

Influence of root dentin treatment on the push-out bond strength of fiber posts

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Abstract The aim of the present study was to assess whether different dentin conditioning protocols with different acids [phosphoric acid, ethylene diamine tetra acetic acid (EDTA), and polyacrylic acid (PAA)] influence the bond strength of fiber posts along the radicular depth when luted with self-adhesive resin cement. Twenty single-rooted teeth were randomly divided into four experimental groups ($n = 5$) according to dentin treatment: *Group 1*: no treatment; *Group 2*: etching with 35 % phosphoric acid for 10 s; *Group 3*: 17 % EDTA application for 60 s; and *Group 4*: conditioning with 25 % PAA for 30 s. RelyX Fiber Posts were luted with the self-adhesive resin cement RelyX Unicem 2 Automix (3M ESPE). Roots were transversally sectioned into nine 1-mm thick specimens, three corresponding to each root third and a push-out test was performed. Data were analyzed by two-way ANOVA and Tukey test ($p < 0.05$). Failure mode was determined and specimens with representative failures for each group were observed under scanning electron microscopy. According to the results, dentin treatment influenced the bond strength ($p < 0.001$), whereas the root third did not ($p > 0.05$). Fiber posts luted after treating dentin with phosphoric acid, and PAA exhibited the highest push-out bond strength values, while the lowest were obtained after EDTA application. Intermediate results were obtained

when dentin was not conditioned. In conclusion, the bond strength of the self-adhesive resin cement RelyX Unicem 2 improves when root dentin is treated with 35 % phosphoric acid or 25 % PAA, before fiber posts luting irrespective of the root depth.

Keywords Fiber post · Smear layer · Push-out · Self-adhesive resin cement · Dentin treatment

Introduction

Fiber reinforced composite (FRC) posts have become the first choice to restore endodontically treated teeth with an important loss of structure due to extensive carious lesions, fractures, defective restorations, or excessive access preparations [1–3].

Several advantages of FRC posts have contributed to their widespread use. Their modulus of elasticity is similar to dentin, allowing a uniform distribution of occlusal stresses in the root dentin and reducing the risk of catastrophic failures, such as root fractures [4]. Moreover, shade and partial translucency of FRC posts confer them favorable optical properties to reproduce the natural aspect in the restored teeth and enable light curing of dual-cure resin cements [5].

Presently, there are numerous dual-cured resin cements available to lute FRC posts, with self-adhesive resin cements (SARCs) being preferably chosen by clinicians [6]. Their attractiveness is based on their simplicity of use and their reduced technique and operator sensitivity, because no etching or denting bonding is required [6, 7]. Moreover, literature suggests that SARCs improve fiber posts retention in comparison with those resin cements that require more bonding steps [3, 6].

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Self-adhesive resin cements contain an organic matrix and inorganic fillers as composites do. The organic matrix consists of multi-functional phosphoric acidic methacrylates or other acidic monomers. The inorganic fillers are composed of combinations selected from barium fluoroaluminoborosilicate glass, strontium calcium aluminosilicate glass, quartz, colloidal silica, and ytterbium fluoride among other glass fillers [8].

Their mechanism of adhesion consists of an initial redox reaction that starts after the acidic monomers are activated by light or self-curing mechanisms. Thus, the monomers demineralize the dentin hydroxyapatite and the infiltration within dentin and the polymerization of the monomers take place [8, 9]. In addition, an acid–base reaction occurs, where phosphoric acidic methacrylates in the organic matrix react with the basic fillers and the hydroxyapatite of root dentin [8, 10]. As a result of this reaction, the pH of SARC increases progressively, and the neutralization is accelerated by water, the by-product of the acid–base reaction [11].

The first SARC, RelyX Unicem, was launched into the market in 2002; thus, it is the most tested in literature and the so-called gold standard for SARCs [8]. A new version was available in 2010, RelyX Unicem 2 with improved fluidity according to manufacturer without changes in the mechanism of adhesion. RelyX Unicem demineralizes dentin very superficially without a distinct hybrid layer or resin tags formation [12, 13]. However, RelyX Unicem has been reported to achieve high and stable bond strength values despite this superficial interaction with dentin substrate, being attributed to its chemical interaction with the calcium from hydroxyapatite [14–16].

However, the main cause of failure for teeth restored with FRC posts is still post debonding [17]. Post space preparation generates a thick smear layer that contains rough debris and remnants of gutta-percha and sealers [18]. Moreover, the mineralized components of this smear layer are able to buffer the pH of the acidic monomers, limiting its diffusion and impairing a proper demineralization of the underlying dentin [19, 20].

Therefore, total or partial removal of the smear layer by different acidic solutions has been proposed to enhance the interaction between resin cement and dentin, and increase the SARCs bond strength [21–24]. However, a few studies have evaluated the effectiveness of the application of these acidic solutions before SARCs insertion in the post space and, according to the results, this effectiveness, seems to be SARC dependent [24, 25].

The aim of this study was to determine the push-out bond strength of RelyX Unicem 2 along the radicular depth after treating the root dentin with a strong acid, such as 35 % phosphoric acid, or mild acids, such as 17 % EDTA and 25 % PAA. Thus, the first null hypothesis was that the

bond strength of RelyX Unicem 2 is the same regardless of the acid applied on the root canal dentin. The second null hypothesis was that this bond strength is similar along the radicular depth.

Materials and methods

Twenty single-rooted human teeth with fully developed apices and extracted for periodontal or orthodontic reasons were selected. The inclusion criteria were absence of caries or root cracks, and previous endodontic treatments, posts, or crowns. Teeth were stored in 0.1 % thymol at 4 °C for a maximum of 3 months.

Roots were sectioned perpendicularly to the long axis of the teeth to a length of 16 mm from the apex using a diamond bur at high speed under copious water cooling.

Specimen preparation

Root canals were endodontically instrumented at a working length of 1 mm from the apex. All root canals were instrumented by the same operator. Canals were treated using a crown-down preparation technique with ProTaper rotatory instruments (Dentsply-Maillefer, Ballaigues, Switzerland) to size F1 or F2. Irrigation was performed using 5.25 % sodium hypochlorite between files and 17 % EDTA solution (Ultradent, South Jordan, UT, USA) for 1 min as a final rinse. Root canals were rinsed with distilled water for 1 min and then dried with ISO-standardized paper points (Dentsply-Maillefer). Afterwards, they were filled with gutta-percha cones (Dentsply DeTrey, Konstanz, Germany) and AH-Plus cement (Dentsply DeTrey, Zurich, Switzerland) with the lateral condensation technique. Once the endodontic treatment was completed, cervical root canal openings were filled with a resin-modified glass-ionomer cement (Vitrebond Plus, 3M ESPE, St Paul, MN, USA) that was light-cured for 40 s (LED Elipar curing light, light intensity of 1200 mW/cm², 3M ESPE, Seefeld, Germany). Specimens were then stored in distilled water for 7 days at 37 °C.

Post luting procedures

Post spaces were prepared to a depth of 10 mm by removing the gutta-percha with Gates-Glidden drills size 2 and 3 (Dentsply-Maillefer), leaving a minimum apical seal of 5 mm.

Dowel spaces were drilled using calibrated drills corresponding to the conical RelyX glass Fiber Post size #1 or #2 (3M ESPE). A final flush of the canal spaces was accomplished using sterile water, and they were dried with paper points (Dentsply-Maillefer).

The prepared roots were equally and randomly divided into four experimental groups ($n = 5$) [26], according to the dentin treatment performed before RelyX Unicem 2 (3M ESPE) application (Table 1): Group 1: No dentin treatment; Group 2: etching with 35 % phosphoric acid (Ultra-Etch, Ultradent) for 10 s; Group 3: treatment with 17 % EDTA gel (SlickGel ES, SybronEndo, Orange, CA, USA.) for 60 s; and Group 4: conditioning with 25 % PAA (Ketac Conditioner, 3M ESPE) for 30 s.

FRC posts were tried-in, cleaned with alcohol, and then luted. The post was seated with constant finger pressure and stabilized under a 750-g static load [23]. This seating force was applied for the first 2 min, leaving the material to set in the self-curing mode and, then, it was light-cured for 40 s.

After the luting procedure, the coronal part of the posts was completely covered with the resin-modified glass-ionomer cement Vitrebond Plus (3M ESPE), and the roots were stored in distilled water for 7 days at 37 °C.

Push-out bond strength test

Roots were embedded in epoxy resin and transversally sectioned with a high precision digitally programmed machine (Isomet 5000, Buehler, Lake Buff, IL, USA). It was pre-set to serially obtain nine specimens 1-mm thick

by means of a water-cooled diamond saw. Three sections corresponded to each root third: apical, middle, and coronal. Each specimen was marked on its coronal side with an indelible marker, and the exact thickness of each segment was measured using a digital caliper of 0.001-mm accuracy (Mitutoyo, Tokyo, Japan).

Before testing, all sections were observed under a stereomicroscope (Olympus SZX7, Olympus Optical Co., Ltd, London, UK) at 30× to detect potential defects caused by cutting. Photographs of both sides of each specimen were taken to calculate the coronal and apical post radius using the software Image J software, v. 1.44, (National Institutes of Health, Bethesda, MD, USA). The coronal dentin surface of each specimen was secured face down with cyanoacrylate adhesive (Loctite Gel, Henkel, Düsseldorf, Germany) to a horizontal stainless steel platform with a central circular perforation. A vertical static load was applied in the apical-to-cervical direction via a metal rod to the post section in each specimen, using a universal load testing machine (Instron 3345, Instron Corp., Canton, MA, USA) at a crosshead speed of 0.5 mm/min. The maximum push-out force (N) for bond failure was recorded. The retentive strength of the post segment was expressed in MPa, by dividing the load at failure in Newtons by the interfacial area (A) of the post fragment, which corresponded to the bonded area, in square millimeters

Table 1 Composition of the materials tested and manufacturers' instructions

| Experimental groups | Composition | Luting procedure |
|--|---|---|
| Group 1: RelyX Unicem 2 Automix (3M ESPE) | Silane treated glass powder, TEGDMA, substituted dimethacrylate, sodium p-toluenesulfonate, 1,12-dodecane dimethacrylate, 1-benzil-5-phenil-barbic-acid, calcium salt, silane treated silica, calcium hydroxide, 2-(phosphonoxy)-1,3-propanediyl bismethacrylate, 2-propenoic acid, 2-methyl-, phosphinicobis (oxy-2,1,3-propan), mixture of other methacrylated glycerol, glycerol-1,3-dimethacrylate, 2-propenoic acid, 2-methyl-, 1,1_- [1-[(phosphonoxy)methyl]-1,2-ethanediyl]ester, sodium persulfate | <ol style="list-style-type: none"> 1. Attach the Endo Tip to the mixing tip “wide” for application in the root canal, insert the Endo Tip as deeply as possible in the root canal and apply RelyX Unicem 2 2. Place the post in the root canal filled with cement; apply moderate pressure to hold it in position 3. Remove excess 4. Light-cure 40 s |
| Group 2: 35 % phosphoric acid gel (Ultradent) + RelyX Unicem 2 Automix (3M ESPE) | Phosphoric acid | <ol style="list-style-type: none"> 1. Etch for 15 s using 35 % phosphoric acid gel 2. Rinse thoroughly with water 30 s and dry slightly with paper points 4. Post luting like group 1 |
| Group 3: EDTA 17 % gel (SybronEndo) + RelyX Unicem 2 Automix (3M ESPE) | Ethylenediaminetetraacetic acid (EDTA) | <ol style="list-style-type: none"> 1. Apply EDTA onto the dentin surface for 60 s 2. Rinse thoroughly with water 30 s and dry lightly with paper points 4. Post luting like group 1 |
| Group 4: 25 % PAA (3M ESPE) + RelyX Unicem 2 Automix (3M ESPE) | Polyacrylic acid | <ol style="list-style-type: none"> 1. Apply PAA onto the dentin surface for 30 s 2. Rinse thoroughly with water 30 s and dry lightly with paper points 4. Post luting like group 1 |

(mm²). The interfacial area was calculated as the lateral surface of a truncated cone using the following formula: $A = (R + r) [h^2 + (R - r)^2]^{0.5}$, where $h = 3.14$, R = coronal post radius, r = apical post radius, and h = root slice thickness.

The failure mode of each debonded specimen was evaluated under the stereomicroscope at 30× magnification and classified as adhesive (between dentin and resin cement or between resin cement and the post), cohesive (within the resin cement or within the post), or mixed failure (a combination of adhesive and cohesive failures). Representative specimens from each experimental group were sputter-coated with gold (Bal-Tec Sputter Coater SCD 005, Bal-Tec GmbH, Witten, Germany) and observed under a scanning electron microscope (SEM) (Phillips XL30 ESEM, FEI Company, Hillsboro, OR, USA) at 100×.

Micromorphological analysis

Eight additional teeth (2 per experimental group) were instrumented and obturated as previously described for the push-out test. Then, the gutta-percha was removed and root dentin was treated as described before for each experimental group. Afterwards, two longitudinal grooves were made on both sides of each root using a diamond disk (Horico GmbH, Berlin, Germany) and with the help of a wax knife positioned into the grooves previously carved the roots were sectioned longitudinally. The specimens were subjected to a drying process by increasingly alcohol immersion dilutions in distilled water for 20 min each (25, 50, 75, 95, and 100 %) according to Perdigão et al. [27] and were gold-sputtered. The surfaces were morphologically analyzed under the same scanning electron microscope at 5000×.

Statistical analysis

Two-way ANOVA was carried out to determine the influence of the independent variables dentin treatment and root depth on the dependent variable bond strength of the resin cement used to lute fiber posts, and posterior comparisons were analyzed by Tukey's test. Statistical analysis was performed by means of the IBM SPSS 19 software (IBM Company, Armonk, NY, USA) with a pre-set alpha of 0.05.

Results

Two-way ANOVA test showed that the variable dentin treatment influenced on the dependent variable bond strength ($p < 0.001$), while the root depth and the

interaction between dentin treatment and root depth did not ($p > 0.05$).

Mean and standard deviations determined for each experimental group are shown in Table 2. The posterior comparisons detected that the specimens treated with phosphoric acid or PAA showed similar and higher bond strength values than those obtained for the untreated group. The lowest bond strengths were achieved when root dentin was conditioned with EDTA.

Failure mode analysis

Figure 1 summarizes the percentage of each failure mode determined for the experimental groups tested.

The most representative failure for the group without dentin treatment was the adhesive failure between the resin cement and the root dentin. As it can be observed in Fig. 2a, after post debonding, the cement remained mainly attached to the post and, thus, no remnants of resin cement were found around dentin walls.

Conversely, the cohesive failure within the cement was the most frequently found when root dentin was conditioned with phosphoric acid, EDTA, and PAA. After push-out test, specimens showed a break within the resin cement mass and consequently, resin cement remnants were clearly visible surrounding the post space walls and also the post (Fig. 2b–d).

Micromorphological analysis

The acidic solutions tested produced distinct effects on root canal dentin. When no acid solution was applied, the dentin walls appeared covered by smear layer and dentinal tubules could not be observed (Fig. 3a). The etching with phosphoric acid completely removed the smear layer and the smear plugs, dentinal tubules could be clearly identified, and peritubular dentin was selectively removed at the tubules entrance (Fig. 3b). By contrast, EDTA (Fig. 3c) was not able to remove the smear layer created after preparing the post space, and therefore, failed to expose dentine tubules resembling untreated dentin. Regarding PAA, this mild acid created a substrate where the smear layer and smear plugs were partially removed (Fig. 3d).

Discussion

According to the results obtained, the first null hypothesis must be rejected, as the dentin treatment influenced the push-out bond strength of FRC posts luted with RelyX Unicem 2. Nevertheless, the second hypothesis has to be accepted, as the root third did not affect the bond strength values achieved.

Table 2 Mean and standard deviations of bond strength results in MPa are shown for each experimental group

| Dentin treatment | Coronal | | Middle | | Apical | | Total | |
|------------------|------------|----------|------------|----------|------------|----------|--------------|----------|
| | Mean (SD) | <i>n</i> | Mean (SD) | <i>n</i> | Mean (SD) | <i>n</i> | Mean (SD) | <i>n</i> |
| None | 13.3 (5.5) | 15 | 12.9 (6.1) | 15 | 15.9 (3.8) | 11 | 13.8 (5.4) b | 41 |
| Phosphoric acid | 19.1 (5.0) | 15 | 21.0 (3.4) | 15 | 18.6 (5.1) | 7 | 19.8 (5.6) a | 37 |
| EDTA | 10.7 (4.5) | 15 | 11.5 (4.3) | 15 | 9.9 (3.0) | 8 | 10.9 (4.1) c | 38 |
| PAA | 20.3 (5.2) | 15 | 19.7 (8.4) | 15 | 14.2 (5.2) | 9 | 18.7 (6.9) a | 39 |

Same letters mean no statistical differences among groups

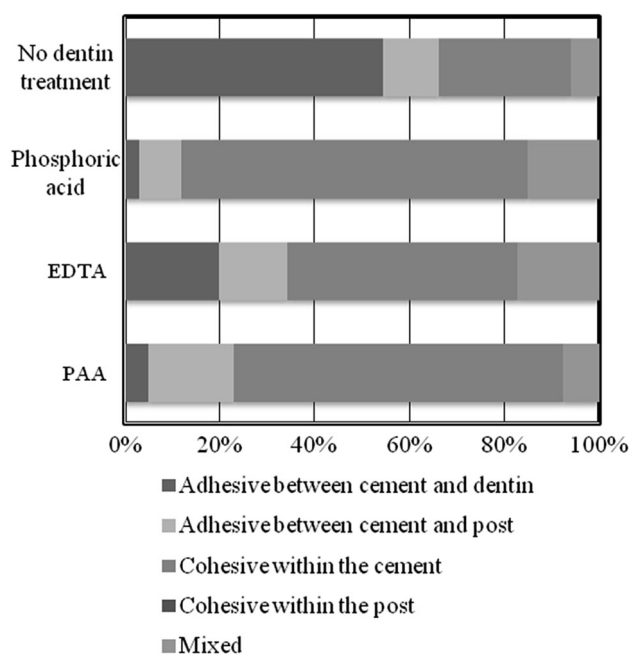


Fig. 1 Failure mode distribution

The push-out bond strength of FRC posts luted with RelyX Unicem 2 increased when smear layer was completely removed after treating dentin with a strong acid like 35 % phosphoric acid or if partially dissolved by a mild acid like 25 % PAA. Conversely, treatment with the mild acid 17 % EDTA produced significantly lower bond strength values.

Previous studies have reported an adequate performance of RelyX Unicem in comparison with total-etch or self-etching adhesives with the conventional resin cements, even when no dentin treatment was performed [28, 29]. However, *in vivo* reports have concluded that post debonding is still the main reason for clinical failure [17, 30].

In the present study, the application of phosphoric acid on root canal dentin completely removed the thick smear layer, and smear plugs created during post space preparation and demineralized the underlying dentin (Fig. 3a, b). This would help the infiltration of the resin cement and improve the micromechanical interlocking, yielding higher

bond strengths than an untreated substrate. The improved performance of the application of phosphoric acid can also be related with the high percentage of cohesive failures (61.8 %) within the cement registered in the present study in comparison with the untreated group which showed mainly adhesive failures (27.13 %). In fact, RelyX Unicem has been reported to perform better in terms of microtensile bond strength on a free smear layer surface in comparison with other SARC [31].

The bonding mechanism of SARC is considered to be similar to that of mild self-etching adhesives based on the adhesion-decalcification concept [12]. According to this, chemical reaction between the phosphoric acidic methacrylates and the hydroxyapatite of root dentin would be relevant [12, 32], as the chemical interaction has been reported to provide hydrolytic stability to this SARC after thermomechanical aging [33], being impaired by the demineralization produced by phosphoric acid. However, in the root canal, the bonding effectiveness of RelyX Unicem 2 would be mainly compromised by the presence of a thick smear layer. According to this, some reports described lower push-out bond strengths for self-etching adhesives when compared with total-etch ones [34, 35], although other reports concluded similar results for both depending mainly on the adhesive tested [13, 33, 36].

Regarding PAA, the application of this mild acid before luting FRC posts with RelyX Unicem 2 was related with higher bond strength values. These results are in accordance with previous studies on radicular dentin [37, 38], although Tonial et al. reported an improvement of bond strength only for Maxcem Elite resin cement with no influence for RelyX Unicem. Furthermore, Faria-e-Silva et al. did not find differences between treating dentin with PAA or not before luting with RelyX Unicem. However, PAA was used in a lower concentration (11.5 %), half of the one used in the present study which could explain the differences found with our results [24]. PAA partially removes the smear layer and the smear plugs (Fig. 3d), and according to Mazzitelli et al. and Pavan et al. [21, 37], it creates an intermediate surface layer that may activate calcium and phosphate ions promoting a stronger chemical interaction between resin cement and dentin. RelyX

Fig. 2 SEM images of representative failure modes for each experimental group. **a** Adhesive failure of a specimen cemented with no dentin treatment (group 1). **b** Cohesive failure of a specimen luted after dentin treatment with phosphoric acid (group 2). **c** Cohesive failure of a specimen cemented after dentin treatment with EDTA (group 3). **d** Cohesive failure of a specimen luted after treatment with PAA (group 4)

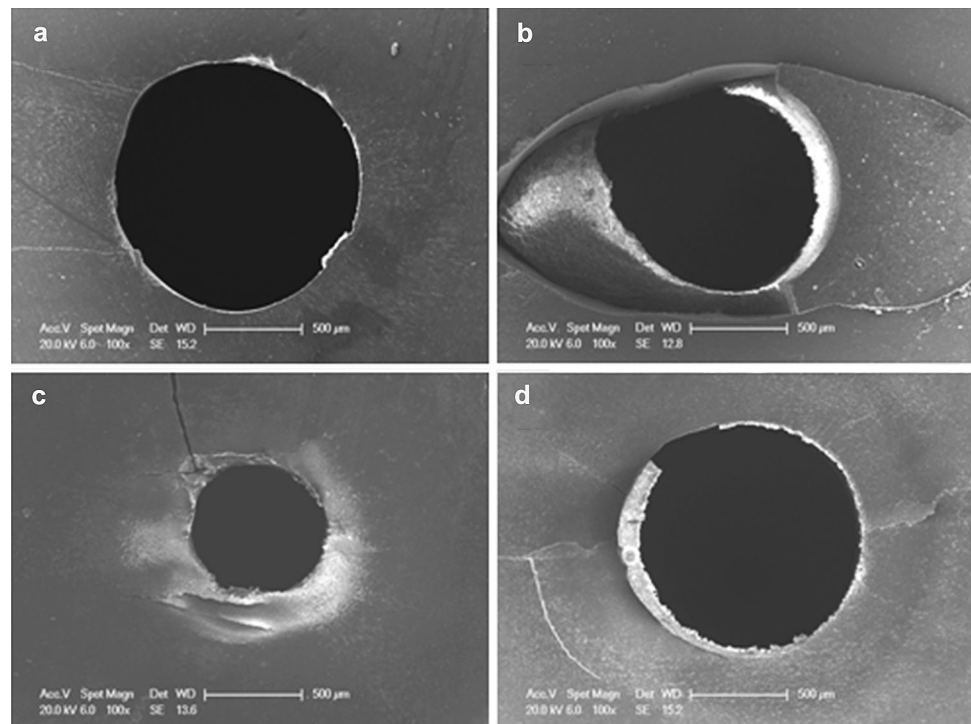
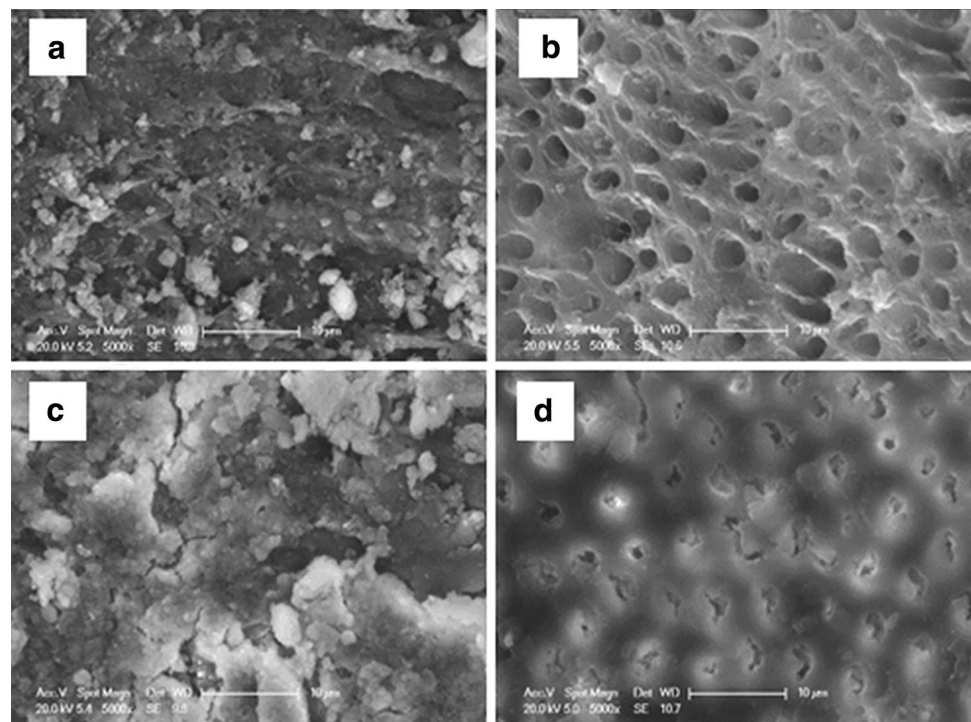


Fig. 3 SEM images that show the root canal dentin morphology of an untreated specimen (a), after treatment with phosphoric acid (b), EDTA (c), and PAA (d)



Unicem 2 contents glass-ionomer filler particles in its composition [39] and mild acids, such as PAA, have been recommended to remove or modify the smear layer prior to GICs cementation to enhance their bond strength to dentin [40]. This improvement in bond strength could be related,

similarly to phosphoric acid application, with the low percentage of adhesives failures between dentin and cement (5.1 %) and the high prevalence of cohesive failures within cement (69.2 %) when compared to untreated specimens.

By contrast, specimens treated with EDTA achieved the lowest bond strength values despite EDTA being also a mild acid. SEM images showed that after EDTA application, the dentin surface was still covered by the smear layer (Fig. 3c). However, EDTA is a chelating agent that depletes Ca levels, modifying the smear layer [41, 42]. Consequently, EDTA would not enhance micromechanical interlocking with the underlying dentin and would also hamper the chemical reaction produced between dentin hydroxyapatite and RelyX Unicem 2, according to previous studies [23, 24].

Regarding the influence of root dentin depth, push-out bond strength values of RelyX Unicem 2 were similar along the root canal depth. The bond strength of resin cements have been described to decrease from the coronal to the apical third [43, 44]. The main reasons described are the light attenuation through the fiber post and the histological characteristics of the root canal, as the number of dentinal tubules decreases along the radicular depth and the presence of sclerotic dentine increases with age [45, 46]. Notwithstanding, literature shows controversial results when RelyX Unicem 2 or its previous version has been tested. Some authors reported higher bond strength values at the coronal and middle thirds than at the apical one [14, 47], whereas others reported that RelyX Unicem behaved homogeneously along the radicular length as found in the current study [26, 48]. The main reason that could explain this homogenous behavior is its dual-cured nature, which allows polymerization where light does not reach [49].

According to the results aforementioned, the application of phosphoric acid or PAA improves the bond strength of RelyX Unicem 2 when luting fiber posts. Notwithstanding, SARC are not a homogenous group neither in composition nor in adhesion mechanism [8, 50]; thus, other SARC should also be tested. In addition, it would be interesting to evaluate how these improved results endure the test of time.

Finally, under the experimental conditions of this study, the following conclusions can be drawn:

- The bond strength of the self-adhesive resin cement RelyX Unicem 2 is increased by treating the root canal dentin with 35 % phosphoric acid or with 25 % PAA before luting fiber post, whereas the conditioning with 17 % EDTA is not beneficial.
- The root depth did not influence the push-out bond strength of this self-adhesive resin cement.

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Conflict of interest The authors declare that they have no conflict of interest.

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