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Bonding self-adhesive resin cements to glass fibre posts: to silanate or not silanate?

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Abstract

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Aim To evaluate the bond strength of self-adhesive resin cements (SARCs) to glass fibre posts with or without a silane coupling agent.

Methodology The SARCs tested were: Maxcem Elite (MXE; Kerr), RelyX Unicem clicker (UNI; 3M ESPE), seT capsule (SET; SDI), and SmartCem 2 (SC2; Dentsply Caulk). The conventional cement RelyX ARC (ARC; 3M ESPE) was evaluated as a reference. Rectangular-shaped flat posts were obtained (Angelus). After silanizing or not the posts, resin cement cylinders were built on the post surfaces. The cylinders were tested in shear after 24 h. Bond strength data were submitted to two-way ANOVA and Student–Newman–Keuls' test (5%). Failure modes were classified under magnification as adhesive failure, mixed failure involving the cement or mixed failure involving the post.

Results For ARC, MXE and SET, the silanated groups had higher bond strengths. For SC2 the silane had no influence, while for UNI silanization decreased the bond strength. The conventional ARC had the lowest bond strength when the posts were not silanated; UNI showed the highest values. When the posts were silanated, SET had the highest values, followed by MXE, ARC and SC2; UNI had the lowest values. A predominance of adhesive failures was detected for all groups, with higher number of mixed failures when the posts were silanated.

Conclusion As the silane impaired or generally had no effect on the bond strength of SARCs to the glass fibre posts, and also as the bond strength of all SARCs was higher than the conventional cement when the posts were not silanated, it seems that silanization of glass fibre posts is not necessary when SARCs are used.

Keywords: bond strength, glass fibre posts, self-adhesive resin cements, silane coupling agent.

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Introduction

The use of resin cements to lute glass fibre posts into root canals is a common clinical procedure. Non-adhesive cementation has been shown to be less reliable for withstanding functional forces than adhesive approaches (Naumann *et al.* 2008). In the last few years, self-adhesive resin cements (SARCs) were introduced in an endeavour to simplify the luting procedures by eliminating the etching, priming and bonding steps. The adhesive properties of SARCs are attributed to acidic monomers that demineralize and simultaneously infiltrate the substrate. Secondary reactions have been suggested to provide additional chemical bonding to tooth tissues (Gerth *et al.* 2006).

One controversial issue regarding the adhesive cementation of fibre posts is the use of silane coupling agents. Organosilanes are bifunctional molecules with one end of the molecule capable of reacting with the inorganic glass fibre and the other copolymerizing with the organic resin. Previous studies report contrasting results regarding the effectiveness of silane coupling agents in enhancing the bonding of resin cements to glass fibre posts (Goracci *et al.* 2005, Perdigao *et al.* 2006, D'Arcangelo *et al.* 2007, Wrbas *et al.* 2007). The association of SARCs with silane agents, on the other hand, has been seldom evaluated (Rathke *et al.* 2009).

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Theoretically, the cementation protocol of fibre posts should not change when using SARCs, i.e. the application of silane is recommended. Recently, phosphate monomer resin cements have been indicated for bonding to polycrystalline ceramics (Mirmohammadi *et al.* 2010). Although not yet investigated, there is a potential chemical interaction between the phosphate ester groups of acidic functional monomers and the silicon oxides on the surface of the glass fibres. The use of silane, thus, could be unnecessary, provided a proper chemical bond occurs, although this effect is still unknown.

The aim of this study was to evaluate the bond strength of four SARCs to glass fibre-reinforced posts with or without silane coupling agent in an attempt to determine whether or not the posts should be silanated when using SARCs. The hypothesis tested was that the silane treatment would increase the bond strength of all resin cements to the posts.

Materials and methods

Four SARCs were tested: Maxcem Elite (MXE; Kerr, Orange, CA, USA), RelyX Unicem clicker (UNI; 3M ESPE, St Paul, MN, USA), seT capsule (SET; SDI, Bayswater, Victoria, Australia), and SmartCem 2 (SC2; Dentsply Caulk, Milford, DE, USA). The conventional dual-cure resin cement RelyX ARC (ARC; 3M ESPE) was tested as a reference. The constituents of the materials are shown in Table 1. Customized rectangular (6 × 5, 2 mm thick) glass fibre-reinforced epoxy resin posts were obtained from Angelus (Londrina, PR, Brazil) and embedded in epoxy resin. The bar geometry was important to obtain specimens on a flat post surface. The specimens were ultrasonically cleansed in distilled water for 10 min and dried with compressed air.

The specimens for the bond strength test were obtained following a detailed experimental set-up described elsewhere (Moraes *et al.* 2008). Briefly, 0.5-mm-thick elastomer moulds with cylinder-shaped orifices (diameter 1.2 mm) were placed onto the post surfaces. In half the number of specimens, a pre-hydrolyzed silane agent (Angelus) was applied to the post surface and air-dried for 60 s before positioning the elastomer mould. Equal volumes of base and catalyst pastes of the cements were mixed for 10 s; the capsules of SET were mixed for 10 s using the Ultramat S mixer (SDI). After manipulation, the orifices were filled with the resin cement and the moulds covered with a polyester strip and a glass slide. The samples were submitted to a constant and uniform 500 g cementation load for 3 min. This procedure was important to simulate the clinical luting procedure and allow effective contact between the cement and post. The specimens were photoactivated for 30 s using a light-emitting diode unit (Radii; SDI) with 600-mW cm^{-2} irradiance.

The samples were stored in distilled water at 37 °C, for 24 h. For the shear test, a thin steel wire (diameter 0.2 mm) was looped around each cylinder and aligned with the bonding interface. The test was conducted on a mechanical testing machine (DL500; EMIC, São José dos Pinhais, PR, Brazil), at a cross-head speed of 0.5 mm min^{-1} until failure. Bond strength values were calculated in MPa. For each material, 40 specimens were tested, i.e. 20 silanated and 20 not silanated. Data were submitted to two-way ANOVA (post treatment versus material). All pairwise multiple comparison procedures were performed by the Student–Newman–Keuls' method ($P < 0.05$). The fractured specimens were examined under optical microscopy at a 100× magnification. Modes of failure were classified as adhesive failure, mixed failure involving the resin cement (remnants of cement in the post surface) or

Table 1 Constituents of the resin cements tested and means (SD) for bond strength

Material	Constituents*	Bond strength, MPa	
		Non-silanated	Silanated
Maxcem Elite	TEGDMA, inert mineral fillers, ytterbium fluoride	5.9 (2.0)^{B,c}	11.2 (3.4)^{A,b}
RelyX Unicem	TEGDMA, substituted dimethacrylate, methacrylated phosphoric acid esters, glass powder, silica	15.2 (2.8)^{A,a}	9.0 (3.6)^{B,c}
seT	Acidic monomer, UDMA, F-Al-Si glass particles	6.9 (1.4)^{B,b}	13.1 (2.4)^{A,a}
SmartCem 2	UDMA, urethane modified Bis-GMA, TEGDMA, Ba-B-F-Al-Si glass particles, amorphous silica	7.8 (1.4)^{A,b}	9.6 (3.3)^{A,bc}
RelyX ARC	TEGDMA, Bis-GMA, functionalized dimethacrylate polymer, ceramic, silica	3.8 (1.4)^{B,d}	9.6 (2.5)^{A,bc}

Distinct capital letters indicate differences in each row; distinct lowercase letters indicate differences in each column. *As disclosed by the manufacturers.

mixed failure involving the post (small portion of the post is removed).

Results

Results for the bond strength test are shown in Table 1. The factors 'post treatment' and 'material' were significant, as well as their interaction ($P < 0.001$). For all conditions, the power of the test was = 1. For ARC, MXE and SET, the silanated groups had significantly higher bond strengths than the non-silanated samples ($P < 0.001$). For SC2, the silane had no significant influence on bond strength ($P = 0.094$). However, for UNI, the silanated group had significantly lower bond strength compared with the non-silanated specimens ($P < 0.001$).

Comparing the resin cements, the conventional ARC had significantly lower bond strength than all the self-adhesive materials when the posts were not silanated ($P < 0.001$); UNI showed the highest values, whilst MXE, SC2 and SET had intermediary values. When the posts were silanated, SET had the highest values, followed by MXE, ARC and SC2, whilst UNI had the lowest bond strength values. The distribution of failures modes is presented in Fig. 1. A predominance of adhesive failures was detected for all groups, irrespective of the resin cement or post treatment. However, a greater number of mixed failures involving the resin cement were detected when the posts were silanated, except for UNI.

Discussion

The present results reveal that when silane was not applied, the conventional resin cement RelyX ARC had lower bond strength to the fibre posts as compared with all other SARCs tested. This finding indicates that a chemical reaction of SARCs with glass fibre posts may indeed occur, probably relying on an ionic interaction between the phosphate ester groups with the oxides on the surface of the fibres. It has been shown that functional groups capable of releasing one or more protons, such as phosphate groups, may bond to metal oxides (Yoshida *et al.* 1999, 2006, Almilhatti *et al.* 2009). The higher bond strength of SARCs may also rely on superficial etching of the post by the acidic monomers.

As mentioned before, silanization would be unnecessary if a true chemical bond of SARCs was established with the glass fibres. The present results indicate that the use of silane actually reduced the bond strength of UNI to the posts. Therefore, the hypothesis tested is rejected. One possible explanation for this result is the silane layer might restrict the interaction of the phosphate monomers with the glass fibres. However, it is expected the covalent linkage of the methacrylate monomers with silane to be stronger than the ionic bond with the glass fibres. Another explanation for the lower bond strength of UNI to silanated surfaces might be related to a higher polarity of this cement as compared with the other materials. As silanization

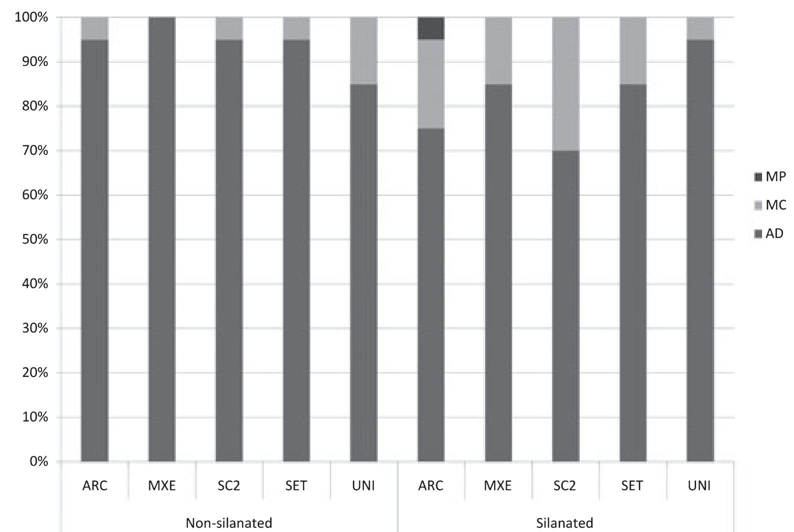

Figure 1 Distribution of failure modes among groups (MP, mixed failure involving the post; MC, mixed failure involving the cement; AD, adhesive failure).

renders the post surface non-polar, the wetting of polar cement would be hindered. This might also explain why silanization had no significant effect on the bond strength of SC2 to the posts.

Another factor that may have interfered with the bonding of UNI to the silanated post surfaces is the fact that this cement is capable of neutralizing its pH in the first 48 h of the setting reaction, whereas other SARCs may still have pH values <4 (Han *et al.* 2007). Constant low pH values may have negative effects on the bonding of SARCs to the tooth structure, but may have favoured the bonding of these cements to the silanated surfaces. In this case, it is speculated that activation of the silane agent is enhanced by acid hydrolysis promoted by the low pH values of the SARCs, improving the formation of siloxane bonds with the glass surface (Foxton *et al.* 2003). For MXE and SET, on the other hand, the use of silane increased the bond strengths. Therefore, it is clear that the bonding mechanism of SARCs to fibre posts is material dependent.

SARCs simplify the luting procedures by eliminating the application of bonding agent to the root canal. From a clinical standpoint, it would be interesting to eliminate the step of post silanization as well. However, when an organosilane is applied, its alkoxy groups are hydrolyzed into silanol groups to bond with silica through the formation of siloxane bonds (Debnath *et al.* 2003, Matinlinna *et al.* 2004). Another effect is the improvement in surface wettability by silane coating: as an intimate contact between the interfacing materials is established, van der Waals' forces may become effective providing a physical adhesion, which contributes to the chemical reactions (Pape & Plueddemann 1991). The organic matrix of the cement then copolymerizes with the methacrylate group of the silane, increasing the bond strengths.

Silanization also increased the number of mixed failures involving the resin cement. This is the further evidence of the enhanced bonding provided by silane. This result is in line with that from D'Arcangelo *et al.* (2007), who showed that non-silanated posts had a relatively smooth surface area, which limited mechanical interlocking between the post and resin cement. Goracci *et al.* (2005) reported that when posts were silanated, only a few modest changes in fracture modes were observed, but the retentive values were significantly higher. For UNI, more adhesive failures were observed when the silane was used as compared with the non-silanated group, reinforcing the chemical interaction of this cement with the post surface.

The bonding of resin cements to fibre posts is usually tested under tensile loading. The present study shows a bond testing set-up in which the bond strength of the cement to the post was tested in shear loading, which may represent the actual forces taking place within the confines of the root canal. This is a simple test, which may provide useful, consistent bonding assessment. The set-up does not take into account the effect of the polymerization stress state in the canal, but the focus was kept on the bond strength of the cements to the post. Under clinical conditions, optimal adhesion between the luting agent and post surface is crucial for the retention of the post into the root canal. Radovic *et al.* (2008) reported that etch-and-rinse or self-adhesive bonding approaches may provide comparable adhesion with root canal dentine. The present results indicate, however, that depending on the material, the bonding mechanism of SARCs may be hindered or improved by application of silane; therefore, care should be taken when combining silane and SARCs. Other SARCs should also be tested, as the combination post system/resin cement may influence the bond strengths (Dimitrouli *et al.* 2011).

Conclusion

As the silane impaired or generally had no significant effect on the bond strength of SARCs to the glass fibre posts, and also as the bond strength of all self-adhesive cements was higher than the conventional cement when the posts were not silanated, it seems that silanization of glass fibre posts is not necessary when SARCs are used.

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References

Almilhatti HJ, Giampaolo ET, Vergani CE, Machado AL, Pavarina AC, Betiol EA (2009) Adhesive bonding of resin composite to various Ni-Cr alloy surfaces using different metal conditioners and a surface modification system. *Journal of Prosthodontics* **18**, 663–9.

D'Arcangelo C, D'Amario M, Prosperi GD, Cinelli M, Giannoni M, Caputi S (2007) Effect of surface treatments on tensile bond strength and on morphology of quartz-fiber posts. *Journal of Endodontics* **33**, 264–7.

Debnath S, Wunder SL, McCool JI, Baran GR (2003) Silane treatment effects on glass/resin interfacial shear strengths. *Dental Materials* **19**, 441–8.

Dimitrouli M, Günay H, Geurtsen W, Lührs AK (2011) Push-out strength of fiber posts depending on the type of root canal filling and resin cement. *Clinical Oral Investigations* **15**, 273–81.

Foxton RM, Nakajima M, Hiraishi N *et al.* (2003) Relationship between ceramic primer and ceramic surface pH on the bonding of dual-cure resin cement to ceramic. *Dental Materials* **19**, 779–89.

Gerth HU, Dammaschke T, Zuchner H, Schafer E (2006) Chemical analysis and bonding reaction of RelyX Unicem and Bifix composites – a comparative study. *Dental Materials* **22**, 934–41.

Goracci C, Raffaelli O, Monticelli F, Balleri B, Bertelli E, Ferrari M (2005) The adhesion between prefabricated FRC posts and composite resin cores: microtensile bond strength with and without post-silanization. *Dental Materials* **21**, 437–44.

Han L, Okamoto A, Fukushima M, Okiji T (2007) Evaluation of physical properties and surface degradation of self-adhesive resin cements. *Dental Materials Journal* **26**, 906–14.

Matinlinna JP, Lassila LV, Ozcan M, Yli-Urpo A, Vallittu PK (2004) An introduction to silanes and their clinical applications in dentistry. *The International Journal of Prosthodontics* **17**, 155–64.

Mirmohammadi H, Aboushelib MN, Salameh Z, Feilzer AJ, Kleverlaan CJ (2010) Innovations in bonding to zirconia based ceramics: part III. *Phosphate monomer resin cements. Dental Materials* **26**, 786–92.

Moraes RR, Correr-Sobrinho L, Sinhoreti MA, Puppin-Rontani RM, Ogliari FA, Piva E (2008) Light-activation of resin cement through ceramic: relationship between irradiance intensity and bond strength to dentin. *Journal of Biomedical Materials Research Part B: Applied Biomaterials* **85**, 160–5.

Naumann M, Sterzenbach G, Rosentritt M, Beuer F, Frankenberger R (2008) Is adhesive cementation of endodontic posts necessary? *Journal of Endodontics* **34**, 1006–10.

Pape PG, Plueddemann EP (1991) Methods for improving the performance of silane coupling agents. *Journal of Adhesion Science and Technology* **5**, 831–42.

Perdigao J, Gomes G, Lee IK (2006) The effect of silane on the bond strengths of fiber posts. *Dental Materials* **22**, 752–8.

Radovic I, Mazzitelli C, Chieffi N, Ferrari M (2008) Evaluation of the adhesion of fiber posts cemented using different adhesive approaches. *European Journal of Oral Sciences* **116**, 557–63.

Rathke A, Haj-Omer D, Muche R, Haller B (2009) Effectiveness of bonding fiber posts to root canals and composite core build-ups. *European Journal of Oral Sciences* **117**, 604–10.

Wrbas KT, Schirrmeister JF, Altenburger MJ, Agrafioti A, Hellwig E (2007) Bond strength between fibre posts and composite resin cores: effect of post surface silanization. *International Endodontic Journal* **40**, 538–43.

Yoshida K, Kamada K, Atsuta M (1999) Adhesive primers for bonding cobalt-chromium alloy to resin. *Journal of Oral Rehabilitation* **26**, 475–8.

Yoshida K, Tsuo Y, Atsuta M (2006) Bonding of dual-cured resin cement to zirconia ceramic using phosphate acid ester monomer and zirconate coupler. *Journal of Biomedical Materials Research Part B: Applied Biomaterials* **77**, 28–33.