their reported classification according to average filler size and the manufacturer.

Flexural Strength

One of the most commonly tested mechanical properties of dental restorative materials is flexural strength (FS), which is considered important for characterizing brittle materials. This type of test generates complex stresses that combine tensile, compressive, and shear stresses when specimens are loaded.²⁹ Several studies examined FS of a number of commercial nanocomposites comparing hybrid and microfilled composites.^{11,13,22,28-36} Direct comparison showed that the FS of nanocomposites was equivalent to or even higher than other composites tested^{11,13,22,29,31,37} with values ranging from 103 to 192 MPa.³⁸⁻⁴⁰ Mitra and others,²² who developed nanocomposite materials in 2003, reported FS values ranging from 153 to 177 MPa. These values were significantly higher than a number of tested hybrid composites, comparable to that of one hybrid mate-

rial and significantly higher than the microfilled composite (Table 2). Similarly, Pontes and others²⁹ reported significantly higher FS of a nanocomposite compared with a hybrid. On the other hand, several investigators reported FS values of several nanocomposites comparable to or significantly lower than a number of hybrid materials but significantly higher than microfilled composites.^{11,31,36,40-42}

A number of studies examined the influence of several factors, such as light polymerization mode, filler content (weight), and degree of conversion (DC) (defined as the percentage of reacted aliphatic C=C bonds from the dimethacrylate monomers present in their polymeric matrices),⁴³ on the FS of nanocomposites. Da Silva and others⁴¹ examined the influence of using three different polymerization modes on the FS of a hybrid and a nanocomposite (Table 2). The results showed that the FS of the tested composites was not influenced by varying the light polymerization mode. Furthermore, FS of the hybrid composite was significantly higher than the nanocomposite tested. Similar conclusions were drawn by Pontes and others,²⁹ who reported no significant effect on FS of a nanocomposite when varying polymerization modes. Similarly, Beun and others³¹ and Rodrigues and others⁴² reported no significant influence of varying polymerization modes on FS of nanocomposites and hybrid composites tested. Degree of conversion has been shown to influence mechanical properties of resin composites.⁴⁴ Beun and others³¹ showed a lower DC when using an LED curing unit for a 10 seconds of curing time compared with a QTH curing unit at a 2-mm depth. The 2-mm thickness is traditionally used and recommended by the ISO standard (4049) for dental resins.⁴⁵ However, in their study, Beun and others³¹ reported that FS was not a discriminating factor used to differentiate the tested composites since the nanocomposites showed comparable FS values to the universal hybrids. Similar conclusions were drawn by Rodrigues and others⁴² regarding the effect of DC on FS. They reported comparable FS values of composites tested that were attributed to the high filler loading of both composites (Table 2).

The influence of filler content on FS of nanocomposites has also been examined comprehensively.^{11,29,31,40,46,47} Rodrigues Junior and others¹¹ showed that there was a positive correlation between the filler weight (FW) and FS of a nanocomposite (FW 84%). The nanocomposite showed intermediate strength values compared to other hybrid composites (FW 74% to 80%) and microfilled

Table 1: Summary of Resin Composites Reviewed, Category, and Manufacturer Information

Resin Composite	Classification	Manufacturer
Filtek Supreme	Nanocomposite	3M ESPE, St Paul, MN, USA
Filtek supreme Z350	Nanocomposite	3M ESPE
Filtek Ultimate	Nanocomposite	3M ESPE
Filtek Supreme Plus	Nanocomposite	3M ESPE
Grandio	Nanocomposite	Voco, Cuxhaven, Germany
Grandio Flow	Nanocomposite	Voco
Esthet X improved	Nanocomposite	Dentsply Caulk, Milford, DE, USA
Premise	Nanocomposite	Kerr, Orange, CA, USA
TPH ³	Nanocomposite	Dentsply Caulk
Concept Advance	Nanocomposite	Vigodent, Rio de Janeiro, Brazil
Ceram X	Nanocomposite	Dentsply Caulk
Tetric EvoCeram	Nanocomposite	Ivoclar Vivadent, Amherst, NY, USA
Venus Diamond	Nanocomposite	Heraeus Kulzer, GmbH, Hanau, Germany
Charisma Diamond	Nanocomposite	Heraeus Kulzer
Ceram X mono	Nanocomposite	Dentsply Caulk
Ceram X Due	Nanocomposite	Dentsply Caulk
Clearfil Majesty	Nanocomposite	Kuraray America Inc, Houston, TX, USA
Ice	Nanocomposite	SDI, Bayswater, Australia
Filtek Z250	Hybrid	3M ESPE
Filtek P60	Hybrid	3M ESPE
Esthet X	Hybrid	Dentsply Caulk
Point 4	Hybrid	Kerr
Charisma	Hybrid	Heraeus Kulzer
Clearfil AP-X	Hybrid	Kuraray America
Amaris	Hybrid	Voco, Cuxhaven
TPH Spectrum	Hybrid	Dentsply Caulk
Venus	Hybrid	Heraeus Kulzer
Filtek Z100	Hybrid	3M ESPE
Tetric Ceram	Hybrid	Ivoclar Vivadent
Prime-Dent	Hybrid	Prime Dental Manufacturing, Chicago, USA
Heliomolar	Microfilled	Ivoclar Vivadent
Helio Fill	Microfilled	Vigodent
Filtek A110	Microfilled	3M ESPE
Durafil	Microfilled	Heraeus Kulzer
Surefil	Packable/hybrid	Dentsply Caulk
Filtek Silorane	Silorane resin	3M ESPE Dental Products, St. Paul, MN, USA

composite (FW 64%). However, it has been reported that the fracture behavior and the structural reliability seem to not be affected in highly filled composites compared with composites with lower filler content, such as microfilled resin composites.⁴² This is because the volume percent content of the fillers may not be markedly different. A number of investigators indicated that filler content and material category had a significant influence on mechanical properties of resin composites, including nanocomposites.^{28,39,47-49} Higher strength was associated with spherical filler particles,⁴⁷ and the

highest values of FS were observed at a filler volume of 60%. Lin and others³⁸ also suggested higher FS values being associated with higher filler content when testing FS of several nanocomposites; however, in their study, spherical filler particles were not associated with higher strength values. Contrary to the results reported by Lin and others,³⁸ Pontes and others²⁹ reported no positive correlation between filler content and FS, which could be related to the fact that different products were examined in the two previously mentioned studies. Lawson and Burgess⁴⁶ attempted to evaluate the

influence of nanofiller weight percent on mechanical properties of experimental resin composites. Three experimental nanocomposites were formulated with different weight percent filler loads (25%, 50%, and 65%). There was an increase in the FS of all experimental composites up to 50% weight content of the fillers.

Ilie and Hickel²⁸ reported that large variations exist between resin composites within the same category. Flexural strength values ranging from 82 to 125 MPa were reported among 72 commercial composites tested (nanocomposites, hybrid, packable, microfilled, and flowable composites). Comparable mechanical properties were found among a number of hybrid and nanocomposites that were expectantly higher than the flowable composites that have a lesser amount of fillers.²⁸

In conclusion, it would be difficult to predict the performance of a single material based on its type. It is reasonable to conclude that the reported FS of nanocomposites was not superior to that of most hybrid composites but was significantly higher than microfilled composites. Furthermore, it should be noted here that the previously mentioned studies used filler weight percent rather than filler volume percent when comparing different composites. It has been mentioned previously that percent filler content is perhaps best expressed in terms of volume because the mechanical properties of resin composites are dictated mainly by their filler volume fraction.¹⁶

Compressive and Diametral Tensile Strength

Compressive strength (CS) and diametral tensile strength (DTS) have been positively correlated in the literature when routinely testing mechanical properties of restorative materials. In both tests, the samples are subjected to a compressive load along different planes. Subsequently, fractures occur due to a combination of tensile and shear stresses.^{50,51} Mitra and others²² reported CS and DTS values of nanocomposites to be comparable to or higher than the tested hybrid and microfilled composites. The CS and DTS of nanocomposites have also been studied by several investigators who reported variability among nanocomposites when compared with other composites.^{1,13,21,22,29,33,40} Several investigators attributed variability and differences in part to the nanofiller content (wt%).^{21,40}

The study by Lu and others¹³ showed comparable values of CS and DTS of a number of nanocomposites and hybrid composites. Similar trends were observed

by de Moraes and others,¹ Pontes and others,²⁹ and Lien and Vandewalle.³⁶ This was attributed to the presence of large individual filler particles in all of them (Table 3).¹

The CS and DTS of a number of nanocomposites were evaluated compared with hybrid, microfilled, flowable, and ormocer-based composites.²⁸ Large variations were observed in the strength values (CS: 103 to 267 MPa; DTS: 32 to 45 MPa). Nanocomposites displayed the highest DTS values and comparable CS values to hybrid composites. It was previously reported that the strongest influence was for the filler volume on the DTS and that the influence of material category was low and influenced mainly the CS.^{29,52,53} Generally, DTS of nanocomposites is at least as good as that of several hybrid composites and higher than a number of hybrid and packable composites. However, Ilie and Hickel²⁸ reported that having a lower modulus of elasticity makes nanocomposites experience more elastic deformation under functional stresses. Therefore, their clinical success is questionable when used in stress-bearing areas.

Fracture Toughness

Fracture toughness (FT) has been occasionally assessed for nanocomposites in addition to the previously mentioned properties.^{22,33,36,54,55} FT is used for assessing brittle materials in order to identify a material's resistance to fracture. It is also used to assess the amount of energy that is needed to cause the propagation of a crack from a well-defined preexisting crack or notch placed in the tested material.⁵⁴ However, due to the sensitivity of the test to the dimensions of the prepared notch or flaw, the results can vary among studies.⁵⁴ Mitra and others²² developed nanocomposites and compared the seven-day FT with hybrid and microfilled composites. The reported values of the two nanocomposites (standard and translucent) were 1.3 and 1.2 MPa√m, respectively, comparable to the hybrid (1.2 MPa√m) and significantly higher than the microfilled (0.9 MPa√m). However, FT of the translucent nanocomposite was significantly lower than other hybrids tested (1.4 MPa√m) (Table 4). Similarly Ilie and others⁵⁴ reported that FT of a nanocomposite (1.46 MPa√m) was significantly higher than microfilled and flowable composites but lower than most hybrid composites. A number of researchers reported that FT of a nanocomposite was comparable to that of several hybrid composites.^{30,33,55} Improved mechanical properties of nanocomposites have been previously attributed to the

Table 2: Flexural Strength (FS, MPa) of Resin Composites (N, Nanocomposite; H, Hybrid; M, Microfilled)

Study	Material	Category	FS (MPa) ^a	Test Conditions
Mitra and others ²²	Filtek A110	M	94 E	Three-point bending test
	TPH spectrum	H	136 D	
	Point 4	H	136 D	
	Esthet X	H	140 C	
	Filtek Supreme standard	N	153 BC	
	Filtek Z250	H	161 AB	
	Filtek Supreme translucent	N	177 A	
Pontes and others ^{29b}	Charisma	H	112, 126, 113 B	Three-point bending test Curing modes: Conventional QTH (400 mW/cm ² for 40 s) Conventional LED (900 mW/cm ² for 20 s) Ramped LED (5-s exposure, then exposure to 900 mW/cm ² for 15 s)
	Filtek Z350	N	138, 142, 149 A	
Lu and others ¹³	Esthet X	H	125 B	Three-point bending test
	Tetric Ceram	H	134 AB	
	Filtek Supreme	N	140 A	
Rosa and others ⁴⁰	Grandio	N	103 B	Three-point bending test
	Esthet X	H	106 AB	
	Filtek Z350	N	123 A	
Lien and others ^{36c}	Esthet X	H	95 C	Three-point bending test
	Filtek Supreme	N	115 AB	
	Filtek Silorane	H	125 AB	
	Filtek Z250	H	137 A	
Rodrigues Junior and others ¹¹	Helio Fill	M	86 D	Three-point bending test 7-d water storage
	Filtek Supreme	N	119 C	
	Charisma	H	127 C	
	Esthet X	H	145 B	
	Filtek Z250	H	168 A	
Rodrigues Junior and others ⁴²	Filtek Supreme	N	135 A	Three-point bending test
	Filtek Z250	H	140 A	
Beun and others ^{31c}	Durafill VS	M	50 B	Three-point bending test Curing modes: Conventional QTH (650 mW/cm ²) LED (450 mW/cm ² for 10 s)
	Filtek A110	M	70 AB	
	Venus	H	80 AB	
	Grandio flow	N	85 AB	
	Tetric Ceram	H	90 A	
	Grandio	N	110 A	
	Point 4	H	110 A	
	Filtek Supreme	N	115 A	
	Filtek Z100	H	120 A	
da Silva and others ^{41b}	Filtek Supreme	N	173C, 185 BC, 190 BC	Three-point bending test Curing modes: Standard (650 mW/cm ² output intensity (30 s) High intensity (1000 mW/cm ² (20 s) Ramped (100 to 1000 mW/cm ² for 10 s + 1000 mW/cm ² for 10)
	Filtek P60	H	225 A, 209 AB, 221 A	

^a Means with the same letters within each study are not statistically different. For studies with multiple values, one letter was used to indicate significant difference between different materials.

^b Multiple values for each material property indicate the reported values under each test condition in the study.

^c Approximate values are used since data were reported using a chart presentation.

Table 3: Compressive Strength (CS) and Diametral Tensile Strength (DTS, MPa) of Resin Composites (N, Nanocomposite; H, Hybrid; M, Microfilled)

Study	Material	Category	CS (MPa) ^a	DTS (MPa)	Test Conditions
Mitra and others ²²	Filtek A110	M	376 B	52 D	NA
	TPH Spectrum	H	378 B	80 B	
	Esthet X	H	422 AB	66 C	
	Filtek Supreme standard	N	426 A	80 B	
	Point 4	H	433 A	76 B	
	Filtek Z250	H	454 A	96 A	
	Filtek Supreme Translucent	N	458 A	87 A	
Lien and others ^{36b}	Filtek Silorane	H	250 C	48 A	NA
	Esthet X	H	320 B	52 A	
	Filtek Supreme	N	360 AB	54 A	
	Filtek Z250	H	390 A	58 A	
Rosa and others ⁴⁰	Esthet X	H	173 A	41 B	NA
	Grandio	N	181 A	42 AB	
	Filtek Z350	N	184 A	50 A	
Lu and others ¹³	Filtek Supreme	N	262 A	54 A	NA
	Esthet X	H	263 A	46 B	
	Tetric Ceram	H	263 A	49 AB	
de Moraes and others ¹	Concept Advance	N		38 B	NA
	Premise	N		40 B	
	TPH	N		53 AB	
	Filtek Z250	H		53 AB	
	Grandio	N		54 AB	
	Filtek Supreme XT	N		58 A	
Pontes and others ^{29c}	Charisma	H		50, 50, 50 B	Curing modes: Conventional QTH (400 mW/cm ² for 40 s) Conventional LED (900 mW/cm ² for 20 s) Ramped LED (exposure for 5 s, followed by exposure to 900 mW/cm ² for 15 s)
	Filtek Z350	N		54, 55, 56 A	

Abbreviation: NA, not applicable.

^a Means with the same letters within each study for each property are not statistically different.^b Approximate values are used since data were reported using a chart presentation.^c Multiple values for each material property indicate the reported value under each test condition in the study.

incorporation of nanofillers into the resin matrix. This in turn leads to a reduction in interparticle space and therefore protection of the organic matrix. Furthermore, nanofillers can act as points that may slow the initiation of or even stop crack propagation.⁵⁴ However, the variations in test protocols and sample preparation among studies may lead to different reported values, making direct comparisons difficult. Generally, the previously mentioned studies indicate that FT of nanocomposites was not superior to that of hybrids but higher than microfilled composites. Further studies using comparable methodology are required to facilitate comparison between materials.

HARDNESS

The hardness of commercial resin composites is a property that is closely related to wear resistance and long-term stability of these materials in the oral environment.⁵⁶ Surface hardness has also been used as an indicator for the degree of monomer conversion by using the hardness ratio of the bottom and top surfaces of tested samples.⁵⁷ Previous literature suggests that a composite is “properly” polymerized when the maximum hardness of the bottom surface is $\geq 80\%$ of the hardness value of the top surface.^{13,21} The hardness of composites has been positively correlated with filler volume %⁵⁸ and with filler weight %.^{21,29,31,46}

Table 4: Fracture Toughness (FT, MPa√m) of Resin Composites (N, Nanocomposite; H, Hybrid; M, Microfilled)

Study	Material	Category	FT (MPa√m) ^a	Test Conditions
Mitra and others ²²	Filtek A110	M	0.9 C	Chevron-shaped notched short rod method, 7-d storage
	Filtek Supreme Translucent	N	1.2 B	
	Esthet X	H	1.2 B	
	Point 4	H	1.2 B	
	Filtek Supreme standard	N	1.3 AB	
	Filtek Z250	H	1.4 A	
	TPH Spectrum	H	1.4 A	
Lien and others ^{36b}	Esthet X	H	0.58 A	Single-edge notched beam method, 24-h storage
	Filtek Supreme	N	0.59 A	
	Filtek Z250	H	0.67 A	
	Filtek Silorane	H	0.68 A	
Rodrigues Junior and others ⁴²	Filtek Supreme	N	1.3 B	Notched bar method, 24-h storage
	Filtek Z250	H	1.5 A	
Hamouda and Abd Elkader ³⁰	Prime Dent	H	6.28 A	Sharp notch bar method, 24-h storage
	Filtek Supreme	N	6.54 A	
Thomaidis and others ⁵⁵	Filtek Ultimate	N	1.20 A	Single-edge notched beam method
	Filtek Z250	H	1.43 A	

^a Means with the same letters within each study are not statistically different.

^b Approximate values are used since data were reported using a bar chart.

Various trends have been reported when microhardness of nanocomposites was compared to other types of resin composites (Table 5). Mota and others²¹ reported a wide range of Knoop microhardness values of nanocomposites that were attributed mainly to differences in the filler content (55 to 123 KHN). Beun and others³¹ reported significantly higher hardness values of the nanocomposites compared to most of the hybrid and microfilled composites tested in their study. Several researchers reported higher hardness values for a number of nanocomposites compared to hybrid composites. This was attributed to higher filler content, large and densely packed filler particles, and resin content of the nanocomposite tested (Table 5).^{1,12,29,59} Similarly Lombardini and others⁵⁷, and Poggio and others⁶⁰ reported greater surface microhardness of nanocomposites tested compared with the hybrid composites, a finding that was statistically significant (Table 5). The hardness values were not influenced by varying polymerization mode or time or sample thickness, something that was also reported by other researchers.^{29,61}

On the other hand, the microhardness of a nanocomposite was found to be inferior to that of a hybrid by several researchers who attributed this to the complex nature of the nanocomposites' filler content, larger filler volume, and greater amount of pigment. These proposed factors may lead to light attenuation yielding a decreased degree of polymerization (Table

5).^{36,42,58,62-64} Cao and others⁶⁵ reported significantly lower Vickers hardness (VH) values of the nanocomposite compared with all tested hybrid composites in their study. Each composite showed a distinct performance in terms of hardness and wear that was attributed to the formulation of each material. Comparable microhardness values were reported by da Silva and others⁴¹ for a nanocomposite and a hybrid. However, using high-intensity light yielded the highest microhardness values. A positive correlation between curing method, depth of cure, curing time, and the hardness of nanocomposites were also reported by others.⁶⁶⁻⁶⁸ Similarly, Marchan and others⁶⁹ reported better microhardness values of tested nanocomposites when light cured for 20 seconds using QTH and LED units compared to 10 seconds. The majority of the nanocomposites produced better VH when cured by LED compared with QTH, the reason for which was unclear. One nanocomposite showed higher VH compared to the other nanocomposites due to its higher filler content (Table 5).

The different values of microhardness reported indicate the influence of the specific formulation of each material, ultimately affecting its hardness behavior.^{40,65} Moreover, different study protocols and testing methods may account for this variability in reported values. Consequently, it would be difficult to accurately compare results. Therefore, further investigations using comparable methodolo-

Table 5: Hardness Values of Resin Composites (N, Nanocomposite; H, Hybrid; M, Microfilled)

Study	Material	Category	Hardness ^a		Test Conditions
De Moraes and others ¹	Concept Advance	N	44	E	Knoop hardness
	TPH	N	54	D	
	Premise	N	62	C	
	Filtek Z250	H	69	B	
	Filtek Supreme XT	N	72	B	
	Grandio	N	111	A	
Pontes and others ²⁹	Charisma	H	40	B	Vickers hardness Curing modes: Conventional QTH (400 mW/cm ² for 40 s) Conventional LED (900 mW/cm ² for 20 s) Ramped LED (exposure for 5 s, followed by exposure to 900 mW/cm ² for 15 s)
	Filtek Z350	N	64	A	
			Top	Bottom	
Poggio and others ^{60b}	Amaris	H	41, 46, 45	36,39,39 D	Vickers hardness Curing times and modes: Standard: 1000 mW/cm ² for 20 s Standard: 1000 mW/cm ² for 40 s Soft start: 0 to 1000 mW/cm ² for 5 s + 1000 mW/cm ² for 35 s
	Filtek Silorane	H	51, 50, 51	45,45,47 C	
	Esthet X	H	52, 59, 60	45,55,56 C	
	Ceram X Mono	N	55, 62, 61	44,58,58 C	
	Filtek Supreme XT	N	82, 82, 84	79,79,81 B	
	Grandio	N	104, 105, 103	101,100,100 A	
Lombardini and others ^{57b}	Amaris	H	44, 44	39,36 D	Vickers hardness Depth of cure and curing time: 2 mm and 3 mm for 40 s
	Filtek Silorane	H	51, 51	45,44 C	
	Esthet X	N	55, 52	44,42 C	
	Ceram X Mono	H	57, 57	55,46 C	
	Filtek Supreme XT	N	83, 82	79,75 B	
	Grandio	N	104, 104	101,91 A	
Lien and others ^{36c}	Esthet X	H	41	C	Knoop hardness
	Filtek Silorane	H	44	C	
	Filtek Supreme	N	57	B	
	Filtek Z250	H	63	A	
Rodrigues Junior and others ⁴²	Filtek Supreme	N	57	43 B	Knoop hardness
	Filtek Z250	H	62	55 A	
Beun and others ^{31c}	Durafill VS	M	19	F	Vickers hardness
	Filtek A110	M	38	E	
	Tetric Ceram	H	40	E	
	Venus	H	45	E	
	Point 4	H	50	D	
	Filtek Supreme	N	60	C	
	Grandio flow	N	60	C	
	Grandio	N	98	B	
	Filtek Z100	H	105	A	
da Silva and others ^{41b}	Filtek Supreme	N	99, A 135, B 94	A	Knoop hardness Curing modes: Standard: 650 mW/cm ² output intensity (30 s) High intensity: 1000 mW/cm ² (20 s) Ramped: 100 to 1000 mW/cm ² (10 s) + 1000 mW/cm ² (10 s)
	Filtek P60	H	103, A 141, B 94	A	

Table 5: Continued.

Study	Material	Category	Hardness ^a		Test Conditions		
Kaminedi and others ⁵⁹	Filtek Z250	H	61	B	Vickers hardness		
	Filtek Z350	N	67	A			
Cao and others ⁶⁵	Charisma Diamond	N	54	C	Vickers hardness following abrasive challenge		
	Filtek Z250	H	79	B			
	Surefil	H	79	B			
	Filtek P 60	H	82	B			
	Clearfil AP-X	H	87	A			
Rastelli and others ⁵⁸	TPH ³	N	53	B	Vickers hardness		
	Filtek Supreme	N	72	A		56	B
	Filtek Z250	H	72	A		71	A
Thome and others ^{62b}	Filtek Supreme	N	Top surface Hardness A3.5: 76, 74, 61 A1: 83, 79, 67 B		Vickers hardness Shade: A1, A3.5 Curing distance: 0, 6, 12 mm		
	Filtek Z250	H	A3.5: 90, 76, 71 A1: 98, 81, 68 A				
Suzuki and others ^{12c}	Tetric Evo Ceram	N	35	D	Knoop hardness		
	Venus Diamond	N	45	C			
	Filtek Supreme XT	N	57	B			
	Grandio	N	80	A			
Marchan and others ^{69b}	Clearfil Majesty	N	33, 27, 28	26, 26, 26	D	Vickers hardness Curing mode and time: QTH: 495 mW/cm ² for 20 s LED: 890 mW/cm ² for 20 s LED: 890 mW/cm ² for 10 s	
	Tetric Evo Ceram	N	34, 33, 30	28, 33, 30	D		
	Ice	N	51, 51, 47	43, 45, 43	C		
	Filtek Z350	N	65, 61, 64	64, 59, 54	B		
	Grandio	N	73, 75, 72	70, 70, 66	A		

^a Means with the same s letters within each study are not statistically different. For studies with multiple values, letter was used to indicate significant difference between different materials.

^b Multiple values for each material property indicate the reported value under each test condition in the study.

^c Approximate values are used since data were reported using a bar chart.

gy should be done in order to be able to directly compare results.

ABRASIVE WEAR

Wear has been defined as the gradual removal of material as a result of the interaction between two surfaces moving against each other.⁷⁰ Wear of resin composite has been reported to be dependent on filler loading and size in addition to the formulation of its resin matrix and the adhesion of fillers to the matrix.²⁷

Several studies investigated abrasive wear of nanocomposites compared with hybrid and microfilled composites by measuring specimen thickness using calipers,⁷¹ assessing surface roughness,¹² and measuring weight loss of tested samples following abrasion.³⁰ Table 6 shows abrasive wear values reported by the studies included in the current literature review.

Mitra and others²² examined the wear rate of nanocomposites (standard and translucent) compared with hybrid and microfilled composites using a three-body wear test. The wear rate of the standard nanocomposite was equivalent to a hybrid but significantly lower than the other hybrid and microfilled composites. The translucent nanocomposite demonstrated equivalent wear values to the microfilled composite but was significantly lower than the other hybrid materials. Comparable results were shown by Cao and others,⁶⁵ who reported significantly lower volume loss of the tested nanocomposite compared with hybrid materials. Similarly, Yesil and others⁷² reported comparable wear rate of a nanocomposite to that of a microfilled and a hybrid composite. Hamouda and Abd Elkader³⁰ reported that the nanocomposite tested in their study demonstrated a significantly lower wear value compared to the hybrid composite that was attributed to the higher filler loading and smaller particle size associated with the nanocomposites. Suzuki and

Table 6: Abrasive Wear of Resin Composites (N, Nanocomposite; H, Hybrid; M, Microfilled)

Study	Material	Category	Abrasive Wear ^a	Test Conditions
Mitra and others ^{22b}	Filtek Supreme standard	N	2.1 C	Three-body wear test Wear determined every 39,000 cycles ($\mu\text{m}/39,000$)
	Filtek Z250	H	2.4 C	
	Filtek A110	M	3.0 B	
	Filtek Supreme Translucent	N	3.1 B	
	Point 4	H	3.4 A	
	Esthet X	H	3.5 A	
	TPH Spectrum	H	3.6 A	
Hamouda and Abd Elkader ³⁰	Filtek Supreme	N	25 B	Two-body abrasion, then measuring weight loss (mg)
	Prime Dent	H	74 A	
Cao and others ⁶⁵	Charisma Diamond	N	6 C	Wear volume loss (mm^3)
	Filtek Z250	H	8 B	
	Filtek P60	H	8 B	
	Clearfil AP-X	H	8 B	
	Surefil	H	10 A	
Yesil and others ⁷²	Point 4	H	7 B	Simulated masticatory wear mechanism using human enamel as opposing cusp (μm)
	Heliomolar RO	M	12 AB	
	Filtek Supreme	N	14 AB	
	Premise	N	19 A	
Suzuki and others ^{12b}	Grandio	N	10 C	Tooth brushing abrasion test (μm)
	Venus Diamond	N	80 B	
	Filtek Supreme XT	N	90 B	
	Tetric Evo Ceram	N	600 A	

^a Means with the same letters within each study are not statistically different.^b Approximate values are used since data were reported using a chart presentation.

others¹² submitted several commercial nanocomposites to an abrasion challenge. It was suggested by the authors that the nanocomposite, which displayed the lowest wear rate, behaves more like a hybrid composite, and its low wear was attributed to its densely packed fillers.⁷³

On the other hand, Mayworm and others⁷⁴ investigated abrasive wear of a nanocomposite and a hybrid before and after storage in saliva for 62 days. Greater abrasive wear of the nanocomposite compared with a hybrid was reported. This was attributed to the larger interparticle space of the former leading to larger wear rates.

Several researchers have suggested that wear resistance behavior of composites is material dependent and cannot be predicted from only a material's filler loading or organic matrix composition.⁷⁵ However, the reported results suggest that several commercial nanocomposites have shown wear values that are lower than several hybrid composites. Also, a number of researchers reported comparable wear values between nanocomposites and microfilled composites that have been reported to display the lowest abrasive wear among resin composites.

SORPTION AND SOLUBILITY

Longevity of a restorative material in the oral environment is directly related to its resistance to degradation.⁷⁶ Therefore, the assessment of solubility and salivary sorption is essential to predict material behavior and clinical performance.^{32,77} The influence of polymerization mode, curing times, storage media, and filler content on sorption and solubility have also been investigated.^{1,32,77-79} Table 7 shows reported results from different studies and special testing conditions.

Da Silva and others⁷⁷ reported significantly higher sorption and solubility of a nanocomposite compared with a hybrid composite tested in their study when using two polymerization modes (conventional and ramped) (Table 7). The ramped polymerization mode was associated with a lower DC in both composites. Higher sorption and solubility of the nanocomposite was explained by the greater surface area of the nanofillers. This makes them more prone to ion leaching and hydrolysis of the silane coupling agent, leading to the filler particles becoming detached and lost. Higher values of salivary sorption of the nanocomposite were attributed to water accumula-

Table 7: Sorption and Solubility Values ($\mu\text{g}/\text{mm}^3$) of Resin Composites (N, Nanocomposite; H, Hybrid; M, Microfilled)

Study	Material	Category	Sorption ($\mu\text{g}/\text{mm}^3$) ^a	Solubility ($\mu\text{g}/\text{mm}^3$)	Test Conditions
da Silva and others ^{77b}	Filtek P60	H	6.7, D 7.04 C	0.38, C 0.43 B	Curing modes: Conventional: 850 mW/ cm ² (10 s) Ramped: 100 to 1000 mW/cm ² (10 s and 1000 for 20 s)
	Filtek Supreme	N	7.35, B 8.74 A	0.41, B 0.49 A	
Kumar and Sangi ^{32b}	Filtek Z250	H	17.3, 23.5 B	1.5, 1.1 B	Storage period: 1 wk 13 wk
	Filtek Supreme translucent	N	18.7, 24.9 B	1.1, 1.0 B	
	Filtek Supreme body	N	23.6, 27.4 A	2.3, 3.6 A	
Shin and others ^{82b}	Grandio	N	11.4, 11.3 F	3.1, 0.8 E	Curing modes: DPSS laser: 400 mW/cm ² Conventional: 800 mW/ cm ²
	Ceram X	N	15.3, 15.4 E	4.6, 0.9 D	
	Tetric Ceram	H	16.6, 15.7 D	5.2, 1.2 C	
	Filtek P60	H	17.4, 18.1 C	2.4, -1.4 F	
	Premise	N	20.7, 21.0 B	6.6, 1.6 A	
	Filtek Z350	N	24.1, 22.8 A	6.3, 1.0 B	
De Moraes and others ¹	Grandio	N	15.1 B	3.1 A	7-d storage in water
	Concept Advanced	N	17.3 B	3.1 A	
	Premise	N	18.1 AB	5.1 A	
	TPH ³	N	26.4 A	2.9 A	
	Filtek Supreme XT	N	29.1 A	2.9 A	
	Filtek Z 250	H	30.7 A	1.9 A	
Almeida and others ^{78b}	Filtek P60	H	2.8, 8.9, 7.3, 4.0 B	2.4, 6.4, 3.4, 2.4 A	Storage media: Artificial saliva Listerine Plax fresh mint Plax
	Filtek Z350	N	14.1, 20.4, 11.9, 14.8 A	3.1, 6.2, 4.4, 3.0 A	

^a Means with the same letters within each study for each property are not statistically different. For studies with multiple values, one letter was used to indicate significant difference between different materials.

^b Multiple values for each material property indicate the reported value under each test condition in the study.

tion at the filler–matrix interface and inside the aggregates of the nanocomposite. Similarly, Kumar and Sangi³² reported significantly higher water sorption and solubility for one nanocomposite compared with the other nanocomposite and a hybrid following 13 weeks of water storage. Furthermore, lower strength values were reported for the nanocomposite that showed the highest sorption and solubility values. The lower strength values of the nanocomposite were attributed to the poor silane penetration of the porous nanoclusters. This made the nanocomposite susceptible to degradation when stored in water. Lower sorption by the hybrid was attributed to the better coupling between filler content and matrix.⁸⁰ On the other hand, Lopes and others⁸¹ demonstrated no influence of varying polymerization mode on sorption and solubility of a nanocomposite. This was attributed to the formation of a densely cross-linked polymer network due to the use of an adequate energy density in all the curing methods used. Similarly, Shin and others⁸² reported

no effect on sorption values of the tested composites when varying polymerization mode. One nanocomposite displayed the lowest sorption and second-lowest solubility compared to a hybrid composite. This was attributed to its high and dense filler content. Similarly, de Moraes and others¹ reported significantly lower sorption of a nanocomposite compared to a hybrid and other nanocomposites tested, while all tested composites displayed comparable solubility values. The authors suggest that results of water sorption and solubility are probably related to the nature of the organic matrix chemical rather than to the filler content of the material (Table 7).

The effect of using different storage media on sorption and solubility of resin composites was assessed by several researchers.^{78,81,83} Almeida and others⁷⁸ and Lopes and others⁸¹ demonstrated an influence of storage media on the sorption of resin composites tested. Negative values of solubility were

reported by researchers for a number of nanocomposites indicating weight gain masking the real solubility. Almeida and others⁷⁸ reported significantly higher sorption of a nanocomposite compared with a hybrid in Listerine (Warner Lambert Health Care, Eastleigh, UK), Plax fresh (Colgate-Palmolive, Guilford, UK), Plax (Colgate-Palmolive), and artificial saliva. The solubility of the nanocomposite and hybrid composites was comparable, with significantly higher values obtained when placed in Listerine and Plax fresh. Similar results regarding water sorption were shown by Curtis and others,⁸⁴ who investigated these properties after different storage periods of a nanocomposite and hybrid composite. It was suggested that the higher water sorption was related to the larger ratio of surface area to volume of the silica nanofillers and the hydrophilic nature of the polymeric matrix.⁸⁵

Goncalves and others⁷⁹ assessed sorption and solubility of a nanocomposite and a hybrid and DC in simulated deep proximal cavities. This was done to investigate composite behavior in a situation similar to a clinical setting. Sorption and solubility were assessed for every 1-mm increment of the 5-mm-deep restoration in three immersion media: distilled water, artificial saliva, and lactic acid. The nanocomposite displayed a lower DC and significantly higher sorption and solubility values than the hybrid. Regardless of media type, the immersion of both resin composites presented an increase in solubility and sorption as a function of depth.

The previously mentioned data generally indicate higher sorption and solubility of nanocomposites. The number of studies investigating sorption and solubility of nanocomposites compared to hybrid and microfilled composites is still limited. Further investigations using test conditions that simulate a clinical setting and compare a wide range of materials are recommended to ascertain the performance of nanocomposites.

CLINICAL STUDIES

The clinical performance of nanocomposites has been investigated in numerous studies and was found to be comparable to that of other resin composites. The majority of these clinical trials used the modified USPHS criteria first described by Cvar and Ryge⁸⁶ and the US Public Health Service's modified Ryge criteria.⁸⁷ These criteria include retention, color match, marginal discoloration, anatomic form, recurrent caries, surface roughness, marginal adaptation, postoperative sensitivity, gross fracture, tooth integrity, gingival health, and proximal contact.

Several clinical studies extending from one to four years reported comparable performance between nanocomposites and hybrid composites in posterior teeth and noncarious cervical cavities.⁸⁸⁻¹⁰² There was no detection of restoration failure, good surface characteristics, good color match, and no postoperative sensitivity.¹⁰³⁻¹⁰⁶ Better polishability and surface gloss retention in favor of the nanocomposites were reported and attributed to the reduced filler plucking and less wear of the nanofillers.¹⁰⁷⁻¹⁰⁹ However, a number of studies reported a certain degree of deterioration in marginal quality over time with minor defects creating surface roughness in all composites tested.^{93,109,110} Türkün and others¹¹¹ reported a high retention rate of the nanocomposite but a better color match of the polyacid-modified composite tested after two years. In a two-year evaluation, it was reported that beyond one year, a negative step occurred due to wear, in addition to staining of the composites tested.⁹⁴ Similarly, Dukic and others¹⁰² reported deterioration of all composites tested after three years with regard to anatomic form, marginal integrity, and marginal discoloration, but these composites were still regarded as being clinically acceptable.

Several long-term clinical performance studies of nanocomposites ranging from five to 10 years have been published. Palaniappane and others^{112,113} evaluated the five-year clinical performance of nanocomposite materials compared to hybrid composites in occlusal and posterior approximal cavities. There was no significant difference in the vertical and volumetric wear between one nanocomposite and a hybrid composite as reported by Palaniappane and others,¹¹² while another nanocomposite material showed lower volume loss compared to the hybrid composites tested by Palaniappane and others¹¹³ after five years. This was explained by the densely packed nanofillers in the nanocomposite that offered protection to the softer resin matrix from the abrasive action of food particles.

Cetin and others¹¹⁵ reported excellent five-year clinical performance of two nanocomposites when compared to two indirect composite materials. No restorations were rated unacceptable in any aspect of the evaluation. A nanocomposite was also compared to a hybrid material after six, eight, and 10 years.^{116,117} The overall success rates of the nanocomposite were 88.1%, 98%, and 80%, respectively, with a comparable performance between the investigated composites.¹¹⁶⁻¹¹⁸ The higher success rate at eight years compared with six years may be due to

the use of different nanocomposite material in each study. Furthermore, the inclusion criteria for recruited subjects differed in that high-carries-risk patients were not excluded from the six-year evaluation period study.¹¹⁶ On the other hand, Frankenberg and others¹¹⁸ included subjects with a high level of oral hygiene, which may have contributed to the reported higher success rate after eight years compared with six years. The authors reported no significant difference in the clinical behavior between the tested composite restorations. Furthermore, significant changes over time were found for all criteria evaluated.

On the basis of the results of the previous studies, it seems reasonable to conclude that there is no evidence yet that the nanocomposites perform clinically better than hybrid composites.

CONCLUSION

The current review of the published literature has shown that commercially available nanocomposite materials do not hold any significant advantage over hybrid composites in terms of strength and hardness. Furthermore, higher sorption and solubility values were found for nanocomposites compared with hybrid composites, and these might influence their clinical performance. On the other hand, the incorporation of nanofillers into resin composite materials was associated with lower abrasive wear of nanocomposites. However, attention should be focused on the resin matrix composition and not only the filler system to be able to assess abrasive wear behavior. In the current review, nanocomposites demonstrated acceptable clinical performance compared with hybrid resin composites for review periods ranging from one to 10 years. However, there was no definitive report of the superior performance of nanocomposites in the majority of evaluation criteria used.

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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Do Nanofilled/Nanohybrid Composites Allow for Better Clinical Performance of Direct Restorations Than Traditional Microhybrid Composites? A Systematic Review

D Angerame • M De Biasi

Clinical Relevance

The effectiveness of direct restorations performed with nanofilled/nanohybrid composites was similar to that obtainable with traditional microhybrid composites. The weight of the available evidence supports the free choice in the clinical setting between these two classes of restorative materials.

SUMMARY

This systematic review was carried out to assess the clinical effectiveness of nanofilled and nanohybrid composites used for direct restorations in comparison with microhybrid composites. The guidelines for the preferred reporting items for systematic reviews and meta-analyses were followed. A search of articles published from July 1996 to February 2017 was performed in PubMed, SciVerse Scopus, Latin American and Caribbean Health Sciences, the Scientific Electronic Library Online,

and the Cochrane Library. The present review selected only randomized controlled trials comparing the clinical performance of a nanofilled or nanohybrid composite for direct restorations with that of a microhybrid composite. The research found 201 studies. Twenty-one articles fulfilled the criteria of the present review. However, the included studies were characterized by great methodological diversities. As a general trend, nanofilled and nanohybrid composites were found to be capable of clinical performance, marginal quality, and resistance to wear similar to that of traditional composites without showing improved surface characteristics. The risk of bias of included studies was judged unclear or high. The clinical performance of nanofilled/nanohybrid composites was found to be comparable to that of traditional composites in the posterior area. The data concerning anterior and cervical restorations were insufficient. With regard to the esthetic properties, there is a compelling

Daniele Angerame, MD, DDS, University Clinical Department of Medical, Surgical and Health Sciences, University of Trieste, Trieste, Italy

*Matteo De Biasi, DDS, MS, PhD, University Clinical Department of Medical, Surgical and Health Sciences, University of Trieste, Trieste, Italy

*Corresponding author: Piazza Ospedale 1, Trieste, I-34125 Italy; e-mail: teiodebiasi@gmail.com

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need for studies on anterior teeth in which the operators are kept unaware of the restorative material. Nanofilled/nanohybrid composites seem to be a valid alternative to traditional microhybrid composites, and at the moment, there is low-level evidence attesting a lack of their superiority.

INTRODUCTION

Among the several resin-based materials used for direct dental restorations, manufacturers offer a wide array of composites suitable for anterior and posterior teeth. These materials greatly differ from each other in terms of characteristics of their inorganic filler, which is known to influence the viscosity and handling of the material,¹ as well as its physical properties,^{2,3} hence affecting the clinical performance of the restoration.^{4,5} The composite strength is maximized when a substantial amount of evenly dispersed filler particles is embedded in the resin matrix.⁶ Even if in a manner that lacks consistency in the plethora of dental literature, resin-based composites are usually classified according to their filler characteristics, such as chemical composition, shape, and especially particle size.⁷

By following the general belief that composites with smaller filler particles prevent the wear of the resin matrix and minimize the surface alteration deriving from the particles' detachment,⁸ several new filler formulations have been proposed. Specifically, the evolution of filler has recently turned to the fabrication of nanofilled and nanohybrid composites, which are regarded as the state of the art in terms of filler formulation.⁷ The size of the filler is surely one of the main determining factors for the most clinically relevant surface properties, such as smoothness and gloss.^{9,10}

Despite the endeavor of the manufacturers that produce nanofilled and nanohybrid composites to grant better initial surface smoothness and provide superior gloss retention, doubt still remains as to whether the clinician should prefer these new-generation materials over traditional universal microhybrid composites.¹¹ A systematic review of *in vitro* studies assessing the difference in surface characteristics between composites with nano- or submicron-sized fillers and conventional composites concluded that, currently, there is insufficient evidence attesting the superiority of nanofilled or submicron materials in terms of surface smoothness and gloss.¹¹ However, laboratory investigations are very abundant in the literature, and this inevitably implies huge methodological variability. The com-

parisons among materials or findings of different studies are frequently impeded by differences in the materials being tested, as well as in the qualitative and quantitative assessment methods of surface characteristics. In light of the aforementioned drawbacks, the reliability of the clinical implications deriving from the information gathered in *in vitro* studies might be questionable.

To delineate evidence-based guidelines for the update and the practice of the clinician involved in restorative dentistry,^{12,13} the aim of this systematic review was to assess the effectiveness of nanofilled and nanohybrid composite resins by selecting randomized clinical trials (RCTs) that compare these materials with traditional composite resins in the middle and long term. The primary outcome measure was the annual failure rate (AFR). The secondary outcome measures were the United States Public Health Service (USPHS) criteria for Clinical Evaluation of Restorations scores, the marginal quality, and the resistance to surface wear.

The present review followed the criteria of the Preferred Reporting Items for Systematic Reviews and Meta-Analyses, the PRISMA statement (<http://prisma-statement.org/>).¹⁴

METHODS AND MATERIALS

Search Methods for Identification of Studies

Each phase of the review was carried out by two calibrated reviewers acting independently, who discussed the cases of disagreement to reach a consensual decision.

The inclusion criteria chosen to consider the trials for the present review are RCTs considering patients who received direct tooth restoration with a nanofilled/nanohybrid composite compared with a traditional one. The following databases were searched for relevant studies: PubMed, SciVerse Scopus, Latin American and Caribbean Health Sciences (LILACS), The Scientific Electronic Library Online (SciELO), and the Cochrane Library. Records from July 1996 to February 2017 were included. There was no restriction in terms of language. The details of the database consultation process are reported in Table 1.

Additional manual research of eligible articles was carried out by searching 1) related citations of selected articles via the PubMed dedicated function, 2) the references of the included articles, and 3) the articles published during the past 10 years in the following scientific journals, which were regarded authoritative because of the topics they treat and their impact factor: *Journal of Dental Research*,

Table 1: Research Algorithms Used for Each Electronic Database

Database	Web Address	Algorithm
PubMed	http://www.ncbi.nlm.nih.gov	(((((nanocomposite) OR nanofilled) OR nanohybrid) OR submicron) AND clinical trial
SciVerse Scopus	http://www.scopus.com	(TITLE-ABS-KEY(((nanocomposite) OR (nanofilled) OR (nanohybrid) OR (submicron)) AND (clinical trial)))
LILACS	http://lilacs.bvsalud.org/en	(nanocomposite or nanofilled or nanohybrid or submicron) AND (clinical trial)
SciELO	http://www.scielo.org	(nanocomposite or nanofilled or nanohybrid or submicron) AND (clinical trial)
Cochrane Library	http://www.thecochranelibrary.com	(nanocomposite or nanofilled or nanohybrid or submicron) AND (clinical trial)

Dental Materials, Journal of Dentistry, and Clinical Oral Investigations.

Study Selection

The duplicated records were removed. Then, the two reviewers simultaneously and independently read the title and abstract of the identified articles to select the articles meeting all these criteria:

- Is it an RCT?
- Does it involve the assessment of direct restorations with nanofilled and/or nanohybrid composites?
- Are the failure rate, the USPHS criteria, and the marginal quality or the surface wear evaluated and reported at the end of the follow-up period?

A restoration was deemed a failure according to the criteria adopted in the trials.

To proceed to the screening of eligible articles, the full text was retrieved if all the criteria were met by the article or if the reviewers could not extrapolate sufficient information from the title and abstract.

Data Extraction

The two reviewers independently filled out a previously designed spreadsheet to perform data extraction. From the selected studies, the two reviewers extracted the following information: study design, length of follow-up, restoration type, outcome of interest, type of analysis, characteristics of the sample, operator(s) performing the interventions, field isolation technique, marginal preparation, lining technique, definition of groups and restorative materials, adhesive strategy, polishing protocol, and final recall rate.

For each experimental group, the AFR of the restorations was calculated according to the following formula:

$$AFR = \frac{\text{failed restorations}}{\text{evaluated restorations} \cdot \text{years of follow-up}}$$

Moreover, to combine the data of the included studies and compare the failure rate of traditional

and nanofilled/nanohybrid composites, the normalized failure index (NFI)¹⁵ was calculated according to the formula

$$NFI = \frac{\sum_i^n (AFR_i \cdot \text{evaluated restorations}_i)}{\sum_i^n \text{evaluated restorations}_i}$$

where n is the number of included studies. NFI calculation was performed distinguishing between the following categories: anterior, posterior, or cervical restoration and traditional or nanofilled/nanohybrid composite. If the same research was identified in distinguished papers, the paper with the longest follow-up period was considered for the calculation.

The continuous secondary outcomes were summarized calculating the mean values and 95% confidence intervals.

If the text of the article reported incomplete information about the data of interest, the corresponding author was contacted via e-mail and asked to provide the missing data, as e-mail has been described as the written method that requires the fewer numbers of attempts and the shortest time to obtain unpublished content.¹⁶ To deal with non-replying authors, a reminder was sent after two weeks. In the case of failure to get in touch with the corresponding author, the data were considered not reported.

Quality Assessment

For the quality assessment of included RCTs, the two reviewers made use of the Cochrane risk of bias tool. The following criteria were taken into consideration:

1. Random sequence generation (protection against selection bias)
 - a. Criterion “met”: the method used to generate the allocation sequence is described in sufficient detail to allow an assessment of whether it should produce comparable groups.

- b. Criterion “unclear”: such information is not reported.
- c. Criterion “unmet”: the method used to generate the allocation sequence is not described or inadequate to produce comparable groups.
- 2. Allocation concealment (protection against selection bias)
 - a. Criterion “met”: patients’ recruitment and assignment were randomized, and the researcher recruiting participants was unaware of the allocation sequence, which was concealed before and until assignment.
 - b. Criterion “unclear”: such information is not reported.
 - c. Criterion “unmet”: the allocation schedule was not kept concealed to the researcher recruiting participants.
- 3. Blinding of participants and personnel (protection against performance bias)
 - a. Criterion “met”: the participants and the personnel involved in the study were kept blind; alternatively, the impossibility of blinding was deemed noninfluential to determine bias.
 - b. Criterion “unclear”: such information is not reported.
 - c. Criterion “unmet”: the participants and the personnel involved in the study were not kept blind.
- 4. Blinding of outcome assessment (protection against detection bias)
 - a. Criterion “met”: the researcher assessing the treatment outcomes was kept blind.
 - b. Criterion “unclear”: such information is not reported.
 - c. Criterion “unmet”: the researcher was not blind to the outcomes.
- 5. Incomplete outcome data (protection against attrition bias)
 - a. Criterion “met”: no dropouts or withdrawals took place, and all outcome data are reported. Alternatively, missing outcome data are evenly distributed among groups and missing for similar reasons.
 - b. Criterion “unclear”: such information is not reported.
 - c. Criterion “unmet”: relevant outcome data are not reported and/or missing data are imbalanced in either number or reasons among groups.
- 6. Selective reporting (protection against reporting bias)
 - a. Criterion “met”: the study protocol is available, and all of the primary and secondary outcomes

that are taken into account in the review have been reported in a prespecified way; if the study protocol is not available, the published reports include all expected outcomes.

- b. Criterion “unclear”: such information is not reported.
- c. Criterion “unmet”: not all of the prespecified primary outcomes of the study have been reported; one or more primary outcomes are reported but were not prespecified or are reported using measurements, methods, or subsets of the data that were not prespecified.
- 7. Protection against other bias.
 - a. Criterion “met”: the study appears to be free of other sources of bias.
 - b. Criterion “unclear”: insufficient information to assess whether an identified problem will introduce bias.
 - c. Criterion “unmet”: there is a potential source of bias related to the specific study design used, or the study stopped early due to some data-dependent process or has been claimed to have been fraudulent.

The validity of the studies was established by classifying each one as follows:

- 1. Low risk of bias: all of the criteria met
- 2. Moderate risk of bias: one or more criteria unclear; the others met
- 3. High risk of bias: one or more criteria unmet

Other methodological aspects were taken into consideration and analyzed, namely, the description of sample size calculation (if present) and the clarity of inclusion and exclusion criteria.

RESULTS

Literature Search

The search found 201 studies; the review of the title and abstract caused the exclusion of 173 of them, as shown in Figure 1. Full-text articles were obtained for the remaining 28, which were all in English.

Seven articles were discarded because they did not fulfil the inclusion criteria of the present review. Two studies with the same first author^{17,18} were excluded from the review because the authors assessed direct and indirect restorations but did not make use of a control group with a direct traditional restorative material for the comparison with nanofilled/nanohybrid composites. Two studies were excluded because they did not consider a control group with a microhybrid composite: in the study of Karaman and others,¹⁹ the authors com-

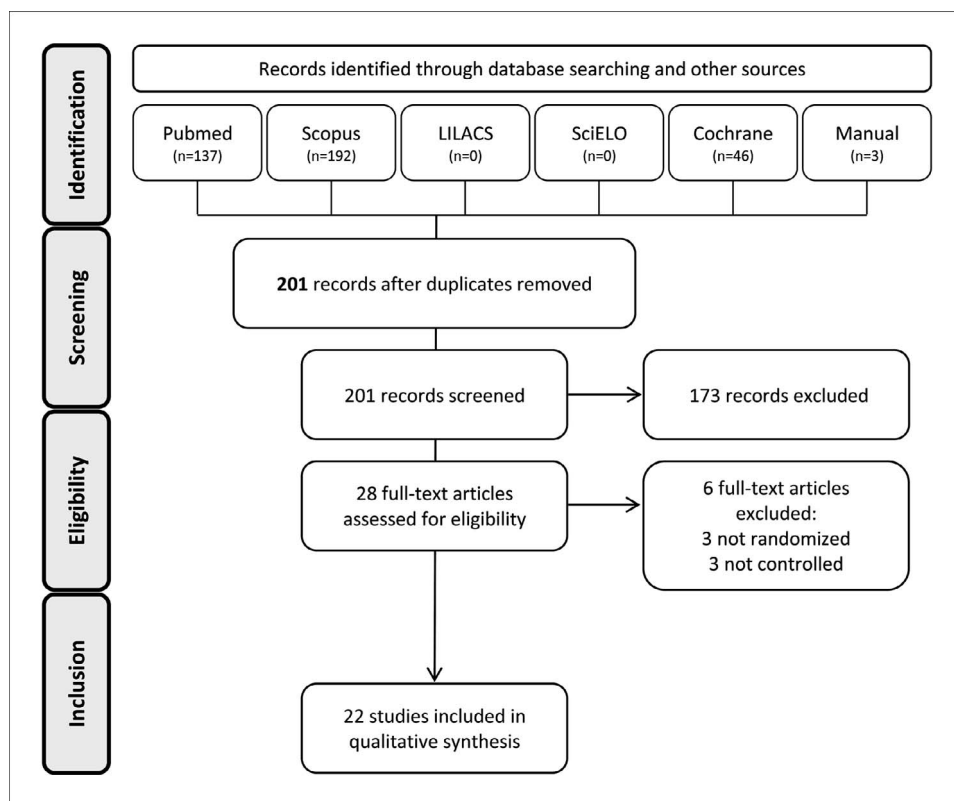


Figure 1. Flow diagram of study inclusion.

pared a nanofilled composite with a flowable nanofilled composite, without further control groups; the study of Türkün and Celik²⁰ used a polyacid modified resin composite (Dyract eXtra, Dentsply, York, PA, USA) as sole control. Three other studies were not randomized.²¹⁻²³

As the selected studies were heterogeneous in terms of several methodological variables and they considered different clinical criteria for analysis, a meta-analysis was not feasible.

Study Characteristics

The data sets arranged in Tables 2, 3, and 4 describe in detail the information obtained from the included studies regarding the primary and secondary outcomes of the present review on anterior, posterior, and cervical restorations, respectively. The oldest study was published in 2006 and the most recent one in 2015. The nationality of the patients involved in the trials and most of the authors were Brazilian (six articles, three trials), German (six articles, two trials), Belgian (four articles, two trials), Swedish (two articles, one trial), Chinese (one article, one trial), Turkish (one article, one trial), and Iranian (one article, one trial). As expected, there was a remarkable variety of materials, techniques, and combination of them across studies. Several of the examined articles

are subsequent reports of the same trial. Despite little differences in the determination of groups, all of the included articles had a split-mouth design.

Evaluation Criteria in the Selected Studies

RCTs appraising the clinical performance of nanofilled or nanohybrid composite restorations as primary objective were the clear majority of included studies.²⁴⁻³² Clinical evaluation of restored teeth was consistently carried out according to one of the modified versions of the USPHS criteria. Given the intrinsic variation among the different versions and the interpretability of the scores, the concept of failure of a restoration was not uniformly shared among the included articles. The attribution of Charlie or Delta scores corresponded to the most common and consistent events that caused a restoration to be classified as failures, such as lack of retention, secondary decay, bulk restoration fracture, restoration fracture with exposed dentin, and pain. Restorations scored as Bravo according to the USPHS criteria were considered functional and, thus, not failed.

In other articles, the researchers performed indirect analyses on positive replicas of the restored teeth. Some of them focused on the assessment of marginal quality via scanning electron microscop-

Table 2: Characteristics of the Included Study on Anterior Restorations							
Author and Year	Population and Follow-up Duration	Outcome of Interest and Type of Analysis	Intervention	Polishing Protocol	Comparison	Conclusions	Quality Assessment
Loguercio, 2007 ²⁹	38 Brazilian adult patients, 114 maxillary anterior teeth followed up for one year	Clinical performance assessed by clinical evaluation and USPHS criteria	Class III restorations Two instructed experienced dentists Rubber dam Bevelled buccal enamel of the cavosurface margins Lining with calcium hydroxide (Dycal, Dentsply) and/or glass ionomer cement (Vitrebond, 3M ESPE, St. Paul, MN, USA)	Sof-Lex Pop-On disks (3M ESPE)	MH: Filtek Z250 (3M ESPE), 38 restorations NF: Filtek Supreme (3M ESPE), 38 restorations Microfilled: Durafill VS (Heraeus Kulzer), 38 restorations AS: Clearfil SE Bond (Kuraray) with or without enamel etching in all groups	Excellent immediate and 12-mo color match of the microhybrid composite resin, which was superior to the nanofilled and microfilled composites tested	No detail of the randomization procedure. Quote: "The resin composite used in each cavity was randomly selected before the beginning of the restorative procedure." It is not specified whether the patients were aware of the materials used for each tooth. The operators performing the restorations were not blind. The outcome data obtained with the two different adhesive protocols (with or without enamel etching) is unclearly and insufficiently reported.
Abbreviations: AS, adhesive system; MH, microhybrid; NF, nanofilled; USPHS, United States Public Health Service.							

py.^{33,34} Another research group took into consideration in different articles the evaluation of the occlusal wear of Class I and II restorations, by three-dimensional laser scanning positive gypsum replicas and observing them with scanning electron microscopy for the analysis of microwear patterns.^{35,36}

Main Findings From the Selected Studies

Table 5 reports the mean AFRs, the failure indices (number of evaluated restorations × AFR), and the NFI calculated from the included studies. The data for all the included studies were combined, distinguishing between anterior, posterior, and cervical restorations. There was only one study²⁹ comparing the clinical performance on anterior teeth of traditional and nanofilled/nanohybrid composites over a period of one year; the NFI for the former was calculated to be 0, with the corresponding value for the latter being 5.26. A larger amount of longer-lasting studies (1 to 10 years of follow-up)^{24-28,31,32,35,36} performed the same comparison in the posterior area. The NFI for traditional composites was equal to 1.20, while that of nanofilled/

nanohybrid composites was 0.91. With respect to cervical restorations of Class V noncarious lesions, only one study was included in the present review³⁰; the NFI for traditional and nanofilled/nanohybrid composites was 0 and 2.68, respectively. A summary of the most relevant findings of each included study is reported below.

The research group of de Andrade and coworkers published four articles^{24,33,37,38} on their 54-month trial, designed to compare the clinical effectiveness of Class I restorations made either with a nanofilled or a nanohybrid composite, using a microhybrid composite control group. Their sample was constituted of 41 adolescent patients in a state of poverty. In synthesis, all of the investigated materials led to acceptable clinical performance, even if the authors reported a trend of better surface smoothness associated with the tested nanofilled composite.

The two-year trial by Arhun and others²⁵ was designed to compare the clinical performance of posterior restorations with a low-shrinkage microhybrid composite with a nanohybrid one in 31 adult patients. The two materials demonstrated similar

and acceptable clinical performance. The authors observed increased surface texture deterioration on the nanohybrid composite restorations.

Dresch and coworkers²⁶ published an article on the comparison among four materials (a nanofilled, a nanohybrid, a packable, and a microhybrid composite) used for Class I and II restorations in 37 dental students. Presenting recall and success rates of 100%, the authors found no difference among materials. Several methodological characteristics of the study raise questions about the reliability of the data in the article, since clarity and rigor were often lacking, especially in the description of the enrollment phase.

In the two-year trial by Ernst and others,²⁷ the clinical performance of a nanofilled composite was compared with that of a microhybrid composite for the restoration of Class II cavities. By comparing the outcome of 112 restorations placed by six different dentists in 50 adult patients, the authors concluded that both restorative materials showed acceptable clinical performance (98% success rate) without observing differences between them.

A German research group presented in five different papers^{28,34,39-41} the findings of a trial investigating the clinical performance of a microhybrid and a nanofilled composite after 2, 4, 6, 8, and 10 years. A private practitioner placed 68 Class II composite restorations in 30 adult patients. At each reevaluation time point, including the last 10-year recall of 29 of the 30 involved patients, there were no differences in the success rate of all restorations between the two tested materials, with an overall success rate equal to 96.9%. The only reported differences in clinical performance between the control microhybrid composite and the nanofilled composite concerned the worse surface smoothness and color match of the latter (Grandio, Voco GmbH, Indian Land, SC, USA).

Loguercio and others²⁹ published the only study presenting outcomes that are of interest in the present review specifically focused on anterior teeth. The authors evaluated the clinical performance of a microhybrid, a nanofilled, and a microfilled composite for the restoration of Class III defects in maxillary anterior teeth. Even if after one year of clinical service high success rates were recorded in all groups (95%-100%), the authors reported better scores for the item "color match" in the microhybrid composite group, compared with the other two.

The research group of Palaniappan and others produced four articles that met the inclusion criteria

of the present review. These two sets of coupled-articles report the findings at subsequent time points of two distinct trials with a similar setup. The first two articles^{35,42} compared the clinical performance and, more specifically, the surface wear of a microhybrid and a nanofilled composite used for the restoration of teeth in the posterior area. Sixteen dental students were involved in the study as patients. The researchers carried out the measurement of surface wear by taking precision impressions of the area of interest of the restored teeth and laser scanning the positive gypsum replicas. The comparisons made after three and five years led to the conclusion that vertical loss in height and volume loss on the restoration surface in the nanofilled group were not significantly different from the microhybrid group. The latter two publications,^{36,43} which were conducted with similar aim and methodology, report the three-year and five-year wear data registered on restorations performed with other materials, namely, a microhybrid, a traditional hybrid, and a nanohybrid. The authors concluded that the wear resistance of the three tested materials complies with American Dental Association specification minimum requirements for posterior composite restorations (vertical loss $<50 \mu\text{m/y}$) and that the nanohybrid composite Tetric EvoCeram showed significantly lower volume loss than the other two materials.

In the study by Qin and others,³⁰ 116 cervical noncarious lesions on anterior and premolar teeth belonging to 46 adult patients were restored either with a microhybrid or a nanofilled composite and followed up for two years. The authors found that the restorations performed with both investigated materials demonstrated acceptable clinical effectiveness in noncarious cervical lesions without significant differences in their clinical performance.

The 18-month trial by Sadeghi and others³¹ compared the clinical performance of Class I restorations received by 35 dental and oral hygiene students. For each patient, a single operator performed one restoration per material type: microhybrid, packable, and nanofilled composite. All materials showed acceptable clinical performance, with 94%-97% success rates; the differences among materials were not significant.

In two different publications reporting the findings of the same trial enrolling 52 patients,^{32,44} van Dijken and Pallesen tested the clinical performance of a microhybrid and a nanohybrid composite used in Class II restorations. This 10-year trial reported a remarkably high recall rate (93%). With a success rate higher

Table 3: *Characteristics of the Included Studies on Posterior Restorations*

Author and Year	Population and Follow-up Duration	Outcome of Interest and Type of Analysis	Intervention	Polishing Protocol
de Andrade, 2014 ²⁴	41 destitute Brazilian adolescent students, 123 permanent molars followed up for 4.5 y	Clinical performance assessed by clinical evaluation and USPHS criteria	Class I restorations One operator "absolute isolation of the operative field" Cavities prepared with carbide burs, no details on margin characteristics Lining with glass ionomer cement (Vitrebond, 3M ESPE) in deep cavities	Multi-bladed bur (FG7714F, KG Sorensen, Cotia, Brazil), rubber cups and points (FlexiCups and FlexiPoints, Cosmedent Inc, Chicago, IL, USA), Enamelize Polishing Paste (Cosmedent Inc), diamond felt disk (FGM Produtos Odontologicos, Joinville, Brazil)
Arhun, 2010 ²⁵	31 Turkish patients, 82 posterior teeth followed up for 2 years	Clinical performance assessed by clinical evaluation and USPHS criteria	Class I and II restorations One clinician of the research team Cotton rolls and saliva ejectors No bevelling Lining with calcium hydroxide (Dycal, Dentsply Caulk) for deep cavities	Fine and super fine diamond points (KG Finishing Kit, Karensen Ltd) and rubber polishing kits (Eveflex Polisher, EVE Ernst Vetter GmbH)
Dresch, 2006 ²⁶	37 Brazilian dental students (42 according to the abstract), 148 permanent molars followed up for one year	Clinical performance assessed by clinical evaluation and USPHS criteria	Class I and II restorations Two calibrated operators Rubber dam Cavities prepared with stainless steel burs, no details on margin characteristics Lining with calcium hydroxide (Dycal, Dentsply) and/or glass ionomer cement (Vitrebond, 3M ESPE)	Fine-grit diamond burs (KG Sorensen) and aluminium oxide polishing paste (Kerr, Orange, CA, USA) in rubber cups on the occlusal surfaces
Ernst, 2006 ²⁷	50 German adult patients, 112 posterior teeth followed up for two years	Clinical performance assessed by clinical evaluation and USPHS criteria	Class II restorations Six experienced dentists placing approximately the same number of restorations Rubber dam Margins, quote: "Occlusal and lateral enamel margins and cervical cementum margins received no bevel preparations, except for cervical enamel margins if enough enamel was left." No lining	Flexible discs (Soflex, 3M ESPE), Enhance polishing tips (Dentsply DeTrey), and polishing brushes (Soflex Brush, 3M ESPE)

Table 3: Extended.

Author and Year	Comparison	Conclusions	Quality Assessment
de Andrade, 2014 ²⁴	MH: Filtek Z250 (3M ESPE), 41 restorations NF: Filtek Z350 (3M ESPE), 41 restorations NH: Esthet-X (Dentsply Caulk), 41 restorations AS: Adper Single Bond 2 (3M ESPE) in all groups	The three tested materials showed similar and acceptable clinical performance in Class I restorations after 12 mo of clinical service.	The methods of the randomization procedure are not described. Unclear allocation concealment. Quote: "To ensure randomness, a drawing was held using sealed envelopes, to establish in which group a certain tooth would be placed." The details of the draw are missing (use of a random sequence, sequential numbered envelopes, assignment procedure, etc). The patients were unaware of the restorative material used for each tooth. The operator performing the restorations was not blinded.
Arhun, 2010 ²⁵	Low-shrinkage MH: Quixfil (Dentsply Caulk), 41 restorations; AS: Xeno III (Dentsply Caulk) NH: Grandio (Voco GmbH), 41 restorations; AS: Futurabond NR (Voco GmbH)	Nanohybrid and low-shrinkage posterior composite restorations demonstrated similar and acceptable clinical performance after two years. Increased surface texture deterioration in nanohybrid composite restorations.	Not properly randomized. Quote: "Interference in the randomization procedure within patients was performed to equally distribute materials into some important variables." Quote: "The distribution of materials and tooth locations were randomly determined by tossing a coin." In trials with relatively small samples, simple randomization often results in an allocation sequence leading to groups that differ, by chance, substantially. The patients were unaware of the restorative material used for each tooth. The operator performing the restorations was not blinded. The authors did not report the reasons for the patients lost to follow-up. The statistical unit of the study is unclear, since some patients participated with more than one couple of restorations.
Dresch, 2006 ²⁶	NF: Filtek Supreme (3M ESPE), 37 restorations; AS: Single Bond (3M ESPE) Packable composite: Pyramid (Bisco), 37 restorations; AS: One Step Plus (BISCO) NH: Esthet-X (Dentsply DeTrey), 37 restorations; AS: Prime & Bond NT (Dentsply DeTrey) MH: Tetric Ceram (Ivoclar Vivadent), 37 restorations; AS: Excite (Ivoclar Vivadent)	Excellent one-year clinical performance and no significant difference among materials	Not properly randomized. Quote: "Interference in the randomization procedure within patients was performed to equally distribute materials into some important variables." The randomization is not meant to be adjusted by the researchers. Quote: "Randomization of the materials was performed on each patient by tossing a coin." In trials with relatively small samples and with more than two groups, simple randomization often results in an allocation sequence leading to groups that differ, by chance, substantially. Not mentioned whether the patients or the operator were aware of the composite type used for each restoration. It is not specified in the materials and methods section how many patients were enrolled, so we have no information on withdrawals/dropouts. The study design is unclear and contradictory: the study enrolled patients requiring at least five Class V restorations, but the authors declare also that 148 restorations were placed in 37 patients (148/37 = 4).
Ernst, 2006 ²⁷	MH: Tetric Ceram (Ivoclar), 56 restorations NF: Filtek Supreme (3M ESPE), 56 restorations AS: Scotchbond 1 (3M ESPE) in all groups	Both restorative materials investigated showed acceptable clinical performance; no significant differences were observed between both types of dental composites.	The allocation sequence was not prepared before the start of the trial. The researchers tossed a coin just before performing each pair of restorations to decide the cavity to start with and then the restorative material. The operators performing the restorations and the patients were not blinded.

Table 3: Continued.

Author and Year	Population and Follow-up Duration	Outcome of Interest and Type of Analysis	Intervention	Polishing Protocol
Krämer, 2015 ²⁸	30 German adult patients, 68 posterior teeth followed up for 10 y	Clinical performance assessed by clinical evaluation and USPHS criteria	Class II restorations One dentist in a private practice Rubber dam Cavities finished with a 25- μ m diamond bur and not bevelled No lining	Super-fine discs (3M ESPE), polishing brushes (Hawe-Neos Dental), and a fluoride varnish (Elmex Fluid)
Palaniappan, 2011 ³⁵	16 Belgian dental student volunteers, 37 molar teeth followed up for five years	Resistance to wear assessed by 3D laser scanning and scanning electron microscopy analysis of positive replicas of restored teeth	Class I and II restorations Two dentists Rubber dam Enamel margins bevelled with diamond-coated bevel tips (Sonic-Sys, KaVo Company, Orange, CA, USA) Lining with glass ionomer cement (Vitrebond, 3M ESPE) to cover preparations closer than 0.5 mm to the pulp	Diamond composite finishing kit (Komet) and Sof-Lex (3M ESPE) finishing and polishing set
Palaniappan, 2012 ³⁶	15 Belgian dental student volunteers, 49 molar teeth followed up for five years	Resistance to wear assessed by 3D laser scanning and scanning electron microscopy analysis of positive replicas of restored teeth	Class I and II restorations Two dentists Rubber dam Enamel margins bevelled with diamond-coated bevel tips (Sonic-Sys, KaVo Company) Lining with glass ionomer cement (Vitrebond, 3M ESPE) to cover preparations closer than 0.5 mm to the pulp	Sof-Lex discs and strips (3M ESPE), polishing kit (Komet, Rock Hill, SC, USA), Prisma gloss paste on polishing cup (Dentsply), and Prisma gloss extra-fine paste on polishing cup (Dentsply).
Sadeghi, 2010 ³¹	35 Iranian dental and oral hygiene students, 105 permanent molars followed up for 1.5 y	Clinical performance assessed by clinical evaluation and USPHS criteria	Class I restorations One operator Cotton rolls No enamel bevel No lining	Microfine diamond finishing burs for contouring and removal of excess restorative material, followed by abrasive aluminium oxide disks

Table 3: Continued. Extended.

Author and Year	Comparison	Conclusions	Quality Assessment
Krämer, 2015 ²⁸	MH: Filtek Z250 (3M ESPE), 32 restorations; AS: Adper Single Bond 2 (3M ESPE) NF: Grandio (Voco GmbH), 36 restorations; AS: Solobond M (Voco GmbH)	After 10 y, Grandio showed worse surface smoothness and color match.	No detail of the randomization procedure is reported. Quote: "fillings to be replaced in different quadrants received at least two different restorations in a random decision" Allocation concealment not mentioned. After contacting the authors, they stated that they made use of envelopes without providing further details. It is not specified whether the patients were aware of the materials used for each tooth. The operator performing the restorations was not blind.
Palaniappan, 2011 ³⁵	MH: Z100 (3M ESPE), 19 restorations NF: Filtek Supreme (3M ESPE), 18 restorations AS: Scotchbond Adhesive (3M ESPE) in all groups	Vertical loss in height and volume loss on the restoration surface in the nanofilled group was not significantly different from the microhybrid group at the five-year recall. Generalized vertical loss (mean; 95% CI): MH, 0.870 $\mu\text{m}/\text{mo}$ [0.830; 0.910]; NF, 0.925 $\mu\text{m}/\text{mo}$ [0.887; 0.963] Generalized volume loss (mean; 95% CI): MH, 0.014 mm^3/mo [0.014; 0.014]; NF, 0.011 mm^3/mo [0.010; 0.011]	The patients were unaware of the materials used for each tooth. The operators performing the restorations were not blind.
Palaniappan, 2012 ³⁶	MH: Gradia Direct Posterior (GC), 16 restorations; AS: UniFil Bond (GC) Traditional hybrid: Tetric Ceram (Ivoclar), 16 restorations; AS: AdheSe (Ivoclar) NH: Tetric EvoCeram (Ivoclar), 17 restorations; AS: AdheSe (Ivoclar)	The wear resistance of the three materials complies with ADA specification of minimum requirements for posterior composite restorations: vertical loss ($<50 \mu\text{m}/\text{y}$). Tetric EvoCeram (NH) showed significantly lower volume loss than the other two materials. Generalized vertical loss (mean; 95% CI): MH, 1.830 $\mu\text{m}/\text{mo}$ [1.777; 1.883]; traditional hybrid, 1.411 $\mu\text{m}/\text{mo}$ [1.364; 1.458]; NH, 1.401 $\mu\text{m}/\text{mo}$ [1.369; 1.433] Generalized volume loss (mean; 95% CI): MH, 0.018 mm^3/mo [0.017; 0.019]; traditional hybrid, 0.017 mm^3/mo [0.016; 0.017]; NH, 0.011 mm^3/mo [0.010; 0.012]	The filling materials were randomized over cavity groups in an unspecified way. After contacting the authors, they stated that they performed a block randomization. No details on allocation concealment. The patients were unaware of the materials used for each tooth. The operators performing the restorations were not blind. It is not specified if the personnel involved in the wear analysis is aware of the materials used for each tooth. After contacting the authors, they stated that the evaluator was kept blind.
Sadeghi, 2010 ³¹	MH: Point 4 (Kerr), 35 restorations Packable composite: Packable Premise (Kerr), 35 restorations NF: Nanofilled Premise (Kerr) AS: OptiBond Solo Plus (Kerr) in all groups	Acceptable clinical performance, no significant difference among materials	No detail of the randomization procedure is reported. Quote: "Three cavities of each patient were randomly restored with three types of light-cured resin composites." No details on allocation concealment. It is not specified whether the operators were aware of the materials used for each tooth. The patients were kept blind. All the restorations are performed in a single increment, but this is usually not advisable except in the case of extremely small cavities filled with low-shrinkage composites.

Table 3: Continued.				
Author and Year	Population and Follow-up Duration	Outcome of Interest and Type of Analysis	Intervention	Polishing Protocol
van Dijken, 2014 ³²	52 Swedish adult patients, 122 posterior teeth followed for 10 y	Clinical performance assessed by clinical evaluation and USPHS criteria	Class II restorations One operator (first author) Cotton rolls and suction device No bevels No lining	Enhance finishing system (Dentsply DeTrey) or brownie points (Shofu Co) and proximal finishing strips
Abbreviations: 3D, three dimensional; ADA, American Dental Association; AS, adhesive system; CI, confidence interval; MH, microhybrid; NF, nanofilled; NH, nanohybrid; USPHS United States Public Health Service.				

than 80% in both groups, the authors concluded that the two materials did not differ in clinical performance.

Risk of Bias Assessment

The item-by-item analysis of the critical points of the quality assessment of included studies according to the Cochrane Quality Assessment tool is reported and justified in Tables 2, 3, and 4. All the included studies showed some flaws, as most of them were judged at high risk of bias and the remaining four at unclear risk of bias, as synthetically depicted in Figures 2 and 3. More specifically, only a few articles^{27,35,36,42,43} properly described an adequate method to generate the allocation sequence, judged capable of producing comparable groups. Further, the issue of allocation concealment has been totally ignored by the included studies, with none of them furnishing information about the procedure for keeping the researcher recruiting participants unaware of the allocation sequence. Also, the risk of performance bias appeared to be relevant in the included studies, because even though the participants involved in the trials were often blind to the restorative material being used, the operative personnel other than the evaluators were never kept blind to the restorative material. The included articles generally fulfilled the criteria to ensure blinding of outcome assessment, selective reporting, and completeness of outcome data, with the exception of two studies, in which a substantial number of dropouts was observed²⁵ or less than one-fourth of enrolled patients were subjected to the analysis.³³ Sporadic minor sources of study-specific methodological biases were identified and are reported in the relevant sections of Tables 2, 3, and 4.

DISCUSSION

The present review demonstrated that there is low-level evidence attesting the absence of differences between the clinical effectiveness of nanofilled/nanohybrid composites and traditional microhybrid composites. Primary and secondary studies that do not provide significant differences between the treatments being compared are often labeled as *negative*, but it is known that a systematic review that does not find evidence of difference is very different from one that finds evidence of no difference.⁴⁵ Indeed, the findings of the present review have some clinical significance, because until a sufficient number of high-quality RCTs are conducted, filling a cavity either with a traditional microhybrid composite or a nanofilled/nanohybrid composite can still be left to the choice of the operator, who can select the material that better matches his or her preferences.

There are, however, some reasons to exercise caution when drawing conclusions from the present review, both in consideration to its primary outcome (AFR) or its secondary outcomes (USPHS scores, marginal quality, and surface wear). It is known that heterogeneity of the data may cause problems when combining the results of a number of studies to provide an overview, for example, with a meta-analysis.¹⁵ Using the NFI is an alternative way to systematically compare results obtained in heterogeneous studies, which weights the sample size and the AFR of the individual studies. Nonetheless, the present review included a reasonable number of studies involving posterior restorations but very few involving cervical and anterior restorations. In these conditions, the mere comparison of NFI values to assess the clinical effectiveness of nanofilled and nanohybrid composite resins can only be indicative in delineating a general trend, until a larger number

Table 3: Continued. Extended.

Author and Year	Comparison	Conclusions	Quality Assessment
van Dijken, 2014 ³²	MH: Tetric Ceram (Ivoclar), 61 restorations NH: Tetric EvoCeram (Ivoclar), 61 restorations AS: Excite (Ivoclar) in all groups	No significant difference between the two tested materials	The restorative material was randomly chosen by casting a coin in a split-mouth design. No details on allocation concealment. The patients were unaware of the restorative material used for each tooth. The operator performing the restorations was not blinded. Blinding of outcome is unclear. Quote: "The restorations were evaluated direct after placement (baseline), six months, and then annually during the following six years by the treating dentist. At different recalls, two calibrated dentists without knowledge of earlier assessments evaluated part of the restorations."

of well-conducted RCTs becomes available and a meta-analysis feasible. Although all the trials that fulfilled the inclusion criteria of the present review reported optimistic findings, with overall success rates ranging from 80% to 100% in relation to the length of the follow-up period regardless of the experimental group, none of them were judged at low risk of bias.

As to the risk of bias assessment, there were some criteria that were never met by the included studies. The random sequence generation or the use of a known random sequence is seldom described or appropriate. The included studies often describe the use of simple randomization procedures achieved

via coin tossing, but this approach is generally not advisable in trials with fewer than 100 subjects per randomized group.⁴⁶ The allocation concealment, which should prevent selection bias in intervention assignment by protecting the allocation sequence before and until assignment and can always be implemented regardless of the study,⁴⁷ was never taken into account in the selected articles. In some articles, a certain tooth is assigned to a designated restorative material by means of a draw of envelopes, but the details of the draw organization and management were not described or retrievable. Moreover, it is known that using envelopes is more susceptible to manipulation than other approach-

Table 4: Characteristics of the Included Study on Cervical Restorations

Author and Year	Population and Follow-up Duration	Outcome of Interest and Type of Analysis	Intervention	Polishing Protocol	Comparison	Conclusions	Quality Assessment
Quin, 2013 ³⁰	46 Chinese adult patients, 116 teeth (not molars) followed up for two years	Clinical performance assessed by clinical evaluation and USPHS criteria	Class V restorations Two experienced dentists Cotton rolls and retraction cords Quote: "The incisal enamel margins of the cervical lesions were bevelled to 1-mm area with a diamond bur at high speed." No lining	Not specified extra-fine diamond point	MH: Clearfil AP-X (Kuraray), 58 restorations; AS: Clearfil SE Bond (Kuraray) NF: Filtek Z350 (3M ESPE), 58 restorations; AS: Adper Prompt (3M ESPE)	Both the Clearfil AP-X and Filtek Z350 restorations demonstrated acceptable clinical effectiveness in noncarious cervical lesions without significant differences in their clinical performance.	No detail of the randomization procedure is reported. Quote: "Each patient received at least one pair of restorations that were randomly allocated." No details on allocation concealment. It is not specified whether the patients or the operators were aware of the materials used for each tooth.

Abbreviations: AS, adhesive system; MH, microhybrid; NF, nanofilled; USPHS, United States Public Health Service.

Table 5: Comparison Between Failure Rate of Restoration With Traditional and Nanofilled/Nanohybrid Composites

Site	Composite Material	Study	Study Duration, y	No. of Evaluated Restorations	No. of Restorations Reported as Having Failed	Mean Annual Failure Rate, %	Failure Index (No. of Restorations × Mean Annual Failure Rate)	Normalized Failure Index
Anterior	Traditional	Loguercio, 2007 ²⁹	1	38	0	0	0	
		Total		38			0	0
	Nanofilled/nanohybrid	Loguercio, 2007 ²⁹	1	38	2	5.3	200.0	
		Total		38			200.0	5.26
Posterior	Traditional	de Andrade, 2014 ²⁴	4.5	31	2	1.4	44.4	
		Arhun, 2010 ²⁵	2	35	2	2.9	100.0	
		Dresch, 2006 ²⁶	1	37	0	0	0	
		Ernst, 2006 ²⁷	2	56	1	0.9	50.0	
		Krämer, 2015 ²⁸	10	32	1	0.3	10.0	
		Palaniappan, 2011 ³⁵	5	19	0	0	0	
		Palaniappan, 2012 ³⁶	5	16	0	0	0	
		Sadeghi, 2010 ³¹	1.5	35	1	1.9	66.7	
		van Dijken, 2014 ³²	10	57	11	1.9	110.0	
		Total		318			381.1	1.20
	Nanofilled/nanohybrid	de Andrade, 2014 ²⁴	4.5	62	3	1.1	66.7	
		Arhun, 2010 ²⁵	2	35	1	1.4	50.0	
		Dresch, 2006 ²⁶	1	74	0	0	0	
		Ernst, 2006 ²⁷	2	56	1	0.9	50.0	
		Krämer, 2015 ²⁸	10	36	1	0.3	10.0	
		Palaniappan, 2011 ³⁵	5	18	0	0	0	
		Palaniappan, 2012 ³⁶	5	17	0	0	0	
		Sadeghi, 2010 ³¹	1.5	35	1	1.9	66.7	
		van Dijken, 2014 ³²	10	57	11	1.9	110	
		Total		390			353.4	0.91
Cervical	Traditional	Qin, 2013 ³⁰	2	58	0	0	0	
		Total		58			0	0
	Nanofilled/nanohybrid	Qin, 2013 ³⁰	2	56	3	2.7	150.0	
		Total		56			150.0	2.68

es.⁴⁸ The last main flaw that threatens the reliability of the findings of the included studies is the risk of performance bias deriving from defective blinding of participants and personnel. Most studies claimed to be “double-blind,” specifically reporting that the patients were unaware of the restorative materials being used on each tooth. Only a few studies did not report this information; however, the blinding of patients is likely to have an impact only on the subjective outcomes (such as postoperative sensitivity) and not on those assessed by the evaluators. What is really noteworthy is that the operator performing the restorations was almost never kept blind to the restorative materials in use; in the other cases, these details were not specified at all, despite the recommendations in the CONSORT Statement to be explicit.⁴⁹ The lack of blinding, in this case, would probably introduce bias, as the operators

placing the restorations could have differentiated their behavior when using different materials, especially whether strong beliefs or prejudices exist among operators. For future investigations, the blinding would be feasible with little effort, for instance by removing the producers’ labels from the bottles and syringes and creating a standard reference color scale for shade choice, by preparing dedicated molds of known dimensions.

There are numerous other sources of variability capable of affecting the results reported in the included studies. In fact, it is known that the material can be a secondary factor for the determination of the prognosis of a restoration.⁵ First, the characteristics of the participants involved in the study are likely to play a major role in determining the success of an adhesive restoration. In the selected studies, the samples varied hugely in terms

of age, culture, social status, wealth, dietary habits, quality of oral hygiene, and so forth; for example, one trial was conducted on Brazilian adolescents living in the suburbs (some of whom were without adequate supply of food),²⁴ another one on German adult patients of a private practitioner,⁴¹ and other ones on dental students.^{26,31} This probably reflects the different aims of the researchers, who wanted to test the performance of the materials in the most controlled conditions or, on the contrary, in the worst possible scenario. It is difficult to comprehend the complex interaction of the multitude of these elements and appraise their relevance since the studies included in the present review involved a relatively small number of patients.

It can be safely assumed that the USPHS criteria are the most widespread and used method to score the performance of tooth-colored restorative materials. One way to deal with ordinal data to produce a meta-analysis is binarization, meaning that some scores were to be considered acceptable and, hence, a clinical success, while the others unacceptable and, thus, restoration failure. This process can be strongly influenced by the arbitrary decision of both authors and reviewers and also because several modifications of the USPHS criteria exist and are further adapted by the authors of primary research. Some versions of the USPHS criteria include the variant of Cvar and Ryge,⁵⁰ the adaptation of Wilson and others,⁵¹ and the color-match modification of Reusens and others.⁵² The use of these multiple versions of the criteria is undesirable because it hinders the summary of the findings of different studies. Even if the evaluators are trained and calibrated, they always make a subjective estimate of the parameters of interest, and there is no guarantee of agreement among evaluators of different trials. This is particularly relevant when the different versions of the scoring system do not share the same amount of rating steps, with some scales contemplating four scores (from Alpha to Delta) and other ones three (from Alpha to Charlie) for the same parameter. Although, at the moment, no better evaluation methods have been proposed to overcome the problems relative to the subjectivity of the appraisal, the reliability of the rating of some items of the evaluation can be easily questioned. Specifically, a substantial difference in opinions is likely to arise when distinguishing among the scores relative to color match and surface roughness; these intrinsically subjective parameters were the most relevant outcomes of interest in the present review. Moreover, there are some methodological details that can

	Random sequence generation (selection bias)	Allocation concealment (selection bias)	Blinding of participants and personnel (performance bias)	Blinding of outcome assessment (detection bias)	Incomplete outcome data (attrition bias)	Selective reporting (reporting bias)	Other bias
Andrade 2012	?	?	-	+	+	+	+
Arhun 2010	-	-	-	+	-	+	-
De Andrade 2011a	?	?	-	+	+	+	+
De Andrade 2011b	?	?	-	+	-	+	+
De Andrade 2014	?	?	-	+	+	+	+
Dresch 2006	-	-	?	+	?	+	-
Ernst 2006	+	-	-	+	+	+	+
Frankenberger 2012	?	?	-	+	+	+	+
Krämer 2009a	?	?	-	+	+	+	+
Krämer 2009b	?	?	-	+	+	+	-
Krämer 2011	?	?	-	+	+	+	+
Krämer 2015	?	?	-	+	+	+	+
Loguercio 2007	?	?	?	+	+	-	+
Palaniappan 2009	+	?	-	+	+	+	+
Palaniappan 2010	+	?	-	+	+	+	?
Palaniappan 2011	+	?	-	+	+	+	+
Palaniappan 2012	+	?	-	+	+	+	+
Qin 2013	?	?	?	+	+	+	+
Sadeghi 2010	?	?	?	+	+	+	-
Turkun 2008	+	-	-	+	+	+	+
van Dijken 2013	?	?	-	?	+	+	+
van Dijken 2014	?	?	-	?	+	+	+

Figure 2. Risk of bias summary: review authors' judgments about each risk of bias item for each included study.

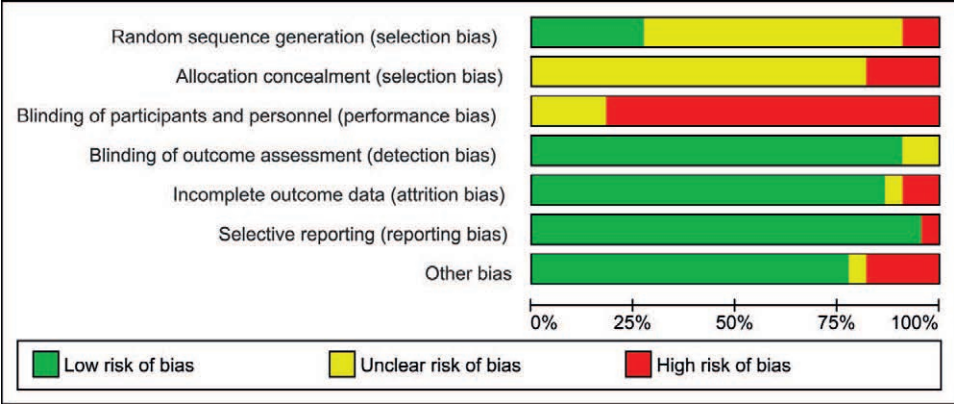


Figure 3. Risk of bias graph: review authors' judgments about each risk of bias item presented as percentages across all included studies.

alter the scores of the USPHS criteria. Examples that support this statement are reported in Table 6.

It is hard to delineate robust evidence in favor of or against the use of nanofilled/nanohybrid composites, also because they belong to a class of materials with numerous commercial products. Furthermore, there is still debate and a certain extent of confusion about the classification of composite resins,⁷ since the distinction between the different classes of materials can be vague and the attribution of a particular composite resin to a single class arduous. Because of the low quality of the evidence found in the present review, it was not possible to carry out any meta-analysis.

One of the limitations of the present review is that it might not have been sensitive enough to locate all

the RCTs published on the clinical performance of nanofilled/nanohybrid composites in comparison to that of microhybrid composites. In fact, it can happen that the words *nanocomposites*, *nanofilled*, *nanohybrid*, or *submicron* do not appear in the title or in the abstract of the article. In the case of trials referring to the materials only with brand names, the probability of the trial to be missed is high; hence, the use of descriptive words that attribute the material to a specific class should be encouraged.

Even if, nowadays, patients are demanding tooth-colored restorations with optimal esthetic properties in the posterior teeth, the most relevant area of the mouth from an esthetic point of view is undoubtedly the anterior area, especially in the maxilla. It is disappointing that a sole trial²⁹ among those that fulfilled the inclusion criteria of the present review was specifically designed to address the issue of the potential benefits of the use of nanofilled composite for Class III restoration of teeth in the esthetic area. The assessment of the hypothetical benefits of nanofilled/nanohybrid materials (ie, possible improved surface luster and prolonged gloss retention) would be particularly useful in this area of the mouth because it is the most esthetically relevant. Nevertheless, the authors reported that the hybrid control composite resin showed an immediate and 12-month color match that was superior to the nanofilled and microfilled composites tested. On the other hand, the nanofilled and microfilled composites obtained the best surface appearance after six months.

CONCLUSIONS

The present review assessed that there are several RCTs attesting that in the posterior area, nanofilled and nanohybrid composites are capable of satisfactory clinical effectiveness, which was similar to that of microhybrid composites. No substantial trend of

Table 6: Factors Other Than the Restorative Material That Could Affect the Evaluation of the Clinical Performance of the Restorations Placed in the Included Studies	
Methodological Item	USPHS Criteria Being Affected
Marginal preparation	Marginal adaptation
	Marginal discoloration
	Color match
	Secondary caries
Field isolation	Secondary caries
	Postoperative sensitivity
Lining	Postoperative sensitivity
Adhesive system	Marginal discoloration
	Color match
	Secondary caries
	Postoperative sensitivity
Polishing protocol	Anatomic form
	Color match
	Surface roughness
	Secondary caries
Abbreviation: USPHS, United States Public Health Service.	

improved surface characteristics, marginal quality, or resistance to wear associated with nanofilled or nanohybrid composites emerged. Data concerning cervical and anterior restorations were extremely scarce.

Considering that the risk of bias was deemed to be unclear or high, the reader should interpret the findings of the present review with caution. The need should be stressed for further well-conducted long-term RCTs comparing nanofilled/nanohybrid composite resins with traditional ones, aiming at decreasing the risk of selection and performance bias.

At this time, the choice of restorative material between nanofilled/nanohybrid and microhybrid composite continues to be up to the clinician performing the restoration.

Disclaimer

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the University of Trieste, in Trieste, Italy.

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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At-home Bleaching With 10% vs More Concentrated Carbamide Peroxide Gels: A Systematic Review and Meta-analysis

JL de Geus • LM Wambier • TF Boing • AD Loguercio • A Reis

Clinical Relevance

Based on the results obtained in this study, similar results are obtained in terms of bleaching effectiveness with different concentrations of carbamide peroxide, but 10% carbamide peroxide produced lower risk and intensity of tooth sensitivity.

SUMMARY

Objective: To perform a systematic review to answer the following research question: Is at-home bleaching in adults with more concentrated carbamide peroxide (CP) gels as effective and safe as bleaching performed with 10% carbamide peroxide gels?

Juliana Larocca de Geus, DDS, MS, PhD, Department of Restorative Dentistry, Paulo Picanço School of Dentistry, Fortaleza, Ceará, Brazil

Letícia Maíra Wambier, DDS, MS, PhD, Department of Dental Materials, State University of Ponta Grossa, Paraná, Brazil

Thaynara Faelly Boing, DDS, MS, PhD, Department of Restorative Dentistry, Guairacá Faculty, Guarapuava, Paraná, Brazil

Alessandro Dourado Loguercio, DDS, MS, PhD, Department of Restorative Dentistry, State University of Ponta Grossa, Paraná, Brazil

*Alessandra Reis, Department of Restorative Dentistry, State University of Ponta Grossa, Paraná, Brazil.

*Corresponding author: Av. General Carlos Cavalcanti, 4748 Ponta Grossa, Paraná, Brazil; e-mail: reis_ale@hotmail.com

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Methods and Materials: A comprehensive search was carried out in the MEDLINE via PubMed, Scopus, Web of Science, LILACS, BBO, Cochrane Library and SIGLE, without restrictions. IADR abstracts (1990 to 2016) and unpublished and ongoing trial registries, dissertations and theses (ProQuest Dissertations and Periodicos Capes Theses Databases) were also searched. The risk of bias of the included studies was analyzed using the Cochrane Risk of Bias tool from the Cochrane Collaboration. We meta-analyzed the data using the random effects model to compare 10% CP and more concentrated CP gels in terms of color change (Δ SGU or Δ E) and risk and intensity of tooth sensitivity (TS). The quality of the evidence was rated using the GRADE approach.

Results: After the database screening, 182 articles remained, and this number was reduced to 17 after examination of the abstracts and/or full texts. Four articles were follow-ups of earlier studies, and thus we collected 13 studies. Ten studies were at unclear risk of bias, while three were at low risk of bias. Lower risk and intensity of TS was observed

for 10% CP. The odds ratio for the risk of TS was 0.41 (95% CI 0.20 to 0.84, $p=0.01$), and the difference in means for TS intensity was 0.44 (95% CI 0.67 to -0.20 , $p=0.0003$). No significant difference was observed in terms of color change in Δ SGU (difference in means 0.29; 95% CI 0.25 to 0.83, $p=0.29$) and for Δ E (difference in means -0.16 ; 95% CI 0.38 to 0.06, $p=0.16$). Except from the Δ SGU, for which the evidence was graded as low quality, the other outcomes were considered at moderate quality.

Conclusions: At-home bleaching with 10% CP showed similar bleaching efficacy with lower risk and intensity of TS in comparison with more concentrated carbamide peroxide gels.

INTRODUCTION

At-home bleaching can be regarded as a popular cosmetic technique for treating dental discoloration since it provides rapid results, employs reduced chair time, and has lower risk of side effects compared to in-office bleaching.¹⁻³

The effectiveness of at-home whitening with 10% carbamide peroxide (CP) has been well reported in the literature.⁴⁻⁷ However, manufacturers have introduced different concentrations of CP (5% to 22%) for at-home bleaching^{8,9} and recommended their use for shorter periods of time.

Due to the continuing release of bleaching gels with different concentrations and protocols, choosing the best product for clinical recommendation is a very challenging task since clinicians should choose a product with similar or superior clinical effectiveness while maintaining patients' safety.

For selection of a bleaching agent, two aspects should be taken into consideration: the whitening efficacy and the risk of side effects. In regard to the former, some clinical studies have shown faster color change for bleaching gels with higher concentrations,¹⁰⁻¹² while other studies did not detect significant differences in groups treated with 10% or more concentrated CP agents.^{4,13} In the same trend, the risk and intensity of tooth sensitivity (TS), which is the most common side effect of bleaching protocols, are shown to be similar^{4,10,13-15} or higher^{5,16-18} for more concentrated CP agents.

Therefore, attempts to reach a consensus to make the choice easier are of clinical interest. Consequently, the aim of this systematic review of the literature was to answer the following PICO question: Is 10% CP gel more effective in terms of color change and

safer in terms of TS than bleaching gels with higher concentrations of CP for at-home bleaching in adults?

METHODS AND MATERIALS

Protocol and Registration

This study protocol was registered at PROSPERO (CRD42016029360), and we followed the recommendations of the PRISMA statement for the report of a systematic review.¹⁹

Information Sources and Search Strategy

The controlled vocabulary (mesh terms) and free key word in the search strategy are in Table 1 and defined based on the following PICOS question reported in the end of the introduction section:

1. Population (P): adult patients submitted to dental bleaching
2. Intervention (I): at-home bleaching with 10% CP
3. Comparison (C): at-home bleaching with more concentrated CP agents
4. Outcome (O): risk and intensity of TS during dental bleaching and color change in shade guide units and in Δ E
5. Study design (S): randomized clinical trials

The outcomes were not used in the search strategy to maximize the sensitivity of the search. To identify trials to be included for this review, we searched the electronic databases MEDLINE via PubMed, Scopus, Web of Science, Latin American and Caribbean Health Sciences Literature database (LILACS), Brazilian Library in Dentistry (BBO), and Cochrane Library. An expert librarian guided the whole search strategy. We hand searched the reference lists of all primary studies for additional relevant publications and the related articles link of each primary study in the PubMed database without restrictions to publication date or languages.

Other sources were also used to identify more articles. We searched the abstracts from the annual conference of the International Association for Dental Research (IADR) and the Brazilian regional division (1990–2016). We explored the gray literature using the database System for Information on Grey literature in Europe (SIGLE). Dissertations and theses were searched for using the ProQuest Dissertations and Theses Full Text database and the Periodicos Capes Theses database.

To locate unpublished and ongoing trials related to the review question, we searched the following

Table 1: *Electronic Database and Search Strategy*

Pubmed (April 14, 2016)	
#1 (tooth discoloration[MeSH Terms] OR "tooth staining"[Title/Abstract] OR "tooth stain"[Title/Abstract] OR "stained tooth"[Title/Abstract] OR "stained teeth"[Title/Abstract] OR "tooth discoloration"[Title/Abstract] OR "tooth discolouration"[Title/Abstract] OR "discolored tooth"[Title/Abstract] OR "discoloured tooth"[Title/Abstract] OR "discolored teeth"[Title/Abstract] OR "discoloured teeth"[Title/Abstract] OR "dental discoloration"[Title/Abstract] OR "dental discolouration"[Title/Abstract])	#2 (peroxides[MeSH Terms] OR tooth bleaching[MeSH Terms] OR bleaching agents[MeSH Terms] OR carbamide peroxide[Supplementary Concept] OR bleaching[Title/Abstract] OR whitening[Title/Abstract] OR "carbamide peroxide"[Title/Abstract] OR "home-use"[Title/Abstract])
#1 AND #2	
Scopus (April 14, 2016)	
#1 (TITLE-ABS-KEY ("tooth discoloration") OR TITLE-ABS-KEY ("tooth staining") OR TITLE-ABS-KEY ("discolored tooth") OR TITLE-ABS-KEY ("stained tooth") OR TITLE-ABS-KEY ("dental discoloration"))	#2 (TITLE-ABS-KEY (peroxides) OR TITLE-ABS-KEY ("carbamide peroxide") OR TITLE-ABS-KEY ("tooth bleaching agent") OR TITLE-ABS-KEY ("dental bleaching") OR TITLE-ABS-KEY ("tooth whitening") OR TITLE-ABS-KEY ("bleaching system") OR TITLE-ABS-KEY ("whitening system") OR TITLE-ABS-KEY ("home-use") OR TITLE-ABS-KEY ("at-home bleaching") OR TITLE-ABS-KEY ("at-home whitening") OR TITLE-ABS-KEY ("home-care bleaching") OR TITLE-ABS-KEY ("home-applied bleaching") OR TITLE-ABS-KEY ("nightguard vital bleaching"))
#1 AND #2	
Web of Science (April 28, 2016)	
#1 Topic: ("tooth discolo*ration") OR Topic: ("discolo*red teeth") OR Topic: ("discolo*red tooth") OR Topic: ("tooth staining") OR Topic: ("stained teeth") OR Topic: ("stained tooth") OR Topic: ("dental discolo*ration")	#2 Topic: (peroxides) OR Topic: ("tooth bleaching") OR Topic: ("bleaching agents") OR Topic: ("tooth bleaching agents") OR Topic: ("carbamide peroxide") OR Topic: ("dental bleaching") OR Topic: ("tooth whitening") OR Topic: ("dental whitening") OR Topic: ("vital bleaching") OR Topic: ("vital whitening") OR Topic: (whitening) OR Topic: (bleaching) OR Topic: ("bleaching techniques") OR Topic: ("bleaching systems") OR Topic: ("whitening systems") OR Topic: ("home use") OR Topic: ("at home bleaching") OR Topic: ("at home whitening") OR Topic: ("home applied bleaching") OR ("home whitening") OR Topic: ("home bleaching") OR Topic: ("night guard vital bleaching") OR Topic: ("home care bleaching")
#1 AND #2	
Lilacs and BBO (April 28, 2016)	
#1 (MH: "tooth discoloration" OR "tooth staining" OR "dicolored tooth" OR "discolored teeth" OR "tooth discolouration" OR "discoloured teeth" OR "discoloured tooth" OR "stained tooth" OR "stained teeth" OR "dental discoloration" OR "descoloração dental" OR "manchamento dental" OR "dentes escuros" OR "escurecimento dental" OR "dientes oscuros" OR "manchas en los dientes" OR "oscurecimiento dental")	#2 (MH: peroxides OR MH: "tooth bleaching agents" OR MH: "tooth bleaching" OR MH: "bleaching agents" OR "carbamide peroxide" OR "peróxido de carbamida" OR "dental bleaching" OR "clareamento dental" OR "blanqueamiento dental" OR "tooth whitening" OR "dental whitening" OR "bleaching systems" OR "whitening systems" OR "sistemas clareadores" OR "vital bleaching" OR "clareamento em dentes vitais" OR "blanqueamiento en dientes vitales" OR "vital whitening" OR "home-use" OR "at home bleaching" OR "at home whitening" OR "clareamento caseiro" OR "blanqueamiento en casa" OR "home-applied bleaching" OR "home whitening" OR "home bleaching" OR "nighguard vital bleaching" OR "night guard vital bleaching" OR "home care bleaching")

clinical trials registries: Current Controlled Trials (<http://www.controlled-trials.com>), International Clinical trials registry platform (<http://apps.who.int/trialsearch>), ClinicalTrials.gov (<http://www.clinicaltrials.gov>), Rebec (<http://www.rebec.gov.br>), and EU Clinical Trials Register (<http://www.clinicaltrialsregister.eu>).

Eligibility Criteria

We included parallel and split-mouth randomized clinical trials (RCTs) that compared the risk and intensity of TS and color change after at-home bleaching with different concentrations of CP in adult patients of any age-group. The efficacy of the

Table 1: Continued.	
#1 AND #2	
Cochrane Library (April 28, 2016)	
#1 MeSH descriptor: [Tooth Discoloration] explode all trees	#7 "dental discolouration":ti,ab,kw (Word variations have been searched)
#2 MeSH descriptor: [Peroxides] explode all trees	#8 "carbamide peroxide":ti,ab,kw or "dental bleaching":ti,ab,kw or "tooth whitening":ti,ab,kw or "bleaching techniques":ti,ab,kw or whitening:ti,ab,kw (Word variations have been searched)
#3 "discoloured tooth":ti,ab,kw or "discoloured teeth":ti,ab,kw or "stained tooth":ti,ab,kw or "stained teeth":ti,ab,kw or "dental discoloration":ti,ab,kw or "dental discolouration":ti,ab,kw (Word variations have been searched)	#9 bleaching:ti,ab,kw or "dental whitening":ti,ab,kw or "bleaching systems":ti,ab,kw or "whitening systems":ti,ab,kw or "vital bleaching":ti,ab,kw (Word variations have been searched)
#3 MeSH descriptor: [Bleaching Agents] explode all trees	#10 "vital whitening":ti,ab,kw or "home-use":ti,ab,kw or "at-home whitening":ti,ab,kw or "at-home bleaching":ti,ab,kw or "home-applied bleaching":ti,ab,kw (Word variations have been searched)
#4 MeSH descriptor: [Tooth Bleaching Agents] explode all trees	#11 "home whitening":ti,ab,kw or "home bleaching":ti,ab,kw or "nightguard vital bleaching":ti,ab,kw or "night-guard vital bleaching":ti,ab,kw or "home-care bleaching":ti,ab,kw (Word variations have been searched)
#5 "tooth staining":ti,ab,kw or "discolored tooth":ti,ab,kw or "tooth discoloration":ti,ab,kw or "discolored teeth":ti,ab,kw or "tooth discolouration":ti,ab,kw (Word variations have been searched)	#12 #1 or #2 or #3 or #4 or #5 or #6 or #7
#6 "discoloured tooth":ti,ab,kw or "discoloured teeth":ti,ab,kw or "stained tooth":ti,ab,kw or "stained teeth":ti,ab,kw or "dental discoloration":ti,ab,kw (Word variations have been searched)	#13 #8 or #9 or #10 or #11
	#14 #12 and #13

bleaching treatment was compared using the ΔSGU (shade guide units) and/or ΔE values.

RCT studies were excluded if studies compared 10% CP with 1) hydrogen peroxide, 2) in-office bleaching, 3) different placebos, 4) whitening tooth-pastes, 5) over-the-counter products, and 6) higher CP concentrations but did not measure any of the outcomes under investigation in this systematic review.

Study Selection and Data Collection Process

Initially, the articles were selected by title and abstracts according to the previously described search strategy. Articles that appeared in more than one database were considered only once. Full-text articles were also obtained when the title and abstract had insufficient information to make a clear decision. Subsequently, three reviewers (JLG, LMW, and TFB) classified those that met the inclusion criteria. To handle such a large number of studies, we used a study ID for each eligible study, combining first author and then year of publication. Data were extracted using customized extraction forms and the following data recorded for each included study:

- Details of the study, including year of publication and author(s)
- Details of study methods, including study design and setting
- Details of participants, including age and gender
- Details of bleaching protocol
- Details of concentration of the bleaching gels

- Details of TS perception using different types of scales
- Details of color evaluation using shade guides and/or spectrophotometers

If there were multiple reports of the same study (ie, reports with different follow-ups), data from all reports were extracted directly into a single data collection form to avoid overlapping data. When data were not reported in the studies, we attempted to contact authors by e-mail at least twice to request the missing information.

When data from multiple bleaching sessions were provided, we made an average of the figures for each bleaching protocol. Concerning color change, we employed the data that represented the immediate result (up to three months postbleaching). When more than one concentrated CP agent was included in the study, their values were combined to make a single entry.

Risk of Bias in Individual Studies

Quality assessments of the selected trials were evaluated by three independent reviewers (JLG, LMW, and TFB) using the Cochrane Collaboration tool for assessing risk of bias in randomized trials.²⁰ The assessment criteria contain six items: sequence generation, allocation concealment, blinding of the outcome assessors, incomplete outcome data, selective outcome reporting, and other possible sources of bias. During data selection and quality assessment, any disagreements between the reviewers were solved through discussion and, if needed, by consulting a fourth reviewer (AR).

For each aspect of the quality assessment, the risk of bias was scored following recommendations as described in the *Cochrane Handbook for Systematic Reviews of Interventions Version 5.1.0* (<http://handbook.cochrane.org>). The judgment for each entry involved recording “yes,” indicating low risk of bias; “no,” indicating high risk of bias; and “unclear,” indicating either lack of information or uncertainty over the potential for bias.

For the outcomes risk, intensity of TS, and color change in shade guide units, studies were at “low” risk of bias if there was adequate sequence generation, allocation concealment, and blinding (key domains). For the objective evaluation of color in ΔE^* , examiner blinding was not considered a key domain, as the foreknowledge of the treatment would not affect the results produced by the instrument tool.

To summarize the risk of bias within a study for each outcome, we followed the directions of the Cochrane Collaboration. An outcome of a study is at low risk of bias when all key domains for that outcome are at low risk of bias. The study was considered at unclear risk when one or more key domain was also unclear, and, finally, the study was at high risk of bias when at least one key domain was at high risk.

Summary Measures and Synthesis of the Results

Data from studies at low or unclear risk of bias were meta-analyzed using Revman 5 (Review Manager version 5, Cochrane Collaboration, Copenhagen, Denmark). Data from eligible studies were either dichotomous (absolute risk of TS) or continuous (intensity of TS, ΔSGU , and ΔE).

The outcomes were summarized by calculating the standardized mean difference for the continuous data and the odds ratio along with the 95% confidence interval (CI) for the dichotomous data. The random effects models were employed. Heterogeneity was assessed using the Cochran Q -test and I^2 statistics. No subgroup analysis was performed. Sensitivity analyses were conducted to investigate the reasons for high heterogeneity whenever detected.

Assessment of the Quality of Evidence Using GRADE

We graded the quality of the evidence for each outcome across studies (body of evidence) using the Grading of Recommendations: Assessment, Develop-

ment, and Evaluation (GRADE) (<http://www.gradeworkinggroup.org>) to determine the overall strength of evidence for each meta-analysis.²¹ The GRADE approach is used to contextualize or justify intervention recommendations with four levels of evidence quality, ranging from high to very low.

The GRADE approach begins with the study design (RCTs or observational studies) and then addresses five reasons (risk of bias, imprecision, inconsistency, indirectness of evidence, and publication bias) to possibly rate down the quality of the evidence (one or two levels) and three to possibly rate up the quality (large effect, management of confounding factors, and dose-response gradient).²¹ Each one of these topics was assessed as “no limitation,” “serious limitations,” and “very serious limitations” to allow categorization of the quality of the evidence for each outcome into high, moderate, low, and very low. “High quality” suggests that we are very confident that the true effect lies close to the estimate of the effect. On the other extreme, “very low quality” suggests that we have very little confidence in the effect estimate and that the estimate reported can be substantially different from what it was measured.

The GRADEpro Guideline Development Tool available online at <http://www.grade-pro.org>) was used to create a summary of finding tables as suggested in the *Cochrane Handbook for Systematic Reviews of Interventions*.²²

RESULTS

Study Selection

After the database screening and removal of duplicates, 1418 studies were identified (Figure 1). After title screening, 182 studies remained, and this number was reduced to 22 after careful examination of the abstracts. After reading the articles, only 17 studies were included in the qualitative analysis.

Characteristics of the Included Articles

The characteristics of the 17 articles selected are listed in Table 2. Four articles were follow-ups of earlier studies, three from Meireles²³⁻²⁵ and one from Matis,¹² totaling 13 studies from a total of 17 articles. The parallel study design was predominantly used in these studies.^{4,5,10,11,15-17,26,27} Four out of the 13 studies used the split-mouth design.^{13,14,18,28}

Three studies used a visual analog scale for pain evaluation,^{10,17,18} and six studies used a numeric rating scale.^{4,5,13,15,27,28} Three studies evaluated

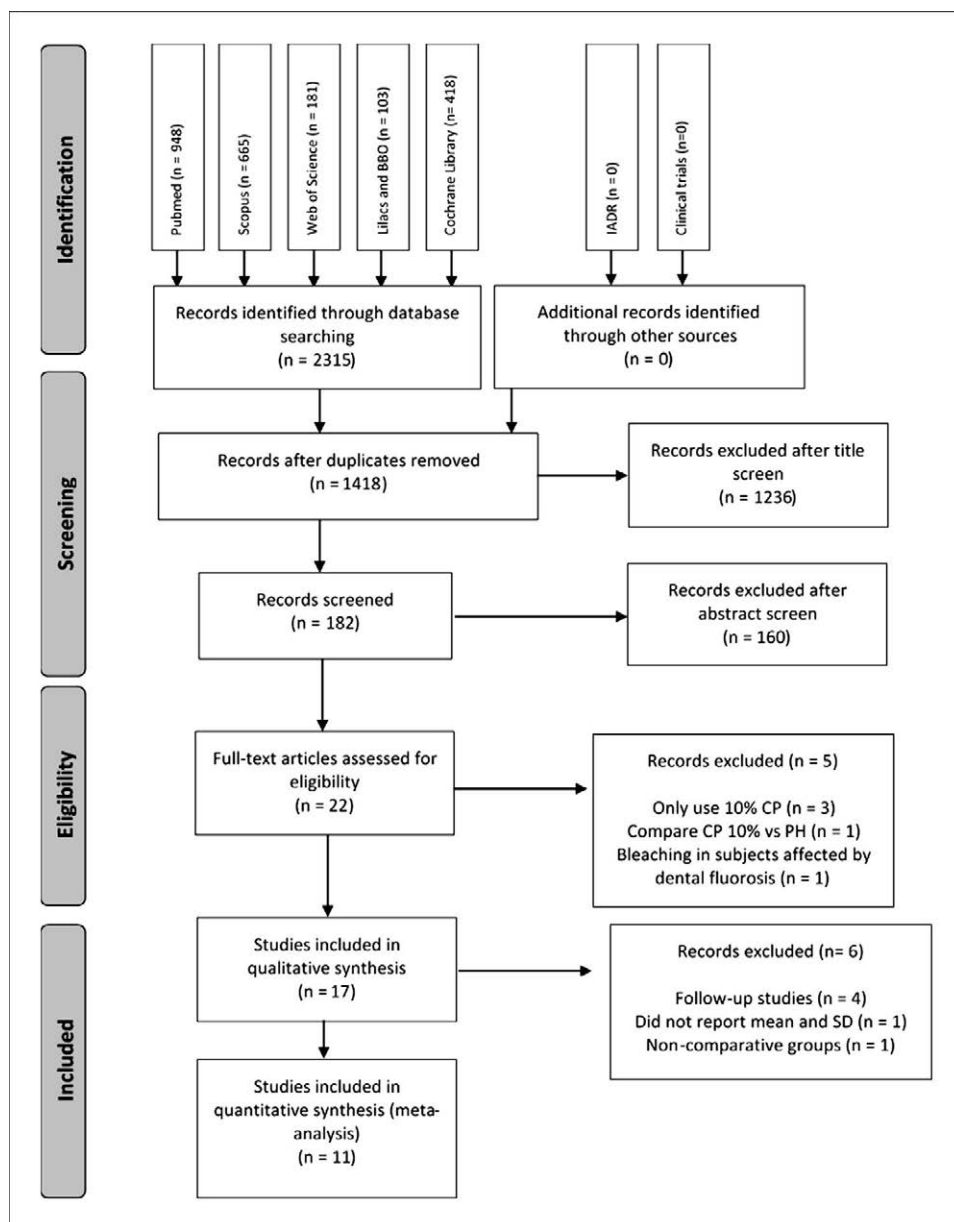


Figure 1. Flow diagram of study identification.

only the risk of TS,^{14,16,26} and one study did not evaluate this outcome.¹¹

For color evaluation, 10 studies used a shade guide.^{4,5,10,11,13,15,17,18,26,28} Eight used an objective instrument (spectrophotometer or colorimeter) for color assessment.^{4,11,13,15,17,18,27,28} Photographs or digital images were used in four studies.^{13,16,27,28} One study did not evaluate the color change.¹⁴

The number of patients per group included in these studies ranged from 10 to 30. The average age of all participants included in the clinical trials was approximately 32.4 years.^{4,13-17} Seven studies did not report this information.^{5,10,11,18,26-28} In one

study, most of the participants were male;¹⁷ in six articles, females predominated.^{4,5,13-16} Six studies did not report this information.^{10,11,18,26-28}

Bleaching Protocol and Features

Bleaching trays with reservoir were used in most of the studies.^{4,11,13,14,16,17,26-28} Three studies used custom-bleaching trays without reservoirs,^{5,15,18} and one study did not report this information.¹⁰

Regarding the bleaching protocol (Table 2), CP with different concentrations, such as 12%,²⁶ 15%,^{10,13,15,16,18,28} 16%,^{4,14,18} 17%,^{11,17} 20%,^{5,16,28} and 28%,²⁷ were used for at-home bleaching.

	Adequate sequence generation?	Allocation concealment?	Examiner blinding?	Incomplete outcome data addressed?	Free of selective reporting?
Basting 2012	+	+	+	+	+
Bernardon et al. 2016	?	?	+	?	+
Braun et al. 2007	?	+	+	?	+
Callan et al. 2008	?	?	+	+	+
de la Peña & Ratón 2013	+	?	?	+	+
Gerlach et al. 2000	?	?	?	+	+
Kihn et al. 2000	?	?	?	+	+
Krause et al. 2008	?	?	+	?	-
Leonard et al. 2002	+	?	?	+	+
Matis et al. 2000	+	+	+	+	+
Matis et al. 2002/2006	?	?	+	-	+
Meireles et al. 2008/2008/2009/2010	+	+	+	+	+
Turkun et al. 2010	?	?	+	+	+

Figure 2. Summary of the risk of bias assessment according to the Cochrane Collaboration tool.

The daily usage time of at-home bleaching gels varied from 20 minutes to overnight and the duration of bleaching in days varied from seven days to six months, but most of the studies performed bleaching for 14 days.^{10,13-16,26} (Table 2).

Assessment of the Risk of Bias

The risk of bias of the included studies is presented in Figure 2. Few full-text studies reported the method of randomization, allocation concealment, and whether the examiner was blinded during color assessment in shade guide units.

In summary, from the 13 studies, ten^{10,11,14-18,26-28} were considered at unclear risk of bias in the key domains of the Cochrane risk of bias tool, and three studies^{4,5,13} were classified at low risk of bias.

Meta-Analyses

All meta-analyses were performed on studies at low and unclear risk of bias and from which the information about the outcome could be extracted. For instance, we could not extract the data from the study of Leonard and others¹⁴ for any of the outcomes. The study of Turkun and others²⁷ compared very different protocols between the two study

groups, being four to six hours daily for 10% carbamide peroxide and only 20 minutes per day for 20% carbamide peroxide. Thus, from 13 studies, only 11 have data to be used at least in one of the outcomes of this study. This explains the variation in the number of studies in the forest plots of the different meta-analyses.

Risk of TS—This analysis was based on nine studies.^{4,5,10,15-18,26,28} The odds ratio was 0.41 (95% CI 0.20 to 0.84; $p=0.01$), which means that two people from the PC 10% will experience the event for every five who will not (Figure 3). Data were not heterogeneous (χ^2 test, $p=0.09$; $I^2=41\%$; Figure 3), which means that all studies included in the analysis share a common effect size.

Intensity of TS—This analysis was based on six studies.^{4,5,10,15,17,28} The standardized difference in means was -0.44 (95% CI -0.67 to -0.20 ; $p=0.0003$). This provides evidence that there is a moderate difference,²⁹ with lower intensity of pain for CP 10% than CP with higher concentrations (Figure 4). Data were not heterogeneous (χ^2 test, $p=0.34$; $I^2=12\%$; Figure 4).

Color Change in Δ SGU—This analysis was based on seven studies.^{4,5,10,11,15,18,26} The standardized difference in means was 0.29, with a confidence interval varying from -0.25 to 0.83 ($p=0.29$). This showed that there was no difference in the color change measured in shade guide units (Figure 5). Data were heterogeneous (χ^2 test, $p<0.00001$; $I^2=85\%$; Figure 5), which means that all studies included in the analysis did not share a common effect size. Through a sensitivity analysis, we did not identify the reason for this high heterogeneity.

*Color Change in ΔE^** —This analysis was based on five studies.^{4,13,15,16,18} The standardized difference in means was -0.16 , with a confidence interval varying from -0.38 to 0.06 ($p=0.16$). These results show that there was no significant difference in the color change measured with a spectrophotometer (Figure 6). Data were not heterogeneous (χ^2 test, $p=0.56$; $I^2=0\%$; Figure 5).

Assessment of the Quality of Evidence—In the summary of findings in Table 3, we can observe that except for the color change in Δ SGU, graded as low in the quality of evidence, the other outcomes were assessed as moderate quality using GRADE. The reasons for downgrading the evidence for Δ SGU were that most RCTs are at “unclear” risk of bias, presence of inconsistency with nonexplained statistical heterogeneity, and imprecision with a high 95% confidence interval, which does not exclude impor-

Table 2: Summary of the Studies Selected for This Systematic Review

Study ID	Study Design [Setting]	Method of Color Assessment	Subjects' Age in Mean $\pm$ SD [Range] (y)	No. of Subjects Male [Total]	Dropouts
Basting (2012) ⁵	Parallel [n.r.]	Shade guide unit (Vita Classical)	n.r. $\pm$ n.r. [18-42]	18 [94]	13
Bernardon and others (2016) ¹⁸	Split-mouth [n.r.]	Shade guide unit (Vita Classical) and spectrophotometer	n.r. $\pm$ n.r. [18-40]	n.r. [50]	n.r.
Braun and others (2007) ¹¹	Parallel [n.r.]	Shade guide unit (Vitapan 3D) and spectrophotometer (Spectroshade)	n.r. $\pm$ n.r. [n.r.]	n.r. [30]	n.r.
Callan and others (2008) ²⁶	Parallel [n.r.]	Shade guide unit (Vita Classical)	n.r. $\pm$ n.r. [n.r.]	n.r. [46]	1
de la Peña & Ratón (2013) ¹⁵	Parallel [University]	Shade guide unit (Vita Classical) and spectrophotometer (Vita Easysshade)	25.9 $\pm$ 5.6 [n.r.]	28 [96]	0
Gerlach and others (2000) ¹⁶	Parallel [n.r.]	Digital images	39 $\pm$ 8.4 [24-57]	6 [36]	4
Kihn and others (2000) ¹⁰	Parallel [University]	Shade guide unit (Vita Lumin)	n.r. $\pm$ n.r. [18-65]	n.r.[57]	4
Krause and others (2008) ¹⁷	Parallel [n.r.]	Shade guide unit (Vitapan 3D) and spectrophotometer (SpectroShade)	31 $\pm$ 4 [n.r.]	16 [30]	n.r.
Leonard and others (2002) ¹⁴	Split mouth [University]	n.r.	23. $\pm$ n.r. [20-30]	0 [20]	1
Matis and others (2000) ¹³	Split-mouth [n.r.]	Shade guide unit (Trubyte bioform), colorimeter (Chroma Meter) and photographic means	50.4 $\pm$ n.r. [26-73]	8 [25]	n.r.
Matis and others (2002, 2006) ^{28, 12}	Split mouth [University]	Shade guide (Vitaescence Esthetic Restorative Masters), photographs and colorimeter (Minolta Chroma Meter)	n.r. $\pm$ n.r. [n.r.]	n.r.[59]	15
Meireles and others (2008, 2008, 2009, 2010) ^{4, 24, 23, 25}	Parallel [n.r.]	Shade guide unit (Vita Classical), and spectrophotometer (Vita Easysshade)	25.3 $\pm$ 7.9 [18-55]	31 [92]	11
Turkun and others (2010) ²⁷	Parallel [n.r.]	Spectrophotometer (Vita Easysshade) and digital images	n.r. $\pm$ n.r. [25-28]	n.r. [20]	0

Abbreviations: CP, carbamide peroxide; delta SGU, shade guide units; delta E (L*, a*, and b*); ID, identification; n.r., not reported; NRS (numerical rating scale): none, mild, moderate, considerate, severe VAS (visual analog scale): a 10-cm horizontal line with words "no pain" at one end and "worst pain" at the opposite end; SD, standard deviation.

^a Opalescence 10%, Ultradent (South Jordan, UT, USA).

^b Opalescence 20%, Ultradent.

^c Power Bleaching 10%, BM4 (Maringá, Brazil).

^d Opalescence 15% (Ultradent).

^e Power Bleaching 16%, BM4.

^f Perfect Bleach 10% (Voco, Cuxhaven, Germany).

^g Perfect Bleach 17%, Voco.

^h NightWhite Classic 10% (Discus Dental, Culver City, CA, USA).

ⁱ Rembrandt Xtra-Comfort 12% (Den-Mat Corporation, Santa Maria, CA, USA).

^j Illuminè Home 15% (Dentsply, Konstanz, Germany).

^k Nupro White Gold 10% (Dentsply).

^l Nupro White Gold 15% (Dentsply).

^m NightWhite Classic 16% (Discus Dental).

ⁿ Whiteness Perfect 10% (FGM, Joinville, SC, Brazil).

^o Whiteness Perfect 16% (FGM).

^p Meta Tray 28% (Remedent, Deurle, Belgium).

Table 2: Extended.

Study ID	Bleaching Tray	Groups/Materials [No. of Subjects]	Bleaching Protocol	Outcomes evaluated		
				Color Change	Tooth Sensitivity (Pain Scale)	Gingival Irritation
Basting (2012) ⁵	Without reservoirs	10% CP ^a [19] 20% CP ^b [21]	2 h daily [21 d]	ΔSGU	Absolute risk and intensity (NRS 0-3)	n.r.
Bernardon and others (2016) ¹⁸	Without reservoirs	10% CP ^a [25] 10% CP ^c [25] 15% CP ^d [25] 16% CP ^e [25]	2 h daily [45 d]	ΔSGU and ΔE*	Intensity (VAS 0-10)	Loe index scores 0-3
Braun and others (2007) ¹¹	With reservoirs	10 % CP ^f [10] 17% CP ^g [10]	2 h daily [7 d]	ΔSGU and ΔE*	n.r.	n.r.
Callan and others (2008) ²⁶	With reservoirs	10% CP ^h [23] 12% CP ⁱ [23]	10%: 2 h twice per day [14 d] 12%: 6-8 h daily [14 d]	ΔSGU	Absolute risk	Number of days
de la Peña & Ratón (2013) ¹⁵	Without reservoirs	10% CP ^a [24] 15% CP ⁱ [24]	1 h daily [14 d]	ΔSGU and ΔE*	Absolute risk and intensity (NRS 0-4)	n.r.
Gerlach and others (2000) ¹⁶	With reservoirs	10% CP ^a [10] 15% CP ^d [11] 20% CP ^b [5]	2 h daily [14 d]	ΔE*	Absolute risk	Absolute risk
Kihn and others (2000) ¹⁰	n.r.	10% CP ^k [28] 15% CP ^l [28]	4 h daily-overnight [14 d]	ΔSGU	Intensity (VAS 0-20)	n.r.
Krause and others (2008) ¹⁷	With reservoirs	10% CP ^l [10] 17% CP ^g [10]	2 h-daily [7 d]	n.r.	Intensity (VAS 0-10)	n.r.
Leonard and others (2002) ¹⁴	With reservoirs	10% CP ^h [20] 16% CP ^m [20]	8-10 h overnight [14 d]	n.r.	Absolute risk	Absolute risk
Matis and others (2000) ¹³	With reservoirs	10% CP ^a [25] 15% CP ^d [25]	Overnight [14 d]	ΔSGU and ΔE*	Intensity (NRS 0-4)	0-4 scale
Matis and others (2002, 2006) ^{28, 12}	With reservoirs	10% CP ^a [30] 15% CP ^d [28] 20% CP ^b [30]	Overnight [6 mo]	ΔE*	Intensity (NRS 0-4)	0-4 scale
Meireles and others (2008, 2008, 2009, 2010) ^{4, 24, 23, 25}	With reservoirs	10% CP ⁿ [46] 16% CP ^o [46]	2 h daily [21 d]	ΔSGU and ΔE*	Absolute risk and intensity (NRS 0-4)	0-4 scale
Turkun and others (2010) ²⁷	With reservoirs	10% CP ^a [10] 28% CP ^p [10]	10%: 6-8 h daily [10 d] 28%: 20 min daily [10 d]	ΔE*	Absolute risk and intensity (NRS 0-4)	Absolute risk

tant harm or benefit (Table 3). For the other outcomes, the evidence was downgraded only for the unclear risk of bias of most RCTs.

DISCUSSION

In the present systematic review, we observed that at-home bleaching with 10% CP produced similar color change and lower risk and intensity of TS than at-home bleaching performed with more concentrated CP concentrations.

From a theoretical point of view, a faster or higher degree of color change was expected to occur with more concentrated CP gels. Chemical theories state that in simplest chemical reactions, an increase in the concentration of reactants may increase the reaction rate. Indeed, a closer look of several primary studies indicated that more concentrated CP products yielded a higher degree of whitening in the first days or week of bleaching,^{4,10,11} but this difference was not maintained at the end of the treatment.

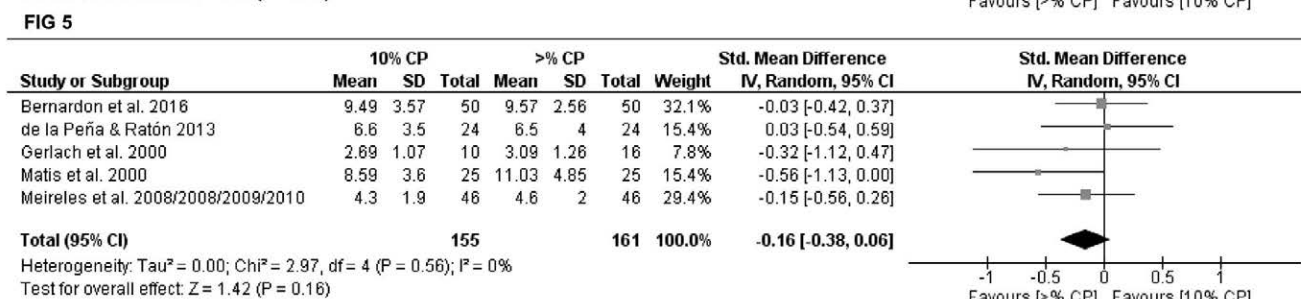
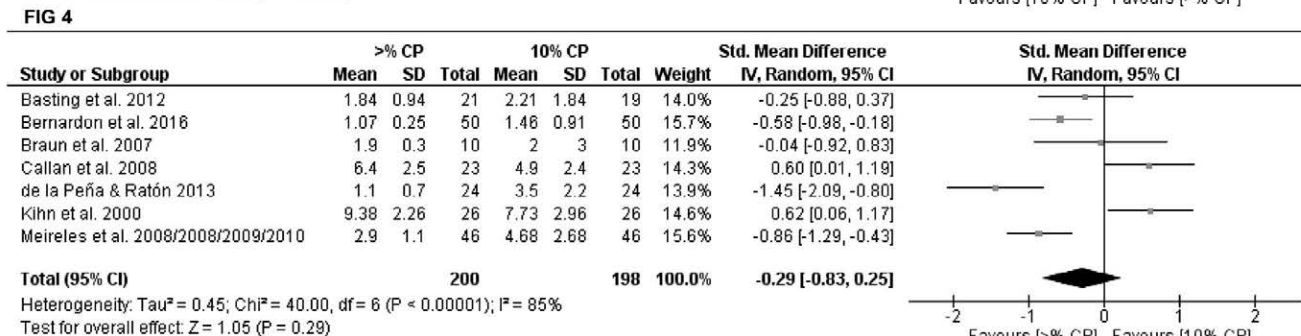
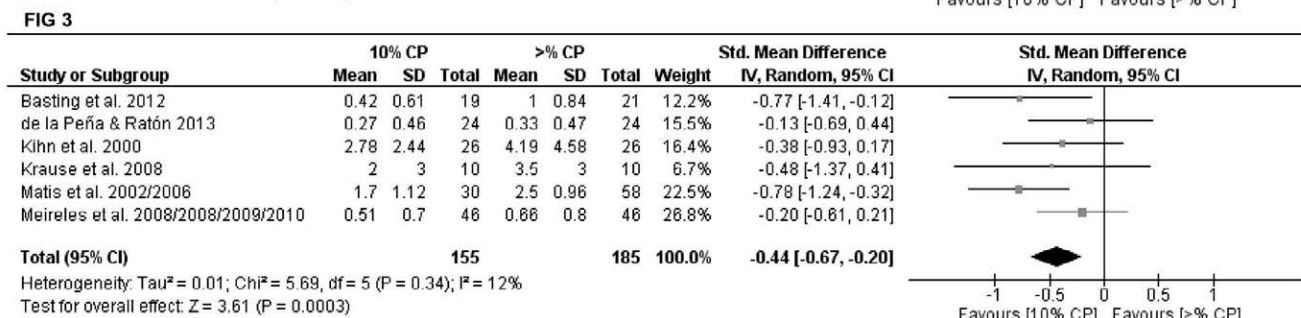
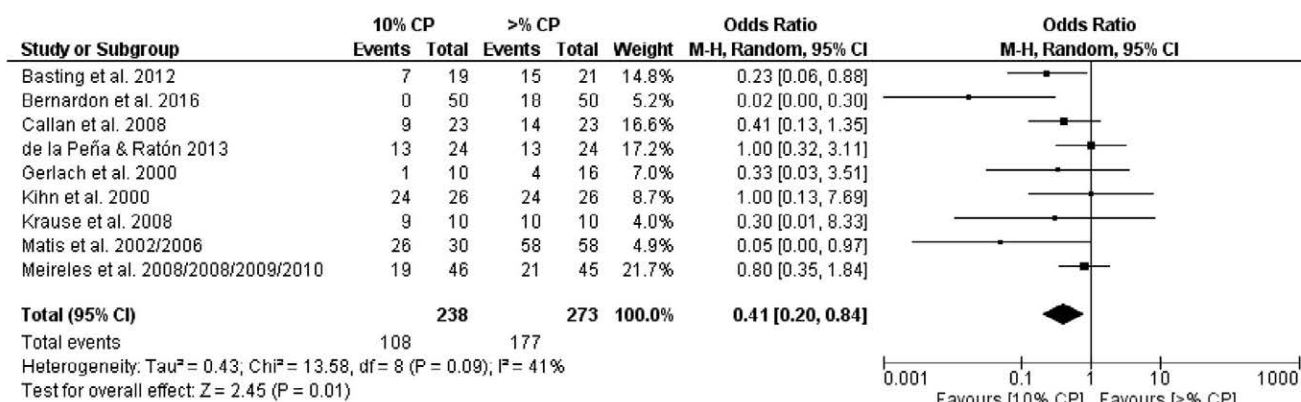


Figure 3. Forest plot of the risk of tooth sensitivity (TS) for 10% CP vs higher-concentration CP (>10% CP).

Figure 4. Forest plot of the intensity of tooth sensitivity for 10% CP vs higher-concentration CP (>10% CP).

Figure 5. Forest plot of the color change in shade guide units for 10% CP vs higher-concentration CP (>10% CP).

Figure 6. Forest plot of the color change in ΔE^* for 10% CP vs higher-concentration CP (>10% CP).

For instance, Matis and others¹³ showed that a 15% CP gel achieved a higher degree of whitening than did the 10% CP gel after two weeks of use. However, by extending the treatment time to six

weeks, the differences in color change or brightness were no longer statistically different. Similarly, Leonard and others¹⁴ concluded that higher CP concentrations achieved faster bleaching, but the

Table 3: Summary of Findings for At-Home Bleaching ^a					
Outcomes	Anticipated Absolute Effects ^b (95% CI)		Relative Effect (95% CI)	No. of Participants (Studies)	Quality of the Evidence (GRADE) ^c
	Risk With More Concentrated CP	Risk With 10% CP			
Risk of TS assessed with dichotomous scale (yes/no) follow-up: mean 2-3 wk	648 per 1000	431 per 1000 (269-608)	OR 0.41 (0.20-0.84)	511 (9 RCTs)	⊕⊕⊕⊖ MODERATE ^d
Intensity of TS (intensity of TS) assessed with pain scales follow-up: mean 2-3 wk	—	SMD 0.44 SD fewer (0.67 fewer to 0.2 fewer)	—	340 (6 RCTs)	⊕⊕⊕⊖ MODERATE ^d
Delta SGU assessed with shade guide units follow-up: mean 2-3 wk	—	SMD 0.29 SD higher (0.25 lower to 0.83 higher)	—	398 (7 RCTs)	⊕⊖⊖⊖ VERY LOW ^{d,e,f}
Delta E assessed with spectrophotometer follow-up: mean 2-3 wk	—	SMD 1.04 SD lower (2.11 lower to 0.04 higher)	—	316 (5 RCTs)	⊕⊕⊕⊖ MODERATE ^d
Abbreviations: CI, confidence interval; CP, carbamide peroxide; OR, odds ratio; RCTs, randomized clinical trials; SGU, shade guide units; SMD, standardized mean difference; TS, tooth sensitivity.					
^a Intervention: 10% CP; comparison: more concentrated CP.					
^b The risk in the intervention group (and its 95% confidence interval) is based on the assumed risk in the comparison group and the relative effect of the intervention (and its 95% CI).					
^c High quality: We are very confident that the true effect lies close to that of the estimate of the effect. Moderate quality: We are moderately confident in the effect estimate: the true effect is likely to be close to the estimate of the effect, but there is a possibility that it is substantially different. Low quality: Our confidence in the effect estimate is limited: the true effect may be substantially different from the estimate of the effect. Very low quality: We have very little confidence in the effect estimate: the true effect is likely to be substantially different from the estimate of effect.					
^d Most RCT are at "unclear" risk of bias.					
^e Nonexplained statistical heterogeneity.					
^f High 95% confidence interval, which does not exclude important harm or benefit.					

results were equivalent, as longer application times were used for the lower CP concentrations.

When bleaching starts, the organic component of dentin has not been oxidized yet; therefore, a higher number of free radicals, available in highly concentrated CP products, have sufficient substrate for oxidization, leading to a higher degree of whitening at the beginning of bleaching. As time passes, the nonoxidized substrate reduces significantly, and the excess of active hydrogen peroxide in more concentrated CP products no longer has much substrate for action; while there are more available in dentin of those bleached with 10% CP. The lower concentration of active hydrogen peroxide in 10% CP gels is compensated for by the repetitive daily at-home bleaching protocol in a nonlinear trend. For instance, when 10% CP was applied in participants for eight hours daily, this group achieved faster bleaching than the group that used the product one hour daily; however, in just two more days, the color change of the one-hour group became similar to that of the eight-hour group.³⁰

Analogously, this also help us understand why a higher risk and intensity of TS was observed for higher-concentrated CP products. The surplus of hydrogen peroxide from highly concentrated prod-

ucts without substrate to oxidize reaches the organic component of the pulp tissue, where it may induce the formation of reactive or reparative dentin.^{31,32} Peroxides diffuse very quickly into dentin, reaching the pulp chamber, but the rate of penetration depends on the concentration and composition of the bleaching agent, the thickness of the hard tissue,³³⁻³⁶ and the application protocol.³⁰ The higher the concentration of the bleaching agent, the greater the aggression to the pulp cells.^{31,32,37}

The damage caused by the hydrogen peroxide leads to the expression of inflammatory mediators, such as substance-P³⁸ and prostaglandins, which have a recognized role in triggering nociceptive impulses for the perception of pain,³⁹ helping us explain why higher-concentrated hydrogen peroxide could be responsible for the higher absolute risk and intensity of TS.

Most of the studies in dental bleaching use shade guides for color evaluation.^{4,5,10,11,13,17,18,26,28} Although these shade guides were designed primarily for shade matching with composite resins, their use is supported in the literature for evaluating bleaching efficacy.^{34,40-42} Compared with the spectrophotometer, the shade guides show better visual correlation and have the potential to allow for more

accurate and consistent monitoring and reliable color of teeth.⁴³

It is worth mentioning that the conclusions herein collected are of moderate quality of evidence, and this means that we are moderately confident in the effect estimate. The true effect is likely to be close to the estimate of the effect, but there is a possibility that it is different. The great limitation observed in this systematic review was the high number of studies at unclear risk of bias. Future studies with well-designed protocols, when added to the results collected so far in future systematic reviews of the same topic, may eventually lead the conclusions to high quality of evidence.

CONCLUSIONS

The 10% CP product demonstrated a significantly lower risk and intensity of TS when compared to higher CP concentrations without jeopardizing color change. In any case, these results should be interpreted with caution since most of the studies included in the meta-analysis were at unclear risk of bias.

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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 State University of Ponta Grossa, Brazil.

Conflict of Interest

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

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