Original Article

Push Out Bond Strength of Self-etch Resin Cement in Canal Obturated with Gutta Percha/bioceramic Sealer

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Abstract

The purpose of this *in vitro* study was to compare the push out bond strength of self-etch resin cement bonded fiber posts after use of two bioceramic sealers in obturation. Forty maxillary incisors were decoronated to the root length of 13 mm and prepared with K3 rotary files up to size 40/.06. All of them were filled with the assigned sealers: control (no sealer, AH Plus™, iRoot SP®, and Nishika Canal Sealer BG®). After seven days, post spaces were prepared and then applied the mixture of primer A and B. The prefabricated fiber posts (FibreKleer® no.2) were cemented with Multilink® N in the prepared root canal. Two cross sections of specimens were performed in 2 mm thick slices, representing two different regions, coronal and middle thirds. Universal Testing Machine was loaded with plunger vertically until specimen failure. Maximum force was recorded as push out bond strength. Failure patterns were observed under a stereomicroscope at 50x magnification. The results showed that, in both root regions, the highest bond strength was found in the control group. Roots filled with AH Plus TM exhibited statistically more bond strength than roots filled with iRoot SP® and Nishika Canal Sealer BG® ($p<0.05$). Failure at the resin cement-dentin interface was prominent in all sealer groups. In conclusion, iRoot SP® and Nishika canal sealer BG® negatively impacted the push out bond strength compared to AH Plus™

Keywords: Bioceramic sealer, Resin cement, Fiber post, Push out bond strength

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Introduction

In teeth with extensive loss of crown structures, intracanal posts were designed to use for retaining coronal restoration. Their retention can be obtained from good adaptation of post to canal wall as well as use of resin cement.[1] In the total etch technique, dentin pretreatment before bonding can be done by applying acid, conditioning the dentin with a primer, and then applying resin. Though this circumstance has been proven to achieve high bond strength, it is difficult to obtain a good outcome due to the challenges of moisture control from a clinical viewpoint.[2] In the search for solving this problem, self-etch resin cement, which combines etching in the form of an acidic monomer with primer, has been recently introduced.[3]

The adhesive mechanism of self-etch resin cement is attributed to the demineralization caused by the acidic monomer after primer application, which modifies the layer over the dentin surface, so called smear layer and allows the infiltration of luting cement into the underlying dentin without water rinsing.[4] This situation is influenced by the variation of the dentin substrate: normal dentin and contaminated dentin, especially root dentin contaminated with root canal sealer after endodontic treatment. Altman and his colleagues[5] demonstrated that when the root surface is contaminated with zinc oxide-eugenol sealer, it deteriorates the retention of fiber posts bonded with resin cement. Moreover, some sealers, such as bioceramic sealers, not only can contaminate the root canal wall but also can modify the dentin surface through hydroxyapatite precipitation.[6]

Nowadays bioceramic root canal sealers have been utilized as revolutionary materials in endodontic treatment. Several forms of this material were introduced to the dental market which vary to the composition inside the products. Some are presented in premixed form, such as iRoot SP® (Innovative BioCeramix, Vancouver, Canada), while others are in two-pasted forms, such as Nishika Canal Sealer BG® (Nippon Shika Yakuhin, Yamaguchi, Japan).[7] The main reaction of this material to dentin is claimed to be its ability to form hydroxyapatite on the root surface, which can refill the gap of root filling as well as penetrate into dentinal tubule.[8] In this circumstance, both the root dentin surface and the chemical composition of dentin can be changed, resulting in changes in reaction with acidic monomers and possibly affecting the intimacy of the post and dentin wall.[6] The question arises whether bond strength of resin cement should be changed after the use of a different form of bioceramic sealer or not.

In the clinical point of view, post bond strength simulation should be created parallel to the bonding interface of post and cement, which present in push out bond strength test.[9] The aim of this study is to compare the push out bond strength of self-etch resin cement bonded fiber posts after use of two bioceramic sealers in obturation.

Materials and methods

Approval of this study was achieved by the ethics committee of Srinakharinwirot University (No. SWUEC-661002).

Sample selection

Forty single-rooted extracted human maxillary incisors with a tooth length of at least 21 mm were collected for the present study. Radiographs were taken in two directions: buccopalatal and mesiodistal views. The teeth with single canal, noncalcified canal, completely formed roots were selected into this study, while the other with root canal treatments, caries, or cracks were excluded.

Specimen preparations

Tooth Decoronation perpendicularly to the long axis was performed by carborundum disc to obtain a 13 mm root length. A size 15 K-file (Dentsply Maillefer, Ballaiques, Switzerland) was inserted into the root canal until visualizing the tip of the instrument at the root apex. That measurement was subtracted 1 mm to obtain the working length. Instrumentation was completed using K3 rotary files (SybronEndo, Orange, CA, USA) up to size 40/.06. 2 mL of 2.5% NaOCl irrigation was performed during instrumentation. After preparation, 10 mL of 17% Ethylenediaminetetraacetic acid (EDTA, SmearClear, Sybron endo, CA, USA) followed by 10 mL of 2.5% NaOCl was used. The final irrigation with 10 mL distilled water was performed to clean the residual of other irrigation. Dried canals were then achieved by paper points.

The specimens were randomly allocated into four groups of ten each according to type of sealers as follows: 1) Control group: no sealer used; 2) AH plus group: AH PlusTM used as sealer; 3) iRoot SP group: iRoot SP® used as sealer; 4) Nishika BG group: Nishika canal sealer BG® used as sealer. The sealers were prepared according to the manufacturer's recommendation as presented in Table 1 and used for single-cone technique obturation using K3 gutta-percha master cone size 40/.06 (SybronEndo, Orange, CA, USA). The specimens were then stored in a closed container with 100% humidity at 37 °C for one week to allow complete sealer setting after sealing Cavit™ (3M ESPE, Seefeld, Germany).

Table 1 *Type and composition of the materials used*

Materials	Type	Composition	
Multilink® N (Ivoclar Vivadent, Liechtenstein) Lot. Z0347C	Self-etch primer	Primer A: initiators	Primer B: -Resin component: HEMA, and methacrylate monomers -Acidic component: phosphonic acid
	Self-etch adhesive	Base and Catalyst: HEMA and Dimethacrylate Filler: barium glass filler, ytterbium trifluoride and spheroid mixed oxide	
	Monobond N	Ethanol, water, 3-MPS, 10-MDP and 10-MDDT	
AH Plus™ (Dentsply DeTrey, Germany Lot. 2202000877	Epoxy resin-based sealer	Paste A: Bisphenol A and F epoxy resin, Calcium tungstate and Zirconium oxide as filler	Paste B: Benzyldiamine, Adamantane amine, Tricyclodecane-Diamine
iRoot SP® (Innovative BioCeramix, Vancouver, Canada Lot. 21002SP	Pure bioceramic sealer	Tricalcium silicate, Dicalcium silicate, calcium phosphate with zirconium oxide as radiopaque and filler	
Nishika canal sealer BG® (Nippon Shika Yakuhin, Yamaguchi, Japan) Lot. M5K	Bioactive glass-based sealer	Paste A: fatty acids, bismuth subcarbonate, and silica dioxide	Paste B: calcium silicate glass (a type of BG) with magnesium oxide silica dioxide base

Fiber post cementation

The 8 mm depth of post space preparation was initially performed using a size #1 Peeso reamer (Mani Inc., Tochigi, Japan) in each canal with a diameter of 0.70 mm, followed by a size #2 FibreKleer™ drill (FibreKleer® 4X, Pentron, Wallingford, USA). Examination of prepared post space was revealed under ×7.5 magnification loupe (Admetec, Deva Medical Supply, Bangkok, Thailand) and with radiograph to prove that there was no gross remnant of material on the wall. A FibreKleer™ 4X glass fiber post, size #2 was selected and tried to fit within the canal.

Before fiber post fixation, rinsing 10 mL of distilled water was done for 60 seconds and post spaces were then dried with paper points. For dentin surface treatment, a mixture of self-etch of multilink (primer A and B) was applied to the root canal walls for 15 seconds. For cementation, silanized fiber posts with Monobond N followed by a mixture of self-etch adhesive (Multilink N, Ivoclar Vivadent AG, Schaan, Liechtenstein) and finally insertion of those posts into the prepared post spaces with light finger pressure. All material compositions were presented in Table 1. Excess cement that extruded was removed and polymerized using a visible light with an intensity of 1,000 mW/cm² curing unit (EliparTM; 3M ESPE, St. Paul, MN, USA) for 20 seconds. Subsequently, all the specimens were kept in the same container.

Bond strength specimen preparation

Each tooth was individually embedded in Polyvinyl Chloride (PVC) pipes in self-cured acrylic resin (Elite SC Tray; Zhermack, Rovigo, Italy), with their long axes perpendicular to the horizontal plane. Each post-cemented root was cut in cross section using the Isomet 100 Precision Saw (Buehler Ltd., Lake Bluff, IL, USA) for two 2 mm thick, representing the coronal and middle thirds of root regions respectively. To examine the completeness of the bonded surface and standardize the diameters of the specimens, examinations and measurements were conducted on both sides of the slices using the EPview™ program (Olympus Imaging Software, Olympus Optical Co., Tokyo, Japan). Specimens showing the presence of bubbles or voids were excluded from the study to standardize the specimens. (Fig. 1).

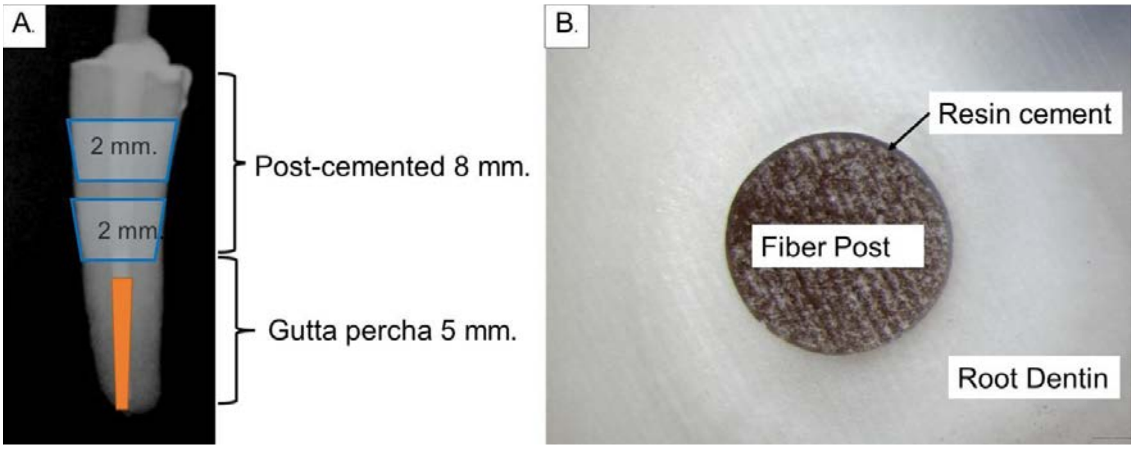

Figure 1 *(A) level of slice of root specimen used in the experiment; (B) at 50X magnification of the post-cemented slice, the surrounding resin cement was coated consistently with no bubbles.*

Push out test

Each slice was loaded vertically with two consecutive plungers, sized 1 mm and 0.8 mm diameter, for coronal and middle thirds, in a Universal Testing Machine (EZ Test Series, Shimadzu, Kyoto, Japan) with a crosshead speed of 0.5 mm/min. The force was applied for the push out test. The maximum force during the specimen failure was recorded and calculated into MPa using the following formula:

$$P = \frac{F}{\pi h(r1 + r2)}$$

where "F" is maximum force (N); "π" is the constant 3.14; "r1" is apical radius; "r2" is coronal radius; and "h" is the thickness of the slice in mm, standardized at 2 mm

Failure mode analysis

Examination of dislodged specimens were done under a stereomicroscope at 50X magnification to clarify into the three failure patterns: 1) failure at dentin: all of material inside the canal moved away from dentin; 2) failure at post: the fiber post moved from the specimen, while the resin cement remained on the dentin wall; 3) failure at dentin and post: dentin is visible in some areas and resin cement present in the others.

Statistical analysis

Statistical analysis was performed using SPSS software (SPSS 20.0; Chicago, IL, USA). One-way ANOVA was used for comparison of the bond strength and the canal diameter of each sealer. In cases of significant differences, Tukey tests were performed for multiple comparisons.

Results

To control the size of each slice, the canal diameters of the coronal and apical sides were measured and presented in Table 2. No statistically significant differences were found on either side of each slice. (P>0.05)

Table 2 *The minimum, maximum, and mean diameters of the coronal and apical sides of the slices*

Regions	Coronal side (mm)			Apical side (mm)		
	Min.	Max.	Mean	Min.	Max.	Mean
Coronal	1.35	1.42	1.38	1.15	1.25	1.20
Middle	1.10	1.16	1.13	0.89	0.96	0.93

The mean and standard deviation of the push out bond strength of each group are presented in Table 3. In both root regions, the control group demonstrated the highest mean push out bond strength. AH Plus group showed significantly higher push out bond strength than those of iRoot SP group and Nishika BG group (P<0.05). No statistically significant difference was found between coronal and middle slices in all groups.

Table 3 *Mean (standard deviation) of push out bond strength, expressed in MPa in the different root*

Group / Regions	Control	AH plus	iRoot SP	Nishika BG
Coronal	11.43 (1.43) [A]	9.59 (0.87) [B]	6.46 (1.36) [C]	6.57 (1.38) [C]
Middle	9.74 (2.08) [a]	7.38 (1.27) [b]	4.73 (1.58) [c]	4.92 (1.36) [c]

**Different superscript letters in the same line indicate statistical differences (P < .05)*

Failure pattern of all slices is shown in Figure 2. The prominent failure pattern was failure at dentin, occurring in 50-60% of the specimens in all sealers. This was followed by failure at dentin and post accounted for 20-40%. Failure at post was observed only not more than 20%. (Fig. 3)

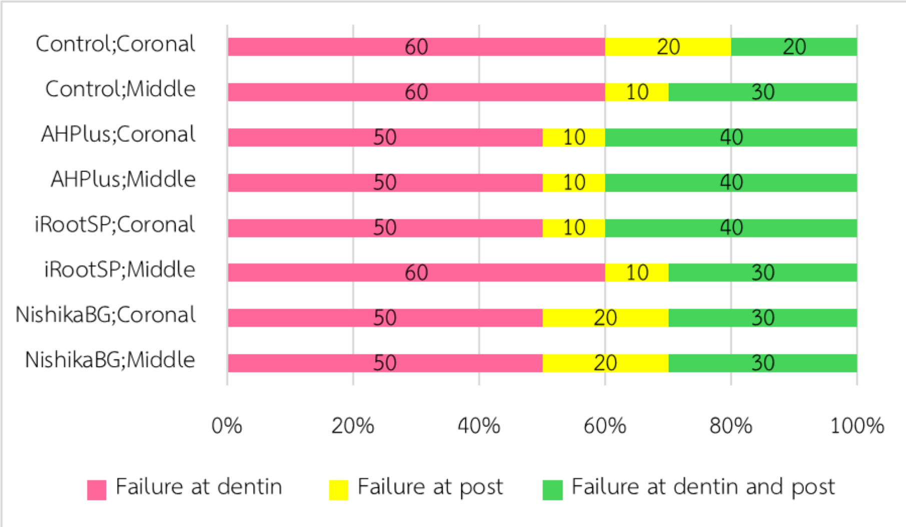

Figure 2 Mode of failure for each group

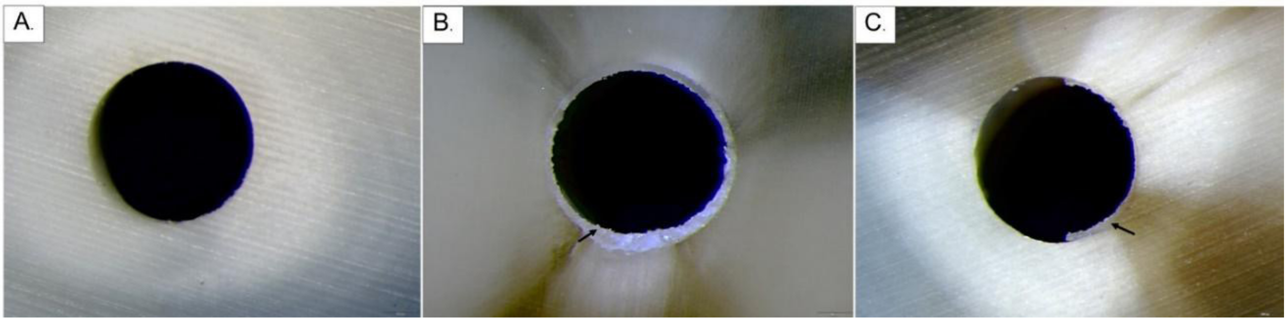

Figure 3 *Stereomicroscopic image of failure modes (A) failure at dentin (B) failure at post (C) failure at dentin and post; arrow indicates resin cement*

Discussion

The control group, which did not use any sealer, demonstrated the highest bond strength, aligning with the results reported by Chadgal *et al.*[10] The explanation for this situation lies that the composition of the smear layer in this group is likewise the smear layer in normal dentin cutting. After the application of primer, the acidic monomer, phosphonic acid, reacts with the superficial part of the smear layer, simultaneously flows into the underlying smear layer, and forms a hybridized smear layer.[11] The bond strength in this group, which was created in unchanged dentin was much higher than the other group.

From the results of this study, AH Plus™ seemed to have some negative effects on the push out bond strength of self-etch resin cement. When the post space was created, the smear layer in this groups was blended with the removed sealer, while a small amount of material was retained on the dentin surface.[12] These circumstances made the acidic monomer in Multilink® N unable to alter the smear layer in the same pattern as in normal dentin, resulting in reduced push out bond strength.

In this experiment, the most negative effect on self-etch resin cement was found in the group of iRoot SP® and Nishika Canal Sealer BG®. This situation may be attributed to residual sealer and hydroxyapatite precipitation on the dentin wall. From the study of Karobari *et al.*[13], more dentine penetration and push out bond strength was found in the bioceramic sealer, which affected the retrievability of this sealer. Chen *et al.*[14] confirmed that iRoot SP® is more difficult to remove in post space preparation than resin-based sealer. The effect of phosphonic acid, an acidic monomer in Multilink N, on the root canal wall should be reduced by the smear layer formed by either iRoot SP® or Nishika Canal Sealer BG®. Yoshii *et al.*[15] reported that Nishika Canal Sealer BG® has similar properties and dentin reaction as other bioceramic sealers. Nishika Canal Sealer BG® have high adaptation, which enhanced the infiltration of the sealer into dentinal tubules.[16] Akcay *et al.*[17] and Chew *et al.*[8] found that iRoot SP® and Nishika Canal Sealer BG® penetrate dentinal tubules more effectively than AH Plus™ , which may cause them to remain more in the root canal post-preparation, resulting in lower push out bond strength of the resin cement.

One of the key factors is the hydroxyapatite precipitation of the bioceramic sealer. Belal *et al.*[18] demonstrated that Ca^{2+} and OH^- released from bioceramic sealer can be detected during and after the setting process, which may be the origin of alkalinity of material and hydroxyapatite formation. The alkaline pH of a bioceramic sealer might buffer the acidity of self-etch primer, reducing its demineralization capability and consequently weakening the bond between the fiber post and root dentin.[19] Motoyama *et al.*[20] revealed that the presence of hydroxyapatite considerably affected the modification effect of acidic monomer.

These results were contradictory to Yuanli *et al.*[21], who reported no difference between iRoot SP® and AH Plus™. One potential explanation could lie in the disparate post-space irrigation methods employed. In this study, the post space underwent irrigation solely with distilled water, whereas Yuanli *et al.*[21] utilized ultrasonic irrigation with Chlorhexidine (CHX) and EDTA. Chen *et al.*[14] demonstrated that ultrasonic irrigation enhances smear layer removal. CHX inhibits matrix metalloproteinases (MMP), thereby not adversely affecting the bond strength of resin cement.[22] Additionally, EDTA can selectively chelate calcium ions, removing hydroxyapatite and facilitating the penetration of the adhesive system's functional monomer through dentinal tubules.[23] Consequently, the amount of sealer remaining on the dentinal wall may be insufficient to significantly impact the bond strength of resin cement.

Considering the failure mode analysis, it could be emphasized that predominant pattern of failure occurred between the resin cement and root canal which indicated the weak bond occurred between the resin cement and the root dentine.[24] The probable reason for this failures may be high C-factor of the resin cement, which presented with more bonded surface than free surface of resin cement. In this situation, it leads to contraction in some parts of the resin cement, inducing and finally dislodging of post with resin cement from the root canal walls.[25]

According to the study by Marcos *et al.*[26], the thickness of the resin cement negatively affects the push out bond strength of the resin cement. The study explained that a thicker resin layer may entrap air voids, increasing the risk of failure and dislodgment. To address this, FibreKleer™ 4X glass fiber post, size #2 is selected to ensure a precise fit within the root canal and to minimize excess resin cement use. A size #1 post would result in a loose fit, necessitating a thicker layer, while a size #3 post would require excessive canal enlargement, risking unnecessary dentin removal. Additionally, the passive placement of posts without mechanical preparation preserves the integrity of the root canal, thereby enhancing clinical outcomes.[27]

Conclusion

Within the limitations of this in vitro study, it can be concluded that all sealer groups reduced the push out bond strength of resin cement to the fiber post and root canal wall. A greater reduction was especially observed when iRoot SP® and Nishika Canal Sealer BG® were used.

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