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ORIGINAL RESEARCH

Shear Bond Strength of Two Adhesive Materials to Eroded Enamel

Tathiane Lenzi, Daniela Hesse, Camila Guglielmi, Ketlin Anacleto, Daniela Prócida Raggio

ABSTRACT

Aim: To evaluate the bond strength of one etch-and-rinse adhesive system and one resin-modified glass ionomer cement to sound and eroded enamel.

Materials and methods: Forty-eight bovine incisors were embedded in acrylic resin and ground to obtain flat buccal enamel surfaces. Half of the specimens were submitted to erosion challenge with pH-cycling model (3x/cola drink for 7 days) to induce eroded enamel. After that, all specimens were randomly assigned according to adhesive material: etch-and-rinse adhesive system (Adper Single Bond 2 – 3M ESPE, USA) or resin-modified glass ionomer cement (Vitro Fil LC – DFL, Brazil). The shear bond testing was performed after 24 hours water storage (0.5 mm/min). Shear bond strength means were analyzed by two-way ANOVA and Tukey post hoc tests ($p < 0.05$).

Results: Adper Single Bond 2 showed the highest bond strength value to eroded enamel ($p < 0.05$), whereas no difference was observed in sound enamel compared with Vitro Fil LC ($p > 0.05$).

Conclusion: Bond strength of etch-and-rinse adhesive system increases in eroded enamel, while no difference is verified to resin-modified glass ionomer cement.

Clinical significance: Adhesive materials may be used in eroded enamel without jeopardizing the bonding quality; however it is preferable to use etch-and-rinse adhesive system.

Keywords: Tooth erosion, Shear bond strength, Dental enamel, Laboratory research.

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INTRODUCTION

Dental erosion is defined as dental mineral loss due to a chemical process of dissolution caused by acids without bacterial involvement.[1] Sources of acids can be attributed both extrinsic and intrinsic ways, and erosive intensity is modified by quality and quantity of saliva.[2] Extrinsic factors mostly comprise the consumption of acidic foods and beverages, such as soft drinks and fruit juices, while eating disorders and gastro esophageal reflux are the major constituents of the intrinsic factors.[3]

These noncarious lesions are considered a challenging dental problem that calls for attention from dentists and patients.[4] In the initial stage of erosive lesions, only the enamel surface is involved and restorations may be placed for esthetic reasons and/or to prevent further progression.[5] In this case, adhesive restorative materials like glass ionomer cements (resin-modified or conventional) and resin composite are generally used in daily clinical practice.

The bond strength is an important indicator of the immediate and long-term performance of restorative materials to dental structure.[6] Under acidic conditions, all restorative materials have shown degradation over time[7,8] Conversely, the composite materials have shown higher durability.[7,9] Although glass ionomer cements (GIC) have properties of bonding to dentin and enamel, biocompatibility and the continuing fluoride release to adjacent structures, seems to have no preventive influence on the erosion after restorations.[10]

However, to the best of our knowledge, this is a pioneering investigation of the bonding performance of adhesive materials to eroded enamel. Therefore, the objective of this *in vitro* study was to evaluate the bond strength of the one etch-and-rinse adhesive system and one resin-modified glass ionomer cement to sound and eroded enamel.

MATERIALS AND METHODS

Teeth Selection and Preparation

Forty-eight freshly extracted bovine incisors were selected, disinfected in 0.5% chloramine and stored in distilled water at 4ºC until use. The roots were removed using low-speed diamond disk in a cutting machine (Labcut 1010, Extec Co, Enfield, USA) and crowns were partially embedded in self-curing acrylic resin inside PVC rings (JET Clássico®, São Paulo, SP, Brazil), with the buccal surfaces facing upward. The buccal surfaces were ground under water with series of SiC-papers ending with 600 grit to obtain flat enamel surfaces.

Erosive Challenge

Specimens were randomly allocated into 2 groups: (1) immersion in artificial saliva during the experimental period (control group—sound enamel, n = 24); (2) exposure to erosion challenge according to a pH—cycling model (eroded enamel, n = 24).

Three pH-cycles were performed each day at 8, 14 and 20 hours for 7 days. In each of them, teeth were immersed in a cola drink (Coca-Cola, [pH—2.6, phosphate—5.43mM 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, RJ, Brazil) for 5 minutes (30 ml per teeth) 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 teeth) between erosive cycles, under agitation and at room temperature. During the remaining time, the teeth were also kept in artificial saliva.[11]

Bonding and Restorative Procedures

Teeth from each enamel substrate (sound or eroded) were randomly reassigned into 2 subgroups according to adhesive material used: etch-and-rinse adhesive system (Adper Single Bond 2, 3M ESPE, St Paul, MN, USA) and resin-modified glass ionomer cement (Vitro Fil LC, DFL, Rio de Janeiro, RJ, Brazil). This resulted in a 2 × 2 factorial experimental design with 12 teeth in each subgroup formed from the crossing of two factors: substrate and adhesive material. The materials were applied following to the manufacturer's instructions (Table 1).

After appropriate pretreatment enamel surfaces according to adhesive material, a teflon matrix was positioned over the all prepared surfaces to build cylinders, resulting in specimens with 2 mm diameter and 4 mm height.

Two increments of resin composite (Opallis, FGM, Joinvile, SC, Brazil) were inserted in the matrix after application of the Adper Single Bond 2. Each increment was light-cured for 40s.

For Vitro Fil LC, the GIC was inserted in the matrix with aid the syringe Centrix® for avoid the inclusion of air bubbles into the material and light-cured for 20s. Surface protection was performed, following manufacturer's instructions. In all cases, light activation was performed using a quartz-tungsten halogen-light unit set at 500 mW/cm^2 (optilux 501, Kerr Corp, Orange, CA, USA). All bonding and restorative procedures were carried out by a single operator at a room temperature of 24°C. The teflon matrix was removed and all specimens were stored in distilled water at 37°C for 24 hours.

Shear Bond Strength Test

The specimens were then attached to the bond universal testing machine (DL 200, Emic, São José dos Pinhais, Brazil) and the shear load was applied parallel to adhesive interface in the base of the cylinders with a chisel-shaped rod at a crosshead speed of 0.5 mm/min until failure. Shear

Table 1: Composition and application mode of the adhesive materials tested

Material and manufacturer	*Composition*	*Application mode*
Adper Single Bond 2 (3M ESPE, St Paul, MN, USA)	35% phosphoric acid HEMA, water, ethanol, Bis-GMA, dimethacrylates, amines, metacrylate functional copolymer of polyacrylic and polyitaconic acids, 10% by weight of 5 nanometer-diameter spherical silica particles.	1. Etch for 15s. 2. Rinse with water spray for 15s, leaving tooth moist. 3. Active application of two consecutive coats of the adhesive with a fully saturated brush tip, for 15s each. Dry gently for 2-5s 4. Light-cure for 10s.
Vitro Fil LC (DFL, Rio de Janeiro, RJ, Brazil)	*Conditioner:* cavity conditioner 20% polyacrilic acid Powder: strontium aluminum silicate, excipients, activators and iron oxide.Liquid: 2-hidroxyethyl methacrylate, polyacrylic and tartaric acid solutions, benzoyl peroxide and camphorquinone. *Primer:* modified methacrylate polyacids, stabilizer, catalyser and ethyl alcohol. *Bond light:* Bisphenol Glycidyl Methacrylate, triethylene glycol dimethacrylate, 2,6-terc-butylphenol, ethyl urethane, B200P, benzyl Dimethyl Ketal, camphorquinone and quantacure EHA	1. Apply Vitro Conditioner for 10s, rinse and gently-air-dry, leaving a moist surface. 2. Apply primer and light-cure for 20s. 3. Dose 2 drops of liquid and one powder scoop, mix up to 25s, apply to enamel surface and light-cure for 20s. 4. Apply finishing gloss (Bond light) to surface protection.

HEMA: 2-hydroxyethylmethacrylate; Bis-GMA: bis-phenol A diglycidyl methacrylate.

bond strength means were calculated and expressed in MPa.

Statistical Analysis

Normal distribution of data was assumed after Kolmogorov-Smirnov test. The shear bond strength means were analyzed with two-way ANOVA using a factorial design with the substrate and adhesive material as variables. Tukey's HSD multiple comparisons statistical test at a 0.05 significance level was used.

RESULTS

Shear bond strength means (MPa) and standard deviations for all experimental groups are displayed in Table 2. The main factors substrate ($p < 0.01$) and adhesive material ($p = 0.01$), as well as, cross-product interaction were statistically significant ($p < 0.01$).

Etch-and-rinse adhesive system (Adper Single Bond 2) showed the highest bond strength value to eroded enamel, whereas no difference was observed in sound enamel compared to resin-modified glass ionomer cement (Vitro Fil LC).

DISCUSSION

Increased erosion prevalence has been clinically observed, especially restricted to enamel.[12,13] Lifestyle changes and rise in consumption of soft drinks/acid foods seem to be the associated factors for this condition.[14,15]

To simulate dental erosion, the dynamic erosive pH-cycling model using cola drink was the method employed in this current study. This beverage has a high erosive potential,[16] due to low pH and low fluoride and calcium concentrations.[11] The protocol adopted simulated a regular intake for individuals considered at risk to dental erosion, which also allowed for a contact of the teeth with saliva, being suitable for remineralization of dental hard tissues.[17]

The substitution of human teeth for bovine dental hard tissues has been recommended for several purposes, especially in adhesion tests.[18-20] Reis et al[21] showed that the human and bovine enamel present similar characteristics when analyzed by scanning electron microscopic. Moreover, bovine teeth are easier to be obtained in large scale in good conditions and they present less composition variation.[22] For these reasons, the bovine enamel was used in current study.

Table 2: Shear bond strength means (MPa) and standard deviations for all experimental groups (*)

	Substrate	
Material	*Sound enamel*	*Eroded enamel*
Adper Single Bond 2	16.7 ± 6.0[a]	25.2 ± 4.8[b]
Vitro Fil LC	15.0 ± 2.6[a]	13.57 ± 1.3[a]

*Different superscript letters indicate statistically significant differences between columns and rows ($p < 0.05$).

The exposition of dental tissues to erosive acids causes mineral loss, in a process known as softening. Micromorphologically, the erosion process leads to a formation of spatial areas with damaged apatite that exhibits local structural alteration, namely broken and/or loosened P-O—Ca atomic linkages.[23] Overtime, as softening progress goes forward into the enamel, the dissolution reaches the point where this layer of enamel is lost completely. The enamel loss can occur by the direct removal of hard tissue by complete dissolution or by the mechanical wear (tooth brushing and/or mastication) of the remaining thin softened enamel layer,[24] that justify the restorative treatment of this substrate. Studies have demonstrated no preventive effect of dental materials on the erosion of adjacent enamel,[11,16] but no previous study evaluated the bonding effectiveness of adhesive materials, as resin-modified GIC and etch-and-rinse adhesive system to eroded enamel.

In the current study, Adper Single Bond 2 showed highest bond strength value to eroded enamel. Since enamel bonding is mainly based on micromechanical interlocking of a low-viscosity resin into microporosities, the preliminary etching with phosphoric acid (strong acid; pH = 0.7) and the increase in porosity of the eroded enamel due erosion process,[25] probably provides a greater penetration depth of the resin into intercrystalline spaces and consequently, allowing better mechanical retention and adhesion in eroded enamel.

Unlike the adhesion mechanism of adhesive systems, based on micromechanical retention, the GIC also is capable of bonding chemically to tooth structure, from the ionic interaction between carboxylic groups of polyacrilic acid with calcium hydroxyapatite. Consequently, the increase of micromechanical retention in eroded enamel promotes little influence on the adhesion of resin-modified GIC, which explains the similar adhesive performance this material in both substrates.[26]

Conversely, no difference in the bond strength values was observed in sound enamel between adhesive materials tested. One previous study[27] compared the bond strength of adhesive system and resin-modified glass ionomer cement to enamel and found no differences between tested materials. Nevertheless, the adhesive materials were bonded to brackets in sound enamel and thus, the results cannot directly be compared to results obtained in current study.

Further studies should be encouraged for a better understanding of the longevity bond of adhesive materials, especially, in eroded enamel, in order to find reliable correlation with clinical situations.

CONCLUSION

Bond strength of etch-and-rinse adhesive system increases in eroded enamel, while no difference is verified to resin-modified glass ionomer cement.

CLINICAL SIGNIFICANCE

Adhesive materials may be used in eroded enamel without jeopardizing the bonding quality; however it is preferable to use etch-and-rinse adhesive system.

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ABOUT THE AUTHORS

Tathiane Lenzi

PhD Student, Department of Pediatric Dentistry, University of São Paulo, Brazil

Daniela Hesse

PhD Student, Department of Pediatric Dentistry, University of São Paulo, Brazil

Camila Guglielmi (Corresponding Author)

PhD Student, Department of Pediatric Dentistry, University of São Paulo, Brazil, e-mail: camigugli@usp.br

Ketlin Anacleto

Private Practice, Department of Pediatric Dentistry, University of São Paulo, Brazil

Daniela Prócida Raggio

Professor, Department of Pediatric Dentistry, University of São Paulo Brazil