Original Article

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Influence of selective acid etching on microtensile bond strength of a self-adhesive resin cement to enamel and dentin

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Abstract

Aim: To evaluate the selective acid etching of enamel and dentin on microtensile bond strength (μTBS) of a self-adhesive resin cement. **Methods:** Forty-eight bovine teeth were selected and had the buccal surface ground to obtain a flat dentin (n=24) or enamel (n=24) surface. Z250 composite resin blocks (4 x 3 x 8 mm) were prepared for cementation at enamel and dentin. Each substrate received three experimental resin cementation strategies: 1) 37% phosphoric acid etching before the application of RelyX Unicem; 2) application of RelyX Unicem alone; 3) RelyX ARC as a control. Samples were light-cured using a halogen light (Optilux 501, 700mW/cm^2), for 40 s. Hourglass-shaped specimens were obtained and submitted to a tensile strength at a crosshead speed of 0.5 mm/min in a universal testing machine. For statistical analysis, data were submitted to ANOVA and Tukey's test at a pre-set alpha (á=0.05). **Results:** RelyX Unicem showed similar bond strength value (16.5 MPa) when compared to the pretreatment with acid etching (11.9 Mpa) and to the conventional resin cement (18.1 MPa) for enamel. All luting strategies presented similar dentin bond strength, but significantly lower than enamel bond strength. **Conclusions:** Acid etching prior to RelyX Unicem application did not improve microtensile bond strength for enamel and dentin.

Keywords: resin cement, bond strength, dental enamel, dentin, dental acid etching.

Introduction

With the development of adhesive dentistry, resin luting cements have been widely used due to their ability to bond indirect restorations to tooth structure[1-2]. These materials can minimize some adverse effects of direct composite restorations, like polymerization shrinkage stress magnitude[3] and gap formation at tooth/restoration interface[4].

Conventional resin luting cements require prior application of bonding systems, which is more susceptible to procedure errors, due to unsatisfactory adhesive curing[5-6], overdrying or overwetting of the dentin for hybridization[7], cement incompatibility to some acidic monomers of the bonding system[8-10]. Self-adhesive resin cements

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were introduced in an attempt to simplify resin luting strategy, in which a single clinical step is achieved[11].

In this process, demineralization/infiltration of enamel and dentin is expected to occur simultaneously, not requiring acid etch and bond system application separately[11]. Some studies state that bond strength values of self-adhesive cements are comparable to those of conventional ones[12-13]. Therefore, bonding performance of these simplified cements on smear layer-covered tooth substrate still remains a concern[14-17]. Also, a chemical reaction is suggested to occur between the self-adhesive resin cement and hydroxyapatite (3M ESPE RelyX Unicem product profile), similar to the reaction of glass ionomer cements[18-19].

Notwithstanding that self-adhesive resin luting cements do not require pretreatment of the dental substrate, acid etching has been proposed for removal of the smear layer in an attempt to enhance the interaction between the cement and the enamel surfaces, thus increasing the bond strength[14,20]. It is not clear if pretreatment with phosphoric acid etching of enamel and dentin is really efficient, and this procedure is largely questioned[11,21].

Since the real efficacy of selective enamel and dentin etching is not clear by the literature, the aim of this study was to evaluate the effect of prior acid etching of enamel and dentin on the microtensile bond strength of a self-adhesive resin luting cement, using a total-etch resin cement as the control. The hypothesis tested is pretreatment with phosphoric acid etching could increase microtensile bond strength of RelyX Unicem to both the dental substrates.

Material and methods

Forty-eighty bovine incisors were selected, cleaned and stored in a 0.5% chloramine T solution at 4°C for no more than 1 week. Roots were sectioned 1 mm below the cementoenamel junction using a double-faced diamond saw (K.G. Sorensen, São Paulo, SP, Brazil). Then, the buccal surface of half of the specimens was ground on a water-cooled mechanical polisher (Metaserv 2000, Buehler, UK Ltd, Lake Bluff, IL, USA) using 320-, 600- and 1200-grit silicon carbide paper (Carbimet Disc Set, #305178180, Buehler, UK Ltd, Lake Bluff, IL, USA) in order to obtain a flat dentin area of at least 8 mm^2, and the other half, to obtain a flat enamel with the same area.

Specimens were randomly assigned into 3 experimental groups according to the luting protocol, *per* tooth substrate (n = 8): 1) RelyX Unicem (3M/ESPE, St. Paul. MN, USA); 2) 37% Phosphoric acid etching (Scotchbond, 3M/ESPE) followed by RelyX Unicem; 3) RelyX ARC, as control (3M/ESPE). Luting agents were mixed and placed according to manufacturer's instructions. For RelyX ARC, the specimen was acid etched, Scotchbond Multi-purpose Plus (3M/ESPE) was applied followed by resin cement application. The composition and manufacturer's information of the resin luting cements are shown in Table 1.

Composite resin blocks (3 mm thick, 8 mm long and 4 mm wide) were built with a microhybrid composite (Z250, shade A3; 3M ESPE) into a silicon mold. Composite was light-cured for 40 s with a halogen light (Optilux 501, Kerr Corp, Orange, CA, USA, at 700mW/cm^2 of irradiance) and then photocured at an EDG unit for 3 min. The composite surface that would be in contact to the luting cement was airborne-particle abraded with 50µm aluminum oxide particles (Asfer Indústria Química Ltda, São Caetano do Sul, Brazil) for 10 s.

Then, a silane couple agent (RelyX Ceramic Primer, 3M/ESPE) was applied on the composite block bonding surface. Resin luting cements were applied according to the tested groups on enamel or dentin using manufacturer's instructions, and then the composite blocks were cemented.

Specimens were light-cured for 40 s from the buccal, lingual and occlusal directions. After storage in distilled water for 24 h, specimens were serially sectioned perpendicular to the bonding interface into slabs with a diamond saw (Isomet 1000, Buehler, UK Ltd, Lake Bluff, IL, USA), and then hourglass-shaped specimens were created with a cross-sectional bonded area of approximately 1 mm^2. The specimens were fixed to the grips of a universal testing machine (EMIC 500, São José dos Pinhais, PR, Brazil) using a cyanoacrylate adhesive (Loctite Super Bonder Gel, Henkel, D Düsseldorf, Germany) and tested in tension at a crosshead speed of 0.5 mm/min until failure. For statistical analysis, the obtained data were analyzed (SAS 9.1, SAS Institute, Inc, NC) and submitted to a two-way ANOVA and Tukey's test at 5 % of significance

Results

There was no statistically significant difference in the bond strength values for the tested conditions at each tooth substrate ($p > 0.05$). However, significant differences were found between enamel and dentin, where enamel showed higher microtensile bond strength means compared to dentin,

Table 1. Materials used in the study with composition and manufacturer's information

Resin cements	Composition	Manufacturer
RelyX Unicem(Batch #270644)	Powder: glass fillers, silica, calcium hydroxide, self-curing initiators, pigments, light-curing initiators, substituted pyrimidine, peroxy compound.Liquid: methacrylated phosphoric esters, dimethacrylates, acetate, stabilizers, self-curing initiators, light-curing initiators.	3M ESPE, St Paul, MN, USA
RelyX ARC(Batch #GU9JG)	Paste A: Silane treated ceramic, Bis-GMA, TEGDMA, photoinitiators, amine, silane treated silica, functionalized dimethacrylate polymer.Paste B: silane treated ceramic, TEGDMA, Bis-GMA, silane treated silica, benzoil peroxide, functionalized dimethacrylate polymer.	3M ESPE, St Paul, MN, USA

regarding the resin luting protocol ($p > 0.05$). These results are shown in Table 2.

Table 2. Means and standard deviations (in MPa) of microtensile bond strength for the tested groups.

	RelyX ARC	RelyX Unicem	Acid Etch + RelyX Unicem
Enamel	18.1 (2.0)Aa	16.5 (3.8)Aa	11.9 (6.6)Aa
Dentin	8.1 (3.6)Ab	9.2 (7.0)Ab	8.3 (3.7)Ab

Mean values followed by different lowercase letters in columns and uppercase letters in rows are significantly different (Tukey's test; $p<0.05$).

Discussion

The resin luting cements used in this study (RelyX Unicem and RelyX ARC) are broadly used on clinical practice for cementation of indirect restorations. Moreover, self-adhesive resin cements have been used because of its simplified resin luting strategy, reducing clinical steps for cementation procedure, although its bond strength it is not totally clarified[5-6,11].

Some studies have shown that the acidity of nonrinsed acidic primers can affect enamel bond strength[22-24]. To improve enamel adhesion, selective phosphoric acid etching prior to the application of self-adhesives resin cements has been suggested[14,20-21]. In this work, the tested hypothesis was rejected, since pretreatment with phosphoric acid etching did not affect enamel bond strength of the self-adhesive resin cement tested. For enamel, similar microtensile bond strength values were found, regarding the fixing system. A possible reason for this fact is that RelyX Unicem would present a low pH at the initial stage of polymerization. Immediately after mixing the self-adhesive cement is very acidic, lower than pH 2 at the first curing minute, reaching at about 5 after this point, according to the manufacturer. This cement only reaches a neutral pH (at about 7). The low pH after mixing promotes only a superficial etching, increasing slightly the surface free energy and improving mechanical bond mechanism. However, it is known that hybrid layer is not formed in this interaction and part of the adhesion of self-adhesive cements occur due to chemical bonding to tooth hydroxyapatite[14,20]. In this manner, enamel prisms would be affected by phosphoric acid deep etching, maybe interfering bond strength. It goes against the majority of studies[14,20-21], which state that micromechanical interaction of resin penetration between the crystallites and enamel rods associated with chemical bonds to hydroxyapatite could explain the higher bond strength. These studies observed an increase on bond strength between enamel and self-adhesive cements after pretreatment with phosphoric acid etching . However, etching time, acid concentration, pH, pKa, hydroxyapatite buffer potential, and orientation of enamel prisms may significantly affect the demineralization of enamel and consequently, the bond strength[25-27].

Bonding to dentin has been referred to be a less reliable strategy, especially when compared to enamel bonding, because of the intrinsic characteristics of dentin, like higher organic content, variations in tubular structure and fluid flow[28-30]. In this work, pretreatment with phosphoric acid etching did not affected RelyX Unicem bond strength to dentin, rejecting the tested hypothesis, maybe because no further cement penetration would occur even with opened and plug-free dentinal tubules[31-33]. Thereby, the viscosity of this cement may hamper the infiltration into dentin[11]. Collagen exposure to phosphoric acid does not seem to improve the bonding values of the hydrophobic auto-adhesive cement[34]. The exact bonding mechanism of these simplified materials remains not clarified. Thus, the results are in agreement with the literature, showing higher bond strength for enamel compared to dentin either with RelyX ARC or RelyX Unicem regardless of pretreatment with phosphoric acid etching for the self-adhesive cement[14,20].

The mechanical test used in this work to evaluate the bond strength values was the microtensile bond test[35]. Some advantages of this methodology are easier specimen collection and uniform loading stress distribution over a small bonded area[36-39]. Sticks, dumbbell bars or hourglass shapes can be the presentation of the microtensile specimens[40-41]. Also, tensile tests are shown to be the most common laboratory tests to evaluate adhesive strength of bonding systems to the tooth substrate[38-39,41]. However, there are some limitations to consider about microtensile bond strength test, as stress on the bonded interface due to the handling, cutting procedure and the way of attachment[40].

The use of bovine teeth could be considered a limitation of the present work. This substrate is not as accurate as human teeth when different interactions between bond system and enamel/dentin are investigated. Thus, bovine teeth were selected because they are a suitable substrate for the tested methodology, easier to collect compared to human teeth and do not rely on ethical problems. Bovine enamel and dentin are adequate alternatives for human teeth, in adhesion tests[42]. However, it has been observed that a good marginal quality is more difficult to ensure with these bovine substrates[4]. In this sense, a successful technique in bovine teeth also tends to be successful in human teeth[42]. Additionally, the use of bovine substrate has been supported by several authors[43-46].

In conclusion, selective acid etching did not improve microtensile bond strength of RelyX Unicem to enamel and dentin. The self-adhesive resin luting cement presented higher bond strength to enamel compared to dentin and similar values when compared to a conventional dual-cured resin cement. The results of this work cannot be directly extrapolated to clinical *in vivo* conditions, since clinical trials are still required.

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