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ORIGINAL ARTICLE

The influence of four dual-cure resin cements and surface treatment selection to bond strength of fiber post

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In this study, we evaluate the influence of post surface pre-treatments on the bond strength of four different cements to glass fiber posts. Eighty extracted human maxillary central incisors and canines were endodontically treated and standardized post spaces were prepared. Four post pre-treatments were tested: (i) no pre-treatment (NS, control), (ii) sandblasting (SA), (iii) silanization (SI) and (iv) sandblasting followed by silanization (SS). Per pre-treatment, four dual-cure resin cements were used for luting posts: DMG LUXACORE Smartmix Dual, Multilink Automix, RelyX Unicem and Panavia F2.0. All the specimens were subjected to micro push-out test. Two-way analysis of variance and Tukey *post hoc* tests were performed (α=0.05) to analyze the data. Bond strength was significantly affected by the type of resin cement, and bond strengths of RelyX Unicem and Panavia F2.0 to the fiber posts were significantly higher than the other cement groups. Sandblasting significantly increased the bond strength of DMG group to the fiber posts.

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INTRODUCTION

The increasing use of fiber posts for restoring endodontically treated teeth with the loss of coronal tooth structure has been proved to be effective and the post in combination with resin cement and restorative material can form a structurally and functionally homogeneous complex with root dentin.[1] Fiber posts are promoted due to their excellent biocompatibility, esthetic and mechanical properties. Fiber post systems that have a similar modulus of elasticity to root dentin are generally preferred to metal post systems and show less microleakage under dynamic loading and fracture patterns availing a retreatment of the fracture.[2–3] Numerous *in vitro* studies have been done to investigate different adhesive systems and pre-treatments of dentin or posts for improving bond strength, and have shown that most failures of fiber posts are caused by bond failure between the post and the dentin.[4–5] Therefore, it is crucial to maximize the bond strength between the resin and root dentin, and between the resin and fiber post.

Many *in vitro* studies have investigated a variety of factors that affect the bonding strength of the fiber post to root dentin. These factors are composed of diameter, length, shape, the surface treatment of the post, cement type, canal region and so on.[6–10] A study performed by Monticelli *et al.*[11] concluded that surface conditioning improves fiber post bonding properties and bond strength of pre-treated fiber posts to restorative materials is satisfactory. Many kinds of pre-treatment have been proposed to enhance the bond strength of fiber posts, such as etching, silanization and air abrasion. Silanization has the advantage of being a convenient chairside operation, but its effect on bond strength was inconclusive. Several studies concluded that the bond strengths between the post and resin cement can be improved by silanizing the post in advance.[12–13] Other studies reported that the use of a silane coupling agent alone did not increase the bond strengths.[14–17] One study reported that the use of a silane coupling agent in combination with air abrasion did not increase the bond strength to the post when luting with resin cement.[18]

Another prospectively efficient pre-treatment is air abrasion. Air abrasion roughens the surface of the fiber post, increases the surface area for bonding and enhances the strength of interlocking.[19–20] Some studies showed that air abrasion on the surface of fiber posts increased the bond strength of posts adhesively luted with dual cure resin cement, especially the combination of air abrasion followed by post silanization.[21–22] However, there is no consensus on the use of air abrasion.[23] Air abrasion can affect both the fibers and matrix; it may reduce bond strength by reducing the quality of the interface between the post and root dentin.

Some studies have reported that the type of adhesive system and root region had a significant influence on the bond strength of the adhesive luting fiber posts.[16,24] Retention of the fiber post to root canal dentin is a function of interlocking, chemical bonding and the applied frictional force which can be reflected by the push-out test.[25] Goracci *et al.*[26] modified the traditional push-out test, and named this the ‘thin-slice’ push-out test and called the result ‘micro push-out bond strength’.[26] In the ‘thin-slice’ push-out test, each root may supply 6–8 specimens, and stress distribution was shown to be more homogeneous in every specimen.

The objective of this *in vitro* study was to evaluate the influence of post surface pre-treatment on the bond strength of four different cements to a glass fiber post tested in a micro push-out test. If

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push-out bond strength is not significantly affected by post surface pre-treatment and different dual cure cements, then this is the void hypothesis.

MATERIALS AND METHODS

Eighty extracted human maxillary central incisors and canines (we obtained informed consent from the donors of the teeth and the experimental studies were approved by the Review Committee for the Use of Human or Animal Subjects of Jilin University) were stored in 0.2% chloramines solution at 4 ℃ for 3 months and then endodontically treated. The canals were prepared with K-files (Kerr, Romulus, MI, USA) using passive step back technique to size #30. The root canals were irrigated with 5 mL 2% chlorhexidine solution followed by 5 mL 17% ethylene diaminetetraacetic acid solution and dried with absorbent paper points (Henan Baistra Industries, Zhengzhou, China). Then they were obturated with gutta-percha point (25–40#; Dentsply Maillefer, Ballaigues, Switzerland) and root canal sealer (Pulpdent, Watertown, MA, USA). The crown of each tooth was removed at 2 mm above the cementoenamel junction with a 0.15 diamond-wafering blade in an Isomet 1 000 slow-speed saw (Isomet 1000TM; Buehler, Lake Bluff, IL, USA) with a continuous supply of distilled water for cooling. Then standardized post spaces were prepared by Pesso-reamers and Glassix glass fiber special burs (Harald Nordin SA, Chailly/Montreux, Switzerland) to 9.5 mm deep, as measured from the cementoenamel junction on the lingual aspect of the tooth. Following post space preparations, the roots were randomly divided into four groups of 20 teeth each, and roots in each group were randomly divided into four subgroups.

Eighty-four radiopaque, translucent Glassix glass fiber composite posts (size no. 4, diameters of 1.5 mm; Harald Nordin SA, Chailly/Montreux, Switzerland) were used for this investigation. Specimens were divided into four groups (n=21) and each group received different surface treatments. In group NS (no surface pre-treatment), no surface pre-treatment was carried out (control group). In group SA (air abrasion), the posts were sandblasted with 50 µm aluminum oxide particle (Microetcher II; Danville Engineering, San Ramon, CA, USA) for 5 s. These particles were ejected from a distance of about 1 cm perpendicular to the post surface at the pressure of 0.28 MPa. In group SI (silanization), the posts were pre-treated with a silane coupling agent Monobond-S (Ivoclar-Vivadent, Schaan, Liechtenstein) using a brush. After being coated with the silane coupling agent for 60 s at room temperature, the posts surfaces were dried. In group SS (air abrasion plus silanization), the glass fiber posts were pre-treated with 50 µm aluminum oxide particle for 5 s followed by the application of a silane coupling agent. Before adhesive procedures, one post of each group (NS, SA, SI and SS) was randomly chosen to be observed by the XL30-FESEM (field emission scanning electron microscope; Philips, Eindhoven, Netherlands). These specimens were evaluated for changes of post surfaces. Then 20 posts in each group were randomly divided into four subgroups, assigned to four dual cure resin cements and bonded into the post spaces: DMG (DMG LuxaCore Smartmix Dual, Hamburg, Germany), Multilink Automix (Ivoclar-Vivadent, Schaan, Lichtenstein), RelyX Unicem (3M ESPE, St Paul, MN, USA) and Panavia F2.0 (Kuraray Medical, Okayama, Japan). All resin cements and corresponding adhesive systems used in this study are listed in Table 1.

Then resin cements were light polymerized for 40 s with a light-emitting diode (LED) curing light (1 000 $mW\cdot cm^{-2}$ output) (3M ESPE, St Paul, MN, USA) directly at a distance of 2 mm. Before performing the micro push-out bond strength test, specimens were stored in distilled water for 24 h at 37 ℃. The coronal part of the 80 roots were sectioned perpendicular to the long axis with an Isomet 1 000 slow-speed saw to make (1.00±0.05) mm-thick slices, and each root provided 6–7 slices. All the slices were numbered and marked indelibly, and their thickness was measured by an ORIENTOOLS digital caliper (Ningbo Joro Electronic, Ningbo, China) with an accuracy of 0.001 mm. The micro push-out tests were performed at a speed of 0.05 $mm\cdot min^{-1}$ by a universal testing machine (1121; Instron, Danvers, MA, USA). The slice was placed on the test machine and the center of the post transect was taken to the center of the push-out pin (diameter: 1.4 mm) to ensure the pin did not contact the surrounding root dentin. Until the posts separated from the root dentin, the load at the peak point of stress–time curve was taken as the point of load failure, and the value was recorded in Newton. The load value recorded in Newton divided by the area of the bonded interface is the bond strength recorded in MPa. The micro push-out bond strength was calculated using the following equation:

$$A = F/\pi dh$$

where A (in MPa) is the calculated micro push-out bond strength, F is the load at failure (in N), π is the constant 3.14, d is diameter of the post and h is the thickness of the slice in mm. All operations were applied by the same operator to ensure standardization.

Tested specimens were observed in fracture mode of bonding interface by the XTL-33 stereo microscope stereomicroscope (×40) (Shanghai Pudan Optical Instrument, Shanghai, China). The fracture mode is divided into the following categories: (i) cohesive failure between adhesive material and the root canal; (ii) cohesive failure between adhesive material and fiber post; (iii) failure within the adhesive material; (iv) failure within the fiber post; and (v) failure within dentin.

Two-way analysis of variance was performed for the comparison of different resin cements and type of pre-treatments of fiber posts. *Post*

Table 1 Luting agents and corresponding adhesive systems used in this investigation

Dual-cure resin cements	Manufacturer	Composition of resins cements	Composition of primers
DMG LuxaCore Smartmix Dual (Batch No. 660718)	DMG, Hamburg, Germany	Acrylic resin, glass powder, silica, urethane dimethacrylate, aliphatic dimethacrylate, aromatic dimethacrylate	No primer available
Multilink Automix (Batch No. N64524)	Ivoclar-Vivadent, Schaan, Liechtenstein	HEMA, dimethacrylate, barium glass, ytterbium silica, trifluoride	Water, HEMA, phosphoric acid acrylate, polyacrylic acid-modified methacrylate resin
Panavia F2.0 (Batch No. 71128)	Kuraray, Osaka, Japan	MDP, dimethacrylate, barium glass powder, sodium fluoride, silica, amine, benzoyl peroxide, sodium aromatic sulfinate	HEMA, 10-MDP, N-methacryl, sodium benzene sulfinate, 5-aminosalicylic, N,N-diethanol p-toluidine, water
RelyX Unicem (Batch No. 426768)	3M ESPE, St Paul, USA	Silica, calcium hydroxide, methacrylated phosphoric ester, glass, dimethacrylate, acetate	No primer available

HEMA, 2-hydroxyethyl methacrylate; MDP, 10-methacryloyloxydecyl dihydrogen phosphate.

Table 2 Mean bond strength values (±s.d.) of investigated post types to different resin cements (*n*=30–35) /MPa

Pre-treatments	DMG LUXACORE Smartmix Dual	Multilink Automix	Panavia F2.0	RelyX Unicem	Mean method
NS	8.44 ± 2.38^{Aa}	8.79 ± 4.11^{ABa}	11.77 ± 2.04^{Ba}	14.77 ± 2.77^{Ca}	11.00 ± 3.94^{a}
SA	13.97 ± 3.50^{Ab}	9.37 ± 2.59^{Ba}	14.37 ± 3.67^{Aa}	16.89 ± 3.88^{Aa}	13.65 ± 4.52^{b}
SI	9.46 ± 1.66^{Aa}	9.83 ± 5.90^{Aa}	16.40 ± 5.92^{Ba}	15.29 ± 3.08^{Ba}	12.75 ± 6.42^{ab}
SS	13.23 ± 5.09^{ABb}	8.95 ± 2.39^{Ba}	12.63 ± 3.65^{ABa}	15.33 ± 4.77^{Aa}	12.53 ± 4.80^{ab}
Mean cement	11.27 ± 5.21^{A}	9.22 ± 4.09^{A}	13.52 ± 4.65^{B}	15.54 ± 3.83^{B}	

s.d., standard deviation; NS, no pre-treatment; SA, sandblasting; SI, silanization; SS, sandblasting followed by silanization.
The same superscripted letters indicate no significant differences ($P<0.05$).
^{a,b,c} Statistically significant differences between pre-treatment methods (within the same cement).
^{A,B,C} Statistically significant differences between cements (within the same pre-treatment method).

hoc comparisons (Tukey B test) were applied. The α-level of significance was 0.05 (two-sided) for all testing. All analyses were performed using SPSS16.0 (SPSS GmbH, Munich, Germany).

RESULTS

Each group was normally distributed. The two-way analysis of variance showed a significant effect for cement type ($F=13.177$; $P<0.001$). There was also a significant interaction between cement and pre-treatment ($F=7.730$; $P<0.001$). Comparing the pooled data of the four cements (Table 2), Panavia F2.0 and RelyX Unicem proved to have significantly higher mean micro push-out bond strength values than DMG LUXACORE Smartmix Dual and Multilink Automix ($P<0.001$). The pooled data of the pre-treatments (Table 2) show that air abrasion of the posts resulted in significantly higher micro push-out bond strength than the control group ($P=0.020$). There were no significantly differences between the pre-treatments. There were no statistically significant differences between pre-treatments for Multilink Automix, Panavia F2.0 and RelyX Unicem. For DMG LUXACORE Smartmix Dual, air abrasion of the post resulted in higher micro push-out bond strengths than the control group ($P=0.008$). There were no further significant differences. There was a tendency for the pre-treatment groups to have higher of micro push-out bond strength than the control group, but this was not statistically significant. In the NS groups and SS groups, both RelyX Unicem and Panavia F2.0 had higher micro push-out bond strength values than DMG LUXACORE Smartmix Dual ($P<0.05$). RelyX Unicem had higher micro push-out bond strength values than Panavia F2.0 ($P=0.047$) and Multilink Automix ($P=0.005$). There were no further significant differences. In the air abrasion group, all the other subgroups had higher micro push-out bond strength values than Multilink Automix ($P<0.05$). In the SS groups, RelyX Unicem had, higher bond strength values compared to Multilink Automix ($P=0.001$) again.

Cohesive failure within the fiber posts and resin cement were not found in each experimental group. Major fracture mode of RelyX Unicem group was cohesive failure within dentin. Adhesively failure

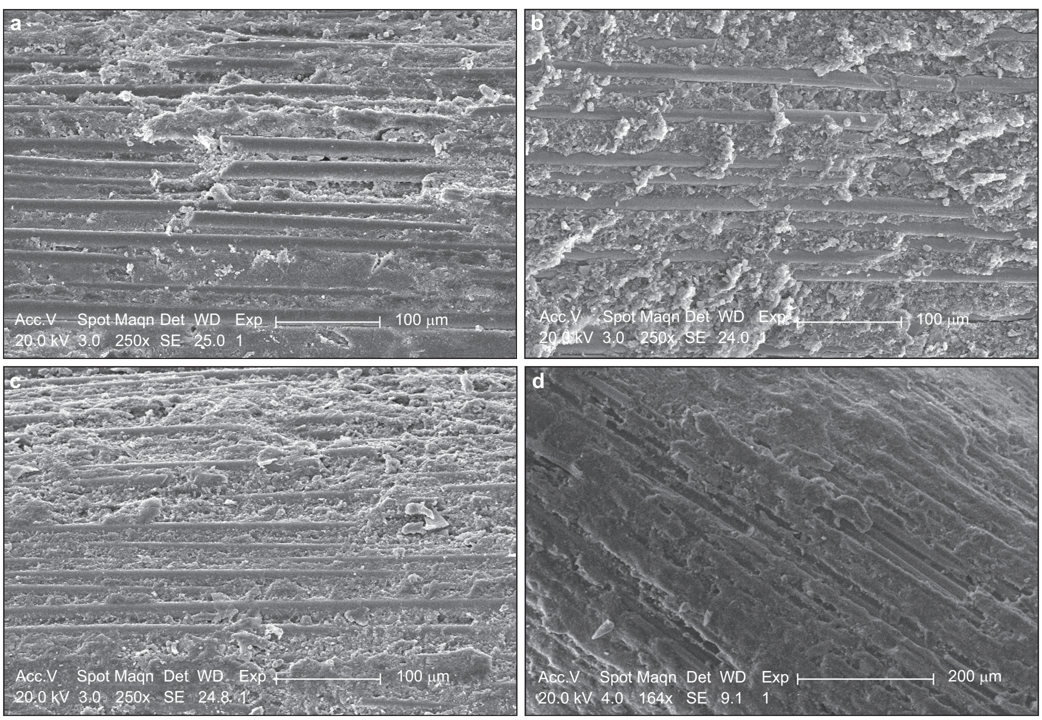

Figure 1 Microstructure of glass fiber post. (**a**) Without pre-treatment; (**b**) with pre-treatment of sandblasting; (**c**) with pre-treatment of sandblasting followed by silanization; (**d**) with pre-treatment of silanization.

between the cement/dentin interface or in a mixed way of these two failure modes was also observed. Major fracture mode of the other groups was adhesive failure between adhesive material and fiber post.

The microstructure of glass fiber posts with the pre-treatment of air abrasion (Figure 1b) was rougher than the control group (Figure 1a). Microstructure of glass fiber post with pre-treatment of sandblasting followed by silanization (Figure 1c) showed little difference between the microstructure of glass fiber posts without pre-treatment. Microstructure of glass fiber posts with the pre-treatment of silanization (Figure 1d) had less fiber exposed than the control group.

DISCUSSION

Micro push-out bond strength is essentially a type of shear bond strength which depends on the degree and stability of interfacial micromechanical interlocking and chemical adhesion. In this study, the influences of post surface pre-treatment on the bond strength of four different cements with a glass fiber post were evaluated using a push-out test. For the push-out test, each root can provide 6–8 specimens, and stress distribution is more homogeneous in every specimen.[26] Therefore, this design was chosen for the present study.

Many *in vitro* studies have investigated and confirmed that a variety of factors affect the bonding strength of fiber post to root dentin. These factors are composed of diameter, length, shape, the surface treatments of the post[16] and root dentin, the type of post,[6] canal regions[6–7] and the type and thickness of luting agent used.[7–8] Various surface pre-treatment methods have been used to improve bond strength with the fiber post, and they can be divided into three categories: (A) increasing the roughness and bonding area of the fiber post to improve micromechanical interlocking, such as air abrasion and etching;[21] (B) promoting chemical bonding, including several kinds of agents, such as a silane coupling agent and a dentin luting agent; and (C) possessing both functions of A and B, such as the Cojet system.

The mechanical theory of bonding considers that the luting agent must run into the space between post surface and dentin and eliminate the adsorption of air on the interface in order to produce a bonding effect.[27] Therefore, mechanical interlock is an important factor on the bonding interface; the clean surface of fiber posts formed by air abrasion can significantly improve the contact angle of the polymer surface and reduce the interfacial energy of the bonding interface. These effects greatly enhance the bond strength. The sandblasted rough surface of fiber posts exposed more fiber (Figure 1b), increasing the bonding area and forming a good micromechanical interlocking at the same time. In this study, the pooled data of the pre-treatments show that air abrasion of the posts resulted in significantly higher micro push-out bond strength than no pre-treatment of the posts ($P=0.020$).

Surface pre-treatment using a silane coupling agent is convenient and will not change the microstructure and performance on the post surface. The use of a silane coupling agent can form 'molecular bridge' between the interface of inorganic substances and organic substances, increase material surface wettability, connect the two different materials together and effectively improve the interfacial bond strength.[28–31]Although the specific effect of the silane coupling agent in improving bond strengths of many kinds of metal and ceramics has been recognized, whether it improves the bonding strength to fiber posts is not clear. An epoxy resin matrix, as the glass fiber post, is a highly crosslinked polymer without silicate; it is difficult to lute with composite resins and tooth structure.[32] The silane coupling agent only provide chemical and mechanical retention through the chemical reaction with silica in glass fiber and luting agent, and cannot provide a good combination with the epoxy resin matrix.[13,33–34] The most relevant finding of the study was that the retentive strength of fiber posts luted with DMG was significantly enhanced with air abrasion. Because air abrasion fiber posts seem to cause many irregularities on the post's surface, the mechanism most likely involved in the enhancement of post bond. Studies have found that surface characteristics of glass fiber posts vary greatly: some glass fiber posts showed more glass fibers exposed on the surface, while others show less or little.[1] D'Arcangelo *et al.*[35] suggested performing a mild form of sandblasting (50 μm Al_2O_3 particles, 2.0 bar, 10 s, 5 cm) to treat fiber post surface and increase post's mechanical retention without decreasing their flexural properties.[35] Fiber posts might show different surface after pre-treatment. Observably changes were noticed on the post surfaces after every surface treatment. Hydrofluoric etching and use of silane produced modest changes on post surfaces. Exposed fibers appear to be free of damaged on their surfaces.[35] When posts were silanized, part of fibers appeared fractured and little resin matrix was exposed. Etching (hydrofluoric acid 9.5% for 15 s) and sandblasting (50 μm Al_2O_3 particles, 2.0 bar, 10 s, 5 cm) appeared to be more effective than silanization in determining micromechanical retention on a fiber post surface.[36] Hydrofluoric etching made more fractured fibers and exposed resin matrix.[36] Sandblasting made a rough surface on post surfaces. Fibers–resin matrix structure of the whole post length showed notable altered.[35,37]

Some scholars believe that the silane coupling agent can significantly improve the bonding effect on fiber posts.[15,33] One study has shown that the use of a silane coupling agent can increase bond strength of quartz fiber posts, but the effect on glass fiber posts was not obvious.[12] In this study, there is no significant difference between silanization group and the control group. The study reaches the same conclusions. This result may be linked to the different surface compositions between glass fiber posts and quartz fiber posts.

RelyX Unicem is convenient dual-curing resin cement and needs no pre-treatment of porcelain and tooth surface. When it combined with dentin, glass ionomer technology was introduced and a new component methacrylated phosphoric ester was added. Each methacrylated phosphoric ester monomer containing two or more PO_4^{3+} and two C=C double bond. Phosphate and Ca^{2+} tooth surface form stable chemical combinations which increases the adhesive force to tooth tissue. Unsaturated double bond determines highly reactive and highly crosslinked. After polymerization, highly crosslinked structure maintains good mechanical properties of resin cements. Therefore, this study shows that the bond strength of RelyX Unicem was significantly higher than the other resin cements and the major failure mode in this group was failure within dentin. Matrix of RelyX Unicem and Panavia F2.0 contain Bis-GMA. Panavia F2.0 containing MDP, its primer containing MDP and HEMA. MDP can modify the smear layer and result in demineralization of dentin. HEMA can effectively fill the gap between the collagen fibers to form a mixed layer.[37–38]

HEMA is also an important component of Multilink Automix. DMG LUXACORE Smartmix Dual is also a dual-cured resin and can remove the smear layer thoroughly, leading to demineralization of dentin. The permeability and bond strength of the resin were affected by demineralization of dentin.

Failure mode can influence the clinical longevity of a post-core system. Major fracture mode of RelyX Unicem group was cohesive failure within dentin (54.2%). This result can be due to that RelyX Unicem provided the highest bond strengths, which resulted in a highest percentage of cohesive failures within the cement. In general, higher bond strengths lead to a higher percentage of cohesive failures.[34,39]

The posts did not undergo thermal cycles and uploads cycles; we may do follow-up experiments to test bond strength of fiber post in simulated oral environment.

CONCLUSION

Within the limitations of this study, it can be concluded that especially when DMG LUXACORE Smartmix Dual is used, air abrasion of glass fiber posts has a significantly helpful effect on the micro push-out bond strength. Silanization of the post surface has no significant effect on the interfacial bond strength between the post and the resin cement. There was no significant difference in bond strength between the silanization group and the control group. Comparing the pooled data of the four cements, Panavia F2.0 and RelyX Unicem proved to have significantly higher mean micro push-out bond strength values than DMG LUXACORE Smartmix Dual and Multilink Automix.

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