Microtensile Bond Strengths of Composite to Dentin Treated with Desensitizer Products

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Purpose: This study was designed to analyze the influence of desensitizing procedures on dentin bond strength.

Materials and Methods: Forty bovine incisors were used, divided into four groups (n = 10): G1: control; G2: Gluma Desensitizer (Heraeus Kulzer); G3: Oxa-Gel (Art-Dent); G4: low-intensity laser (MMOptics). The buccal surface was wet ground flat with 180-, 400- and 600-grit silicon carbide abrasive paper to expose midcoronal dentin and create a uniform surface. After the application of the desensitizing agents to the exposed dentin, the specimens were etched with 35% phosphoric acid for 30 s, and an adhesive (Single Bond) was applied and light cured. A 4-mm high crown of composite resin (Filtek Z250) was then built up. Specimens were trimmed to an hourglass shape with cross sections of 1 mm². Each specimen was individually fractured by a microtensile testing machine at a crosshead speed of 0.5 mm/min. The data, recorded in MPa, were analyzed with one-way ANOVA and the Duncan test (p = 0.05).

Results: Specimens treated with dentin desensitizers (except Gluma) yielded significantly lower mean bond strengths than nontreated control specimens. The mean values in MPa (±SD) were: G1: 13.4 (6.2); G2: 13.2 (4.8); G3: 7.15 (4.3); G4: 7.21 (4.6).

Conclusions: Among the desensitizing agents studied, only Gluma Desensitizer did not detrimentally influence the bond strength values. It is a useful material for dentin desensitization.

Keywords: dentin, desensitizer, microtensile bond strength.

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Professional interest in the causes and treatment of dentinal hypersensitivity has existed for the past 150 years.²² Dentinal hypersensitivity is a common complaint in adults

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Reprint requests: Ana Cecilia Corrêa Aranha, University of São Paulo, Piracicaba Dental School, Av. Limeira, 901, 13414-900 SP, Brazil. Tel: +55-19-3412-5340, Fax: +55-19-3412-5340. e-mail: acca@usp.br and is one of the most painful and least successfully treated chronic problems of the teeth.⁹ Dentin sensitivity or cervical dentinal sensitivity has been defined as a short and sharp pain arising from exposed dentin, typically in response to chemical, thermal, tactile or osmotic stimuli, which cannot be explained as a result of other forms of dental lesions or pathology.⁸ The pain-producing stimuli can elicit a range of sensations from mild discomfort to extreme pain from a stimulus that normally would be regarded as harmless.^{5,8,9}

The dentin exposure may occur either by removal of enamel or denudation of the root surface by loss of the overlying cementum and periodontal tissues.^{11,13}

Exposure of the root area may have many causes. Chronic trauma from tooth brushing, tooth flexure due to occlusal loading, parafunctional habits, acute and chronic inflammatory gingival and periodontal diseases, periodontal surgery, and acidic dietary components are commonly cited as major causes of gingival recession that will probably lead to a cervical lesion.^{1,14}

Table 1	The desensitizing agents,	composition, and	I manufacturers
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Table 1 The desensitizing agents, composition, and manufacturers			e. rbehalt
Group	Product	Composition	Manufacturer
G2	Gluma Desensitizer	361 mg (35%) HEMA 51 mg (5%) glutaraldehyde	Heraeus Kulzer
G3	Oxa-Gel	3% monohydrogen-monopotassium oxalate	Art-Dent
G4	Low-intensity laser therapy (LILT)	red beam (660 nm)/ 3.8 J/cm ² for 10 s/ Gallium Aluminum Arsenide (GaAlAs)	MMOptics

According to Brännström's² well-accepted hydrodynamic theory, it is possible to make the fluid inside the dentinal tubule move and consequently stimulate mechanoreceptors at the pulp-dentin interface, causing the sensation of pain. Thus, the concept of tubular occlusion as a method of dentin desensitization is a logical conclusion of the hydrodynamic theory.²

The fact that many of the agents used clinically to desensitize dentin are also effective in reducing dentin permeability tends to support the hydrodynamic theory. The most commonly used agents in the treatment of dentin sensitivity can be broadly classified by their modes of action: anti-inflammatory drugs, protein precipitants, tubule occluding agents, tubule sealants, and recently, laser treatment.13

There are controversies with regard to the restoration of noncarious cervical lesions. A restoration may be necessary if the integrity of the tooth is threatened, the pulp is exposed, the location of the lesion makes it difficult to design a partial denture, the defect is esthetically inacceptable to the patient, or in order to decrease stress and strengthen the tooth.^{1,3,12} In these particular cases, the restoration of the cervical lesion must be a continuation of the dentinal tissue to avoid injuries to the periodontal tissue.

A question which intrigues researchers and clinicians is: after one week of applying desensitizing products without achieving any substantial result, what should be done to reduce pain in cervical lesions? One plausible option is to restore the exposed dentin surface, but what would happen to the bond strength values? Is it necessary to first remove the desensitizer remaining on the dentin surface with a diamond bur?

The purpose of this study was to examine the bond strength of a composite resin built up on surfaces that had first been treated with different desensitizing agents.

MATERIALS AND METHODS

Forty freshly extracted bovine incisors were collected, cleaned, and stored in an isotonic saline solution containing 0.3% sodium azide. The roots were removed from the crown approximately 4 mm apical to the cementoenamel junction using a slow-speed diamond saw under water spray. The buccal surface of each tooth was wet-ground flat in a mechanical grinder with 180-, 400-, and 600-grit silicon carbide abrasive paper in order to expose midcoronal dentin and create a uniform dentin surface for bonding. The specimens were randomly assigned to 4 groups, corresponding to the desensitizing products to be applied (n = 10): G1: control, no desensitizer was applied; G2: Gluma Desensitizer (Heraeus Kulzer, South Bend, IN, USA); G3: Oxa-Gel (Art-Dent Com. e Prod. Odontológicos, Araraquara, SP, Brazil); G4: low-intensity laser (MMOptics, São Carlos, SP, Brazil). The area upon which the desensitizers were applied was delimited as 7 mm long and 7 mm wide on the buccal surface. The desensitizing agents used in this study are described in Table 1.

Application of Desensitizers

G1, control

Control specimens were not treated and remained in distilled water during the period in which the desensitizing agents were applied to the other groups.

G2. Gluma Desensitizer

Gluma Desensitizer was applied with cotton pellets using a gentle but firm rubbing motion and left for 30 s to dry thoroughly.

G3. Oxa-Gel

Oxa-Gel agent, a gel of 3% monohydrogen-monopotassium oxalate, was applied for 2 min and then dried carefully just to remove the excess.

G4. low-intensity laser therapy (LILT)

The equipment used was a 660-nm diode, low-intensity laser system, with an energy density 3.8 J/cm², applied for 10 s in contact mode. The application was repeated three times, with an interval of 72 h between applications, according to the dentin hypersensitivity protocol of LELO (Special Laboratory of Lasers in Dentistry) at the University of São Paulo.

After the desensitizing procedures, specimens were stored in distilled water for 1 week at 37°C until the restoration procedure.

Restoration Procedure

One week after the desensitizing agents were applied, the dentin surface was acid etched for 30 s (35% phosphoric acid, 3M/ESPE, St Paul, MN, USA) and rinsed for the same time, then gently air thinned without desiccating the dentin. After acid etching, the adhesive (Single Bond, 3M/ESPE) was applied with a brush tip and polymerized for 10 s. A 4-mm high crown of composite resin (Filtek Z250, 3M/ESPE) was built up on the bonded surface in three increments, each one approximately 1.30 ± 0.3 mm thick and polymerized for 40 s (Optilux 501, Demetron, Danbury, CT, USA). The light was tested for light output before each use with a Demetron radiometer (model 100). After restoration, specimens were stored in distilled water at 37°C.

Microtensile Bond Test

The restored teeth were placed in a low-speed sectioning machine (Buehler Isomet 100; Buehler, Lake Bluff, IL, USA) and serial slices 1 mm thick were made with a diamond saw, perpendicular to the bonded surface. Using a fine finishing diamond bur #1099 FF (KG Sorensen, Barueri, SP, Brazil) in a high-speed handpiece under air/ water spray coolant, the slices were trimmed to an hourglass shape to enable uniform stressing of the smallest cross-sectional area (0.8 to 1.0 mm²). The cross-sectional interface area of each specimen was measured using a digital caliper (Mahr, Esslingen, Germany). From each tooth, 6 slices were obtained, but the 2 peripheral slices were excluded, totaling 40 specimens per group.

Specimens were then glued to an acrylic grip with a cyanocrylate adhesive (Super Bonder, Henckel Loctite, São Paulo, SP, Brazil). The grip with the specimen was then fitted into an MT jig (Fig 1), which was held in a universal testing machine (EMIC, São José dos Pinhais, PR, Brazil). A tensile force exerted by a load cell of 100 N at a cross-head speed of 0.5 mm/mim was applied. The microtensile bond strength was calculated and expressed in MPa.

After the microtensile bond strength test, each specimen was observed using a light stereoscope (Meiji 2000, Meiji Techno, Saitama, Japan) at 35X magnification to observe the type of fracture (cohesive or adhesive).

Statistical Analysis

The means of each group were calculated and the data were subjected to one-way ANOVA. The Multiple Comparison Duncan test was chosen for observing differences among groups (p = 0.05).

Scanning Electron Microscope (SEM) Examination

Three additional samples were prepared for SEM examination to analyze the morphological pattern of the hybrid layer in specimens treated with desensitizing agents prior to restoration. The specimens were immersed in 0.5 M hydrochloric acid for 30 s to demineralize the dentin and then washed carefully with tap water for the same length of time. Afterwards, the samples were immersed in 1% sodium hypochlorite to remove the collagen network and expose the hybrid layer. These samples were glued to aluminum stubs and sputter coated with gold. Samples were observed in a DSM 900 SEM (Zeiss, Jena, Germany).



Aranha et

Fig 1 The MT jig holding an hourglass-shaped specimen in the universal testing machine (EMIC).

RESULTS

One-way ANOVA detected differences in bond strength values and the multiple comparison Duncan test showed statistical differences among the groups (p = 0.05). Gluma Desensitizer did not interfere in the bond strength or formation of a hybrid layer. On the other hand, Oxa-Gel and LILT resulted in significantly lower bond strengths than Gluma and the control group. The mean values and standard deviations are shown in Table 2. Analyzing the type of fracture, it could be seen under the light stereoscope that 92% of the failures were adhesive, whereas 8% of the samples failed cohesively. Considering the groups separately, 86.3% of the control specimens showed adhesive failure, and cohesive failure was found in 13.6% of the specimens. For the Gluma group, 90% of the failures were adhesive and 10% cohesive. Analyzing the Oxa-Gel samples, all failures were adhesive; in the LILT group, 94.4% of failures were adhesive and 5.6% were cohesive. Thus, adhesive failures were predominant for all groups tested.

Scanning electron microscopic images of the additional samples are depicted in Figs 2 to 5. The SEM images of the tooth/restoration interface for the control group show the formation of a hybrid layer (Fig 2). After the application of Gluma Desensitizer, the scanning electron micrograph (Fig 3) shows an interface similar to that found in the control group. However, samples treated with Oxa-Gel (Fig 4) display a gap between the dentin and the restoration which is larger than that found in the Gluma Desensitizer and the control groups. In laser-irradiated samples (Fig 5), the quality of the hybrid layer formed is similar to that of the Gluma and control groups.

DISCUSSION

The method developed by Sano et al²³ for testing the microtensile bond strength of dentin and enamel has a number of potential advantages: higher interfacial bond

Aranha et al

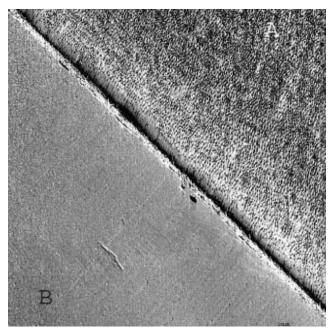


Fig 2 A representative scanning electron micrograph (100X) of the tooth/restoration interface from the control group. Note the formation of a hybrid layer (A = dentin; B = composite resin).

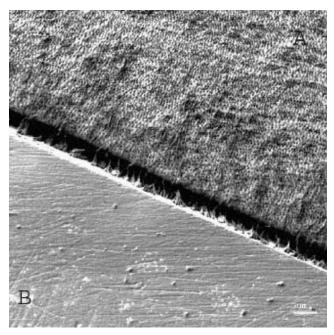


Fig 4 SE micrograph (100X) showing the interface of tooth/ restoration after the application of 3% monohydrogen-monopotassium (Oxa-gel) before the restoration. Note that the gap between dentin and restoration is bigger than the one shown for Gluma Desensitizer and the control group, even though the hybrid layer is visible (A = dentin; B = composite resin).

strengths can be measured, it is possible to determine regional bond strengths, means and variances can be calculated for single teeth, it permits testing of bonds to irregular surfaces and very small areas, and finally, it facilitates ex-

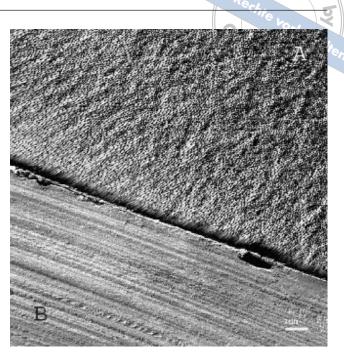


Fig 3 SE micrograph (100X) showing the interface of tooth/ restoration after the application of Gluma Desensitizer (A = dentin; B = composite resin).

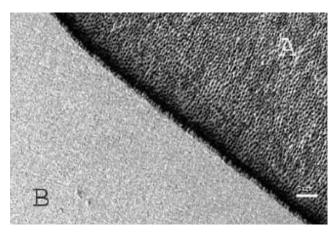


Fig 5 SE micrograph (100X) showing the interface of tooth/restoration after LILT. Note the quality of the hybrid layer formed (A = dentin; B = composite resin).

Table 2	Bond strength	means and	standard	deviations
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Groups	G1 (Control)	G2 (Gluma)	G3 (Oxa-Gel)	G4 (LILT)
Mean values (MPa)	13.4ª	13.2ª	7.15 ^b	7.21 ^b
Standard deviations	6.2	4.8	4.3	4.6

Same superscript letters indicate statistically equal means.

amination of the failed bond by scanning electron microscopy.^{16,17} Another advantage of the microtensile test is that it yields more adhesive than cohesive failures, as seen in this study, where 92% of the failures were adhe-

sive and 8% were cohesive. Considering the groups separately, adhesive failures were predominant in all groups tested. However, adhesive failure does not always indicate good bonding performance. Recent systems with good adhesive performance tend to show cohesive failure in the adhesives if high microtensile bond strengths are obtained. Since some authors recommend this method,^{16,23} it was chosen to test the effect of desensitizing agents applied to treat dentinal/cervical hypersensitivity before the restoration was placed. Desensitizing agents have also been used as a method for preventing postoperative sensitivity after the restoration procedure.⁶

The clinical management of dentin hypersensitivity on an exposed dentin surface and postoperative sensitivity after restoration with composite resin has long been a challenge to clinicians. The characteristic brief, sharp pain that occurs is explained by the indirect excitation of intradental nerves due to fluid shifts within exposed dentin tubules.^{2,8} Based on this mechanism, a number of products and procedures have been advocated, but none of them has proved to be completely effective.13 Even with the improvement of adhesive materials, restoring a cervical lesion is a procedure that requires great care. Deficient margins and loss of material are commonly observed in cervical restorations.¹² In restorative dentistry, the application of desensitizing agents has traditionally been preferred to the restoration of such lesions. However, in particular cases, the restoration of cervical lesions may be necessary, for example, in cases of recurrent sensitivity. On the other hand, the advent of adhesive dentistry has already caused profound changes in dental practices, and the development of new dentin adhesive systems and new composite resins is likely to result in Class V restorations with better marginal adaptation.^{12,20,23}.

In the present study, the period of one week was chosen, because this is the period in which patients usually return to the office, complaining of pain. In addition, it is not recommendable to restore a surface that has just been treated with a desensitizing agent.

Previous studies indicated that adhesive resins do not bond well to oxalate-treated dentin because the dentin surface, including the tubule orifices, is covered with calcium oxalate crystals. These crystals are a result of the reaction between the potassium oxalate and the ionized calcium in the dentin fluid or on dentin.¹⁹ Pashley et al¹⁶ conducted a study to test the hypothesis that the application of a combined technique of a potassium oxalate gel and adhesive agents on exposed dentin surfaces would be effective in reducing its permeability and would not undermine bond strength. SEM images showed that the application of potassium oxalate gel resulted in the formation of crystals inside the tubules rather than on the surface. Examination of the bonded interfaces demonstrated that the crystals inside the tubule allow the formation of a typical hybrid layer on top of dentin surfaces. Resin monomers penetrated into the tubules, filling the spaces around the crystals, forming resin tags with jagged features. The authors concluded that the application of a potassium oxalate gel to exposed dentin prior to an adhesive restoration may be a useful method.

In the study by Pashley et al,¹⁶ the surface was etched with 32% phosphoric acid, allowing the entrance of the oxalate into the dentinal tubules. In this study, the surface was not acid etched before the application of the potassium oxalate as the manufacturer recommends. The acid etching was only conducted after the application of the desensitizer as part of the restoration procedure. Nevertheless, the formation of a hybrid layer (Fig 2) could be seen in SEM images. The acid etching procedure removes the smear layer and depletes the crystals of calcium oxalate from the dentin surface, allowing the formation of a hybrid layer. On the other hand, the deposition of calcium oxalate crystals on the dentinal surface could neutralize the etching procedures and consequently might inhibit the formation of a sufficient hybrid layer, interfering with the subsequent bonding procedure. Pashley et al¹⁶ recommend acid etching before and after the application of the oxalate gel to deplete calcium from the top surface of the dentin. According to the authors, the oxalate products tend to migrate into the tubules until calcium is available to form the calcium oxalate crystals. If calcium oxalate crystals are formed below the surface, they should not interfere with the subsequent bonding procedure and yet provide tubule occlusion in association with the dentin bonding agent. The current study shows the need for depleting the surface of all calcium oxalate crystals by cleaning it or even acid etching it, as the values for the potassium oxalate desensitizer, Oxa-gel, were statistically lower than those for the control group.

Recently, Tay et al²⁶ showed that microtensile bond strengths (μ TBS) were significantly lower compared with the control when oxalates were used before the specimens were acid etched; in contrast, when oxalates were used after acid-etching, μ TBS were similar to controls not treated with oxalate. They hypothesize that effective tubular occlusion and dentin bonding may be simultaneously achieved by depleting dentin surfaces of calcium with acids before desensitizer application.

Gluma Desensitizer is applied in a simple, one-step procedure for treating and also preventing dentinal hypersensitivity.⁴ This product is identical to the third step (primer) of the dentin bonding agent, Gluma Bond. The manufacturers state that the Gluma-patented glutaraldehyde/ HEMA formula acts in seconds to seal dentinal tubules. preventing the fluid shifting recognized as the primary cause of localized hypersensitivity. Clinically, Gluma Desensitizer may be successful in reducing or eliminating dentin sensitivity due to the fixative effect of 5% glutaraldehyde on the superficial odontoblastic layer, resulting from precipitation of plasma proteins.⁴ Glutaraldehyde has long been used as a fixative and sterilizing agent.⁹ According to some studies, HEMA, one of the components of Gluma Desensitizer, well known for its water solubility, may promote deep penetration of the glutaraldehyde component into the tubules.^{10,24}

The satisfactory results achieved with Gluma desensitizing agent occurred because the product is normally recommended for use under restorations to reduce postoperative sensitivity, after the dentinal smear layer has been removed and before cementation procedures; it has not been found to affect bond strength values of adhesive systems.^{7,25} As seen in Fig 3, no gap at the dentin/restoration interface can be seen when Gluma Desensitizer was used, similar to the control specimen.

According to the manufacturer's instructions, Gluma Desensitizer does not require removal of smear layer in order to desensitize, but most dentin bonding agents require removal in order to achieve a hybrid layer. As there were no statistical differences between the control group and the Gluma Desensitizer group, this material is recommended for application under composite resin restorations to prevent postoperative sensitivity and to protect the exposed dentinal surface against dentin hypersensitivity.

Olsen¹⁵ proposed that low-intensity laser therapy exerts its effect by stimulating the sodium/potassium pump in cell membranes, which maintains the potential difference across the membrane. Stimulation of the pump is thought to hyperpolarize the membrane, thus increasing the pain threshold. The irradiation with low intensity laser does not alter the morphology of the surface, in contrast to high-intensity lasers which ablate and modify the dentinal structure. The exact action mechanism of low-intensity laser in dentin hypersensitivity is not thoroughly understood.15,21 Further research needs to be conducted. The findings of this study indicate that low-intensity laser therapy should be used in cases of pain and can be used adjunct to other therapies (Fig 5). However, in view of the lower bond strength values achieved in this study, further analysis should be carried out

In conclusion, this study provides the clinician with information about desensitizing agents applied one week prior to restorations on exposed dentin surfaces. Among the agents studied, Gluma Desensitizer showed good bond strength values similar to those showed by the control group. Oxa-Gel and LILT resulted in lower bond strength values which did not differ statistically significantly between them. The results suggest that some desensitizers may have a negative effect on dentin bonding strengths if not chosen properly. The type of restoration should be considered before the application of a desensitizer.

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Clinical relevance: Dentin desensitizers should be correctly chosen in order not to affect the quality of the restoration.

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