

Effects of Epoxy Resin and Calcium Silicate-Based Root Canal Sealer on Fiber Post Adhesion

Swesty Mahardhini¹, Ratna Meidyawati², Dewa Ayu Nyoman Putri Artiningsih², Milati Amalia¹

1. Conservative Dentistry Residency Program, Faculty of Dentistry, Universitas Indonesia.

2. Department of Conservative Dentistry, Faculty of Dentistry, Universitas Indonesia.

Abstract

This study aimed to evaluate the effects of epoxy resin and calcium silicate-based root canal sealer on fiber post adhesion strength.

Thirty single-rooted lower premolars were divided into three groups (n = 10 for each group): Group 1, no root canal filling; Group 2, epoxy resin root canal sealer; and Group 3, calcium silicate root canal sealer. After all teeth were prepared with rotary instruments, Groups 2 and 3 were filled with gutta-percha points and root canal sealer. A fiber post was cemented in all tooth, then 2-mm disc samples were taken from the middle root section and subjected to a push-out bond strength test. Data were analyzed using one-way ANOVA and Bonferroni test.

The highest average adhesion strength was exhibited by the control group (57.142 ± 12.205 MPa), followed by the epoxy resin group (44.455 ± 10.347 MPa) and then the calcium silicate group (28.009 ± 9.562 MPa). There was a significant difference in push-out bond strength between the groups ($p < 0.006$).

The teeth that were endodontically treated with epoxy resin root canal sealer exhibited better fiber post adhesion strength compared to the teeth that were treated with calcium silicate root canal sealer.

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Introduction

Endodontically treated teeth may experience large structural loss of coronal hard tissue. This can be due to an increase in stress load on the cervical area, which can cause crown or root fracture. Therefore, intra-radicular posts are often needed to increase the retention of tooth restorations after endodontic treatment.^{1,2} Restoration with post and core material reinforcement can reduce the likelihood of cervical crack development when axial force is applied. This treatment increases hoop tensile stress resistance at the thin margin, thus reducing the formation of fractures originating from this area.³

One type of intra-radicular post used by many dentists today is the fiber-reinforced post (FRP). This type of post is made from microfibers

with high flexural strength and an elastic modulus approximating that of dentine, which allows the tooth load to pass evenly to the entire part of the root, resulting in a lower risk of root fracture.^{4,5} Fiber posts can adhere securely to the dentine wall of the root canal and form a homogeneous unit known as a monoblock system.⁶ The bonding mechanism is obtained from micromechanical bonds through the formation of a hybrid layer from a demineralized dentine surface and the formation of a resin tag in the open dentinal tubules produced by a resin bonding system.² Meanwhile, adhesive bonding can occur through polymerization between fiber posts and resin cement because both have similar matrix resin contents.

Although their success rate is relatively high, the use of fiber posts for restoration can also result in various clinical failures, the most common of which is the detachment/ debonding of the post due to adhesion failure.^{2,7} Various factors can affect adhesion, including the preparation and cleaning of the post chamber. For example, a new smear layer is produced by the preparation and cleaning of gutta-percha and resin cement residues or root canal cement

*Corresponding author:

Prof. Ratna Meidyawati, Dr., drg., Sp.KG(K)
Department of Conservative Dentistry, Faculty of Dentistry,
Universitas Indonesia.
Jl. Salemba Raya no.4 Jakarta 10430, Indonesia
E-mail: meidyawati58@gmail.com; drg.swesty@gmail.com

inside the root canal chamber with a rotary instrument. This secondary smear layer is composed of dense, thick debris originating from residual root canal cement or gutta-percha that was solidified/plasticized by the friction created by the heat of the bur. More specifically, the secondary smear layer contains dentine powder, root canal sealer, and residual gutta percha within the root canal chamber.⁸

Epoxy resin and calcium silicate-based root canal cements are two of the most widely used nowadays for root canal obturation on endodontic treatments. Epoxy resin-based cement biocompatible and antimicrobial, with low solubility, good sealing ability, and many other good properties.⁹ Calcium silicate-based root canal sealer contains sources of calcium, phosphate, and hydroxyl ion-releasing material; these components form bioactive calcium hydroxide, which can form a crystalline hydroxyapatite mineral structure so that it forms a better bond with dentine.¹⁰ However, one of the disadvantages of calcium silicate-based root canal sealer is that it is quite difficult to clean from the root canal once it has hardened.¹¹

An experimental study by Serafino (2004) showed that gutta-percha and root canal sealer cement cannot be completely removed from the wall of the root canal, especially in anatomically irregular areas.¹² A study by Chen (2018) showed that there are differences in the cleanliness of the smear layer on the root canal dentin surface after gutta-percha removal and post space preparation in teeth that were previously filled with several different types of root canal cement.² Based on these two studies, it can be concluded that the use of different types of root canal cement appears to affect adhesion between root canal dentine, resin cement, and fiber posts.

The present study aimed to evaluate the effects of endodontic treatment with epoxy resin and calcium silicate root canal sealers on fiber post adhesion strength.

Materials and methods

A total of 30 post-extracted mandibular single-rooted premolars indicated for orthodontic treatment were used in this study (ethical clearance: 138/ethical approval/FGUI/XII/2019, protocol number: 051471219). The selection of these samples was based on the following

inclusion criteria: single-rooted mandibular premolar tooth, fully formed, straight, root canal length of 20 ± 2 mm, and macroscopically intact.

After the working length of each tooth was determined, the crown of each tooth was cut using a diamond disc to obtain a similar root length of 15 mm for all tooth samples. Only teeth with the same root diameter as the stainless steel K-File No. 15 (Dentsply Maillefer, Switzerland) were used. The samples were prepared with ProTaper Next (Dentsply Maillefer, Switzerland) rotary instruments up to X4 along the specified work length (Figure 1A–B).

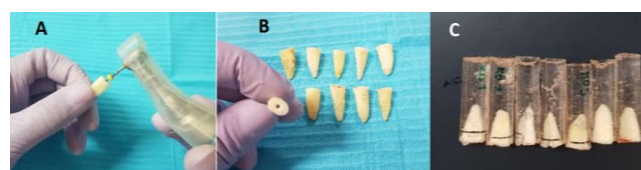


Figure 1. Tooth sample preparations. **A.** The crown of each tooth was cut with a diamond disc, then the root canal was prepared using a ProTaper Next rotary file. **B.** Tooth samples after root canal preparation. **C.** Tooth samples after the root canals had been filled, incubated, then planted inside a mixture of acrylic solution poured into plastic tubing, and then left for 1x24 hours until set. Then, the sample tooth within each acrylic tube was cut in the middle part of the root with a cutting machine, producing a 2-mm-thick disc sample.

Each change in instruments was followed by irrigation with a 2.5% NaOCl solution. After preparation, the root canals were irrigated with 5 ml of 17% EDTA (MD Cleanser, Meta Biomed, South Korea) and then dried with paper points. The teeth were randomly sorted into three experimental groups ($n = 10$ for each group): Group 1 (control group), tooth samples without root canal fillings/obturation; Group 2: tooth samples that had root canal fillings with gutta-percha and epoxy resin-based root canal cement (AH-Plus, Dentsply Maillefer, Switzerland); and Group 3: tooth samples that had root canal fillings with gutta-percha and calcium silicate-based root canal cement (Ceraseal, Meta Biomed, South Korea).

All root canal cements were manipulated according to the manufacturer's recommendations and then inserted into the root canal. A master cone gutta-percha with a 0.4-mm-diameter tip and a 6% taper (X4 ProTaper

Next, Dentsply Maillefer, Switzerland) was inserted into the root canal according to the working length and then compressed with a heated plugger. The cavity was closed with a temporary restoration Cavit G (3M ESPE, German). Then, the sample was incubated at 37°C for seven days.

After the incubation process, the gutta-percha was partially removed with Gates-Glidden Drill #3 (MANI, Japan), 10 mm from the coronal part of the tooth. Then, the post preparation process was carried out with Post Drill #1 (Postec Plus, Ivoclar Vivadent, Switzerland). Next, the root canals were irrigated with 17% EDTA and 2% chlorhexidine for one minute each and then dried with paper points. Prefabricated Fiber Post #1 (Postec Plus, Ivoclar Vivadent, Switzerland) was inserted into the prepared root canals to evaluate its fitness at the root end area in accordance with the post-preparation length. Next, the post was cemented with self-etch resin cement (Multilik-N, Ivoclar Vivadent, Switzerland). The fiber post was then inserted into the root canal and polymerized with curing light for ten seconds until it hardened. The excess fiber post was cut parallel to the cervical surface of the tooth with a highspeed fissure bur.

All teeth were embedded in a custom-made resin mold in a vertical position (Figure 1C). After the resin was completely set, a 2-mm-thick disc sample representing the middle third of the root canal was cut in a horizontal plane perpendicular to the root axis with an automatic cutting machine under constant water spray. The acrylic tube was cut between 9 mm and 11 mm from the apical root, resulting in ten specimens per group.

The dislodgement resistance and mode of failure of each specimen was evaluated, and the adhesion strength was analyzed using the push-out bond strength technique with a universal testing machine (Shimidzu AG-5000E, Kyoto, Japan; Figure 2A). The disc samples were placed in metallic jigs with gaps so that the fiber posts would fall when vertical force was applied. A plunger tip with a 0.8-mm diameter was used to apply vertical force to the fiber post. The diameter of the plunger tip was adjusted to the diameter of the fiber post on the sample to achieve an even distribution load in 60–85% of the fiber post area without touching the area of resin and root canal cement (Figure 2B). Vertical force was applied to the apical direction at a speed of 0.5 mm/min continuously until it reached the maximum load, which was sufficient to push the fiber post and disrupt the fiber post-dentin bond. The push-out bond strength values are presented in N/mm² (units equivalent to MPa).

The mode of failure between the fiber post, resin cement, and dentine was examined under a stereomicroscope camera (Nikon SMZ800, Japan) at ×30 magnification. Each specimen was evaluated and classified into one of three failure modes described by Skidmore et al.: adhesive failure (no material left on the root canal wall), cohesive failure (material present on the entire root canal wall), or mixed failure (material present in patches on the root canal wall).¹³

The quantitative data were statistically analyzed with a one-way ANOVA and a Bonferroni post hoc test with a significance level of $p < 0.05$. All statistical analyses used SPSS 24 software.

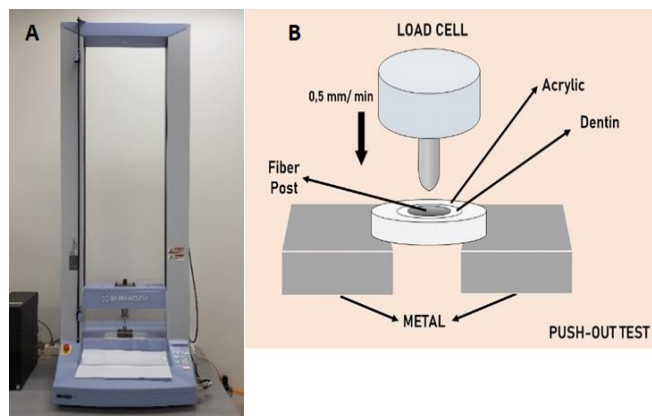


Figure 2. A. Universal testing machine (Shimidzu AG 5000E). B. Schematic illustration of the push-out bond strength test.

Results

All the specimens showed measurable adhesion to root dentine. The results of the one-way ANOVA on the fiber post push-out bond strength test data are presented in Table 1. There was a statistically significant difference in adhesion strength ($p < 0.05$) between the two control groups (epoxy resin vs. calcium silicate root canal cement). The highest average adhesion strength was exhibited by the control group (57.142 ± 12.205 MPa), followed by the epoxy resin group (44.455 ± 10.347 MPa) and then the calcium silicate group (28.009 ± 9.562 MPa).

Group	n	Mean (± SD)	p-value
Control	10	57.142 (± 12.205)	0.000*
AH-Plus	10	44.455 (± 10.347)	
Ceraseal	10	28.009 (± 9.562)	

Table 1. Mean and significance values for push-out bond strength (Mpa) in control, epoxy resin (AH-Plus), and calcium silicate (Ceraseal) groups. *One-way ANOVA (p < 0.05) showed significant differences.

A Bonferroni post hoc test was used to determine the significance of the intergroup significance. Based on the results presented in Table 2, there were significant differences (p < 0.05) between all the tested pairs of groups; specifically, there were significant differences between the control group and the epoxy resin group (p = 0.041), between the control group and the calcium silicate group (p = 0.000), and between the epoxy resin group and the calcium silicate group (p = 0.006).

	Control	AH-Plus	Ceraseal
Control		0.041*	0.000*
AH-Plus			0.006*
Ceraseal			

Table 2. Significance of differences in push-out bond strength between control, epoxy resin (AH-Plus), and calcium silicate (Ceraseal) groups. *Post-hoc Bonferroni (p < 0.05) showed significant differences.

	Adhesion failure	Cohesive failure	Mixed failure	Total
Control	0 (0%)	1 (10%)	9 (90%)	10 (100%)
AH-Plus	1 (10%)	1 (10%)	8 (80%)	10 (100%)
Ceraseal	2 (20%)	1 (10%)	7 (70%)	10 (100%)
Total	3 (10%)	3 (10%)	24 (80%)	30 (100%)

Table 3. Cross-tabulation of bonding failure between control, epoxy resin (AH-Plus), and calcium silicate (Ceraseal) groups.

The mode of failure between the fiber post, resin cement, and dentine was examined under a stereomicroscope camera and then classified into one of three failure modes described by Skidmore et al. The bond failure percentages of the groups are presented in Table 3. In all groups, the most frequent types of bonding failure were mixed adhesion-cohesion failure (90% in the control group, 80% in the epoxy resin group, and

70% in the calcium silicate group), cohesion failure (10% in all groups), and adhesion failure (0% in the control group, 10% in the epoxy resin group, and 20% in the calcium silicate group).

Discussion

Fiber post retention depends on interactions between root canal dentin, resin cement, and post surfaces.¹⁴ Most fiber post restoration failures are caused by inadequate bonding.² Therefore, the success of fiber post cementation depends on the bond and good adhesion to the dentin of the root canal wall. The adhesion strength of a dentin-cement, resin-fiber post is influenced by various factors, one of which is the presence of a secondary smear layer consisting of residual root canal cement or gutta-percha from previous root canal treatment and post preparation.^{8,15}

In the present study, a push-out test was carried out by applying pressure to the surface of a dentin-cement, resin-fiber post, simulating the actual clinical condition of an occlusal load on the tooth. This testing method was chosen because it provides an accurate, reliable estimation of resistance to bonding that is easily interpreted; furthermore, this test can detect and analyze maximum load with a machine and tools that are relatively easy to use.^{16,17}

Epoxy resin-based and calcium silicate-based cements are the two types of materials most widely used for root canal obturation. Epoxy resin-based root canal sealer has the advantage of good sealing ability, and it is also said to be easier to remove during post preparation and cleaning. Meanwhile, calcium silicate-based root canal cement is more popular due to its superior bioactive ability to form bonds and initiate mineralization and the formation of new hard tissues. However, a previous study reported that the use of calcium silicate/bioceramic root canal cement produces a lower level of cleanliness compared to epoxy resin root canal cement and zinc oxide after fiber post preparation.⁵ This is thought to influence the bonding of restorative material adhesives, including the bonding of resin cement to dentin after fiber post placement.

Strong fiber post adhesion to the root canal wall is achieved through the micromechanical retention of demineralized surfaces and the formation of resin tags. However, post chamber preparation with a high-speed drill creates a

secondary smear layer consisting of gutta-percha and root canal cement, which covers the dentin surface and may reduce micromechanical retention and fiber post bonding strength.¹⁸ Therefore, the root canal dentin surface must be effectively and thoroughly cleaned before fiber post cementation. As stated previously, root canal cement is one component of the secondary smear layer. Each type of root canal cement has unique physical and chemical properties and involves a particular level of difficulty to clean; to date, the effects of these factors on fiber post adhesion strength have remained uncertain. To determine these effects, the present study analyzed the effect of the type of root canal cement used for root canal treatment—particularly epoxy resin versus calcium silicate root canal sealer—on the adhesion strength of fiber posts to the dentin walls of the root canal.

According to the results presented in Tables 1 and 2, there were significant differences in mean push-out bond strength (i.e., fiber post adhesion strength) between all test groups. The highest push-out bond strength was exhibited by the control group (no root canal cement), followed by the AH-Plus (epoxy resin-based) group and then the Ceraseal (calcium silicate-based) group. The superior strength of the control group was consistent with the results of several previous studies.^{9,19} This result was likely attained because open dentinal tubules without residual root canal cement allow for maximum resin cement penetration.⁹

The fiber post adhesion strength of the tooth that was previously filled with AH-Plus root canal cement was higher than that of the group that had previously used Ceraseal root canal cement. Cohen et al. reported that epoxy resins in resin-based root canal cements, such as AH-Plus, did not interfere with the activation of free radicals in composite resins, such that resin-based root canals had no adverse effects on resin adhesion.²⁰ In a study by Cecchin et al., it was reported that the high adhesion strength of a root canal cement was due to its epoxy resin composition, which was similar to a resin cement composition.²¹ In the present study, the epoxy resin root canal cement was easier to clean than calcium silicate root canal cement during post space preparation. This is in accordance with those of previous studies comparing the root canal cleanliness of several types of cement after endodontic treatment.² It is suspected that a

cleaner dentin surface of the root canal after post preparation corresponds to better penetration of resin cement into the root canal wall as well as higher bonding ability/adhesion of the fiber post to the root canal wall.

The Ceraseal group exhibited the lowest adhesion strength compared to the other groups. This was likely because Ceraseal is a calcium silicate-based root canal cement with high bonding strength due to the micromechanical tag produced by the biomineralization process. This type of root canal cement is reported to have stronger adhesion to root canal dentin walls than other popular root canal cements.^{22,23} Calcium silicate cement interacts with dentinal tubule fluid, creating an intrafibrillar apatite deposit and forming a tag-like structure with dentin, which provides further bonds between the root canal cement and the dentine surface. At the dentine interface, the root canal forms a layer of ion and mineral exchange from the root canal cement into the dentine; this layer is called the mineral infiltration zone. In addition, previous *in vitro* studies have reported cases in which the intratubular diffusion of calcium silicate formed mineral blockages in the dentinal tubules, which can interfere with any other adhesive materials used thereafter.²⁴ The same bonding strategy has also been applied to the monoblock concept of root canal obturation to produce a good seal in the coronal and apical areas.²⁵ This strong bond between the dentine and the root canal cement is likely to make this type of cement difficult to clean with conventional techniques, especially when the cement has hardened.¹¹ Thus, the dentin tubules on the root canal surface can become clogged such that the adhesive system produced by the fiber post cementation cannot provide an optimal bond because the micromechanical bonds cannot form properly. As a consequence, the calcium silicate cement exhibited the lowest adhesion strength, as indicated by the push-out bond strength, compared to the other two groups.

Stereomicroscopic observations (30x magnification) revealed bonding failures after the push-out bond strength test in all groups (Figure 3).

Based on the results shown in Table 3, the most common type of bonding failure across all groups was mixed adhesion-cohesion failure, followed by cohesion failure and then adhesion failure. The high percentages of mixed failure

and cohesion failure indicate that the bonds between the dentin and the resin cement were relatively good in most of the specimens, as parts of the resin cement remained attached to the root canal walls even though maximum pressure was applied to the fiber posts. The absence of adhesion failure in the control group indicated that the bonding of the resin cement to the root canal walls was good, presumably due to the absence of residual root canal sealers that could interfere with micromechanical bonds. The number of adhesion failures in the calcium silicate group, which was higher than in the other groups, was likely due to the calcium silicate layer. Calcium silicate is difficult to clean because it binds to the root canal wall, thus blocking the micromechanical bond between the resin cement and the dentin of the root canal wall. In all groups, a small proportion of the samples exhibited cohesive failure. This failure may have been caused by imperfect pretreatment procedures or procedural errors in the application of the bonding material to the post surface before the cementation stage.

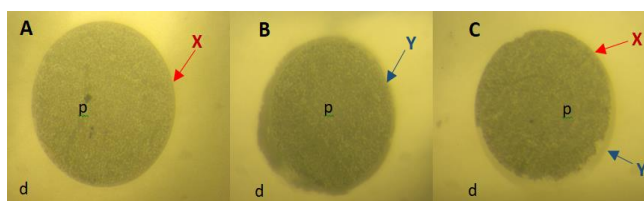


Figure 3. Stereomicroscopic images (x30 magnification) for bonding failure analysis. d: dentin, p: fiber post. **A.** Example of adhesive failure, with no material left on the root canal wall (mark X). **B.** Example of cohesive failure, with material present on the entire root canal wall (mark Y). **C.** Example of mixed failure, with patches of material on the root canal wall (mark X for adhesive failure, mark Y for cohesive failure).

Conclusions

Based on the results of this study, it can be concluded that the endodontically treated teeth that had previously been treated with epoxy resin-based root canal sealer exhibited better fiber post adhesion strength than those previously treated with calcium silicate-based root canal sealer. The choice of epoxy resin-based root canal sealer may be taken into consideration in daily clinical applications,

especially when a fiber post is planned to be used after endodontic treatment.

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Declaration of Interest

The authors declare that they have no conflicts of interest to disclose.

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