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Deproteinization stabilises dentin bonding of self-adhesive resin cements after thermocycling

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ABSTRACT

This study examined the effect of deproteinization on the microtensile dentin bond strength (μTBS) and nanoleakage (NL) of conventional and self-adhesive resin cements after 24 h or after 20,000 thermocycles. Occlusal dentin of thirty-two human molars were distributed into four groups according to the type of cement used: conventional or self-adhesive; and the strategy of luting: RelyX ARC/Single Bond 2 (RAc) following the manufacturer's instructions (control), RelyX ARC/Single Bond 2 (RAd) applied after dentin deproteinization; RelyX U200 (RUc) following the manufacturer´s instructions (control); RelyX U200 (RUd) applied after dentin deproteinization. The specimens were cut into non-trimmed dentin–composite sticks and the half sticks of each group were subdivided into two subgroups: 24 h water storage and after 20,000 thermal cycles, before microtensile bond test. For NL, 5 bonded sticks from each subgroup were prepared and analyzed under SEM. Three-way ANOVA showed that the dentin deproteinization increased the μTBS of both cements, although the RAd group showed a decrease on the μTBS after thermocycling. Chi-square test showed significant loss of specimens by premature failure for the groups after thermocycling, except for the RUd group. The dentin deproteinization improved the initial μTBS and decreases the NL of both cements tested, but, after thermocycling, this technique is only effective for RelyX U200.

1. Introduction

Currently, resin cements are the most indicated luting materials in the cementation of indirect composite resins and ceramic restorations to tooth structures. These cements showed advantageous properties like low water sorption, low solubility and high color stability [1], bond to enamel and dentin [2] and dual-curing mode [3–5]. These cements have been widely used for fixation of inlays, onlays, crowns, posts, and veneers [6] because of their enhanced mechanical properties, ease of handling and good esthetic qualities [3].

These luting systems may be classified as conventional resin cements or self-adhesive resin cements, depending on the bonding strategies [7–9]. The conventional resin cement requires the previous application of adhesive systems, that can be an etch-and-rinse or a self-etch system, while the self-adhesive cements requires no pretreatment of tooth surfaces [6,10–12]. Despite this attractive concept for cementation, scientific evidence has shown low retentive power of this cementation strategy for indirect restorations [13–15] and the superficiality of the interaction between the cement and dentin are the critical points in this new approach for self-adhesive cements [6,7,15]. Researches investigating pretreatment of dentin with acid solutions show controversial results.

Some studies have reported that the removal of the smear layer improved the interaction between self-adhesive resin cement and dentin [16–18]. On the other side, it has been shown that the

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Table 1
Resin cements, their application protocols and strategy of cementation.

	Composition	Control	Deproteinized
RelyX ARC/Single Bond 2 (3M/ESPE, Seefeld, Germany)	**RelyX^{MR} ARC:** TEGDMA, Bis-GMA, ether zircônia/ sílica filler, functional dimethacrylate polymer Adper Single BondTM 2: Ethanol, Bis-GMA, silanated silica filler, 2-HEMA, GDM, copolymer of polyacrylic and polyitaconic acidse UDMA.	1. Apply of 37% H_3PO_4 Gel (Condac 37%/FGM, Joinville, SC, Brazil) for 15 s 2. Rinsing with air spray 3. Dry with absorbent paper, keeping dentin moisture 4. Apply of two consecutive coats of Single Bond 2 for 15 s with gently agitation 5. Gently air thin for 5 s to evaporate the solvent 6. Light cure adhesive for 10 s 7. Apply the silane on the previously ragged surface of resin block 8. Mix cement for 10 s and apply on silanized surface of resin blocks 9. Wait 3 min and light cure each surface/margin for 40 s	1. Apply 37% H_3PO_4 Gel (Condac 37%/ FGM, Joinville, SC, Brazil) for 15 s 2. Rinsing with air spray + apply 5% NaOCl (Vetec, Rio de Janeiro, RJ, Brazil) for 2 min 3. Rinsing with air spray for 30 s 4. Dry with absorbent paper, keeping dentin moisture 5. Apply two consecutive coats of Single Bond 2 for 15 s with gently agitation 6. Gently air thin for 5 s to evaporate the solvent 7. Light cure adhesive for 10 s 8. Apply the silane on the previously ragged surface of resin block 9. Mix cement for 10 s and apply on silanazed surface of resin blocks 10. Wait 3 min and light cure each surface/margin for 40 s
RelyX U200 (3M/ESPE, Seefeld, Germany)	**RelyX^{TM} U200:** Silane treated glass powder, substituted dimethacrylate, 1-benzyl- 5 -phenyl-barbic-acid, calcium salt, 1,12-dodecane dimethycrylate, sodium p-toluenesulfinate, silane treated silica, calcium hydroxide	1. Apply the silane on the previously ragged surface of resin block 2.Mix cement for 10 s and apply on silanized surface of resin blocks 3.Wait 3 min and light cure each surface/margin for 20 s	1. Apply 37% H_3PO_4 Gel (Condac 37%/ FGM, Joinville, SC, Brazil) for 15 s 2. Rinsing with air spray 3. Apply 5% NaOCl (Vetec, Rio de Janeiro, RJ, Brazil) for 2 min 4. Rinsing with air spray for 30 s 5. Dry with absorbent paper, keeping dentin moisture 6. Apply the silane on the previously ragged surface of resin block 7. Mix cement for 10 s and apply on silanized surface of resin blocks 8. Wait 3 min and light cure each surface/margin for 20 s

pretreatment with phosphoric acid can result in reduction of the bond strength since the exposition of collagen fibrils after dentin demineralization reduced the penetration of the cement [18,19]. The collagen removal after etching with phosphoric acid could be an option to eliminate this mechanical barrier and improve the penetration [20] and the chemical interactions between resin cements and the hydroxyapatite of the dentin [21], providing optimized results.

Some studies have shown that collagen removal has a beneficial effect in the cement-dentin adhesion [19,22] but to the extent of our knowledge no experiments have been performed to evaluate this technique after aging under thermocycling. Therefore, this *in vitro* study examined the effect of deproteinization on the microtensile bond strength (μTBS) and nanoleakage (NL) between conventional and self-adhesive resin cements and dentin surfaces after 24 h or 20,000 thermocycles. The following hypotheses of study were tested: 1) the use of dentin deproteinization will not result in differences in the adhesive performance; 2) there is no difference between adhesive performance when a conventional and a self-adhesive resin cements was compared and; 3) thermocycles will not result in differences in the adhesive performance.

2. Materials and methods

2.1. Tooth preparation

Thirty-two freshly extracted human non-carious third molars were used in this study after obtaining the patients informed consent for their use, under a protocol approved by the Institution. The teeth were stored in 0.01% thymol solution at 4 °C for no more than 1 month. A flat dentin surface was exposed on each tooth after wet grinding of the occlusal enamel on #100- and # 400-grit SiC paper mounted in a polishing machine (Aropol 2V-Arotec SA, São Paulo, SP, Brazil). Dentin surfaces were exposed and inspected under ×80 magnification to ensure that no enamel remnants were left (Leica DM 1000 Leica Microsystems GmbH-Wetzlar, Germany). The exposed dentin surfaces were further polished on wet #600-grit silicon-carbide paper for 20 s to produce a standardized smear layer. After that, each tooth was individually fixed to a sectioning machine (Isomet 1000, Buehler Ltd. Lake Bluff, USA) and teeth roots were removed using a diamond disc under cooling, and to obtain dentin discs with 4 mm thick.