Evaluation of Sealer Remnants Using a Bioceramic Sealer Single-cone Technique after Post Space Preparation and its Influence on the Adhesion of Fibre Posts in vitro

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Objective: *To compare calcium silicate–based endodontic sealer and epoxy resin–based sealer remnants on root canal walls after post space preparation and their influence on the bond strength of fibre posts fixed with a dual-cured resin cement.*

Methods: *Thirty-six extracted single-root mandibular premolars were instrumented and divided randomly into two equal groups with different endodontic sealers. iRoot SP (Innovative BioCeramix, Vancouver, Canada) was employed in the experimental group and AH Plus (Dentsply Sirona, Charlotte, NC, USA) was used in the control group. Sealer remnants were observed under an endodontic microscope after root canal therapy and post space preparation. Fibre posts were fixed with dual-cured resin cement. Specimens were taken at each third of the post space. The push-out bond strength was measured using a universal testing machine and fracture modes were assessed. Statistical analysis was performed using an independent samples t test and one-way analysis of variance.*

Results: *There was no statistically significant difference in bond strength of fibre posts between the control and experimental group ($P > 0.05$); however, sealer remnants were observed in 38.9% of the samples treated with iRoot SP and none of the samples treated with AH Plus. The major fracture mode in samples treated with iRoot SP was adhesive failure between the resin cement and the post, and no adhesive failure between the resin cement and dentine occurred at the site of the sealer remnant. The presence of iRoot SP remnants on the root canal walls after post space preparation did not interfere with bonding.*

Conclusion: *iRoot SP is a viable option for root canal obturation before fibre post cementation.*

Key words: *AH Plus, bond strength, calcium silicate–based sealer, endodontic sealer, fibre post, iRoot SP*

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Fibre posts are recommended for ensuring retention of teeth that have been compromised by extensive coronal destruction and treated endodontically[1,2]. Resin cement is commonly used to fix fibre posts in place; however, one of the main concerns with this technique is the uncertain nature of the bond strength between the fibre post and dentine, which is closely related to the high sensitivity of the adhesive technique[3].

Endodontic sealer is commonly used during root canal obturation; thus, careful consideration of the endodontic sealer used during the obturation process but before fibre post cementation is required, as adhesion of the resin cement to the root dentine may be affected by the endodontic sealer, which may impact retention of the fibre post after treatment[4,5]. It is commonly accepted that eugenol-based sealers should be avoided because the eugenol may inhibit polymerisation of the resin-based cement or modify the surface of the polymerised resin[6], decreasing the bond strength of fibre posts to the root dentine[6-10]. In addition, sealers

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containing calcium hydroxide are difficult to remove from dentinal walls, and can also reduce the bond strength[11,12].

AH Plus (Dentsply Sirona, Charlotte, NC, USA) is an epoxy resin–based sealer that is considered the gold standard due to its excellent adhesive properties, sealing ability and non-interference with the bond strength of fibre posts fixed using adhesive resin cements[13,14]. For this reason, the performance of new endodontic sealers is often compared against that of AH Plus. In recent years, iRoot SP (Innovative BioCeramix, Vancouver, Canada), a calcium silicate–containing, injectable, premixed sealer with good physicochemical and biological properties, has become an increasingly popular alternative sealant for root canal obturation[15-17].

During the setting process, iRoot SP can form tag-like structures consisting of either sealer itself, or crystals formed by the reaction of calcium hydroxide present in iRoot SP with phosphate ions present in the dentine[18-20], producing a high bonding capacity between the dentine and the filling material, which consequently means removal is more difficult[21,22]. It has been shown that iRoot SP can penetrate dentine tubules to a depth of more than 1 mm[6,23], and any sealer remnants in the dentine tubules might affect the bond strength since bonding is mainly based on micromechanical retention through the diffusion of monomers into the dentinal tubule[24]. Besides the sealer in the dentine tubules, large amounts of sealer may still remain on the root canal walls even after fibre post preparation, because the material becomes very hard when set and is difficult to remove[25]. Thus, the influence of sealer remnants on the adhesion of fibre posts must be evaluated.

Few studies have examined the influence of iRoot SP sealer on the adhesion of fibre posts[4,9,10,26]; however, the results were conflicting. Some studies found that fibre posts displayed lower bond strength when iRoot SP was used compared with AH Plus[4,9,10], whereas others found that both sealers showed equal bond strength[26,27]. These conflicting results may be due to the different morphology of the root canals, differences in treatment and the use of different cement resins. Therefore, whether the bond strength of fibre posts can reach the same level of bond strength after using iRoot SP or AH Plus as endodontic sealer remains inconclusive. There have also not been any reports on whether large amounts of sealer remnants on the root canal walls after post space preparation affects the adhesion of fibre posts.

The purpose of this in vitro study was to assess the effect of the calcium silicate–based endodontic sealer iRoot SP on the push-out bond strength of fibre posts cemented with a dual-cured resin cement and observe the relationship between the sealer remnants and the debonding of fibre posts. The null hypothesis was that the type of endodontic sealer would have no influence on the adhesion of fibre posts, even if there might be large amounts of sealer remnants on the root canal wall.

Materials and methods

Experimental design

This study was approved by the Biomedical Ethics Committee of Peking University School of Stomatology under the protocol PKUSSIRB-201734051. The sample size was calculated based on the following formulae: n = f(α, β/2) × 2 × σ2 / d2, where σ is the standard deviation, and f(α, β) = [Φ-1(α) + Φ-1(β)]2, where Φ-1 is the cumulative distribution function of a standardised normal deviate. The power was set at 80% and the level of statistical significance was set at 0.05. The minimum sample size was determined to be 18 samples in each group. Each procedure in the study was performed by a trained operator.

A total of 36 human permanent mandibular premolars extracted for orthodontic reasons were collected and immersed in 0.1% thymol solution. All the selected teeth had a straight root with a single canal with a fully formed apical foramen and were free of caries lesions and cracks (Fig 1a). The teeth were decoronated below the cementoenamel junction with a high-speed bur (TR-13; MANI, Utsunomiya, Japan) under water to obtain a standard root length of 14 mm (Fig 1b). The roots were observed under an endodontic microscope (Carl Zeiss, Gottingen, Germany) to make sure there were no cracks. The initial apical width was no more than the diameter of a size 20 K-file (MANI) in order to reduce the variation in size between the root canals. The diameters (buccal-lingual and mesial-distal) of the root canals at orifice level were measured with digital calipers (Mitutoyo, Kawasaki, Kanagawa, Japan) and the length-width ratio was calculated for each specimen.

The specimens were divided randomly into two groups by way of a coin toss according to different endodontic sealers and corresponding obturation techniques. Group 1 (n = 18, experimental group) used iRoot SP sealer with the single-cone technique (Fig 1c), and Group 2 (n = 18, control group) used AH Plus sealer with the warm vertical condensation technique (Fig 1d).

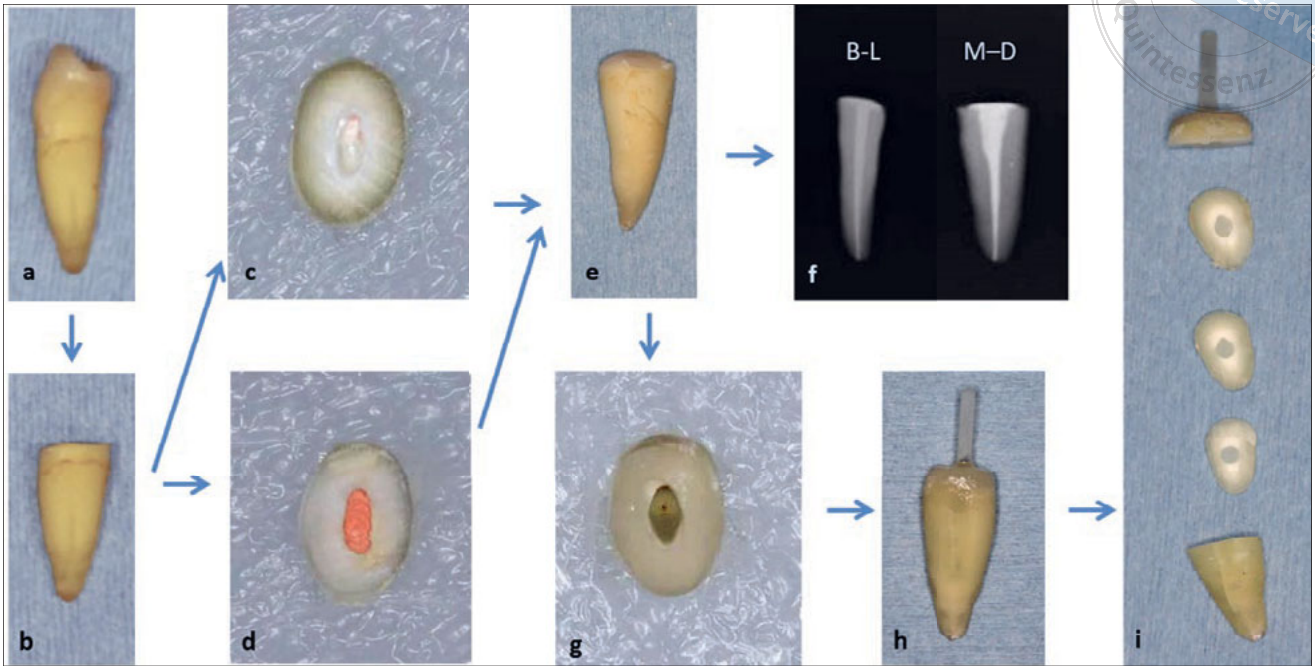

Fig 1 Operation procedure. **(a)** Mandibular premolar; **(b)** decoronated; **(c)** root canal obturation with iRoot SP using the single-cone technique; **(d)** root canal obturation with AH Plus using the warm vertical condensation technique; **(e)** coronal access cavity sealed with resin; **(f)** radiographs showing buccal-lingual (B-L) and mesial-distal (M-D) expansion; **(g)** post space preparation; **(h)** fibre post adhesion; **(i)** three specimens for each tooth.

Both groups had several round (length-width ratio < 1.2) and oval root canals (length-width ratio ≥ 1.2). In Group 1, there were 7 round root canals and 11 oval root canals, and in Group 2, there were 6 round root canals and 12 oval root canals.

Endodontic treatment

The working length was set as 1 mm shorter than the length of a size 10 K-file (MANI) in the tooth when its tip is placed at the apical foramen. The canals were instrumented with ProTaper Next rotary instruments (Dentsply Sirona) up to X3, with 2.5% sodium hypochlorite (NaOCl) irrigation (1 ml, after using each file) through a side-vented needle (239; Paikedun, Suzhou, China), followed by treatment in an ultrasonic bath (P5 Newtron; Satelec, Merignac, France) with 2.5% NaOCl for 20 seconds, three times. Subsequently, the canals were immersed in 17% ethylenediaminetetraacetic acid for 1 minute, followed by a final flush with 5 ml of 2.5% NaOCl, and then dried with absorbent paper points (Dayading, Beijing, China).

In Group 1, iRoot SP was inserted with the syringe provided by the manufacturer in the middle third of the canal, and then one 35#/0.04 gutta-percha point (Dayading) was covered with a thin layer of sealer and slowly inserted. The gutta-percha point was cut off at the root canal orifice with a heat carrier (B&L Biotech, Ansan, South Korea). In Group 2, AH Plus was prepared according to the manufacturer's recommendations and the warm vertical condensation technique was applied. Each root canal was obturated with one 35#/0.04 gutta-percha point covered with AH Plus and several 25#/0.02 gutta-percha points (Dayading) if necessary to ensure that the apical 4 mm was tightly filled. The gutta-percha points were removed 4 mm from the apical foramen with a heat carrier, and then the root canal was backfilled with warm gutta-percha.

Finally, for all specimens, Clearfil SE Bond (Kuraray Medical, Tokyo, Japan) and P60 resin (3M ESPE, St Paul, MN, USA) were used to seal the coronal access cavity (Fig 1e), and radiographs were taken from both the buccal-lingual and mesial-distal expansions to ensure there was no vacuole within the root filling materials (Fig 1f). The specimens were stored at 37°C under 100% humidity for 7 days.

Fibre post cementation

After storage, the P60 resin was removed with a diamond bur. The gutta-percha and its surrounding sealer material was removed with 1# to 3# Peeso reamers

(MANI), leaving 4 mm of filling material in the apical third. Post spaces were prepared to a depth of 9 mm with a blue size (diameter of 1.2 mm) post space preparation drill (RTD, St Egeven, France) (Fig 1g). The post spaces were then observed under an endodontic microscope, and if sealer remained in the canals, this was recorded. Before cementation, the post spaces were treated in an ultrasonic water bath for 1 minute and then dried with absorbent paper points. The CORECEM adhesive resin cement system (RTD) was injected into the post spaces with a syringe until the resin cement spilled out of the root canal orifice. Fibre posts (RTD) with a diameter of 1.2 mm were inserted to full depth with bulldog forceps and finger pressure. Excess cement was removed and the cement was cured for 40 seconds with an ultraviolet–light-emitting diode (UV-LED) curing light (Fig 1h). The roots were then incubated in 100% humidity at 37°C for 24 hours.

Push-out test

Each root specimen was sectioned transversally with a cutting machine (Isomet 1000 Precision Saw; South Bay Technology, San Clemente, CA, USA) under water irrigation, and slices with a thickness of 1 mm at the cervical, middle and apical thirds of the created post space were obtained at depths of 2.0, 4.5 and 7.0 mm from the root canal orifice, respectively (Fig 1i). Images of both sides of the slice were captured with a camera attached to an endodontic microscope. Slices with evident bubbles or voids were discarded from the study.

The push-out bond strength was measured using a universal testing machine (Instron 5969; Instron, Norwood, MA, USA). Loading was performed at a crosshead speed of 0.5 mm/min, with a metallic plunger with a diameter of 0.8 mm in the crown-to-apex direction until the post was completely dislodged from the slice, which is in line with a previous study[28]. The maximum force at failure (F) was recorded in Newtons (N), and the thickness of the slice (H) was measured in mm with digital calipers. The push-out bond strength (P) in megapascals (MPa) of each slice was calculated using the following equations: $P = F/S$; $S = C \times H$ and $C = \pi \times D$, where S is the area of the bonded interface (mm^2); C is the periphery of the post (mm) and D is the diameter of the post (mm).

Fracture mode analysis

All push-out specimens were assessed under 16× magnification with an endodontic microscope for the fracture analysis. The fracture modes were divided into four categories (Figs 2a to d): adhesive failure between resin cement and dentine (the post was enveloped by resin cement); adhesive failure between resin cement and the post (no cement visible around the post); cohesive failure in the post, i.e., the fracture occurred in the fibre post; and mixed failure i.e. post, cement and dentine visible on the debonded area.

Scanning electron microscopy (SEM) (S-4800; Hitachi, Tokyo, Japan) was used to observe the bonding interface and fracture surface. The specimens were immersed in 2.5% glutaraldehyde for 24 hours at 4°C and a gradient of dehydration in 30%, 50%, 70% and 90% ethanol for 10 minutes each and finally immersed in 100% ethanol for 30 minutes. Subsequently, the samples were dried naturally and fixed with electrically conductive silicone for examining.

Statistical analysis

Statistical analysis was conducted with an independent samples *t* test and one-way analysis of variance (ANOVA), using a factorial design with the type of sealer (AH Plus and iRoot SP) and the root canal region (cervical, middle and apical), followed by post-hoc Tukey multiple comparisons using SPSS statistics software (version 20.0; SPSS, Armonk, NY, USA) for Windows. The level of significance was set at $\alpha = 0.05$. The results were considered statistically significant when $P < 0.05$.

Results

The mean bond strength values (in MPa) and standard deviation values for fibre posts that were cemented in the root canals previously obturated with different sealers at the cervical, middle and apical regions were determined (Table 1). Within the same root canal region, there was no statistically significant difference in bond strength between the two sealers, whether in the cervical, middle or apical portion ($P = 0.67$, $P = 0.14$ and $P = 0.78$ for the cervical, middle and apical portions, respectively). When using the same sealer, the highest bond strength was obtained in the cervical portion for both AH Plus and iRoot SP (22.78 ± 3.80 MPa and 23.88 ± 4.15 MPa, respectively), which was significantly higher than for the middle portion (19.74 ± 3.49 MPa and 20.90 ± 4.03 MPa, respectively) ($P = 0.030$ and $P = 0.048$ for AH Plus and iRoot SP, respectively) and the apical portion (18.96 ± 4.86 MPa and 18.82 ± 4.55 MPa, respectively) ($P = 0.07$ and $P < 0.001$ for AH Plus and iRoot SP, respectively).

In the AH Plus group, there were two major fracture modes: adhesive failure between the resin cement and

Table 1 Mean bond strengths (MPa) and standard deviations for the sealers in different canal regions.

Group	Root canal region			Overall
	Cervical	Middle	Apical	
AH Plus	22.78 ± 3.80^{aA}	19.74 ± 3.49^{aB}	18.96 ± 4.86^{aB}	20.49 ± 4.34^{a}
iRoot SP	23.88 ± 4.15^{aA}	20.90 ± 4.03^{aB}	18.82 ± 4.55^{aB}	21.24 ± 4.64^{a}

Uppercase letters correspond to the comparison in the rows (among root canal regions) and lowercase letters correspond to the comparison in the columns (among sealers). The same letters indicate no significant differences between the compared groups ($P > 0.05$) and vice versa.

Table 2 Mode failure percentages for each group (n = 18).

Sealer	Region	Failure mode			
		Adhesive cement–dentine	Adhesive cement–post	Cohesive in post	Mixed
AH Plus	Cervical	0.0%	33.3%	27.8%	38.9%
	Middle	5.6%	44.4%	16.7%	33.3%
	Apical	16.7%	38.9%	5.6%	38.9%
iRoot SP	Cervical	0.0%	50.0%	33.3%	16.7%
	Middle	0.0%	50.0%	27.8%	22.2%
	Apical	5.6%	50.0%	5.6%	38.9%

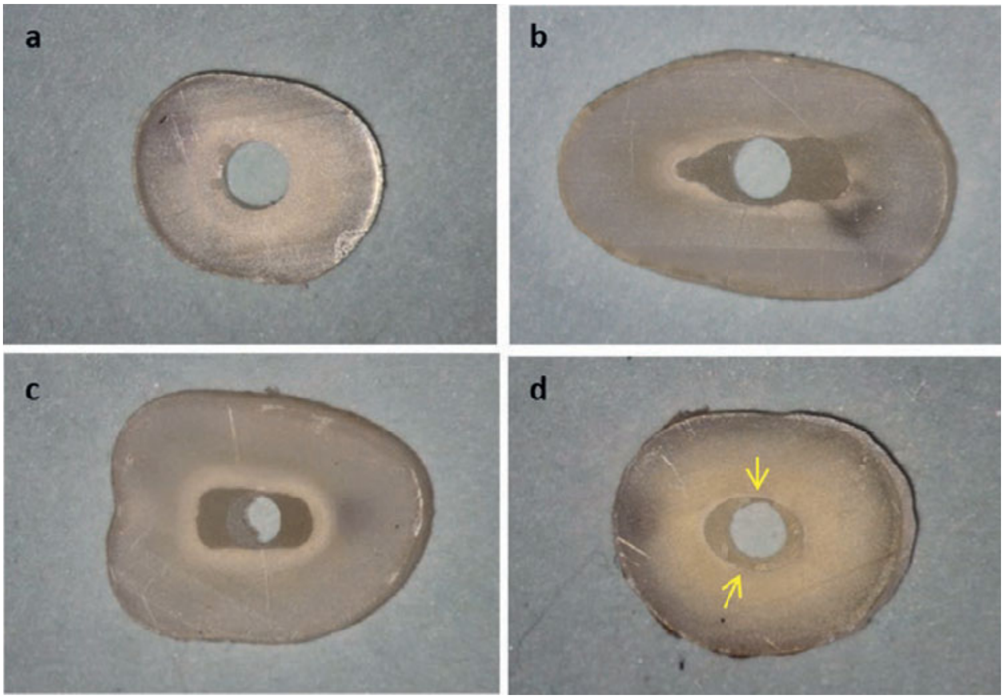

Fig 2 Fracture mode categories. **(a)** Adhesive failure between resin cement and dentine. **(b)** Adhesive failure between resin cement and the post. **(c)** Cohesive failure in the post. **(d)** Mixed failure (arrows indicate the location for debonding between the adhesive resin and dentine).

the post, and mixed failure. In the iRoot SP group, the major fracture mode was adhesive failure between the resin cement and the post (Table 2). In both the AH Plus and iRoot SP groups, compared with the cervical and middle specimens, there were more adhesive failures between the cement and dentine and fewer cohesive failures in the post in the apical specimens. In the specimens that showed mixed failure, the most common location for debonding between the adhesive resin and the dentine was where the adhesive resin was thin and weak, as shown by the arrows in Fig 2d.

After post space preparation, sealer could still be seen on the root canal walls in 38.9% (7/18) of root canals with iRoot SP obturation (Fig 3a), whereas no sealer was seen in the AH Plus group. All seven of the root canals containing sealer remnants were oval, with the location of the remnants being near the vertex of the long axis of the ellipse. Of these seven root canals, six had sealer remnants in the cervical portion, seven in the middle portion, and three in the apical portion. The adhesive resin was also found to combine tightly with the sealer (Fig 3b), and there was no debonding

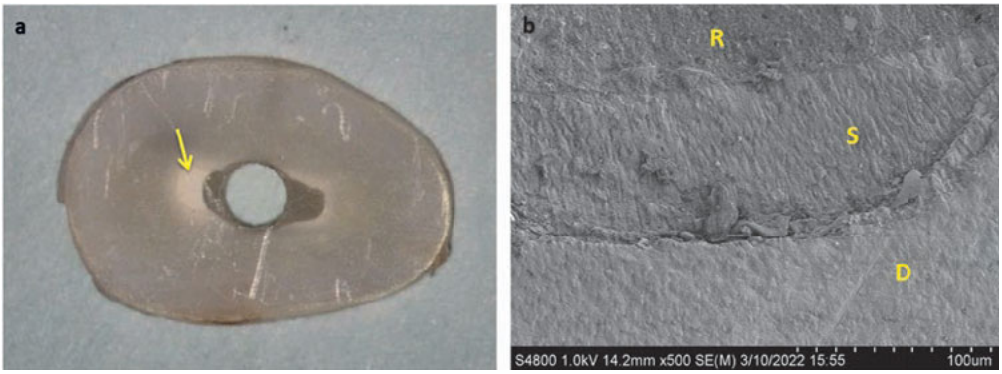

Fig 3 Sealer remnants on root canal walls after post space preparation in the iRoot SP group.
(a) Sealer remnants were located near the vertex of the long axis of the ellipse (arrow point).
(b) The adhesive resin (R), the sealer (S) and the dentine (D) were bound tightly together after push-out testing.

of adhesive resin on the root canal wall where sealer remnants were present.

Discussion

To evaluate the bond strength of materials, several techniques can be used, including conventional tensile strength testing, as well as pull-out and push-out tests. The push-out test is widely used when evaluating the bond strength of fibre posts and was employed in the present study, since it can be used in confined areas such as root canal walls and give a precise indication of the location(s) in which failures occurred[8, 29].

The present study has shown that it is difficult to remove excess iRoot SP completely from the root canal system after post space preparation, whereas all excess AH Plus can be removed. iRoot SP was seen in each third of the root canal, mainly in the cervical and middle regions. From the cervical to the apical region, the shape of the cross-section of the root canal changes gradually from oval to circular, the diameter of the root canal becomes progressively smaller and the penetration depth of the sealer in the dentine tubule gradually becomes less[30], making it easier to remove the sealer in the apical region.

Most previous studies have demonstrated that iRoot SP has a negative influence on the bond strength of fibre posts[9,10,31,32], and that any sealer remnants could prevent contact between the resin cement and the collagen matrix in dentine and change the wettability, permeability, pH and reactivity of the dentine, affecting the resin–dentine bond strength[9]; however, this was not shown to be the case in the present study, and the authors did not observe any difference in the bond strength to root dentine whether iRoot SP or AH Plus sealer was used, which is in line with the findings reported by He et al[26] and Özcan et al[27]. One possible explanation for this could be the use of different study designs and methodologies in the present study and others, for example in relation to the obturation technique, post space irrigation methods, and brand of cement and fibre post used[33]. The retention of fibre posts depends mainly on adhesion, including the adhesion between cement and dentine and between cement and the post. In the present study, most dislocations occurred between cement and the post, which means that as long as the bond strength between the cement and dentine was greater than that between the cement and the post, there would be no difference in bond strength of fibre posts between the iRoot SP and AH Plus groups, even if there was some difference in bond strength between cement and dentine between the two groups. Although sealer remnants were found along the root canal walls, it may not be enough to have an impact on the bond strength of fibre posts cemented with the CORECEM adhesive resin system.

This study showed that the bond strength of the fibre posts in the cervical portion was higher than that in the middle and apical portions when both AH Plus and iRoot SP were used, which is consistent with previous studies[8,10,34,35]. One possible explanation for this is the morphological differences within the dentine along the canal, such as the reduction in the number, density and diameter of dentine tubules from the cervical to the apical portion[36,37]. Since adhesion relies mainly upon micromechanical retention, higher bond strength is expected to be achieved in the cervical portion[10,27]. Another factor that may explain the lower bond strength in the deepest portion of the root canal is the difficulty accessing this area. For example, insertion of the acid etching solution and adhesive system was impaired[38] and there was likely poor UV curing of the resin composite in regions distant from the light source, with incomplete curing likely contributing to the diminished bond strength of fibre posts[5,26,38].

Finally, the major fracture modes with AH Plus were found to be adhesive failure between the resin cement and the post and mixed failure, whereas with iRoot SP, half of the specimens experienced adhesive fracture between the resin cement and the post, implying that this group had better adhesion with the dentine walls, which is in accordance with Özcan et al[27] but in contrast with the majority of previous studies, where most failures were either of the adhesive at the cement–dentine interface[8,9,26] or mixed failure[30]. The possible explanations for the different distribution of fracture mode include the type of luting cements used, the approach taken to treatment of the post space, and difference in the pre-treatment of the fibre post. Fibre posts that have been airborne-particle abraded and pre-treated with silane might experience fewer adhesive failures between the cement and posts, although this warrants further investigation[39-41].

The main limitation of the present study was the fact that different obturation techniques were used for different sealers; however, the study design is based on clinical application. AH Plus with the warm vertical condensation technique is considered the gold standard[13,14], whereas warm vertical condensation was not advised in conjunction with iRoot SP by some researchers[42]. The single-cone technique is employed by most dental practitioners when iRoot SP is used[17], as the technique is easy and fast and the most important is the clinical success rate. The success rate of root canal therapy using a single-cone technique with iRoot SP was 90.9%, which fell within the range found in previous studies[43]. To eliminate the influence of different obturation techniques, another group with the use of iRoot SP in conjunction with the warm vertical condensation technique could be added; however, this approach is rarely used in clinical settings and might have limited clinical significance.

Conclusion

Within the limitations of the present in vitro study, it can be concluded that the presence of remnants of iRoot SP sealer after post space preparation has a limited effect on the bond strength of fibre posts to dentine. iRoot SP used with the single-cone technique is a viable option for root canal obturation before fibre post cementation.

Conflicts of interest

The authors declare no conflicts of interest related to this study.

Author contribution

Drs Di QIAO and Meng Meng ZHU contributed to the experiments, data collection and analysis. Dr Di QIAO drafted the manuscript; Drs Jie PAN and Di QIAO contributed to the study design. All the authors approved the manuscript.

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