Eroded dentin does not jeopardize the bond strength of adhesive restorative materials

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Abstract: This *in vitro* study evaluated the bond strength of adhesive restorative materials to sound and eroded dentin. Thirty-six bovine incisors were embedded in acrylic resin and ground to obtain flat buccal dentin surfaces. Specimens were randomly allocated in 2 groups: sound dentin (immersion in artificial saliva) and eroded dentin (pH cycling model - 3× / cola drink for 7 days). Specimens were then reassigned according to restorative material: glass ionomer cement (Ketac™ Molar Easy Mix), resin-modified glass ionomer cement (Vitremer™) or adhesive system with resin composite (Adper Single Bond 2 + Filtek Z250). Polyethylene tubes with an internal diameter of 0.76 mm were placed over the dentin and filled with the material. The microshear bond test was performed after 24 h of water storage at 37°C. The failure mode was evaluated using a stereomicroscope (400×). Bond strength data were analyzed with two-way ANOVA and Tukey's *post hoc* tests ($\alpha = 0.05$). Eroded dentin showed bond strength values similar to those for sound dentin for all materials. The adhesive system showed the highest bond strength values, regardless of the substrate ($p < 0.0001$). For all groups, the adhesive/mixed failure prevailed. In conclusion, adhesive materials may be used in eroded dentin without jeopardizing the bonding quality. It is preferable to use an etch-and-rinse adhesive system because it shows the highest bond strength values compared with the glass ionomer cements tested.

Descriptors: Tooth Erosion; Dental Materials; Shear Strength; Dentin.

Introduction

An increase in erosive tooth wear[1,2] associated with a decline in dental caries incidence[3] has attracted research interest to this field. Lifestyle changes and the consumption of soft drinks / acidic foods seem to be the factors responsible for this condition.[4,5] In the initial stages, the erosion lesions are limited to the enamel; however, dentin exposure can also occur as the lesions progress.[6] In such cases, adhesive restorative materials, such as glass ionomer cements and resin composites, are able to reestablish tooth contour, function and aesthetics, and protect the exposed dentin.[7]

Several studies have evaluated the modification that occurs on the surface of restorative materials after an erosive challenge. Deleterious effects have been documented in properties such as hardness,[8] wear depth,[9,10] morphology[11] and surface roughness.[12] However, there is a lack of studies

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focusing on the bond strength properties of dental materials applied to eroded dentin.

The erosive challenge leads to greater wear[13] and reduction in hardness[8] of the dentin substrate with the formation of spatial areas with damaged apatite that exhibit local structural alteration, namely broken and/or loosened P-O--Ca atomic linkages.[14] The lower hydroxyapatite and calcium content is expected to interfere with the bond strength of glass ionomer cements due to their mechanism of action. Alterations such as dentinal tubule opening, removal of dentinal plugs and the organic portion of intertubular dentin, that increase the tubules' diameter and cause collagen exposure, have also been reported.[15]

Because the erosion challenge causes alterations in dentin, we hypothesize that restorative materials may act differently in sound and eroded substrates. However, to the best of our knowledge, no previous study has investigated the bond strength of adhesive materials to eroded substrates. While a number of studies focus on erosive tooth wear prevalence,[1,2] few seek information about their treatment. Therefore, the aim of this study was to evaluate the bond strength of adhesive restorative materials to sound and eroded dentin.

Methodology

Tooth selection and preparation

Thirty-six bovine incisors stored at 4°C were used in this study. The roots were removed using a low-speed diamond disc in a cutting machine (Labcut 1010, Extec Co., Enfield, USA), and crowns were embedded in self-curing acrylic resin inside PVC rings (JET Clássico®, São Paulo, Brazil). The exposed buccal surfaces were ground under water with 320 grit SiC paper to obtain a flat dentin surface and further polished with 600 grit SiC paper for 60 s to create a standardized smear layer.[16]

Erosive Challenge

Specimens were randomly allocated into 2 groups:

- (1) immersion in artificial saliva during the experimental period (control group - sound dentin, n = 18);
- (2) erosion challenge according to a pH-cycling model (eroded dentin, n = 18).

Three pH-cycles were performed at 8, 14 and 20 hours for seven days. Teeth were immersed in a cola drink (Coca-Cola, [pH: 2.6, phosphate: 5.43 mM Pi, calcium: 0.84 mM Ca^{2+}, fluoride: 0.13 ppm F, titratable acid: 40.0 mmol/L OH^- to pH 5.5 and 83.6 mmol/L OH^- to pH 7.0], Spal, Porto Real, Brazil) for 5 min (30 mL per tooth) and were kept in artificial saliva (1.5 mmol L^{-1} $Ca[NO_3]_2.4H_2O$, 0.9 mmol L^{-1} $NaH_2PO_4.2\ H_2O$, 150 mmol L^{-1} KCl, 0.1 mol L^{-1} Tris buffer, 0.03 ppm F, pH 7.0, 30 mL per tooth) between erosive cycles, under agitation and at room temperature. During the remaining time, teeth were also kept in artificial saliva.[9]

Restorative procedures

Teeth from each dentin substrate (sound or eroded) were randomly reassigned into 3 subgroups according to adhesive restorative material used:

- glass ionomer cement - GIC (Ketac™ Molar Easy Mix),
- resin-modified GIC (Vitremer™) or
- etch-and-rinse adhesive system associated with resin composite (Adper Single Bond 2 + Filtek Z250).

This resulted in a 2 × 3 factorial experimental design with 6 teeth in each subgroup formed from the crossing of two factors:

- substrate and
- material.

Table 1 displays the components and application mode of adhesive restorative materials used in the experiment.

After surface pretreatment, polyethylene tubes (Micro-bore®Tygon S-54-HL Medical Tubing, Saint-Gobain Performance Plastics, Akron, USA) with an internal diameter of 0.76 mm and a 1.0-mm height were filled with one of the restorative materials and placed on the bonded area. Tubes were covered with a matrix strip and gently pressed with a glass slide. For each tooth, 3 specimens were built up. Surface protection was performed for glass ionomer cement according to the manufacturer's instructions. For

Table 1 - Adhesive materials: manufacturer, composition, application mode and surface protection.

Material and manufacturer	Composition	Application mode	Surface protection
Ketac™ Molar Easy Mix (3M ESPE, Seefeld, Germany)	**Ketac™ Conditioner:** polyacrylic acid (25%) **Powder:** calcium aluminum-lanthanum-fluorosilicate glass, acrylic acid-maleic acid copolymer, pigments **Liquid:** water, acrylic acid-maleic acid copolymer, tartaric acid	**(1)** Apply Ketac Conditioner for 10 s; **(2)** rinse with a copious amount of water; **(3)** gently air-dry (5 s), leaving a moist surface; **(4)** dose 1 drop of liquid and 1 powder scoop, mix up to 30 s; **(5)** apply to dentin surface	Solid petroleum jelly
Vitremer™ (3M ESPE, Seefeld, Germany)	**Primer:** Vitrebond copolymer, HEMA, ethanol and photoinitiators **Powder:** fluoroaluminosilicate glass, potassium persulfate and ascorbic acid **Liquid:** polyalkenoic acid, HEMA, dimethacrylate, photoinitiator, water	**(1)** Apply primer for 30 s; **(2)** gently air-dry with syringe; **(3)** light-cure for 20 s; **(4)** dose 1 drop of liquid and 1 powder scoop, mix up to 45 s; **(5)** apply to dentin surface; **(6)** light-cure for 40 s	Finishing gloss
Adper Single Bond 2 (St. Paul, USA)	HEMA, water, ethanol, Bis-GMA, dimethacrylates, amines, methacrylate functional copolymer of polyacrylic and polyitaconic acids, 10% by weight of 5 nanometer-diameter spherical silica particles	**(1)** Etch for 15 s; **(2)** rinse with water spray for 15 s, leaving tooth moist; **(3)** active application of two consecutive coats of the adhesive with a fully saturated brush tip, dry gently for 2-5 s; **(4)** light-cure for 10 s; **(5)** apply one increment of composite resin and light-cure for 20 s	Not recommended
Filtek Z 250™ (St. Paul, USA)	Bis-GMA, TEG-DMA, zirconia, silica		

HEMA: 2-hydroxyethylmethacrylate; Bis-GMA: bis-phenol A diglycidyl methacrylate; TEG-DMA: triethylene glycol dimethacrylate.

the light-cured materials, halogen light curing was used (Jetlite 4000 Plus, J. Morita USA Inc., USA) with a 600 mW/cm² power density.

After storage in distilled water at 37°C for 24 h, the polyethylene tubes were removed using a surgical blade, resulting in cylindrical specimens with a cross-sectional area of approximately 0.45 mm². Specimens were examined under a stereomicroscope at 20× magnification, and those with interfacial gaps, bubble inclusion or other defects were excluded and replaced.

Microshear bond strength test (μSBS)

The specimens were then attached to the microshear bond universal testing machine (Kratos Industrial Equipment, Cotia, SP, Brazil), and a shear load was applied with a thin steel wire (0.20 mm diameter) at a crosshead speed of 1.0 mm/min until failure. Care was taken to keep the restorative material cylinder in line with the center of the load cell and the wire loop parallel to the load cell movement direction and to the bonding interface. The microshear bond strength was calculated and expressed in MPa.

Failure mode

The failure mode was determined with a stereomicroscope with 400× magnification (Discovery V20, Zeiss, Oberkochen, Germany) and classified as adhesive/mixed failure (presence of dentin or resin composite/GIC adjacent to interface) or cohesive failure (failure in dentin or resin composite/GIC).

Statistical analysis

The experimental unit in the current study was the tooth. Thus, the mean μSBS values of all specimens from the same tooth were averaged for statistical analysis. A normal data distribution and an equality of variances were assumed after Kolmogorov-Smirnov and Barlett's tests. The μSBS means were analyzed by two-way ANOVA and Tukey *post hoc* test. The significance level was set at $p < 0.05$. All statistical analyses were performed using Minitab 16 software (Minitab Inc., State College, USA).

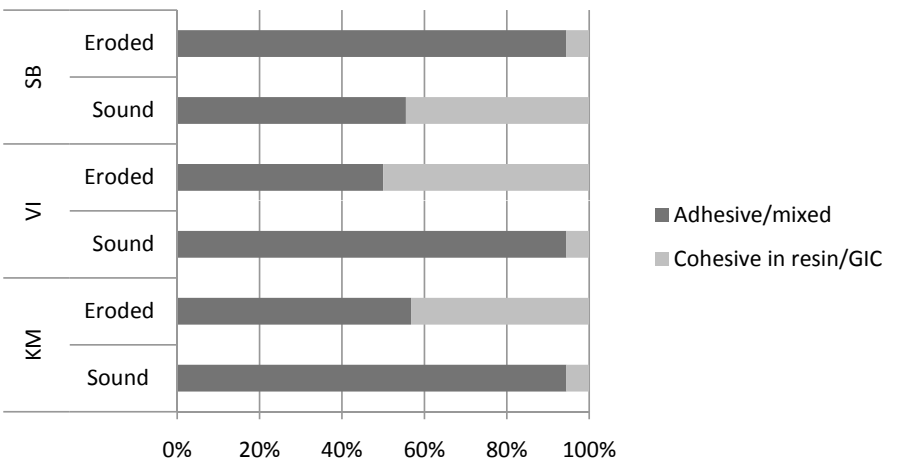

Figure 1 - Distribution (%) of failure mode for experimental groups (SB: Adper Single Bond 2; VI: Vitremer™; KM: Ketac™ Molar Easy Mix).

Results

The microshear bond strength means (MPa) and standard deviations for all experimental groups are presented in Table 2. Only the main factor (adhesive material) was statistically significant ($p < 0.0001$). The adhesive system showed the highest bond strength values, regardless of the substrate. No difference in bond strength was found between eroded or sound dentin using any of the materials.

The distribution of the failure mode is summarized in Figure 1. For all groups, adhesive/mixed failure prevailed. No cohesive failure in dentin was observed.

Table 2 - Microshear bond strength means and standard deviations (MPa) for the experimental groups.

Substrate	Material		
	Vitremer	Adper Single Bond 2	Ketac Molar Easy Mix
Sound dentin	13.9 ± 7.1[b]	17.1 ± 3.4[a]	7.5 ± 1.4[b]
Eroded dentin	9.4 ± 6.3[b]	17.5 ± 4.2[a]	9.8 ± 3.0[b]

Different superscript letters indicate statistically significant differences ($p < 0.05$).

Discussion

Increased erosion prevalence has been clinically observed, particularly in enamel.[1,2] Although deep lesions are less common, without controlling etiological factors, they can extend to dentin. Such situations require a restorative procedure, and it is important to know the performance of adhesive restorative materials in this type of substrate.

To simulate dental erosion, dynamic erosive pH-cycling using a cola drink was employed. This beverage has a high erosive potential due to its low pH and low fluoride/calcium concentrations.[4] Although *in vitro* models are unable to thoroughly simulate the oral environment, especially with respect to important aspects of the erosion process, such as salivary flow, pellicle formation and buffering capacity,[17] the adopted protocol simulates a typical intake of individuals considered to be at risk for dental erosion.[13]

The replacement of human teeth with bovine dental hard tissues has been recommended, especially for studies that evaluate bonding mechanisms.[18,19] Schilke *et al.*[20] showed that human and bovine dentin present similar characteristics when analyzed by scanning electron microscopy, such as number and diameter of tubules per mm² and presence of a collagen matrix. Moreover, bovine teeth are easier to obtain in large numbers in good condition, and they present less variation in composition.[21] Bovine dentin can be a suitable substitute for human dentin in bonding tests. For these reasons, bovine dentin has been chosen for the present study.

Furthermore, the microshear test was used to evaluate bond strength because the literature affirms that the "micro" tests are the best at verifying the performance of contemporaneous materials due to the high bond strength values, which are not possible to measure with precision in "macro" tests (such as shear and tensile bond strength tests).[22] The microshear bond strength test showed some advan-

tages—a reduced demand during the specimen preparation, the standardization of bond test areas by the use of a tube with a known diameter and the use of fewer teeth to perform the study—all of which justify its choice.[23]

The erosion process can lead to the removal of dentinal plugs and organic intertubular dentin, resulting in an increase in tubule diameter and collagen exposure.[15] These differences between sound and eroded dentin might interfere with the bonding properties of adhesive materials. In spite of that, this difference in performance was not observed in the current study. Eroded dentin showed similar bond strength values compared with sound dentin. It is speculated that the adhesive materials were able to promote complete micromechanical interlocking in a similar way for both substrates, even when a greater degree of demineralization had occurred, as in the case of the eroded dentin.

The adhesive system showed better bonding performance to dentin, corroborating a previous study,[24] and this difference was noted for both substrates. This can be attributed to the mechanism of the etch-and-rinse adhesive system, which results in smear layer removal, allowing the resin to penetrate the tubules and to infiltrate the underlying demineralized dentin.[25] It is also known that the bonding mechanism in dentin is carried out by hybrid layer formation, which is composed of residual hydroxyapatite, resin and collagen. After etching, the resin monomers penetrate the water-filled spaces between dentin collagen fibers that used to be filled with hydroxyapatite crystals. In terms of the dentin substrate, it is hypothesized that the mineral content may be not as essential as it is for the enamel when using an etch-and-rinse strategy.

The bonding performance of the resin-modified GIC was similar to that of the conventional GIC, and the erosive process also did not interfere with the bond strength. Despite the presence of resin monomers that may enhance physical properties, the bonding mechanism for resin-modified GIC does not differ much from that for the conventional GIC. The former still presents the chemical adhesion to dental structures that is unique to glass ionomer cements. Some studies demonstrated higher bond strengths values for the resin-modified GIC and have associated this finding to the use of an acidic primer.[26,27] This component is able to modify the smear layer and wet the dental surface to improve the interaction between the material and the substrate. However, this result was not observed in the current study, possibly because of the prior application of polyacrylic acid to the dentin substrate before the use of the conventional GIC. This step is recommended to increase the bond strength because it promotes a superficial cleaning, which enhances the micromechanical and chemical interaction between the GIC and the hydroxyapatite.[28]

Although the higher demineralization of eroded dentin due to the lower hydroxyapatite and calcium content did not negatively influence the bond strength of glass ionomer cements, this aspect may be related to changes in the failure pattern. A higher percentage of cohesive GIC failure was observed in the eroded substrate. The reasons are not clear, but some reports suggest that there was no correlation between the cohesive material failure and the bond strength.[29] It was suggested that the failure mode was affected by the material properties of all components of the bonded joint—i.e., GIC, hybrid-like layer and dentin—and the mechanics of the test assembly. Thus, the eroded dentin, softer and more porous than sound dentin and associated with the presence of possible air bubbles in GIC that can act as stress points, increased the likelihood of cohesive fracture within the cement.[24]

Conversely, in this study, few cohesive fractures in GICs in sound dentin were observed, in contrast with a previous study[24] that used a microtensile test. The microshear bond test does not require cutting procedures during the specimens' preparation phase; the cutting may cause damage to brittle materials and can result in fractures prior to adhesive failure.[30]

Furthermore, because the chemical interaction might be beneficial in reducing the hydrolytic degradation and thus enhancing the restoration longevity, further studies should be conducted to obtain a better understanding of the durability of the bond to eroded dentin to find a reliable correlation with clinical situations.

Conclusion

Adhesive materials may be used in eroded dentin without jeopardizing the bonding quality. It is preferable to use an etch-and-rinse adhesive system because it shows the highest bond strength values compared with the glass ionomer cements tested.

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