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Push-out bond strength of a self-adhesive resin cement used as endodontic sealer

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Objectives: The aim of the present study was to investigate the bond strength of RelyX Unicem (3M) to root canal dentin when used as an endodontic sealer. ***Materials and Methods:*** Samples of 24 single-rooted teeth were prepared with Gates Glidden drills and K3 files. After that, the roots were randomly assigned to three experimental groups (n = 8) according to the filling material, (1) AH Plus (Dentsply De Trey GmbH)/Gutta-Percha cone; (2) Epiphany SE (Pentron)/Resilon cone; (3) RelyX Unicem/Gutta-Percha cone. All roots were filled using a single cone technique associated to vertical condensation. After the filling procedures, each tooth was prepared for a push-out bond strenght test by cutting 1 mm-thick root slices. Loading was performed on a universal testing machine at a speed of 0.5 mm/min. One-way analysis of variance and Tukey test for multiple comparisons were used to compare the results among the experimental groups. ***Results:*** Epiphany SE/Resilon showed significantly lower push-out bond strength than both AH Plus/Gutta-Percha and RelyX Unicem/Gutta-Percha ($p < 0.05$). There was no significant difference in bond strength between AH Plus/Gutta-Percha and RelyX Unicem/Gutta-Percha ($p > 0.05$). ***Conclusions:*** Under the present *in vitro* conditions, bond strength to root dentin promoted by RelyX Unicem was similar to AH Plus. Epiphany SE/Resilon resulted in lower bond strength values when compared to both materials. (*Restor Dent Endod* 2014;39(4):282-287)

Key words: Bond strength; Push out test; Resin-based cement; Root canal filling materials

Introduction

Root canal fillings are performed to seal the root canal system in order to prevent microorganisms and/or their toxic products from reaching the periodontal tissues.[1] Adhesive properties of endodontic sealers to dentin is an important aspect of filling materials because it minimizes the risk of material detachment from dentin during restorative procedures or masticatory function, ensuring proper sealing and consequently, the clinical success of endodontic treatment.[2] AH Plus (Dentsply De Trey GmbH, Konstanz, Germany) is considered a "gold-standard" endodontic sealer and is frequently used as a control material for research purposes and some previous studies have examined the potential of adhesive resins as root canal filling materials, with favorable *in vitro* results.[3-9]

Recently, a thermoplastic synthetic resin polymer, namely Resilon (Epiphany, Pentron, Wallingford, CT, USA) appeared as a promising material for root canal filling.[10,11] Resilon cones are used with Epiphany sealer, a dual polymerized cement associated with a monocomponent primer. This material has shown appropriate biocompatibility and more resistance to leakage than gutta-percha for filling root canals.[12-14]

RelyX Unicem (3M, St Paul, MN, USA) is a self-adhesive universal resin cement, to be used without surface pre-treatment, which has been also recently introduced in the market. Its efficiency is based on its antimicrobial effect and its potential for inducing mineralization.[15] The manufacturer claims that the organic matrix consists of newly developed multifunctional phosphoric acid methacrylates which are able to react with the basic fillers in the bulk cement and the hydroxyapatite of the hard tooth tissue.

Several *in vitro* studies have reported bond strength values of different adhesive systems used in combination with a luting composite to both enamel and dentin.[16-20] In view of these desirable features, this material could be used as an endodontic sealer. A recent study demonstrated good results when associating RelyX Unicem to Resilon cones.[20] However, up to the present date, little information regarding its potential to be used as an endodontic sealer is available in the literature.

Therefore, the purpose of this study was to assess the push-out bond strength of RelyX Unicem associated with gutta-percha to root dentin and to compare with those obtained for AH-Plus/gutta-percha and Epiphany SE/Resilon. The null hypothesis tested in this study is that there are no differences in the bonding effectiveness among the different tested materials.

Materials and Methods

Thirty-nine extracted single-root human teeth presenting similar dimensions and circular canals were randomly selected, under a protocol approved by the local Ethical Committee, and stored in distilled water at 4℃. To standardize the working length, a #15 K-file (Dentsply-Maillefer, Ballaigues, Switzerland) was inserted into the root canal until it could be visualized at the apical foramen. The working length was determined by subtracting 1 mm from this length. After measurement, the length of all roots was standardized to 13 mm to prevent the introduction of confounders which might contribute to variations in the preparation procedures.[21]

All teeth were instrumented using #3 and #2 Gates Glidden drills in the cervical portion of the canal. Then, the root canals were instrumented using K3 rotary instruments (Sybron Endo, Orange, CA, USA) to a size #25/0.06 up to the working length. Irrigation with 0.5 mL 2% chlorhexidine gel was used before each instrument and 1 mL 0.9% saline solution after each instrument. The smear layer was then removed with 3 mL 17% EDTA for 3 minutes. A total of 3 mL 0.9% saline solution was then used for 3 minutes as a final rinse and after that, each canal was dried with paper points. Obturation procedures were performed by using the single gutta-percha cone technique. Twenty-four roots were randomly distributed using a computer algorithm (http://www.random.org) into 3 groups for obturation using one of the three sealers:

Group 1 (n = 08): The roots were filled with AH Plus sealer (Dentsply De Trey GmbH) and 0.06 taper gutta-percha (Konne, Belo Horizonte, MG, Brazil).

Group 2 (n = 08): The roots were filled with Epiphany SE/Resilon sealer and 0.06 taper Resilon points (Pentron).

Group 3 (n = 08): The roots were filled with RelyX Unicem (3M ESPE, Seefeld, Germany) and 0.06 taper gutta-percha (Konne).

All groups were filled with the specific sealing agent (mixed according to the respective manufacturer's instructions) and a single #25 0.06 tapered master cone associated with vertical warm condensation. The sealers were prepared according to the manufacturers' instructions. On completion of these procedures, the specimens were radiographed at different angles to verify the quality of the filling procedure and presence of voids. The specimens were placed in 100% humidity at 37℃ for 7 days to ensure complete setting of the sealer.

Preparation of root slices for push-out bond strength testing

The middle portion of each root was horizontally sectioned into three 1 ± 0.1 mm-thick slices by using a low-speed saw with a diamond disk under continuous water irrigation. This process created 24 slices for each tested group. The root filling of each sample was loaded with a 0.45 mm-diameter stainless steel cylindrical plunger. The plunger tip was sized and placed to touch only the root filling. The load was applied in an apical-coronal direction to avoid any constriction interference caused by root canal taper during the test. Loading was performed on a universal testing machine (Instron, Norwood, MA, USA) at a crosshead speed of 0.5 mm/min until debonding occurred. The bond strength was determined by using a real-time computer software program (BlueHill2, Instron, Barueri, SP, Brazil), which plotted a load/time curve during the test. Bond failure load was noted when a sharp decline was observed on the graph and/or complete dislodgement of the root filling material was noted. The maximum failure load was recorded in newtons (N) and was used to calculate the push-out bond strength in megapascals (MPa) according to the following formula.

$$\text{Force (MPa)} = \text{Force (N)} / \text{Area}.$$

The adhesion (bonding) surface area of each section was calculated from $(\pi r_1 + \pi r_2) \times L$ and L was calculated from $\sqrt{(r_1 - r_2)^2 + h^2}$, where π is the constant 3.14, r_1 is the smaller radius, r_2 is the larger, and h is the thickness of the section in mm.[22]

Shapiro-Wilkand normality test and Levenes´ variance homogeneity test revealed that data was normally distributed, and there was homogeneity of variance among the groups. One-way analysis of variance (ANOVA) and *post-hoc* Tukey test were used for data analysis ($p < 0.05$).

SEM Imaging

Five teeth from each group were selected for morphologic examination. Epoxy resin replicas were prepared for SEM analysis.[23] After 48 hours, the roots were longitudinally sectioned using a slow-speed diamond saw under water cooling to expose the interfaces between the gutta-percha/sealer and dentin. Because of the small diameter of the filling material and the amount of structure lost during cutting, only one half of each tooth was available for the analysis.

The cut surfaces were ground with increasingly finer grit SiC papers (#600, #1,200, #2,000), followed by polishing with 6 and 3 μm diamond pastes under copious water irrigation. The specimens were ultrasonicated in deionized water for 10 minutes between each polishing step. Polyvinyl siloxane (3M ESPE, St. Paul, MN, USA) impressions of the surfaces were taken and these were replicated with Epo-Thin epoxy resin (Buehler Ltd., Lake Bluff, IL, USA). The replica technique was used to avoid artifacts produced during the preparation for SEM examination.

The replicas were secured with carbon tape to aluminum stubs, placed on the Peltier (cooling) stage of a field emission-ESEM (XL-30 ESEM-FEG, Philips, Eindhoven, The Netherlands) and examined without coating at 15 kV using the gaseous secondary electron mode at 4℃ and 5.9 Torr to achieve a 95% relative humidity.

Results

The mean push-out test values for each group are demonstrated in Table 1. ANOVA revealed significant differences among the groups ($p < 0.001$). Multiple paired comparisons showed that Groups 1 (AH Plus + gutta-percha) and 3 (RelyX Unicem + gutta-percha) had significantly higher bond strength than Group 2 (Epiphany + Resilon) (Tukey's test, $p < 0.001$). There was no significant difference in bond strength between Group 1 (AH Plus + gutta-percha) and Group 3 (RelyX Unicem + gutta-percha). Figures 1 and 2 show examples of bonded and non-bonded interfacial failures.

Table 1. Push-out bond strength values (Mean ± SD, Unit: MPa) for the different tested groups

Group	Push-out bond strength
AH Plus + Gutta-Percha	3.80 ± 1.94^A
Epiphany + Resilon	0.29 ± 0.15^B
RelyX Unicem + Gutta-Percha	4.56 ± 2.25^A

Different letters represent significant differences between the groups ($p < 0.05$).
SD, standard deviation.

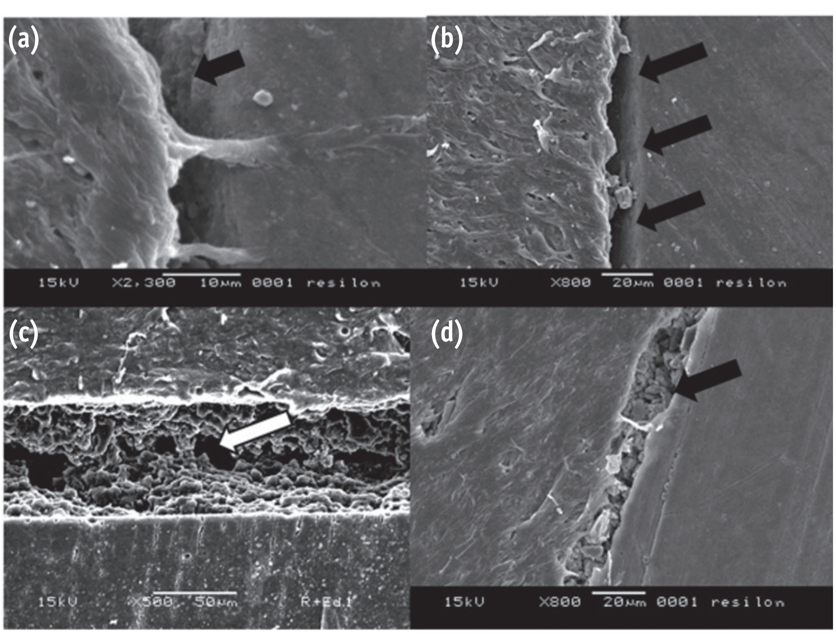

Figure 1. The types of interfacial failure in the Resilon/Epiphany group. (a) A micrograph at higher magnification showing the most commonly identified type of interfacial failure of the bonded intraradicular dentin (arrow); (b) A view of the previous micrograph, at lower magnification, showing the interfacial failure-along the surface (debonded intraradicular dentin); (c) A type of interfacial failure that was identified in specimen 2, in which the root fillings probably showed gaps after the polymerized process; (d) The Epiphany sealer contraction material.

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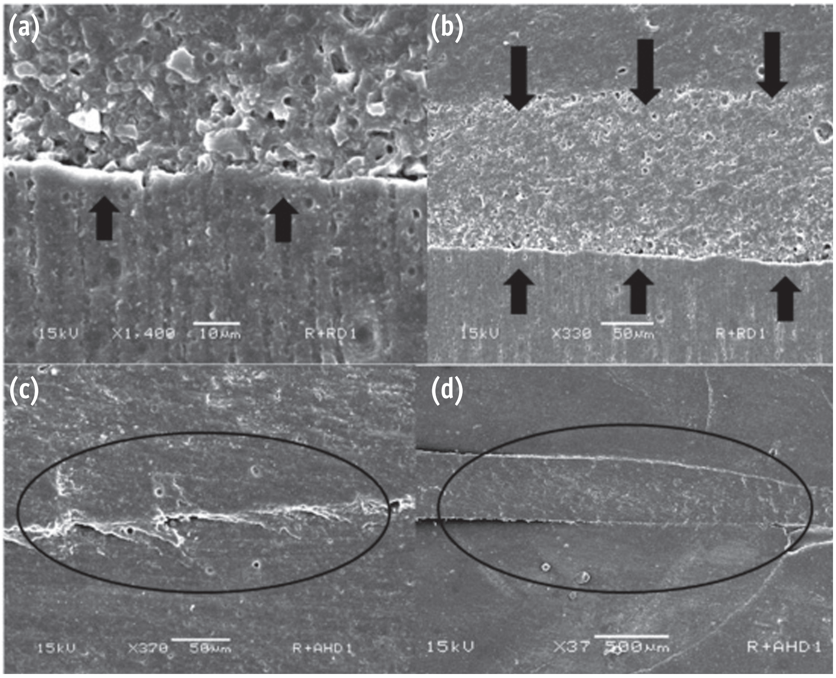

Figure 2. Images illustrating the types of interface taken from the AH-Plus/Gutta-Percha and RelyX/Resilon groups. (a) A micrograph at higher magnification showing the most commonly identified type of interfacial good adaptation of RelyX to the bonded intraradicular dentin (arrow); (b) A view of the previous micrograph, at lower magnification, showing the bonded interfacial surface; (c) The type of bonded interfacial surface of AH-Plus sealer; (d) A view of the previous micrograph, at lower magnification, showing the good adaptation of AH-Plus gutta-percha group.

Discussion

The push-out testing method allows bond strength measurements of adhesive materials to root canal dentin. The results of the present study showed higher bond strength for the RelyX Unicem and AH-Plus groups compared to the Resilon/Epiphany group ($p < 0.05$). Therefore, the tested null hypothesis was rejected. Previous studies demonstrated higher bond strengths in teeth filled with gutta-percha and AH Plus compared with those filled with Resilon/Epiphany.[24] These results can be explained by the high-quality properties of epoxy based root canals sealers, including very low shrinkage while setting and long-term dimensional stability.[25]

To the best of the author's knowledge, this is the first attempt to evaluate the bond-strength of RelyX Unicem when used as a root canal sealer. No statistical difference was observed between the RelyX Unicem and AH Plus ($p > 0.05$). The good bond strength results obtained for RelyX Unicem observed in the present study was also found in a previous study using the coronal dentin surface during restorative procedures.[26] The intense chemical interaction of RelyX Unicem with hydroxyapatite seems to be clinically relevant and explains the good mechanical properties of the product.[15] These results could open a new gateway for the use of RelyX Unicem during the root canal procedures.

Variations in particle size present in endodontic sealers are of great importance regarding mechanical characteristics, such as tensile bond strength, shear bond strength and marginal adaptation to dentin. The inorganic particles have a size distribution within a few microns (Figure 2a).[15] In the present study, only the middle portion of the roots was used in the push-out test because these areas have generally more favorable conditions for adhesion of root canal sealers than the apical portion. Future studies, however, should evaluate RelyX Unicem in the apical portion of the root and in other conditions, such as different chemo-mechanical preparation.

Some concerns regarding the use of this material as an endodontic sealer are related to possible difficulties in removing the sealer from the root canal, which may be a problem for endodontic retreatment. However, this cement is actually indicated for post cementation and can be easily removed when necessary. In fact, post cementations are performed in the cervical and medium third, which are more accessible areas during endodontic retreatment when compared to the apical portion of root canal. If used as an endodontic sealer, the apical portion will be filled with this material and for this reason, future studies should be aimed to evaluate the ability to remove this material from the apical portion during endodontic retreatement.

The result of the Resilon group was in disagreement with a previous study that found significantly higher mean bond strength to root dentin in the Resilon with Epiphany group in comparison with the gutta-percha and Grossman sealer group.[27] A potential factor that may account for this dissimilarity is the zinc oxide-eugenol component present in Kerr Pulp Canal sealer.[27] On the other hand, no substantial bond strength was found between AH-Plus and Gutta-percha pellets. The bond strength was measured only between gutta-percha and the sealer.[27] The idea of monoblock formation seems realistic nor only for Resilon but also for gutta-percha.[27]

Chlorhexidine gluconate (CHX) has been suggested as an alternative irrigant solution in endodontics. CHX has good antimicrobial activity and acts on the cell walls of microorganisms causing disarrangement in the intracellular components. Previous studies demonstrated that this solution associated to 17% EDTA significantly increased bond strength to root dentin when compared to the association of sodium hypochlorite and 17% EDTA.[22,28-30] Therefore, the association of CHX and 17% EDTA was selected as irrigant solution in the present study.

Conclusions

Under the conditions of this study, it can be concluded that Epiphany showed lower bond strength to root dentin than AH Plus and RelyX Unicem. From a clinical point of view, the present results are favorable for the RelyX Unicem and AH Plus. However, future studies should be conducted with RelyX Unicem evaluating its biocompatibility, bacterial leakage, solubility and removability in cases of retreatment.

Conflict of Interest: No potential conflict of interest relevant to this article was reported.

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