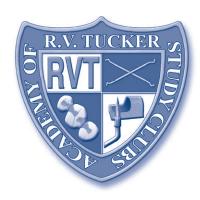
# OPERATIVE DENTISTRY







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### OPERATIVE DENTISTRY

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

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### Dental Education From a Private Practicing Dentist's Point of View

Dan B Henry, DDS, Private Practice

Dental education in the United States has gone through several major changes from the beginnings of dentistry as a trade or vocation learned through apprenticeships or on-the-job training or practiced by self-taught barber surgeons to the current system of dental school training. The evolution to the pinnacle of excellence in restorative and preventive arts did not happen overnight or by accident. Dr Horace H. Hayden and Dr Chapin A. Harris, two dental practitioners in Baltimore, Maryland, were instrumental in founding the first dental school in the world, the Baltimore College of Dental Surgery, in 1840. This served as a prototype for the formation of dental schools in other American cities, which, in turn, led to the development of a formal foundation for dental education in America.

These early dental schools were two-year programs with nonstandardized requirements for acceptance or graduation. As technology and new procedures were developed and curriculums expanded, dental schools developed into four-year upperlevel degree programs associated with colleges and universities. National standards for acceptance into dental school and graduation from dental school were adopted. Because specialty disciplines did not exist, early dental schools had a general restorative focus, and few specialty disciplines were incorporated into a general dental curriculum. Students were taught to do specialty procedures as general dentists. Deans of dental schools were typically general dentists with a broad understanding of restorative dentistry done in a private-practice setting. There was an unobstructed vision to teach dental students

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Dan B Henry

to practice competent comprehensive general restorative dentistry. The schools also had an excellent relationship with the private practicing community, and used private practitioners as part-time instructors. In addition, state/university support for dental education was at a much higher level than it currently is. Most graduates went into solo general restorative practices.

Now most dental school deans are specialists and have little or no private practice/general restorative dentistry experience. This, the author suspects, is due in part to the fact that universities recruit deans that have advanced degrees. Because of the decrease in state/federal funding, deans must concentrate a large part of their time on developing revenue sources for the dental school to avoid the situation in the 1970s and 1980s when a number of dental schools closed because of funding cuts. In the past few years, however, there has been a resurgence in privately funded dental schools. At the same time, many dental schools have dramatically increased their commitments to research, shifting resources

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away from a primary mission of teaching general restorative dentistry. Research grant money from government and industry is a significant source of funding and support for these programs. The size and scope of specialty residencies and departments have also increased. The net result is that more influence for curriculum development comes from a specialist perspective. Though restorative/operative dentistry was at one time the backbone of dental education, many dental schools have limited or eliminated these departments, absorbing them into other departments.. The unintended consequence of this refocus is a disjointed educational experience for one of the foundational skill sets for general private practice. Combine this situation with a significant national decrease in the pool of dental school instructors and you have a crisis within dental education in this country.

The downside of all of this, and it all boils down to money, is that the current graduate has less understanding of complete comprehensive restorative treatment and how to organize an effective long-term treatment plan right out of school than his or her counterpart of 30 years ago. Current graduates need more exposure to the art and science of ethical needs-based treatment planning. They need a better understanding of the consequences of all restorative options along with the technical skills to deliver all available restorative options with competence. They need dedicated time to learn from and be mentored by ethical and competent private-practice restorative dentists in an ethically sheltered environment.

The author suspects that new ideas within nontraditional frameworks will be a familiar theme in dental education well into the future. Dental schools will need to have an increased emphasis in general practice and become even more community based within their clinical facilities. This will be a better fit with government-funded mandates for access to care programs and with the overall needs of dental students and the public at large.

Dental schools should take advantage of the full eight years most students spend in college and dental school combined. By restructuring and by emphasizing different areas within the first two years of college, a revised pre-dental curriculum can be created that could be completed during the first two years of undergraduate college. Students could then be accepted into dental school at the completion of the sophomore year of college. This would give six years for a combined dental school and upper-level college curriculum that would lead to both a BS and

either a DDS or DMD degree. Incoming class sizes could be increased to compensate for those who decide not to continue to the DDS/DMD or are found to be unsuitable to continue to the DDS degree after achieving their BS degree.

The benefits of moving dental school into the last two years of college are multiple. Most important there would be a longer learning experience directed by the dental school. Liberal arts, humanities, and business classes could be more effectively incorporated into an overall strategy for developing a dentist with a balanced life approach to the art and science of dental practice. Another benefit is that upper-level science classes, such as organic chemistry or genetics, could be tailored for dental students. This would give dental students more meaningful information and would eliminate time spent duplicating basic science instruction, which is what happens in the current dental school curriculum. In addition, this program design would give dental schools more time to better evaluate a student's ability to continue to the DDS degree. The added two years would allow for a shift from the current emphasis on grades to a total evaluation of the student's intellect and character for determining future ethical success within the profession. It should also give dental schools better options for taking responsibility to eliminate those who do not have the skill sets to be competent or ethical dentists. Thus, it should allow for better selection of those persons who would make great teachers.

Within a dental program such as this, the first three years would be preclinical and the last three years would be devoted primarily to clinical training. Students could also have clinical exposure during the first three years by assisting fifth-year students in the clinic and starting to have patient interactions in other ways. The fourth and fifth years would be completed in a traditional dental school clinic setting. These two years would be similar to the traditional dental school clinical experience. Students would learn the art and science of restorative dentistry and would be required to pass competency evaluations in all disciplines by the end of the fifth year

The sixth and final year of the curriculum would be spent away from dental school in a community clinic. Students would be under the supervision of credentialed ethical private-practicing dentists in the area. Depending on the size, a clinic would have one or more full-time dental school instructors from a supporting dental school. Their job would be to monitor treatment, teach, and the run the community clinic on a day-to-day basis. By spending the last year of dental school in a community clinic under the supervision of private-practicing dentists who would act in a mentoring role, dental students would have a more meaningful transition from the academic environment of dental school to the practical world of private practice in an ethically mentored environment.

These clinics would need government funding. Federal funds could be used to build the facility with federal/state support to operate the clinic. Revenues earned from fee-for-service and Medicaid patients would remain with the sponsoring dental school. Because this would not be a residency program, but the last year of dental school, students would not be entitled to stipends. These three conditions—government funding, fee-for-service collections, and no residency stipend—should make the clinics at least revenue neutral. Benefits again should be obvious. The government would get the most benefit for the use of taxpayer dollars. Dental schools would have an additional funding source, at least to the point

that the clinic would be revenue neutral. Students would have the benefit of a full year's exposure to ethical, private-practicing clinicians and a more meaningful transition into private practice. Access to dental care for the underserved would be improved. Finally, the community at large would gain an asset.

### CONCLUSION

The future of dentistry in the United States remains fluid. By developing patient-centered solutions for how dental students are selected, trained, and licensed, dentistry will be positively affected. If solutions are not developed that bring dentists and dental students into communities where they become part of the solution to access to care, those outside of dentistry will fill the void. Public opinion will force government policy makers to make changes that will fragment the profession. As a consequence, oral health and general health will suffer in America.

# The Tucker Technique: The Proximal Hollow Grind to Address a Root Concavity

### TA Hess

### Clinical Relevance

This case report describes a modification of the Tucker technique to manage a concavity of a proximal surface when a traditional cast gold box form would be too invasive. A conservative and esthetic alternative to this box form is the proximal hollow grind.

### **SUMMARY**

Cast gold inlays have long been used to conservatively restore compromised tooth structure. When the mesial or distal proximal surfaces are indicated for restoration and a cast gold restoration is desired, traditionally a box is prepared with an external bevel. Often a root concavity does not allow for a standard box form or the external and/or internal bevels. A proximal hollow grind can be utilized to address limits of standard inlay or onlay preparation design.

### **INDICATIONS**

Cast gold inlays have long been used to conservatively restore compromised tooth structure. When the mesial or distal proximal surfaces are indicated for restoration and a cast gold restoration is desired, traditionally a box is prepared with an external bevel. With a single proximal box on a bicuspid or

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maxillary first molar where the oblique ridge is not broken, often an internal bevel is placed in the dentin to create a guiding plane for accurate seating of the casting.<sup>2</sup> The internal bevel ensures a tight gingival seal upon cementation. Often a root concavity does not allow for a standard box form or the external and/or internal bevels. A proximal hollow grind can be utilized to address limits of standard inlay or onlay preparation design.<sup>3</sup>

### **TECHNIQUE**

Teeth 4 and 5 were observed to have mesial and distal decay secondary to excessive consumption of highly acidic energy beverages. Tooth 4 was restored with a MOD amalgam, and a composite buildup was placed in tooth 5 for a future inlay restoration. Twelve months later, the patient returned with a fractured tooth 4–MOD amalgam despite what appeared to be sufficient occlusal depth of the preparation (Figures 1 and 2). The patient elected to have teeth 4 and 5 restored with MOD cast gold inlay restorations to resist failure due to fracture.<sup>4,5</sup>

The occlusion was evaluated and anesthesia administered. A rubber dam retainer (Hu-Friedy,

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Figure 1. Preoperative condition.

Chicago, IL, USA) was applied to tooth 3, and holes with approximately 3.5 mm of space between each were punched in the heavy-weight rubber dam (Hygenic Dental Dam Latex; Coltène/Whaledent, Cuyahoga Falls, OH, USA). The rubber dam was placed for optimal isolation and access by extending from tooth 3 to tooth 11. Typically all of the existing restorative material and any caries would be removed. However, in this case the operator elected to utilize the existing composite buildup on tooth 5. The fractured amalgam was removed from tooth 4 with a #6 high-speed round bur (Midwest/Dentsply International, York, PA, USA). A band and retainer (Tofflemire; Water Pik Inc, Fort Collins, CO, USA) without gingival wedging was applied to tooth 4, and dual-cure composite (ParaCore; Coltène/Whaledent) with its associated adhesive was used to build up to close-to-original contours.

An essential concept of the Tucker technique is the use of a buildup. The buildup allows conservative cavity preparation because the operator does not need to extend occlusal and axial walls to address undermining caries or extensions of previous restorations. Ideal taper, smoothness, and proportions can be created.

Initial occlusal preparation of both tooth 4 and 5 was performed using a #56 fissure carbide bur (Midwest/Dentsply International). The mesiobuccal line angle of tooth 4 was used as a guide for preparation to minimize the buccal extension and the display of gold to maintain esthetics. On both tooth 4 and 5, depth of the central groove area was reduced to approximately 1.5 mm and that of the buccal and lingual walls 2.5 mm as a result of the inclines of the cusps. The #56 carbide was used to create an angulation of approximately 3°-5° on each



Figure 2. Bitewing radiograph illustrating thickness of tooth 4 amalgam.

of the occlusal walls, and therefore an ideal preparation taper of  $6^{\circ}$ - $10^{\circ}$  was produced.

The mesial of tooth 5 was initially prepared using the #56 bur. Once the mesial concavity was visualized the decision to prepare a mesial hollow grind was made. A relatively simple preparation with rounded internal angles was made with a #7404 bur (Brasseler USA, Savannah, GA, USA).3 Retention of the casting was maximized by keeping the #7404 bur at a consistent perpendicular angle to the pulpal wall as the bur was carried interproximally. Because of the diameter of the #7404 bur, the buccal and lingual extensions were finalized with a #7901 flame finishing bur (Midwest/Dentsply International). The #7901 bur is used to avoid contact with the adjacent tooth. Care was taken to create enough axial depth in the buccal and lingual extensions for bulk of gold during casting and accuracy of fit when seating and finishing. The #7901 bur was held perpendicular to the pulpal floor and was used in the painting stroke from the mesiobucco-gingival point angle and in blending occlusally to the bucco-occlusal line angle with a minimal lean toward the buccal. The #7901 bur was then used to finish from the mesiolinguogingival point angle to the linguo-occlusal line angle with slightly more angulation toward the lingual than was used toward the buccal. Ideal draw was achieved without unesthetic extension to the mesial buccal of tooth 5.

The mesial proximal draw of tooth 5 guided the preparation of its distal box. The #56 bur was used to establish a proximal box to a depth of 1.5 mm gingival to the pulpal floor, or 4 mm from the proximal-occlusal cavosurface on the mesial and distal of tooth 4 and distal of tooth 5. The mesiobuccal extension of

tooth 4 was kept conservative by planning the draw of tooth 4, including axial and proximal walls, to allow seating of tooth 4 before tooth 5.

The proximals, excluding the mesial of tooth 5, were trued using hand instrumentation. The 42S off angle chisel (G Hartzell & Son, Concord, CA, USA) was utilized to smooth the pulpal and gingival walls. Mesial proximal axial line angles are placed with the 42S off angle chisel, and distal proximal axial line angles are placed with the 43S off angle chisel (G Hartzell & Son). The mesial axial wall was smoothed with the 43S off angle chisel and the distal axial line angle with the 42S off angle chisel. External bevels (0.5 mm) were placed using a beveled cylinder carbide bur (H248-009; Axis, Coppell, TX, USA) and planed with the #233 Tucker gingival margin trimmer (G Hartzell & Son) on the mesial and the #232 Tucker gingival margin trimmer on the distal (Figure 3). The Tucker gingival margin trimmers have an angulation of  $30^{\circ}$  rather than the  $45^{\circ}$ angulation of non-Tucker gingival margin trimmers (G Hartzell & Son). Forty-five-degree gingival margin trimmers are used to place the internal bevel on restorations with a single proximal box. Internal bevels will increase retention and resistance form.

A small gingival retraction cord (#0 Ultrapak; Ultradent Products Inc, South Jordan, UT, USA) soaked in 25% aluminum chloride solution (Hemodent: Premier, Plymouth Meeting, PA, USA) was tucked into the sulci of teeth 4 and 5. Next a braided cord (#2 Gingi-Pak; Belport Co Inc, Camarillo, CA, USA) was placed into the sulci above the smaller cord for five minutes prior to being removed. Upon removal the preparations were impressed using only light-body polyvinyl siloxane (Flexitime; Heraeus, South Bend, IN, USA) syringed around the preparations and into a double arch tray (Check Bite; GC America, Alsip, IL, USA). Upon setting the impression was removed and inspected and judged to have sufficient detail. It was noted that the impression material capturing the mesial concavity of tooth 5 obscured the gingival margin when viewed directly from above (Figure 4).

Provisionalization was achieved with a piece of temporary stopping (Hygenic Temporary Dental Stopping; Coltene/Whaledent) heated with a lighter and placed with a Woodson 2 composite instrument (Hu-Friedy) into the proximals so that the acrylic (DuraSeal; Reliance Dental Mfg Co, Worth, IL, USA) would not contact soft tissue. Care was taken not to aggressively pack the stopping and create separation of the teeth. The temporary stopping was kept below

the contact so that the acrylic would lock in. Acrylic was added using a liquid/powder technique and a disposable brush (Benda Brush; Centrix, Shelton, CT, USA). While the acrylic was soft the patient was instructed to bite and go into excursive movements. Once the acrylic was set the #0 gingival retraction cords were removed from the sulci. The patient was advised that he would be unable to floss this area prior to the seating appointment.

Seating involved anesthesia, removal of the provisionals, and placement of a heavy-weight rubber dam. Gold castings (JRVT Gold 77% Au, 1% Pd, 13% Ag: Jensen Industries Inc, North Haven, CT, USA) were tried in together to verify fit and proximal contacts. Unfortunately, there was inadequate contact between the castings (Figure 5). The distal of tooth 5 was chosen to add to because it looked slightly undercontoured. Gold was added to the distal of tooth 5 using solder (650 Fine; Jensen Industries Inc) and flux (Brown Fluoride Flux; Jensen Industries Inc). A mixture of water, phosphoric acid, and urea (Prevox Liquid; Ivoclar Vivadent Inc, Amherst, NY, USA) was heated in a fume hood and used to remove the oxidation layer from the soldering process.

The inlay castings were seated one at a time beginning with tooth 4. Tooth 4 was seated first so that the mesiobuccal aspect could be accessed for ideal finishing. Separate mixes of zinc phosphate cement (Fleck's Cement; Myerstown, PA, USA) for each tooth were used to give adequate time for finishing of tooth 4. A resin-modified glass ionomer or self-adhesive modified resin cement could have been used.<sup>6,7</sup> The zinc phosphate was slaked with a small amount of powder in the liquid until the liquid appeared clear and was then mixed according to the manufacturer's directions. Cement was applied to the castings, and an orange wood stick (Pearson Dental, Sylmar, CA. USA) along with light tapping with a Gourley mallet was used to seat the castings. A shortened orange wood stick was then used between the castings and lower premolars until the hydraulic pressure of cementation had dissipated. A series of sandpaper disks (medium garnet, fine sand, and fine cuttle) (EC Moore, Dearborn, MI, USA), linen strips (Moyco Dental, Philadelphia, PA, USA), and polishing powders were used to refine the tooth-to-gold interface. Wet #4 laboratory pumice (Kerr Corp, Romulus, MI, USA) was used next with a ribbed prophy cup (Young Dental, Earth City, MO, USA). A light touch rotating from casting to tooth was employed to avoid the uneven removal of tooth and gold if the pumice was used too aggressively or for too long.



Figure 3. Final preparations.

The tooth 5 inlay was then seated utilizing the same steps described above. After the #4 laboratory pumice, wet 15-µm aluminum oxide powder (Micro Abrasives Corp, Westfield, MA, USA) with a new ribbed prophy cup was used. Rinsing and drying of the castings and teeth were performed between

polishing steps to prevent incorporating scratches late in the sequence. Final polishing was performed dry with 1-µm aluminum oxide powder (Micro Abrasives Corp), again with a new ribbed prophy cup (Figures 6 and 7).

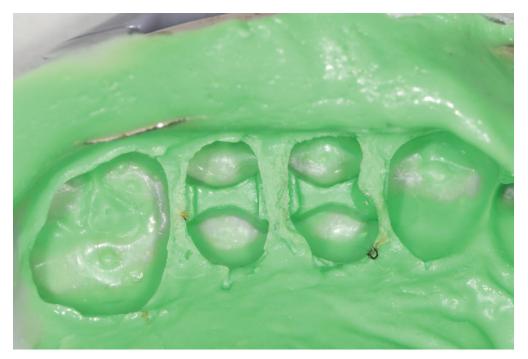


Figure 4. Impression.



Figure 5. Try-in of castings with open contact.

Once the rubber dam was removed the occlusion was verified with bite registration tape (AccuFilm; Parkell, Edgewood, NY, USA) lightly coated with petroleum jelly (Vaseline; Unilever, Englewood Cliffs, NJ, USA) to improve the visibility of marks obtained. The patient was released after occlusion

was confirmed with the patient supine and then again upright.

### Acknowledgements

Castings are by Mr Gary Burk (Issaquah, WA, USA). The preparation designs are based on those of Dr RV Tucker. The



Figure 6. Final restoration—occlusal view.



Figure 7. Final restoration—buccal view.

2013 Tucker Institute Syllabus, Drs RV Tucker, RD Tucker, DM Miya, and WK Johnson, provide the guidance and details for the technique steps. The author acknowledges support from his study club mentor, Dr Robert Ward.

### **Conflict of Interest**

Dr Hess, author of this manuscript, certifies that he has no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article except for personal interests in The Academy of RV Tucker Study Clubs.

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### Cracked Tooth Syndrome in an Unrestored Maxillary Premolar: A Case Report

S Batalha-Silva • R Gondo • SC Stolf LN Baratieri

### **Clinical Relevance**

Cracked tooth syndrome is an uncommon finding in unrestored teeth. However, these phenomena appear to be found with increasing frequency, creating a challenge for a correct diagnosis. Direct-bonded restorations may be a safe and conservative option for teeth affected by incomplete cracks.

### SUMMARY

Cracked tooth syndrome is known to occur most frequently in heavily restored teeth. Nevertheless, when the symptoms occur in intact teeth, there is difficulty in obtaining a correct diagnosis because it is difficult for the dentist to find where the crack is located. This clinical report describes the diagnostic procedures and the direct bonded composite resto-

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ration used to restore an incompletely fractured unrestored maxillary premolar in a 22-year-old female patient. To achieve a correct diagnosis, the following were performed: periapical and bitewing radiographs, percussion and thermal vitality tests, a bite test, and the placement of a stainless steel band. Once the symptoms ceased with band placement, cone beam computed tomography, transillumination, macro photographs, and isolation with a rubber dam helped to visualize the crack line along the occlusal surface involving distal and mesial marginal ridges. The crack was traced using a high-speed tungsten carbide bur until the fracture line was not visible. The tooth was restored with a direct composite resin, associated with a total-etch adhesive system, and the symptoms were immediately eliminated.

### INTRODUCTION

Fractures are one of three major conditions in human teeth that cause pain. However, when a tooth fracture is incomplete, the presentation is more subtle and frequently remains undiagnosed because signs and symptoms are often confusing and are not sufficiently recognized by clinicians. Cracked tooth

syndrome (CTS) is described as an incomplete fracture of a vital posterior tooth involving enamel and dentin and possibly the dental pulp. It may cause a complete fracture, reaching the dental pulp and/or periodontal ligament.

The term *cracked-tooth syndrome* was first used by Dr Cameron in 1964 and was defined based on three clinical observations.<sup>2</sup> First, a patient complained of pain upon application of cold or pressure to a tooth that had been recently restored with a mesio-occlusal inlay, and there was relief of the pain a year later when the distal cusp broke off. even though sensitive dentin was then exposed. In the second observation, some posterior teeth had abscessed with small shallow restorations and no periodontal disease; however, there was evidence of rarefactions at the apex. Upon extraction and examination with magnification, many of these teeth were found to have cracks extending from the mesial, distal, or both marginal ridges, and the patients had a previous history of pain or occlusal adjustment. The third observation came from three other cases where, after extraction of fractured teeth, patients shortly thereafter complained of pain in another tooth. These latter suspect teeth were covered with crowns and complete fractures were prevented. Other authors had previously described the incomplete fracture as "cuspal fracture odontalgia,"3 "fissured fractures,"4 and "greenstick fracture of the tooth crown."5

The predominant symptom of a cracked tooth is discomfort from chewing pressure, especially with hard foods of a certain consistency. 1,2,6-9 This pain may have been present for several months previously, 10-12 so the patient might have adapted to the pain by learning to avoid the painful side when biting tough foods. The pain is sharp and brief, usually lasting as long as the pressure persists and ceasing when the force ends. This can be explained by dentinal tubular fluid flow, which is a result of the movement of the tooth fragments away from and toward each other. 13 These movements also can stretch and possibly disrupt dentinoblastic processes along the fracture plane. In vital teeth, depending on the depth of the crack and its duration, an additional symptom may include sensitivity to thermal changes, particularly to colder temperatures.<sup>2,7</sup>

The predisposing factors and etiology of CTS are still not properly understood and have a complex and multifactorial aspect. Usually, the cracks occur in restored teeth, with a direct relationship to the size of the restoration. 11,14 Teeth with restorations have

a 29 times greater risk for cracks when compared with intact teeth, <sup>15</sup> and there is a higher incidence of CTS in teeth with restored marginal ridges. <sup>8</sup> However, intact or minimally restored teeth can also be compromised by incomplete fractures, with some studies <sup>16,17</sup> finding an increased incidence of the syndrome, ranging from 13% to 74% in healthy teeth. <sup>9,18-22</sup> An *in vitro* study confirmed that unrestored molars could withstand higher stresses under mechanical and thermal loads than restored molars with amalgam and composite restorations. <sup>23</sup>

It is interesting that CTS can affect multiple teeth in the same patient. <sup>2,24-27</sup> Cameron treated 50 patients with CTS, and seven patients had lost another tooth due to fracture. In another study, nine patients (28%) exhibited incomplete fractures in more than one tooth, ranging from two to six. <sup>11</sup> A couple of case reports described bilateral cracked teeth in intact maxillary molars and premolars. <sup>24,25</sup> In those cases, the authors mentioned there was evidence of tooth wear, habits of chewing very hard nuts, <sup>24</sup> wear facets, hypertrophy of the masseter muscles, and extremely steep cusp-fossa relationships, which could have acted as a splitting force on maxillary teeth. <sup>25</sup>

The following clinical case describes the occurrence of CTS in an unrestored maxillary premolar. After the use of simple and sophisticated tools for diagnosis, the crack was traced with a carbide bur and a direct-bonded composite resin restoration was performed.

### **CLINICAL CASE REPORT**

A 22-year-old female patient presented to the Department of Operative Dentistry at the Federal University of Santa Catarina complaining of discomfort with the maxillary left premolar during mastication of soft food (Figure 1). Her medical history revealed no systemic problems. The patient reported that she had suffered a road traffic accident 2 years prior but with no major trauma to the head region. Tooth 24 was intact with no periodontal or endodontic disease, based on clinical and radiograph exams (Figure 2). No symptoms related to cold sensitivity were present, but some pain presented when chewing sweet food. The patient was asked to bite a wooden wedge and complained of pain when the biting pressure was released (Figure 3). Transillumination (Microlux, Addent, Danbury, CT, USA) showed a fracture line on the occlusal surface in a mesiodistal direction, affecting both marginal ridges (Figure 4). Because it was not clear whether this



Figure 1. Initial view of the nonrestored tooth 24.

crack was causing all of the symptoms, and to exclude others sources of pain, a stainless steel orthodontic band was cemented on the tooth and the patient tested the tooth for 21 days (Figure 5).

The placement of the orthodontic band immediately eliminated all symptoms. The band was removed after 21 days and, in an attempt to assess the depth and length of the crack, cone beam computed tomography was conducted. The images localized the crack at the same position as was observed in macro photographs and transillumination (Figures 4 and 6). The tooth was isolated with a rubber dam, where the crack became more evident due to dehydration (Figure 7). A high-speed tungsten carbide bur was used to create a conservative cavity preparation and the crack was removed until no fracture line was visible. The crack involved enamel



Figure 2. Periapical radiograph of left maxillary premolars. No significant findings were present.



Figure 3. Bite test using a wooden wedge revealed pain in tooth 24.

and the external dentin. A fiber optic light was used to confirm crack removal during the cavity preparation (Figure 8). A provisional restoration (Bioplic, Biodinâmica, Londrina, Brazil) was placed and the patient tested the tooth for two weeks (Figure 9). Given that no more symptoms were present, the mesial-occlusal-distal (MOD) defect was restored (Figure 10) with a direct composite resin (Filtek Z350 XT shade A2E, 3M ESPE, St Paul, MN, USA), using a total-etch adhesive system (Adper Single Bond 2, 3M ESPE).

### **DISCUSSION**

By eliminating the symptoms before and after the restorative treatment, and by making the crack visible, it may be concluded that the intact maxillary premolar in the current study suffered from CTS.



Figure 4. A crack was observed under transillumination on the occlusal surface of tooth 24, from a mesial to distal orientation.



Figure 5. A stainless steel orthodontic band was cemented to confirm the diagnosis. The patient could bite without pain after this procedure.

In a more recent clinical investigation of 154 cracked teeth, most of the affected teeth (89.6%) were intact or minimally restored. The Similarly, another study found that 40% of incomplete fractures were in healthy teeth or in teeth with a single occlusal restoration. This is not a recent event. Hiatt, In an earlier clinical study, found a high percentage (74%) of incompletely fractured teeth with no restoration or without a significantly weakened tooth due to a Class I restoration. Therefore, the possibility of an unrestored cracked



Figure 6. A cone beam computed tomography showing the crack in a mesial-distal direction, involving the mesial, occlusal, and distal faces.

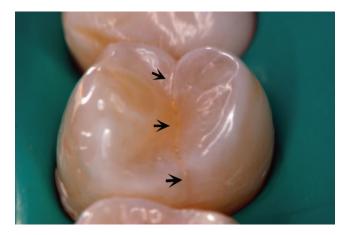


Figure 7. Using a rubber dam, the crack was better visualized by giving a contrasting color to background and keeping the tooth dehydrated.

tooth should be considered, regardless of the location of the tooth or the presence and size of a restoration.

An unintentional bite on a very hard and small object, like a seed, is the most common cause for CTS. $^{1,6,20}$  This event can immediately cause an

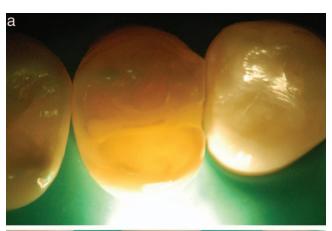




Figure 8. (a): Transillumination was useful to confirm the defect removal during the cavity preparation, until no more cracks were visualized. (b): Completed MOD cavity preparation.



Figure 9. The tooth received a provisional restoration, and the patient tested the tooth for 21 days.

excessive masticatory load, due to the small contact area. As a result, the tooth may crack or fracture. 20,21 The same can happen during heavy impact in an accident involving the mandible and maxilla, as is believed to have happened in this present clinical report. Patients with healthy physiologic occlusion are inherently more confident to chew and might unexpectedly exert an excessive masticatory force on a hard object when compared with patients with periodontal disease. Patients with periodontal disease are naturally more cautious with biting due to the presence of mobile teeth, and consequently, these patients are less prone to splitting teeth due to mastication. 6

Another etiological factor for the presence of CTS in unrestored teeth is excessive occlusal forces. 2,14,19 Cameron proposed that the force exerted to cause the crack seems to be a prolonged hammering.<sup>2</sup> Almost 60% of the cases provided histories of discomfort from one month to 10 years, and few patients could remember a sudden force. However, these patients had usually suffered complete fractures.<sup>2</sup> In addition, most patients exhibit worn occlusal surfaces and canines, which are evidence of interferences, interceptive contacts, or bruxism.<sup>2,8,10,24,25,29,30</sup> Occlusal trauma also can be present when there are severe occlusal premature interferences from working and nonworking sides, teeth adjacent to an unrestored edentulous region, or malposed teeth. 6,10,19 The general dental exam of occlusion should observe disclusion movements. Patients with anterior disclusion rarely have a fractured tooth, unless it occurs during a "masticatory accident." The incidence of fracture is greater in extreme Angle Class II occlusal relationships because anterior disclusion is poor or nonexistent, relegating shearing forces to the posterior teeth. 10



Figure 10. After the symptoms were eliminated, a direct bonded MOD composite restoration was performed.

In this current clinical report, it is possible that the anatomical features contributed to the crack occurrence. Cameron<sup>2</sup> affirmed that premolars and molars are frequently fractured and split in a mesiodistal direction into buccal and lingual fragments. Bifurcated roots and the occlusal relationship with antagonist teeth are crucial for the incidence of the CTS. 7,20,21,31 Teeth with deep cusps and steep fossae are more vulnerable to fracture due to the wedge effect from antagonist cusps, resulting in compressive forces in the cusps and tension forces in the pits. 1,6,18,20,25 This wedge effect has been suggested as a major cause of broken intact teeth, 14,18,31 especially in maxillary premolars with deep grooves, 11,20,21,32 as presented in this current clinical case. Another causative factor, suggested by Hiatt, <sup>18</sup> is an internal structural weakness that might exist between cusps at calcification sites that have failed to coalesce. These developmental weaknesses are noted in the formation of pits, fissures, grooves, and lamellae, creating a point of structural fatigue that tends to spread or separate cusps. 18

This clinical condition, when present in intact teeth, carries a puzzling challenge to the professional, when compared with restored teeth, because it has been recommended to remove all of the restorative material in order to visualize fractures in teeth with CTS. <sup>2,8,16,19-21,27</sup> The crack in an unrestored tooth is frequently hard to see during a common clinical examination. Moreover, it is even harder to trace the crack with a bur. Fortunately, simple tools may help find where the crack is located, such as transillumination, application of dyes, isolation with rubber dam, macro photographs, and surgical loupes or microscopes, <sup>8,11,19,20,24,28,30,33,34</sup> although these tools cannot reveal the extent or depth of a

fracture.35 The use of a rubber dam enhances the visibility of these cracks because it isolates the tooth with a contrasting color and keeps the area free of saliva, removing peripheral distractions.8 However, these tools should be used in association with bite tests: This type of test is the most reliable for reproducing the symptoms of CTS because biting pain is present in more than 80% of the cases. 9,22,27 Transillumination, for instance, dramatizes all cracks to the point that craze lines (shallow cracks confined to enamel that are nonsymptomatic and do not require treatment) appear as serious structural cracks, confounding the visualization of symptomatic cracks. 1,19,36,37 In this current clinical case, another confounding factor was centrally located cracks, because they seem to follow the lines of the dentinal tubules, more closely approximating the dental pulp and causing more severe symptoms. 2,17

Although the crack in the current case was visible by transillumination, the placement of an orthodontic stainless band was indicated to confirm the diagnosis. This procedure has been advocated for unrestored cracked teeth and for those teeth where the existence of a crack can only be presumed by the symptoms. 1,7,16,20,35,38 As shown in this present clinical report, the stainless steel band aided in a correct diagnosis and appeared to be the most conservative approach. The band serves as a splint; if the pain on chewing stops, the diagnosis is confirmed. After cementation of the band, the biting test is repeated and the patient should not feel any pain. The patient is then instructed to use the tooth normally, and is reexamined after two to four weeks. The band should be cemented with a glass ionomer or carboxylate cement and fit tightly, sometimes requiring adjustment so as not to interfere with occlusion. 1,16,20 If the patient is able to chew normally with the band in place, the crack should be removed and a restoration can be placed. If pain or sensitivity to temperature has not ceased, then endodontic therapy has to be considered, because the crack may extend close to the pulp or may even involve it. 1,16,35 If endodontics is indicated, the orthodontic band should be used to reinforce the tooth during endodontic treatment. 1 It is not costeffective to subject the patient to a restoration without knowing whether the pulpal condition is reversible; however, it is much more logical to apply a provisional restoration and/or a stainless steel band, because it eliminates this source of error if the pulp is involved. 16

In an attempt to confirm whether the visible crack was in dentin and possibly responsible for symptoms,

cone beam computed tomography was performed. Although a cone beam exam may not detect a small defect, such as an incomplete fracture, an in vitro study concluded that this approach was more successful than digital radiography in detecting cracks smaller than 0.2 mm in thickness.<sup>39</sup> This technique involves a single 360° beam scan in which the x-ray source and a reciprocating detector synchronously move around the patient's head. This produces submillimetric resolution, ranging from 0.4 mm to as low as 0.125 mm, 40 and is a noninvasive tool for assessing the length of the fracture. 41 Some limitations of this computed tomography are related to artifacts that may appear as dark zones or streaks around metallic materials, implants, and, to a lesser extent, endodontic filling materials, which present similarly to root fractures and thus lead to false positive readings. 42,43 Clinicians should use cone beam computed tomography only when the need for imaging cannot be answered adequately by other, simpler tools, because this modality has a higher radiation dose than conventional dental radiography, especially in the case of children or young adults.44 Although the use of cone bean imaging is a relatively new technique for detecting cracks causing symptoms of CTS, a recent clinical case<sup>41</sup> reported a very similar condition in an unrestored upper premolar, which appeared as a faint craze line on the distal marginal ridge and extending mesiodistally, involving the lingual pulp horn of tooth 24.

Several therapies have been proposed to treat painful cracked teeth; however, the specific therapy depends on the severity of symptoms and location of the crack. 7,45,46 Many authors have suggested an occlusal reduction to provide relief from occlusal stresses in centric and lateral relationships. 18,19,47 However, the reduction or elimination of occlusal contacts is temporary and is not sufficient because 1) the tooth could be loaded by a food bolus while chewing and occlusal reduction does not eliminate the risk of fracture, <sup>2,18,20</sup> and 2) this procedure also involves the removal of healthy, sound tooth tissue. 12 If the affected cusps fracture off spontaneously during removal of the filling, treatment is straightforward and consists of replacing the lost tooth substance and overlaying the remaining cusps. 16

Before restorative procedures, careful tracing of the crack to decide the next line of approach has been advocated.<sup>35</sup> It is possible that the crack disappears into the dentin; however, if the crack continues in a pulpal direction, it is likely that the root is involved.<sup>19</sup> A clinical study has recommended an interim restoration using traditional glass ionomer

cements after the removal of restorations and/or cracks from the affected tooth to sedate the pulp and help with the healing response. After this procedure, the patient should be asked to record whether the pain disappeared and to be alert for possible postoperative sensitivity. In case of continuous pain or persisting thermal sensitivity, an endodontist should evaluate the tooth for endodontic treatment. According to Abbott, three months is the time indicated to assess the pulpal status of reversible pulpitis caused by CTS. The pulpal status of reversible pulpitis caused by CTS.

In early clinical studies, the treatment of CTS was the placement of a complete crown restoration. <sup>1,2,16,17,19,20,35,48</sup> Conversely, it seems that full crowns are less effective in preserving the pulpal vitality. In a one-year clinical investigation, 84.4% of cracked teeth were treated with crowns; half of those teeth needed root canal treatment, considering that around 60% of the cases were initially intact teeth. 17 In another short-term clinical evaluation of cracked teeth, approximately 20% needed endodontic treatment after crown placement within six months of service. 49 Even when the root canal treatment is performed in advance of crown placement, this association has a survival rate of only 85.5% based on a two-year clinical evaluation of root-filled, cracked teeth. 50 Opdam and others<sup>46</sup> noted that painful cracked teeth often presented a long period of persisting pain, but gradually reduced postoperative sensitivity, and that maintaining the vitality of the dental pulp might improve the long-term prognosis of the tooth.

A more invasive treatment includes a higher loss of tooth structure and may result in extraction. 46,51 Some reasons for extraction may be pulpitis, restorative failures, and endodontic needs in CTS. First, this is related to the crack itself, because it can be deep enough to invade the pulp chamber, and later this can be confirmed during endodontic opening, because the fracture line is detectable from inside the pulp chamber. <sup>29,30,46</sup> Second, a scanning electron microscopy investigation<sup>52</sup> determined that all symptomatic cracks appear to be extensively contaminated by bacteria and may be the cause of pulpitis after the treatment of cracked teeth. Third, an additional factor that may contribute to pulpitis is the direct diffusion into the pulp of dentin adhesive components used to restore cracked teeth. <sup>30</sup> Finally, the indication of a more invasive restorative treatment, such as a full crown, might be accompanied by a higher loss of teeth in the long term as a result of extraction.46

As in this current clinical case, a direct-bonded composite resin restoration can be a successful

treatment for a painful cracked tooth. 29,46,51 It is possible that this procedure has the potential to bond the affected cusps,<sup>29</sup> creating less sensitivity during mastication due to cusp reinforcement.<sup>23</sup> Behle,<sup>51</sup> relying on modern bonding restorative materials, stimulated thought on minimally invasive dentistry because clinical experience indicates that even symptomatic cracked teeth can be restored without full coverage by injecting a flowable composite into the crack area, which is especially true for low-force areas, such as with premolars. Simonsen<sup>53</sup> noted that crowns can be considered overtreatment in some situations when a restorative decision is based on expedience or economic advantage. Moreover, indirect restorations can result in more loss of sound tooth tissue and may increase the risk for pulpal complications due to the need for temporary restorations.<sup>29</sup> Cuspal coverage with direct composite resin had no failures after a seven-vear clinical investigation<sup>46</sup> of painful cracked teeth, whereas the mean annual failure rate for no-cuspal coverage direct composite restorations was only 6%. It is possible that the direct composite resin has some level of "shock-absorbing effect" by increasing cuspal stiffness and by redistributing occlusal loads away from the crack and toward the axial walls and down the long axis of the tooth. 12

Nevertheless, for slightly larger defects, the aim of minimizing flexure of the compromised cusp also can be achieved with success using conservative, bonded partial-coverage ceramics<sup>54</sup> or composite onlays.<sup>30</sup> These are great choices for true esthetic demands.

### **CONCLUSIONS**

The prevention and early recognition of cracked tooth syndrome is essential for avoiding more injuries and preventing the progression of cracks into the pulp or root. Simple tools, such as transillumination, bite tests, macro photographs, and isolation with a rubber dam, are useful for locating incomplete fractures. Bonding options, such as with direct composite restorations, should be considered for restoring cracked teeth because they are quick, low-cost, conservative, and readily available restorative techniques, and they have proven to bond to compromised cusps.

### **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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### 10-year Follow-up of Natural Crown Bonding After Tooth Fracture

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### Clinical Relevance

Fracture of anterior teeth by trauma has become frequent in the permanent dentition, and bonding of the tooth fragment seems to be an excellent and reliable treatment. It is a quick and simple procedure that reduces concerns about tooth color and the longevity of composite restorations.

### **SUMMARY**

The aim of this article is to discuss relevant considerations about crown bonding and describe a clinical case in which a tooth fragment and direct composite resin were used to successfully restore a fractured anterior tooth. Clinical examinations showed good esthetics and periodontal health after 10 years of follow-up.

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### INTRODUCTION

The purpose of this case report is to demonstrate the bonding of a lower canine tooth that fractured obliquely and to assess its longevity.

### **DESCRIPTION OF THE TECHNIQUE**

A 30-year-old male patient had an accident while surfing and fractured the crown of the lower right canine tooth (27) obliquely, involving enamel, dentin, and pulp (Figures 1 and 2). After the trauma, the patient kept the tooth fragment in his mouth and then stored it in real saline solution. Endodontic treatment was conducted because of the prolonged exposure of the pulp. During all phases of endodontic treatment the fragment was kept in the solution in a closed flask. The patient had undergone direct restorative esthetic treatment to close the diastema in teeth 6, 7, 8, 9, 10, and 11 with composite resin approximately 5 years earlier. This fracture revealed the strength of that composite buildup, which suffered no alterations in the accident. Because the patient was young and long-term follow-up was possible, bonding of the tooth fragment was proposed. The patient agreed, and treatment was conducted.

The attachment of the fragment was checked for fit, and then the tooth was isolated with a 212 clamp and rubber dam (Figure 3); 2 mm of root canal filling

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Figure 1. Tooth fragment.

was removed with a round number 4 carbide bur using a slow-speed handpiece. Phosphoric acid at 35% was applied for 15 seconds for etching on both the fragment and tooth and then removed by flushing with water for 30 seconds. Primer and adhesive were applied on the remaining tooth structure followed by application of a layer of Z100 composite resin. Subsequently, the fragment was placed and excess material removed (Figure 4).

The adhesive system and composite resin were cured at the same time so that the adhesive layer would not interfere with the fitting of the fragment. The composite resin Z100 was chosen because it had better flow than other resins available. The curing stage needed special attention to ensure that the light would reach the deeper portions of the tooth; thus, the light irradiation time (SDI, Victoria, Australia) was 60 seconds on each surface, the buccal and lingual. Excess material was removed using a number 12 scalpel blade. Finishing was done with diamond burs and abrasive discs in areas that allowed their use; occlusal adjustment was done with articulating paper (Figure 5).



Figure 2. Oblique fracture.



Figure 3. Absolute isolation.

Recall appointments were conducted annually for two years. At that point, the patient returned reporting the debonding of the fragment, which was then rebonded using dual-cure composite Biscore along with the adhesive system Scotchbond Multipurpose Plus with activator, in a self-cure chemical reaction. The patient remains scheduled for annual recall appointments (Figure 6).

### POTENTIAL PROBLEMS

Dental trauma is quite frequent and is considered a public health problem.<sup>1</sup> It may cause esthetic, physical, and psychological problems.<sup>2</sup> The therapeutic challenge in cases of dental trauma is assessing the extent of the damage and any other implications that will define the treatment needed. Type and extent of the fracture, presence or absence of pulp exposure, root involvement, and condition of the dental fragment must be taken into account when determining the restorative treatment.<sup>3</sup>



Figure 4. Fragment reattached.



Figure 5. Complete dental bonding.

Several authors have reported that morphologic buildup of traumatized teeth can be done with direct restorations of composite resin, ceramic crowns, or even tooth replacement in case of irreversible loss. <sup>4–7</sup> Bonding the fragment is the most conservative approach. By using the fragment, however, it is possible to restore function, tooth contour, surface texture, and a color compatible to the natural tooth.

The bonding option is possible because of adhesive dentistry resources and thus became the first choice of treatment for dental fractures.<sup>8–11</sup> In those cases where the fragment does not present a good fit, it is necessary to use adhesive restorative techniques to fill the gap.<sup>12, 13</sup> This clinical procedure is conservative, has a low cost, demands less clinical time, and is more convenient for the patient.

Although dental bonding has its advantages, there are some limitations that should be considered when choosing this procedure. The longevity of this technique is not predictable and the patient must be aware of its limitations. If the fragment is not kept in a moist environment, color change may occur compromising the esthetics and resulting in a visible line of fracture.<sup>3, 14</sup> Advantages and limitations of the procedure to be performed should be discussed with the patient when deciding which treatment will be carried out.

### ADVANTAGES AND DISADVANTAGES

Bonding a tooth fragment is an excellent treatment option for treating traumatic injuries of the anterior teeth. It is a quick and simple procedure that eliminates concerns about tooth color and temporary restorations. The fragment maintains the color, translucency, shine, texture, and shape of the original tooth.<sup>15</sup> Furthermore, the original frag-



Figure 6. Restoration at the 10-year follow-up.

ment is more resistant to stains and abrasion than resin restorations and causes fewer periodontal problems.<sup>16</sup> The procedure has a lower cost compared with a ceramic crown. Because the technique is simple, the fracture is easily solved, keeping the patient's well-being in mind.

The main disadvantage of dental bonding seems to be that the longevity of this procedure is not predictable, and in time, debonding of the fragment may occur. To increase the durability of the restoration, rehydration of the fragment has been proven to work. It is considered a key step in the treatment of fractured teeth and should be routinely conducted to avoid any flaws in the restoration of teeth that could compromise the bonding and esthetics; the fragment cannot regain its original color when it is dehydrated. The limitations can be controlled by strictly following the adhesive protocol in order to ensure good fracture resistance and to maintain a satisfactory esthetic appearance without compromising the remaining dental tissue. The same strictly following the same satisfactory esthetic appearance without compromising the remaining dental tissue.

### **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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### Patient Age and Dentists' Decisions About Occlusal Caries Treatment Thresholds

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### Clinical Relevance

Despite advances in cariology, variations in treatment threshold regarding when to intervene into occlusal carious lesions still exist among dentists. Patient age affects dentists' decisions about when to intervene surgically.

### **SUMMARY**

Objectives: This study was performed to 1) quantify dentists' treatment thresholds for occlusal primary caries; 2) determine if the patient's age affects dentists' decisions to surgically treat these carious lesions; and 3) test

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the hypothesis that patients', dentists', and practices' characteristics are significantly associated with surgical enamel intervention.

Methods: The study used a cross-sectional design consisting of a questionnaire survey in Japan. This study queried dentists working in outpatient dental practices who were affiliated with the Dental Practice-Based Research Network Japan (JDPBRN), which aims to allow dentists to investigate research questions and share experiences and expertise (n=282). Participants were asked whether they would surgically intervene in a series of cases depicting occlusal caries. Each case included a photograph of an occlusal surface displaying typical characteristics of caries penetration and written descriptions of adult and pediatric patients at high caries risk.

Results: In a case of a carious lesion within inner enamel, the proportion of dentists who indicated surgical intervention was significantly higher in adult patients (48%) when compared with pediatric patients (34%; p < 0.01). Logistic regression analysis showed that using a dental explorer for the diagnosis

of primary occlusal caries, type of practice, practice busyness, and percentage of patients who self-pay were significantly associated with dentists' decisions to intervene surgically into the inner enamel carious lesion.

Conclusions: These findings demonstrate that more than one-third of participants chose to intervene surgically into inner enamel carious lesions, and patients' age affects dentists' decisions about when to intervene surgically (clinicaltrials.gov registration number NCT01680848).

### INTRODUCTION

The diagnosis and treatment of primary dental caries are common procedures in general dental practice and are topics of extensive research.<sup>1,2</sup> Small-scale studies have shown that substantial variation exists among clinicians in restorative treatment thresholds.<sup>3-8</sup> At present, the only thresholds that can be definitely identified as inappropriate are those that call for surgical treatment when noncavitated caries is confined to enamel, because of the potential for enamel lesions to arrest or reverse.<sup>9</sup>

Previous studies by the Dental Practice-Based Research Network (DPBRN) and Dental PBRN Japan (JDPBRN), which included practitioners from the United States, Scandinavia, and Japan, revealed substantial variation among dentists in restorative treatment thresholds based on radiographic interproximal lesion depth. When the interproximal cavity is located in the enamel, findings for intervention proportions were as follows: Scandinavia, 0%-21%; the United States, 40%-75%<sup>10</sup>; and Japan, 47%-74%,<sup>11</sup> depending on patients' caries risk status on various clinical scenarios.

Regarding occlusal enamel carious lesions, several studies using a series of clinical photographs of the occlusal surface of a mandibular first molar have documented wide variation in the proportion of dentists who would intervene surgically into enamel when the caries is located in the inner half of the enamel: for adult patients, 6% in Sweden, 43%-8% in Scandinavian countries, and 63%-77% in the United States. 12 These results show that the treatment thresholds of occlusal primary caries differ among populations. However, no studies conducted in Japan have quantified differences in dentists' treatment thresholds for occlusal carious lesions, nor have they investigated differences between adult and pediatric patients.

The purposes of this study were to 1) quantify Japanese dentists' restorative treatment thresholds for occlusal primary caries; 2) determine if patient's age affects dentists' decisions to surgically treat these carious lesions; and 3) test the hypothesis that patients', dentists', and practices' characteristics are significantly associated with surgical enamel intervention.

### **METHODS AND MATERIALS**

### Study Design

We conducted a cross-sectional study consisting of a questionnaire survey in Japan between May 2011 and February 2012. We used the same questionnaire used in previous studies. <sup>10,13</sup> Four dentists and clinical epidemiologists translated these questionnaires into Japanese. The translated version of this questionnaire is available at http://www.dentalpbrn.org/uploadeddocs/Study%201(Japanese%20Version).pdf. Dentists were asked about assessment of caries diagnosis and treatment, treatment thresholds by hypothetical scenarios with clinical photographs, and patient and dentist background data. <sup>11</sup>

The network regions of the JDPBRN represent all seven districts in Japan (Hokkaido, Tohoku, Kanto, Chubu, Kansai, Chugoku-Shikoku, and Kyushu). Similar to the DPBRN, 12,14 every region has a regional coordinator who distributed and gathered the questionnaires. Dentists were asked to complete the questionnaire by hand and return to the assigned regional coordinator in a preaddressed envelope. Upon receipt, the regional coordinator reviewed the questionnaire for completeness.

### **Participants**

This study queried dentists working in outpatient dental practices who were affiliated with JDPBRN to investigate research questions and to share experiences and expertise (n=282). Participants were recruited from the JDPBRN Web site and mailings among those who indicated that they perform some measure of restorative dentistry at their practice. All participants provided informed consent prior to participation in this study.

The JDPBRN is a consortium of dental practices with a broad representation of practice types, treatment philosophies, and patient populations, having a shared mission with DPBRN, 14 which subsequently evolved to become the National Dental PBRN (http://NationalDentalPBRN.org). The recent establishment of the JDPBRN created an opportunity to make international comparisons. 11



Figure 1. Level of lesion severity/ depth for occlusal lesion. Reprinted with permission from the Norwegian Dental Journal (1997; 107: 66–74)<sup>15</sup>.

**Questions 1 and 2**: For each question, circle the letters that correspond to the treatment codes you would recommend for each of the four cases. You may circle more than one treatment code per case.

- 1. Suppose the patient is a 30-year-old woman with no relevant medical history. She has no complaints and is in your office today for a routine visit. She has been attending your practice on a regular basis for the past 6 years and has 12 teeth with existing dental restorations, heavy plaque and calculus, multiple Class V white spot lesions, and is missing five teeth.
- 2. Suppose the patient is a 12-year-old child with no relevant medical history. The patient is in your office today for the first time for a routine visit. She has five restorations and moderate plaque. A rubber dam cannot be used.

**Treatment codes**: a. No treatment today, follow the patient regularly, b. In-office fluoride, c. Recommend nonprescription fluoride, d. Prescription for fluoride, e. Use sealant or unfilled resin over tooth, f. Chlorhexidine treatment, g. Minimal drilling and sealant, h. Minimal drilling and preventive resin restoration, i. Air abrasion and a sealant, j. Air abrasion and preventive resin restoration, k. Amalgam restoration, l. Composite restoration, m. Indirect restoration.

### Hypothetical Scenarios With Clinical Photographs and Patient Background Data

Participants indicated their treatment decision from options presented for cases described in the questionnaire. A series of four clinical photographs of the occlusal surface of a mandibular first molar, together with a description, were presented portraying increasing depths of cavitation (Figure 1). We inquired about the treatment decision for each case with high caries risk under two different patient age scenarios (30 and 12 years old). The exact wording of each case scenario is provided in Figure 1. The same photographs showing occlusal surfaces with increasing caries severity were used in previous studies with the following descriptions: Case 1 had a white or discolored enamel surface, no cavitation. No radiographic signs of caries. Case 2 had minor loss of tooth substance with a break in the enamel surface or discolored fissures with gray/opaque enamel and/or caries confined to the enamel. No radiographic signs of caries. Case 3 had moderate loss of tooth substance and/or caries in the outer third of the dentin according to the radiograph. Case 4 had considerable loss of tooth substance and/or caries in the middle third of the dentin according to the radiograph (Figure 1).4,12,15 Cases 1 and 2 were enamel lesions located in the outer and inner enamel, respectively. Cases 3 and 4 were dentin lesions located in the outer and middle thirds of the dentin. For each case and each scenario, the respondent provided treatment codes in a "check all that apply" format (see Figure 1).

### Variable Selection

To identify characteristics associated with occlusal restorative treatment threshold, theoretical models employed in previous studies were used. 10,11,16,17 In addition, explanatory variables were extracted, consisting of four categories: dentists' individual characteristics (years since graduation from dental school, race/ethnicity, gender), practice setting (type of practice and busyness, patient waiting time for restorative dentistry, city population [government ordinance-designated city with population greater than 700,000 or not]), patient characteristics (dental insurance coverage, percentage of patients who selfpay, age and racial/ethnic distributions), and procedure-related characteristics (percentage of patient contact time spent each day doing restorative procedures, esthetic procedures, and extractions; whether or not caries risk is assessed as a routine part of treatment planning; percentage of patients

examined using a dental explorer for primary occlusal caries diagnosis, and receiving diet counseling).

### Statistical Analysis

Description of Treatment Thresholds—Treatment recommendations were classified into the following categories: I) no treatment (a. no treatment today, follow the patient regularly), II) preventive treatment (b. in-office fluoride, c. recommend nonprescription fluoride, d. prescription for fluoride, e. use sealant or unfilled resin over tooth, f. chlorhexidine treatment), III) minimally invasive treatment (g. minimal drilling and sealant, h. minimal drilling and preventive resin restoration, i. air abrasion and a sealant, j. air abrasion and preventive resin restoration), and IV) restorative treatment (k. amalgam restoration, l. composite restoration, m. indirect restoration). The overall variable had values with the following definitions: 1) no treatment (if only option I was endorsed), 2) preventive only (if only option II was endorsed), 3) minimally invasive (if option III was endorsed), and 4) restorative treatment (if only option IV was endorsed). We also determined the numbers (percentage) of dentists who would perform a nonsurgical treatment (1. no treatment and 2. preventive only) or surgical treatment (3. minimally invasive and 4. restorative treatment) for each case, 1 through 4. Chi-square tests were performed to assess the association between treatment thresholds and patient age.

Factors Affecting Decision to Intervene Into Inner Enamel Lesions—Descriptive analysis was conducted via univariate regression analysis for explanatory variables associated with dentists' use of a surgical treatment for inner enamel (Case 2). Subsequently, multiple logistic regression analysis was conducted to examine the relationship between explanatory variables and the decision to perform surgical treatment into inner enamel. Odds ratios were calculated together with the 95% confidence intervals (CIs). All analyses were performed using SPSS Statistics (version 19.0, IBM Corporation, Somers, NY, USA), with statistical significance set at p < 0.05.

### **RESULTS**

### **Demographic Information of Participants**

Questionnaires were distributed to 282 dentists; 189 (67%) were ultimately collected. Demographic characteristics of study participants are shown in Table 1.<sup>11</sup> The mean number of years elapsed since graduation from dental school was  $18.5 \pm 9.9$ ;

participants were predominantly male (n=154, 82%). Race/ethnicity was almost entirely Asian (n=186, 99%). With regard to type of practice, 41% (n=77) of participants were employed by another dentist. Regarding practice busyness, 40% (n=72) of dentists were able to provide care to all, but the practice was overburdened, while 33% (n=59) provided care to all, but the practice was not overburdened. Most dentists (n=159, 84%) used a dental explorer to diagnose primary occlusal caries. The percentage of patients who self-pay was 9%.

### **Treatment Thresholds**

Table 2 shows the distributions of treatment recommendations. In the adult-patient scenario, the percentage of participants who would perform surgical treatment decreased in the following order: case 4~(98%) > case~3~(76%) > case~2~(48%) > case~1~(14%). The same order was reported in the pediatric model: case 4~(97%) > case~3~(65%) > case~2~(34%) > case~1~(9%).

In case 2, the percentages of participants who would perform surgical treatment for adult and pediatric patients were 48% (n=88) and 34% (n=63), respectively, and 76% (n=140) and 65% (n=121) in Case 3. The proportion of dentists who indicated surgical intervention was significantly higher for the adult patient model than for the pediatric patient model in both case 2 (p<0.01) and case 3 (p<0.05).

### Factors Affecting the Decision to Intervene Surgically in Occlusal Inner Enamel Lesions

The results of the multiple logistic regression analysis are shown in Table 3. In the case 2 adult patient model, three factors were significantly associated with the decision to intervene surgically in enamel (odds ratios [95% CI]): type of practice 0.26 (0.11-0.62), percentage of patients who self-pay 1.04 (1.01-1.07), and practice busyness 3.72 (1.05-13.19). In the case 2 pediatric patient model, two factors were significantly associated with dentists' decision to intervene surgically in inner enamel (odds ratio [95% CI]): type of practice 0.38 (0.15-0.93) and using a dental explorer for a primary occlusal caries diagnosis 8.32 (1.84-37.71).

### DISCUSSION

In this study, most dentists chose not to restore an enamel lesion in the absence of dark brown pigmentation (as shown in case 1). Approximately one-third to one-half of participants chose to intervene surgi-

Number (%) or Mean ± SD  Positists' individual characteristics  Years since graduation from dental school (year)' (n=185)  Racoetimicity, n (%) (n=188)  Asian  186 (98.9)  White  1 (0.5)  Gender (male), n (%) (n=187)  Fractice busyness, n (%) (n=189)  Fractice busyness, n (%)	Table 1: Distribution of Dentists', Practices', Patients', and Dental Procedures' Characteristics of F others, 2012) <sup>11</sup>	Participants (Kakudate and
Years since graduation from dental school (year)* (n−185)         18.5 ± 9.9           Racelethnicity, n (%) (n=188)         166 (98.9)           White         1 (0.5)           Native Hawaiian or Other Pacific Islander         1 (0.5)           Gender (male), n (%) (n=187)         154 (82.4)           Practice busyness, n (%) (n=181)         157 (82.4)           Practice busyness, n (%) (n=181)         19 (10.5)           Provided care to all, but the practice was overburdened         72 (39.8)           Provided care to all, and the practice was not overburdened         59 (32.6)           Not busy enough         31 (17.1)           Wailing time for restorative dentistry, min* (n=182)         12.7 = 10.3           City population (government ordinance-designated city), n (%) (n=189)         76 (40.4)           Type of practice, n (%) (n=188)         77 (41.0)           Self-employed by another dentist         77 (41.0)           Self-employed without partners but share costs of office and/or assistants, etc         3 (1.6)           Self-employed without partners but share costs of office and/or assistants, etc         3 (1.6)           Self-employed as a partner in a complete partnership         3 (1.6)           Petient characteristics, %         8.5 ± 20.3           Dental insurance coverage* (n = 183)         88.5 ± 20.3 <t< td=""><td></td><td>Number (%) or Mean ± SD</td></t<>		Number (%) or Mean ± SD
Asian   186 (98.9)	Dentists' individual characteristics	
Asian   186 (86.9)	Years since graduation from dental school (year)* (n=185)	18.5 ± 9.9
Asian   186 (86.9)	Race/ethnicity, n (%) (n=188)	
White   1 (0.5)		186 (98.9)
Native Hawaiian or Other Pacific Islander	White	1 (0.5)
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Too busy to treat all people requesting appointments   19 (10.5)	Practice busyness, n (%) (n=181)	
Provided care to all, but the practice was not overburdened		19 (10.5)
Provided care to all, and the practice was not overburdened		
Not busy enough   31 (17.1)		59 (32.6)
Waiting time for restorative dentistry, min <sup>a</sup> (n=182)		31 (17.1)
City population (government ordinance-designated city), n (%) (n=189)         76 (40.4)           Type of practice, n (%) (n=188)         77 (41.0)           Employed by another dentist         77 (41.0)           Self-employed without partners and without sharing of income, costs, or office space         105 (55.9)           Self-employed without partners but share costs of office and/or assistants, etc         3 (1.6)           Self-employed as a partner in a complete partnership         3 (1.6)           Patient characteristics, %         0           Dental insurance coverage® (n=183)         88.5 ± 20.3           Percentage of patients who self-pay® (n=183)         8.6 ± 16.6           Patient age distribution, y°         11.18 (n=183)           1-18 (n=183)         16.1 ± 13.2           19-44 (n=188)         24.8 ± 11.0           45-64 (n=183)         30.4 ± 11.2           65+ (n=183)         28.5 ± 17.4           Racial/ethnic distribution®         30.3 ± 1.2           Black or African American (n=184)         0.3 ± 1.2           Black or African American (n=184)         0.01 ± 0.07           Asian (n=185)         98.9 ± 7.4           Native Hawaiian or Other Pacific Islander (n=184)         0.02 ± 0.2           Others (n=184)         0.07 ± 7.4           Dental procedure characteristics	· · · ·	
Type of practice, n (%) (n=188)		
Employed by another dentist         77 (41.0)           Self-employed without partners and without sharing of income, costs, or office space         105 (55.9)           Self-employed without partners but share costs of office and/or assistants, etc         3 (1.6)           Self-employed as a partner in a complete partnership         3 (1.6)           Patient characteristics, %         8.5           Dental insurance coverage* (n=183)         88.5 ± 20.3           Percentage of patients who self-pay* (n=183)         8.6 ± 16.6           Patient age distribution, y*         1.18 (n=183)           1-18 (n=183)         16.1 ± 13.2           19-44 (n=188)         24.8 ± 11.0           45-64 (n=183)         30.4 ± 11.2           65+ (n=183)         28.5 ± 17.4           Racial/ethnic distribution*         30.3 ± 12           Black or African American (n=184)         0.3 ± 12           Black or African American (n=184)         0.04 ± 0.2           American Indian or Alaska Native (n=184)         0.01 ± 0.07           Asian (n=185)         98.9 ± 7.4           Native Hawaiian or Other Pacific Islander (n=184)         0.02 ± 0.2           Others (n=184)         0.7 ± 7.4           Dental procedure characteristics         2.7 ± 7.2           Percentage of patient contact time spent each day doing extractions* (		
Self-employed without partners and without sharing of income, costs, or office space         105 (55.9)           Self-employed without partners but share costs of office and/or assistants, etc         3 (1.6)           Self-employed as a partner in a complete partnership         3 (1.6)           Patient characteristics, %            Dental insurance coverage* (n=183)         88.5 ± 20.3           Percentage of patients who self-pay** (n=183)         8.6 ± 16.6           Patient age distribution, y**            1-16 (n=183)         16.1 ± 13.2           19-44 (n=188)         24.8 ± 11.0           45-64 (n=183)         30.4 ± 11.2           65+ (n=183)         28.5 ± 17.4           Racialethnic distribution**            White (n=184)         0.3 ± 12           Black or African American (n=184)         0.04 ± 0.2           American Indian or Alaska Native (n=184)         0.01 ± 0.07           Asian (n=185)         98.9 ± 7.4           Native Hawaiian or Other Pacific Islander (n=184)         0.02 ± 0.2           Others (n=184)         0.7 ± 7.4           Dental procedure characteristics           Percentage of patient contact time spent each day doing restorative procedures* (n=183)         28.7 ± 14.2           Percentage of patient contact time spent each day doing esthetic pro		77 (41.0)
Self-employed without partners but share costs of office and/or assistants, etc   3 (1.6)		
Self-employed as a partner in a complete partnership   3 (1.6)		
Patient characteristics, %  Dental insurance coverage® (n=183) 88.5 ± 20.3  Percentage of patients who self-pay® (n=183) 8.6 ± 16.6  Patient age distribution, y® 1.18 (n=183) 16.1 ± 13.2  19-44 (n=188) 24.8 ± 11.0  45-64 (n=183) 30.4 ± 11.2  65+ (n=183) 30.4 ± 11.2  88.5 ± 17.4  Racial/ethnic distribution® 28.5 ± 17.4  Black or African American (n=184) 0.3 ± 1.2  Black or African American (n=184) 0.04 ± 0.2  American Indian or Alaska Native (n=184) 0.01 ± 0.07  Asian (n=185) 88.9 ± 7.4  Native Hawaiian or Other Pacific Islander (n=184) 0.02 ± 0.2  Others (n=184) 0.07 ± 7.4  Dental procedure characteristics  Percentage of patient contact time spent each day doing restorative procedures® (n=183) 8.8 ± 6.2  Caries risk is assessed as a routine part of treatment planning, n (%) (n=189) 49 (25.9)  Percentage of patient contact time spent each day doing extractions® (n=183) 8.8 ± 6.2  Caries risk is assessed as a routine part of treatment planning, n (%) (n=189) 49 (25.9)  Percentage of patient in whom a dental explorer was used for a primary occlusal caries lesion, n (%) (n=189) 1.24 51 (27.0)  25-49 12 (6.3)  50-74 20 (10.6)  75-99 29 (15.3)  100 (every time) 47 (24.9)  Percentage of patients who receive diet counseling® (n=183) 21.4 ± 27.2		` '
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1-18 (n=183)		
19-44 (n=188)       24.8 ± 11.0         45-64 (n=183)       30.4 ± 11.2         65+ (n=183)       28.5 ± 17.4         Racial/ethnic distribution <sup>a</sup>		16.1 + 13.2
45-64 (n=183)       30.4 ± 11.2         65+ (n=183)       28.5 ± 17.4         Racial/ethnic distribution*		
65+ (n=183)       28.5 ± 17.4         Racial/ethnic distribution <sup>a</sup> 0.3 ± 1.2         White (n=184)       0.3 ± 1.2         Black or African American (n=184)       0.04 ± 0.2         American Indian or Alaska Native (n=184)       0.01 ± 0.07         Asian (n=185)       98.9 ± 7.4         Native Hawaiian or Other Pacific Islander (n=184)       0.02 ± 0.2         Others (n=184)       0.7 ± 7.4         Dental procedure characteristics       Percentage of patient contact time spent each day doing restorative procedures <sup>a</sup> (n=183)       28.7 ± 14.2         Percentage of patient contact time spent each day doing esthetic procedures <sup>a</sup> (n=185)       4.5 ± 7.2         Percentage of patient contact time spent each day doing extractions <sup>a</sup> (n=183)       8.8 ± 6.2         Caries risk is assessed as a routine part of treatment planning, n (%) (n=189)       49 (25.9)         Percentage of patients in whom a dental explorer was used for a primary occlusal caries lesion, n (%) (n=189)       30 (15.9)         1-24       51 (27.0)         25-49       12 (6.3)         50-74       20 (10.6)         75-99       29 (15.3)         100 (every time)       47 (24.9)         Percentage of patients who receive diet counseling <sup>a</sup> (n=183)       21.4 ± 27.2		
Racial/ethnic distribution <sup>a</sup> White (n=184) 0.3 ± 1.2  Black or African American (n=184) 0.04 ± 0.2  American Indian or Alaska Native (n=184) 0.01 ± 0.07  Asian (n=185) 98.9 ± 7.4  Native Hawaiian or Other Pacific Islander (n=184) 0.02 ± 0.2  Others (n=184) 0.7 ± 7.4  Dental procedure characteristics  Percentage of patient contact time spent each day doing restorative procedures <sup>a</sup> (n=183) 28.7 ± 14.2  Percentage of patient contact time spent each day doing esthetic procedures <sup>a</sup> (n=185) 4.5 ± 7.2  Percentage of patient contact time spent each day doing extractions <sup>a</sup> (n=185) 4.5 ± 7.2  Percentage of patient contact time spent each day doing extractions <sup>a</sup> (n=183) 8.8 ± 6.2  Caries risk is assessed as a routine part of treatment planning, n (%) (n=189) 49 (25.9)  Percentage of patients in whom a dental explorer was used for a primary occlusal caries lesion, n (%) (n=189)  0 (never) 30 (15.9)  1.24 51 (27.0)  25-49 12 (6.3)  50-74 20 (10.6)  75-99 29 (15.3)  100 (every time) 47 (24.9)  Percentage of patients who receive diet counseling <sup>a</sup> (n=183) 21.4 ± 27.2		
White (n=184)       0.3 ± 1.2         Black or African American (n=184)       0.04 ± 0.2         American Indian or Alaska Native (n=184)       0.01 ± 0.07         Asian (n=185)       98.9 ± 7.4         Native Hawaiian or Other Pacific Islander (n=184)       0.02 ± 0.2         Others (n=184)       0.7 ± 7.4         Dental procedure characteristics       Percentage of patient contact time spent each day doing restorative procedures³ (n=183)       28.7 ± 14.2         Percentage of patient contact time spent each day doing esthetic procedures³ (n=185)       4.5 ± 7.2         Percentage of patient contact time spent each day doing extractions³ (n=183)       8.8 ± 6.2         Caries risk is assessed as a routine part of treatment planning, n (%) (n=189)       49 (25.9)         Percentage of patients in whom a dental explorer was used for a primary occlusal caries lesion, n (%) (n=189)       30 (15.9)         1-24       51 (27.0)         25-49       12 (6.3)         50-74       20 (10.6)         75-99       29 (15.3)         100 (every time)       47 (24.9)         Percentage of patients who receive diet counseling³ (n=183)       21.4 ± 27.2		20.0 2
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American Indian or Alaska Native (n=184)  Asian (n=185)  98.9 ± 7.4  Native Hawaiian or Other Pacific Islander (n=184)  0.02 ± 0.2  Others (n=184)  0.7 ± 7.4  Dental procedure characteristics  Percentage of patient contact time spent each day doing restorative procedures³ (n=183)  28.7 ± 14.2  Percentage of patient contact time spent each day doing esthetic procedures³ (n=185)  4.5 ± 7.2  Percentage of patient contact time spent each day doing extractions³ (n=185)  4.5 ± 7.2  Percentage of patient contact time spent each day doing extractions³ (n=183)  8.8 ± 6.2  Caries risk is assessed as a routine part of treatment planning, n (%) (n=189)  Percentage of patients in whom a dental explorer was used for a primary occlusal caries lesion, n (%) (n=189)  0 (never)  30 (15.9)  1-24  51 (27.0)  25-49  12 (6.3)  50-74  20 (10.6)  75-99  29 (15.3)  100 (every time)  Percentage of patients who receive diet counseling³ (n=183)  21.4 ± 27.2		
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Native Hawaiian or Other Pacific Islander (n=184)  Others (n=184)  Dental procedure characteristics  Percentage of patient contact time spent each day doing restorative procedures <sup>a</sup> (n=183)  Percentage of patient contact time spent each day doing esthetic procedures <sup>a</sup> (n=185)  Percentage of patient contact time spent each day doing extractions <sup>a</sup> (n=185)  Percentage of patient contact time spent each day doing extractions <sup>a</sup> (n=183)  Caries risk is assessed as a routine part of treatment planning, n (%) (n=189)  Percentage of patients in whom a dental explorer was used for a primary occlusal caries lesion, n (%) (n=189)  0 (never)  30 (15.9)  1-24  51 (27.0)  25-49  50-74  20 (10.6)  75-99  29 (15.3)  100 (every time)  Percentage of patients who receive diet counseling <sup>a</sup> (n=183)		
Others (n=184)  Dental procedure characteristics  Percentage of patient contact time spent each day doing restorative procedures <sup>a</sup> (n=183)  Percentage of patient contact time spent each day doing esthetic procedures <sup>a</sup> (n=185)  Percentage of patient contact time spent each day doing esthetic procedures <sup>a</sup> (n=185)  Percentage of patient contact time spent each day doing extractions <sup>a</sup> (n=183)  Respectively:  Percentage of patient contact time spent each day doing extractions <sup>a</sup> (n=183)  Respectively:  Percentage of patients in whom a dental explorer was used for a primary occlusal caries lesion, n (%) (n=189)  O (never)  O (never)  1-24  51 (27.0)  25-49  50-74  20 (10.6)  75-99  29 (15.3)  100 (every time)  Percentage of patients who receive diet counseling <sup>a</sup> (n=183)  21.4 ± 27.2		
Dental procedure characteristics  Percentage of patient contact time spent each day doing restorative procedures <sup>a</sup> (n=183)  Percentage of patient contact time spent each day doing esthetic procedures <sup>a</sup> (n=185)  Percentage of patient contact time spent each day doing extractions <sup>a</sup> (n=183)  Percentage of patient contact time spent each day doing extractions <sup>a</sup> (n=183)  Rank ± 6.2  Caries risk is assessed as a routine part of treatment planning, n (%) (n=189)  Percentage of patients in whom a dental explorer was used for a primary occlusal caries lesion, n (%) (n=189)  0 (never)  1-24  25-49  51 (27.0)  25-49  50-74  20 (10.6)  75-99  29 (15.3)  100 (every time)  Percentage of patients who receive diet counseling <sup>a</sup> (n=183)  21.4 ± 27.2	,	
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Caries risk is assessed as a routine part of treatment planning, n (%) (n=189)       49 (25.9)         Percentage of patients in whom a dental explorer was used for a primary occlusal caries lesion, n (%) (n=189)         0 (never)       30 (15.9)         1-24       51 (27.0)         25-49       12 (6.3)         50-74       20 (10.6)         75-99       29 (15.3)         100 (every time)       47 (24.9)         Percentage of patients who receive diet counseling <sup>a</sup> (n=183)       21.4 ± 27.2		
Percentage of patients in whom a dental explorer was used for a primary occlusal caries lesion, n (%) (n=189)         0 (never)       30 (15.9)         1-24       51 (27.0)         25-49       12 (6.3)         50-74       20 (10.6)         75-99       29 (15.3)         100 (every time)       47 (24.9)         Percentage of patients who receive diet counseling <sup>a</sup> (n=183)       21.4 ± 27.2		
0 (never)       30 (15.9)         1-24       51 (27.0)         25-49       12 (6.3)         50-74       20 (10.6)         75-99       29 (15.3)         100 (every time)       47 (24.9)         Percentage of patients who receive diet counseling <sup>a</sup> (n=183)       21.4 ± 27.2		` '
1-24       51 (27.0)         25-49       12 (6.3)         50-74       20 (10.6)         75-99       29 (15.3)         100 (every time)       47 (24.9)         Percentage of patients who receive diet counseling <sup>a</sup> (n=183)       21.4 ± 27.2		
25-49       12 (6.3)         50-74       20 (10.6)         75-99       29 (15.3)         100 (every time)       47 (24.9)         Percentage of patients who receive diet counseling <sup>a</sup> (n=183)       21.4 ± 27.2		` ,
50-74       20 (10.6)         75-99       29 (15.3)         100 (every time)       47 (24.9)         Percentage of patients who receive diet counseling <sup>a</sup> (n=183)       21.4 ± 27.2		
75-99 29 (15.3) 100 (every time) 47 (24.9) Percentage of patients who receive diet counseling <sup>a</sup> (n=183) 21.4 ± 27.2		` ,
100 (every time) 47 (24.9)  Percentage of patients who receive diet counseling <sup>a</sup> (n=183) 21.4 $\pm$ 27.2		` ,
Percentage of patients who receive diet counseling <sup>a</sup> (n=183) 21.4 ± 27.2		, ,
		` ,

	Case 1		Case 2		Case 3		Case 4	
	30 Years (n=185)	12 Years (n=185)	30 Years (n=185)	12 Years (n=186)	30 Years (n=184)	12 Years (n=185)	30 Years (n=183)	12 Years (n=183)
No treatment	103 (56%)	73 (39%)	41 (22%)	28 (15%)	14 (8%)	12 (6%)	1 (1%)	0 (0%)
Preventive only	57 (31%)	96 (52%)	56 (30%)	95 (51%)	30 (16%)	52 (28%)	3 (2%)	6 (3%)
Minimally invasive	18 (10%)	12 (6%)	57 (31%)	47 (25%)	66 (36%)	77 (42%)	24 (13%)	37 (20%)
Restorative	7 (4%)	4 (2%)	31 (17%)	16 (9%)	74 (40%)	44 (24%)	155 (85%)	140 (77%)
Nonsurgical treatment <sup>a</sup>	160 (86%)	169 (91%)	97 (52%)	123 (66%)	44 (24%)	64 (35%)	4 (2%)	6 (3%)
Surgical treatment <sup>b</sup>	25 (14%)	16 (9%)	88 (48%)	63 (34%)	140 (76%)	121 (65%)	179 (98%)	177 (97%)
p value <sup>c</sup>		0.19		<0.01		< 0.05		0.75

Nonsurgical treatment: no treatment and preventive only.

cally when the image pictured minor loss of tooth substance with a break in the enamel surface or discolored fissures with gray/opaque enamel (case 2) and most dentists (two-thirds to three-fourths) when the image pictured moderate loss of tooth substance and/or caries in dentin (case 3). Almost all participants chose to intervene surgically when considerable loss of tooth substance with caries involving dentin was visible (case 4).

In the presence of enamel surface integrity, caries lesions present in the enamel and/or dentin can be managed via remineralization therapies, <sup>18,19</sup> although the extent of remineralization is limited by the caries risk of the individual environment, as explained in the concept of caries balance. <sup>20,21</sup>

Consensus has been reached regarding the potential for noncavitated enamel lesions to reverse, and the restorative intervention of noncavitated caries confined to enamel is inappropriate. Therefore, the authors think interventions to the enamel lesion shown in case 1 and 2 are not necessary.

According to the result of the same scenario survey (N=519) by US DPBRN,  $^{12}$  the percentage of dentists who indicated surgical treatment in patients with high caries risk in cases 1, 2, and 3 were 25% (n=129), 77% (n=394), and 94% (n=482), respectively. However, in that study, subgroup analysis revealed that almost all dentists in Scandinavia chose not to restore lesions that were limited to enamel; restorative treatment was indicated pre-

Table 3: Factors Affecting Dentists' Decision to Intervene Surgically Into Inner Enamel Lesions According to Patient's Age<sup>a</sup> Variable **Adult Patient Scenario Pediatric Patient Scenario** OR 95% CI p Value OR 95% CI p Value Lower Upper Lower Upper Type of practice Employed by another dentist 1 Self-employed without partners and without sharing 0.26 0.11 0.62 0.002 0.38 0.15 0.93 0.034 of income, costs, or office space 1.07 Percentage of patients who self-paya 1.04 1.01 0.021 1.03 1.00 1.07 0.057 Practice busyness, n (%) Too busy to treat all people requesting appointments 1 1 Provided care to all, but the practice was overburdened 1.37 0.39 4.78 0.620 2.22 0.47 10.44 0.310 Provided care to all, and the practice was not overburdened 3.72 1.05 13.19 0.042 4.35 20.94 0.067 0.90 1.58 0.38 6.49 0.528 10.59 0.452 Not busy enough 1.92 0.35 Using a dental explorer for a primary occlusal 1.64 0.57 4.69 0.360 8.32 1.84 37.71 0.006 caries diagnosis<sup>b</sup> (reference: no use)

Surgical treatment: minimally invasive and restorative.

Chi-square test.

Abbreviation: CI, confidence interval.

<sup>&</sup>lt;sup>a</sup> Adjusted for gender, years elapsed since graduation from dental school, waiting time for restorative dentistry, city population, patient age distribution, and percentage of patient contact time spent each day doing restorative, esthetic, and extractions procedures and conducting caries risk assessment and percentage of patients who receive diet counseling in both adult and pediatric patient models. C statistic (area under the receiver-operating characteristic [ROC] curve) is 0.73 in the adult patient model and 0.70 in the pediatric patient model. Statistically significant odds ratios are highlighted in italic font.

<sup>b</sup> Continuous variable.

dominantly for occlusal surfaces that involved dentin. A previous study in Scandinavia reported a similar finding. 4 Given these reports, the Japanese dentists' treatment thresholds may be said to fall somewhere between US and Scandinavia levels. The current treatment strategy in Scandinavia is based on the diagnosis of caries activity, identification of the main causal and predisposing factors in individual cases, and assessment of caries risk. 12 This Scandinavian situation is a result of dental school education about cariology, current restrictive criteria for placement of the first restoration in Scandinavian dental practices, and high and predictable recall frequency among Scandinavians. 12,22

Results of multiple logistic regression analysis suggested that type of practice, percentage of patients who self-pay, practice busyness, and using a dental explorer for primary occlusal caries diagnosis were significantly associated with the decision to intervene surgically in an enamel lesion (case 2). A previous study by the US DPBRN using the same scenario also revealed that decisions to intervene surgically were associated with type of practice.<sup>12</sup> Further, usage of a dental explorer was associated with decisions to intervene surgically into proximal enamel carious lesions. 11 Taken together, these findings suggest that further dissemination of information on the appropriate use of dental explorers may help reduce surgical intervention into enamel.

In general, practitioners may treat patients who do not follow directions fully and who may not return for several years, thereby making a remineralization approach less effective. 20,21 That may be the reason why dentists feel hesitant to take a nonsurgical approach with patients, exemplified by the 12-yearold patient scenario. However, in the present study, the proportion of dentists who indicated surgical intervention into enamel was significantly higher for adult patients than for pediatric ones. Elderton's empirical work about the restorative cycle may underly why the profession is concerned about the adverse effects of intervening surgically before it becomes necessary. 23 Previous studies suggested that dentists intervened earlier in adult patients than in pediatric patients<sup>24,25</sup> in the 1980s. In addition, in the early 1990s, the concept of minimally invasive dentistry advanced rapidly. 26,27 Therefore, it is possible that the participants believed that delayed surgical intervention among pediatric patients may improve first molar longevity.

This study featured a relatively wide variety of participants, with respondents hailing from all over

Japan. The age and gender distribution of this study sample was similar to the actual distribution in Japan,<sup>28</sup> thereby enhancing the generalizability of the findings. However, the study findings should be evaluated with caution. First, participants were not selected by random sampling. Second, given the cross-sectional nature of our study, causative relationships between factors and use of an enamelbased surgical treatment threshold were difficult to assess. Lastly, a clinical photograph and a patient scenario cannot replicate all of the nuances that can be perceived in a real tooth and a patient caries risk status; we therefore cannot state with certainty that the decision-making context provided by this questionnaire entirely duplicated the real-world clinical context.

### CONCLUSION

More than one-third of the participants chose to intervene surgically into occlusal enamel. The translation of research findings to clinical practices is a complex matter. <sup>29,30</sup> In an effort to improve dentists' clinical decision-making regarding occlusal enamel intervention, results of this study will be communicated to dentists, and the effect of this dissemination will be evaluated for its impact on routine clinical practice.

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### **Conflict of Interest**

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

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## In Vitro Progression of Artificial White Spot Lesions Sealed With an Infiltrant Resin

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### Clinical Relevance

Infiltration of initial *in vitro* enamel lesions by resin seems to reduce or even stop the progression of carious lesions.

### **SUMMARY**

This study assessed the ability of an infiltrant resin (Icon, DMG Chemisch-Pharmazeutische Fabrik GmbH, Hamburg, Germany) to prevent artificial lesion progression *in vitro* when used to impregnate white spot lesions and also

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assessed the effect of saliva contamination on resin infiltration. Enamel specimens (n=252) were prepared and covered with nail varnish, leaving a window of sound enamel. After demineralization (pH 5.0; four weeks), specimens were divided into six groups (n=42 per group): group 1, 2% fluoride gel (positive control); group 2, resin infiltrant; group 3, resin infiltrant + fluoride gel; group 4, no treatment (negative control); group 5, resin infiltrant application after saliva contamination; and group 6, resin infiltrant + fluoride gel after saliva contamination. Specimens from each group were cut perpendicular to the surface, and one-half of each specimen was exposed to a demineralizing solution for another four weeks. The other half was set aside as a record of initial lesion depth and was used later in the determination of lesion progression. Lesion progression and infiltrant penetration were measured using confocal laser scanning microscopy (CLSM) and transverse microradiography (TMR). For lesion depth, based on CLSM, groups 2 and 3 showed the least changes when submitted to demineralization challenge, followed by group 1, then groups 5 and 6, and finally group 4. There were no significant differences between groups 2

and 3 or groups 5 and 6 in their ability to inhibit further lesion progression (p < 0.05). Based on TMR, groups 2 and 3 also showed the fewest changes when submitted to demineralization challenge, followed by group 5, then groups 1 and 6, and finally group 4. In terms of mineral loss as measured by TMR, all groups that contained fluoride (groups 1, 3, and 6) show less percentage change in mineral loss than the groups that did not contain fluoride (groups 2, 4, and 5). It can be concluded that infiltrant penetration into early enamel lesions inhibited further demineralization in vitro, especially in the presence of fluoride. Saliva contamination decreased the ability of the infiltrant to prevent further demineralization, but the presence of fluoride seemed to counteract this effect.

### INTRODUCTION

Dental caries is one of the most prevalent chronic diseases globally, with individual susceptibility lasting a lifetime. Early recognition of the disease process (before cavitation) is important to implement intervention in an attempt to stop and even reverse the disease process (remineralization of the noncavitated lesion). Within the modern concept of dental caries management, prevention and hardtissue preservation are the primary goals, and dentists are encouraged to prefer a more conservative and biological rather than a surgical approach. 1 Common nonoperative treatment for enamel caries includes fluoride application, sealants, and behavioral modification. For pits and fissures, mainly on the occlusal surfaces of permanent molars, sealing with light-curing resins has been shown to be an effective preventive measure.<sup>2,3</sup> A promising alternative therapy to arrest caries lesions on proximal surfaces might be the infiltration of subsurface lesions with low-viscosity, light-curing resins. Early white spots have increased enamel porosity. Since porosities of enamel caries lesions act as diffusion pathways for acids and dissolved minerals, infiltration of these pores with low-viscosity resins might occlude the pathways and thus hamper or arrest caries progression.4 These same porosities can also be the ideal loci for infiltration of adhesives.<sup>5</sup>

Reduction in pore volume after the sealing of artificial initial enamel lesions has been reported in several studies<sup>5-14</sup> either by dental adhesives<sup>15-17</sup> or by fissure sealants. However, dental sealants and adhesives are not optimized for high penetrability and have therefore shown only superficial penetra-

tion into natural enamel lesions. 18 Special resins, optimized for rapid capillary penetration (so-called infiltrants), penetrate significantly deeper. 19 In laboratory experiments, resin-infiltrated enamel lesions without a covering resin coat showed a significantly reduced lesion progression in a demineralizing environment compared to untreated lesions. 14,15 The aim of caries infiltration is to saturate the porous lesion body with a low-viscosity resin (infiltrant) that is subsequently hardened with blue light. 4,18,19 Thereby, diffusion pathways for cariogenic acids are blocked and lesions sealed. However, in contrast to conventional caries sealing, <sup>20,21</sup> with this technique, the diffusion barrier is created inside the lesion and not on the surface, facilitating clinical application, especially in the interproximal space.<sup>22</sup> Recently, a resin based on this concept was introduced to the market (Icon, DMG Chemisch-Pharmazeutische Fabrik GmbH, Hamburg, Germany). This material, composed of triethylene-glycol-dimethacrylate-based resin, bisphenol A glycerolate dimethacrylate, camphorquinone, and ethyl 4-(dimethylamino) benzoate and ethanol, has an extremely high penetration coefficient that facilitates deeper penetration. However, little is known about the performance of the new infiltrant resin on the progression of white spot lesions<sup>23</sup> or the effect that salivary contamination might have on the penetration of the infiltrant. Studies show that one second of contact between saliva and etched enamel is enough to noticeably modify enamel topography.<sup>24</sup> Furthermore, Taskonak and Sertgoz reported that etched enamel absorbs salivary components, decreasing surface energy and impairing potential adhesion.<sup>25</sup> Therefore, it can be assumed that infiltrant, as it is based on the same principle, would likely suffer the same consequences of saliva contamination.

As a noninvasive treatment, the use of topical fluoride associated with plague removal is indicated to promote lesion remineralization.<sup>26</sup> Remineralization is the natural repair process for noncavitated lesions and relies on calcium and phosphate ions, assisted by fluoride, to rebuild a new surface on existing crystal remnants in subsurface lesions remaining after demineralization. Fluoride ions incorporate into remineralizing enamel/dentin, changing carbonated apatite to a fluoroapatite-like form that is more acid tolerant and makes the hard tissues more acid resistant.26 To date, there are no data comparing the preventive effect of resin infiltrant, fluoride application, or a combination of the two on lesion progression. Therefore, the purpose of this study was to assess the in vitro ability of an

infiltrant resin to impregnate artificial white spot lesions to prevent lesion progression when done alone and also in conjunction with fluoride treatment. Additionally, as a secondary objective, this study assessed the effect of saliva contamination on resin infiltration. The null hypotheses tested were 1) that caries progression was not altered by the different treatments tested 2) that there was no effect of saliva contamination on resin infiltration.

### **METHODS AND MATERIALS**

### Sample Preparation

Enamel slabs (approximately  $5 \times 5 \text{ mm}^2$ ) were prepared from the facial aspect of bovine incisors stored in an agueous 0.1% thymol solution. The slabs were cut from the middle third of the labial coronal surfaces. Initially, the pulpal surface of the slabs was flattened in a grinding machine (Roto-Pol31/Roto-Force4 polishing unit, Struers, Westlake, OH, USA). Next, the external/experimental surface was sequentially flattened with 500-, 1200- and 4000-grit silicon carbide grinding papers (MD-Fuga, Struers) and then polished (1-µm diamond suspension; Struers). Specimens with white spots, cracks, or any other defect were discarded. The remaining specimens were mounted on an acrylic rod with sticky wax, and their baseline surface microhardness (SMH) was determined by the mean length (Lb) of five indentations placed 100 µm apart from each other in the center of the specimen using a Knoop diamond indenter with a load of 50 g and a dwell time of 15 seconds (2100 HT, Wilson Instruments, Norwood, MA, USA).<sup>27</sup> Only specimens with baseline SMH between 300 and 350 KHN were selected for the study (n=252).

### **Preparation of Caries-Like Lesions**

The enamel surface of each specimen was partly covered with acid-resistant nail varnish, leaving an experimental window of sound enamel of about  $5 \times 3$ mm. Specimens were then demineralized using a hydroxyethylcellulose (HEC) acid gel. The gel was prepared by adding HEC (Sigma 54290, Cellosize QP-40, 80-125 cP, at a ratio of 140 g/L) to a pH 5.0adjusted solution containing 0.05 M lactic acid. The solution was continuously stirred until the HEC was partially hydrolyzed (about 30 minutes). The gel was then poured into a container and placed into an incubator set at 37°C for about 24 hours. Specimens were placed into a second, sealable container, and fully hydrolyzed (ie, clear) HEC gel was poured over them. The specimens were demineralized at 37°C for four weeks. The SMH of the enamel specimens was

measured again (SMH1). Indentations were spaced  $100~\mu m$  from each other and from the baseline indentation sites.

### **Treatment of White Spot-Like Lesions**

All specimens were balanced according to microhardness values (SMH1) and divided into six groups (n=42 per group; Figure 1). In group 1 (positive control), enamel specimens were treated with a fluoride gel (2% NaF, neutral ssodium fluoride) for 4 minutes, which was removed by rinsing the specimens under running distilled water for 2 minutes. For all groups that received the infiltrant (groups 2, 3, 5, and 6), the indirect staining technique<sup>28</sup> was used prior to application of the infiltrant: the surface was dried with compressed air for five seconds and etched for five seconds with 37% phosphoric acid, and specimens were stored in an ethanolic solution of 0.1% Rhodamine B isothiocyanate (RITC) for 12 hours to dehydrate and label all accessible porosities with the red fluorophore. Because etching with phosphoric acid removes the surface layer in bovine teeth, HCl was not used. Group 2 specimens were dried using compressed air for 10 seconds, and pure infiltrant (ICON preproduct) was applied onto the lesion surface. In order to ensure that inhibition of lesion progression was achieved only by infiltration of the lesion body and not by a superficial resin layer, after five minutes resin surplus was removed from the surface using a cotton roll, and the material was then light cured for 60 seconds (530 mW/cm<sup>2</sup>; Astralis 5; Ivoclar Vivadent, Schaan Liechtenstein). To bleach all red fluorophore that had not been enclosed by infiltrant. specimens were stored in 30% hydrogen peroxide solution for 12 hours at 37°C. Subsequently, specimens were washed with water for 60 seconds. Group 3 specimens were first treated the same way as group 2, and then fluoride gel (2% NaF, neutral sodium fluoride) was applied for 4 minutes. Subsequently, the fluoride gel was removed by rinsing in distilled water for two minutes. Group 4 specimens served as the negative control and received no treatment. Group 5 specimens were treated the same way as group 2 (dry, acid etch, and label with red fluorophore) and then exposed to saliva before placing the resin infiltrant (after the acid-etching step). Stimulated, frozen pooled whole human saliva collected under Indiana University Institutional Review Board (IRB) approval (IRB no. 1105005588) was microbrushed onto the selected specimens, left undisturbed for five seconds, and gently air blown for three to five seconds. Subsequently, resin

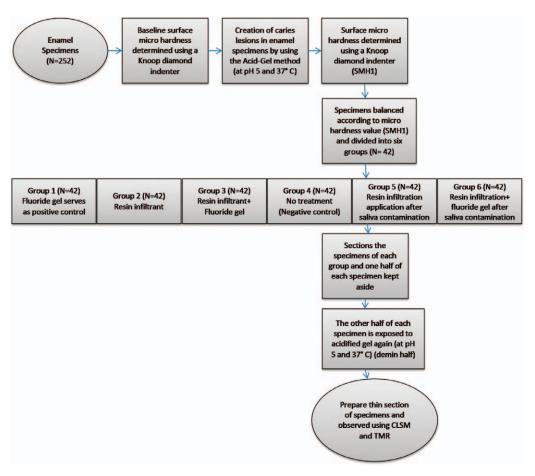


Figure 1. Schematic representation of specimen preparation.

infiltrant application and storing specimens in 30% hydrogen peroxide took place as previously described.

In group 6, specimens were dried, acid etched, labeled with red fluorophore, and then exposed to saliva. The resin infiltrant application and storing in 30% hydrogen peroxide took place after the saliva exposure. This was followed by a four-minute application of fluoride gel (2% NaF, sodium fluoride gel), which then was removed by rinsing in distilled water for 2 minutes.

In order to evaluate the progression of sealed lesions in a demineralizing environment, specimens from each group were cut perpendicular to their surface using a hard-tissue microtome yielding two halves for each lesion. One-half of each specimen (initial half) was set aside to be used later as a record of initial lesion depth when determining lesion progression. For the remaining specimen halves (demineralization half), the cut surface of each specimen was covered with nail varnish and treated as per above, and the specimens exposed to the

hydroxyethylcellulose gel for another four weeks to simulate a cariogenic environment.

### Confocal Laser Scanning Microscopy and Transverse Microradiography

The nail varnish covering the cut surface of each of the demineralization specimen halves was removed with acetone. For examination by confocal laser scanning microscopy and transverse microradiography (TMR), thin sections (100 µm) of both initial and demineralization halves of each specimen were prepared. First, specimens were observed using a confocal laser scanning microscope (Olympus Fluoview FV1000 MPE, Olympus, Center Valley, PA, USA) at the Indiana Center for Biological Microscopy (Indianapolis, IN, USA). To visualize the pore structures of the uninfiltrated portion of the lesions, both halves of each specimen were immersed in 50% ethanol solution containing 100 µM sodium fluorescein (Sigma Aldrich, Saint Louis, MO, USA) for 10 minutes. Subsequently, specimens were thoroughly washed in deionized water for three minutes and

Group	n		Confocal Lesion Depth (in µm	)
		Initial Mean (SE)	After-Challenge Mean (SE)	% Change <sup>a</sup> Mean (SE)
1. Fluoride (F)	42	110.1 (2.7)	146.4 (4.1)	33.9 (3.1) A
2. Icon	42	116.2 (3.3)	121.0 (3.2)	4.3 (0.6) в
3. lcon + (F)	42	106.9 (3.1)	111.0 (3.1)	4.0 (0.6) в
4. No treatment	42	109.7 (2.2)	172.3 (3.7)	58.3 (3.3) c
5. Saliva contamination + Icon	42	116.1 (3.7)	165.0 (5.3)	42.3 (2.2) D
6. Saliva contamination + Icon + (F)	42	108.4 (3.2)	155.1 (4.8)	43.4 (2.7) D

visualized with a confocal laser scanning microscope; a 488-nm excitation light and 500-545-nm emission filter band was used to visualize fluorescein isothiocyanate, and a 559-nm excitation laser and 570-650-nm emission filter band was used for RITC. Images were recorded with a lateral resolution of 1024  $\times$  1024 pixels (635  $\times$  635  $\mu m)$  and analyzed using Metamorph imaging software (Molecular Devices, Downingtown, PA, USA).

At three defined points per image (depending on the lesion depth indicated by a 50-um grid), both initial lesion depth and resin penetration depth were measured and their mean values calculated. The lesion depth was defined as the distance from the surface of the specimen to the point where the prism cores were no longer fluorescent. 14 Lesion depth was measured for each specimen individually. In the case of complete infiltration, the initial lesion depth was the same as the resin penetration depth. If the lesion depth could not be exactly determined in "demin" halves due to complete infiltration, it was assumed that the lesion had not progressed. Subsequently, thin sections were examined using TMR.<sup>29</sup> Lesion depths as well as integrated mineral loss for both lesion halves were measured using TMR software (TMR for Windows, version 2.0.27.2, Inspektor Research System, Amsterdam, The Netherlands). Lesion progression was then evaluated by subtracting lesion depths of paired "initial" halves from "demin" halves. 15 The outcomes measure was depth of penetration of the resin infiltrant for both halves of the specimens and lesion depth and mineral loss on both halves of the specimens.

#### **Statistical Analysis**

For each treatment group, the significance of the lesion depth and mineral loss changes between the treatment and demineralization periods was tested using Wilcoxon signed rank tests. The treatment groups were compared for differences in percentage

change in lesion depth and percentage change in mineral loss using Wilcoxon rank sum tests. Resin penetration depth was calculated as the average penetration from the two halves of each specimen. Penetration depth was also compared between groups using Wilcoxon rank sum tests.

#### **RESULTS**

With the new indirect staining technique, the confocal microscopic images obtained in dual fluorescence mode showed red-infiltrated structures (RITC), whereas porous structures (noninfiltrated part of the lesion) appeared green due to staining with NaFl. Nonporous structures, like sound enamel, showed no fluorescence and were displayed dark.

A total of 10 specimens were damaged during preparation for TMR. Thus, averages of 39-40 specimens from each group were analyzed by TMR. Based on confocal lesion depth, group 2 (resin infiltrant) and group 3 (resin infiltrant + fluoride) showed the fewest changes when submitted to the demineralization challenge, followed by group 1 (fluoride), group 5 (saliva contamination + resin infiltrant), and group 6 (saliva contamination + resin infiltrant + fluoride), then group 4 (negative control, no treatment; Table 1). There were no significant differences between groups 2 and 3 or groups 5 and 6 in their ability to inhibit further lesion progression.

When analyzing lesion depth using TMR, group 2 (resin infiltrant) and group 3 (resin infiltrant + fluoride) showed the fewest changes when submitted to the demineralization challenge, followed by group 5 (saliva contamination + resin infiltrant), then group 6 (saliva contamination + resin infiltrant + fluoride) and group 1 (fluoride), then group 4 (negative control, no treatment; Table 2).

In terms of mineral loss as measured by TMR, all groups that included fluoride (groups 1, 3, and 6)

Table 2:	Change in	Transverse	Microradiography	Lesion	Depth	and N	1ineral	Loss	Measurements .	After	Demineraliza	tion
	Challenge											

Group	n	TMR Lesion Depth (in μm)			TMR Mi	neral Loss (in vol%	vol% × μm)	
		Initial Mean (SE)	After-Challenge Mean (SE)	% Change <sup>a</sup> Mean (SE)	Initial Mean (SE)	After-Challenge Mean (SE)	% Change <sup>a</sup> Mean (SE)	
1. Fluoride (F)	41	155.1 (1.8)	197.1 (3.0)	27.6 (2.2) A	2267 (49)	3076 (111)	37.2 (5.1) A	
2. Icon	40	187.4 (3.3)	190.9 (3.4)	1.9 (0.3) в	2546 (70)	3910 (109)	57.1 (5.5) в	
3. lcon + F	42	195.1 (3.6)	197.3 (3.5)	1.2 (0.2) в	2852 (87)	3605 (87)	31.4 (5.0) A	
4. No treatment	39	155.2 (2.4)	221.8 (4.1)	<b>43.8 (3.0)</b> c	2467 (61)	3991 (133)	63.8 (5.8) в	
5. Saliva contamination + Icon	40	177.5 (3.0)	207.5 (3.2)	17.8 (2.2) D	2477 (63)	3811 (101)	57.1 (5.5) в	
6. Saliva contamination + Icon + F	40	151.9 (3.4)	187.8 (3.1)	24.9 (2.2) A	2428 (82)	3259 (70)	39.7 (5.3) A	

<sup>&</sup>lt;sup>a</sup> Percentage change after challenge within group significantly different at p<0.0001. Statistical significance (p≤0.03) between groups indicated by different letters.

Table 3: Measurements of Infiltrant Resin Penetration by Confocal Microscope<sup>a</sup>

Group	n	Lesion Depth	Infiltrant Resin Penetration <sup>a</sup>					
Icon	42	116.2 (3.3)	112.8 (2.9) A					
Icon + F	42	106.9 (3.1)	103.0 (2.9) в					
Saliva contamination + Icon	42	116.1 (3.7)	110.1 (3.3) ав					
Saliva contamination + Icon + F	42	108.4 (3.2)	106.0 (2.5) AB					
a Statistical significance (p =0.004) be	two	n aroune indi	nated by different					

<sup>&</sup>lt;sup>a</sup> Statistical significance (p=0.004) between groups indicated by different letters.

showed less percentage change in integrated mineral loss than the groups that did not include fluoride (groups 2, 4, and 5; Table 2). Resin penetrated

significantly deeper in group 2 (resin infiltrant;  $p{=}0.0041$ ) than in group 3 (resin infiltrant + fluoride), but there were no other statistically significant differences in penetration depth among other groups (Table 3). Although saliva did not interfere with the penetration of resin, the resin layer was not homogeneous, as seen in Figure 2, and thus there was greater lesion progression in salivacontaminated groups (Tables 1 through 3).

#### DISCUSSION

Resin infiltration of enamel lesions aims to reduce or even stop the progression of white spot lesions based on the available clinical and laboratory stud-

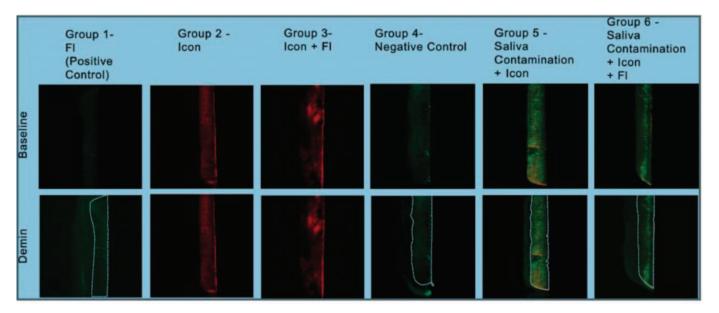


Figure 2. Representative confocal laser scanning microscopy (CLSM) images of corresponding lesion halves of all groups. On the images of the "demin" half, the extent of the corresponding baseline lesion is indicated by a dotted line. Group 1 shows slight progression of lesion, and groups 2 and 3 infiltrated the complete lesion body with resin. Here, no progression of lesion depth could be observed using CLSM. Group 4, the untreated control in the "demin" half, progressed significantly compared to the baseline. In groups 5 and 6, the lesion is not homogeneously infiltrated, and progression of lesion depth could be observed. Notice the inhomogeneity of the resin layer in groups 5 and 6; the inhomogeneous layer of resin tags appears as a mix of red and green (ie, yellow) or more green.

ies. $^{11,15,19,23,30,31}$  This novel technique might bridge the gap between noninvasive and minimally invasive treatment of initial dental caries, postponing as long as possible the need for a restoration.<sup>23</sup> Fluoride's ability to inhibit or even reverse the initiation and progression of dental caries is well documented.<sup>32</sup> As a prophylactic, the frequent use of fluorides for the noninvasive treatment of initial enamel lesions is generally recommended, and remineralization of the lesion may be obtained by improving the patient's oral hygiene and the use of fluoride toothpaste. The aim of the current study was to evaluate the synergistic effect of treatment with the resin infiltration technique and remineralization of enamel caries with fluoride gel on the progression of initial dental caries. According to the results of this study, all the treatments tested hampered lesion progression although at different levels. The first null hypothesis of the study was rejected, as caries progression differed among the different treatments (fluoride, resin infiltration + fluoride, or resin infiltration). After the new acid challenge, lesion depth values for the groups treated with resin infiltrant and resin infiltrant + fluoride were similar and exhibited less change than specimens in the group treated with fluoride only (Tables 1 and 2). This is in agreement with other studies indicating that resin infiltration can assist in hampering the progression of dental caries. 19,28,30 However, in relation to mineral loss, the resin infiltrant + fluoride group showed less change in mineral loss values compared with the resin infiltrant group alone (Table 2).

To date, no study has shown the effect of saliva contamination on resin infiltration. In this investigation, when the lesions were contaminated with saliva prior to the application of the resin infiltrant, there was no effect on the penetration of the infiltrant. However, due to saliva contamination, the lesion was not homogeneously infiltrated, and the lesions were not protected from further progression (Tables 1 and 2). Thus, in agreement with a previous study, this indicates that not only a deep infiltration but also a homogeneous resin layer within the lesion body is essential for a leakproof seal. 14 In terms of mineral loss, group 6 (saliva contamination + resin infiltration + fluoride) showed less change in mineral loss than group 5 (saliva contamination + resin infiltration), indicating that in the presence of fluoride, saliva contamination did not have a strong negative effect. Although short-term contamination with saliva did not alter the penetration of the infiltrant, it reduced

the ability of resin infiltration to hamper the progression of early enamel lesions, and thus proper isolation should be performed during application of resin infiltrant. In case of inadvertent saliva contamination, an application of topical fluoride is recommended. It should be taken into consideration that an artificial bovine enamel lesion model was used and that the mean lesion depths of the initial lesions were on average 110  $\mu m$ . This limits the validity of the study because, under clinical situations, the enamel lesions to be resin infiltrated are usually deeper (500-900  $\mu m$ ). More studies are needed to confirm the efficacy of resin infiltration in conjunction with fluoride treatment in clinical conditions.

#### CONCLUSION

Resin infiltrant, especially in combination with fluoride, has great potential for inhibiting further progression of small white spot lesions. Saliva contamination can negatively affect lesion progression, but fluoride can counteract this effect.

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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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## Immediate Adhesive Properties to Dentin and Enamel of a Universal Adhesive Associated With a Hydrophobic Resin Coat

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#### **Clinical Relevance**

Multi-mode adhesives may be optimized when they are used as self-etch adhesives on dentin with a separate enamel etching step.

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#### **SUMMARY**

Objectives: To evaluate the effect of acid etching and application of a hydrophobic resin coat on the enamel/dentin bond strengths and degree of conversion (DC) within the hybrid layer of a universal adhesive system (G-Bond Plus [GB]).

Methods: A total of 60 extracted third molars were divided into four groups for bondstrength testing, according to the adhesive strategy: GB applied as a one-step self-etch adhesive (1-stepSE); GB applied as in 1-stepSE followed by one coat of the hydrophobic resin Heliobond (2-stepSE); GB applied as a two-step etch-and-rinse adhesive (2-stepER); GB applied as in 2-stepER followed by one coat of the hydrophobic resin Heliobond (3-stepER). There were 40 teeth used for enamel microshear bond strength (uSBS) and DC; and 20 teeth used for dentin microtensile bond strength (µTBS) and DC. After restorations were constructed, specimens were stored in water (37°C/24 h) and then tested at 0.5 mm/min (μTBS) or 1.0 mm/min (μSBS). Enamel-resin

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and dentin-resin interfaces from each group were evaluated for DC using micro-Raman spectroscopy. Data were analyzed with two-way analysis of variance for each substrate and the Tukey test ( $\alpha$ =0.05).

Results: For enamel, the use of a hydrophobic resin coat resulted in statistically significant higher mean enamel  $\mu SBS$  only for the ER strategy (3-stepER vs 2-stepER, p<0.0002). DC was significantly improved for the SE strategy (p<0.00002).

For dentin, the use of a hydrophobic resin coat resulted in significantly higher dentin mean  $\mu$ TBS only for the SE strategy (2-stepSE vs 1-stepSE, p < 0.0007). DC was significantly improved in groups 2-stepSE and 3-stepER when compared with 1-stepSE and 2-stepER, respectively (p < 0.0009).

Conclusions: The use of a hydrophobic resin coat may be beneficial for the selective enamel etching technique, because it improves bond strengths to enamel when applied with the ER strategy and to dentin when used with the SE adhesion strategy. The application of a hydrophobic resin coat may improve DC in resindentin interfaces formed with either the SE or the ER strategy. On enamel, DC may benefit from the application of a hydrophobic resin coat over 1-stepSE adhesives.

#### INTRODUCTION

A new family of dental adhesives, known as "universal" or "multi-mode" adhesive systems, has been recently introduced. These novel adhesives give the dentist the opportunity to decide which adhesive strategy to use: etch-and-rinse (ER) or self-etch (SE). This versatile new adhesion philosophy advocates the use of the simplest option for each strategy (ie, one-step self-etch [1-stepSE] or two-step etch-and-rinse [2-stepER] adhesive).

It has been reported that 1-stepSE adhesives result in water permeability in dentin,<sup>4-6</sup> as well as osmotic blistering in enamel, which may affect clinical durability.<sup>7</sup> Due to equivalent water contents between 1-stepSE adhesives and the new multi-mode adhesive solutions, degradation of the bonding interface might also occur with the latter. In fact, a recent *in vitro* study<sup>3</sup> showed that a multi-mode adhesive applied as a 2-stepSE resulted in higher bond strengths and increased degree of conversion, compared with other simplified universal adhesives. On the other hand, prior application of phosphoric acid with multi-mode

adhesives improved the bond strength to enamel but negatively affected the dentin hybridization quality.<sup>8</sup> It is still controversial whether acid etching prior to the application of 1-stepSE adhesives affects the respective dentin bond strengths.<sup>9-12</sup>

One-step SE adhesives result in thin adhesive layers that are prone to polymerization inhibition by oxygen. The monomer solutions are composed of high concentrations of hydrophilic and/or ionic resin monomers. The presence of up to 40% water in the composition of SE adhesives triggers the dissociation of the weak acidic methacrylate monomers into ionized forms for permeation into the smear layer and underlying mineralized dentin. 14,16

However, excess water may reduce the performance of adhesives by inhibiting the optimal copolymerization of the adhesive monomers, <sup>17,18</sup> leading to phase separation. <sup>19,20</sup> These mechanisms compromise the final structure of the polymer and its mechanical properties, <sup>21</sup> accelerating degradation and resulting in lower resin-dentin bond strength. <sup>22-24</sup> Therefore, the placement of an additional hydrophobic resin coat has been advocated to increase the performance of 1-stepSE adhesives both *in vitro* <sup>25-28</sup> and clinically. <sup>29,30</sup> Due to the recent introduction of multi-mode adhesives, there is no evidence of the effect of a hydrophobic bonding resin on their behavior.

Dissolved hydroxyapatite crystals and residual smear layer are incorporated in the hybridized complex of SE adhesives. <sup>31,32</sup> Except for very acidic SE systems, <sup>33,34</sup> the whole extension of the demineralized dentin depth is impregnated with resin monomers, preventing the technique sensitivity characteristic of bonding to moist etched dentin. <sup>35-37</sup> A disadvantage of the SE approach, specifically one-step adhesives, is the reduction in enamel bonding effectiveness. <sup>38,39</sup> The increase in surface area in intact and ground enamel obtained with SE adhesives is lower than that achieved with phosphoric acid. <sup>39</sup> The performance of 1-stepSE adhesives improves when enamel is etched with phosphoric acid. <sup>10</sup>

This study compared the enamel microshear ( $\mu SBS$ ) and dentin microtensile bond strengths ( $\mu TBS$ ) of the multi-mode adhesive G-Bond Plus (GB; GC Corporation, Tokyo, Japan) (also available as G-ænial Bond), applied as an ER or as a SE adhesive, in combination with one coat of the hydrophobic resin Heliobond (Ivoclar Vivadent, Schaan, Liechtenstein). The *in situ* degree of conversion (DC), in enamel and dentin, of the adhesive interface was also evaluated. The hypotheses tested were that the application of a hydropho-

bic resin coat after GB would not influence 1) the enamel and dentin bond strengths; 2) the DC of the adhesive at the enamel- and dentin-resin interfaces.

#### **METHODS AND MATERIALS**

#### **Tooth Selection and Preparation**

A total of 60 extracted, caries-free, human third molars were used. The teeth were collected after obtaining the patients' informed consent under a protocol approved by the local Ethics Committee Review Board. The teeth were disinfected in 0.5% chloramine, stored in distilled water, and used within six months after extraction.

A flat occlusal dentin surface was exposed in 20 teeth after wet grinding the occlusal enamel with #180-grit silicon-carbide (SiC) paper for 60 seconds. The exposed dentin surfaces were further polished with wet #600-grit SiC paper for 60 seconds to standardize the smear layer. These teeth were used for dentin microtensile bond strength ( $\mu$ TBS) and measurement of *in situ* degree of conversion in dentin-resin interfaces.

Four flat enamel surfaces (buccal, lingual, and proximals) were exposed in 40 teeth after wet grinding the enamel with #180-grit SiC paper for 60 seconds. The enamel surfaces were further polished with wet #600-grit SiC paper for 60 seconds. Twenty teeth were used for enamel microshear bond strength ( $\mu$ SBS), whereas the remaining 20 teeth were used for measurement of  $in\ situ$  DC in enamelresin interfaces.

#### **Experimental Design**

The enamel (n=40) and dentin (n=20) specimens were randomly assigned into four groups according to the combination of the independent variables: adhesive strategy (ER or SE) and the hydrophobic resin coating (with or without). All teeth for each test ( $\mu$ SBS,  $\mu$ TBS, and DC) were randomized in block (http://www.sealedenvelope.com). A person not involved in the research protocol performed this procedure using computer-generated tables.

In all groups, the universal adhesive system GB (GC Corporation, Tokyo, Japan) was used: 1) GB applied as a 1-stepSE adhesive; 2) GB applied as in 1-stepSE followed by one coat of a hydrophobic resin coat (Heliobond, Ivoclar Vivadent, Schaan, Liechtenstein) (2-stepSE); 3) GB applied as a 2-stepER adhesive; and 4) GB applied as in 2-stepER followed by one coat of Ivoclar Vivadent Heliobond (3-stepER). All details regarding the adhesive composition are displayed in Table 1.

#### **Restorative Procedure**

The adhesive system was applied according to the manufacturer's instructions, except that the manufacturer does not recommend dentin etching with phosphoric acid. Please refer to Table 1 for more details.

After the bonding procedures, all teeth received a nanofilled composite restoration (Filtek Z350, 3M ESPE, St Paul, MN, USA) in two increments of 2 mm each. Each increment was light-polymerized for 40 seconds using an LED light-curing unit set at 1200 mW/cm² (Radii-cal, SDI Limited, Bayswater, Victoria, Australia). A radiometer (Demetron LED Radiometer, Kerr Sybron Dental Specialties, Middleton, WI, USA) was used to check the light intensity after finishing five specimens.

#### Specimen Preparation for Dentin µTBS

After storage of the restored teeth in distilled water at 37°C for 24 hours, the dentin specimens were longitudinally sectioned in mesiodistal and buccallingual directions across the bonded interface with a low-speed diamond saw (Isomet, Buehler Ltd, Lake Bluff, IL, USA) to obtain resin-dentin sticks with a cross-sectional area of approximately 0.8 mm² measured with a digital caliper (Digimatic Caliper, Mitutoyo, Tokyo, Japan). All specimens, from each tooth, were used for the  $\mu TBS$  evaluation, except that two sticks were randomly selected for measurement of the  $in\ situ\ DC$ .

Resin-dentin bonded sticks were attached to a grooved Geraldeli jig $^{40}$  (Odeme Biotechnology, Joaçaba, Santa Catarina, Brazil) with cyanoacrylate adhesive and tested in tension (Model 5565, Instron, Canton, OH, USA) at 0.5 mm/min until failure. The most peripheral sticks were discarded. The  $\mu TBS$  were calculated by dividing the load at failure by the cross-sectional bonding area.

The failure mode was classified as cohesive (C; failure exclusively within dentin or resin composite), adhesive (A; failure at the resin-dentin interface), or mixed (M; failure at the resin-dentin interface that included cohesive failure of the neighboring substrates). The failure mode analysis was performed under a stereomicroscope at 100× magnification (Model SZ40, Olympus, Tokyo, Japan).

#### Specimen Preparation for Enamel µSBS

Prior to applying the adhesive, each tooth was mounted in a polyvinyl chloride ring filled with acrylic resin (AutoClear, DentBras, Pirassununga, São Paulo, Brazil), displaying the buccal enamel

Materials (Batch numbe	Composition r)		SE Strategy				
Heliobond (N37749)	Bis-phenol glycidyl methacrylate (bis-GMA), triethylenedimethacrylate (TEGDMA)		Without Hydrophobic Bonding Resin	With Hydrophobic Bonding Resin			
G-Bond Plus (1102221)	Acetone, dimethacrylate, triethylenedimethacrylate (TEGDMA), 4-methacryloyloxyethyl trimellitic acid (4-MET), phosphoric acid ester monomer, silicon dioxide, photo initiator	Dentin	<ol> <li>Apply adhesive using a microbrush.</li> <li>Leave undisturbed for 10 s after the application.</li> <li>Dry thoroughly for 5 s with oil-free air under maximum air pressure. Use vacuum suction to prevent splatter of the adhesive.</li> <li>Light-cure for 10 s.</li> </ol>	<ul> <li>5. After applying the adhesive in the SE mode, apply a very thin layer of Heliobond with a microbrush.</li> <li>6. Air blow to achieve an optimally thin layer.</li> <li>7. Light-cure for 10 s.</li> </ul>			
		Enamel	<ol> <li>Apply adhesive using a microbrush</li> <li>Leave undisturbed for 10 s after the application.</li> <li>Dry thoroughly for 5 s with oil free air under maximum air pressure. Use vacuum suction to prevent splatter of the adhesive.</li> <li>Light-cure for 10 s.</li> </ol>	<ul><li>5. After applying adhesive in the SE mode, apply a very thin layer of Heliobond with a microbrush.</li><li>6. Air blow to achieve an optimally thin layer.</li><li>7. Light-cure for 10 s.</li></ul>			
Filtek Z350 (7WN)	Bis-GMA, UDMA (urethane dimethacrylate), TEGDMA, Bis-EMA (ethoxylated bisphenol-A dimethacrylate), silanated silica, silanated zirconia, photoinitiators	N/A	N/A				

surface on the top of the cylinder. The delimitation of the bonding area was performed according to Shimaoka and others (2011). An acid-resistant, double-faced adhesive tape (Adelbras Ind e Com Adesivos Ltda, São Paulo, Brazil) was perforated with a Hygenic Ainsworth-style rubber-dam punch (Coltene, Alstätten, Switzerland).

After the application of the adhesive system, polyethylene Tygon tubes (Tygon Medical Tubing Formulations 54-HL, Saint Gobain Performance Plastics, Akron, OH, USA) with an internal diameter of 0.8 mm were sectioned to obtain 0.5 mm-high matrices. A second lateral section was made to reduce the external diameter of the matrix. Each matrix was positioned over the double-faced tape with the lumen coincident with a perforation. An operator trained in the µSBS technique positioned seven to nine matrices per tooth. Resin composite was carefully packed inside each tube, and a clear Mylar matrix tape was placed over the filled tube and pressed gently into place. Resins were lightcured for 20 seconds. All restorative procedures were made under magnifying loupes.

After storage of the restored teeth in distilled water for 24 hours at 37°C, the Tygon tubes and the double-faced adhesive tape were carefully removed, exposing the composite cylinders. Each specimen was examined under a stereomicroscope at  $10\times$  magnification to identify those with evidence of air bubbles or gaps at the interface, which were discarded.

The specimens were attached to a shear-testing fixture (Odeme Biotechnology) and tested in a universal testing machine (Kratos IKCL 3-USB, Kratos Equipamentos Industriais Ltda, Cotia, São Paulo, Brazil). A thin wire (0.2 mm diameter) was looped around the base of each composite cylinder, making contact with half of its circumference, always keeping the setup aligned (resin-enamel interface, the wire loop, and the center of the load cell) to ensure the correct orientation of the shear forces.  $^{42}\,\mathrm{A}$  shear load was applied at a crosshead of 1 mm/min until failure. The  $\mu\mathrm{SBS}$  values were calculated by dividing the load at failure by the surface area (mm²) to determine the shear bond strength in megapascals.

Materials (Batch number)	ER Strategy					
Heliobond (N37749)	Without Hydrophobic Bonding Resin	With Hydrophobic Bonding Resin				
G-Bond Plus (1102221)	<ol> <li>Apply 34% phosphoric acid gel (Scotchbond Universal Etchant, 3M ESPE, St Paul, MN, USA) for 10 s.</li> <li>Rinse for 5 seconds and gently dry.</li> <li>Apply adhesive using a microbrush. Leave undisturbed for 10 s after the application</li> <li>Dry thoroughly for 5 s with oil free air under maximum air pressure.</li> <li>Light-cure for 10 s.</li> <li>Note: The manufacturer does not recommend dentin etching with phosphoric acid.</li> </ol>	<ul> <li>6. After applying the adhesive in the ER mode, apply a very thin layer of Heliobond with a microbrush.</li> <li>7. Air blow to achieve an optimally thin layer.</li> <li>8. Light-cure for 10 s.</li> </ul>				
	<ol> <li>Apply 34% phosphoric acid gel for 10 s.</li> <li>Rinse for 5 seconds and gently dry.</li> <li>Apply adhesive using a microbrush. Leave undisturbed for 10 s after the application.</li> <li>Dry thoroughly for 5 s with oil free air under maximum air pressure.</li> <li>Light-cure for 10 s.</li> </ol>	<ul> <li>6. After applying the adhesive in the ER mode, apply a very thin layer of Heliobond with a microbrush</li> <li>7. Air blow to achieve an optimally thin layer</li> <li>8. Light-cure for 10 s</li> </ul>				

#### Degree of Conversion in situ

Two resin-dentin bonded sticks from each tooth prepared for  $\mu$ TBS and the remaining 20 teeth prepared for  $\mu SBS$  were used for measurement of in situ DC in enamel. The adhesive interface of each bonded stick was wet polished with #1500-, #2000-, and #2500-grit SiC paper for 15 seconds each. Then they were ultrasonically cleaned for 20 minutes in distilled water and stored in water for 24 hours at 37°C prior to performing the DC readings. The micro-Raman spectroscopy analysis was carried out using Senterra equipment (Bruker Optik GmbH, Ettlingen, Baden-Württemberg, Germany). The micro-Raman spectrometer was first calibrated for zero and then for coefficient values using a silicon specimen. Specimens were analyzed using the following micro-Raman parameters: 20-mW neon laser with 532-nm wavelength, spatial resolution of  $\approx 3$  µm, spectral resolution  $\approx 5$  cm<sup>-1</sup>, accumulation time of 30 seconds with 6 coadditions, and magnification of 100× (Olympus UK, London, UK) to a beam diameter of  $\approx 1$  µm. Spectra were taken at the dentin-adhesive and enamel-adhesive interface at three different sites for each specimen. The average value of the measurements taken from the same tooth was used for statistical purposes. Spectra of uncured adhesives were taken as reference. Postprocessing of spectra was performed using the dedicated Opus Spectroscopy Software, version 6.5

(Bruker Optik GmbH). The ratio of double-bond content of monomer to polymer in the adhesive was calculated according to the following formula:

$$DC(\%) = \left(1 - rac{R_{(cured)}}{R_{(uncured)}}
ight) imes 100,$$

where R is the ratio of aliphatic and aromatic peak areas at  $1639~\rm cm^{-1}$  and  $1609~\rm cm^{-1}$  in cured and uncured adhesives, respectively. The *in situ* DC values of all resin-dentin and enamel-bonded specimens from the same tooth were averaged for statistical purposes.

#### **Statistical Analysis**

A power analysis was calculated separately for enamel  $\mu SBS$  and for dentin  $\mu TBS$  using  $\alpha=0.05$  and a power of 80%, using mean bond strengths reported in the literature for G-Bond, the predecessor of GB.

The resin-dentin  $\mu TBS$  and resin-enamel  $\mu SBS$  of all specimens (with adhesive/mixed failure) from the same tooth were averaged for statistical purposes. Similarly, the same procedure was performed for the DC measurements so that the experimental unit in this study was the tooth. Specimens with cohesive and premature failures were not included in data analysis. Data from  $\mu TBS$  and  $\mu SBS$  were analyzed separately using two-way analysis of variance

(ANOVA) (adhesive strategy vs hydrophobic resin coat) and a Tukey *post hoc* test at  $\alpha = 0.05$ . For *in situ* DC, the data were analyzed with three-way ANOVA (adhesive strategy vs hydrophobic resin coat vs. substrate) and a Tukey *post hoc* test at  $\alpha = 0.05$ .

#### **RESULTS**

#### **Bond Strengths**

The use of a hydrophobic resin coat resulted in statistically significant higher mean enamel  $\mu SBS$  for the ER strategy (3-stepER vs 2-stepER, p<0.0002; Table 2). All other groups had a statistically similar mean  $\mu SBS$  (p>0.05; Table 2).

For dentin, the use of a hydrophobic resin coat resulted in a significantly higher mean  $\mu$ TBS for the SE strategy (2-stepSE vs 1-stepSE, p<0.0007; Table 2) but not for the ER strategy. All other mean  $\mu$ TBS were statistically similar (p>0.05; Table 2).

For dentin  $\mu$ TBS, the majority of the specimens (86.6% to 95.1%) showed adhesive/mixed failures (Table 3). For enamel  $\mu$ SBS, the majority of the specimens (86.0% to 96.6%) showed adhesive/mixed failures (Table 3).

#### **Degree of Conversion**

The use of a hydrophobic resin coat significantly improved the DC in dentin irrespective of the bonding strategy (p<0.0009; Table 4). In enamel, an increase in the DC was observed only in the SE strategy (p<0.00002; Table 4). The highest DC was found in dentin for both strategies when the hydrophobic bonding resin was used.

#### **DISCUSSION**

SE dental adhesives are a sophisticated blend of hydrophilic and hydrophobic reactive monomers and mono-functional comonomers, polymerization initiators, at least two solvents including water, crosslinking monomers, stabilizers, and filler particles. <sup>14</sup> Contemporary SE adhesives contain specific monomer molecules that combine unsaturated polymerizable functions with carboxylate or phosphate acidic groups. <sup>14,43</sup>

The composition of GB or G-ænial Bond, a multimode adhesive, is similar to that of 1-stepSE adhesives. GB is a new version of the 1-stepSE adhesive G-Bond (GC Corporation). The UDMA monomer was replaced in the newer version with a different dimethacrylate monomer; whereas, the concentration of the phosphate monomer was increased<sup>8</sup> to make the monomer solution more acidic

(pH=1.5 vs pH=2.0, respectively, for GB and G-Bond). The newest version is, therefore, a mild one-bottle SE adhesive indicated as a "total" SE adhesive or as a "partial" SE adhesive with selective enamel etching. The respective manufacturer does not recommend using GB on phosphoric acid-etched dentin. Nevertheless, we used this adhesive experimentally as an ER adhesive due to the fact that some clinicians may etch dentin inadvertently.

Concerns about incomplete adhesive infiltration of ER adhesives into the collagen network of the demineralized zone prompted research on the hermetic seal of the hybrid layer against silver particles. Reports on water permeation and sorption within the hybrid layer and adhesive resin films are related to polymer hydrophilicity. The acidic monomer solution in SE adhesives must be able to simultaneously demineralize and infiltrate dentin to form a hybrid layer that has been shown to have fewer defects than that of ER adhesives. In fact, Hanabusa and others described more defects within the dentin hybrid layer formed by GB when used as an ER adhesive than when used as a SE adhesive.

Our results showed that the use of a hydrophobic resin coat in dentin increased the mean bond strengths for the SE approach, which has been reported before in several studies with other SE adhesives. The monomer solutions from 1-stepSE adhesives lack a nonsolvated resin coating, which makes them permeable membranes. This permeable structure would allow rapid dentinal fluid transudation across the polymerized adhesive, specifically in 2-hydroxyethyl-methacrylate (HEMA)-containing adhesives.

The mono-functional comonomer HEMA acts as a solvent and helps prevent hydrophilic and hydrophobic phase separation. <sup>20,43,48</sup> However, HEMA may retain water, compromising the DC and resulting in hydrolytic degradation. Together these factors reduce the durability of the adhesive interface over time. <sup>4,48</sup>

Theoretically, the absence of HEMA in GB, as well as in its predecessor G-Bond, may result in longer stability of the resin-dentin interface compared with adhesives that contain HEMA<sup>49</sup> because water sorption is more pronounced when HEMA is present.<sup>47</sup> On the other hand, the lack of HEMA may result in phase separation at the interface, previously observed with G-Bond,<sup>48</sup> which may be a limiting factor for improved performance of the material.

Table 2: Mean and Standard Deviation (MPa) of Microshear (μSBS) and Microtensile Bond Strength (μTBS) of the Experimental Groups, as Well as the Statistical Significance<sup>a</sup>

Adhesive System	Groups	μSBS Enamel	μTBS Dentin			
G-Bond Plus	Self-etch without bond	14.7 ± 1.1 B	13.4 $\pm$ 1.3 b			
	Self-etch with bond	15.0 ± 2.7 B	20.4 ± 1.5 a			
	Etch-and-rinse without bond	15.9 ± 2.8 B	19.1 ± 0.7 a			
	Etch-and-rinse with bond	20.6 $\pm$ 3.4 A	17.6 ± 1.2 a			
<sup>a</sup> Identical uppercase	<sup>a</sup> Identical uppercase (enamel) and lowercase (dentin) letters indicate the means are not statistically different (Tukey test; p<0.05).					

The mean dentin  $\mu$ TBS obtained in our study with GB was somewhat lower than those obtained by other research groups. 8,50 It has been demonstrated that the mechanical properties of GB are lower than those of other 1-stepSE adhesives. 47 In 2002 and 2005, Takahashi and others 23 and Reis and others, 24 respectively, reported that there is a positive correlation between dentin microtensile bond strengths and the ultimate tensile strength of the adhesive, which would explain the low dentin bond strengths obtained with GB in the present study.

Additionally, factors related to the methodology may have accounted for this difference in the mean μTBS. In the Hanabusa and others study, 8 authors only used nine central sticks from each tooth to reduce substrate variability, whereas in our study we used sticks from the entire interface. One study reported lower µTBS for peripheral specimens than for centrally located specimens.<sup>51</sup> Furthermore, the cross-head speed in our study was 0.5 mm/min, whereas in the Hanabusa and others study<sup>8</sup> it was 1.0 mm/min. A comparative study reported higher uTBS for Clearfil SE Bond when greater cross-head speed was used. A more uniform stress-time pattern was observed for 1 mm/min. 52 In the case of the 2011 Goracci and others study, 50 although the authors used the same cross-head speed as in our study, they

applied GB on dentin under agitation, not following the manufacturer's instructions (Table 1). It has been shown that dynamic application of SE adhesives results in greater bond strengths. <sup>53,54</sup>

The DC measured in the dentin-resin interfaces was significantly higher when a hydrophobic resin coat was used. This difference was expected, according to the previous literature finding<sup>55,56</sup> where 1-stepSE was compared with 2-stepSE adhesives. However, an increase in DC has not been previously reported after adding an extra hydrophobic resin coat to a specific 1-step adhesive.

One-step SE and 2-stepER adhesives may exhibit droplets from water attraction and osmosis through the cured adhesive layer. Osmosis droplets are observed at the transition between the adhesive layer of 1-stepSE and 2-stepER adhesives and the composite resin material. This transition contains uncured monomers from the oxygen-inhibition layer, which would have resulted in decreased DC in our study. The hydrophobic resin coat would have copolymerized with the uncured surface of the adhesive layer from the 1-step adhesive, resulting in higher DC due to the consumption of residual double bonds.

For enamel, we would have expected an increase in bond strengths compared with the original G-

Table 3: Number and Percentage of Specimens (%) According to Fracture Pattern Mode From the Experimental Groups in the Microshear (μSBS) and Microtensile Bond Strength (μTBS) Tests

Adhesive System	Adhesive Strategy	Hydrophobic Resin Coat	Fracture Pattern (μSBS)			
			Α	С	A/M	PF
G-Bond Plus	Self-etch	Without	33 (57.9)	6 (10.5)	16 (28.1)	2 (3.5)
		With	39 (61.9)	5 (7.9)	17 (27.0)	2 (3.2)
	Etch-and-rinse	Without	26 (49.1)	3 (5.7)	23 (43.3)	1 (1.9)
		With	39 (66.1)	1 (1.7)	18 (30.5)	1 (1.7)
G-Bond Plus	Self-etch	Without	42 (80.8)	2 (3.8)	3 (5.8)	5 (9.6)
		With	56 (90.3)	0 (0.0)	2 (3.2)	4 (6.5)
	Etch-and-rinse	Without	44 (72.2)	1 (1.6)	14 (22.9)	2 (3.3)
		With	42 (82.4)	0 (0.0)	6 (11.8)	3 (5.8)

Table 4:	Mean and Standard Deviation (%) of In Situ Degree of Conversion (DC) Values of the Experimental Groups, as Well as
	the Statistical Significance <sup>a</sup>

Adhesive System	Groups	DC% Enamel	DC% Dentin				
G-Bond Plus	Self-etch without bond	72.7 ± 4.9 D	58.3 ± 4.8 F				
	Self-etch with bond	83.1 ± 4.2 B	92.3 $\pm$ 5.2 A				
	Etch-and-rinse without bond	81.8 $\pm$ 3.7 B,C	69.3 ± 5.5 E				
	Etch-and-rinse with bond	79.4 $\pm$ 2.9 C	94.8 $\pm$ 4.5 A				
a Identical uppercase letters indica	a Identical uppercase letters indicate the means are not statistically different (Tukey test; $p$ <0.05)						

Bond applied as a SE adhesive, because pH has decreased to 1.5 in the multi-mode version. For the original G-Bond (pH=2.0), in a 2006 study Perdigão and others<sup>61</sup> obtained 18.3 MPa to ground enamel, whereas in 2007 De Munck and others<sup>62</sup> obtained a mean enamel μTBS of 19.8 MPa. In 2013, Reis and others<sup>63</sup> obtained a mean of 17.2 MPa for G-Bond to ground enamel. For GB (pH=1.5) Hanabusa and others<sup>8</sup> obtained a mean enamel µTBS of 23.1 MPa. In our study, we obtained slightly lower enamel bond strengths with GB, 14.7-15.0 MPa. This may have been the result of the wider bonding areas required in the µSBS as compared with the µTBS. The µSBS test has resulted in lower enamel bond strengths for the SE adhesives Clearfil SE Bond and Adper Prompt L-Pop when compared with their μTBS values.<sup>64</sup> It has been reported that μTBS values tend to be higher because the defect concentration in the small cross-sectional interfacial areas is lower.<sup>65</sup>

Although Pashley and Tay<sup>39</sup> reported that the cohesive strength of the adhesive layer might be more relevant than the etching potential of an enamel adhesive for acidic SE adhesives, 10,66 the hydrophobic resin coat application, which increases the cohesive strength of the adhesive layer, was not able to improve the enamel-dentin bonds in our study. This similarity in enamel bond strengths with and without the hydrophobic resin coat may be a result of the poor enamel-etching pattern obtained with the application of the SE adhesive, because the ability of the acidic agent to chemically etch enamel is directly related to the respective enamel bond strengths. The additional hydrophobic resin coat is unable improve bond strengths<sup>27,63</sup> because it has no influence in the etching pattern.

In spite of the similarity in mean enamel µSBS, DC was significantly higher when GB was used in SE mode with the extra hydrophobic resin coat (2-stepSE). In fact, a good correlation was previously observed between bond strengths and DC when SE adhesives were applied on enamel<sup>67</sup> but not when ER adhesives were applied.<sup>68</sup> Two factors may have

played a role in this increase in DC for the 2stepSE compared with the 1-stepSE on enamel. First, the presence of residual uncured monomers on the top of the cured GB adhesive would have caused lower DC. This was probably reduced with the extra hydrophobic coat. Second, the evaporation of all water from the interface, which prevents the approximation of the polymer chains, would have been more difficult to accomplish in the 1-stepSE group due to the presence of microscopic osmotic blisters. The hydrophobic resin coat application may have aided in the evaporation of solvents and residual water.

One of the limitations of our study has to do with the lack of thermal fatigue or long-term water storage. Thermal fatigue may not be relevant because the susceptibility of adhesives to thermal fatigue depends on the specific composition of each adhesive. However, water storage may correlate better with clinical behavior. In fact, Heintze and others reported that dentin  $\mu TBS$  of adhesive systems after water storage for six months showed a good correlation with marginal discoloration in clinical Class V restorations. With this in mind, future studies in our laboratory will analyze the effect of long-term water storage on the  $in\ vitro$  performance of the multi-mode adhesive GB.

We failed to reject the first null hypothesis, given that the application of a hydrophobic resin coat after GB increased the enamel  $\mu SBS$  for the ER adhesion strategy and the dentin  $\mu TBS$  for the SE adhesion strategy. We also failed to reject the second null hypothesis because the application of a hydrophobic resin coat after GB increased *in situ* DC for enamelresin interfaces when GB was used in SE mode and for dentin-resin interfaces for both the SE and the ER adhesion strategies.

#### **CONCLUSIONS**

Within the limitations of this *in vitro* study, the use of a hydrophobic resin coat may be beneficial for the selective enamel etching technique, because it improves bond strengths to enamel when applied with the ER strategy, and to dentin when used with the SE

adhesion strategy. The application of a hydrophobic resin coat may improve DC in resin-dentin interfaces formed with either the SE or the ER strategy. On enamel, DC may benefit from the application of a hydrophobic resin coat over 1-stepSE adhesives.

#### **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 Evaporation on the Shelf Life of a Universal Adhesive

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#### **Clinical Relevance**

Evaporation of adhesive ingredients due to repeated opening of the bottle adversely influences the shelf life of a universal one-step adhesive only when more than 50% of the solvent and evaporable ingredients have been evaporated.

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#### **SUMMARY**

Objectives: The purpose of this study was to evaluate how evaporation affects the shelf life of a one-bottle universal adhesive.

Methods: Three different versions of Scotch-bond Universal (SBU, 3M ESPE, Seefeld, Germany) were prepared using a weight-loss technique. SBU0 was left open to the air until maximal weight loss was obtained, whereas SBU50 was left open until 50% of evaporation occurred. In contrast, SBU100 was kept closed and was assumed to contain the maximum concentration of all ingredients. The degree of conversion (DC) was determined by using Fourier transform infrared spectroscopy on different substrates (on dentin or glass plate and mixed with dentin powder); ultimate microtensile strength and microtensile bond strength to dentin were measured as well.

Results: DC of the 100% solvent-containing adhesive (SBU100) was higher than that of the 50% (SBU50) and 0% (SBU0) solvent-containing adhesives for all substrates. DC of the adhesive applied onto glass and dehydrated dentin was higher than that applied onto dentin. Even though the ultimate microtensile strength of SBU0 was much higher than that of SBU50 and SBU100, its bond strength to dentin was significantly lower.

Conclusions: Evaporation of adhesive ingredients may jeopardize the shelf life of a one-bottle universal system by reducing the degree of conversion and impairing bond strength. However, negative effects only became evident after more than 50% evaporation.

#### INTRODUCTION

In dental clinical practice, the shelf life of adhesives is important. Shelf life can be defined as the period during which adhesive systems retain optimum bonding efficacy. Because the composition of adhesives gradually changes over time, manufacturers always provide an expiration date. The adhesive should not be used after this date, as the efficacy can no longer be guaranteed. More specifically, the adhesive composition may change over time due to hydrolysis or polymerization of the monomers, degradation of the additives (initiators/stabilizers), or evaporation of ingredients. The same period and the same period and

Thanks to their easy application procedure, onestep self-etch adhesives are commonly used in dental practice today. A long shelf life is important for their bonding effectiveness, but because of their specific composition, this class of adhesives may be more prone to reduced shelf life than conventional multistep systems. First, one-step self-etch adhesives typically have their adhesive monomers mixed with water, which may lead to hydrolysis of the ester bond of the methacrylic group. 3,4 Moreover, the acidic pH in one-step self-etch adhesives may accelerate this hydrolysis process.<sup>3,5</sup> Long storage times and high temperatures may also promote hydrolysis.<sup>3,5</sup> Previous research showed that hydrolysis of the monomers may indeed reduce the bonding efficacy of selfetch systems.<sup>1</sup>

Second, ingredients may evaporate by repeated opening of the bottle. Not only do organic solvents easily evaporate due to their high vapor pressure but small quantities of low-molecular-weight monomers do also.6 Evaporation is also enhanced by high temperatures. It is clear that the amount of solvent in an adhesive may be different between the first and last use. Unlike composites, dental adhesives contain solvents, which serve different purposes, like dissolving the monomers, ionizing the functional monomers, and facilitating infiltration of the resin in dentin.<sup>2</sup> Perdigao and coworkers<sup>7</sup> studied the effects of repeated opening of a two-step etch-and-rinse adhesive on bond strength. They left the bottle open for one minute, two times a day for three weeks, and determined the bond strength. They found lower bond strengths after three weeks for the acetonecontaining adhesive and concluded that solvent evaporation may reduce the shelf life of adhesives.

Apart from that study, only a few studies have evaluated how ingredient evaporation affects the bonding effectiveness of adhesives. The objective of this study was to evaluate how ingredient evaporation affects degree of conversion (DC), ultimate microtensile strength (U $\mu$ TS), and bond strength of a commercial, universal, one-component, one-step self-etch adhesive. To better understand how ingredient evaporation affects the shelf life of this adhesive, polymerization efficacy was investigated on different substrates. The null hypothesis tested was that evaporation does not affect DC, U $\mu$ TS, and microtensile bond strength.

#### **METHODS AND MATERIALS**

#### **Adhesive Preparation**

Three different versions of Scotchbond Universal (SBU, 3M ESPE, Seefeld, Germany) were prepared (Table 1). First, the content of a fresh bottle of SBU was divided into three amber vials, which were protected from light by aluminum foil. The first vial was capped immediately. This version was assumed to have 100% solvent and was called SBU100. The second vial was left open in a dark room at room temperature (20°C) until its weight was constant for at least two days (this took more than 14 days). This vial was assumed to have 0% solvent (SBU0). The last vial was left open until it had 50% of the weight loss as calculated from the second vial, which took more than five days at room temperature. This adhesive version was called SBU50. According to the manufacturer, SBU contains around 20 to 30 wt% solvent (ethanol and water), and the percentage of weight loss in SBU0 and SBU50 was 22.7 wt% and 11.3 wt%, respectively.

All adhesives were then kept capped in the refrigerator until use. The pH of SBU100, SBU50, and SBU0 was 2.93, 2.79, and 2.15, respectively (measured in triplicate, Inolab pH Level 2, WTW GmbH, Weilheim, Germany).

#### **Degree of Conversion**

Each adhesive was applied onto six different substrates and the DC of the adhesives was measured by Fourier transform infrared spectroscopy (FTIR). Four samples were prepared per group (n=4).

• Group 1 (glass): The adhesives were rubbed on a glass slide for 20 seconds and then air-blown for 10 seconds according to the manufacturer's instruc-

Table 1: Composition of the Universal Adhesive Used in This Study							
Adhesive Composition Instructions for Application							
Scotchbond Universal (SBU) Lot 468651	BisGMA 15-25 wt%, HEMA 15-25 wt%, DMDMA 5-15 wt%, ethanol 10-15 wt%, water 10-15 wt%, silane-treated silica 5-10 wt%, copolymer of acrylic and itaconic acid 1-5 wt%, methyl ethyl ketone <0.5 wt%, CQ ~2 wt%, EDMAB <2 wt%	Apply the adhesive and rub it in for 20 seconds, gently air-blow for 5 seconds until no more movement of the adhesive, then light-cure for 10 seconds.					
Abbreviations: BisGMA, bisphenol A diglycidyl ether dimethacrylate; CQ, camphorquinone; DMDMA, decamethylene dimethacrylate; EDMAB, ethyl 4-(dimethylamino)benzoate; HEMA, 2-hydroxyethyl methacrylate.							

tions. The adhesive was covered by another glass slide to avoid an oxygen-inhibition layer, and light-cured.

- Group 2 (glass 37°C): The adhesives were applied as in group 1 onto a glass slide, which was preheated on a hot table up to 37°C, and then covered by another preheated glass slide.
- Group 3 (dentin): Human third molars with no caries (collected after obtaining informed consent approved by the Commission for Medical Ethics of Katholieke Universiteit Leuven) were stored in 0.5% chloramine/water at 4°C and used within three months after extraction. Midcoronal dentin discs were cut from extracted human molars. The adhesives were applied as in group 1, covered by a glass slide, and light-cured.
- Group 4 (dentin 37°C): The adhesives were applied as in group 3 to a dentin disc preheated on a hot table up to 37°C, and then covered by another preheated glass slide.
- Group 5 (dehydrated dentin): The adhesives were applied as in group 3 to a dentin disc, which was dehydrated in an oven at 37°C for 24 hours, and then kept in vacuum for at least 1 hour.
- Group 6 (dentin powder): Dentin powder was prepared by grinding human dentin with a grinder (A10, IKA, Staufen, Germany) according to a previously described protocol. After mixing the adhesive with 10 wt% dentin powder, it was further treated as in group 1.

The specimens were cured with a Bluephase 20i light-emitting diode unit (Ivoclar-Vivadent, Schaan, Liechtenstein) for 10 seconds at 1000 mW/cm² in high mode and stored at 37°C for 24 hours in a dry condition. DC was measured by attenuated total reflectance FTIR (Vertex 70, Bruker Optik GmbH, Ettlingen, Germany) in triplicate and calculated as the ratio of peak intensities of the aliphatic 1640 cm<sup>-1</sup> and aromatic 1610 cm<sup>-1</sup> peak in cured and uncured materials. FTIR spectra between 4500 and 400 cm<sup>-1</sup> were recorded at room temperature at 32 scans per sample and a resolution of 4 cm<sup>-1</sup>. DC was calculated according to the following formula:

$$\mathrm{DC} = \left(1 - \frac{\mathrm{R}\; \mathrm{cured}}{\mathrm{R}\; \mathrm{uncured}}\right) \times 100$$

where R is the ratio of intensities of the peak at  $1640 \text{ cm}^{-1}$  and  $1610 \text{ cm}^{-1}$ .

#### **Ultimate Microtensile Strength**

Ten polymerized adhesive specimens were prepared (n=10) to determine the UμTS. Each adhesive was applied in a  $2 \times 2 \times 8$  mm<sup>3</sup> silicone mold, which was covered with a glass slide. The specimens were cured for 20 seconds through the glass slide with the Bluephase 20i (Ivoclar-Vivadent) light-emitting diode unit with the output of 1000 mW/cm<sup>2</sup>. After dry storage for 24 hours in the dark at 37°C, the adhesive sticks were trimmed at the middle of the stick to an hourglass shape with a diameter of 1.1 mm using a cylindrical, extra-fine grit (15 µm) diamond bur in a water-cooled high-speed handpiece mounted in a MicroSpecimen Former (University of Iowa, Iowa City, IA, USA). For SBU0, additional samples with a diameter of 0.8 mm were trimmed. The diameter of each specimen was measured using a stereomicroscope with a resolution of 1 µm at a magnification of 20× (400-NRC, Leitz, Germany). A cross-section area of about 1 mm<sup>2</sup> (SBU100 and SBU50) and about 0.5 mm<sup>2</sup> (SBU0) was obtained. Each specimen was attached to a modified notched Ciucchi's jig with cyanoacrylate glue (Model Repair II Blue, Dentsply-Sankin, Tochigi, Japan) and stressed at a crosshead speed of 1 mm/min until failure in a universal testing device (LRX, Lloyd, Hampshire, UK) to determine the UµTS.

#### Microtensile Bond Strength (µTBS)

After removal of the occlusal third of the crown of human third molars with a diamond saw (Isomet 1000, Buehler, Lake Bluff, IL, USA), a standardized bur-cut smear layer was created by means of a high-speed medium-grit diamond bur (100  $\mu$ m; 842, Komet, Lemgo, Germany) mounted in a MicroSpecimen former (University of Iowa). Per adhesive

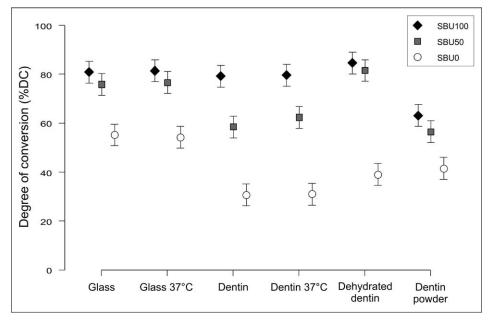


Figure 1. Degree of conversion (DC in %) of the adhesive versions: 100% solvent-containing adhesive (SBU100), 50% solvent-containing adhesive (SBU50), and 0% solvent-containing adhesive (SBU0) (mean and standard deviation).

group, eight teeth were used (n=8). As per the manufacturer's instructions, the adhesives were applied onto dentin, rubbed for 20 seconds, air-blown for 10 seconds, and light-cured for 10 seconds (output of 1000 mW/cm<sup>2</sup>, Bluephase 20i, Ivoclar Vivadent). Composite build-ups were made with Z100 MP composite (shade A3; 3M ESPE) in three layers, the first being 1-mm thick and then two subsequent 2-mm thick layers. Each composite layer was cured for 20 seconds. After 24 hours of storage in distilled water at 37°C, the teeth were sectioned perpendicularly to the interface by means of an automated water-cooled precision diamond saw (Accutom-50, Struers, Ballerup, Denmark) to produce rectangular  $1 \times 1$  mm sticks. Up to six central sticks were used for  $\mu TBS$  testing. Each stick was attached to a modified notched Ciucchi's jig9 with cyanoacrylate glue and stressed at a crosshead speed of 1 mm/min until failure in the LRX (Lloyd) testing device to determine the µTBS. The mode of failure was determined by stereomicroscopy. Representative composite and dentin fragments were observed by scanning electron microscopy (SEM; JSM-5600, JEOL, Tokyo, Japan).

#### **Statistical Analysis**

To statistically assess DC, a linear mixed effects model taking into account multiple testing of the same specimens was constructed using statistical software (R 2.13.2 and nlme package, R Foundation

for Statistical Computing, Vienna, Austria). In this model substrate, adhesives and their interaction were included. To further investigate these interactions, 95% confidence intervals were calculated (R 2.13.2 and AICcmodavg package). All tests were performed at a significance level of  $\alpha$ =0.05.

The U $\mu$ TS and  $\mu$ TBS data were analyzed by oneway analysis of variance and Tukey's multiple comparison test ( $\alpha$ =0.05).

#### RESULTS

#### **Degree of Conversion**

Irrespective of the group, SBU100 always exhibited the highest DC, and SBU0 had the lowest DC (Figure 1). The linear mixed-effect model showed that both adhesive and substrate significantly affected DC (p<0.0001 for both). However, depending on the adhesive, a different effect was observed on the various substrates, as a significant interaction (p<0.0001) was observed.

SBU100 had the highest DC when applied on dehydrated dentin substrate (DC=84%) and the lowest DC when mixed with dentin powder (DC=63%). DC in this last group was significantly lower than that in the other groups. There were no statistically significant differences between the other groups: glass, glass  $37^{\circ}$ C, dentin, dentin  $37^{\circ}$ C, and dehydrated dentin.

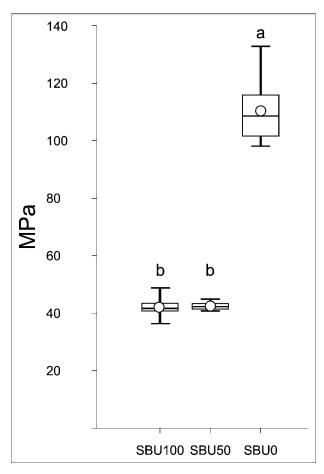


Figure 2. Ultimate microtensile strength of the adhesive versions: 100% solvent-containing adhesive (SBU100), 50% solvent-containing adhesive (SBU50), and 0% solvent-containing adhesive (SBU5) (mean and standard deviation). The same letters indicate no statistically significant difference (p>0.05).

For SBU50, the highest DC was also observed when applied on dehydrated dentin (DC=81%), and this DC was not significantly different from that in the group with glass and glass 37°C substrates (Figure 1). DCs on glass, glass 37°C, and dehydrated dentin were significantly higher than the DCs on dentin or dentin 37°C and when mixed with dentin powder. The lowest DC, which was 56%, was observed in the dentin powder group.

For SBU0, the highest DC was obtained when applied on a glass substrate (DC=55%), and this was not significantly different from that in the glass 37°C group. When applied on dehydrated dentin and mixed with dentin powder, DC was significantly lower than in the glass and glass 37°C groups, but still higher than the DC in the dentin and dentin 37°C group. The lowest DC observed was 30% when applied on dentin.

#### **Ultimate Microtensile Strength**

The  $U\mu TS$  of SBU0 adhesive samples with a 1-mm constriction could not be determined as the cyanoacrylate glue always failed first. Therefore, the  $U\mu TS$  of SBU0 was determined in additional samples with 0.8-mm constriction. With a  $U\mu TS$  of 110.4 (±10.4) MPa, the  $U\mu TS$  of SBU0 was more than twice as high as that of SBU50 and SBU100 (U $\mu TS$  of 42.5  $\pm$  1.4 and 42.1  $\pm$  3.3, respectively) (Figure 2). The  $U\mu TS$  of SBU0 was significantly higher than that of SBU100 and SBU50.

#### Microtensile Bond Strength

The  $\mu$ TBS of the different experimental adhesives to dentin are shown in Figure 3. No pretesting failures were recorded. The  $\mu$ TBS of SBU100 and SBU50 was not statistically significantly different. However, SBU0 obtained a significantly lower bond strength than SBU50 and SBU100.

#### DISCUSSION

So-called universal adhesives, which have been claimed to be applicable onto different substrates (tooth, alloys, ceramics, composites) have recently regained attention, as the latest generation of universal adhesives consists of only a single bottle. With their simple and short application procedure, they represent an attractive substitute for previous universal systems that were often very laborious and required different application steps and primers depending on the bonding substrate. Because this new class of adhesives meets the demands of dentists, who no longer need to buy different adhesives, it can be foreseen that they will probably become very popular. Considering the still increasing share of adhesive applications used in general dental practice, the way evaporation affects shelf life may be very important for universal adhesives, as this type of adhesives will be opened very frequently.

In this study, we left the bottle open until there was no longer weight loss due to evaporation (SBU0). In total, there was 23% weight loss compared with the original adhesive. Assuming that the main cause for the weight loss was the evaporation of the solvent, this corresponded well to the amount of solvent (20-30 wt%) in the adhesive according to the manufacturer.

First, we tested how evaporation affects DC. In general, evaporation led to decreased DC, which corresponds to previous research, in which it was reported that small amounts of solvent increase the polymerization degree.<sup>11,12</sup> The negative effect of

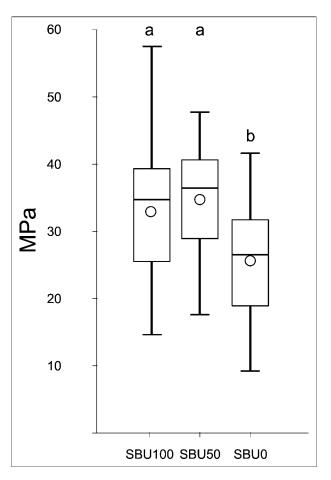


Figure 3. Microtensile bond strength of the adhesive versions: 100% solvent-containing adhesive (SBU100), 50% solvent-containing adhesive (SBU50), and 0% solvent-containing adhesive (SBU50) (mean and standard deviation). The same letters indicate no statistically significant difference (p>0.05).

reduced solvent concentrations may be attributed to increased viscosity, with hampered diffusion of monomers, growing polymer-chain segments, and radicals. 13 Alternatively, this phenomenon could be explained by a decreased glass-transition temperature when a solvent is mixed with the adhesive monomers. 14 At a constant temperature, mixtures with a lower glass-transition temperature will obtain higher degrees of polymerization.<sup>14</sup> To verify this hypothesis, we added 10 wt% of ethanol (absolute ethanol, 99.99%; CAS: 64-17-5; VWR, Haasrode, Belgium) to SBU0 and applied it on a glass plate as in group 1. This resulted in a dramatic increase of the DC of SBU0 from 55.1% ( $\pm 1.2$ ) to 74.8% ( $\pm 1.8$ ), which confirmed that solvent evaporation jeopardized polymerization and that the lower DC was not due to monomer degradation, premature cure, or any other side effect of the evaporation.

However, the effect of evaporation was highly dependent on the type of substrate, which demonstrates that the conversion rate depends on a complex interplay of factors. When applied on a glass plate, 50% evaporation did not significantly reduce DC, whereas on dentin, DC was significantly decreased. This should be attributed to the inhibitory effect of remaining water in dentin<sup>15</sup> as SBU50 did not exhibit reduced DC on dehydrated dentin. The inhibitory effect of water on the DC can be explained by the fact that in SBU50, ethanol had most probably been evaporated due to its high vapor pressure and that any remaining solvent would have been water. As result, the solvent in SBU50 could not be removed anymore, and further dilution of the monomers due to additional water uptake from dentin led to a reduced DC.

Nevertheless, when all solvent was evaporated, SBU0 performed equally on hydrated and dehydrated dentin. We hypothesized that the drop in DC on dehydrated dentin compared with SBU50 and compared with SBU0 on glass was due to the fact that the dehydrated dentin absorbed all remaining water upon application of the solvent, thereby increasing the viscosity to the point that polymerization was hindered. A balanced amount of solvent in the adhesive thus seems imperative to obtain high conversion degrees. <sup>13,16</sup>

To evaluate the effect of the constituents (collagen, hvdroxyapatite)<sup>17</sup> of dentin on the polymerization rate, we mixed dentin powder with the adhesive. 18,19 However, the DC of SBU100 mixed with dentin was significantly lower than on glass or dentin, probably due to the fact that the dentin powder acted as filler particles,<sup>20</sup> which may prevent activated polymer strands from meeting. As reported in literature, temperature is an important factor that influences the degree of polymerization.<sup>21</sup> Preheating the substrate did not seem to influence DC in this study. Whereas a significant positive effect of higher temperatures on DC was previously reported in studies<sup>21,22</sup> in which DC was measured shortly after curing, DC of the samples in our study was only determined 24 hours after light-curing.

We also tested how evaporation affects the intrinsic mechanical strength of the adhesive resin. Unlike SBU100 and SBU50, the UµTBS of SBU0 could not be tested, as the intrinsic mechanical strength of these adhesive samples surpassed that of the cyanoacrylate glue to fix samples to the µTBS jig. Therefore, we also tested adhesive samples with a 0.8 mm constriction, even though it was previously reported that the bonding surface may influence the

bond strength. 23 To our surprise, the UµTS of SBU0 was more than twice as high as that of SBU50 and SBU100, in spite of its inferior DC. A plausible explanation may be the higher monomer/volume ratio in SBU0 compared with the other versions. In addition, it is also conceivable that the solvent in SBU50 and SBU100, which was difficult to remove with air-blowing once the adhesive was applied in the mold, significantly deteriorated the mechanical properties of the adhesive resin. It was previously reported that remaining solvent softened the polymer. 11,15,24 The remarkably higher mechanical strength may also be due to a higher cross-linked polymer structure in SBU0, as small low-molecular weight monomers, like 2-hydroxyethyl methacrylate (HEMA), may have evaporated. HEMA is a monomethacrylate, which will lead to a less cross-linked polymer<sup>2</sup> and thus to polymers with inferior mechanical strength.

Nevertheless, in spite of the superior  $U\mu TS$  of SBU0, the bond strength to dentin was significantly worse than that of SBU100 and SBU50. This result once again shows that adhesion to tooth tissue is not related to mechanical strength alone but also to the interaction with the tooth substrate and the DC. Complete evaporation of water from an adhesive with a self-etch strategy would be problematic as the functional monomers need to be ionized before they can interact with hydroxyapatite in dentin. In addition, the increased viscosity after evaporation may adversely influence the interaction with dentin. Last, the inferior bonding performance of SBU0 may also be partially associated with the low DC on dentin.

To conclude, evaporation of adhesive ingredients did affect the DC, U $\mu$ TS, and microtensile bond strength of a universal adhesive, and the null hypothesis must thus be rejected. However, repeated opening of the bottle will only jeopardize the clinical performance of a universal one-step adhesive when more than 50% of the solvent and evaporable compounds have been evaporated. In our study, it took more than five days before 50% was evaporated (at room temperature), in spite of using an open bottle. It is thus unlikely that shelf life in a clinic will be impaired by evaporation, especially when the adhesive is used according to the manufacturer's instructions and when the adhesive bottle is recapped after every use.

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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 Long-term Simulated Pulpal Pressure on the Bond Strength and Nanoleakage of Resin-luting Agents With Different Bonding Strategies

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#### **Clinical Relevance**

Simulated pulpal pressure presented different effects on the long-term adhesive performance of the resin cements and should be considered when choosing a resin-based luting cement.

#### **SUMMARY**

This study evaluated the effects of simulated hydrostatic pulpal pressure (SPP) on the microtensile bond strength ( $\mu TBS$ ) to dentin and

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nanoleakage patterns produced by self-adhesive luting agents after 12 months. Three selfadhesive luting agents (RelyX Unicem [UN], RelyX U100 [UC], and Clearfil SA Luting [SA]) and three conventional luting agents (Rely X ARC [RX], Panavia F [PF], and a two-step selfetching adhesive system [Clearfil SE Bond] associated with Panavia F [PS]) were evaluated. One hundred twenty-three human molars were abraded to expose occlusal surfaces. Resin cements were used to lute cylindrical composite blocks to the teeth either subjected or not to SPP. Sixty specimens were subjected to 15 cm H<sub>o</sub>O of SPP for 24 hours before and 24 hours or 12 months after cementation procedures. Afterward, restored teeth were serially sectioned into beams with a cross-sectional area of 1 mm<sup>2</sup> at the bonded interface and were tested in tension (cross-head speed of 1 mm/ min). Failure mode was determined using scanning electron microscopy (SEM). Data were statistically analyzed by three-way analysis of variance and post hoc Tukey test (p=0.05). Two additional teeth in each group

were serially sectioned into 0.9-mm-thick slabs, which were submitted to a nanoleakage protocol with AgNO3 and analyzed with scanning and transmission electron microscopes. The µTBS values of the etch-and-rinse group (RX) were negatively influenced by SPP and long-term water storage with SPP. After 12 months, UC and SA presented premature failures in all specimens when submitted to SPP. SPP increased silver deposition in most groups in both evaluation times. The hydrostatic pulpal pressure effect was material dependent. The storage time without SPP did not affect bond strength. However, long-term SPP influenced the performance of the etch-and-rinse and self-adhesive cements regarding µTBS and nanoleakage pattern, except to UN.

#### INTRODUCTION

Resin-based luting agents were introduced to overcome the inherent problems of zinc phosphate cements and provide better handling and esthetic properties. To promote adhesion to tooth structures, conventional resin cements use etch-and-rinse or self-etching adhesive systems.<sup>2</sup> Dentin is the main substrate available for adhesion in prosthetic procedures, especially in vital teeth. Dentin is a hydrated hard tissue in the vital state, when there is an outward flow of dentinal fluid through the dentinal tubules with a positive pulpal pressure, estimated to be approximately 15 cm H<sub>2</sub>O.<sup>3</sup> Water presents deleterious effects for adhesive procedures, such as the plasticization of the polymer chains, leading to compromised mechanical properties and hydrolytic degradation of resin and collagen fibrils.4-8

The etch-and-rinse multistep adhesive technique has been considered to be complex and sensitive. This technique produces a complete smear layer and smear plug removal, increasing outward flow of dentinal fluid. In addition, an incompletely infiltrated hybrid layer has been reported. Some adhesive systems behave as semipermeable membranes and can allow outward fluid flow through the dentin-adhesive interface, even after polymerization. These disadvantages probably account for a higher incidence of postoperative sensitivity after bonding procedures, pulpal damage, and premature degradation of the resin-dentin interface. These

Self-etching adhesive systems were developed in an attempt to reduce the technique sensitivity of etch-and-rinse systems. Self-etching monomers simultaneously etch and infiltrate dentin, providing micromechanical retention after polymerization.<sup>20</sup> The maintenance of smear plugs when self-etching adhesive systems are used minimizes moisture contamination by dentinal fluid transudation when compared with the use of etch-and-rinse adhesives.<sup>21</sup> Simplified single-step all-in-one self-etching adhesives have been reported to allow water diffusion through the adhesive interface even after polymerization.<sup>13,14,16,22</sup>

A new type of luting material that does not require any pretreatment of the tooth surface with adhesive systems has been developed, the so-called selfadhesive cement. 23-25 This material aims to combine the favorable properties of conventional (zinc phosphate, glass ionomer, and polycarboxylate cements) and resin-luting agents. 25 After the first self-adhesive cement was developed (RelyX Unicem; 3M ESPE, St Paul, MN), it rapidly gained popularity among clinicians because of its simplified "mistake-free" application technique. Application on smear layercovered substrates maintains dentin permeability in very low levels, 15 contributing to reduced postoperative sensitivity and lower susceptibility to moisture degradation.26 Its interaction with subjacent dentin and enamel has been suggested to occur through formation of a hybridized complex and chemical interaction with hydroxyapatite.<sup>27</sup> However, limited information is available with regard to the bonding mechanism and nanoleakage of such products when they are applied to teeth under simulated pulpal pressure.  $^{25,28-30}$  Within this context, prolonged storage time with simulated pulpal pressure may compromise the sealing ability and mechanical properties of the bonded interface over time.

The aim of this study was to evaluate the effects of simulated pulpal pressure (SPP) on bond strength and nanoleakage in resin-dentin interfaces produced by different cementation strategies after 12 months. The research hypothesis was that bond strength provided by self-adhesive resin cements in indirect restorations is not as susceptible to the outward flow of dentinal fluid as regular resin-luting agents (resin cements/adhesive systems). In addition, it was anticipated that the changes in the nanoleakage patterns at the adhesive interface as a consequence of SPP are similar for multistep and self-adhesive resin cements.

#### **METHODS AND MATERIALS**

#### **Tooth Preparation**

One hundred sixty-eight recently extracted cariesfree third molars stored in 0.1% thymol (Symrise GmbH, Holzminden, Germany) solution at 4°C for no

longer than 3 months were used in this study. Teeth were obtained by protocols that were approved (SISNEP/384) by the review board of Guarulhos University (Guarulhos, São Paulo, Brazil). After disinfection and removal of soft tissues, flat middle depth coronal dentin surfaces were exposed with 600-grit SiC paper (3M of Brazil Ltd, Sumare, Brazil) under running water to create a standardized smear layer. Teeth had their roots removed using a diamond saw (ISOMET, Buehler, Lake Bluff, IL) 2 mm below the cement-enamel junction. Pulpal tissue was gently removed so as not to damage the predentin region.

Teeth were assigned to four experimental groups (with or without SPP and immediate or 12 months), which were distributed into 24 experimental subgroups (n=7) according to the luting technique, pulpal pressure, and storage time. Three selfadhesive cements, RelyX Unicem (UN), RelyX U100 (UC), Clearfil SA Luting (SA), and two conventional luting agents, one that uses a two-step etch-and-rinse adhesive (RelyX ARC [RX]) and one that uses a one-step self-etching adhesive (Panavia F [PF]), were used in this study. An additional group included the use of a two-step self-etching primer adhesive system (Clearfil SE Bond) prior to the application of Panavia F (PS). Luting agents were mixed and placed according to manufacturers' instructions (Table 1).

To simulate pulpal pressure on the dentin surface, each tooth was bonded to a Plexiglass platform (3 cm × 3 cm × 0.3 cm) penetrated by an 18-gauge stainless-steel tube and fixed with cyanoacrylate adhesive (Loctite Super Bonder Gel; Henkel, Düsseldorf, Germany). The pulp chamber was filled with distilled water via polyethylene tubing connected to a syringe barrel with 10 mL of distilled water and suspended 15 cm from the tooth crown. Thus, each specimen was connected to a hydraulic pressure device that delivered 15 cm water pressure. The teeth were kept under hydrostatic pressure for 48 hours or 12 months, starting 24 hours before luting procedures.

#### Luting Procedures for Microtensile Bond Strength (µTBS)

Five teeth of each group were used for the  $\mu TBS$  evaluation. Four-millimeter-thick composite resin discs 12 mm in 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 (700 mW/mm²) for 40 seconds with a halogen light

(Optilux 501; Kerr Corp, Orange, CA). 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. The composite surface was airborne-particle abraded with 50-µm aluminum oxide particles (Asfer Indústria Química Ltda, São Caetano do Sul, Brazil) for 10 seconds. Before 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).

The cementation procedures were randomly processed. Excess water was removed with cotton pellets. Care was taken not to dehydrate dentin surfaces. The application of adhesive systems (when necessary) and luting agents was made according to the manufacturer's instructions, and the composite resin disc was pressed on the cement using digital pressure, which was sustained until light curing was performed from the buccal and lingual sides. Specimens were exposed to light from the same halogen curing unit for 40 seconds on the buccal, lingual, and occlusal directions. Bonded specimens were stored in distilled water for 24 hours or 12 months, and the specimens submitted to SPP were kept under constant pulpal pressure during the same storage time.

#### **Luting Procedures for Nanoleakage Analysis**

Two specimens were prepared for each group. After luting agents were mixed and applied onto flat dentin surfaces, a polyester strip was placed over the luting agent and was used to apply proper digital pressure while the luting agent was light activated for 40 seconds. Afterward, a thin layer of a low-viscosity resin composite (Clearfil Majesty Flow, Kuraray Med. Inc, Kurashiki, Okayama, Japan) was applied and light activated for 40 seconds. After similar storage conditions described above (24 hours or 12 months, with or without SPP), teeth were sectioned perpendicular to the adhesive-tooth interface into 0.9-mm-thick slabs using a diamond saw (Isomet 1000; Buehler).

Bonded slabs were coated with two layers of nail varnish applied up to within 1 mm of the bonded interfaces. To rehydrate specimens and avoid desiccation artifacts,<sup>32</sup> they were immersed in distilled water for 20 minutes prior to immersion in the tracer solution of ammoniacal silver nitrate for 24 hours. Tooth slabs were placed in the tracer solution in total darkness for 24 hours, rinsed thoroughly in distilled water, and immersed in a photo-developing solution for eight hours under a fluorescent light to reduce

Туре	Manufacturers (Lot Number)	Delivery System (Cement)	Composition	Application Technique	
Dual- polymerizing	RelyX ARC (GEHG) + Adper Single Bond	Automatic dispenser, two pastes, hand	Cement: bis-GMA, TEGDMA polymer, zirconia/silica filler	a (15 s); b (15 s); c; d; e; i	
resin cement +	2 (7MY); 3M ESPE,	mixed for 10 s	Etchant: 35% H <sub>3</sub> PO <sub>4</sub>	(10 s); mix	
two-step etch- and-rinse adhesive system	ch- St Paul, MN		Adhesive: bis-GMA, HEMA, UDMA, dimethacrylates, ethanol, water, camphorquinone, photoinitiators, polyalkenoic acid copolymer, 5-nm silica particles	— cement; apply mixture	
Dual- polymerizing	Panavia F (paste A, 00249D; paste B,	One-step self-etching adhesive + resin	Primer A: HEMA, 10-MDP, 5-NMSA, water, accelerator	h (A+B) (leave	
resin cement + 1-step self-	0027A) + ED Primer (primer A, 00262A;	cement, dual polymerizing two	Primer B: 5-NMSA, accelerator, water, sodium benzene sulphinate	undisturbed for 60 s); mix	
etching adhesive	primer B, 00137A); Kuraray Medical, Inc, Tokyo, Japan	pastes, hand mixed -	Paste A: 10-MDP, silanated silica, hydrophobic aromatic and aliphatic dimethacrylate, hydrophilic dimethacrylate photoinitiator, dibenzoyl peroxide	mixture; i (40s)	
			Paste B: silanated barium glass, sodium fluoride, sodium aromatic sulfinate, dimethacrylate monomer, BPO		
Dual- polymerizing	Panavia F (paste A, 00249D; paste B,	Two-step self-etching adhesive + ED Primer + resin cement, dual polymerizing two	Primer: MDP, HEMA, hydrophilic dimethacrylate, dl- camphorquinone, N,N-diethanol p-toluidine, water	f (20 s); e; g; i (10 s); h (ED	
resin cement + two-step self- etching	0027A), Clearfil SE Bond (00788A); Kuraray Medical, Inc		Bond: MDP, Bis-GMA, HEMA, hydrophobic dimethacrylate, dl-camphorquinone, N, N-diethanol p-toluidine, silanated colloidal silica	Primer); e; mix cement; apply mixture; i (40 s)	
adhesive system		pastes, hand mixed -	Paste A and Paste B: as described above		
Dual- polymerizing self-adhesive	RelyX U100 (366321) 3M ESPE	Clicker dispenser two pastes, hand mixed	Base: glass fiber, methacrylated phosphoric acid esters, dimethacrylates, silanated sílica, sodium persulfate	Mix cement; apply mixture; i (40 s)	
resin cement		_	Catalyst: glass fiber, dimethacrylates, silanated sílica, p-toluene sodium sulfate, calcium hydroxide	_ ` `	
Dual- polymerizing self-adhesive	merizing (365979) 3M ESPE mecha		Powder: glass powder, silica, calcium hydroxide, self-curing initiators, pigments, light-curing initiators, substituted pyrimidine, peroxy compound	Automix cement; apply mixture i (40 s)	
resin cement			Liquid: methacrylated phosphoric esters, dimethacrylates, acetate, stabilizers, self-curing initiators, light-curing initiators		
Dual- polymerizing self-adhesive resin cement	Clearfil SA luting (008AA), Kuraray Medical, Inc	Dual polymerizing two pastes, hand mixed	Paste A: MDP, Bis-GMA, TEGDMA, Hydrophobic aromatic dimethacrylate dl-Camphorquinone, benzoyl peroxide, initiator, silanated barium glass filler, silanated colloidal silica	Automix cement; apply mixture, i (40 s)	
			Paste B: Bis-GMA, hydrophobic aromatic dimethacrylate, hydrophobic aliphatic dimethacrylate, accelerators, pigments, surface treated sodium fluoride, silanated barium glass filler, silanated colloidal silica		

Abbreviations: 10-MDP, 10-methacryloxydecyl dihydrogen phosphate; 4-META, 4-methacryloyloxyethyl trimellitate anhydride; 5-NMSA, N-methacryloxyl-5-aminosalicylic acid; Bis-GMA, bisphenol A diglycidyl ether methacrylate; HEMA, 2-hydroxyethyl methacrylate; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate.

silver ions into metallic silver grains within voids along the interface.

### **Scanning and Transmission Electron Microscopy**

For scanning electron microscopy (SEM) analysis, specimens were fixed in Karnovsky's solution and

embedded in epoxy resin (Epoxycure, Buehler). Afterward, they were polished with 400-, 600-, 1200-, and 2400-grit SiC paper and 6-, 3-, 1-, and 0.25-µm diamond paste (Arotec, Cotia, SP, Brazil). Then, specimens were dehydrated in ascending ethanol series and coated with carbon (MED 010, Balzers Union, Balzers, Liechtenstein). Resin-dentin

<sup>&</sup>lt;sup>a</sup> Application technique = a: acid etch; b: rinse surface; c: dry with cotton pellet; d: apply one bottle adhesive; e: gently air dry; f: apply primer; g: apply adhesive; h: apply mixture; i: light polymerize; j: autopolymerize.

interfaces were observed with an SEM (LEO 435 VP, LEO Electron Microscopy Ltd, Cambridge, UK) operated in the backscattered electron mode. After SEM analysis, representative leakage patterns at the cement-dentin interfaces produced by each system were photographed at 500× magnification.

For transmission electron microscopy (TEM) analvsis, specimens were examined with the TEM to compare silver uptake patterns along the resindentin interfaces. Undemineralized specimens were fixed in Karnovsky's solution, postfixed in osmium tetroxide, dehydrated in an ascending ethanol series, and embedded in epoxy resin (Dr Spurr, Electron Microscopy Sciences, Hatfield, PA). Representative 90-nm-thick ultrathin sections were prepared with an ultramicrotome (Leica UC6; Leica Microsystems, Wetzlar, Germany) and collected on 100-mesh copper grids. Without additional staining, they were observed in a TEM (Zeiss EM 900; Zeiss, Munich, Germany) operated at 80 kV. Silver deposition patterns were compared among the different luting products and storage conditions. Because of differences in hybrid layer thickness and in the magnifications required to characterize the interfaces, no attempt was made to quantify the silver deposits. Representative images were chosen to depict the most frequently observed aspect of the resin-dentin interfaces in the different testing conditions.

#### μTBS Evaluation

After the storage time was completed (24 hours or 12 months), the restored teeth were serially sectioned perpendicular to the adhesive-tooth interface into slabs and the slabs into beams with a cross-sectional bonded area of approximately 1 mm<sup>2</sup> using a diamond saw (Isomet 1000; Buehler). 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 cross-head speed of 1 mm/min until fracture occurred. Maximum tensile load was divided by specimen cross-sectional area to express results in units of stress (MPa). Five beams were selected from each restored tooth, and the average value for each tooth was used in the calculations. Bond strength values were evaluated statistically using three-way analysis of variance (ANOVA) and Tukey post hoc test (p=0.05). Pretest failures were not included in the statistical analysis. 33,34 Statistical analyses were performed using a statistical software program (SAS for Windows V8; SAS Institute, Inc, Cary, NC). Failure modes were determined by examination of fractured specimens with an SEM (LEO 435 VP; LEO Electron Microscopy Ltd). Fractured specimens were mounted on aluminum stubs and gold-sputter coated (MED 010; BAL-TEC AG, Balzers, Liechtenstein) prior to viewing at different magnifications. Failure mode at the fractured interface was classified into one of four types: CD (cohesive failure in Dentin), AD (adhesive failure between hybrid layer and dentin), CC (cohesive failure in the cement), or ADR (adhesive failure between the luting agent and composite resin). Instead of classifying failures as mixed, the area percentage of each type of failure in each specimen was recorded.

#### **RESULTS**

#### μTBS

The mean µTBS values are presented in Table 2. Three-way ANOVA revealed a significant difference for the factor "cements" (p=0.00001), for the factor "pulpal pressure," and for the interaction "cements × pulpal pressure  $\times$  storage time" (p=0.00028). For RX, SPP significantly reduced µTBS both at 24 hours and 12 months. However, the negative effects of SPP were more pronounced after 12 months. Storage in water for 12 months without SPP did not influence negatively µTBS values of RX. SPP and long-term storage did not affect µTBS values of PS and UN. When SPP was applied, a considerable reduction in μTBS of PF was recorded at both storage times. Even though the self-adhesive cement UC was positively influenced by SPP at 24 hours, the association of long-term storage and SPP was catastrophic to UC and SA, which presented 100% of pretest failures.

Without SPP at 24 hours, RX and PS presented the highest bond strength values (Table 2). PF, UN, UC, and SA did not present significant differences. With SPP at 24 hours, the highest µTBS values were produced by RX, PS, and UC, which did not differ among them. However, no difference in µTBS values was observed among UC, UN, and SA. PF showed the lowest µTBS but with no significant difference from UN and SA. Without SPP at 12 months, RX presented the highest bond strength values, followed by PS. The lowest  $\mu TBS$  at 12 months without SPP were recorded for PF, UN, UC, and SA, which did not differ among them. After 12 months of storage with SPP, the highest µTBS values were recorded for PS. RX, UN, and PF did not differ from each other after 12 months with SPP. UC and SA did not withstand the challenge and could not be tested.

Distribution of failure modes is presented in Figure 1. Cohesive failure in resin cement and

Table 2: Mean Bond Strength Values in MPa (Standard Deviation) for the Different Resin Cements Applied on Dentin With and Without Simulated Pulpal Pressure and Stored in Water for 24 Hours and 12 Months<sup>a</sup>

Product Type	Resin Cement	24 Hours		12 Months	
		No Pressure	Hydrostatic Pressure	No Pressure	Hydrostatic Pressure
Two-step etch-and-rinse adhesive/resin cement	RelyX ARC + Single Bond (RX)	53.0 (8.6) Aa	34.8 (11.3)ABb*	67.9 (11.0) Aa*	18.6 (17.9) Bb
Two-step self-etching adhesive/resin cement	Panavia F + Clearfil SE Bond (PS)	45.5 (6.9) Aa	38.0 (10.2) Aa	48.4 (14.6) Ba	57.5 (7.9) Aa*
One-step self-etching adhesive/resin cement	Panavia F (PF)	14.1 (4.6) Ba	7.8 (1.4) Db	22.5 (4.3) Ca	12.0 (4.2) Bb
Self-adhesive cement	RelyX Unicem (UN)	17.5 (7.4) Ba	20.4 (7.6) BCDa	18.5 (8.4) Ca	16.2 (10.5)Ba
Self-adhesive cement	RelyX U100 (UC)	14.2 (5.6) Ba	24.2 (2.3) ABCa*	19.9 (6.3) Ca	0.0 Cb
Self-adhesive cement	Clearfil SA Luting (SA)	13.1 (11.1) Ba	14.3 (5.3) CDa*	23.8 (16.2) Ca	0.0 Cb

<sup>&</sup>lt;sup>a</sup> Means followed by different upper case letters (columns), lowercase letters (rows within each hydrostatic pressure condition), and asterisks (rows within each time of storage) are significantly different by Tukey test at the 5% confidence level.

adhesive failure were predominantly observed in RX at 24 hours and PS at both times, regardless of the application of pulpal pressure. At 12 months, RX presented predominantly adhesive failure (AD). On the other hand, the predominant failure mode for all other groups was adhesive between dentin and the luting agent.

#### Nanoleakage

SPP increased nanoleakage in all groups, except for the self-adhesive cements UN and UC at 24 hours. The influence of storage time was material dependent. When RX was applied without SPP, silver deposition was observed in some regions within the hybrid layer (Figure 2). RX with SPP revealed silver deposition within the entire thickness of the hybrid layer and tags (Figure 2). After 12 months, increased silver deposition was observed for RX, increasing considerably more when it was submitted to SSP (Figure 2). PS with or without SPP presented small silver deposition at the bottom of the hybrid layer (Figure 3). After 12 months, no increase in silver deposition was observed for PS without SPP, and a moderate increase was observed with SSP. Some regions of the interface produced by PF without SPP presented adhesive layer, hybrid layer, and tags

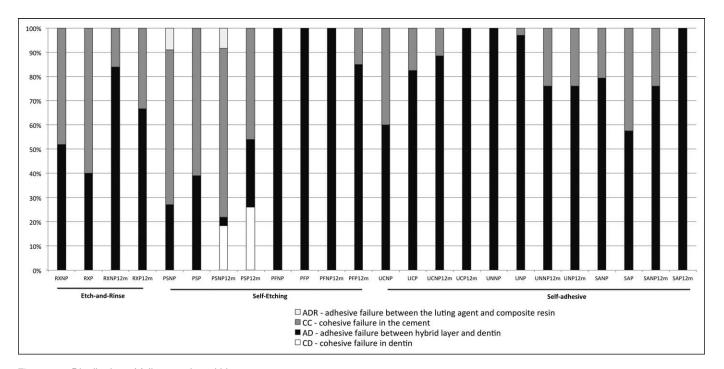


Figure 1. Distribution of failure modes within groups.

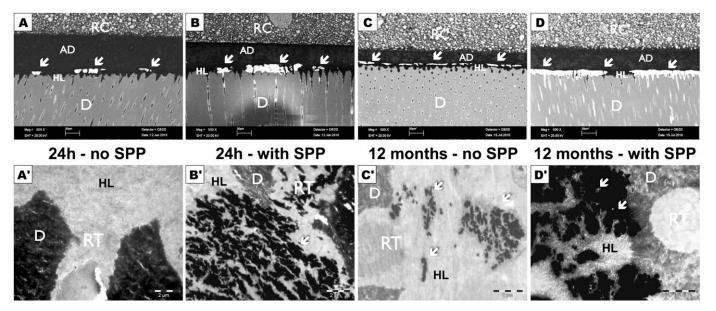


Figure 2. Representative backscattered SEMs and TEMs of the cement-dentin interfaces produced by the two-step etch-and-rinse system RelyX ARC (RX).

completely impregnated by silver (Figure 4). PF with SPP presented gaps between the cement layer and dentin (Figure 4). After 12 months, PF with and without SPP presented higher silver deposition than observed at 24 hours.

The self-adhesive cements presented lower silver impregnation than the other cement systems at 24 hours (Figures 5 through 7). UN, UC, and SA presented almost no or very little silver deposition

at the cement-dentin interface after 24 hours, with or without SPP (Figures 5 through 7). After 12 months of storage without SPP, a small increase in silver deposition was observed for SA and UC. However, when they were stored with SPP, gaps were frequently observed. The cement-dentin interface could not be observed for SA and UC after 12 months with SPP, because specimens did not survive SEM/TEM preparation procedures.

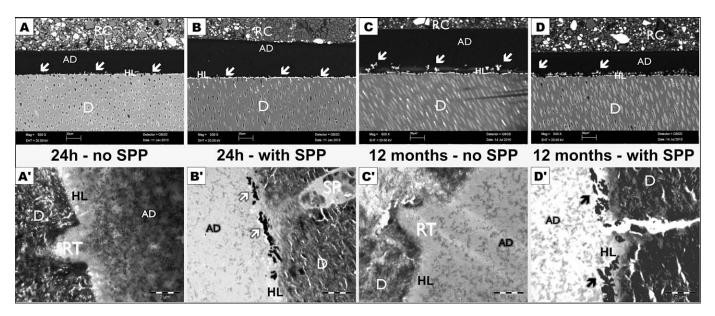


Figure 3. Representative backscattered SEMs and TEMs of the cement-dentin interfaces produced by the two-step self-etching system Panavia F + Clearfil SE Bond (PS).

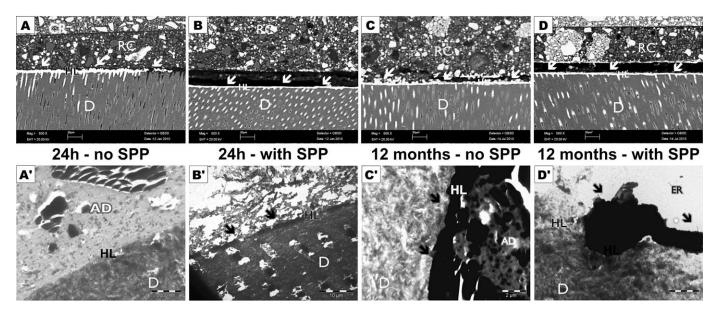


Figure 4. Representative backscattered SEMs and TEMs of the cement-dentin interfaces produced by the one-step self-etching system Panavia F (PF).

#### **DISCUSSION**

In this study, the etch-and-rinse group presented significantly decreased  $\mu TBS$  when it was submitted to SPP either after 24 hours or 12 months. This finding can be attributed to the diffusion of water into the hybrid layer, as noted by the silver deposition within the hybrid layer (Figure 2). As a consequence, the mechanical properties of the polymer within dentin are compromised by swelling

and decreased frictional force between the polymer chains, a process known as plasticization.  $^{5,19,35}$ 

Even in the absence of SPP, all conventional cement systems presented a certain degree of nanoleakage, which was located mainly within the hybrid layer (Figures 2 through 4). The degree of silver deposition depends on the adhesive used, application mode, and composition. The presence of water on the adhesive composition plays an important role in both total- and self-etching techniques, as water-based

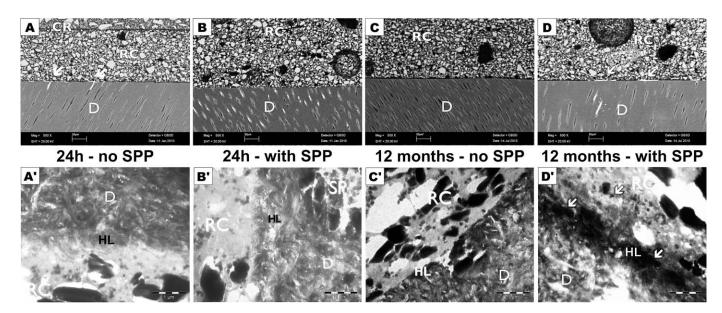


Figure 5. Representative backscattered SEMs and TEMs of the cement-dentin interfaces produced by the self-adhesive cement RelyX UNICEM (UN).

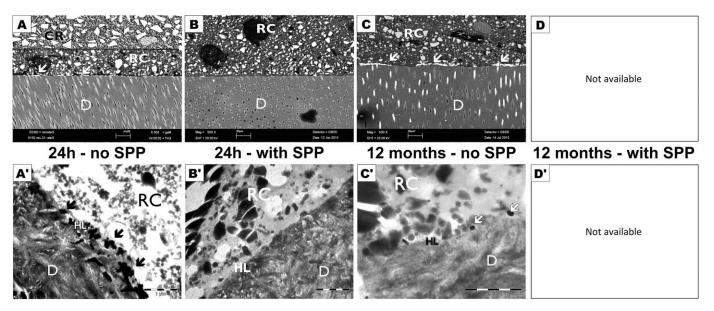


Figure 6. Representative backscattered SEMs and TEMs of the cement-dentin interfaces produced by the self-adhesive cement RelyX U100 (UC). Figures 6D/6D' are not presented because specimens did not resist electron microscopy preparation procedures after being stored for 12 months with simulated pulpal pressure.

adhesives used in total-etch systems can solvate dried matrices and reexpand dentin collagen. Probably, water was not eliminated during the adhesive procedures, producing the nanoleakage patterns observed in Figures 2A and 2A'. Besides the presence of water in their composition, the two-step etch-andrinse adhesive produces a semipermeable membrane due its high concentration of hydrophilic monomers and solvents. <sup>13,14</sup> Hydrophilic resin monomers attract

water molecules and permit their movement from dentin across the adhesive layer through water channels. Such a statement is confirmed in Figures 2B and 2D, which show an evident increase in nanoleakage, probably due to fluid flow produced by SPP. This water-filled channel has been considered to be a site of hydrolytic degradation and crack propagation during μTBS testing. 38,39

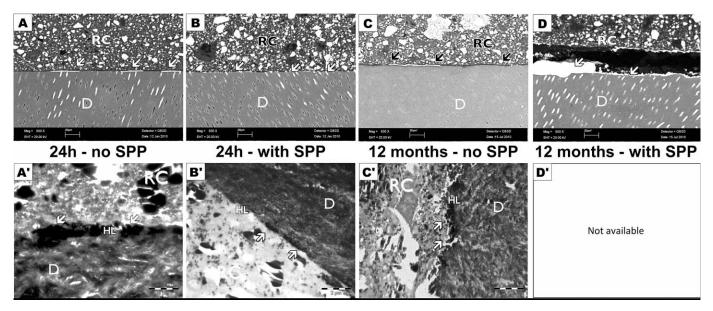


Figure 7. Representative backscattered SEMs and TEMs of the cement-dentin interfaces produced by the self-adhesive cement Clearfil SA Luting (SA). Figure 7D' is not presented because specimens did not resist electron microscopy preparation procedures after being stored for 12 months with simulated pulpal pressure.

Source	df	SS	MS	f	p Value
Cement	5	25383.1963793	5076.6392759	63.6640	0.00001
Pressure	1	2735.7870911	2735.7870911	34.3084	0.00001
Time	1	14.7899862	14.7899862	0.1855	0.67154
Cement × Pressure	5	4084.4360507	816.8872101	10.2442	0.00001
Cement × Time	5	1267.3700452	253.4740090	3.1787	0.01074
Pressure × Time	1	1299.0906566	1299.0906566	16.2914	0.00028
Cement × Pressure × Time	5	2209.5092822	441.9018564	5.5417	0.00031
Error	96	7655.1477991	79.7411229		
Total	119	44649.3272904			

Table 3: Three-Way Analysis of Variance<sup>a</sup>

Another phenomenon that contributes to reduction in µTBS values is water diffusion from the underlying hydrated dentin structure across the polymerized hydrophilic adhesive layer via an osmotic gradient during the slow setting process of these resin cements. 13 According to Hiraishi and others, 15 continuous water uptake via the adhesive layer could result in an unstable porous region, increasing the degradation along the interface between the adhesive and resin cement. As a result, this bonding interface turns into a weak link when pulpal pressure is simulated.<sup>2,40</sup> Porous regions were observed in fractured specimens of RX with SPP, suggesting the presence of water channels (Figure 2E,F,G). At higher magnification, nonuniform globular structures were identified, which might suggest poor polymerization (Figure 2E,F,G). In addition, backscattered SEM micrographs of PF subjected to SPP after 24 hours and 12 months revealed silver deposition between the adhesive cement and resin (Figure 4), suggesting permeability within this system.

When SPP was applied for 24 hours or 12 months, PF presented a significant reduction in µTBS values. Previous studies have reported a negative influence of pulpal pressure for Panavia F. <sup>2,15</sup> This performance is probably associated with a high concentration of hydrophilic and ionic resin monomers in ED Primer, resulting in the formation of a highly permeable layer after polymerization. 13,41 Because of its high permeability, 15 primed dentin allows water to diffuse from dentin across the hybrid layer and form water droplets in the interface (Figure 4), resulting in low μTBS. Hydrophilic monomers, such as HEMA, can attract water, which impairs the cement polymerization, reducing mechanical properties. 41 This statement can be confirmed by the high amount of silver deposition (Figure 4), even without SPP. As previously demonstrated, 42 for an ideal bonding performance, the water concentration must be sufficient to

provide adequate ionization of the acidic monomers but without lowering the resin concentration too much to optimize their bonding efficacy to dentin. The high water content at the interface during polymerization setting probably contributed to the reduction in monomer concentration when PF was submitted to SPP. This fact can explain the lower µTBS associated with gap formation at the interface. The failure modes for PF were exclusively adhesive between resin cement and dentin. The fracture of PF groups with SPP occurred frequently on top of the hybrid layer, where it was not possible to visualize the tubular lumen, suggesting a poorly cured adhesive layer. Besides, it could be speculated that inhibition of the polymerization of the luting agent (Panavia F) could occur due to the presence of acidic monomers within the ED Primer composition. 41 However, as light activation was performed immediately upon luting, this effect is probably negligible.<sup>28</sup>

Infiltration of hydrophobic monomers from Clearfil SE Bond into primed dentin along with direct light activation of the adhesive system probably resulted in a better monomer conversion and stronger network polymer within the hybrid and adhesive layers, resulting in the significantly higher μTBS of the PS group in comparison to the values of PF group and in the highest µTBS after 12 months with SPP. This hydrophobic layer can reduce permeability of dentinal fluids between dentin and the luting agent and result in better long-term performance. 2,43,44 In addition, the presence of smear plugs is very important to reduce significantly the rate of fluid flow through the interface even in the presence of intrapulpal pressure, in vivo and in vitro. 14,16,45

The self-adhesive resin cements maintained their  $\mu TBS$  when submitted to SPP for 24 hours. Three different self-adhesive materials were used in this study. The self-adhesive cements RelyX U100 (UC)

<sup>&</sup>lt;sup>a</sup> Coefficient of variation = 35.588%

and RelyX Unicem (UN) were developed by the same manufacturer and are marketed under the same name in some countries. According to the manufacturer, the only difference between these products is the delivery system. While UN requires an activator, triturator, and applicator, UC can be hand mixed. Another self-adhesive cement used was Clearfil SA Luting, which needs to be hand mixed. When SPP is applied, water transudation increases the acidic monomers' aggressiveness, improving smear layer dissolution and dentin demineralization. 42 It also optimizes the acid-basic reactions, allowing better setting.26 The favorable µTBS and very low silver impregnation (Figures 5 through 7) observed for selfadhesive resin cements have been attributed to the micromechanical retention and chemical interaction between monomer acidic phosphate groups and dentin-enamel hydroxyapatite. 15,25,26 This possible improvement produced by water was evidenced by the µTBS values and favorable nanoleakage patterns observed when UC and UN were initially submitted to SPP (Figures 5 and 6).

However, despite the favorable  $\mu TBS$  results observed at 24 hours with and without SPP, and after 12 months without SPP, long-term storage with SPP resulted in debonding of the indirect restorations cemented with UC and SA prior to testing. The low initial pH of UN and UC (pH<2 in the first minute, according to the manufacturer) and SA (pH 2-3 in the first minute, according to the manufacturer), produces almost no demineralization and hybrid layer formation on dentin surface. \$^{18,28,46-48}\$ The present findings demonstrated that this interaction was not strong enough to resist 12 months of storage with SPP for UC and SA.

During polymerization of the self-adhesive resin cements (UN and UC), an increase in pH from 1 to 7 is observed as a consequence of the reaction between phosphate groups and both alkaline filler particles and hydroxyapatite from enamel and dentin, to neutralize resin acidity. 49,50 The presence of calcium hydroxide can also act in pH neutralization. The pH neutralization results in water formation and a more hydrophilic cement, which enhances the cement's wetting ability on the dentin surface and the cement tolerance to water. Water is crucial for self-adhesive luting agents to release hydrogen ions required for smear layer demineralization<sup>51</sup> and is also reused in the reaction between multifunctional acidic phosphate monomers and alkaline filler particles. Such a phenomenon is speculated to be responsible for a change in the nature of the cement from hydrophilic to hydrophobic, which is thought to improve adhesive stability. This hydrophobic characteristic is desired for bonded interfaces, because it can prevent hydrolytic degradation and consequently improve the long-term durability of indirect restorations. After 12 months of storage with SPP, UN demonstrated bond strength stability and very low silver deposition throughout the study.

Except for the materials that presented a high number of pretest failures, all groups presented acceptable bond strengths throughout the evaluation period. However, the groups that presented the highest bond strength values are more indicated for cementation of indirect restoration in lessretentive cavity preparations.

#### CONCLUSION

Based on the results of this study, it was concluded that the hydrostatic pulpal pressure effect was material dependent. Storage in water for 12 months without simulated pulpal pressure did not affect the bond strength for all materials. However, long-term storage with simulated pulpal pressure influenced the performance of the etch-and-rinse adhesive system RelyX and the self-adhesive cements U100 and Clearfil SA luting regarding  $\mu TBS$  and nanoleakage patterns. Panavia F+ Clearfil SE Bond and Unicem presented bond strength stability and low silver deposition throughout the study regardless of the application of simulated pulpal pressure.

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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 Toothbrushingmouthrinse-cycling on Surface Roughness and Topography of Nanofilled, Microfilled, and Microhybrid Resin Composites

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### Clinical Relevance

The use of alcohol-containing mouthrinses, associated with toothbrushing, may increase the roughness and topography changes of resin composites over time. The microhybrid resin composite was more susceptible to increased roughness than were the nanofilled and microfilled composites.

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### **SUMMARY**

The purpose of this study was to evaluate the influence of toothbrushing-mouthrinse-cycling (TMC) on the surface roughness and topography of three resin composites with different filler particle systems (Z350, nanofilled [Nf]; Durafill, microfilled [Mf], and Empress Direct, microhybrid [Mh]). Twenty specimens of each resin composite (8.0 mm diameter and 2 mm height) were randomly divided into four groups (n=5) according to the mouthrinses: alcohol-free (Plax - P) and alcohol-containing (Listerine - L and Plax Fresh Mint - PM) and artificial saliva (control - AS). The specimens were submitted to TMC for nine

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weeks. A surface roughness tester and a threedimensional profilometer were used to measure the roughness (Ra) and the topography (Sa) before and after TMC. The data were analyzed by multifactor analysis of variance and Tukey post hoc test ( $\alpha$ =0.05). In all media, Mh presented greater roughness than Mf (p < 0.05). The highest value of roughness was presented by Mh immersed in L (p < 0.05). The lowest values of roughness were presented by Mf (p < 0.05). The three resin composites presented the highest roughness after immersion in mouthrinses containing alcohol (PM and L) (p < 0.05). For the three resin composites, the increase in roughness was noticeable after the fifth week. Topographic analysis showed that the smoothest surfaces were present after immersion in AS.

### INTRODUCTION

Resin-based composites are currently the most used material in the field of restorative dentistry. 1 Basically, these materials are composed of three chemically different phases: a polymeric matrix of dimethacrylate monomers; filler particles (dispersed phase); and an organosilane, a coupling agent that bonds the fillers to the polymeric matrix. The most used method of classifying resin-matrix composites is based on their filler particle system: hybrid (0.5-3) μm), microhybrid (0.4-1 μm), and microfilled (0.04-0.4 µm). A new class of resin-matrix composite, with filler particles exclusively in the nanoscale, ie, from 0.1 to 100 nm size range, was recently made available. Nanocomposites claim to combine the mechanical behavior of hybrid and microhybrid composites with the improved surface properties of microfilled composites (lower roughness and best gloss retention). 2,3 Recent in vitro 4-6 and clinical studies<sup>7,8</sup> have shown that nanocomposites present similar or better behavior when compared with hybrid composites.

As a polymer-based material, resin-based composites may undergo degradation when exposed to the oral environment. This degradation process may lead to several drawbacks, such as an increase in wear and surface roughness. 9,10 Among other aspects, the surface topography of resin composites plays a crucial role on the accumulation of dental biofilm. 11,12 Additionally, it is known that increasing dental biofilm deposits may lead to periodontitis and secondary caries lesions around tooth-resin composite interfaces. 13

Toothbrushing is the most used and efficient mechanical method for removing dental biofilm from all accessible tooth surfaces. <sup>14,15</sup> However, published studies have shown that this method may cause tooth <sup>16,17</sup> and resin composite abrasion. <sup>18</sup> Specifically with resin composites, this abrasion increases the surface roughness, accelerating the staining produced by pigments from beverages and interfering with color appearance over time. <sup>19-21</sup>

In addition to toothbrushing, mouthrinses are widely used to complement the cleaning of the oral cavity and as an antimicrobial agent to prevent and control periodontal diseases. Irrespective of the therapeutic indication, these substances have been used without professional prescription in an attempt to reduce halitosis and to improve the freshness of the oral cavity.<sup>22</sup> In general, mouthrinses contain salts, hydrogen peroxides, antimicrobial agents, pigments, emulsifiers, solvents, acids, and alcohol, diluted in an aqueous solution.<sup>23</sup> Previous studies have shown that alcohol-containing mouthrinses negatively influenced the properties of resin composites, such as sorption and solubility, a roughness, hardness, and color change. Although previous studies have analyzed the effect of toothbrushing and mouthrinses on the roughness of many resin composites, 3,5,10,16,18,20,25,26 little information is available with regard to the association of these mechanisms.<sup>30</sup> Therefore, the purpose of this study was to evaluate the roughness and the topography of resin-based composites submitted to toothbrushing-mouthrinse-cycling (TMC). The research hypotheses were: 1) microfilled and nanofilled composites will present lower roughness and less topographic change than will a microhybrid resin composite, and 2) the alcohol-containing mouthrinses will produce higher roughness and greater topographic change in the resin composites.

### **METHODS AND MATERIALS**

Three resin composites with different types of filler particles were analyzed: Filtek Z350 (nanofilled – Nf), Durafill (microfilled – Mf), and Empress Direct (microhybrid – Mh). Their compositions are depicted in Table 1. Three mouthrinses, chosen based on their alcohol concentration, were used in the current study (Table 2): one alcohol-free (Plax – P) and two containing alcohol (Listerine – L and Plax Fresh Mint – M). Artificial saliva was used as a control (AS). The pH of all substances was measured in triplicate using a pH meter (HI 3220, Hanna Instruments, Woonsocket, RI, USA). The pH was measured by placing a 1.5-cm diameter glass pH

Resin Composite	Composition	Shade
Filtek Z350 (nanofilled)	Filler: 59.5 vol% combination of aggregated zirconia/silica cluster ranging from 0.6 to 1.4 $\mu$ m with primary particles size, 5-20 nm, and nonagglomerated 20-nm silica filler	А3
	Polymeric matrix: Bis-GMA, Bis-EMA, UDMA, and TEGDMA	
Empress Direct	Filler: Ba-Al-fluorosilicate glass from 0.4 to 0.7 μm (52 vol%)	А3
(microhybrid)	Polymeric matrix: Dimethacrylate, Bis-GMA, UDMA	
Durafill (microfilled)	Filler: 40 vol% combination of silicon dioxide cluster ranging from 0.04 to 0.07 μm with prepolymerized particles, size 10 to 20 μm	А3
	Polymeric matrix: Bis-GMA, UDMA, and TEGDMA	

electrode (HI 1131, Hanna Instruments) into 20 mL of each substance.

### **Specimen Preparation**

Twenty disc-shaped specimens (8.0 mm diameter and 2 mm height) for each resin composite were built up using a Teflon ring matrix. After the matrix was filled, the material was covered with a polyester strip and a glass slab, with a device providing compression (500 g) for 30 seconds to eliminate porosities. The specimens were then light-activated at a central and at four overlapped points on their surfaces, using a quartz-tungsten-halogen unit (Optilux 501, Demetron Inc, Danbury, CT, USA) with an irradiance of 650 mW/cm<sup>2</sup> for 40 seconds. After 24 hours in water storage at 37°C, all of the specimens were sequentially polished with silicon carbide papers: 1200- and 4000-grit (Arotec, Cotia, SP, Brazil) under constant water irrigation (DPU 10, Struers, Ballerup, Denmark). After polishing, the specimens were randomly divided into four groups (n=5) according to the mouthwash and control group (artificial saliva).

### **Surface Roughness Analysis**

All of the specimens had their surface roughness evaluated using a surface roughness tester (Surftest SJ 201, Mitutoyo, Tokyo, Japan). Five traces of roughness, in different locations, using a 0.8-mm cutoff and a speed of 0.1 mm/s, were recorded for each specimen. The average surface roughness (Ra,  $\mu$ m) was determined for each specimen. The Ra parameter was obtained using the following equation:

$$R\alpha = \frac{1}{L} \int_{0}^{L} |f(x)| dx$$

where the roughness curve is expressed in y = f(x), and L is the sample length.

### **Topographic Analysis**

The topographic analysis was performed using a three-dimensional (3D) profilometer (Form Talysurf 60i, Taylor Hobson, Leicester, UK). For each specimen, an area of 1 mm $^2$  (1 × 1 mm) was scanned with a 20-nm z-resolution, employing 4000 steps in the x-axis and a spacing of 2  $\mu$ m in the y-axis. The roughness of the 3D reconstructed images was obtained using the 3D Sa parameter (average absolute deviation of the surface), using the following equation:

Table 2: Composition and Characteristics of the Substances Used in This Study						
Substance	Alcohol Content, (v/v%)					
Artificial saliva	KCI, NaCl, MgCl, CaCl, nipacin, carboxymethyl, cellulose, sorbitol, and deionized water	6.4	0			
Listerine	Ethanol, benzoic acid, eucalyptol, menthol, methyl salicylate, thymol	4.1	26.9			
Plax Fresh Mint	Triclosan 0.03%, sodium fluoride 0.025%, gantrez 0.2%	6.5	6			
Plax	Triclosan 0.03%, cetylpyridinium chloride 0.05%	5.7	0			

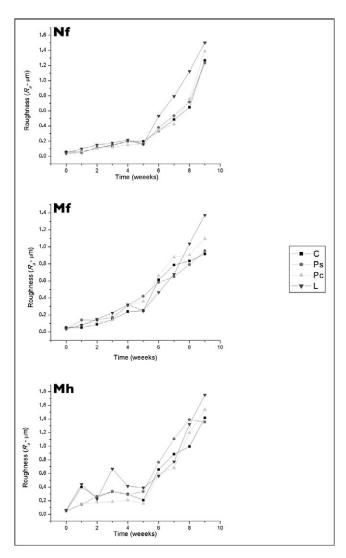


Figure 1. Increase in mean surface roughness (Ra,  $\mu m$ ) as a function of time (weeks).

$$Sa = \frac{1}{MN} \sum_{k=0}^{M-1} \sum_{l=0}^{N-1} |z(x_k; y_1)|$$

where z is the height of the measured point in the coordinates x and y.

### Toothbrushing-Mouthrinse-Cycling

In the first week, the specimens were submitted daily to TMC as follows: storage for 4 hours in 2 mL of artificial saliva at 37°C, brushing (20 strokes [Oral B 30, Procter & Gamble, São Paulo, SP, Brazil]), storage in artificial saliva at 37°C for 8 hours, brushing (20 strokes [Oral B 30, Procter & Gamble]), and storage in artificial saliva at 37°C overnight (12 hours). Brushing was carried out by using a toothbrushing machine (MEV2, Odeme Biotechnolo-

gy, Joacaba, SC, Brazil) and a toothpaste slurry in a proportion of 1:2 by weight (18 g of Paradontax [GlaxoSmithKline, Rio de Janeiro, RJ, Brazil] and 36 mL of distilled water). After each brushing, the specimens were abundantly rinsed with distilled water and immersed in 2 mL of mouthrinse or artificial saliva (control) for 2 minutes. The specimens were then washed in distilled water and replaced in artificial saliva. From the second to the ninth week, the specimens were maintained in artificial saliva for seven days and submitted to 280 strokes of brushing and immersed for 28 minutes in mouthrinse or artificial saliva (control). The surface roughness was reevaluated at the end of each week and the topography reevaluated after the ninth week.

### **Statistical Analysis**

The obtained data were analyzed using Statgraphics Centurion XVI software (StatPoint Technologies Inc, Warrenton, VA, USA). Initially, the normal distribution of the errors and the homogeneity of variances were checked by Shapiro-Wilk test and Levene test. Based on these preliminary analyses, the roughness data were analyzed by multifactor analysis of variance (MANOVA) and Tukey *post hoc* test. The baseline and final values of Ra and Sa were submitted to linear regression analysis. All analyses were performed at a significance level of  $\alpha$ =0.05.

### **RESULTS**

The results of the MANOVA showed that the three independent factors (resin-based composite, mouthrinse, and time of evaluation), as well as all of the interactions, were found to be significant (p<0.05). Figure 1 presents the roughness from the baseline to the ninth week for each resin composite in all media. It can be noted that, in general, the increase in roughness was notable after the fifth week. Table 3 shows the mean roughness at the end of the experimental protocol. In all media, Mh presented

Table 3: Final Means (Standard Deviations) of Roughness (Ra,  $\mu m$ )<sup>a</sup>

Media	Nanofilled	Microfilled	Microhybrid			
Artificial saliva	1.27 (0.06) <sup>a,A</sup>	0.92 (0.09) <sup>a,B</sup>	1.42 (0.22) <sup>a,b,C</sup>			
Plax	1.24 (0,09) <sup>a,A</sup>	0.95 (0.05) <sup>a,B</sup>	1.36 (0.19) <sup>a,A</sup>			
Plax Fresh Mint	1.39 (0.10) <sup>a,b,A</sup>	1.09 (0.05) <sup>a,B</sup>	1.54 (0.20) <sup>b,A</sup>			
Listerine	1.50 (0.07) <sup>b,A</sup>	1.37 (0.17) <sup>b,A</sup>	1.75 (0.24) <sup>c,B</sup>			

<sup>&</sup>lt;sup>a</sup> In columns, means followed by the same lowercase letters are not statistically different (Tukey test, p>0.05). In rows, means followed by the same uppercase letters are not statistically different (Tukey test, p>0.05).

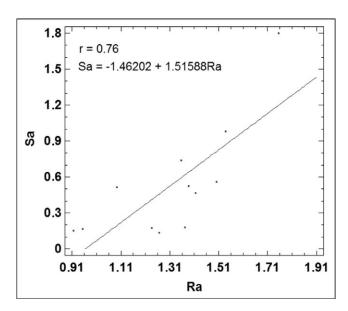


Figure 2. Regression line (linear model) of Ra vs Sa (r=0.76, Sa = -1.46202 + 1.51588Ra).

higher roughness values than Mf (Tukey test, p < 0.05). The highest value of roughness was presented by Mh immersed in L (Tukey test, p < 0.05). The lowest values of roughness were presented by Mf (Tukey test, p < 0.05). The three resin composites presented the highest roughness after immersion in alcohol-containing mouthrinses (PM and L) (Tukey test, p < 0.05). Figure 2 shows the plot of Ra against Sa. Linear regression analysis found a significant correlation (p = 0.0038) between the two parameters (r = 0.76, Sa = -1.46202 + 1.51588Ra)

Representative 3D reconstructed images showing the topography of the resin composites at the end of the experimental protocol are shown in Figure 3. It can be noted that specimens immersed in AS presented the smoothest surfaces.

### **DISCUSSION**

Many published studies have separately analyzed the *in vitro* influence of brushing<sup>3,5</sup> and mouthrinses<sup>26,27</sup> on the surface changes of resin composites. However, it is well known that the degradation of resin-based materials in the oral environment is a complex process, which involves mechanical and chemical mechanisms.<sup>10,24</sup> This was the rationale to employ TMC in the present study. This was performed in an endeavor to simulate actual conditions in the oral environment.

Several published studies have shown that microfilled and nanofilled resin composites present the lowest roughness immediately after polishing. 2-4,31 Based on this, clinicians assume that these materials are the most adequate for building up anterior restorations. Moreover, the characteristic of the filler particle system (concentration, size, and shape) is the most crucial factor affecting the wear of resin composite. 25,32 These aspects conducted the choice of the resin composite used in the present study (nanofilled, microfilled, and microhybrid). This was done in an attempt to investigate whether the type of resin composite is really a matter of concern on the clinical protocol for resin composite restorations. Although it is not a dental clinical procedure, the polishing of all specimens with 1200- and 4000-grit silicon carbide papers at the beginning of the experimental protocol aimed to ensure that all specimens had similar initial roughness values so the final results represented the actual material behavior.<sup>5</sup> In the present study, the coefficient of variation of the groups ranged from 4.6 to 15.4. These values can be seen as the overall data presenting a low variability, thereby supporting the sample size of n=5.

The clinical relevance of surface roughness can be demonstrated in two ways. Firstly, this property is strongly related to the bacterial colonization of surfaces located in the oral environment. A surface roughness above 0.2 µm has been reported to increase the colonization and adhesion of bacteria on composite surfaces. Moreover, it has been established that a higher surface roughness (difference between peaks and valleys) provides a reduced possibility of dislodging the oral biofilm, 33,34 a periodontal health concern. Secondly, an increase in roughness can interfere with changes in color and gloss of composite restorations, 5,18,35 an esthetic concern.

Analysis of Table 3 and Figure 3 indicates that in all media, Mf presented lower roughness and less topographic change than Mh. Moreover, after immersion in AS and L, Nf also presented lower roughness and fewer topographic changes than Mh. These findings lead to the acceptance of the first research hypothesis. The final values of roughness ranged from 0.92 to 1.75 µm and are in agreement with previously published results. 2,5,28 Among other factors, the roughness of resin composites is directly related to the features of their filler particle systems, ie, amount, size, shape, hardness, and interparticle spacing. <sup>25,36</sup> These aspects can be used to explain the poorest behavior presented by Mh. Even when considering that filler particles and polymeric matrix are bonded with a silane-coupling agent, the first

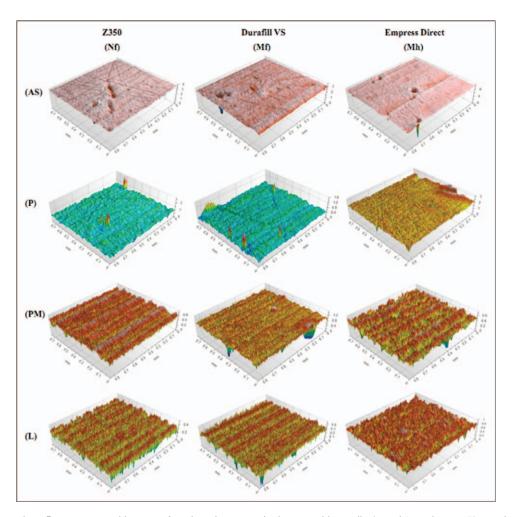


Figure 3. Representative 3D reconstructed images of each resin composite immersed in media (α and β angles = 45°, z axis in μm).

may be plucked out during brushing. Thus, it is possible that, due to the greater size, the filler particle of Mh (0.4-0.7 µm) protruded more through the composite surface than did the filler particle of the Nf (5-20 nm) and Mf (0.05-0.07 µm). This extended protrusion provided longer cantilevers, leading to higher angular moments that facilitated the fillers being pulled out from the material.<sup>37</sup> The variation in roughness seen in Figure 1 may reinforce this hypothesis. It is possible that, in some measurements (first and third weeks), the roughness of this resin composite was influenced by the deep valleys produced for some filler particle dislodgment and in a subsequent measurement, the wear of polymeric matrix around these valleys produced a less rough surface.

With the exception of the L group, the final roughness of Nf was always statistically higher than the roughness values of Mf. However, it can be noted that until the fifth week the increasing roughness of

Nf was slightly lower than that of Mf (Figure 1b,c). The higher concentration of filler particles of Nf (59.5 vol%) may be used to explain this behavior. It is known that when there is a higher content of smaller filler particles (as found with Nf), the distance between neighboring particles is small, which may act as a barrier against polymeric matrix wear. 37,38 Thus, the current authors hypothesized that the polymeric matrix of Nf was less worn than that of Mf during the first stage of TMC, which had only 40 vol% of filler particles. On the other hand, it is possible that the continuing TMCs increased the removal of zirconia/silica clusters of 0.6 to 1.4 μm, inducing fatigue cracks between fillers and organic matrix and causing gradual matrix destruction and increasing the loss of material.<sup>39</sup> Consequently, the final roughness of Nf was increased.

Although Mf had larger filler particles among the analyzed materials (prepolymerized fillers of 10 to 20  $\mu$ m), its roughness was the lowest (Table 3). This can

be explained by the fact that these prepolymerized particles are also formed by monomers (isofillers). Consequently, it is possible that these particles were worn away during TMC, rather than by being plucked out, so that the final roughness of Mf reflected only the size of its primary filler particles of 0.04 to 0.07  $\mu m$  (40 to 70 nm). The smoothest surfaces were provided by Mf in all media (Figure 3), which might serve as representative of this phenomenon.

Since the alcohol-containing mouthrinses produced greater roughness and topography changes in the resin composites (Figure 3; Table 3), the second research hypothesis of the present study was accepted. This result was expected and can be explained by the plasticizing effect of ethanol. This polar solvent penetrates into the resin composite, causing material swelling, pulling apart the polymeric matrix chains, and decreasing its crosslinking density, resulting in a decrease in wear resis $tance^{27,40,41}$  and potentiating the deleterious effects of brushing. The findings of Almeida and others<sup>42</sup> may reinforce this discussion. These authors found that the sorption and solubility of a hybrid and a nanofilled resin composite were higher after immersion in alcohol-containing mouthrinses and claimed that this was due to the swelling of their polymeric matrixes produced by the ethanol contained in the mouthrinses and increasing the elution of nonreacted monomers and oligomers from those materials.

Listerine was the medium that produced the greatest increase in roughness in the three resin composites (Table 3). In addition to the greater content of ethanol (26.9 v/v%), it is possible that its low pH (4.1) contributed to this result. It is well established that the ester groups present in dimethacrylate monomers, such as those present in the resin composites in the current study (ie, Bis-GMA, UDMA, Bis-EMA, and TEGDMA), can undergo degradation through hydrolysis in environments with low pH. 43 This hydrolysis may produce surface erosion and dissolution, negatively affecting the wear, hardness, and surface integrity by softening the matrix and causing a loss of structural ions. 44 It is possible that these aspects act synergistically to potentiate the negative effects of toothbrushing, thereby increasing the roughness of the resin composites.

The data provided in Figure 1 are noteworthy. Although the roughness slowly increased from the first to the fifth week for the three resin composites (approximately 15% to 20% of the total roughness),

there was a notable increase from the fifth to the ninth week. Moreover, the final values of roughness (Table 2) were far greater than the values found in other studies that evaluated toothbrushing  $^{10,39,45}$  or mouthrinse immersion. 26,28,46,47 However, Sadaghiani and others<sup>46</sup> found that when mouthrinses and toothbrushing were combined, the effect on the roughness of modified glass-ionomer restorative materials was at least twice that obtained after immersion in mouthrinses alone. The authors theorized that a superficial layer of material was removed during each episode of toothbrushing, exposing a fresh surface to be attacked by the mouthwash at the next immersion. They further indicated that this interaction between chemical erosion and mechanical abrasion was considered to have contributed to the progressive roughening of the surfaces. Thus, it is reasonable to speculate that the effect described by the former study took place in the present study, since TMC combined toothbrushing and immersion in mouthrinses. The similarities among all curves depicted in Figure 1 suggest a common roughening effect that could be used to reinforce this possibility.

### CONCLUSIONS

Within the limitations of the present study, it can be concluded that:

- Models that involve the combined use of toothbrushing and mouthrinse immersion could be more useful to obtain results that are clinically more accurate for roughening resin composites;
- Microfilled resin presented the best behavior (lower roughness and less topographic changes), suggesting that it is more adequate to be used in the surface layer of anterior restorations; and
- Alcohol-containing mouthrinses can increase the roughness of resin composites. Thus, clinicians, especially periodontists, should consider this when prescribing these substances to their patients.

### 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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# Survival Rate, Load to Fracture, and Finite Element Analysis of Incisors and Canines Restored With Ceramic Veneers Having Varied Preparation Design

CD Bergoli • JBC Meira • LF Valandro MA Bottino

### **Clinical Relevance**

Fracture loads of canines and maxillary central incisors are not affected by the veneer preparation design (eg, conservative or conventional preparation with palatal chamfer). However, conventional preparation generated higher tensile stress in the ceramic veneer.

### **SUMMARY**

Purpose: To evaluate the survival rate, success rate, load to fracture, and finite element analysis (FEA) of maxillary central incisors and canines restored using ceramic veneers and varying preparation designs.

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Methods and Materials: Thirty human maxillary central incisors and 30 canines were allocated to the following four groups (n=15) based on the preparation design and type of tooth: Gr1 = central incisor with a conservative preparation; Gr2 = central incisor with a conventional preparation with palatal chamfer; Gr3 = canine with a conservative preparation; Gr4 = canine with a conventional preparation with palatal chamfer. Ceramic veneers (lithium disilicate) were fabricated and adhesively cemented (Variolink Veneer). The specimens were subjected to  $4 \times 10^6$ mechanical cycles and evaluated at every 500,000 cycles to detect failures. Specimens that survived were subjected to a load to fracture test. Bidimensional models were modeled (Rhinoceros 4.0) and evaluated (MSC.Patrans 2005r2 and MSC.Marc 2005r2) on the basis of their maximum principal stress (MPS) values. Survival rate values were analyzed using the Kaplan-Meier test ( $\alpha = 0.05$ ) and load to fracture values were analyzed using the Student *t*-test ( $\alpha = 0.05$ ).

Results: All groups showed 100% survival rates. The Student *t*-test did not show any difference between the groups for load to fracture. FEA showed higher MPS values in the specimens restored using veneers with conventional preparation design with palatal chamfer.

Conclusion: Preparation design did not affect the fracture load of canines and central incisors, but the veneers with conventional preparation design with palatal chamfer exhibited a tendency to generate higher MPS values.

### INTRODUCTION

Solving esthetic problems in the anterior teeth is a challenge for restorative dentistry. For many years, full coverage restorations were the most highly preferred option for treating esthetic defects. However, such restorations are invasive and cause great loss of tooth structure.

Preparations for ceramic veneers conform to the concept of conservative dentistry with minimally invasive procedures. This has contributed to the widespread use of this technique.<sup>2</sup> In addition, this technique has been used successfully to correct such defects as stains, small fractures, and diastemas and to improve a patient's esthetic condition.<sup>2-5</sup>

Laboratory studies have shown similar load to fracture values between specimens restored using ceramic veneers and varying preparation designs and between healthy teeth or teeth restored using full crowns.<sup>6,9</sup> Besides, researchers have shown survival rates greater than 90% in the first 5 years of clinical function for teeth restored using ceramic veneers, <sup>10-15</sup> concluding that ceramic veneers are a good alternative for restoring anterior teeth.

Clinical studies present the strongest evidence for a material or a restorative technique. However, such studies are expensive and not easy to conduct. Therefore, laboratory studies are important tools for obtaining data because they permit comparison of different materials under standardized conditions. The aging protocols of mechanical cycling subject the specimens to load, temperature, and humidity conditions similar to those experienced in the oral environment. In addition, this test is able to induce crack propagation to the point of catastrophic failure, as observed in clinical function. The such standard stand

Nondestructive tests such as finite element analysis (FEA), which are associated with *in vitro* tests,

permit a better understanding of the different phenomena that occur in the tested specimens. <sup>19,20</sup> FEA offers a better overview of the stress distribution at an interface or inside a material, thus permitting an accurate check of the system's behavior. <sup>21</sup>

Although clinical studies have reported good survival rates for teeth restored using ceramic veneers, there have been no studies determining which preparation design is best suited for canines and central incisors specifically. In addition, there are no studies elucidating the principal cause of ceramic veneer failures by aging the specimens through mechanical cycling or evaluating the behavior of different teeth restored using laminate veneers on the basis of survival rates.

Hence, this study aims to evaluate the survival rate and mechanical behavior of canines and maxillary central incisors restored using ceramic veneers and varying preparation designs, by using mechanical cyclic aging, load to fracture test, and FEA. The following hypotheses were tested: (1) canines restored using ceramic veneers and varying preparation designs will show the same values of load to fracture and survival rate; (2) maxillary central incisors restored using ceramic veneers and varying preparation designs will show the same values of load to fracture and survival rate; and (3) tensile stress distribution will be similar in all the models, independent of the preparation design.

### **METHODS AND MATERIALS**

The product names, manufacturers, chemical compositions, and batch numbers of the materials used in the study are listed in Table 1.

# Selection, Embedding, and Standardization of Specimens

Sixty human teeth (30 canines and 30 maxillary central incisors) were selected for the study. Teeth were analyzed at  $4\times$  magnification using the following selection criteria: no caries or previous restorations, no cracks, and presence of completely formed apexes. After the selection process, teeth were cleaned, disinfected, and stored in distilled water (4°C) until use.

To simulate the periodontal ligament, the labiolingual and mesiodistal dimensions were recorded at three different points on the root of each tooth. Utility wax was then liquefied at a temperature of 70°C and applied on the root with a paintbrush up to 3 mm below the cementoenamel junction. New

Material	Manufacturer	Chemical Composition	Batch No.	
IPS e.max Press	Ivoclar Vivadent, Schann, Liechtenstein	Principal component: SiO <sub>2</sub> ; additional components: Li <sub>2</sub> O, K <sub>2</sub> O, MgO, ZnO, Al <sub>2</sub> O <sub>5</sub> .	P87414	
Total Etch	Ivoclar Vivadent, Schann, Liechtenstein	37% phosphoric acid	R22282	
Porcelain Conditioner	Dentstply, Petrópolis, RJ, Brazil	10% hydrofluoric acid	229431B	
Excite F	Ivoclar Vivadent, Schann, Liechtenstein	Contains phosphoric acid acrylate, 2- hydroxyethyl methacrylate, dimethacrylate, highly dispersed silicone dioxide, initiators, stabilizers, and potassium fluoride in an alcohol solution	R23729	
Variolink Veneer	Ivoclar Vivadent, Schann, Liechtenstein	Is composed of dimethacrylates, silicon dioxide; and ytterbium trifluoride; additional content: catalysts, stabilizers, and pigments; total content of inorganic filler is 40 vol%; filler particle sizes range from 40 nm to 300 nm	P71541	
Monobond Plus	Ivoclar Vivadent, Schaan, Liechtenstein	Alcohol solution of silane methacrylate, phosphoric acid methacrylate, and sulphide methacrylate	R26662	

measurements of the root dimensions, at the same points as those previously measured, were taken until a homogeneous wax thickness of 0.3 mm was obtained.<sup>22</sup>

The specimens were then embedded, up to the same level as the wax, in metal matrices containing autopolymerizing acrylic resin. After the acrylic resin had cured, the specimens were removed from the matrices; the wax was detached from the root surface and removed from the space created in the acrylic resin. Later, an elastomeric material (Impregum, 3M ESPE, St Paul, MN, USA) was manipulated and inserted into this space. The specimen was then repositioned and the excess polyether was removed using a scalpel.

# Randomization and Preparation of the Specimens

After the specimens had been standardized, they were randomly allocated to the following four groups based on preparation design and type of tooth: Gr1 = maxillary central incisor with a conservative preparation; Gr2 = maxillary central incisor with a conventional preparation with palatal chamfer; Gr3 = canine with a conservative preparation; and Gr4 = canine with a conventional preparation with palatal chamfer. Randomization was performed by numbering the canines and the incisors from 1 to 30 and generating two sequences of 30 numbers by using a randomization program (Random Allocation, developed by M. Saghaei, Department of Anesthesia, University of Medical Sciences of Isfahan, Isfahan,

Iran) to obtain homogeneous groups, thus reducing the possibility of bias in the future results.

Before starting the tooth preparation, a silicone mold of each specimen was obtained to control the veneer preparation thickness. Teeth were prepared by a single trained operator. The conservative preparation involved reducing the facial surface by 1 mm. The conventional preparation with palatal chamfer involved reducing the facial surface by 1 mm and the incisal edge by 2 mm; the chamfer's height and width were 1 mm each. Preparations were extended to the cementoenamel junction and the margins were confined to the enamel. Preparations were executed with a regular rotatory diamond instrument (4137F, KG Sorensen, Cotia, Brazil) and finishing procedures were executed with a fine-grain diamond rotatory instrument (4138FF, KG Sorensen). Each diamond instrument was discarded after preparing three specimens.

# Modeling Procedures and Obtaining of Ceramic Veneers

Specimens were molded using an elastomeric material (Express XT, 3M ESPE), and the master casts were obtained in a Type IV dental stone (Elite Rock, Zhermack, Badia Polesine, Italy). On each master cast, the laminate veneer was built up with vegetal wax (GEO, Renfert, Hilzingen, Germany) by using the mold obtained before the tooth preparation. Thereafter, ceramic restorations were fabricated using lithium disilicate ceramic ingots (IPS e.max Press, Ivoclar Vivadent, Schaan, Liechten-

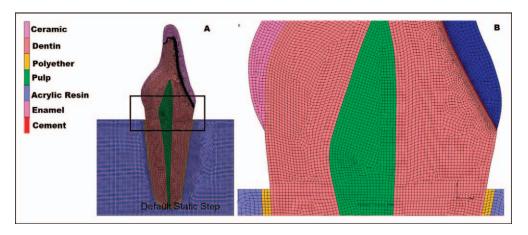


Figure 1. (A): Mesh obtained (canine with palatal chamfer). (B): Closer view showing the adequate relation between the elements.

stein) in accordance with the manufacturer's instructions.

### **Cementation Procedures**

The enamel and dentin were conditioned with 37% phosphoric acid (Total Etch, Ivoclar Vivadent) for 20 seconds and 15 seconds, respectively. Surfaces were washed with distilled water and dried with absorbent papers. The total-etch adhesive system (ExciTE F, Ivoclar Vivadent) was applied to the surfaces for 20 seconds, air-sprayed, and then photoactivated (Radii Cal, SDI, Bayswater, Australia) for 20 seconds.

The ceramic surfaces were etched with 10% hydrofluoric acid (Porcelain Conditioner, Dentsply, Petropolis, Brazil) for 20 seconds; a silane coupling agent (Monobond Plus, Ivoclar Vivadent) was applied and allowed to react for 3 minutes. A resin cement (Variolink Veneer, Ivoclar Vivadent) was then applied on the internal surface of each laminate veneer. The restorations were cemented onto the corresponding teeth, excess resin cement was removed, and photoactivation (Radii Cal, SDI) was performed for 20 seconds on each face. All cementation procedures were performed by a single trained operator.

# **Mechanical Cyclic Aging and Periodic Evaluation**

After the cementation procedures were completed, specimens were positioned at a 45° inclination and immersed in water at 37°C in a fatigue simulator (Erios ER 11000, Erios, São Paulo, Brazil). A stainless steel piston with a flat surface was positioned on the incisal portion of each specimen;  $4\times10^6\,\mathrm{cycles}$  were induced with a load of 100 N, at a

frequency of 4 Hz. Between the piston and the specimen, a polyester matrix strip was positioned.

After every 500,000 cycles, the specimens were analyzed in a stereomicroscope (Discovery V-20, Zeiss, Göttingen, Germany) for the following outcomes: ceramic veneer irreparable fracture, ceramic veneer decementation, and ceramic veneer cracks. If an event was noted, the specimen received a score that was then used to calculate the survival rate.

### **Load to Fracture Evaluation**

The specimens that did not fail during mechanical cycling were positioned in a universal testing machine (EMIC DL 2000, EMIC, São José dos Pinhais, Brazil) at a 45° inclination. A stainless steel piston with a flat surface was used to induce a load until the specimens fractured.

### **Failure Mode Evaluation**

All fractured specimens were analyzed using a stereomicroscope (Discovery V-20, Zeiss), and the type of failure was classified based on the following criteria: type I = veneer decementation; type II = ceramic veneer fracture without fracture of coronal structure; type III = coronal dental fracture; Type IV = reparable root fracture above the simulated periodontal ligament; and type V = catastrophic root fracture below the simulated periodontal ligament.

### **Finite Element Analysis**

Bidimensional models were obtained via computerassisted design software (Rhinoceros 4.0). The dental structures were modeled using values from the literature, <sup>23,24</sup> and other structures (periodontal ligament, acrylic resin, resin cement, laminate

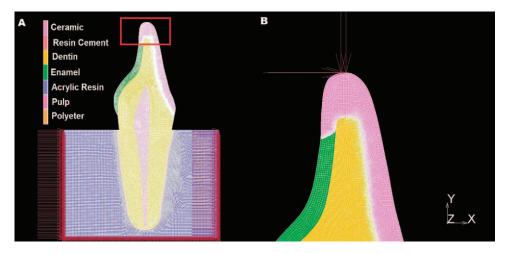


Figure 2. (A): External elements of acrylic resin were fixed at the x, y, and z axes. (B): Force applied on the elements at the incisal portion.

veneer) were modeled by replicating the laboratory characteristics and dimensions.

The models were extracted to a preprocessing software (MSC.Patran 2005 r2) and the mesh conditions (Figure 1) were generated by quadrilateral elements (QUAD 4) in the plane strain condition. The number of elements varied for each model as follows: 29,769 elements for canine with conventional preparation with palatal chamfer; 30,723 elements for canine with conservative preparation; 30,456 elements for central incisor with conventional preparation with palatal chamfer; and 29,327 elements for central incisor with conservative preparation.

The external elements of the acrylic resin had their movement restricted in all axes (x, y, and z) (Figure 2A), and a load of 100 N was applied on the incisal region at a 45° inclination (Figure 2B). The materials were considered isotropic, homogeneous, and linear, and their properties were provided in accordance to the literature (Table 2). Finally, the models were analyzed using a postprocessing software (MSC.Marc 2005 r2) to verify the distribution and direction of maximum principal stress (Max  $\sigma_1$ ) in the ceramic veneer restoration and on the dentin surface.

### **Statistical Analysis**

Initially, the sample size was calculated. Based on a standard deviation of 150 N, with differences of 200 N between groups, conferring a significance level of 5% and a power sample of 95%, the sample size estimated was 12 specimens per group. However, as the power sample would be almost 99% with a

sample size of 15 specimens, we selected 15 specimens per group.

Survival rates were evaluated by Kaplan-Meier and log-rank tests ( $\alpha = 0.05$ ). The load to fracture values were evaluated using the Student *t*-test ( $\alpha = 0.05$ ). One Student *t*-test was performed to compare values between canines, and another Student *t*-test was performed to compare values between central incisors. Statistix 8 (Analytical Software, Tallahassee, FL, USA) software was used for data analysis.

### **RESULTS**

The restorations did not fail after mechanical cycling, generating Kaplan-Meier survival rates of 100% for all the groups and no statistically significant differences between them. Student *t*-tests did not show any influence of preparation design on the load to fracture values (Table 3). The failure mode analysis (Table 4) showed the highest number of catastrophic root fractures (below periodontal liga-

Table 2: Materials, Elastic Modulus, and Poisson Coefficient						
Material	Elastic Modulus Poi (GPa)					
Dentin <sup>25</sup>	18.6	0.31				
Resin cement <sup>26</sup>	2.6	0.33				
Acrylic resin <sup>27</sup>	2.7	0.35				
Enamel <sup>28</sup>	46.8	0.3				
Lithium disilicate based ceramic (IPS.emax Press) <sup>a</sup>	95	0.3				
Dental pulp <sup>29</sup>	0.003	0.45				
Polyether <sup>30</sup>	0.05	0.45				
<sup>a</sup> Manufacturer's information (Ivoclar	Vivadent, Schaan, Lietch	teinstein).				

Table 3: Mean, Standard Deviations (Newtons), and Statistical Values After Student t-Test ( $\alpha$ =0.05) for the Fracture Load Values<sup>a</sup>

Preparation		Teeth
	Canine	Central Incisor
Conservative	705.6 (131.8) <sup>B</sup>	519.3 (171.2) <sup>b</sup>
	( <i>p</i> =0.193)	( <i>p</i> =0.166)
Palatal chamfer	638.8 (141.9) <sup>B</sup>	441.9 (123.4) <sup>b</sup>

<sup>&</sup>lt;sup>a</sup> Identical letters represent similar statistical results (comparisons between columns).

ment) (Figure 3), followed by coronal dental fractures.

By FEA, groups subjected to the conventional preparation with palatal chamfer showed higher tensile stress values in ceramic restorations (Figure 4) compared with the other groups, when using the restorative material (lithium disilicate ceramic). All models showed tensile stress values in the palatal cervical region of the root; models subjected to conventional preparation with palatal chamfer showed slightly higher values than the other groups (Figure 5).

The directions of the tensile stress vectors both in the ceramic veneer and in the root dentin are shown in Figures 6 and 7, respectively.

### **DISCUSSION**

Some methodologic aspects are important in guaranteeing the quality and reliability of a study. A reliable randomization process and an adequate sample size are examples of aspects that increase the authenticity of a study. <sup>31</sup> In this study, the use of a computer program to randomize the specimens reduced the possibility of bias interfering with the results. <sup>31</sup> Besides this, the power analysis performed was important to avoid any misinterpretation of the results, by rightly rejecting the null hypothesis when it is indeed false. <sup>32</sup>

After mechanical cyclic aging and periodic evaluations, the Kaplan-Meier test did not show statistically significant differences between the groups, thereby confirming the first and second hypotheses of this study. The survival rate values observed in this study (100%) differ from the values observed by Stappert and others. However, Stappert and others cycled their specimens by using an ellipsoidal curve pattern with horizontal and vertical components that generated an impact on the palatal surface. This pattern of cycling could have induced too much stress on the specimens and therefore may explain the difference in the study results.

On the other hand, when we compared the survival rates of this study with those of clinical studies, we observed similarities between the values.  $^{10,12,14,15,33,34}$  Smales and Etemadi  $^{14}$  Guess and Stappert  $^{10}$  and D'arcangelo and others  $^{34}$  did not record any catastrophic failures in their specimens during the first 4 years of clinical service. The  $4\times10^6$  mechanical cycles performed in our study correspond to 4 years of clinical function,  $^{18}$  thus reinforcing the similarities between the studies.

The high survival rate values, observed both in this *in vitro* study and in clinical studies, could be related to the excellent adhesion achieved between both the resin cement-ceramic interface and the resin cement-dental substrate interface in this type of restoration. For ceramic, the surface treatment protocol is well established in the literature, showing high and stable bond-strength values. <sup>35-38</sup> As for the tooth substrate, the adhesive procedure performed during the ceramic veneer cementation is basically on the enamel. This guarantees a good pattern of union and is mentioned by some authors as the reason for the success of this restorative technique. <sup>12,14,33,34,39,40</sup>

Another factor that could have influenced the survival rates in this study is the methodology used to detect cracks in the restoration. Evaluation under

Table 4: Amount and Percentage of Failure Mode for Each Group						
Preparation Groups			Mode of Failure	1		
	Type I	Type II	Type III	Type IV	Type V	
Canine with conservative preparation	0	0	4 (27%)	1 (6%)	10 (67%)	
Canine with palatal chamfer	1 (6%)	1 (6%)	0	5 (34%)	8 (54%)	
Central incisor with conservative preparation	0	0	3 (11%)	1 (6%)	11 (73%)	
Central incisor with palatal chamfer	1 (6%)	1 (6%)	2 (13%)	3 (21%)	8 (54%)	
Total	2 (3%)	2 (3%)	9 (15%)	10(17%)	37 (62%)	

<sup>&</sup>lt;sup>a</sup>Type I, veneer decementation; type II, ceramic veneer fracture without fracture of coronal structure; type III, coronal dental fracture; type IV, reparable root fracture above the simulated periodontal ligament; type V, catastrophic root fracture below the simulated periodontal ligament.

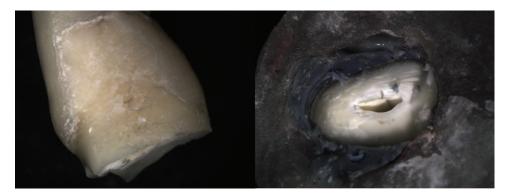


Figure 3. Representative image of an irreparable root fracture.

a stereomicroscope, without the use of a dye solution may not be capable of detecting cracks.

In relation to the load to fracture values, the Student t-tests did not show any statistically significant difference between the preparation designs, independent of the type of tooth restored, thereby confirming the first and second hypotheses of the study and corroborating the findings of some studies.  $^{6,41,42}$  On the other hand, the findings of other studies did not agree with our results.  $^{7,9,43,44}$  The fact that these studies used different design parameters, such as a different region of applied load during the load to fracture test, no aging of the specimens, use of different ceramic materials for tooth restoration, and difference in the thickness of ceramic restorations, could have contributed to the absence of similarities between the results.

The failure mode analysis showed a predominance of root fractures (Table 4). These data are in accordance with findings of other laboratory studies that used the incisal edge to apply the load during the load to fracture test. <sup>6,7,41,42</sup> However, this failure mode was not observed in clinical studies, which show a predominance of ceramic veneer fractures and decementation failures. <sup>10,12,14,15,33,34</sup> This could be a limitation of the load to fracture test. Hence, reproducing failures similar to those seen in clinical function could be an area of improvement for future studies.

We conducted an FEA using a two-dimensional (2D) model instead of a three-dimensional model. Although three-dimensional models can better reproduce the clinical characteristics, in some cases, this model can hinder the obtainment of a fine mesh, especially in thin regions (resin cement and ceramic finish lines). Moreover, there are studies validating the use of 2D models to evaluate restored teeth. Magne and Douglas stated that the mechanical events that occur in teeth restored using

laminated veneers are in the vestibulolingual plane, thereby supporting the use of a 2D plane strain model for evaluating the scenario.

In the present study, FEA showed a difference in the tensile stress distribution between the models, thereby rejecting the third hypothesis of this study. Independent of the type of tooth, FEA showed that specimens prepared with the conventional design with palatal chamfer generated higher tensile stress values in ceramic restorations compared with teeth

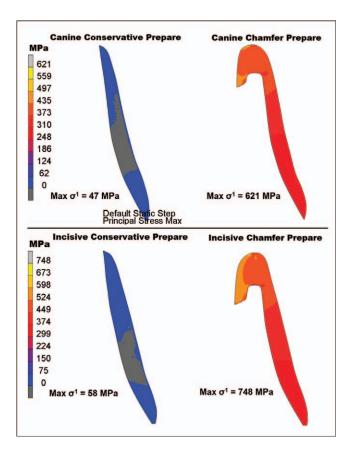


Figure 4. Maximum principal stress distribution in the ceramic veneer.

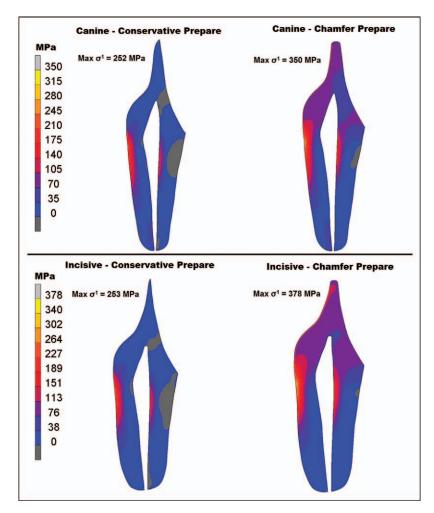


Figure 5. Maximum principal stress distribution in the root dentin.

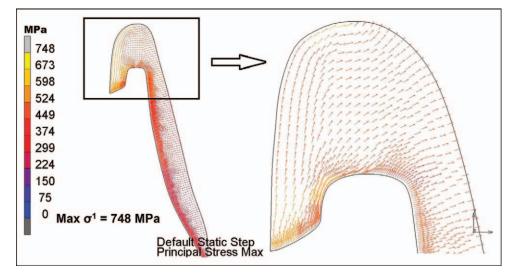


Figure 6. Values and direction of the maximum principal stress vectors in the ceramic veneer.

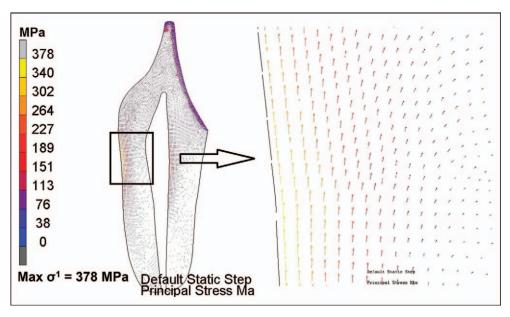


Figure 7. Values and direction of the maximum principal stress vectors in the root dentin.

subjected to the conservative preparation (Figure 4). This result is in accordance with that of the Magne and Douglas<sup>45</sup> study but not the study performed by Zarone and others (2005).<sup>49</sup> The difference observed between the present study and Zarone's study could be related to the fact that Zarone used Von Mises stress for analysis, fixed the movement of the elements located on the external surface of the root, and simulated a laminate veneer with a 0.5 mm thickness.

The differences in the tensile stress distribution of the models did not influence the mode of failure between the groups. However, it is possible to suppose that teeth prepared with the conventional design with palatal chamfer present a higher possibility of suffering a restoration fracture. This is corroborated by the direction of the tensile stress vectors seen in the veneer (Figure 6). Although no relation to any mode of failure could be established in the present study, the tensile stress distribution and tensile stress vector directions were in agreement with the mode of failure observed in clinical studies. <sup>10,11,34,50</sup>

The FEA also showed a tensile stress concentration in the root dentin (Figure 5). This distribution, in addition to the tensile stress vector direction in the root dentin, can explain the pattern (Figure 3) and incidence (Table 4) of root fractures observed in this study. The incidence of root fractures could be related to the mechanical properties of the ceramic material used to fabricate the restorations (lithium

disilicate, IPS e.max Press, Ivoclar Vivadent) and to the quality of adhesion achieved between the interfaces, turning the lithium disilicate into an interesting material for ceramic veneer restoration. It is possible that if a fragile material (a feldspathic ceramic) had been used, the mode of failure and the results of this research might have been different.

### CONCLUSION

Based on the results we conclude that (1) conservative preparations and conventional preparations with palatal chamfer, whether performed on canines or maxillary central incisors, generate similar load to fracture and survival rate values; (2) conventional preparation with palatal chamfer generates higher maximum principal stress concentration in the laminate veneer compared with conservative preparation.

### **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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# Preliminary Results of the Survival and Fracture Load of Roots Restored With Intracanal Posts: Weakened vs Nonweakened Roots

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### Clinical Relevance

When restoring roots with an adequate quantity of dental tissue, a cast post and core or fiber post may be used. But when weakened roots are being restored, fiber posts with a wider cervical emergence diameter appear to be the best clinical solution, even though the clinical prognosis is doubtful.

### **SUMMARY**

Purpose: To evaluate the fracture load and survival rate of weakened and non-weakened roots restored with different intracanal posts.

Methods: Eighty teeth (16 mm) were prepared to a length of 10 mm with a custom drill. Fifty roots were weakened with a tapered diamond

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drill, and 30 roots were not. The specimens were embedded with acrylic resin up to 3 mm from the coronal aspect, and the periodontal ligament was simulated. The 50 weakened roots were restored with (n=10) CPC-gold (cast post and core made of gold alloy), CPC-Ni (cast post and core made of Ni-Cr alloy), FP (glass fiber posts), FP-W (glass fiber posts with a wider coronal diameter), and FP-CR (fiber posts relined with composite resin). The 30 nonweakened roots were restored with (n=10) CPC-gold, CPC-Ni, and FP. All of the posts were adhesively cemented. All of the specimens were mechanically cycled (37°C, 45°, 130 N, 2.2 Hz, and 1.5 million pulses) and evaluated after every  $5 \times 10^4$  cycles to evaluate the presence of cracks as a primary outcome (event). The specimens that survived cycling were subjected to a fracture load test (load application on the palatal aspect at a 45° inclination). Failure mode was classified as favorable (above the simulated bone level) and catastrophic (below the simulated bone level). Survival rates were estimated using the Kaplan-Meier method. Fracture load data were

analyzed using the Kruskal-Wallis test ( $\alpha$ =0.05) for weakened roots, one-way analysis of variance, and Tukey test (p<0.05) for non-weakened roots, and Student t-test (p<0.05) compared nonweakened vs weakened roots for the same post system.

Results: For the preliminary survival results, FP-W showed a higher survival rate when compared with CPC (gold/Ni). For the fracture load (N), the statistical analysis (p<0.0001) presented differences among the weakened groups: CPC-gold (541.4) = CPC-Ni (642.6) > FP (282.2) = FP-W (274.1) = FP-RC (216.6). No differences were observed for the groups that were nonweakened (majority of favorable failures): CPC-gold (459.3) = CPC-Ni (422.0) = FP (347.9). Weakened roots restored with CPC-gold promoted high values of load fracture and unfavorable failure rates.

Conclusion: Cast post and cores or fiber posts can be used for restoring nonweakened roots. However, for weakened roots, a fiber post with a wider cervical emerging diameter appears to be a better alternative when compared with cast post and cores.

### INTRODUCTION

Depending on the degree of destruction of coronary tooth structure, endodontically treated teeth often require anchorage to retain a restoration. Thus, the two primary functions of intracanal posts are 1) to provide retention for a coronal restoration, which replaces the lost crown structure, <sup>2,3</sup> and 2) to transmit minimal stress to the tooth structure, so as to prevent root fracture.

The ideal elastic modulus (E) for a retainer should be as similar as possible to that of dentin; however, different materials with different elastic moduli can be indicated as retainers (E dentin = 20 GPa, E noble metal alloy = 90 GPa, E titanium = 190 GPa, E glass fiber = 20-40 GPa, E composite = 5-25 GPa, E polyethylene fiber = 3.2 GPa). Studies involving finite element analysis and photoelastic analysis showed that cast post and cores or prefabricated rigid posts (stainless steel, titanium, and zirconia) transfer higher stresses to the surrounding dentin when compared with fiber retainers. Hus, it is believed that a rigid retainer can be associated with high rates of unfavorable failures in  $in\ vitro^{11-13}$  and  $in\ vivo\ studies.$ 

The restoration of endodontically treated teeth becomes more complicated if the root is excessively weakened, because of the absence of the crown or loss of intraradicular dentin.  $^{15,16}$  In these cases, teeth are unable to support high levels of forces during mastication  $^{17-19}$  and have a higher risk of catastrophic fractures.  $^{20}$ 

Several materials have been associated with fiber posts to restore weakened roots, including fiber ribbons, accessory posts, <sup>21</sup> and composite resins. <sup>22</sup> Those materials have achieved favorable results for both bond strength and load fracture and usually present favorable failures in *in vitro* studies. <sup>16,23-26</sup>

The behavior of roots restored with retainers with different E may have different outcomes under cyclic loading; thus, mechanical cycling has been used to evaluate possible outcomes, taking into account different scenarios (roots or teeth restored with different materials and E).27-29 Even with the limitations of in vitro studies, mechanical fatigue tests are carried out in a humid environment as a method that best approximates the clinical behavior of different materials and restorative techniques, reproducing, for example, the effects of restorative materials with different E on the whole toothrestoration assembly. Fatigue is a type of test that may lead to fracture of a structure after repeated loads that can be explained due to microscopic crack propagation from areas of stress concentration, typically in regions of microscopic or molecular structural defects. 30-33

Furthermore, survival analysis can be used to estimate survival and to interpret the function of an event; this function can also be used to compare different groups and to assess the relationship between explanatory variables and the frequency of the event of interest. Some clinical studies have used this technique to evaluate therapies for restoring endodontically treated teeth, taking into account such variables as the type of canals, number of remaining walls, and types of retainers.

In the case of *in vitro* studies, this analysis can be used to evaluate the probability of restored teeth surviving a period of evaluation under certain test conditions (eg, mechanical cycling parameters). Within this context, few studies have evaluated the *in vitro* survival rate of weakened roots restored with different intracanal retainers by the Kaplan-Meier method.

Thus, this *in vitro* study had the following objectives: 1) evaluate the fracture load of weakened and nonweakened roots restored with different intracanal retainers after mechanical cycling and 2) evaluate the main method of survival of the

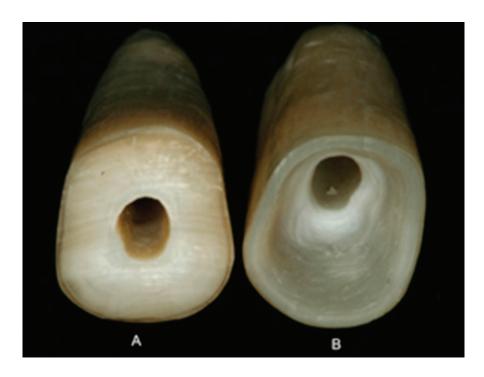


Figure 1. Images of nonweakened roots (A) and weakened roots (B).

specimens, using the Kaplan-Meier test, after 1.5 million cycles.

Thus, the scientific hypotheses were the following:
1) nonweakened roots restored with fiber cores
would not present a difference in the fracture load
values when compared with cast post and cores; (2)
weakened roots restored with cast post and cores
would present lower values of fracture load when
compared with fiber posts; (3) nonweakened roots
would present superior load when compared with
weakened roots, independent of the restorative
approach; and (4) nonweakened roots would present
superior survival rates in relation to weakened roots.

### **METHODS AND MATERIALS**

### **Selection and Preparation of Specimens**

Bovine incisors were analyzed with a loupe (4× magnification, EyeMag Pro S, Carl Zeiss, Gottingen, Germany) to detect possible failures (fractures, fissures, or cracks) that might bias the results (exclusion criteria). After specimen selection (N=80), the coronal portions of each tooth were removed to standardize the length of the specimens at 16 mm. As another inclusion criteria, the mesiodistal and buccolingual dimensions of the cervical portion of the roots were measured with a caliper (Starrett 727, Starrett Indústria e Comercio Ltda, Itu, Brazil), and only teeth that exhibited values between 4 and 6 mm were included in the study to standardize the size of the roots.

Afterward, the specimens were divided into eight (n=10) groups according to the restorative approach and the presence or lack of canal weakening. Randomization was accomplished by numbering the specimens from 1 to 80, and eight random sequences of 10 numbers were generated by the computer program Random Allocator (developed by M. Saghaei, Department of Anesthesia, Isfahan University of Medical Sciences, Isfahan, Iran).

All specimens were initially prepared at a length of 10 mm with the No. 2 drill of the fiber post system White Post (White Post DC, FGM, Joinville, Brazil). In the weakened groups, the intracanal dentin was removed using a diamond-tapered drill (4137, KG Sorensen, Cotia, Brazil). Weakening was performed in the most coronal portion of the root up to 6 mm deep, standardizing the remaining dentin thickness at 0.5 mm (Figure 1B) to simulate the most hostile scenario of endodontically treated teeth.

To simulate the periodontal ligament, all roots from the apex to 3 mm apical from the coronal surface were coated with a 0.3-mm-thick layer of wax (Lysanda, São Paulo, Brazil) that was liquefied in a container at a standardized temperature of 70°C (to check the thickness of wax, we used a digital caliper to measure the distance mesiodistal and buccolingual before and after immersion of the root in the wax).

Subsequently, the drill of the post system was inserted into the canal and connected to a surveyor

(B2, BioArt, São Carlos, Brazil), keeping the whole tooth and drill parallel to the y-axis. Acrylic resin (VIPI Flash, VIPI, Pirassununga, Brazil) was placed in a PVC cylinder (h: 20 mm, Ø: 12 mm), and each root was embedded in resin up to 3 mm apical from the most coronal portion.

After polymerization of the resin, the roots were separated from the resin and the wax was removed. Then, an elastomeric material (Impregum F, 3M-Espe, Seefeld, Germany) was inserted into the "false socket," the root was repositioned in the PVC, and the excess elastomeric material was removed with a scalpel blade. 44

# Production and Cementation of Intracanal Posts

Gold and Ni-Cr Alloy Cast Post and Core (CPC-Gold/CPC-Ni)—Conventional procedures were performed for the cast post and core (CPC) production: patterns of the intracanal post space and core were made with acrylic resin (Duralay, Reliance Dental Mfg Co, Worth, IL) followed by casting, as recommended by the manufacturer (gold alloy, La Croix 2, La Croix Dental Alloys, Rio de Janeiro, Brazil; Ni-Cr alloy, Wironia Light, Bego, Bremen, Germany). The cores had their dimensions standardized by using identical plastic matrices. 45,46

The CPCs were evaluated for adaptability and cemented as follows: 1) posts were cleaned with isopropyl alcohol and the post root portion was airabraded with aluminum oxide particles (110 µm, pressure: 2.8 bars, 10 mm distance, and 15 seconds); 2) root dentin was conditioned with 37% phosphoric acid (Condac 37, FGM) for 15 seconds, washed with water for 15 seconds, and the excess water was removed with No. 80 paper points (Dentsply, Petrópolis, Brazil); 3) a photo-cured adhesive agent (Ambar, FGM) was applied with a microbrush, and the excess was removed with paper cones and photo activated for 20 seconds with a high-power LED (1200 mW/cm<sup>2</sup>, RadiiCal, SDI, Australia); and 4) equal amounts of the dual-cured resin cement pastes (AllCem, FGM) were mixed and inserted into the canals using a No. 40 Lentulo spiral (Dentsply Maillefer, Catanduva, Brazil), followed by insertion of the post into the root canal, excess cement was removed with a microbrush, and the remaining resin was photo activated for 10 seconds in the incisal surface and another 10 seconds at each face. The specimens were stored in distilled water at 37°C for 24 hours prior to the metal crown production.

Conventional Prefabricated Fiber Posts (FP)—The No. 2 fiber posts (White Post DC No. 2) were

positioned in the canal, and the coronal part was cut, preserving a 5-mm height from the coronal portion. The fiber posts were cleaned with isopropyl alcohol, and the surface received an MPS-based silane coupling agent (Prosil, FGM). Cementation was carried out as mentioned previously.

Afterward, the core construction was performed with a composite resin (Oppalis, FGM). First, a layer of resin was applied on the post and coronal dentin and photo activated for 30 seconds. Then, resin was inserted inside standard plastic templates (as used for CPCs), which was positioned on the post and dentin, followed by photo activation (20 seconds) on each face, and the matrices were removed. The specimens were stored in distilled water (37°C) for 24 hours prior to the metal crown production.

Wider Prefabricated Fiber Posts (FP-W)—The dowels used for this group were the No. 2 Double Cone Special (White Post DC No. 2E). The preparation of the specimens was similar to the FP groups. These posts are considered "special" because of their wider cervical diameter, which is recommended for roots with enlarged root canals. The post cementation was carried out as previously mentioned.

Fiber Posts Relined With Composite Resin (FP-CR)—Fiber posts (White Post DC No. 2) were used for this group.

The following procedures were performed: 1) the fiber posts were cleaned with isopropyl alcohol and silanized as mentioned previously; 2) the walls of the intracanal dentin were isolated by applying hydrosoluble insulation (K-Y gel, Johnson & Johnson, São José dos Campos, Brazil) to avoid composite resin bonding; 3) the composite resin was condensed inside the canal, the post was positioned, and the resin was light cured for 20 seconds from the occlusal surface; 4) the assembly was removed from the canal, photo activated for 40 seconds, and reinserted to verify adaptation; 5) the post/composite resin assembly was conditioned with 37% phosphoric acid for 60 seconds, washed with distilled water for 15 seconds, and dried with absorbent paper: 6) a thin layer of adhesive was applied and photo activated for 40 seconds; and 7) the adhesive was applied on the intraradicular dentin, and the cementation procedures were performed as mentioned previously for other groups restored with fiber posts.

### **Preparation and Cementation of Metal Crowns**

Eighty metal crowns were fabricated according to the anatomy of a maxillary canine, as follows: 1) each prosthetic preparation was molded with a vinylpolysiloxane material (addition silicone, Elite Double 8, Zhermack, Rovigo, Italy), and a master die was fabricated (Durone, Dentsply); 2) each crown was waxed on the die (Newwax, Technew, Campo Grande, Brazil) using standardized maxillary canine plastic templates; and 3) casting was accomplished using the lost-wax technique as recommended by the manufacturer (nonnoble alloy, Wironia Light).

The crowns were examined for adaptation, and the inner surfaces were air-abraded with aluminum oxide (110  $\mu$ m; Blue Equipment, São José do Rio Preto, Brazil) and adhesively cemented, using the same adhesive system and resin cement described previously. A load of 5 kg was applied on the crown by means of a static press during cementation, and the excess cement was removed.

### **Mechanical Cycling**

For mechanical cycling, the specimens were subjected to the following protocol:  $45^{\circ}$  angle to the long axis of the root, water immersion ( $\pm 37^{\circ}$ C), pulse load of 0 N to 130 N, frequency of 2.2 Hz, and 1,500,000 pulses on the crown at a point located 2 mm below the incisal edge on the lingual aspect of the specimen.

According to Wiskott and others,<sup>47</sup> 1 million cycles are equivalent to one year of clinical service; therefore, the present study simulated approximately 1.5 years of clinical service.

For the survival analysis, the roots were evaluated for the presence of cracks. After each set of 50,000 cycles, all roots were evaluated by a calibrated operator. The specimens were monitored to check the time and the approximate size of the failure (if it occurred). The evaluation of the failure was performed with a loupe (4× magnification, EyeMag Pro S, Carl Zeiss). The final mode of failure was observed with a stereomicroscope (Discovery V20, Carl Zeiss). The specimens that survived mechanical cycling were submitted to a monotonic fracture load and subsequent failure analysis.

### **Fracture Load Test**

Each specimen was positioned on a device that was aligned at an angle of 45° to the long axis of the tooth. A universal testing machine (DL-1000, Emic, Sao Jose dos Pinhais, Brazil) was used to apply a constant load at a crosshead speed of 1 mm/min until failure. The threshold of failure was defined as the point at which the force (N) reached a maximum value at root fracture, bending of the post, or detachment of the core-post assembly.

### **Failure Analysis**

The failures that occurred during the tests were classified as favorable (above the 3 mm corresponding to the simulated bone level) and unfavorable (below the simulated bone level). For failure analysis, the roots were stained with pens (blue marker, Faber-Castell, São Carlos, Brazil), and the ink was partially removed using a cotton ball and 70% alcohol to visualize the fractures. The procedure was performed before and after the fracture load test.

### **Data Analysis**

Considering that every final failure comes from an initial failure, events such as root fracture were measured as a "crack" event. Events of marginal misfit, when not associated with root cracks, were not considered an outcome.

Survival rates were estimated with the Kaplan-Meier technique, using STATA 12.0 (Stata Corporation, College Station, TX, USA). The differences between the survival rates according to the study groups were analyzed using the log-rank test  $(p \le 0.05)$ .

For fracture load data, the Shapiro-Wilk test did not show a normal distribution for the weakened roots' data; consequently, a nonparametric test (Kruskal-Wallis,  $\alpha$ =0.05) was used to analyze it. In contrast, data from other conditions had normal distribution: nonweakened roots were compared among themselves (one-way analysis of variance ANOVA and Tukey tests, p<0.05).

The Student *t*-test (p<0.05) compared nonweakened vs weakened roots for the same post system.

### **RESULTS**

### Mechanical Cycling: Descriptive Analysis and Survival Preliminary Results (Kaplan-Meier Method)

Among the weakened roots, five specimens fractured after 1.5 million cycles: two failures occurred in the FP group (50,000 and 100,000 cycles, unfavorable and favorable failures, respectively), one failure in the CPC-gold group (700,000 cycles, unfavorable), and two failures in the FP-CR group (150,000 and 1,150,000 cycles, favorable and unfavorable failures, respectively). For the nonweakened groups, two specimens fractured in the FP group (50,000 and 110,000 cycles, both favorable failures). The mechanical failures during cycling are presented in Table 1.

Table 1: Qualitative assessment of the failures after fracture load and during mechanical cycling for the outcome fracture

			STUDY GROUPS											
			-	Non-weak	banaz					akened			I	
			CPC- gold (n=10)	CPC- Ni (n=10)	FP (n=	8)	CPC- gold (n=9)	CPC- Ni (n=10)	FP (ı		FP-W (n=10)	FP- (n:		TOTAL
	Pattern of	Favorable	4	7	6		0	0	C	)	1		1	19
	Failure	Unfavorable	6	3	2		9	10	8	;	9		7	54
TER		Mesial crack	7	7	7		10	9	9	)	7	(	5	62
AI	Failure	Buccal crack	3	1	4		4	2	4		4	3	3	25
FAILURES AFTER FRACTURE LOAD	place	Distal crack	5	5	11		8	8	6	)	9	(	5	58
		Lingual crack	1	0	1		2	1	C	)	0	(	)	5
		Fracture post	0	1	0		0	0	C	)	0	(	)	1
田田	Failure	Mesiodistal	5	6	9		8	7	5	i	6	4	1	50
	mode	buccolingual	0	0	0		0	0	C	)	0	(	)	0
	Amount and pattern of	Favorable (F)	-	-	2 failur (1 & 2)	es	-	-	1 fai (4		-	1 fa		4
G ING	failure	Unfavorable (U)	-	-	-		1failure	-	1 fai (5		=	1 fa	ilure	3
FAILURES DURING MECHANICAL CYCLING					failure fa	ilure 2	failure 3		failure 4	failure 5		failure 6	failure 7	
DI		Mesial crack	-	-	X	X	X	-	X	X	-	X	X	7
CA	Failure	Vestibular crack	-	-	-	-	-	-	-	-	-	-	-	-
E E	place	Distal crack	-	-	X	X	X	-	X	X	-	X	X	7
A H		Lingual crack	-	-	-	-	-	-	-	-	-	-	-	-
EC		Fracture post	-	-	-	-	-	-	-		-	-	X	1
2	Failure	Mesiodistal	-	-	X	X	Х	-	Х	X	-	х	х	7
	mode	buccolingual	-	-	-	-	-	-	-	-	-	-	-	-

CPC-gold, cast post and core made of gold alloy; CPC-Ni, cast post and core made of Ni-Cr alloy; FP, glass fiber posts; FP-W, glass fiber posts with a wider coronal diameter; FP-CR, fiber posts relined with composite resin.

For the survival analysis, three approaches were performed (Table 2).

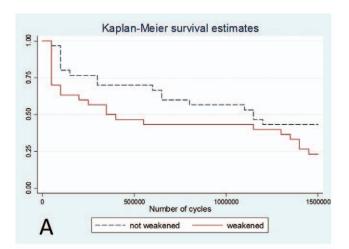
1) Comparison between groups according to root condition, weakened vs nonweakened (Figure 2A)—KM analysis showed no difference between the two conditions (log-rank test, p=0.09). However, the survival rate was 0.43 (SEM = 0.09) for nonweak-

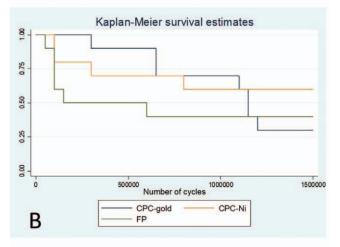
ened roots and 0.23 (SEM = 0.07) for weakened roots; thus, the probability that weakened roots exceed 1.5 million cycles without showing cracks (event/primary outcome) was 23%, while the probability for the nonweakened roots was 43%. The median survival for weakened roots was 350,000 cycles (EP = 205,396). For the nonweakened roots, the median was 1,150,000 cycles (EP = 273,252).

Table 2: Mean (±standard error) of the data survival rates and Log-rank test<sup>a</sup>

	Weakened r	oots canals
	Yes	No
Restoring strategies	Mean (SE)	Mean (SE)
CPC-Gold	750000 (200499.4) <sup>a, B</sup>	1070000 (123733.6) <sup>a, A</sup>
CPC-Ni	710000 (190368.1) <sup>a, B</sup>	1030000 (190814) a, A
FP	670000 (215893.5) a, AB	710000 (209141.1) <sup>a, A</sup>
FP-W	1085000 (201128.1) <sup>A</sup>	-
FP-CR	955000 (198172.9) <sup>AB</sup>	-

Different lowercase letters indicate differences between the types of weakened; Different capital letters indicate differences between restoration strategies.





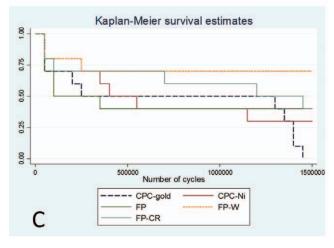


Figure 2. Survival curves estimated by using the Kaplan-Meier method for the occurrence of cracks according to condition of root (A), according to type of strategy (nonweakened condition of the root) (B), and according to strategy type (weakened roots) (C).

- 2) Comparison of restoring strategies for nonweak-ened roots (Figure 2B)—It is noted that the survival curves of the strategies were similar (log-rank test, p=0.54, CPC-gold = CPC-Ni = FP) and that they had similar survival rates estimated during the observation time.
- 3) Comparison of restoring strategies for weakened roots (Figure 2C)—It is noted that the only probabilities of survival of the specimens of the FP-W group had higher survival rates when compared with the specimens of both of the CPC groups (logrank test, p=0.004).

### **Fracture Load**

Table 3 presents the average values and standard deviation as well as statistical inferences.

For the groups with nonweakened roots, the one-way ANOVA showed that the restoring strategy was not significant (p=0.558; study groups were statistically similar).

For the groups with weakened roots, the Kruskal-Wallis test showed that the restorative strategy was significant (p=0.000; CPC-gold = CPC-Ni > FP > FP-W > FP-CR).

The Student *t*-tests (Table 3) indicated that weakened and nonweakened specimens did not differ statistically for each restoring strategy.

### **Failure Analysis**

The failures during mechanical cycling are shown in Table 1 and Figure 3A-F.

After the fracture load test, the nonweakened specimens had 39% unfavorable fractures (Table 1; Figure 4A,D), whereas the weakened specimens had 95% unfavorable fractures (Table 1; Figure 4B,C).

The location of failures was evaluated during mechanical cycling and after the fracture load tests. It is noted that the vast majority of failures were cracks in the mesial and distal surfaces of roots in different radicular thirds.

### **DISCUSSION**

The first research hypothesis of this current study was accepted, since no differences were noted between the three restoring strategies (CPC-gold, CPC-Ni, FP) when the roots were not weakened. These results are in agreement with those of Maccari and others, <sup>12</sup> Mitsui and others, <sup>3</sup> and Ni and others, <sup>48</sup> all of whom also observed no differences between metal, ceramic, and fiber posts. However, those studies contrast with those of Özcan and

Table O.	11/	dard deviation	·		(11)	J Tl. a!a	T+
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	Weakened r		
	Yes	No	
Restoring strategies	Mean (SD)	Mean (SD)	P value*
CPC-Gold	541.4 (227.4) <sup>a, A</sup>	459.3 (111.1) <sup>a, A</sup>	p = 0.135
CPC-Ni	642.6 (219.5) <sup>a, A</sup>	422.0 (151.9) <sup>a, A</sup>	p = 0.221
FP	282.2 (64.7) <sup>a, B</sup>	347.9 (91.8) <sup>a, A</sup>	p = 0.120
FP-W	274.1 (51.3) <sup>B</sup>	-	-
FP-CR	216.6 (63.6) <sup>B</sup>	-	-

Different lowercase letters indicate differences between the types of weakened (Kruskal-Wallis; a=0.05); Different capital letters indicate differences between restoration strategies; \* P-value from Student t test, comparing weakened vs non-weakening for each restoring strategy.

Valandro<sup>49</sup> and Kaur and others,<sup>13</sup> since cast post and cores and titanium posts had higher values of fracture load when compared with fiber posts in the latter studies.

The disagreement between the results of this current study with the findings of Özcan and Valandro<sup>49</sup> may be that the latter study did not use crowns to cover the cores. Without the protection of the crown, the core becomes more susceptible to failure and the load is transmitted directly to the post/core assembly.<sup>50</sup> According to Kaur and others, <sup>13</sup> the disagreement between the current results may be attributed to the greater removal of dentin

conducted by the researchers in that study during preparation of specimens restored with cast post and cores, contrasting with the more conservative preparation performed in the present study. While the specimens in the study by Kaur and others had enlarged canals, <sup>13</sup> the current study used only the drill of the post system in the preparation of roots restored with cast post and cores.

Finite element analysis studies have shown that greater volumes of retainers present greater stress concentration in the dental structure. <sup>4,6</sup> The greater volume of the retainer and higher stress concentration may have been responsible for generating a

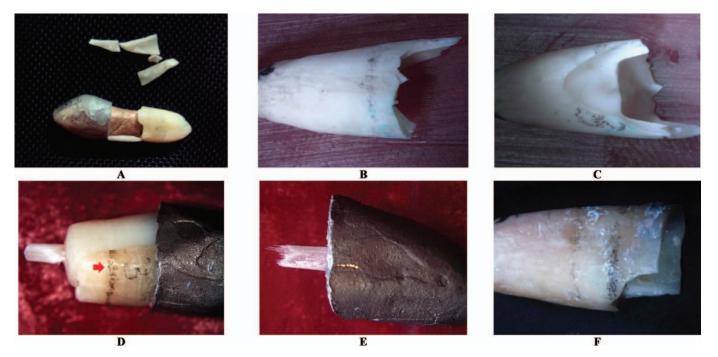


Figure 3. Representative images of failures occurring during mechanical cycling. (A) Unfavorable fracture in CPC-gold weakened group (failure 3 as typed by Table 1). (B) Unfavorable fracture in FP weakened group (failure 5 as typed by Table 1). (C) FP weakened from the labial view (failure 5 as typed by Table 1). (D) FP-CR: unfavorable fracture (arrow indicates the bone limit simulated; failure 7 as typed by Table 1). (E) FP-CR: fracture of post/core (failure 6 as typed by Table 1). (F) Fracture favorable in FP weakened group (failure 1 as typed by Table 1).

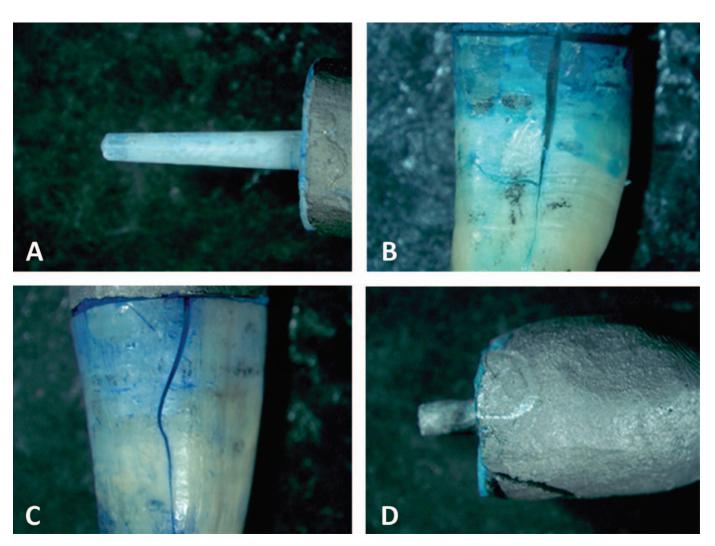


Figure 4. Representative images of failures occurred after the fracture load test. (A) FP nonweakened: displacement post/core/crown. (B) CPC-gold weakened: crack on the mesial surface. (C) FP weakened: crack on the distal surface. (D) CPC-Ni nonweakened: fracture of the cast and post core.

wedge effect and the unfavorable failures for Kaur and others,  $^{13}$  something also observed by other studies,  $^{6,50,51}$  a result that was not found in the present study, in which the tooth structure was preserved and a predominance of favorable failures was achieved.  $^{2,12,52-55}$ 

When considering the weakened roots, the cast post and core groups had higher mean values of fracture load when compared with the groups restored with fiber posts (Table 3); therefore, the second hypothesis was rejected.

The current results partially agree with those of Silva and others, <sup>22</sup> since the fracture load values of fiber posts were similar; however, there is disagreement in relation to the roots restored with cast post and cores. While Silva and others <sup>22</sup> had lower values of load to fracture for the specimens restored with

cast post and cores, the present study demonstrated higher values for the same condition, which may be associated with the greater depth of weakening in the Silva and others study, <sup>22</sup> which maintained the thickness of remaining dentin of 1 mm up to an intracanal depth of 9 mm. Studies regarding widely weakened roots <sup>20,21,23,26,56</sup> restored with cast post and cores presented results similar to that study, considering fracture load and the predominance of unfavorable failures. Cast post and cores had higher fracture load values and more unfavorable failures than the groups restored with fiber posts and fiber posts associated with materials that had an E similar to that of dentin (composite resin, accessory posts, and fiber ribbons).

When roots are restored with fiber posts or fiber posts associated with materials with properties

compatible to dentin, a more homogeneous stress distribution is observed on the dental structure when compared with cast post and cores. 4,6,57,58 Even though the tensions on roots restored with fiber posts are concentrated in the cervical third, 8,59,61 weakening was performed in this present study up to an intraradicular depth of 6 mm, which could have increased the number of unfavorable failures.

When evaluating the failures in weakened roots, 95% of the failures were unfavorable (45 from 47). Favorable failures occurred in the groups restored with fiber posts (FP-CR and FP-W). Bonfante and others<sup>21</sup> had more unfavorable failures for fiber posts and cast post and cores. Such divergence of results could be associated with the difference in weakening performed in these studies. While 0.5 mm of the dentin walls was maintained in the present study, Bonfante and others<sup>21</sup> maintained a thickness between 1.25 and 1.5 mm in the mesiodistal direction and 2.25 and 2.5 mm in the buccolingual direction (ie, more dentinal structure to support the applied loads).

When comparing weakened and nonweakened roots with the same retainer (Table 3), the t-test (p<0.05) did not show a difference between the weakened and nonweakened groups. Hence, the third hypothesis was rejected.

The current results are in agreement with Silva and others<sup>22</sup> regarding weakened and nonweakened roots restored with fiber posts. However, the current results presented divergences regarding the CPC-Ni strategy, since Silva and others<sup>22</sup> noted higher mean values of fracture load for nonweakened roots restored with cast posts than for weakened roots.

While the Student t-test showed no difference between the groups in terms of fracture load, the failure analysis showed a predominance of unfavorable failures in the weakened roots when compared with nonweakened roots, a result also observed in many studies. <sup>12,21,22</sup> This result can be explained by the preservation of dental structure. <sup>50,53,55,56</sup>

The fourth hypothesis was rejected, since the survival analysis showed no differences between the weakened and nonweakened roots (Figure 2A). On the other hand, the restorative strategies for the nonweakened condition (Figure 2B) were similar. For the weakened roots (Figure 2C), the estimated survival was higher for the FP-W strategy when compared with the CPC (gold and Ni) strategies. The discussion of the results was based on studies regarding fracture load and finite element analysis,

since the authors were unaware of the existence of a study to assess the estimated survival rate of weakened and nonweakened roots restored with intraradicular retainers.

Studies have stated that weakened teeth restored with root posts with an E similar to that of dentin have a more homogeneous stress distribution on the dental structure, promoting favorable failures when compared with more rigid posts. 4,6,8,58,59 In this context, Maccari and others, 12 Kivanç and others, 19 Bonfante and others, 21 Silva and others, 22 and Akkayan and Gülmez 62 demonstrated a high percentage of catastrophic root failures when using rigid posts (metallic and ceramic) when compared with prefabricated fiber posts after fracture load tests, supporting the findings found in the current study for FP-W vs CPC strategies.

Regarding the failure pattern obtained in the weakened and nonweakened roots, it is feasible to notice that, according to Table 1, fractures were mainly on the mesial and distal surfaces. To assess the effects of a load applied at an angle of 45°, it is essential to understand that this force can be decomposed into two components by means of the Parallelogram Law. <sup>63</sup> Decomposing the force into a vector in a Cartesian axis (Figure 5), one obtains the Cartesian components Fx and Fy (Figure 6A).

Concerning the effects of the components Fx and Fy on the specimens tested, the axial force component Fx produces compressive loading, generating stress uniformly distributed in the cross section (Figure 6B). Component Fy carries transverse force leading to bending of the structure, producing normal stresses of tensile and compression (Figure

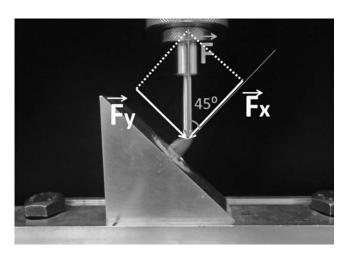


Figure 5. Diagram of the vectors decomposition on the Cartesian coordinate system of the original force F (45°) on the specimen.

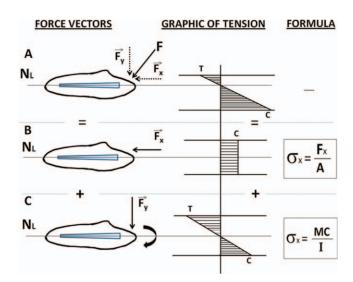


Figure 6. Diagram of the normal stresses (A), compression normal stress (B), and bending stress (C). Force vectors acting on the tooth structure expressed by tension graphs and their formulas. F, original force  $45^\circ$ ; Fx, horizontal component of F; Fy, vertical component of F; NL, neutral line; T, tensile; C, compression;  $\sigma_{\chi^*}$ , normal stress in Fx, sectional area; M, bending moment; C of the formula, distance of the neutral line to the fiber experiencing most F in the specimen; I, moment of inertia of area. Inside the tooth, the retainer is defined with a blue post.

6C). The force Fy induced shear stress in parallel planes to the longitudinal axis of the structure due to the transverse loading (Figure 7).

In the case of a dental element restored with intracanal posts, while Fx causes only compressive stresses uniformly distributed in the transverse plane (Figure 6B), Fy causes tensile stresses on the lingual surface and compression on the buccal surface (Figure 6C) and zero stress in the center of the dental element. In this area, there is the maximum shear stress caused by the component Fy (Figure 7).<sup>63</sup> Since the post is placed in the root canal, in the central area of the tooth, it is subject to minimum tensile and minimum compressive stress and maximum shear stress. This shear zone can be shifted to the region where the volume of the structure is larger. 64 In the case study, bovine roots with a trapezoidal geometric shape with the larger base facing to the buccal surface (Figure 1B) have been used. Therefore, the shear zone would be moved to the buccal area.

Given this information, it is feasible to believe that a sequence of events can lead to the ultimate prevalent failure in most failures (mesial-distal fracture mode with full coronal displacement; Table 1; Figure 3A-D,F; Figure 4B,C). Because of the shear stress, it may occur primarily at the interface of adhesive failure (post/dentin), which leads to

decementation of the restored assembly (post-crown). Thus, the retainer would be loose in the canal, being no longer a unique structure. The consequence of this is that the buccal wall of the roots suffer higher stress (compression) compared with the lingual wall.

The combination of all of the factors mentioned above may have led to a mesiodistal fracture mode closer to the buccal face (Figure 8A-E). However, it is important to note that more studies should be conducted to confirm the failure hypothesis. The strain gauge method can be a useful methodology, although caution must be taken in interpreting *in vitro* studies evaluating the restoration of endodontically treated teeth.

This present study has some limitations. Loading of materials by mechanical cycling is the *in vitro* mechanical procedure that is closest to the real conditions of aging, although it is still not possible to accurately reproduce the *in vivo* environment. Mechanical cycling has fixed execution protocols, such as value and load direction, humidity, and temperature, which limit the simulation of the *in vivo* real conditions. Thus, through a load considered of intermediate value by the authors, the specimens were aged to verify the behavior of the restorations within the period of evaluation, something that might not be possible using a lower load of cycling.

The fracture load test has limitations as well: load application until failure of the structure, high values of loading (not correspondent to *in vivo*), and high standard deviations are present in studies that use this methodology.

Further longitudinal studies evaluating survival and/or randomized controlled trials should be performed to define clinical predictability and plausibility of restoring therapies for weakened roots.

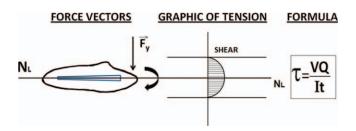


Figure 7. Diagram of the shear stress acting on the tooth structure expressed by the graph of tension and its formula.  $\tau$ , shear stress; V, load (in this case represented by the value of Fy); Q, static moment of area; I, moment of inertia of the area; I, thickness of the flat section area.

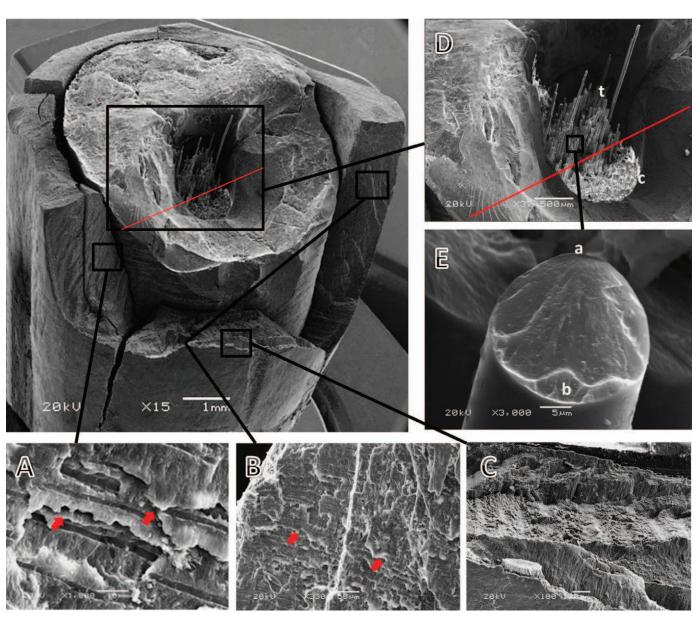


Figure 8. Representative micrographs of a specimen from group 8 (FP-CR) fractured during mechanical cycling. We can see the debonding of the restoring complex and fracture, which occurred in the buccal region, as explained by biomechanical principles cited previously. The red line refers to the neutral line of bending. (A,B). Micrograph of the fractures at the mesial and distal regions, respectively. It notes the red arrows through the peculiar characteristics related to this nature of fracture. The difference in plane for the "tear" structure and the presence of dentinal tubules. (C) Micrograph of buccal dentin wall. It appears to be a characteristic situation of "kneading" (typical "compression curl") of the buccal structure after breaking of the opposed lingual portion occurred by tensile stress. (D) Micrographs of the region of the fiber post. The red line refers to the neutral line in the post. It notes the difference between the areas of tensile (lingual "t") and compression (buccal "c") of the post. (E) Micrograph of a fiber situated in the tensile zone (lingual) of the post. By surface analysis, the fracture orientation appears to have occurred from "a" to "b." The "b" point represents the typical "compression curl" (tell-tale feature), which appears in the bend structure.

### CONCLUSION

- The tested restoring strategies were similar when restoring nonweakened roots. Thus, roots with an adequate amount of remaining dental structure can be restored using any evaluated intracanal post system.
- Taking into account the survival rate findings for the weakened roots, the fiber post with a wider
- cervical emerging diameter appears to be the best restorative alternative.
- The cast post and cores (gold and Ni) promoted higher values of fracture load of weakened roots and higher levels of unfavorable fractures.
- Weakened roots are highly susceptible to cracks and catastrophic failures in relation to nonweakened roots.

### Conflict of Interest

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

(Accepted 20 August 2013)

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# American Academy of Gold Foil Operators' 2014 Distinguished Member of the Year Award



Dr. Barry O Evans

n presenting this year's Distinguished Member Award to Dr. Barry Evans, we could begin by talking about the definition of the word "distinguished", which means "to be made conspicuous by excellence". Dr. Barry Evans exemplifies conspicuous excellence in several areas of his career and life and this award acknowledges him for his skill as a gold foil operator and his accomplishments and service to this organization.

Award

Dr. Evans' commitment to his profession can be seen in his long term membership and participation in several dental organizations, in which he has held several offices. He is a member of the Academy of Restorative Dentistry, the American College of Dentists, a Fellow in the International College of Dentists and a Fellow in the Academy of General Dentistry. He is a Past President of the Academy of RV Tucker Study Clubs, Past President of the American Academy of Gold Foil Operators, Immediate Past President of the Associated Ferrier Study Clubs and currently serves as Treasurer of the American Academy of Gold Foil Operators. He has been a long term member of 7 different study clubs and has mentored 4 study clubs. He has presented numerous essays, table clinics and papers and was honored as AAGFO Outstanding Clinician of the Year in 2007.

Dr. Evans was a member of the Seattle RV Tucker Cast Gold Study Club for 33 years, where he honed his skills in cast gold restorative work. He has been a member of the Alex Jefferies Study Club since 1983 and has had a venerable list of mentors, including Dr. Dick Tucker. Dr. Evans has been heard to say that what makes him a good dentist is "bulldog determination" and where this can be seen time after time is in the work he accomplishes in study club. There is no better example of what "distinguished member" means than the example a clinician like Dr. Evans sets; working with concentration, diligence, humility and respect for his patients to create beautiful, long-lived restorations which set a standard of excellence for his fellow study club members to follow. His roles as student and mentor are of one piece and he is above all, a constant learner. A common thread which unites the recipients of this award is that in addition to having superlative clinical skills which their fellow dentists emulate, they are all natural teachers by virtue of their ability to generously share information with their fellow clinicians.

Dr. Evans practices in Portland, Oregon where he continues to be an influence in Oregon dentistry with his involvement and support of OHSU School of

Dentistry. Barry and his wife Yvonne have two children, Arianna and Bryce (who is also a practicing dentist in Seaside, Oregon) and four grandchildren. In acknowledging how important family support is to all of us in our dental careers, Barry shares this award and the admiration of his fellow AAGFO

Members with his wife Yvonne. Thank you, Barry, for all you do for us!

Submitted by Dr Carol Klingensmith Presented at the 2014 AAGFO Meeting Portland, OR May, 10 2014

### **Departments**

## **Faculty Positions**



# University at Buffalo, The State University of New York

### Clinical Assistant or Associate Professor (HS), Assistant or Associate Professor (HS)

The Department of Restorative Dentistry, School of Dental Medicine at the University at Buffalo is seeking applicants for multiple positions in Operative Dentistry and/or in Prosthodontics. Positions are full-time, clinical or tenure-track.

Must have for all positions: (1) D.D.S. or D.M.D. or equivalent degree, (2) eligibility for licensure in NYS and (3) teaching and clinical experience. Must have for the prosthodontist position: (1) a certificate from an ADA accredited advanced program in prosthodontics at the time of employment, (2) training in implant dentistry and (3) board certification or serious intent toward board certification in Prosthodontics. For tenure track: evidence of scholarly activity and an established independent research program. An MS degree or higher preferred for all positions.

Salary and rank are commensurate with the candidate's qualifications and experience. A faculty practice opportunity is available for NYS licensed individuals.

Interested applicants should submit a letter of intent on-line with CV and 3 letters of reference from individuals who are familiar with the applicant's career. Positions available immediately.

All applications must be completed on-line at the following website: www.ubjobs.buffalo.edu/applicants/Central?quickFind=57124

Any questions, please contact Jill Uebelhoer in Restorative Dentistry at (716) 829-2862 or e-mail at: jgu@buffalo.edu.

The University at Buffalo is an Equal Opportunity / Affirmative Action Employer/Recruiter.



### Assistant/Associate Professor of General Dentistry Creighton University School of Dentistry

Creighton University School of Dentistry is seeking candidates for a full-time teaching research Assistant or Associate Professor in the Department of General Dentistry. Responsibilities include participation in didactic, pre-clinical, and clinical instruction. The position also involves engaging in research and service activities in alignment with the mission and purpose of the school and University.

Candidates must possess a DDS or DMD and be eligible for and willing to complete all requirements for licensure in the State of Nebraska. Advanced training in Dental Materials or Operative Dentistry preferred.

Creighton University is an equal opportunity employer and strives to create and maintain a diverse educational community. Interested candidates should fill out an online application at: https:// careers.creighton.edu "Faculty"



# The Effect of Three Desensitizing Agents on Dentin Hypersensitivity: A Randomized, Split-mouth Clinical Trial

CRG Torres • TM Silva • BM Fonseca ALLS Sales • P Holleben • R Di Nicolo AB Borges

### **Clinical Relevance**

The desensitizing agents tested provided rapid and effective clinical relief of dentin hypersensitivity.

### SUMMARY

The aim of this study was to evaluate the efficacy of three desensitizing agents to provide relief to dentin hypersensitivity after one session in a four-week follow-up. Forty selected patients participated in a double-blind study following a split-mouth model. One application of the desensitizing agents (A, Ad-

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Ana Luísa Leme Simões Sales, DDS, MSc student, UNESP— Univ Estadual Paulista, Institute of Science and Technology, Department of Restorative Dentistry, São José dos Campos, Brazil mira Protect [Voco]; B, Bifluorid 12 [Voco]; and C, Colgate Pro-Relief in office [Colgate Palmolive]) was performed in three different quadrants for each patient. Each tooth was evaluated by tactile and evaporative stimuli, and the sensitivity response was measured using the Visual Analogue Scale. Evaluations were performed at baseline, immediately after

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treatment, and after one, two, three, and four weeks. The application of Kruskal-Wallis and Dunn multiple comparisons tests (5%) for both tactile and evaporative stimuli showed that all agents presented a significant desensitizing effect. In groups A and B this relief was maintained for four and three weeks, respectively, as measured by tactile stimulus and for four weeks with evaporative stimulus. The desensitizing effect for group C was maintained for two weeks for both tactile and evaporative stimuli. It is concluded that all desensitizing agents tested were effective in reducing sensitivity compared to baseline values. One application of Admira Protect and Bifluorid 12 presented a longer-lasting desensitizing effect than did Colgate Pro-Relief (applied in the office) on both tactile and evaporative stimuli.

### INTRODUCTION

Dentin hypersensitivity is characterized by short-term pain, intense and subtle, caused by thermal stimulus (such as that associated with water ingestion and hot or cold food) or by chemical (pH alteration) or mechanical (excessive pressure during tooth brushing and/or inappropriate brush use) actions in dentin areas exposed to the intraoral environment. This exposure can be a consequence of enamel loss through abrasion, abfraction, or erosion or it can result from root surface exposure caused by gingival recession, periodontal treatment, or the combination of both factors. 3-6

It was previously demonstrated<sup>7,8</sup> that one in six people, both men and women, are affected by dentin hypersensitivity, with a greater incidence in elderly people. Other studies<sup>9-11</sup> reported that the prevalence of dentin hypersensitivity in the population varies from 4% to 57%. This great variation is attributed mainly to the existence of various diagnosis methods and criteria of sample selection from the population.<sup>12,13</sup> In addition, with the increase in the population's life expectancy and the greater retention of elderly people's teeth as a result of preventive care, a rise in cervical hypersensitivity may be expected.<sup>6,14</sup>

The hydrodynamics theory proposed by Brännström<sup>15</sup> in the 1960s is that most often accepted as an explanation for painful dentin transmission. According to this theory, when a stimulus is applied to the dentin, a flow movement occurs in the tubules. The dentin flow movement toward the pulp—or flow in the opposite direction—causes a mechanical

deformation of the nerve fibers that are inside the tubule or of the dentin/pulp interface, which is transmitted as a painful sensation.

The desensitizing agents can be divided into groups based on their occlusive or neural action. 16 The occlusive agents can work by different mechanisms, as the precipitation of proteins that are present in the fluid inside the tubule, precipitation of amorphous particles over the dentin and/or inside the tubule, or through mechanical action promoted by the formation of a superficial pellicle penetrating (or not penetrating) into the dentin tubules. 17 The neural blocking method consists of the direct diffusion of potassium ions through the dental structure, raising its concentration in the pulp tissue and blocking the anoxic action (nerve impulse conduction) by the altering of its action potential. 18,19

Independent of the mechanism of action, the objective of hypersensitivity treatment is the immediate interruption of the pain.<sup>20,21</sup>

The desensitizing agents can be applied by a professional (in-office treatment) or used by the patient at home. 16,22 The great variety in both the treatment types and products for dentin sensitivity can be related to the fact that this problem is hard to treat or to the fact that there is not a desensitizing agent that is good enough to eliminate the patient's discomfort. 21,23 Therefore, the aim of the present study was to investigate the clinical efficacy of three different desensitizing agents in reducing dentin hypersensitivity over a four-week period. The null hypotheses tested were that 1) the desensitizing agents tested are not able to reduce the pain resulting from dentin hypersensitivity and 2) the desensitizing actions do not differ among the tested groups when tactile and evaporative stimuli are applied.

### **METHODS AND MATERIALS**

The study protocol was approved by the local ethics committee.

### **Patient Selection**

Forty patients who presented with some degree of sensitivity in at least three quadrants of the mouth were selected, yielding a total of 225 teeth. All patients received detailed information, both orally and in written form, and signed the appropriate informed consent forms outlining the purpose of the study.

The study inclusion criteria were that the patients had to be in good general health, be at least 18 years old, and have a minimum of one tooth that was sensitive in each of the three different quadrants of the mouth. The study excluded patients who were using desensitizing agents; were receiving periodontal treatment or had received nonsurgical periodontal treatment in the last three months; were receiving anti-inflammatory, psychotropic, or antidepressant drugs and analgesic medication; were pregnant or lactating; or had an allergy to any of the components in the treatment materials used in the study. In addition, patients with eating disorders and regurgitation or chronic diseases or who had received orthodontic treatment in the previous three months were also excluded. Also excluded were those with teeth having a painful condition involving the pulp and periapical region; those with any active caries or deep cervical lesions that required restoration; those with teeth having large restorations or who had been treated in the last three months; and those with abutment teeth for fixed and removable prostheses; as well as patients who had any fractured or cracked teeth.

### **Evaluation of Sensitivity**

Two different operators worked in this study. One operator evaluated the response of each tooth to tactile and air stimuli for each patient and then measured and recorded the sensitivity. The second operator, who did not know the baseline sensitivity values for the teeth, applied the desensitizing agents to the teeth according to the manufacturers' instructions. The researcher who applied the treatment had no access to the sensitivity scores, which provided for the double-blind nature of the study.

The teeth were cleaned with pumice and a rotary brush using a low-speed handpiece. For diagnosis, the quadrants were isolated with cotton rolls and the dental surface dried with cotton pellets. Then the teeth were subjected to mechanical and evaporative stimuli. For the mechanical test, a relatively constant mild force using manual pressure was applied using a dental explorer in the mesiodistal direction across the cervical area of each tooth to determine the patient's tactile response. Immediately after the stimulus, the operator requested that the patient score the pain using a Visual Analogue Scale (VAS) coupled with a Numeric Rating Scale (NRS)

The VAS/NRS scale used a plastic ruler 15 cm long, with a groove of 10 cm in the middle of its long axis and a button which the patient could move inside the groove from one extreme to the other. On

each side of the ruler there was a different scale. On the side that was shown to the patient, corresponding to the VAS scale, "no pain" was written on the left extreme and "intolerable pain" written on the right extreme.<sup>25</sup> After each stimulus, the patient moved the button to the position representing how much pain he/she was feeling. On the opposite side, not visible to the patient, there was a NRS scale, corresponding to the VAS but in numeric values. The NRS consisted of a span of 10 cm, with markings each 1 mm. 26 After each patient marked on the VAS side the intensity of his sensitivity, moving the button to the appropriate position, the operator determined the sensitivity score by checking the number on the NRS side, corresponding to the mark determined by the patient on the VAS scale. The number corresponded to the distance (in centimeters) from the initial point (no pain) to the point the patient marked.

Five minutes after the mechanical test,  $^{27,28}$  the sensitive teeth were isolated from the adjacent teeth mesially and distally using cotton rolls in order to determine the response to the evaporative stimulus. A one-second blast of air from a dental unit syringe at 40-65 psi and a temperature of 19°C  $\pm$  5°C applied 1-3 mm away from and perpendicular to the exposed buccal cervical areas of exposed dentin was used,  $^{29}$  while adjacent teeth were protected with gloved fingers and cotton rolls to prevent false-positive results.  $^{27}$  The pain was recorded using the same VAS scale.

The sensitivity test was recorded by a calibrated examiner. Calibration procedures were performed using a dental mannequin, jet air/water, a stopwatch, and a dental explorer. The duration of the calibration process (training and calibration exercises) was approximately 20 hours. The order in which teeth were assessed in each patient was maintained at each visit. The examiner and the patient were blinded from the type of treatment performed in each quadrant. Each quadrant containing at least one sensitive tooth was randomly assigned to each of three treatments by lot. Each volunteer had at least one hypersensitive tooth in each quadrant. All sensitive teeth of each quadrant received the same treatment.

### **Application of Desensitizing Agents**

For application of desensitizing agents, the teeth were cleaned with cotton pellets and dried with air. The operating field was isolated by means of cotton rolls and suction. Only one application of the products was carried out, according to the manufac-

Group	Composition	Method of Use		
Admira Protect (Voco)	Monomers (bisphenol A diglycidyl ether dimethacrylate, 2-hydroxyethyl methacrylate);	<ol> <li>Remove excess water with an oil-free air jet. Do not overdry dentine.</li> </ol>		
	organic acids; and ormocer	2. Apply on all dentine surfaces for 20 s.		
		3. Disperse with a faint air jet.		
		<ol><li>Light-cure with a conventional polymerization device for 10 s.</li></ol>		
	_	<ol><li>Apply a second layer; disperse it with a faint air jet and light-cure for 10 s.</li></ol>		
	_	<ol><li>Remove the oxygen-inhibited layer with a cotton pellet.</li></ol>		
Bifluorid 12 (Voco)	Fluoride varnish made of synthetic resin; 6%	1. Apply a thin layer over the surface for 10 s.		
	sodium and 6% calcium fluoride	2. Dry with air.		
Colgate Sensitive Pro-Relief (Colgate-Palmolive)	Hydrated silica, calcium carbonate, glycerin, arginine 8%, water, bicarbonate, flavor, cellulose	<ol> <li>Place enough paste for one procedure in a clean dappen dish.</li> </ol>		
	gum, sodium saccharin, FD&C blue no 1	<ol><li>Fill a rotary cup with paste and run rotary cup at low to moderate speed.</li></ol>		
	_	3. Apply the product to sensitive areas for 3 s and then repeat.		

turers' instructions (Table 1). The following desensitizing agents were applied: Admira Protect (Voco, Cuxhaven, Germany); Bifluorid 12 (Voco) and Colgate Sensitive Pro-Relief in office (Colgate Palmolive, Colgate-Palmolive Company, New York, NY, USA).

The present study used at least three quadrants, characterizing a "split-mouth" study. In each quadrant different desensitizing agents were randomly applied. The study did not include a "placebo" group for ethical reasons.

After application, the patients were instructed to avoid food and liquid intake for two hours and to avoid alcoholic beverages, brushing and flossing for 12 hours. The effectiveness of the products was tested immediately after desensitizer application with the VAS scale. The follow-up examinations using the same tactile and evaporative stimuli were performed after seven, 14, 21, and 28 days.

### **Statistical Analysis**

The data were submitted to the nonparametric Kruskal-Wallis and Dunn multiple comparison tests. The significance level was set at 5%. The software program Statistix for Windows (version 8.0, Analytical Software, Tallahassee, FL, USA) was used for the calculations.

### **RESULTS**

The percentage of variations of the subject responses for the treatment groups at baseline; immediately after treatment; and in the first, second, third, and fourth weeks are presented in Table 2. This table corresponds to differences in sensitivity values of each recall compared to baseline. The results obtained for negative values correspond to a reduction in pain sensitivity. The positive values account for an increased sensitivity value to painful stimuli. When differences in sensitivity values compared to the baseline did not occur, it was perceived to indicate no variation in pain. All desensitizing agents showed decreased levels of sensitivity following the revaluations after next recalls. In general, 50%-60% of the treated teeth continued showing reduced pain after four weeks for all agents.

The mean VAS scores for the treatment groups after receiving tactile and air stimuli are presented in Tables 3 and 4, respectively. The level of hypersensitivity to tactile stimulus for each desensitizer was compared among the different recalls using Kruskal-Wallis and Dunn tests (Table 3, columns). Significant differences were observed for all tested agents. For Admira Protect, an immediate and significant reduction was observed, and this difference was maintained throughout the additional four weeks of evaluation. Bifluorid 12 presented a significant desensitizing effect immediately after application, which was maintained for three weeks, although an increase in values was observed after four weeks. For Colgate Pro-Relief a significant desensitizer effect was noticed immediately, and reduced values were retained for two weeks. After

Table 2: Variations of the Subject Responses of the Treatment Groups Immediately After and at the First, Second, Third, and Fourth Weeks (in Percentage)

							Variat	e VAS S	Scores									
		Evaporative, %									Tactile, %							
	Negative Ranks <sup>a</sup>			Pos	itive Ra	nks <sup>b</sup>	Ties <sup>c</sup>			Negative Ranks <sup>a</sup>		Positive Ranks <sup>b</sup>		nks <sup>b</sup>	Ties <sup>c</sup>			
	Α	В	С	Α	В	С	Α	В	С	Α	В	С	Α	В	С	Α	В	С
Immediately	61	57	47	9	19	18	28	23	33	68	63	61	3	6	5	27	30	32
1 wk	57	52	54	9	17	14	32	30	30	67	63	64	6	4	5	26	32	29
2 wk	54	64	52	18	13	15	27	21	32	59	56	60	16	15	14	24	28	25
3 wk	55	56	52	14	20	20	30	23	26	55	57	53	18	17	14	26	24	32
4 wk	57	60	52	13	17	20	28	21	26	57	54	50	15	12	14	26	32	35

Abbreviation: VAS, Visual Analogue Score.

three weeks, the values were not significantly different from baseline.

In order to compare the performance among all desensitizers at each recall, Kruskal-Wallis and Dunn tests were applied (Table 3, rows). At the evaluation performed immediately after the application of the products, Admira Protect and Bifluorid showed a better performance than did Colgate Pro-Relief. However, in the further evaluations, the results were not significantly different.

The level of hypersensitivity to evaporative stimulus for each desensitizer separately was compared among the different recalls using Kruskal-Wallis and Dunn tests (Table 4, columns). For all desensitizers, significant differences were observed. For Admira Protect and Bifluorid 12, an immediate significant reduction was observed, and this difference was maintained during the four weeks of evaluation. For Colgate Pro-Relief a significant

desensitizing effect was observed immediately and maintained for two weeks, although an increase was observed after three weeks.

### DISCUSSION

Pain sensation can be caused by different stimuli, such as chemical, mechanical or thermal stimuli, applied on exposed dentin under oral conditions. Several studies used a probe tip as a tactile stimulus because it causes the movement of the dentinal fluid as a result of the compression of the dentin. In addition to promoting the evaporation of the fluid inside the tubules, the air blast also decreases the temperature at the dentin surface. Both effects cause the movement of dentinal fluid from opened tubules. Using tactile and evaporative stimuli, the sensitivity level can be determined by VAS. This is considered the most appropriate method with which to diagnose pain levels because it allows for the

Table 3: Mean Visual Analogue Scores (VASs) and Percentage of Hypersensitivity Reduction for the Treatment Groups After Receiving Tactile Stimuli over Four Weeks

Recalls	Admira Protect Mean ± SD <sup>b</sup>	Bifluorid 12 Mean ± SD	Colgate Pro-Relief Mean ± SD	K-W <sup>a</sup> (df=2)
Baseline	3.59 ± 3.08 aA	3.63 ± 3.07 aA	3.35 ± 3.11 aA	$p = 0.787, H^d = 0.47$
Immediately	0.93 ± 1.85 bA	1.67 ± 2.31 bAB	1.92 ± 2.88 bB	p = 0.014, H = 8.46
1 wk	1.54 ± 2.27 bA	1.82 ± 2.29 bA	1.65 ± 2.43 bA	p = 0.344, H = 2.12
2 wk	1.82 ± 2.45 bA	1.82 ± 2.46 bA	1.86 ± 2.46 bA	p = 0.985, H = 0.02
3 wk	2.04 ± 2.76 bA	2.05 ± 2.60 bA	1.97 ± 2.70 abA	p = 0.864, H = 0.29
4 wk	1.84 ± 2.52 bA	2.27 ± 2.60 abA	2.06 ± 2.80 abA	p = 0.255, H = 2.72
K-W <sup>c</sup> (df=5)	$p = 0.000^{c}$	$p = 0.000^{c}$	$p = 0.002^{c}$	
	H = 47.04	H = 25.79	H = 18.69	

<sup>&</sup>lt;sup>a</sup> Results of Kruskal-Wallis test for the comparison among the desensitizers for the same recall.

<sup>&</sup>lt;sup>a</sup> Percentage of hypersensitivity reduction in relation to the baseline values for each product.

<sup>&</sup>lt;sup>b</sup> Percentage of hypersensitivity growth in relation to the baseline values for each product.

<sup>&</sup>lt;sup>c</sup> Percentage of maintenance hypersensitivity in relation to the baseline values for each product: A) Admira, B) Bifluorid 12, and C) Colgate ProRelief.

b Different lowercase letters in columns indicate significant differences among each recall for the same desensitizer, while different capital letters in rows indicate significant differences among the desensitizers for the same recall.

<sup>&</sup>lt;sup>c</sup> Results of Kruskal-Wallis test for the comparison among the different recalls for the same desensitizer.

d Kruskal-Wallis H statistic.

Stimuli	over Four Weeks			
Recalls	Admira Protect Mean ± SD <sup>b</sup>	Bifluorid 12 Mean ± SD	Colgate Pro-Relief Mean ± SD	K-W <sup>a</sup> (df=2)
Baseline	$3.46 \pm 2.85 \text{ aA}$	$3.37\pm2.64$ aA	$2.75 \pm 2.80 \text{ aA}$	p = 0.260, H= 2.69
Immediately	$1.72\pm2.30~bA$	$2.34\pm2.89$ bA	$1.72 \pm 2.51 \text{ bA}$	p = 0.441, H= 1.63
1 wk	1.86 $\pm$ 2.15 bA	$2.44\pm2.68$ bA	$1.68 \pm 2.43 \text{ bA}$	p = 0.352, H= 2.08
2 wk	$1.92\pm2.60$ bA	1.90 $\pm$ 2.57 bA	$1.58\pm2.35\;\text{bA}$	p = 0.819, H = 0.39
3 wk	$2.19\pm2.71$ bA	$2.08\pm2.69~\text{bA}$	$1.79 \pm 2.61 \text{ abA}$	p = 0.969, H = 0.06
4 wk	1.90 $\pm$ 2.50 bA	$1.78\pm2.52$ bA	1.80 ± 2.68 abA	p = 0.909, H = 0.19

Table 4: Mean VAS Scores and Percentage of Hypersensitivity Reduction for the Treatment Groups After Receiving Evaporative Stimuli over Four Weeks

 $p = 0.000^{\circ}$ 

H = 25.86

 $p = 0.000^{c}$ 

H = 23.19

K-W<sup>c</sup> (df=5)

translation of the subjective feedback into objective data.<sup>29</sup>

In the present study, the first null hypothesis was rejected, since the three agents tested showed efficacy in reducing sensitivity, even with a single application. The reduction of pain sensitivity was in general greater than 50% (52%-60% and 54%-57% evaporative and tactile stimuli, respectively).

The second null hypothesis tested was also rejected when tactile stimulus was applied, as the patients reported significantly higher sensitivity relief immediately after the application of Admira Protect, compared to Colgate Sensitive Pro-Relief. For the evaporative stimulus, there were no significant differences among the tested groups. Indeed, it was previously reported that the type of stimulus can influence the painful response. 29,33,34 The tactile stimulus was applied prior to the evaporative since the use of air blast results in reversible dehydration of dentin. Although the interval between stimulus applications should be of sufficient duration to minimize interactions between stimuli, the appropriate interval is not known.<sup>35</sup> Thus, it was expected that a five-minute interval between the application of evaporative and tactile stimuli would be appropriate, according to previous studies.<sup>27,28</sup>

Colgate Sensitive Pro-Relief consists of arginine, a positively charged amino acid, in physiological pH, with bicarbonate as a pH buffer and calcium carbonate. This kind of agent was developed in an attempt to seal patent dentinal tubules and, consequently, to relieve hypersensitivity. Its action is based on the natural role of saliva, which contains arginine and calcium carbonate and provides calcium and phosphorus ions to migrate to patent tubules

and create a precipitate of salivary glycoproteins and calcium phosphate, occluding the tubules. Studies<sup>23,36</sup> have shown that the plug composed of arginine, calcium carbonate, and phosphate within dentin tubules effectively reduced the fluid dentinal flow and, consequently, minimized sensitivity. Previous clinical data<sup>37,38</sup> also demonstrated the effectiveness of this product in reducing sensitivity. In the present study, the single application of the professional desensitizing paste Colgate Sensitive Pro-Relief, simulating an in-office application, resulted in an immediate reduction of sensitivity, but at the third-week assessment, the values increased and were not significantly different from the baseline values.

 $p = 0.024^{c}$ 

H = 12.89

The application time may interfere with the efficacy of this agent. A scanning electron microscopy (SEM) analysis<sup>36</sup> of exposed dentin tubules treated with Colgate Sensitive Pro-Relief for 30 seconds showed a partial tubule lumen obliteration, with reduction of the dentin permeability (69.8%). On the other hand, when the application time was increased to one minute, the tubule obliteration was complete.<sup>39</sup> Nevertheless, Petrou and others<sup>39</sup> stated that even though the tubules were obliterated, the fluid movement inside the dentin tubules was not completely inhibited; hence, the pain may persist.<sup>39</sup> Thus, it should be necessary to undergo multiple applications in seeking a longer-lasting protective coating. Therefore, the in-office use should be associated with the at-home toothpaste.

Various fluoride-based products have been tested as desensitizing agents, with different formulations, such as sodium fluoride, stannous fluoride, sodium monofluorophosphate, and fluorosilicate.<sup>22</sup> Fluoride

<sup>&</sup>lt;sup>a</sup> Results of Kruskal-Wallis test for the comparison among the desensitizers for the same recall.

<sup>&</sup>lt;sup>b</sup> Different lowercase letters in columns indicate significant differences among each recall for the same desensitizer, while different capital letters in rows indicate significant differences among the desensitizers for the same recall.

<sup>&</sup>lt;sup>c</sup> Significant differences.

d Results of Kruskal-Wallis test for the comparison among each recall for the same desensitizer.

can be added in varnishes, since they are common agents used as a dentin sensitivity treatment, providing a barrier that seals the exposed dentin. <sup>2,23,40</sup> The fast-drying varnish that adheres to the surface makes it possible to achieve long-term intensive fluoridation, retaining the fluoride as long as possible on the surface so that the fluoride is able to act. However, although the varnish can produce an immediate desensitizing effect, it has been stated<sup>2,23</sup> that these materials exhibit low adhesion that is easily removed by saliva or by the abrasion caused by brushing. The fluoride varnish Bifluorid 12 is composed of 6% calcium fluoride and 6% sodium fluoride. It creates a barrier by precipitating calcium fluoride on the dentin surface and causes the occlusion of the dentin tubules. 36,40,41

In the present study, the application of Bifluorid 12 reduced dentin hypersensitivity. According to the manufacturers, the sodium fluoride dissociates, releasing F<sup>-</sup> ions, which diffuse into the tubules and are precipitated as calcium fluoride as a result of the high calcium concentration in dentinal fluid and saliva. The calcium fluoride present in the varnish composition diffuses into the tubules and seals the canal with a semipermanent protective layer. Therefore, the calcium fluoride present in Bifluorid 12 is added to close the dentin tubules mechanically, in combination with the calcium fluoride buildup by the sodium fluoride reaction to the calcium of dentin. Previous authors<sup>42</sup> reported the effectiveness of this product in reducing sensitivity during a one-month assessment study and found some degree of sensitivity reduction even after 12 months. The tubule occlusion was demonstrated immediately after the application of Bifluorid 12 using SEM. However, after one month, some tubules had reopened. 43 This may be due to the low bonding of varnish to dentin, since it can be peeled off of the dentin surface, reducing its mechanical desensitizing effect,43 and this may be the reason for the reduction of relief effect after three weeks when tactile stimulus was applied in this group. According to the manufacturers, for a more durable effect the application should be repeated two or three times at intervals of seven days. Nevertheless, in the present study, one single application was effective to relieve sensitivity for four weeks in most of the cases.

Admira Protect is a desensitizer that is based on bisphenol A diglycidyl ether dimethacrylate and 2hydroxyethyl methacrylate monomers, organic acids, and ormocer. Ormocer materials contain inorganic-organic copolymers and inorganic silanated filler particles. According to the manufacturers, this material acts in a manner similar to that of a selfetching adhesive. It bonds to the dentin and penetrates into the tubules, creating resinous tags and a polymer layer over the surface. It is able to seal the dentin surface, reducing fluid flow. 38,44 In contrast to fluoride varnish, the tubules are sealed with a light-curing material.

In the present study, Admira Protect exhibited the best performance relative to hypersensitivity reduction detected by tactile stimulus when the evaluation was performed immediately after application. A previous SEM investigation showed the efficacy of this agent in the obliteration of the dentinal tubules. Moreover, its desensitizing effect was maintained during the four-week evaluation. This longer-lasting effect may be related to the product's components. The resinous monomers are able to adhere to dentin, forming a hybrid layer. In addition, Admira Protect contains fillers, which may promote higher resistance to abrasion, avoiding the removal of the product layer by tooth brushing.

Clinically, several treatments and products to reduce the dentin hypersensitivity have been tested. All desensitizing agents tested in the present study reduced the dentin sensitivity after one application during the four-week evaluation. However, the efficacy of the products varied among the studies and the methodologies employed. Thus, the results from different clinical trials must be considered with caution, since the dentin hypersensitivity analysis involves subjective aspects.

### CONCLUSION

Based on the methodology used, it can be concluded that

- All desensitizing agents tested were efficient in reducing hypersensitivity. They were equally effective when tactile and evaporative stimuli were applied at each recall, although Admira Protect presented a significantly better immediate effect with tactile stimulus.
- Admira Protect and Bifluorid 12 showed a longerlasting desensitizing effect in relation to Colgate Pro-Relief on both tactile and evaporative stimuli after one application.

### **Conflict of Interest**

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

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# Influence of Cavity Preparation, Light-curing Units, and Composite Filling on Intrapulpal Temperature Increase in an *In Vitro* Tooth Model

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### Clinical Relevance

Ten to 15 seconds of light-curing with a high-power density unit (>1200 mW/cm<sup>2</sup>) is recommended to keep the intrapulpal temperature increase within a safe range.

### **SUMMARY**

This study examined the effect of both the tooth substance and restorative filling materials on the increase in pulp chamber temperature when using light-curing units with different power densities.

The tip of a temperature sensor was positioned on the pulpal dentinal wall of the buccal side of a maxillary premolar. Metal tubes were inserted in the palatal and buccal root of the tooth, one for water inflow and the other for water

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outflow. Polyethylene tubes were connected from the metal tubes to a pump to control the flow rate. For the unprepared tooth group (group 1), the tooth was light-cured from the buccal side using two light-curing units (three curing modes): the VIP Junior (QTH, BISCO, Schaumburg, IL, USA) and the Bluephase LED light-curing units (two modes: LED<sub>low</sub> and LED<sub>high</sub>; Ivoclar Vivadent, Schaan, Liechtenstein). The power densities of each light-curing unit for the LED  $_{\rm low}$ , QTH, and LED  $_{\rm high}$  modes were 785 mW/cm², 891 mW/cm², and 1447 mW/ cm<sup>2</sup>, respectively. All light-curing units were activated for 60 seconds. For the prepared tooth group (group 2), a Class V cavity, 4.0 mm in width by 4.0 mm in height by 1.8 mm in depth in size, was prepared on the buccal surface of the same tooth for the temperature measurement. The light-curing and temperature measurements were performed using the same methods used in group 1.

The cavity prepared in group 2 was filled with a resin composite (Tetric N Ceram A3 shade, Ivoclar Vivadent) (group 3) or a flowable composite (Tetric N Flow with A3 shade, Ivo-

clar Vivadent) (group 4). The light-curing and temperature measurements were performed for these groups using the same methods used for the other groups.

The highest intrapulpal temperature  $(T_{MAX})$  was measured, and a comparison was conducted between the groups using two-way analysis of variance with a post hoc Tukey test at the 95% confidence level.

The  $T_{MAX}$  values were as follows: 38.4°C (group 1), 39.0°C (group 2), 39.8°C (group 3), and 40.3°C (group 4) for the  $LED_{low}$  mode. For the QTH mode, the  $T_{MAX}$  values were 40.1°C (group 1), 40.4°C (group 2), 40.9°C (group 3), and 41.4°C (group 4). For the  $LED_{high}$  mode, the  $T_{MAX}$  values were 43.3°C (group 1), 44.5°C (group 2), 44.7°C (group 3), and 45.3°C (group 4). The statistical analysis revealed the following: the  $T_{MAX}$  values were arranged by mode in the following manner:  $LED_{low} < QTH < LED_{high}$  (p < 0.05) and group 1 < group 2  $\leq$  group 3  $\leq$  group 4 (p < 0.05).

### INTRODUCTION

Light-curing of composite materials is an indispensable process in many direct and indirect restorative procedures. Many dental materials, such as restorative composites, bonding agents, luting materials for indirect restorations and orthodontic appliances, pit and fissure sealants, temporary restorations, and even some bleaching agents, employ light-curing units for the polymerization or activation of the materials. High-power density light-curing units were recently released onto the market. These lights allow materials to cure in a shorter period of time. However, they also generate more heat, which can be detrimental to the vitality of the pulp tissues.<sup>1</sup>

Zach and Cohen² reported in 1965 that temperature increases of 5.5°C and 11°C resulted in necrosis rates of 15% and 60%, respectively, of the pulpal tissues when examined after three months. In that trial, the teeth in five rhesus monkeys were heated with a soldering gun at a temperature of 275°C (±50°C) for five to 20 seconds. However, it is highly questionable as to whether the values obtained in the monkeys can be applied to humans. In a clinical study on human patients,³ a 9-15°C temperature increase did not cause histologically confirmed pulpal necrosis after three months. In that study, heat was applied to the occlusal surface of six premolars and six molars with individually fitted supports until the subjects complained of toothache.

The contralateral tooth was extracted, and the temperature increase in the pulp was measured using the same parameters as were used under the in vivo conditions. After three months, the other teeth were extracted and examined histologically. The results suggested that the pulpal tissues could tolerate a temperature rise above 5.5°C without damage. In that study, the elevated temperature was only sustained for a short duration. There was no evidence of a critical temperature rise that would cause irreversible damage to the pulpal tissues after exposure to heat-generating processes. Therefore, it is important to be cautious and accept 5.5°C as a cutoff value, knowing that the pulp can in all likelihood withstand greater increases in temperature without irreversible damage.

The increases in pulpal temperature can be affected by the dentin thickness,<sup>4-6</sup> the duration of light exposure, and the type of light-curing device used in the curing process.<sup>7-9</sup> The role of the tooth substance in the temperature rise during the light-curing process has not been studied systematically. Although many composites are placed without or with only little cavity preparation, there is no information available on the differences in the temperature increases produced by light-curing units in a prepared vs a nonprepared cavity.

Placing composites into a cavity and curing them with light-curing units has been reported to increase the pulpal temperature. 4,7,8,10-17 However, the temperature increases in only the tooth alone caused by light-curing units should be measured separately to determine the increase in pulpal temperature separately from that of the composite itself during the course of the light-curing process. There is limited information available regarding the increase in specific pulpal temperature as a result of the light-curing process of composites themselves.

This current study examined the effect of both the tooth substance and restorative filling materials on the increase in pulp chamber temperature when using light-curing units with different power densities.

The null hypotheses were as follows: First, pulp temperature does not increase with different power density light units. Second, pulp temperature does not increase when light-curing intact teeth, cavity-prepared teeth, or cavities filled with resin composites of different viscosities.

### **METHODS AND MATERIALS**

The experimental design used in this study was based on a previous study by Park and others.<sup>1</sup>

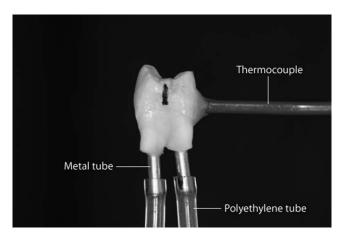


Figure 1. The premolar connected with a thermocouple and metal tubes.

# **Insertion of Thermocouples and Connection to Water Flow**

Fifteen maxillary premolars with two separate roots, without caries or restorations, were used. The roots were cut to half of their length to expose the canal spaces and to allow for metal tube insertion. After confirming that the root canals were free of debris, two metal tubes (diameter, 2 mm) were inserted into the apices of both roots to a depth of approximately 2 mm and fixed into position using XP BOND bonding agent (Dentsply DeTrey GmbH; Konstanz, Germany) and Unifil LoFlo Plus flowable resin composite (GC; Tokyo, Japan). Then, two polyethylene tubes, one for water outflow and one for water inflow, were connected to the metal tubes.

On the palatal side of the premolar, a horizontal hole (diameter, 2 mm) was drilled into the pulp chamber using a cylindrical diamond bur (FG 8614, Intensiv, Grancia, Switzerland). After beveling the orifice, the enamel surrounding the hole was etched with phosphoric acid (Total Etch, Ivoclar Vivadent, Schaan, Liechtenstein) for 30 seconds and rinsed with water. XP BOND was applied to the etched enamel, gently air-dried, and then light-cured for 10 seconds (Bluephase, 1130 mW/cm<sup>2</sup>). A K-type thermocouple (CHAL-003; OMEGA Engineering Inc, Stamford, CT, USA) was positioned in the hole, with attention being paid to place the tip in contact with the dentin on the buccal wall of the pulp chamber. The thermocouple was fixed in position using Unifil LoFlo Plus and was light-cured for 30 seconds (Figure 1). A radiograph was taken to confirm the position of the thermocouple (Figure 2). Other thermocouples were placed in the water bath and the air above the water bath. All three thermocouples were connected to a computer via a data logger

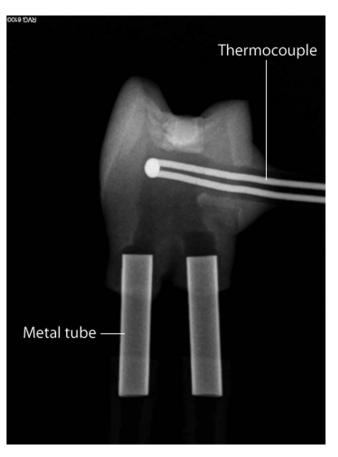


Figure 2. X-ray of the premolar with a thermocouple and metal tubes.

(Agilent 34970A, Agilent Tech, Santa Clara, CA, USA). Software (Agilent BenchLink DataLogger, version 1.4) was used to measure the temperatures at a frequency of 1 Hz.

A polyethylene tube was connected to the pump to serve as a water outlet, and a tube for water inflow was placed in the water bath containing deionized water (Figure 3). To mimic tooth blood flow, the flow rate of water was controlled using a regulator in the pump. The flow rate was set at 40-50  $\mu L/\text{min}$ .

### Measuring the Power Density of the Lightcuring Units

The tested light-curing units were as follows: a halogen lamp, VIP Junior (QTH, BISCO, Schaumburg, IL, USA), and a Bluephase LED light-curing unit (two modes:  $\mathrm{LED}_{\mathrm{low}}$  and  $\mathrm{LED}_{\mathrm{high}}$ , Ivoclar Vivadent). The diameter of the light-curing tip (light guide) was 9.8 mm in the VIP Junior and 9.0 mm in the Bluephase. The power density of the light-curing units was measured using an integration sphere and its software (Gigahertz-Optic GmbH, Puchheim, Germany). The integration power of the  $\mathrm{LED}_{\mathrm{low}}$ ,

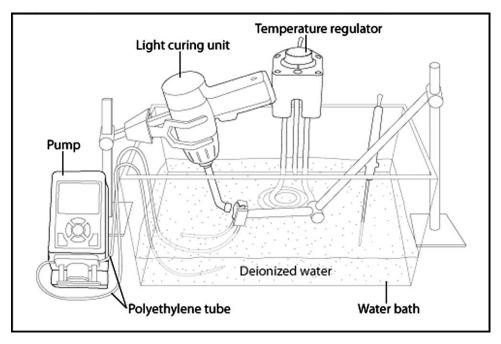


Figure 3. Schematic diagram of experimental setup.

QTH, and LED $_{\rm high}$  modes was 499.2 mW, 672 mW, and 920.4 mW, respectively, and the glass-fiber bundle area was 0.75 cm $^2$  in the VIP Junior and 0.64 cm $^2$  in the Bluephase. The power density of each light-curing unit was calculated by dividing the integration power (mW) of each light-curing unit by the fiber bundle area (cm $^2$ ). Therefore, the power densities of each light-curing unit for the LED $_{\rm low}$ , QTH, and LED $_{\rm high}$  modes were 785 mW/cm $^2$ , 891 mW/cm $^2$ , and 1447 mW/cm $^2$ , respectively.

### **Measurement of Pulpal Temperature**

Before light-curing, the temperatures in the water and air were stabilized to 39°C and 27°C, respectively.

Highest Pulp Temperature ( $T_{MAX}$ ) Measurements Before Cavity Preparation (Group 1)—Fifteen maxillary premolars, which were already prepared for intrapulpal temperature measurements, were used. When the pulp temperature was stabilized at 32°C  $\pm$  0.2°C, a tooth was exposed to the three curing modes (LED $_{\rm low}$ , QTH, and LED $_{\rm high}$ ).

For each light-curing unit, the distance from the light tip to the tooth was set to 4 mm using a metal spacer. All light-curing units were activated for 60 seconds. The pulp, water, and air temperature data were stored in a computer every second from the start of the light-curing procedure and for a total of three minutes.

The highest pulp temperature in each measurement was registered. The resulting 15  $T_{\rm MAX}$  data sets for each light-curing unit were used for a statistical comparison.

 $T_{MAX}$  Measurement After Cavity Preparation (Group 2)—For the same teeth used in group 1, a 4.0-mm (width)  $\times$  4.0-mm (height)  $\times$  1.8-mm (depth) cavity was prepared on the buccal surface of the same tooth, and the temperature of the prepared cavity was measured. After an X-ray of the tooth was taken from the proximal side, the image was digitized, and the software, Analysis FIVE (SIS, Bergisch-Gladbach, Germany), was used to measure the distance between the pulp space and cavity floor. The remaining dentin thickness was between 0.9 mm and 1.0 mm (Figure 4).

For each light-curing unit, the same procedures as described in group 1 were repeated for the pulp temperature measurement. The resulting 15  $T_{\rm MAX}$  data sets for each light-curing unit were used for statistical comparison.

 $T_{MAX}$  Measurement After Cavity Filling with Composites (Group 3)—Using the 15 teeth that had been used in group 2, the cavities were coated with a glycerine-based liquid strip gel (Ivoclar Vivadent) to allow easy removal of the composite from the cavity after curing and filled with a resin composite (RC, Tetric N Ceram, A3, Ivoclar Vivadent). After insertion of the composite it was light-cured, and the temperature was recorded using the same proce-

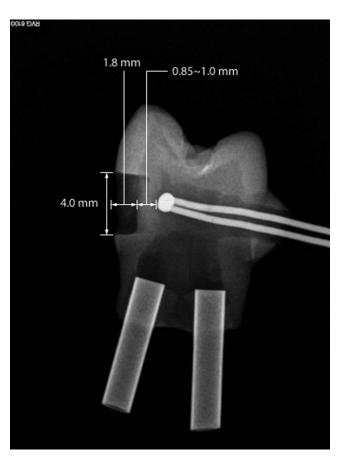


Figure 4. X-ray of the prepared premolar with a thermocouple and metal tubes.

dures as described for group 1. After each measurement, the cured composites were removed from the cavity, and the gel was washed out from the tooth surface with water. After the tooth surface had been rapidly air-dried, a liquid strip gel was applied again to the cavity surface for another composite filling. In this way, the same tooth could be used for different experiments. The resulting 15  $\rm T_{MAX}$  data sets for each light-curing unit were registered and used for statistical analysis.

 $T_{MAX}$  Measurement After Cavity Filling with Flowable Composites (Group 4)—For the 15 teeth used in group 3, the composite fillings were removed from the cavities using an explorer and hand instrument, and the cavities were coated with a glycerine-based liquid strip gel. Then, the cavities were filled with a flowable composite (FC, Tetric N Flow, A3, Ivoclar Vivadent), light-cured, and the temperature was recorded using the same procedures as described in group 1. After each measurement, the cured composites were removed from the cavities and new composites were filled into the cavities for different light-curing units using the

Table 1:	: Summar	y of Study	Design <sup>a</sup>	
	Cavity Preparation	•	Light-Curing	Temperature Recording, min
Group 1	Х	Χ	LED <sub>low</sub> (60 s)	3
			VIP Junior (60 s)	3
			LED <sub>high</sub> (60 s)	3
Group 2	0	Χ	LED <sub>low</sub> (60 s)	3
			VIP Junior (60 s)	3
			LED <sub>high</sub> (60 s)	3
Group 3	0	Tetric N	LED <sub>low</sub> (60 s)	3
		Ceram	VIP Junior (60 s)	3
			LED <sub>high</sub> (60 s)	3
Group 4	0	Tetric N	LED <sub>low</sub> (60 s)	3
		Flow	VIP Junior (60 s)	3
			LED <sub>high</sub> (60 s)	3
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 $<sup>^</sup>a$  Cavity dimension: 4.0 mm (width)  $\times$  4.0 mm (height)  $\times$  1.8 mm (depth). LED $_{low}$ · Bluephase low power, 785 mW/cm²; QTH: VIP Junior, 891 mW/cm²; and LED $_{high}$ · Bluephase high power, 1447 mW/cm².

same methods as described for group 3. The resulting 15  $T_{MAX}$  data sets for each light-curing unit were registered and used for statistical analysis.

The study design is summarized in Table 1.

### **Statistical Analysis**

The  $T_{\rm MAX}$  for each light-curing unit and the  $T_{\rm MAX}$  for groups 1, 2, 3, and 4 were compared using two-way analysis of variance (ANOVA) and post hoc Tukey test using a 95% confidence level.

### **RESULTS**

Figures 5, 6, and 7 show the relationship between the pulpal temperatures as a function of time.

The results of the  $T_{\rm MAX}$  analyses are listed in Table 2. The  $T_{\rm MAX}$  ranged from 38.4°C (group 1) to 40.3°C (group 4) for the LED<sub>low</sub> mode, from 40.1°C (group 1) to 41.4°C (group 4) for the QTH, and from 43.3°C (group 1) to 45.3°C (group 4) for the LED<sub>high</sub> mode. The results of the two-way ANOVA revealed that there were significant differences in the  $T_{\rm MAX}$  between light-curing units ( $p{<}0.05$ ) and between the groups ( $p{<}0.05$ ). There was no interaction among them ( $p{>}0.05$ ) (Table 3). The post hoc test indicated the following orders: LED<sub>low</sub> < QTH < LED<sub>high</sub> ( $p{<}0.05$ ) and group 1 < group 2 ≤ group 3 ≤ group 4 ( $p{<}0.05$ ).

### **DISCUSSION**

There were significant differences in the  $T_{\rm MAX}$  among the light-curing units using different power

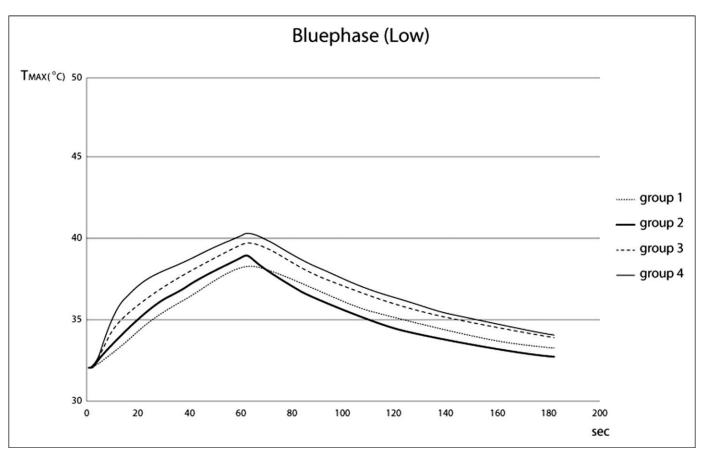


Figure 5.  $T_{MAX}$  vs time for the Bluephase in low mode.

densities. The  $T_{MAX}$  for the LED $_{high}$  mode was highest, and the  $T_{MAX}$  for the QTH mode was higher than that for the LED $_{low}$  mode. These results indicate that the pulpal temperature increased with the increase of the light-curing unit's power. Therefore, the first null hypothesis was rejected.

There was a significant difference in the  $T_{MAX}$ between groups 1 and 2. In accordance with the common belief that enamel and dentin are good thermal insulators, 18,19 these results indicate that the pulpal temperature increases more in a prepared tooth than in an unprepared tooth. Therefore, the second null hypothesis was also rejected. This indicates that enamel and dentin are effective thermal insulators. In clinical situations, the lightcuring unit tends to be placed closer than 4 mm to the prepared surface of a tooth, which can cause the intrapulpal temperature to increase more than indicated by this present study. Therefore, it is recommended that clinicians be cautious in ensuring that they do not place the light-curing unit too close and use the light for too long in one application when working with a prepared tooth.

There was a significant difference in the  $T_{\rm MAX}$  between groups 1 and 3 and between groups 1 and 4 for all light-curing units. This suggests that the composite generates an exothermic reaction during the light-curing process, which is consistent with the findings of previous studies.  $^{14}$ 

McCabe<sup>20</sup> reported that polymerization of a resin composite resulted in an increase in temperature caused by the exothermic reaction process and radiant heat from the light curing unit. The temperature differences in the  $T_{MAX}$  between groups 1 and 3 were approximately 1.4°C (LED $_{\rm low}$ ),  $0.8^{\circ}\mathrm{C}$  (QTH), and  $1.4^{\circ}\mathrm{C}$  (LED<sub>high</sub>). The differences between groups 1 and 4 were 1.9°C (LED<sub>low</sub>), 1.3°C (QTH), and 2.0°C (LED<sub>high</sub>). Considering that the  $T_{MAX}$  differences between groups 1 and 3 and between groups 1 and 4 were not very great between the  $\mathrm{LED}_{\mathrm{low}}$  and  $\mathrm{LED}_{\mathrm{high}}$  modes, most of the heat energy produced by the light-curing unit might be responsible for the increase in temperature of the tooth itself, and the effects on the filling material might be limited. 16,17 It is interesting to note that the  $T_{\rm MAX}$  differences between groups 1

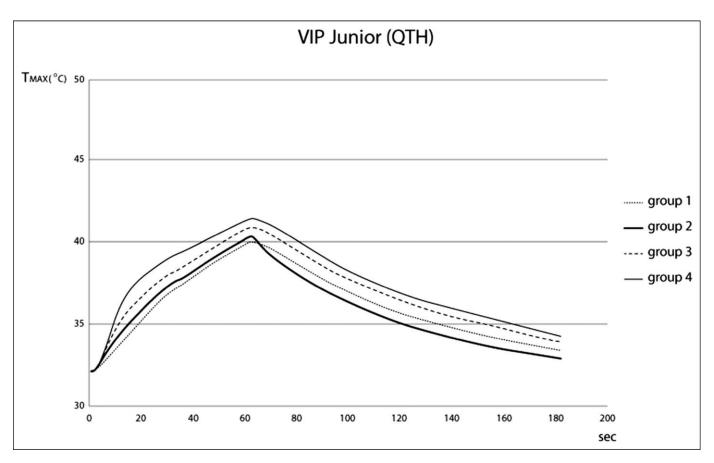


Figure 6.  $T_{MAX}$  vs time for the VIP Junior (QTH).

and 3 and between groups 1 and 4 were relatively lower in the QTH mode when compared with the  $\rm LED_{low}$  and  $\rm LED_{high}$  modes. These results might be related to the greater effectiveness of LED vs QTH modes in composite polymerization due to the efficiency in camphorquinone activation provided by the LED curing units.  $^{21-23}$ 

Even though the  $T_{\rm MAX}$  for group 3 was a little higher than that of group 2, there was no significant difference between them. This indicates that even though there are exothermic reactions in composite resins, they are not remarkable or significant. It can be assumed that the composite resins functioned as a thermal insulator, so a sudden increase in pulp temperature was prevented. Previous reports<sup>4,6</sup> indicate that when the composite is cured, a lower increase in temperature occurs in the pulp than occurs when the remaining dentin thickness is thicker. It is recommended that more care should be taken with composite filling procedures in deep cavities to avoid an increase in the pulpal temperature.

Even though the  $T_{MAX}$  in group 4 was a little higher than that of group 3, there was no significant difference between these values for all light-curing units. This is not in line with the findings of a study by Al-Qudah and others, <sup>24</sup> which reported that the temperature rise with a flowable composite was significantly higher than that found with hybrid or packable composites. Those researchers reported that a flowable composite with a higher proportion of resin available for polymerization can explain the higher temperature rise (compared to what is observed with other types of composites). This difference between the present study and the previous study may be due to the differences in the composition of the test materials and/or the test setup.

According to Zach and Cohen,<sup>2</sup> the critical threshold temperature increase to cause a pulp problem was 5.5°C. As the initial temperature in the pulp was 32°C in the present study, the amount of the temperature increase in group 4 was approximately 8.3°C, 9.4°C, and 13.3°C for the LED<sub>low</sub>, QTH, and

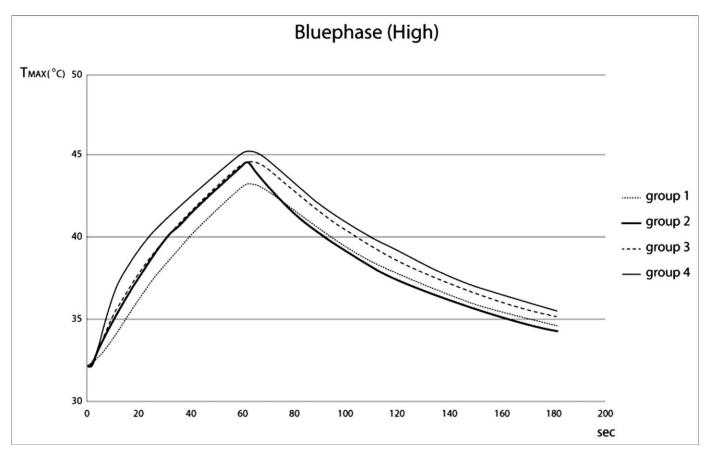


Figure 7.  $T_{MAX}$  vs time for the Bluephase in high mode.

LED<sub>high</sub> modes, respectively, after 60 seconds of light activation. Figure 5, 6, and 7 indicate that the critical threshold of 5.5°C was reached after 35 seconds for the LED<sub>low</sub> mode, after 27 seconds for the QTH mode, and after 20 seconds for the LED<sub>high</sub> mode in group 3. Park and others¹ suggested that curing devices with a high power density (>1200 mW/cm²) should only be activated for a short duration (<15 seconds) to reduce the risk of a potentially detrimental increase in pulpal temperature, even in teeth without a cavity preparation. In group 4, the temperature increased more than 5.5°C after 24 seconds, 18 seconds, and 14 seconds using

the  $\rm LED_{low}$ , QTH, and  $\rm LED_{high}$  light-curing modes, respectively (Figure 5a-c).

According to the total energy concept, the energy density should be approximately 16,000 mW/cm² to obtain good curing results in resin-based composites. <sup>25</sup> At an energy density >17,000 mW/cm², no further improvements in mechanical properties were found. In the present study, the power densities of each light-curing unit for the LED<sub>low</sub>, QTH, and LED<sub>high</sub> modes were 785 mW/cm², 891 mW/cm², and 1447 mW/cm², respectively. Therefore, the proper curing time, based on the total energy concept, would be about 11 seconds for LED<sub>high</sub>, 18 seconds for QTH,

Table 2:	The Average Value of Highest Pulpal Temperature, °C (T <sub>MAX</sub> ) <sup>a</sup>										
Groups	1 No Cavity Preparation	2 Cavity Preparation	3 Cavity Preparation + Tetric N Ceram	4 Cavity Preparation + Tetric N Flow							
LED <sub>low</sub>	38.4 ± 1.0	39.0 ± 0.8	39.8 ± 0.8	40.3 ± 0.9							
QTH	40.1 ± 1.1	40.4 ± 1.2	40.9 ± 0.9	41.4 ± 0.9							
LED <sub>high</sub>	43.3 ± 1.5	44.5 ± 1.3	$44.7 \pm 0.9$	45.3 ± 1.2							
a LED <sub>low</sub> : Bi	<sup>a</sup> LED <sub>low</sub> : Bluephase low power, 785 mW/cm <sup>2</sup> ; QTH: VIP Junior, 891 mW/cm <sup>2</sup> ; and LED <sub>high</sub> : Bluephase high power, 1447 mW/cm <sup>2</sup> .										

Table 3: Two-way Analysis of Variance (ANOVA) of Curing Lights and Steps									
Source	Sum of Square	df	Mean Square	F	<i>p</i> -Value				
Curing_Light (C)	840.963	2	420.482	368.220	0.000				
Groups (G)	77.726	3	25.909	22.689	0.000				
Interaction (C)(G)	4.836	6	0.806	0.706	0.645				
Error	191.844	168	1.142						
Total	1115.370	179							

and 20 seconds for LED $_{\rm low}$ . In Table 4, the results of intrapulpal temperature after the 10 seconds ( $T_{\rm 10sec}$ ), 20 seconds ( $T_{\rm 20sec}$ ), and 30 seconds ( $T_{\rm 30sec}$ ) of light-curing are listed. When considering the results of this current study and the total energy concept, 10-15 seconds of light-curing would be recommended for LED $_{\rm high}$ , with approximately 20 seconds for the LED $_{\rm low}$  and QTH.

Recently, high-intensity light-curing units have been used more frequently, but a downside to this instrument is that the pulpal temperature may increase significantly. Therefore, care should be taken when using high-intensity light-curing units for a long duration.<sup>1</sup>

Operational measures that may be helpful in reducing the temperature increase include the use of base materials<sup>7</sup> and modulation of the light intensity,<sup>26</sup> as well as the curing tip design and diameter.<sup>4</sup> Another variable would be the distance between the light tip and the tooth. In the present study, the distance from the light tip to the tooth was set at 4 mm.

Further studies are needed to determine if a temperature change of more than 5.5°C is detrimental to the pulp tissue. This is because the light-curing time often exceeds 60 seconds in many clinical cases

of indirect restorations, and there have been no clinical reports yet that indicate that such a lightcuring procedure harms the pulp tissue. Pulp damage is also of concern in terms of bleaching procedures. To achieve bleaching effects of teeth in a shorter period of time, an acceleration of the degradation of hydrogen peroxide is needed, and light sources, such as halogen, LEDs, and lasers, have been used to activate the hydrogen peroxide. Existing studies reveal that activation of bleaching agents by heat, lights, or laser may have an adverse effect on pulpal tissue due to an increase in intrapulpal temperature exceeding the critical value of 5.5°C.<sup>27</sup> However, there is no clear evidence regarding whether application of heat increases the frequency and severity of postoperative tooth hypersensitivity in vital bleaching procedures since randomized, controlled clinical studies addressing this question are still lacking.<sup>27</sup>

Liquid strip was used as a separating medium. It has a negligible thickness and can be washed out from the tooth surface with simple water irrigation. In the pilot study, the effect of the liquid strip on the temperature increase was tested, and it was confirmed that it did not affect the results. To prove its inertness, it would have been more preferable to place one group in the study design in which

Time	Groups	1 No Cavity Preparation	2 Cavity Preparation	3 Cavity Preparation + Tetric N Ceram	4 Cavity Preparation + Tetric N Flow
T <sub>10sec</sub>	LED <sub>low</sub>	33.24	33.79	34.77	35.68
	QTH	33.73	34.41	35.14	36.07
<u> </u>	LED <sub>high</sub>	34.13	35.33	35.62	36.95
T <sub>20sec_</sub>	LED <sub>low</sub>	34.52	35.23	36.15	37.35
	QTH	35.57	36.10	36.92	38.07 <sup>a</sup>
<u> </u>	LED <sub>high</sub>	36.51	37.91 <sup>a</sup>	38.05 <sup>a</sup>	39.53 <sup>a</sup>
T <sub>30sec</sub>	LED <sub>low</sub>	35.67	36.40	37.26	38.15 <sup>a</sup>
	QTH	37.00	37.38	38.05 <sup>a</sup>	39.12 <sup>a</sup>
	LED <sub>high</sub>	38.58 <sup>a</sup>	40.03 <sup>a</sup>	40.08 <sup>a</sup>	41.25 <sup>a</sup>

composites were placed into the cavity without the strip. However, this was not done because it would not be possible to remove the composites from a cavity without damaging the tooth structure without the liquid strip.

### CONCLUSIONS

The intrapulpal temperature increased more when using the higher intensity light-curing unit. Composite curing by light-curing units increased intrapulpal temperature. The light-curing time should be controlled depending on the power density of light-curing units to limit the intrapulpal temperature increase to within a safe range.

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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 Different Surface Treatments and Adhesives on Repair Bond Strength of Resin Composites After One and 12 Months of Storage Using an Improved Microtensile Test Method

ST Eliasson • J Tibballs • JE Dahl

### **Clinical Relevance**

Repairing and extending composite restorations is enhanced by mechanical roughening and applying freshly made silane on the old composite filling and the use of an adhesive rendering a thin bonding layer.

### **SUMMARY**

Objectives: To evaluate the effect of surface treatments and bonding systems on the repair bond strength between composite materials after one and 12 months of storage, using an improved microtensile test method.

Methods: A total of 72 composite cylinders (Tetric Evo Ceram, Ivoclar) were fabricated,

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stored in distilled water for two weeks followed by thermal cycling (5000 times between 5°C and 55°C), and served as substrate. The cylinders were mechanically roughened using 320-grit silicon carbide sandpaper, etched with 37% phosphoric acid gel, rinsed with water, and divided equally into three experimental groups: group 1, unchanged surface; group 2, sandblasting of the surface (CoJet tribochemical silica sand, 3M ESPE; Microetcher II, Danville Engineering Inc); and group 3, surface silane coating (Bis-Silane, BISCO Inc). Eight control cylinders were prepared and underwent similar aging as the substrate. Each experimental group was divided into subgroups that received the following bonding systems: one-step self-etching adhesive (AdheSE One, Ivoclar Vivadent), two-step self-etching adhesive (Clearfil SE, Kuraray America),

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and three-step etch-and-rinse adhesive (Adper Scotchbond Multi-Purpose, 3M ESPE). Fresh composite (Tetric Evo Ceram, Ivoclar) was placed and cured on top of the prepared substrate cylinders. The specimens were placed in distilled water for a week and thermocycled the same way as before. Eight composite control cylinders were also stored and thermocycled for the same period of time. Half of the cylinders in each test group were tested at one month and the second half at 12 months. The cylinders were serially sectioned in an automatic cutting machine, producing 10 to 20  $1.1 \times 1.1$ -mm test specimen beam from each cylinder. Specimens were prepared for microtensile testing and the tensile strength calculated based on the force at fracture and specimen dimension. The fracture surfaces were examined under a stereomicroscope and the type of fracture noted.

Results: The mean tensile strength of composite control was 54.5  $\pm$  6.0 MPa at one month and 49.6  $\pm$  5.1 MPa at 12 months. The mean tensile strength for the repaired groups ranged from 26.4  $\pm$  6.8 MPa to 49.9  $\pm$  10.4 MPa at one month and 21.2  $\pm$  9.9 to 41.3  $\pm$  7.5 at 12 months. There was a statistical difference between all groups (p < 0.05) at one month. This difference was less pronounced at 12 months. The highest repair strength was obtained in the group having a silane-coated surface and Clearfil, the two-step self-etching adhesive. Clearfil also had the highest repair strength within each surface treatment group. There was a tendency for lower tensile strength at 12 months compared with one month. Most fractures were of the adhesive type; the highest number of cohesive fractures, 16% at one month and 12% at 12 months, were in groups with the highest tensile strength.

Conclusion: The best repair bond strength was achieved by using freshly mixed silane solution on the substrate in addition to an adhesive, rendering a thin bonding layer.

### INTRODUCTION

The replacement of failed restorations is a major dental health care expense and accounts for roughly half of restorative dental work.<sup>1-4</sup> Removing faulty bonded-composite restorations is a demanding and time-consuming task. It has been demonstrated in a clinically simulated study that more than twice as much tooth structure was lost when removing

composite restorations than comparable amalgam restorations.<sup>5</sup> As a consequence, a more conservative and minimally invasive approach—repair rather than replacement of the whole restoration—has been suggested when possible.<sup>2,6,7</sup> This approach and philosophy has gradually been adopted by most Western dental schools.<sup>8-12</sup> Furthermore, some clinical evidence has been presented that repairing composites increases the longevity of the restorations.<sup>13</sup>

Since the introduction of resin composite materials, researchers have explored methods to repair composite restorations by adding new composite to the old. <sup>14</sup> New composite can possibly be retained to old composite either through chemical bonding to the filler particles and the organic matrix or through micromechanical bonding to irregularities in the prepared surface. 15 It is, however, generally accepted that as composite ages and water uptake occurs, there is a significant reduction in available carboxyl double bonds for chemical polymerization to new composite. 16-18 In the last two decades various reports have been published on the repair strength of resin composites. The vast majority of these investigations deal with different surface treatments of the original substrate to be repaired. This includes mechanical roughening with various grits of diamond burs and sandpaper, 19-31 abrasion with pumice,<sup>32</sup> sandblasting with aluminum oxide or silicatized sand,<sup>22,24-42</sup> etching with various concentrations of hydrofluoric acid,<sup>22,25-27,31,41,43-45</sup> application of 38% hydrogen peroxide,<sup>35</sup> and silane application. <sup>19,26,28,30,35,43,46,47</sup> In all these investigations some adhesive was used as a wetting agent in the repair process, in addition to using only flowable composite as an adhesive<sup>36</sup> and preheating the repairing composite.<sup>34</sup> In the majority of these studies, except in some using shear bond testing, the adhesive testing is performed shortly after the repair process without aging the repaired specimens. From none of these studies has a preferred method emerged to repair bisphenol A glycidyl methacrylate (Bis-GMA)-based composite resin restorations.

Storage in water for different periods is the most common procedure to age the composite to be repaired, and in a few studies thermal cycling, using extreme mouth temperatures, has been applied. Some less clinically oriented aging methods have also been used, like boiling the specimens for several hours or immersing them in citric acid for a week.

Most of the recent studies on repair of composites have examined the repairability of silorane-based restoratives. 49-52 In one recent study, however,

Table 1: Materials Used in the Inv	vestigation		
Product	Manufacturer	Lot No.	Expiration Date
Tetric Evo Ceram caps, shade B2	Ivoclar Vivadent AG, 9494 Schaan, Liechtenstein	N70113	2014-01
Tetric Evo Ceram syringe, shade A2	Ivoclar Vivadent AG, 9494 Schaan, Liechtenstein	P02083 and P11483	2014-12
AdheSE One F	Ivoclar Vivadent AG, 9494 Schaan, Liechtenstein	N58194	2012-09
Clearfil SE Bond	Kuraray America Inc New York, NY 10038, USA	Primer: 01043A	2013-04
		Bond:01557A	2013-04
Adper Scotchbond Multi-Purpose	3M ESPE Dental Products, St Paul, MN	Etch: N231977	2014-01
	55144-1000, USA	Primer: N236935	2014-01
		Adhesive: N229564	2013-11
Bis-Silane	BISCO Inc, Schaumburg, IL 60193, USA	Part A: 1000008430	2012-07
		Part B: 1000008431	2012-07
CoJet system	3M ESPE Dental Products, St Paul, MN 55144-1000, USA	CoJet sand: 355331	2012-04

repairability of many different composites was investigated.<sup>25</sup> It was concluded that none of the surface treatments tested could be recommended as a universally applicable repair technique. The effect of adhesive layer thickness on bond strength between composite and dentin has been investigated,<sup>53-56</sup> but no work on the effect of the thickness of the adhesive layer when repairing composite could be located.

The main objective of this *in vitro* investigation is to evaluate the repair bond strength between composite materials using microtensile testing of specimens united by different bonding systems and surface treatments. In addition to three different bonding systems representing self-etch and three-step etch-and-rinse systems, three different surface pretreatments<sup>10-12</sup> were used. The tested null hypotheses were as follows: 1) the repair bond strength is independent of the type of the bonding system; 2) the repair bond strength is independent of surface pretreatment; 3) the durability of the repair bond strength decreases over the course of time.

### **METHODS AND MATERIALS**

All the restorative materials used in this study are listed in Table 1. The procedure and preparation of the composite cylinders is summarized in Table 2. A total of 72 Tetric Evo Ceram (Ivoclar Vivadent, Schaan, Liechtenstein) composite cylinders, 10 mm in diameter and 6 mm in height, were fabricated in Teflon molds. The composite cylinders were incrementally built in three layers and each layer cured for 40 seconds with a Demetron A2 corded LED curing light (Kerr Corp, Orange, CA, USA). The light output was measured at 1100 mW/cm² (Norwegian Radiation Protection Authorities, Österaas, Nor-

way). A Mylar strip and glass slide was used at both ends of the Teflon mold to achieve flat-ended specimen blocks. In addition, as a control group, eight composite cylinders of the same diameter and 12 mm in height were incrementally fabricated.

After polymerization, the cylinders were immediately stored in distilled water for a total of two weeks. <sup>57</sup> The cylinders were further aged by thermal cycling 5000 times between 5°C and 55°C, with a dwell time of 20 seconds and transfer time of three seconds. The 72 cylinders were all surfaced flat with a 320-grit silicon carbide sandpaper disc (Struers, Copenhagen, Denmark) under running water for 5 seconds to obtain a flat surface with standardized roughness.

For cleaning purposes, all the experimental composite cylinders were acid etched with 37% phosphoric acid gel for 15 seconds and rinsed with water for another 15 seconds. The aged cylinders were randomly divided into three experimental groups to have the following surface treatments: Group 1— The 320-grit sandpaper finish unchanged. Group 2— The cylinders were coated with CoJet tribochemical silica sand (3M ESPE, St Paul, MN, USA) using an intraoral sandblaster (Microetcher II, Danville Engineering Inc, San Ramon, CA, USA) for 20 seconds at a distance of about 5 mm. Residual sand was removed by a stream of air for five to 10 seconds. Group 3—The cylinders were coated with Bis-Silane (BISCO Inc, Schaumburg, IL, USA) two-part silane porcelain primer. The two parts were mixed and applied to the test surfaces with a small brush for 30 seconds and gently dried with air for five to 10 seconds to evaporate the solvent. A fourth group contained the eight control cylinders.

Base specimens	Tetric Evo Ceram shade B2 cylinders (Diam: 10 mm, height 12 mm)		Tetric Evo Ceram shade B2 cylinders (Diam:10 mm, height 6 mm)  ter storage and thermo cycling [5000X (5 and 55 °C, dwell time: 20 sec, transfer time 3 sec)] total 14 days								
Aging	Wa	ter storage a	nd thermo cy						al 14 days		
Surface treatment 1				Me	chanical rougl	hening with sa	andpaper, 320	grid			
Rinsing				Acid etch (	37% phospho	ric gel for 15	sec) + water rir	rse (15 sec)			
Surface treatment 2			Non			CoJet			Bis-Silane		
		AdheSE	Clearfil	Scotchbond	AdheSE	Clearfil	Scotchbond	AdheSE	Clearfil	Scotchbond	
		Apply +	Primer 20	Etchant	Apply +	Primer 20	Etchant	Apply +	Primer 20	Etchant	
		brush 20 sec.	sec	15 sec.	brush 20 sec	sec.	15 sec.	brush 20 sec.	sec.	15 sec.	
Bonding procedure		Air flow	Air flow	Rinse 15 sec.	Air flow	Air flow	Rinse 15 sec.	Air flow	Air flow	Rinse 15 sec.	
		LC 10 sec.	Bond	Air 5 sec.	LC 10 sec	Bond	Air 5 sec.	LC 10 sec.	Bond	Air 5 sec.	
			LC 10 sec	Adhesive		LC 10 sec.	Adhesive		LC 10 sec.	Adhesive	
				LC 10 sec.			LC 10 sec.			LC 10 sec.	
New composite						Evo Ceram sh					
Aging	W	0	,	cling (5000X b		*			fer time)		
7,611,6		tot	al 10 days (1 i	month specime	ens) and 11 m	onths storage	(12 months sp	pecimens)			
Cutting			Preparir	ng square test s	pecimen rods	s approximate	ly 1.1 X 1.1 mr	n			
Test specimen	Combined	4 -	1b	4 -	2-	2b	2.	3a	3b	3c	
designation	Control	1a	10	1c	2a	20	2c	3a	30	3C	
Number of test	4.5										
specimens (1 month)	45	41	41	45	43	44	64	42	58	75	
Number of test	44		40	40	4.4	64			50		
specimens (12 months)	44	52	40	40	41	61	58	57	59	53	

Each experimental group was further divided into subgroups, each receiving a different bonding system for repair: a—AdheSE One (Ivoclar Vivadent), a onestep self-etching adhesive; b—Clearfil SE Bond (Kuraray America Inc, New York, NY, USA), a two-step self-etching adhesive, and c—Adper Scotchbond Multi-Purpose (3M ESPE), a three-step etchand-rinse adhesive. All the adhesives were applied according to the manufacturers' recommendations for placement of composite restorations.

After surface treatment and adhesive application, the original mold was carefully placed over the cylinder and a second mold fitted on the top. The aged composite cylinders were then repaired using Tetric Evo Ceram, shade A2, in three incremental layers, the same way as the original specimens, resulting in 12-mm high specimens. After this, the cylinders were placed in distilled water for a week and then thermocycled 5000 times and finally stored in water for a total of 10 days. Control cylinders were also stored and thermocycled for same period of time. The eight cylinders in each test group were then divided into two groups to be tested either immediately (one month) or after a year of storage in distilled water (12 months). During the 12-month storage period, water was regularly replaced.

The composite cylinders were mounted on an automatic cutting machine (Isomet, Buehler Ltd, Lake Bluff, IL, USA) equipped with a thin, water-cooled diamond blade. The specimens were serially

sectioned perpendicularly to the bonding surface, both in the x-axis and the y-axis, producing a number of square test specimen rods approximately  $1.1 \times 1.1$  mm. Ten to 20 test specimens were obtained from each composite cylinder. The test specimens were cleaned ultrasonically in distilled water for 3 minutes. After the cleaning procedure the test specimen rods were examined light microscopically at a magnification of  $40\times$  for voids and imperfections in the composite and the adhesive interface and for evaluation of interface thickness. Only flawless specimen rods were tested. The width and thickness of each test specimen was measured to the nearest 0.01 mm using a calibrated digital caliper (Mitutoyo Co, Kawasaki, Japan).

A novel method was developed to attach the specimen rods to the bond testing machine to secure straight alignment of the rods and uniform distribution of the tensile forces throughout the specimens. The 1.1-mm size of the test specimen rods was selected to fit into the female (hollow) end of 2-mm commercially available male/female extension screws (ELRA AS, Oslo, Norway) (Figure 1). An extension screw was fitted to each end of the test specimen rods and secured with cyanoacrylate glue (Locktite 435, Henkel Norden, Gothenborg, Sweden). A custom-fit mold was made to ensure alignment of the screws to the long axis of the specimen.

Each test specimen was mounted in a calibrated universal testing machine (Lloyd Instruments Ltd,

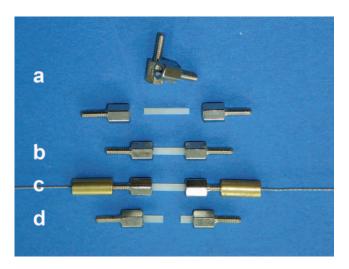


Figure 1. (a): The specimen rod to be tested and the 2-mm male/ female extension screws used. (b): The specimen rod fitted, aligned, and secured with cyanoacrylate glue. (c): The specimen assembly attached to the steel wires of the testing machine. (d): The specimen fractured at the repair joint.

Model LRX, Fareham, England) using specially attached steel wires designed to transmit pure tensile forces to the specimen. The microtensile testing was conducted at a crosshead speed of 1 mm/min until fracture. The tensile bond strength of each test specimen was calculated in megapascals by dividing the imposed force (in newtons) at fracture by the cross-sectional bond area (in millimeters squared). The test specimens were kept moist throughout the preparation of specimens and the test procedure.

The fracture surfaces were examined under a stereomicroscope (American Optical, Buffalo, NY, USA) at 40× magnification to determine whether the

failure region was within the adhesive zone or out of it. The adhesive zone was defined as the interface between the old and the new composite. The failure modes were classified as cohesive or adhesive.

The probability of failure in the test specimens was assessed by means of a distribution plot, and the significance of differences was evaluated by a mixed-model analysis of variance (ANOVA) and the Kolmogorov-Smirnov test. <sup>58</sup>

### **RESULTS**

The results are presented in Tables 3 to 5. The mean tensile strength of the unrepaired composite control group was  $54.5 \pm 6.0$  MPa at one month and  $49.6 \pm$ 5.1 MPa at 12 months. The lowest mean tensile strength in the repaired groups was for group 1c, mechanical roughening with sandpaper + Scotchbond Multi-Purpose, both at one month (26.4±6.8 MPa) and 12 months (21.2  $\pm$  9.9 MPa). This amounts to 48.4% of the strength of the control composite at one month and 42.7% of the strength at 12 months. The highest mean tensile strength for both storage periods was for group 3b, mechanical roughening + silane + Clearfil:  $49.9 \pm 10.4$  at one month and 41.3 $\pm$  7.5 at 12 months, amounting to 91.6% and 83.3% of the control composite strength, respectively. There was a general reduction in the mean tensile strength for the repaired groups after 12 months of storage, amounting from 14.6% to 21.8% of the one-month tensile strength values. In general, Clearfil had the strongest repair strength within each surface treatment group. The mean tensile strength of the unrepaired control composite decreased 8.9% during the same storage period. Statistical calculations

Table 3: Results of Microtensile Testing in the Various Groups of Surface Treatment and Bonding Systems After One Month and 12 Months

Surface	Control	Mechani	ical Rough	nening (MR)		MR + CoJ	et		MR + Silar	ne
Treatment		1a AdheSE	1b Clearfil	1c Scotchbond	2a AdheSE	2b Clearfil	2c Scotchbond	3a AdheSE	3b Clearfil	3c Scotchbond
Bonding system										
Mean μTF (SD) (1 mo) <sup>a</sup>	54.5 (6.0)	28.6 (8.6)	40.2 (9.6)	26.4 (6.8)	40.5 (12.5)	45.4 (11.2)	35.6 (7.2)	43.2 (10.0)	49.9 (10.4)	35.2 (11.0)
Mean μTF (SD) (12 mo) <sup>a</sup>	49.6 (5.1)	24.1 (7.3)	33.6 (8.4)	21.2 (9.9)	32.9 (8.5)	36.8 (10.7)	30.4 (8.3)	33.8 (6.6)	41.3 (7.5)	28.2 (6.2)
Reduction in Mean μTF	8.9%	15.7%	16.4%	19.7%	18.8%	18.9 %	14.6%	21.8%	17.2%	19.9%
Mean μTF in % of contr. (1 mo) <sup>b</sup>	100%	52.5%	73.8%	48.4%	74.3%	83.3 %	65.3%	79.3%	91.6%	64.6%
Mean μTF in % of contr. (12 mo) <sup>b</sup>	100%	48.6%	67.7%	42.7%	66.3%	74.1 %	61.3%	68.1%	83.3%	56.9%

<sup>&</sup>lt;sup>a</sup> Mean microtensile force and standard deviation in MPa after one month (1 mo) and 12 months (12 mo).

<sup>&</sup>lt;sup>b</sup> Mean microtensile force in % of the mean value of control specimens after one month (1 mo) and 12 months (12 mo).

Surface Control, Treatment %	,	Mechanical Roughening (MR)				MR + Cc	Jet	MR + Silane		
	1a AdheSE, %	1b Clearfil, %	1c Scotchbond, %	2a AdheSE, %	2b Clearfil, %	2c Scotchbond, %	3a AdheSE, %	3b Clearfil, %	3c Scotchbond, %	
Bonding system										
Cohesive fracture (1 mo)	100	4	2	2	0	7	0	5	16	4
Cohesive fracture (12 mo)	100	6	5	3	2	11	3	7	12	2

using ANOVA and Kolmogorov-Smirnov tests gave similar significance levels for all comparisons. At one month, there was a statistical difference between all groups, which was less pronounced at 12 months (Table 5). There was a tendency for lower tensile strength after one year compared with one month in all groups. However, the difference was not statistically significant. At both observation times, the mean strength of the control composite was significantly higher than that of the strongest repair.

The thickness of the bonding layer using Adper Scotchbond Multi-Purpose varied somewhat but appeared mostly to be approximately 175 µm; the AdheSE One layer was approximately 20 µm; and Clearfil SE, less than 5 µm.

The percentage of cohesive fractures for each group is presented in Table 4. All the cohesive fractures occurred in the old composite. Most cohesive fractures for the repair groups, 16% of specimens at one month and 12% at 12 months, were in group 3b, which also had the highest mean repair strength for both storage periods; group 2b had 7% at 1 month and 11% at 12 months and came in second

in mean tensile repair strength. Other groups had fewer or no cohesive failures.

### DISCUSSION

Shear bond strength tests have been widely used when measuring adhesion to dental structures or dental materials. The wide acceptance of this testing method is due to its relative simplicity when compared with tensile strength methods. Some authors have found that conventional shear bond testing produced stress concentrations in the substrate or adherend, leading to cohesive failures when testing adhesive joints and therefore giving misleading results.<sup>59,60</sup> This has led to increased use of the microtensile test where, in a very small specimen, comparatively more uniform loading stress distribution is obtained and the tensile forces concentrated in the adhesive interface are tested. 61,62 In a review on microtensile bond testing, Pashley<sup>62</sup> stated that microtensile testing offered versatility not obtainable with other methods even though it is more labor intensive. Poitevin and others<sup>63</sup> reported on composite adhesive strength to dentin and concluded that microtensile testing was a reliable laboratory test in

	Between Groups at One Month and 12 Months*								
Groups	1a	1b	1c	2a	2b	2c	3a	3b	3с
control	A, b	A, b	A, b	A, b	A, b	A, b	A, b	A, b	A, b
1a		A, b	A, ns	A, b	A, b	A, b	A, b	A, b	A, b
1b			A, b	A, ns	A, ns	A, ns	A, ns	A, b	A, b
1c				A, b	A, b	A, b	A, b	A, b	A, b
2a					A, ns	A, ns	A, ns	A, b	A, ns
2b						A, b	A, ns	A, b	A, b
2c							A, ns	A, b	A, ns
3a								A, b	A, b
3b									A, b
3c	•	•		•	•		•		•

Abbreviation: ns. difference between groups not statistically significant.

Uppercase letter represents one month and lowercase letter represents 12 months storage. Similar letters represent statistical difference (p<0.05).

ranking contemporary adhesives on their bonding effectiveness. Recently, a microshear test was introduced to measure bond strength between dentin and resin composite, intended as an alternative to microtensile testing. The main reason given for using the microshear test was it required a less demanding and time-consuming specimen collection. Only one investigation on composite repair using microshear bond testing was found.

Several authors<sup>66</sup> have found some evidence for correlation of laboratory bond strength with clinical retention rates of class V restorations. In a recent study, Heintze<sup>67</sup> found that both macrotensile and microtensile bond strength tests correlated better with clinical retention of cervical restorations than macroshear and microshear testing. He recommended shear testing to be abandoned due to critical and inadequate stress distribution and unreliable correlation to clinical outcome. Because no universal agreement exists on bonding methods, these authors decided after preliminary investigation to use microtensile strength testing.

For this investigation an easier and much less time-consuming method was developed to measure the tensile bond strength, where straight alignment of the specimen rods glued into commercially available and inexpensive extension screws was obtained. The specimen/extension screw assembly, screwed to the aligned steel wires attached to the testing machine, directed the pulling force longitudinally from the ends of the specimen. Under these testing conditions a vast majority of specimen rods broke in the repair junction. In preliminary testing of the methods and materials used in this study, one flat side of the specimen was glued directly to the jig of the testing machine, resulting in cohesive fractures much more often than when the extensionscrew setup was tested. This was attributed to the possibility that the forces might have been less uniformly distributed throughout the specimen than in the new method.<sup>68</sup> In all the other comparable investigations <sup>19,22,25,43,69</sup> on repair, where microtensile testing was used, cohesive failure is reported as very high, up to 95%. The number of cohesive fractures reported in some of these studies is surprising, because it could be anticipated that the adhesive joint would be the weakest link. If the cohesive fractures are relatively few, as in this study, the results more likely demonstrate the true repair strength of the adhesive joint between the old and the new composites. In general, more cohesive fractures can be expected as the repair strength approaches the fracture strength of the composite used, as observed in this study. However, when the repair strength measured is only half the cohesive strength of the composite used, and two-thirds of the fractures are reported as cohesive, the testing procedure must be questioned.<sup>19</sup>

When the three bonding systems tested in this study are compared separately within each surface treatment group, it is obvious that Adper Scotchbond Multi-Purpose, the three-step adhesive, gave the lowest repair microtensile bond strength.

The three-step adhesives have been classified as the criterion standard in adhesive technology when placing resin composite restorations, especially large posterior restorations in load-bearing areas. 70-72 The great success this group of materials has experienced is possibly because the relatively thick and more flexible adhesive layer serves as a shock absorber between tooth and composite when the restoration is under masticatory stresses. 53,56 Another explanation could be that it contains less hydrophilic monomers than the more acidic self-etching adhesives. High hydrophilicity of an adhesive system may impair the long-term durability of the adhesive, because hydrophilic monomers tend to absorb more water, in time weakening the bond. 16,26 One study 43 on repair of composites reported early signs of degradation for hydrophilic self-etching adhesive repair bond compared with a hydrophobic three-step adhesive at six months. In most studies 24-48 the specimens are tested immediately or a few weeks after the composite repair, and no studies 23,43 could be found with storage periods exceeding six months. In this study the specimens were tested after one and 12 months of storage in water in addition to thermocycling. Between the testing periods, a general reduction in mean tensile strength values, 16% to 22%, was observed for all the repaired groups (Table 4). These differences, however, were not statistically significant. A slight reduction, 8.9%, was also observed for the control composite specimens between the storage periods.

When gluing hard pieces together it is of utmost importance to wet both surfaces with the appropriate glue and bring the pieces as tightly together as possible. Evaluating the results and mode of failure from this study, it is postulated that the adhesive film thickness plays a role when repairing resin composite with resin composite. The components in the Clearfil system are considerably more fluid than those in the AdheSE One and the Adper Scotchbond Multi-Purpose adhesives, therefore producing a thinner adhesive layer. Almost all the Adper Scotchbond Multi-Purpose and the AdheSE One specimens failed

in the adhesive layer, and many specimens showed adhesive material on both the repaired and the new composite, indicating that the tensile strength of the adhesive itself directed the repair tensile strength at failure. In the Clearfil specimens, the fracture appeared to occur more between the old composite and the adhesive, except for group 3b, the silane-Clearfil group that showed the strongest mean repair bond, which had almost 92% of the mean cohesive strength of the control composite at one month and more than 83% at 12 months. There the fracture often appeared both between the old and the new composite and the adhesive in the same specimen. Coelho and others<sup>56</sup> observed lower microtensile bond strength values for Single Bond (3M ESPE) as the adhesive layer between dentin and composite was increased from one layer to three layers; whereas, no correlation could be found for the more fluid Clearfil SE (Kuraray). In that investigation, however, the majority of specimens for both adhesive systems presented mixed cohesive/adhesive failure mode. 56

In this investigation three surface treatments of the aged composite were used. The purpose of a surface treatment is to increase the surface energy and/or the surface roughness. A common practice for mechanical roughening of composite specimens is to use silicon carbide sandpaper with a specific grit, giving a standardized surface roughening. Use of 320-grit sandpaper has been selected by several investigators, 23,69,73 the same grit selected for this study. Both of the other two surface treatments added sandblasting the surface with CoJet silica-coated alumina particles or silane application to the 320-grit sandpaper roughening, which significantly improved the mean bond strength (p<0.05), except for group 2b at 12 months. The CoJet particles are designed to penetrate and be embedded in the surface of the substrate and leave it partially coated with silica.<sup>74</sup> It is possible that the embedded particles act as microretention for the new composite, explaining the improved bond strength. Sandblasting using alumina particles has also been reported to improve repair bond strength. 25,43 In one study<sup>22</sup> no difference was found between sandblasting with aluminum oxide and silica coating with CoJet. It was postulated that similar surface roughness pattern resulted in similar mechanical retention.

It is a known fact that silane bonds well to silicabased materials and also to resin composites. Many resin composites contain filler materials that consist of siliceous compounds. This can explain why silanization of old composite improves bonding to new composite as demonstrated in this study. Some studies<sup>22,28,43</sup> are, however, in disagreement with our findings. One-bottle prehydrolyzed silane solutions have a relatively short shelf life and gradually become less reactive after opening of the bottle, preventing optimal adhesion. In this study a freshly mixed (two-bottle) silane system was used. The two solutions are mixed just before use to allow hydrolysis of the silane to secure a fresh reactive solution. Lundvall and others found a much higher bond strength when repairing porcelain with composite using two-bottle silane, whereas one-bottle silane showed similar bond strength as the group without silane.

Using air abrasion in the mouth requires an intraoral sandblaster and it can be difficult to achieve in the dental operatory. Using silane as an adhesion promoter is a simple method that requires no extra equipment and, according to this study, achieves comparable or better results. Within the limitations of this study, a two-bottle silane adhesion promoter in addition to a thin layer of adhesive would be the treatment of choice when repairing resin composite.

### **CONCLUSIONS**

The results obtained did not support the three null hypotheses that were claimed. The best repair bond was achieved by using freshly mixed silane solution in addition to an adhesive rendering a thin bonding layer.

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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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# Effects of Heat Treating Silane and Different Etching Techniques on Glass Fiber Post Push-out Bond Strength

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### Clinical Relevance

Surface treatment enhances bond strength of a fiber post. Heat treating silane improves its efficiency.

### **SUMMARY**

The aims of this study were to compare two pretreatment methods of a fiber post and to evaluate the effect of heat treatment to applied silane on the push-out bond strength for different levels of root. In this *in vitro* study, 40 glass fiber posts were divided into five groups (n=8) according to the kind of surface treatment applied. They were then inserted into extracted and endodontically treated human canines using a self-etch resin cement (Panavia F2.0, Kuraray, Japan). Group HF+S =

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hydrofluoric acid (HF) etching and silane (S) application; group HF+S+WP = HF etching and heat-treated silane application and warmed posts (WP); group  $H_0O_0+S = hydrogen$  peroxide etching and silane application; group  $H_0O_0+S+WP = hydrogen peroxide and heat$ treated-silane application and warmed post; and group C, the control group, received no pretreatment. After completion of thermal cycling (1000 cycles, 5-55°C), all specimens were cut horizontally to obtain three sections. Each section was subjected to a push-out test, and the test results were analyzed using two-way analysis of variance, post-hoc Tukey honestly significant difference test, and a paired sample *t*-test ( $\alpha$ =0.05). It was found that bond strength was not statistically influenced by the kind of etching material used (p=0.224), but was significantly affected by heat treatment of applied silane (p < 0.001). The interaction between these two factors was not statistically significant (p=0.142). Group HF+S+WP showed the highest bond strength (12.56±1.73 MPa) (p<0.05). Scanning electron microscopy revealed the effect of the different treatments

on the surface characteristics of posts. In the four pretreated groups, the bond strength decreased significantly from the coronal to the apical root canal sections ( $p \le 0.05$ ). The results of this study show that the use of heattreated silane significantly enhances the pushout bond strength of the fiber posts to root. HF acid etching with heat-treated silane application led to the highest bond strength.

### INTRODUCTION

Endodontically treated teeth may weaken due to caries, fracture, previous restorations, and access cavity preparation. Casting posts are frequently used to restore such teeth that have an insufficient coronal structure; the aim is to provide adequate retention for future prosthetic restorations. 1-3 Traditional metallic cast posts are gradually being replaced by fiber posts. 4,5 The latter have several mechanical and clinical advantages, such as esthetic appeal, corrosion resistance, easier removal for endodontic retreatment, faster insertion, and decreased probability of catastrophic root fracture.<sup>3,5,6</sup> The last of these advantages is the most important and is mainly attributed to the elastic modulus of fiber posts, which is closer to the modulus of dentin and improves the distribution of occlusal stresses to the root dentin. Failure of fiber posts and core restorations usually occurs from debonding or, less often, from fracture of the fiber post.<sup>2,4,5</sup> In the case of inadequate bond strength, debonding occurs between the fiber post/resin and/or the resin/root canal dentin interfaces. <sup>2,7</sup> The matrix of fiber posts is usually composed of highly cross-linked epoxy resin with a high degree of conversion. Silanes do not generally bond well with the epoxy matrix. A chemical bond via application of silane may be obtained only with the exposed glass fibers of the post.<sup>2</sup> Several factors are known to affect the retention of posts, such as post length and design, type of luting agent, cementation procedure, and preparation, shape, and condition of the post space.8-10

Various pretreatment methods involving the use of mechanical or chemical agents have recently been proposed for improving resin bonding to fiber posts. <sup>11-13</sup> Although several researchers propose the use of silane coupling agents to form a strong bond between the composite resin and the fiber post, there are several different views about the efficiency of post silanization. Further, silane application is known to be a technique-sensitive step. Numerous factors influence the effectiveness of silane, such as

its composition (pH, presence of solvent, molecular size) and the application method used. The chemical reaction between silane and an inorganic surface may be enhanced and catalyzed by acid treatment of the fiber-post surface or by silane heating, <sup>14</sup> particularly in the case of two-component silane solutions, which have been shown to be more sensitive to heating. <sup>15</sup>

Surface etching of fiber posts using chemical agents such as hydrofluoric acid (HF), hydrogen peroxide ( $\rm H_2O_2$ ), hydrochloric acid, and potassium permanganate produces a rough surface and therefore increases mechanical retention between the post and the resin luting agent. Although the use of HF achieves this purpose, it may damage glass fibers and affect the integrity of the post.  $\rm H_2O_2$  etching is a considerably milder technique, in addition to being simple, effective, and clinically feasible for enhancing bond strength.  $^{2,16}$ 

The effect of  $\rm H_2O_2$  on bond strength has been evaluated through microtensile tests. <sup>16,17</sup> However, because a push-out test is more reliable than a microtensile test, <sup>18</sup> in this study the former was used to evaluate bond strength. In studies thus far, warm air has been used for heat treatment of a silanated fiber post surface. Heat treatment of silane with warm water may result in the formation of a thin layer of silane and may enhance bond strength.

The present *in vitro* study was performed to compare the effects of two kinds of chemical etching pretreatments of the fiber-post surface on the pushout bond strength and to determine whether the heat treatment of the applied silane solution with warm water could increase the bond strength to exceed that resulting from a room-temperature airdrying procedure. The null hypotheses tested in this study were as follows: 1) heat treatment of silane solution does not affect the push-out bond strength of fiber posts, 2) there is no difference in bond strength between different etching techniques, and 3) there is no difference in bond strength at different root levels.

### **METHODS AND MATERIALS**

### **Specimen Preparation**

Forty freshly extracted non-endodontically treated human maxillary canines were extracted for periodontal reasons. Inclusion criteria were absence of severe root curve, decay, and cracks; existence of at least a 14-mm root length from cemento-enamel junction to apex. Exclusion criteria were completely oval and wide canals and defect formation in specimens during preparation. All external debris was eliminated with an ultrasonic scaler. Teeth were stored in a 0.9% saline solution at 4°C for no longer than four months. To disinfect, specimens were immersed in 2.5% NaOCl for 15 minutes. Each tooth was marked at a 14-mm distance from the apex. The crown portion of each tooth was removed at this mark with a diamond disk under water cooling. The root canals were then shaped to size 30 with a 0.06 taper (K-Files, Maillefer, Ballaigues, Switzerland) and, after irrigation with normal saline, were filled with gutta-percha (Diadent, Tianjin, China) and a non-eugenollic sealer (AH26, Dentsply-Maillefer). Specimens were fixed vertically and embedded in autopolymerizing clear acrylic resin (Triplex, Ivoclar Vivadent, Schaan, Liechtenstein) surrounded by a wax mold. Before post placement, excess guttapercha was removed with Gates Glidden burs #3 (Gates Drills, Mani Inc, Utsunomiya, Japan), leaving 4 mm of it intact in the apical portion. Prefabricated conical shape glass fiber posts #3 (FRC Postec Plus, Ivoclar Vivadent) were used in this study. Their polymer matrix was composed of aromatic and aliphatic dimethacrylates. They also contained ytterbium trifluoride. The root canals were then prepared with a reamer #3 that was available in the FRC Postec Plus kit. The prepared spaces were rinsed with 2.5% NaOCl to remove the smear layer from the root canal walls. Final irrigation was done with normal saline; excess water was then removed with air and paper points. Posts were cut horizontally at a mark made 10 mm from the end. After the complete seating of posts was confirmed, they were divided into five groups (n=8) and prepared in different manners. In group HF+S (HF + silane), the posts were exposed to 9.5% HF (Porcelain Etchant, Bisco, Schaumburg, IL, USA) for 60 seconds, irrigated with water for 15 seconds, then gently air-dried. To enhance chemical bonding, a silane solution (Bis-silane, Bisco) was applied on the post surface in two layers by a microbrush (Multibrush Multi-colors, Denbur Inc, Oak Brook, IL, USA) and gently air-dried for 60 seconds at room temperature following the manufacturer's instructions. In group HF+S+WP (HF + silane + warmed post), pretreatment of posts was performed as explained for group HF+S followed by immersion of the silanated posts in warm water (45°C) for 10 seconds and then gentle air-drying with warm air. The water temperature was adjusted and fixed with a Hanau Compound Heater (Hanau Engineering Co, Inc, Buffalo, NY, USA). Warm air (45±5°C) was generated from a blow dryer (Heat-Blo Guns, Fisher Scientific, Pittsburgh, PA, USA) blown onto the

silane applied to the post surface for 30 seconds. In group  $\rm H_2O_2+S$  ( $\rm H_2O_2$  + silane), the posts were immersed in 10% hydrogen peroxide for 20 minutes, followed by the same procedures as described for group HF+S. In group  $\rm H_2O_2+S+WP$  ( $\rm H_2O_2$  + silane + warmed post), pretreatment of the posts was the same as for group  $\rm H_2O_2+S$ . The final procedure was immersion of silanated posts in warm water (45°C) for 10 seconds and then gentle air-drying with warm air. In group C, no post surface pretreatment was carried out.

A self-etch resin cement (Panavia F2.0, Kuraray, Osaka, Japan) was used to cement the posts. Equal amounts of ED Primer II liquids A and B (Kuraray) were mixed and applied to the prepared root space with a microbrush tip for 30 seconds; the excess was removed by paper points. Equal amounts of Panavia F2.0 base and catalyst paste were mixed on a pad with a plastic instrument and applied to the prepared space with a lentulo spiral #30 (Maillefer). The posts were then coated with luting cement and inserted into the prepared canal spaces using finger pressure. The excess cement was immediately removed with a microbrush. The luting agent was polymerized with a light curing unit (Demi-LED Light curing system, Kerr Corp, Orange, CA, USA) with 400 mW/cm<sup>2</sup> output for 40 seconds. The specimens were stored in water at room temperature for 24 hours and subjected to thermal cycling (1000 cycles, 5°-55°C, 30-second dwell time).

### **Push-out Testing**

Each specimen was sectioned perpendicular to its long axis with a slow speed diamond saw (Accutom-50, Struers, Copenhagen, Denmark). After discarding the first 0.5-mm slice, three 2-mm-thick sections of each root were obtained. The thickness of each slice was measured and recorded using a digital caliper (SC-6 Digital Caliper, Mitutoyo Corp, Tokyo, Japan) with an accuracy of 0.01 mm. Each specimen was positioned and fixed onto the stainless steel jig, which had a central hole to support the dentin and acrylic resin. A compressive load was applied apicocoronally on the apical surface of each slice by a cylindrical metallic pin on a Universal Testing Machine (Zwick Roell Z020, Zwick, Germany). Because of the tapered type of the post, three different pin sizes (1.1, 0.9, and 0.7 for the coronal, middle, and apical sections, respectively) were used for push-out testing. The load was applied with a crosshead speed of 0.5 mm/min until failure occurred. Push-out bond strength was expressed in megapascals (MPa) by dividing the load at failure

Table 1: Mean Push-out Bond Strengths (MPa) and Standard Deviations (SDs) for Experimental and Control Groups <sup>a</sup>						
Group <sup>a</sup>	Mean (SD)					
	Root Levels			Total <sup>b</sup>		
	Apical	Middle	Coronal			
HF+S	6.94 (4.07)	8.43 (1.65)	9.34 (3.58)	8.24 (1.49) <sup>y</sup>		
HF+S+WP	14.64 (4.57)	10.96 (7.9)	12.08 (2.98)	12.56 (1.73) <sup>z</sup>		
$H_2O_2+S$	6.44 (2.11)	6.41 (4.68)	11.64 (2.62)	8.16 (1.38) <sup>y</sup>		
$H_2O_2+S+WP$	6.76 (1.89)	9.69 (2.59)	14.38 (10.51)	10.28 (3.57) <sup>yz</sup>		
Control	9.13 (2.55)	10.53 (2.23)	6.96 (5.52)	8.87 (1.31) <sup>y</sup>		

<sup>&</sup>lt;sup>a</sup> HF+S, hydrofluoric acid + silane; HF+S+WP, hydrofluoric acid + silane + warmed post; H<sub>2</sub>O<sub>2</sub>+S, hydrogen peroxide + silane; H<sub>2</sub>O<sub>2</sub>+S+WP, hydrogen peroxide + silane + warmed post; control, no pretreatment.

Means with different superscript letter show a statistically significant difference between groups (p<0.05).

(Newtons) by the surface area (mm<sup>2</sup>). The total bonding area for each segment was calculated using the following formula:

$$\pi(\mathbf{R} + \mathbf{r})[\mathbf{h}^2 + (\mathbf{R} - \mathbf{r})^2]^{1/2}$$

(R=Radius of the canal at the coronal surface of the slice, r=radius of the canal at the apical surface of the slice, h=height of the slice.)

### Failure Mode Evaluation

After the posts were dislodged, they were observed under a stereomicroscope (Lomo SF-100, MBC-10, Moscow, Russia) (36×), to determine the type of failure. Failures were classified in three groups: 1) cohesive failure in post or dentin, 2) adhesive failure between the resin and post or the resin and dentin, and 3) mixed failure.

### Scanning Electron Microscope Analysis

Four posts were selected in order to evaluate their surface conditions after acid etching. For this evaluation two posts were immersed in  $10\%~\rm H_2O_2$  for 20 minutes, and two posts were exposed to  $9.5\%~\rm HF$  for 60 seconds. The prepared specimens were then immersed in  $96\%~\rm ethanol$  and gently air-dried. Each post was sputter-coated with gold alloy (SCD 005 Sputter coater, Bal-Tec Co., Balzers, Vaduz, Liechtenstein, Germany) and analyzed under a scanning electron microscope (SEM, Philips XL30, Philips Eindhoven, Netherlands).

### **Statistical Analysis**

The two-way analysis of variance (ANOVA) was used to evaluate the effect of independent factors (different chemical etching and heat treatment of silane solution) on the dependent variable (push-out bond strength). Moreover, post-hoc Tukey honestly signif-

icant difference (HSD) test and paired sample t-test were used. The significance level was set at  $\alpha$ =0.05.

### **RESULTS**

The mean and standard deviation (SD) values of the push-out bond strength for each of the five groups are shown in Table 1. Two-way ANOVA revealed that the type of chemical agent used for etching the post surface had no significant effect on bond strength (p=0.224), whereas the bond strength was significantly increased via heat treatment of the silane solution using warm water (p<0.001). The interaction between these two factors was not statistically significant (p=0.142).

Use of HF for etching in combination with the heat treatment of the silane solution produced the best overall results. The Tukey HSD test displayed significant differences between group HF+S+WP and group HF+S (p=0.002) and between group H $_2$ O $_2$ +S (p=0.001)and group C (p=0.009). The difference between group HF+S+WP and group H $_2$ O $_2$ +S+WP was not significant (p=0.20). By use of the paired sample t-test between the four pretreated groups and ignoring the control group, the coronal third of the root (10.88±6.08 MPa) showed significantly greater bond strength than the middle third (9.21 ± 4.53 MPa) and the apical third (8.78 ± 4.36 MPa) at p=0.05 and p=0.022, respectively.

Evaluation of the failure mode revealed that the most frequent failure was mixed (77.5%), followed by adhesive failure (14%), and cohesive failure in the post (8.5%). There was no cohesive failure in dentin.

### **SEM Analysis**

SEM evaluation revealed that the post surface morphology was altered after etching with 9.5% HF and  $10\%~H_2O_2$ . The surface treatments dissolved the post resin matrix and created microspaces among

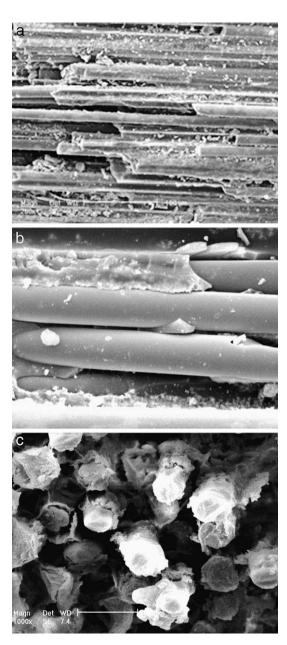


Figure 1. SEM photomicrographs of the post surface and cross-section of the post treated with 9.5% HF. (a): Post surface, 200×, bar =  $100\mu m$ . (b): Post surface 1000×, bar =  $10\mu m$ . (c): Cross-section of the post, 1000×, bar =  $10\mu m$ .

the exposed fibers. Treatment with 9.5% HF had a greater impact on the post surface. In the cross-section view it was revealed that more superficial fibers were exposed with the HF pretreatment because larger amounts of the resin matrix were removed to a greater depth (Figures 1 and 2).

### **DISCUSSION**

The present study evaluated the push-out bond strength of a glass fiber post to the root canal dentin

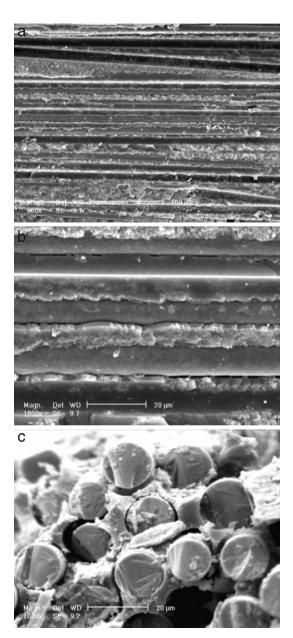


Figure 2. SEM photomicrographs of the post surface and cross-section of the post treated with 10% H $_2$ O $_2$  (a):.Post surface, 200%, bar =  $100\mu$ m. (b): Post surface 1000%, bar =  $100\mu$ m. (c): Cross-section of the post 1000%, bar =  $10\mu$ m.

with different surface pretreatments. There were no significant differences between the groups prepared with HF and those prepared with  $\rm H_2O_2$ . The second null hypothesis was accepted. Based on the SEM analysis, treatment with 9.5% HF dissolved the resin matrix more extensively and to a greater depth than did treatment with 10%  $\rm H_2O_2$ . Because of the more corrosive nature of HF, dissolution of resin matrix and glass fibers creates a rough surface that causes penetration of resin composite into the microporos-

ities and leads to an increase in bond strength. However, the etching effect of H<sub>2</sub>O<sub>2</sub> depends on its capacity to partially dissolve the resin matrix, breaking epoxy resin bonds and exposing the surface of fibers to silanization through a mechanism of substrate oxidation. 15,19 This effect, in which only the epoxy resin is dissolved with no effect on glass fibers, may reflect the insignificantly lower bond strength of H2O2 groups. These findings are in contrast to the results of the study by Vano and others, 19 which reported an increased bond strength after H<sub>2</sub>O<sub>2</sub> application compared with HF application. Several reasons, such as type of test performed, could be responsible for these conflicting results. Vano and colleagues used microtensile testing to evaluate the bond strength of different composite core buildups and pretreated fiber posts. In our study we used push-out testing, which has been shown to provide a better estimation of bond strength in the root canal than conventional shear tests do. 1 Moreover, the large and complex C-factor (ratio of bonded to nonbonded surfaces) in long. narrow post spaces is a complicating factor resulting in formation of numerous interfacial gaps. 6 Several factors may be associated with the detrimental effect of H<sub>2</sub>O<sub>2</sub> on bond strength; H<sub>2</sub>O<sub>2</sub> is capable of generating the hydroxyl radical, an oxygen-derived free radical, and leaves behind an oxygen-rich surface that inhibits polymerization of resin.<sup>20,21</sup> The formation of such a surface in the narrow space of the root canal and nonreleased stress may have led to the lower bond strength in the root region in the present study.

Voids and air bubbles may prevent proper cementation of the post, thus causing its debonding. D'Arcangelo and others<sup>22</sup> reported that using the Lentulo spiral to apply the luting resin enhances bond strength. In the present study, the resin cement was inserted in the canal with a Lentulo spiral and applied on the post surface simultaneously to reduce voids and increase the displacement resistance of fiber posts.

Thermocycling has been considered an essential aspect of dentin adhesion testing. It has been shown that thermocycling results in a significant decrease in the flexural strength of fiber posts. <sup>23</sup> Purton and others<sup>4</sup> reported no significant differences between thermocycled and non-thermocycled specimens in regards to the forces required to cause post-retention failure. Nevertheless, in the present study thermocycling was used to simulate the clinical condition.

Silanes improve the bond of composite resin to porcelain; however, the use of silanes to enhance bond strength between composite resin and the fiber post is controversial.<sup>23</sup> Silanes provide the chemical bond between the inorganic matrix of luting agent and the organic surface of the fiber post, protect the fibers from damage during handling, and improve the catalytic and wettability characteristics of the fiber post surface. Moreover, they enhance the chemical resistance of the fiber resin interface to hydrolysis. 18 The results of this study showed that silane heat treatment significantly increases bond strength (p < 0.001). Thus, the first null hypothesis can be rejected. Similar results have been reported by Monticelli and others. 15 In their research, the bond strengths of a two-component silane and a onecomponent ethanol-based silane were increased by heat treatment via application of warm air. For optimal adhesion to the fiber post structure, only a monolayer of the silane is required to convert the ceramic surface from an Si-OH to a methacrylate appearance, but usually multiple layers form. 25 A thicker silane interphase may become the weak link of the bond. The innermost layer is cross-linked and provides a strong siloxane bond, whereas the outermost and intermediate layers are only physically adsorbed to the innermost layer and contain many oligomers, which can be easily washed away by organic solvents or water.<sup>26</sup> To provide heat treatment and removal of the weakly bound oligomers, in this study warm water was used to wash the post surface after silane treatment. This technique may produce a bond that is much more hydrolytically stable than if the silane were simply applied and left to dry.

Because of the dependence of core stability to postroot adhesion, the bond strength evaluation at each level of the root seems to be important; therefore, one of the aims of the present study was to compare the bond strength at different levels of the root. It seems that the post retention in the root canal greatly depends on frictional sliding rather than micromechanical and chemical adhesion. 6 In this study, the conical FRC Postec Plus posts were used to eliminate the effect of sliding friction. Although a special reamer with a similar-sized fiber post was used, complete adaptation in the coronal third was impossible. In areas where there is good adaptability between the post and dentin, sliding friction and micromechanical interlocking are important; in areas where the adaptability is low, chemical bonding could be more effective.

This study was performed with the purpose of improving the bond strength between the post and cement by use of different post surface treatments.

In the present study mixed failure was the most frequent type of failure, followed by adhesive failure, between the post and cement. This finding is consistent with the result reported by D'Arcangelo and others, <sup>22</sup> which confirmed that the post-cement interface is weaker than the dentin-cement interface, and special attention should be given to it.

### CONCLUSION

Based on the results of this *in vitro* study, heat treatment of silane had a significant effect on the push-out bond strength. There were no significant differences in the mean bond strength between pretreatment with HF and pretreatment with  $H_2O_2$ .

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### Note

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### **Conflict of Interest**

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

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## Effects of Light Activated In-office Bleaching on Permeability, Microhardness, and Mineral Content of Enamel

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### Clinical Relevance

The use of LED/laser with in-office bleaching gel does not produce severe alterations in dental enamel.

### **SUMMARY**

The aim of this study was to evaluate the permeability (PE), microhardness (KHN), and mineral change in enamel after LED/laser activated in-office bleaching. For PE, the coronal portion of premolars (n=51) was subjected to bleaching with 35% hydrogen peroxide

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(Whiteness HP Maxx, FGM Dental Products, Joinville, SC, Brazil). The samples were stained via the histochemical method, which involves a copper sulphate solution and rubeanic acid. The penetration of dye into the enamel was measured. The KHN of enamel was assessed before treatment, immediately after the bleaching treatment, and again after one week. The calcium and phosphorus content were analyzed with a scanning electron microscope with energy-dispersive X-ray (JSM 6360LV, Jeol Ltd, Tokyo, Japan). The data set from each test was subjected to appropriate parametric statistical analysis ( $\alpha$ =0.05). No significant differences were observed for PE in NLA and LA compared to the control group (p=0.98), as well as for calcium (p=0.16) and phosphorus (p=0.80) content. Significant reduction of KHN after bleaching occurred for both groups (p < 0.001). After immersion in artificial saliva, the KHN of the enamel for all groups was similar to that seen before bleaching. Light activation during in-office bleaching does not produce significant changes in the enamel compared to a non-light-activated technique.

### INTRODUCTION

With a growing awareness of dental esthetic options, there is subsequently a greater demand for cosmetic solutions. Within this context, vital tooth bleaching is one of the cosmetic dental procedures requested most often by patients who want a more pleasant smile. At-home bleaching systems are the most frequently recommended treatment for vital teeth. However, as reported by Marson and others, some patients do not adapt to the technique, mainly because they prefer not to use a bleaching tray or do not like to wait two to three weeks to see the results of their treatment. These patients might request a method that produces more immediate results, such as the in-office bleaching technique.

Since the introduction of in-office bleaching protocols, the use of curing lights has been recommended to accelerate the action of the bleaching gel.<sup>3</sup> The theoretical advantage of a light source is its ability to heat the hydrogen peroxide (HP), thereby increasing the kinetic energy of the molecules and the rate of decomposition of oxygen to form oxygen-free radicals and enhance the bleaching outcome.<sup>4,5</sup>

Even though the body of research is not definitive on the use of light-enhanced bleaching, with many conflicting results having been published, 2,6-14 patients often demand its use due to media coverage. Many clinicians look upon light-activated bleaching as an important factor for patient satisfaction and recognize that many current systems use light activation in the process of tooth whitening.

Thus, it is relevant to compare the side effects of light-activated vs non-light-activated bleaching in order to discover whether the benefits can be said to outweigh the side effects. For instance, it has been consistently reported that light-activated bleaching is associated with increased tooth sensitivity. 9-11,13,14 Although this can be due to significant increases in pulpal temperatures produced by light activation, 15-<sup>18</sup> one cannot rule out the fact that modifications to the enamel structure caused by the association of light activation and HP might be responsible for this increased sensitivity. Therefore, the aim of this study was to evaluate the effects of in-office bleaching associated with light emitting diode (LED)/laser light activation on the permeability, microhardness, and mineral content of enamel.

### **METHODS & MATERIALS**

Seventy-seven caries-free premolars, stored in distilled water and with no coronal cracks or enamel malformations, were randomly selected from a pool

of extracted teeth of unknown origin. The use of extracted human teeth followed a protocol that was reviewed and approved by the local ethics committee of the local university under protocol 82/2009.

### **Enamel Permeability**

Fifty-one premolars were scraped of any remaining soft tissues and polished with a pumice slurry. Subsequently, a 7.0 mm<sup>2</sup> circular area was isolated on the labial surface of each tooth by applying cyanoacrylate resin (Super Bonder, Loctite, São Paulo, SP, Brazil) and three layers of nail varnish (Nati Esmaltes, Nati, Moóca, SP, Brazil) to the remaining surfaces of the tooth. Afterwards, specimens were randomly divided in three groups and kept in distilled water at room temperature until exposure to the bleaching agent.

In the non-light-activated (NLA) group, the 35% HP gel (Whiteness HP Maxx, FGM Dental Products, Joinville, SC, Brazil) with a pH of 6.5 was applied in the exposed enamel area following the manufacturer's directions. The pH of the gel was measured in triplicate with a pH meter (pHmetro Nova Técnica NT-PHM, Piracicaba, SP, Brazil). Three 15-minute applications were performed in each bleaching session. Two consecutive bleaching sessions with no interval were performed. In the light-activated (LA) group, the same procedure was repeated; however, LED/laser energy (Whitening Lase Light Plus, DMC Odontológica, São Carlos, SP, Brazil) was used following the manufacturer's directions. This light source was made of a matrix of LEDs with a wavelength of 470 nm and three infrared laser diodes with a wavelength of 830 nm and a radiant energy of 200 mW. The tooth surfaces were activated for one minute. The device was turned off for two minutes. This procedure was repeated three times for each 15-minute application of the gel. Specimens from the control group were not submitted to any bleaching protocol and were maintained in distilled water until immersion in the dye.

After the aforementioned procedures, specimens of all groups were immersed in 35 mL of 10% copper sulfate aqueous solution and kept in a vacuum (Bomba de Vácuo e Compressor de Ar, Pump37, Exipump, Paulinia, SP, Brazil) for five minutes under 20 psi. This procedure was done to remove air from the dental substrate and allow for the highest dye penetration. Specimens were soaked in 35 mL of the solution for an additional 10 days. After being dried with absorbent paper, specimens were immersed in a 1% dithiooxamide acid alcoholic solution with a pH of 4.6, following the same protocol

as outlined for the copper sulfate solution. Afterwards, specimens were rinsed with distilled water for 15 seconds, dried, and stored in a covered vial containing ammonia vapor for 7 days. Copper ions were revealed by the dithiooxamide acid, resulting in specific colorations that ranged from dark blue to black, depending on the amount of copper ion penetration.<sup>19</sup> Each specimen was embedded in self-curing acrylic resin and sticky wax and three buccolingual tooth sections of 700 µm were cut longitudinally at the middle of the exposed area using a low-speed diamond saw (Isomet 1000, Buehler, Lake Bluff, IL, USA). Slabs were then slightly abraded and polished by hand with silicon carbide abrasive papers of decreasing coarseness up to approximately 300 µm.

The sections were examined at 40× magnification with a light microscope (Olympus model BX 51, Olympus Optical Co, Tokyo, Japan). Digital images of the sections were recorded, and dye penetration along the enamel thickness was evaluated with an image-analyzing system (Image Tool 3.0 software, University of Texas Health Science Center, San Antonio, TX, USA) by one trained evaluator. Permeability was quantitatively analyzed as the percentage of copper ion penetration over the total enamel thickness. Three measurements of dye penetration were performed per tooth. The average of these measurements was the outcome value for each tooth. The data were submitted to a one-way analysis of variance (ANOVA). Multiple comparisons were made utilizing the Tukey test at a significance level of 0.05.

### **Enamel Microhardness**

Twenty human premolars had their roots cut with a diamond saw (Isomet 1000, Buehler) under water cooling. Each tooth crown was embedded in a self-curing methylmethacrylate resin (AutoClear, Dentbras, Pirassununga, SP, Brazil). Enamel buccal surfaces were flattened with wet 600-, 1000-, 1200-, 2000-, and 2200-grit aluminum oxide abrasive papers (3M, Sumaré, SP, Brazil). They were then polished with 1-µm grit and 0.25-µm grit diamond pastes with a polishing machine (Aropol E, Arotec SA, Cotia, SP, Brazil) until a 3-mm long by 3-mm wide flat enamel surface was achieved.

The specimens were kept in artificial saliva (0.0625% KCl, 0.0865% NaCl, 0.0056% MgCl<sub>2</sub>, 0.0166% CaCl<sub>2</sub>, 0.0804% Na<sub>2</sub>HPO<sub>4</sub>, 0.0326% KH<sub>2</sub>PO<sub>4</sub>, 4.274% sorbitol, 0.0004% NaF, 0.1% C<sub>6</sub>H<sub>5</sub>COONa, 2% carboxymethylcellulose, and distilled water)<sup>20</sup> at room temperature for one week and

were then randomly divided into two groups (NLA and LA groups; n=10). These enamel specimens were submitted to the same bleaching procedure described in the enamel-permeability section. Microhardness measurements were taken before initial exposure to the bleaching agents (baseline), immediately posttreatment, and one week after immersion in artificial saliva. Specimens were positioned to record the Knoop hardness (KHN) with a load of 25 g during five seconds in the microhardness tester HMV (HMV2, Shimadzu Co, Tokyo, Japan). Three measurements were performed on each specimen in each evaluation period, and these values were averaged to determine the KHN of each specimen at each evaluation period. The data from the microhardness tester were analyzed by repeated measures via two-way ANOVA and Tukev test at a significance level of 0.05, considering treatment and evaluation time (baseline, after treatment, and one week in artificial saliva) as the main factors. The repeated measure was the evaluation time.

### Mineral Evaluation by Scanning Electron Microscope

Six premolars had their roots removed from the crowns with a diamond saw (Isomet 1000, Buehler) under water cooling. Three buccal to lingual tooth sections were cut longitudinally using a low-speed diamond saw. The fragments were isolated with cyanoacrylate resin (Super Bonder, Loctite) with the exception of the buccal areas, and each fragment was assigned to a different treatment. No bleaching procedure was performed on one fragment (control), while the two other fragments were submitted to LA and NLA bleaching as described in the enamel permeability test. Six fragments from different teeth were used for each group.

Specimens were dried in a desiccator containing colloidal silica for 24 hours at 37°C. Specimens were then mounted on stubs and sputter-coated with a 10-nm gold layer to be analyzed in SEM (JSM 6360LV, Jeol Ltd, Tokyo, Japan) in the secondary electron mode with dispersive X-ray spectrometry energy (EDX). The ratings of EDX quantified the levels of calcium and phosphorus of the enamel surfaces. The levels of calcium and phosphorus were evaluated by a one-way ANOVA and Tukey test for multiple comparisons at a significance level of 0.05.

### **RESULTS**

The means and standard deviations of the dye penetration (%), calcium (%), and phosphorus (%) are reported in Table 1. The one-way ANOVA did not

Table 1: Means and Standard Deviations of Dye Penetration (%), Calcium (%), and Phosphorus (%) in Enamel Surfaces for the Experimental Conditions\*

Groups	Dye Penetration	Calcium	Phosphorus
Control	$9.9 \pm 4.2^{a}$	$21.4 \pm 4.2^{a}$	$14.7 \pm 2.53^a$
NLA	$9.3\pm2.9^{a}$	$24.5 \pm 3.0^{a}$	$14.0 \pm 2.9^{a}$
LA	9.4 ± 2.9 <sup>a</sup>	$24.2 \pm 4.2^{a}$	14.1 ± 2.1 <sup>a</sup>

Abbreviations: LA, light-activated; NLA, non-light-activated.

\* Comparisons are valid only within columns. Identical letters indicate statistically similar means (p>0.05).

show any statistical difference among groups with regard to the dye penetration (p=0.98), calcium (p=0.16), and phosphorus (p=0.80). Representative images of enamel after permeability evaluation are seen in Figure 1.

With regard to enamel microhardness data (Table 2), the two-way ANOVA detected that the interaction treatment vs the evaluation period (p=0.25) and the main factor treatment (p=0.514) were not statistically significant. On the other hand, the main factor evaluation period was significant (p<0.001). Both groups showed reductions in enamel microhardness after bleaching, which recovered to baseline values after 1 week of immersion in artificial saliva (Table 2).

### DISCUSSION

It has been reported that HP and its byproducts can easily pass through enamel and dentin and reach the pulp tissue. This can occur in approximately five to 15 minutes. <sup>21</sup> Further proof of this rapid passage is that dentin changes color next to the pulp as fast as it does next to the dentin-enamel junction. <sup>22</sup> Although there is some acceptance that such fast passage is due to an increase in enamel permeability

Table 2: Means and Standard Deviations of Enamel
Microhardness for the Experimental Conditions

Groups	Baseline (Control)	Posttreatment	One Week After
NLA	$359.5 \pm 35.0^{Aa}$	341.6 ± 25.5 <sup>Bb</sup>	375.2 ± 31.9 <sup>Ac</sup>
ΙA	358.3 + 30.1 <sup>Aa</sup>	319.8 + 53.0 <sup>Bb</sup>	372.5 + 26.6 <sup>Ac</sup>

Abbreviations: LA, light-activated; NLA, non-light-activated.

\* Uppercase letters indicate comparisons within rows. Lowercase letters

produced by the oxidizing HP agent, <sup>19,23</sup> the results of this study do not support such a concept.

We did not detect significant differences in the enamel permeability specimens among the three groups. Although enamel is both extremely hard and dense, it also has a porosity<sup>24</sup> that allows HP penetration. It is likely that HP and its byproducts penetrate into the enamel through these porosities by a diffusion process, similar to what occurs after fluoride application.<sup>25</sup>

So far, only a few studies in the literature have attempted to investigate enamel permeability after bleaching using the histochemical coloring method herein employed. Although earlier studies demonstrated that this methodology was capable of detecting differences between the enamel permeability of bleached and unbleached specimens, the clinical relevance of these results is yet to be addressed.

The slightly acidic pH of the gel employed in this study, around 6.5, may also explain why decreases in the calcium and phosphorus levels were not detected. Although this finding is in agreement with other studies that evaluated mineral content by EDX and Fourier transform infrared spectroscopy, <sup>26,27</sup> it is contrary to others that detected reductions in the mineral levels after bleaching. <sup>28-30</sup> It is likely that

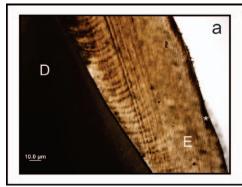






Figure 1. Dye penetration in enamel in (A): control group; (B) light-activated group; and (C) non-light-activated group. (D): Dentin. (E): Enamel. \* represents the penetration of the dye.

<sup>\*</sup> Uppercase letters indicate comparisons within rows. Lowercase letters indicate comparisons within columns. Identical letters indicate statistically similar means (o>0.05).

this controversy may be due to differences in the bleaching protocol, number of sessions, period of evaluation, and bleaching product employed. More pronounced morphologic alterations might be observed if bleaching gels with a lower pH were employed.<sup>31</sup>

With regard to the microhardness measurement, a significant decrease in enamel microhardness was observed immediately after bleaching for both groups. Although literature on this topic is controversial, 32 in vitro studies frequently report drops in enamel microhardness when measurement is done soon after bleaching. 33-35

When enamel microhardness was evaluated one week after immersion in artificial saliva, no significant alteration was observed for either bleached group. The hardness reductions induced by bleaching under *in vitro* conditions usually resolve themselves due to the remineralizing impact of saliva. The closer we get to the oral conditions, the less likely it is that there will be any loss of microhardness. For instance, the only *in vivo* study that evaluated microhardness after bleaching did not report any loss of microhardness.

Despite the fact that the benefits of light activation on bleaching effectiveness are still controversial in the literature, 2,6-14,18 many clinicians still perform this procedure due to media coverage and patient request. Based on the findings of this study, we cannot consider light-activated bleaching to be more detrimental to enamel than the chemical bleaching alone. It seems reasonable to conclude that either bleaching alone or associated with light activation produce similar effects on the enamel surface.

### CONCLUSION

Although the benefit of light-activated bleaching is still doubtful in terms of color change, light-activated bleaching did not increase enamel permeability and did not reduce the mineral content and microhardness of enamel.

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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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