Comparative Study on Fiber Post Adhesion according to Calcium Silicate and Resin-based Endodontic Sealer

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Comparative Study on Fiber Post Adhesion according to Calcium Silicate and Resin-based Endodontic Sealer

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감사의 글

먼저 보존과 학위 과정과 수련 생활을 잘 마무리할 수 있도록 인도해주신 하나님께 감사드립니다.

또한 부족한 제가 학위 논문을 작성할 수 있도록 세심하게 도움을 주신 노병덕 지도교수님께 진심으로 감사드립니다. 연구 설계를 하고 실험을 진행하면서 여러 방면에서 부족함과 어려움을 느끼는 순간이 많았습니다. 그 때마다 교수님께서는 저에게 조언을 아끼지 않으시고 따뜻한 격려를 보내주시며 지도해 주셔서 제가 학위 논문을 무사히 완성할 수 있게 되었던 것 같습니다. 또한, 학위 과정뿐만 아니라, 임상 시간, 혹은 case presentation 시간에도 때로는 다정하고, 때로는 엄격한 조언을 주시며 제가 성장할 수 있도록 지도해주신 교수님께 이 지면을 빌어 다시 한 번 존경과 감사의 말씀을 드립니다.

또한, 많이 부족한 제 논문을 바쁘신 와중에도 심사해주시고, 세심하게 논문에 대해 지도해 주시며 다방면으로 조언을 주신 신유석 교수님, 김도현 교수님께도 깊이 감사드립니다.

3 년 전 처음 보존과 레지던트를 합격했을 때부터 지금에 이르기까지 어엿한 보존과 의사로 성장할 수 있도록 이끌어 주신 박성호 교수님, 김의성 교수님, 정일영 교수님, 박정원 교수님, 신수정 교수님, 김선일 교수님, 강수미 교수님께도 감사의 말씀을 드립니다. 교수님들의 가르침을 통하여 진료나 학문적인 측면, 그리고 보존과 의사로서 가져야 할 마음가짐을 배울 수 있었습니다.

무엇 하나 저 혼자 해낸 것이 없었으며, 이 논문이 완성되기까지 순간순간 마다 교수님들, 의국 선후배, 동기들, 연구원 선생님들의 도움이 있었기 때문에 가능했던 것 같습니다. 특히, 의국 생활을 하면서 즐거울 때나 힘들 때나 서로 힘이

되어주고 응원해주었던 우리 보존과 의국 동기와 선후배 의국원들에게 고맙다는 인사를 전하고 싶습니다.

마지막으로 6 년의 치과대학 생활과 4 년의 전공의 수련이라는 긴 시간동안 아낌없는 사랑으로 격려해주시고 지원해주신 사랑하는 아버지, 어머니, 그리고 동생에게도 감사의 말씀을 전합니다.

2022 년 12 월

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Abstract

Comparative Study on Fiber Post Adhesion according to Calcium Silicate and Resin-based Endodontic Sealer

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(Directed by Professor Byoung-Duck Roh, D.D.S., M.S.D., Ph.D.)

This study aimed to evaluate the influence of the type of endodontic sealers and the time of fiber post cementation after root canal filling on the glass-fiber post adhesion to root dentin with self-adhesive resin cement.

Sixty-six human mandibular premolars were decoronated, and root canal instrumentation was done chemo-mechanically. The teeth were randomly divided into six groups according to the type of sealer and time of post cementation : (1) AH Plus with post cementation immediately after root canal filling (AH-IM), (2) AH Plus with post cementation 48 hours after root canal filling (AH-DE) (3) Ceraseal with post cementation immediately after root canal filling (CE-IM), (4) Ceraseal with post cementation 48 hours after root canal filling (CE-DE), (5) Endoseal with post cementation immediately after root canal filling (EN-IM), (6) Endoseal with post cementation 48 hours after root canal filling (EN-DE). After the post cementation, 2-mm-thickness slices were produced, and the push-out bond strength test was performed. The failure mode was examined under a stereomicroscope. The sectioned surface was also examined by a scanning electron microscope. The fluorescence marking process was done in the two teeth from each group for examination by a confocal laser scanning microscope. Data were analyzed using the ANOVA test followed by post hoc Tukey's test and independent t-test. The statistical significance level was set at $p<0.05$.

In the case of immediate post cementation after root canal filling, AH-Plus showed significantly higher push-out bond strength than Ceraseal and Endoseal. In the case of delayed post cementation, there was no significant difference according to the type of sealer. Only AH-Plus showed significantly different bond strength between the two different post cementation time, whereas Ceraseal and Endoseal did not show significance. The predominant failure type was the adhesive failure between resin cement and radicular dentin (88%).

The gap distance was wider in the case of immediate post cementation than in delayed post cementation in all sealer groups, while a significant difference was only in the Ceraseal group. In the case of immediate post cementation, there was no significant difference among the three types of sealer. In contrast, there was a significant difference between AH-Plus and Endoseal in delayed post cementation.

Therefore, our finding suggests that AH-plus can be a better choice for root canal filling than calcium silicate-based sealer when immediate post cementation is planned. Sufficient setting time for the root canal filling with calcium silicate-based sealer is needed to get better fiber post retention.

Keywords: endodontic sealer; calcium silicate; epoxy resin; self-adhesive resin cement; fiber post; bond strength; SEM; CLSM

Comparative Study on Fiber Post Adhesion according to Calcium Silicate and Resin-based Endodontic Sealer

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I. Introduction

Endodontic treatment is a process to induce the recovery of periapical tissue through the removal of bacteria, infective dentin, and necrotic pulp tissue in the root canal. Thus, appropriate canal filling material and endodontic sealer which can adequately seal the root canal system are required. Epoxy resin, zinc oxide-eugenol, calcium hydroxide, glass ionomer, and calcium silicate can be such base materials for endodontic sealers (Vilas-Boas et al., 2018). Recently commercialized calcium silicate-based endodontic sealer has alkaline pH, biocompatible properties, formation of hydroxyapatite, root canal sealing action by bonding to dentin, and expansion during setting with moisture (Alsubait, 2021).

In the case of teeth with severe damage to the crown due to dental caries or fractures, dentists need to make maintenance and resistance forms during preparation using post and core materials after root canal treatment to restore the function and aesthetics of the teeth (Hayashi et al., 2006). Typically, cast posts or ready-made metal posts have been used for this purpose. However, metal posts have a higher elastic modulus than dentin, which can cause cracks or fractures in teeth and aesthetic problems (Figueiredo et al., 2015). Among the various types of posts, fiber posts have flexibility similar to dentin and low rigidity, which may lead to stress dispersion and reduce the root fractures through adhesion with dentin and be used as an alternative to metal posts, increasing its usability (Santos-Filho et al., 2014).

The adhesion mechanism between previously treated teeth and fiber posts can be explained in chemical and micro-mechanical ways (Zicari et al., 2008). The success of previously treated teeth with intracanal posts depends on the appropriate choice of canal filling material, sealer, and the type of intracanal post. Most fiber post-failure occurs at the interface between the root dentin and resin cement (Monticelli et al., 2003), and the type of sealer can affect the bond strength of the fiber post and the root dentin.

Studies on the adhesion interface characteristics between fiber post and root canal dentin in the case of root canal filling using epoxy resin and calcium silicate-based sealer are insufficient and controversial. AH-plus showed significantly higher push-out bond strength than Endosequence BC sealer regardless of post cementation time (Vilas-Boas et al., 2018). On the other hand, another study showed similar pull-out bond strength between AH-plus and BC Hiflow sealer on 7 days delayed post cementation after canal filling (Alsubait, 2021).

Thus, this study aimed to evaluate and compare the push-out bond strength of glass-fiber posts and the adhesion surface between the post and dentin according to the type of endodontic sealers and the time of post cementation. The adhesion surfaces were observed by stereomicroscope, scanning electron microscope, and confocal laser scanning microscope. The null-hypotheses were as follow: (1) There is no significant difference between the push-out bond strength of fiber posts cemented in root canals filled with calcium silicate-based sealer and epoxy resin-based sealer. (2) There is no significant difference in the push-out bond strength of fiber posts cemented in root canals according to the post cementation time. (3) There is no interaction between the two factors; the type of sealer and post cementation time for push-out bond strength.

II. Materials and methods

1. Specimen preparation

This study was approved by the Institutional Review Board of Yonsei University Dental Hospital (approval number: 2-2021-0089). Sixty-six extracted single-rooted mandibular premolars with linear canals were stored in 0.9% sodium chloride (NaCl). The sample teeth were assigned from the Human Derivatives Bank of Yonsei University Dental Hospital. Teeth with dental caries, curved canal, resorption, crack, open apex, and previously treated were excluded. Fifty-four teeth were used for the push-out bond strength test. Twelve teeth were used for confocal laser scanning microscopy analysis. External debris was removed, and the crown portion was mechanically removed to the cemento-enamel junction with a diamond bur. The minimum root canal length should be 14mm.

2. Endodontic treatment

Working length was determined by subtracting 1mm from the length of a #10 K-file after observing its tip at the apical foramen, and canal patency was confirmed. All root canals were prepared up to MAF #40 K-file. Canal preparation was done by Profile (Dentsply Sirona, Ballaigues, Switzerland) up to MAF #40 with the crown-down technique. Canal enlargement on the coronal and middle portion was done by #2,3 Gates-Glidden (Mani Inc., Tochigi-ken, Japan) drills. After each instrumentation, canal irrigation was carried out with 1ml 2.5% sodium hypochlorite (NaOCl). Final irrigation was done with 1 ml of 17% EDTA solution for 1 minute, followed by 3 ml of 2.5% NaOCl. Sterile saline was used for the final flush, and the canal was dried with paper points. Specimens were randomly distributed into three groups, and each group was divided into two subgroups according to the type of endodontic sealer and time of fiber post cementation (n=9 each) as follows (Table 1). Root canal filling was done, followed by the manufacturers' instructions for each type of sealer (Table 1, 2).

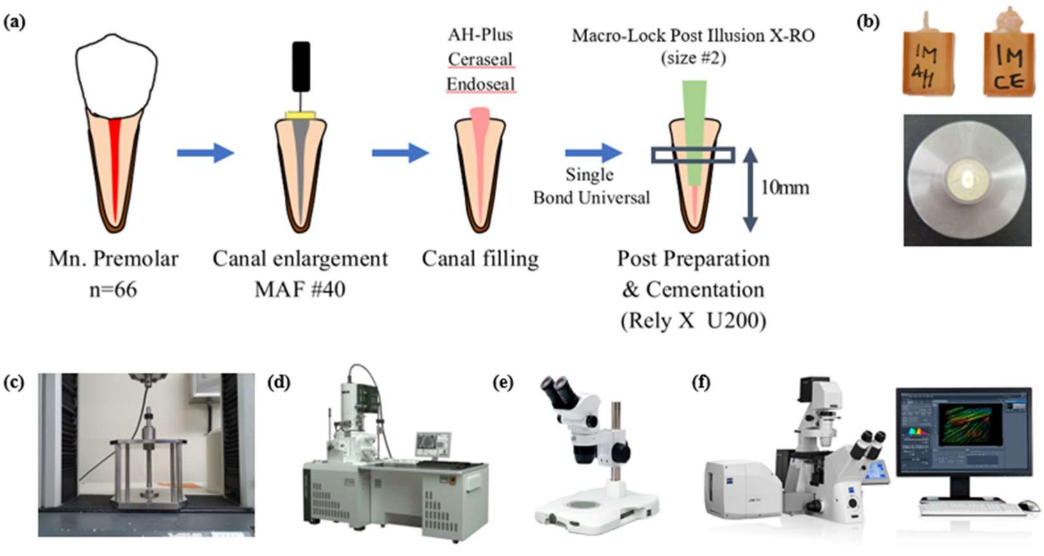

Figure 1. Scheme of the experiment. (a) Procedure of root canal treatment (b) Disc formation by embedding with acrylic resin, (c) Push-out bond strength test with the universal machine (Instron series IX, ITW, MA, USA), (d) Scanning electron microscope analysis (JEOL-7800F, JEOL Ltd., Tokyo, Japan), (e) Stereomicroscope for observation of failure mode (SZ 61, Olympus Ltd., Tokyo, Japan), (f) Confocal laser scanning microscope analysis (LSM 700, Carl Zeiss, Oberkochen, Germany).

Group (n=11 each)	Sealer	Canal filling technique	Post cementation time
AH-IM	AH-plus	Continuous wave technique	Immediately after canal filling
AH-DE			48 hours after canal filling
CE-IM	Ceraseal	Sealer-based technique	Immediately after canal filling
CE-DE			48 hours after canal filling
EN-IM	Endoseal	Sealer-based technique	Immediately after canal filling
EN-DE			48 hours after canal filling

Table 1. Group distribution of the specimens. The specimens were divided into three groups according to the endodontic sealers (AH:AH-plus, CE: Ceraseal, EN: Endoseal) and each group was divided into two subgroups (n=11 each, IM: immediately post cementation, DE: delayed post cementation) according to the post cementation time after canal filling. Nine teeth were used for push-out bond strength test and two teeth were used for CLSM analysis for each subgroup.

Materials	Type of sealer	Composition
AH-plus® (DeTrey/Dentsply, Konstanz, Germany)	Epoxy resin-based	Paste A: epoxy resin; calcium tungstate; zirconium oxide; aerosol; iron oxide
		Paste B: 1-adamantane amine; 'N,N-dibenzyl-5-oxa- nonandiamine-1,9; TCD- diamine; calcium tungstate; zirconium oxide; aerosol; silicone oil
Ceraseal (Meta Biomed Co., Cheongju, Korea)	Calcium silicate-based	Tricalcium silicate, dicalcium silicate, calcium aluminate, zirconium oxide, thickening agent
Endoseal MTA (Maruchi, Wonju, Korea)	Calcium silicate-based	Calcium silicate, calcium aluminate, calcium aluminoferrite, calcium sulfates, radiopacifier, thickening agent
Single bond universal (3M ESPE, St. Paul, MN, USA)		10-MDP phosphate monomer, Vitrebond copolymer, HEMA, dimethacrylate resins, filler, silane, initiators, ethanol, water
Monobond N (Ivoclar Vivadent, Liechtenstein, Germany)		Alcohol solution of silane methacrylate, phosphoric acid methacrylate, sulfide methacrylate
Rely X U200 (3M ESPE, St. Paul, MN, USA)		Base: Methacrylate monomers containing phosphoric acid groups, methacrylate monomers, initiators, stabilizers, rheological additives
		Catalyst: Methacrylate monomers, alkaline fillers, silanated fillers, initiator components, stabilizers, pigments, rheological additives. Zirconia/silica fillers

Table 2. Composition of the materials used in the experiment

3. Fiber post cementation

Macro-Lock Post Illusion X-RO (size #2, RTD Inc., St. Egreve, France) was used for post cementation. Fiber posts were cemented immediately after root canal filling for the specimens of immediate post cementation or 48 hours after root canal filling for the specimens of delayed post cementation. Gates Glidden drills with size #2 and #3 were used to remove the filling material except for apical 4mm. Canal preparation was performed with #1 starter and #2 finishing post drill (RTD Inc., St. Egreve, France) to remove the remaining root canal filling materials except for the apical 4mm by the manufacturers' instruction. The prepared post space was rinsed with 2.5% sodium hypochlorite and distilled water for the final irrigation and dried using paper points. The posts were disinfected with alcohol, and were silanized with Monobond N (Ivoclar Vivadent, Liechtenstein, Germany. The canal space was bonded with Single Bond Universal (3M ESPE, St. Paul, MN, USA). After the application of resin cement with Rely X U200 (3M ESPE, St. Paul, MN, USA), the post was seated gently with finger pressure, and the extra cement was removed. The cement was light-cured for 20 seconds. All specimens were stored at room temperature and 100% humidity for 24 hours before further specimen preparation.

4. Disc samples preparation

Resin molds (12×12×14mm) were made by CAD and 3D printing; designed by Meshmixer (Autodesk Inc., San Rafael, CA, USA) and printed using a 3D printer (Nextdent 5100, Nexdent BV, Soesterberg, Netherlands) with 3D printing resin (Mazic D SG, VERICOM Co., Ltd., Chuncheon, Korea). The root portion of the teeth was embedded vertically into the mold with clear acrylic resin (Ortho-Jet, Lang Dental Manufacturing Co., Wheeling, IL, USA) (Figure 2).

To maintain the post's vertical direction, the post was placed in the center of the mold when observed from the top, perpendicularly to the base. The post was parallel to all axial walls when observed from the front. Through this, it was possible to cut teeth perpendicular to the post and dental axis when sectioning the specimens.

After the acrylic resin was completely set, each specimen was sectioned in a horizontal plane perpendicular to the root axis using a low-speed precision diamond saw (TOPMET Metsaw-LS, R&B, Daejeon, Korea) under constant water cooling. Then, 2mm-thickness samples were taken.

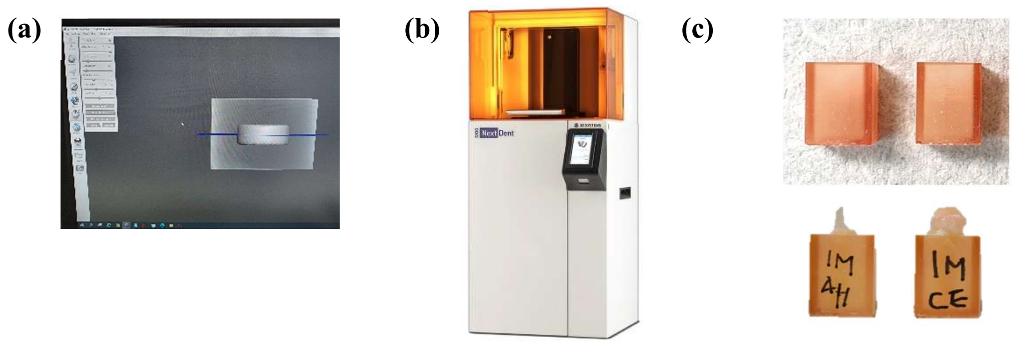

Figure 2. Fabrication of resin mold. (a) CAD design for resin mold by Meshmixer (Autodesk Inc., CA, USA) (b) 3D printing machine (Nextdent 5100, Nexdent BV, Soesterberg, Netherlands) (c) Resin mold after CAD/3D printing process(upper) and the tooth specimen embedded with acraylic resin in the mold (lower).

5. Push-out bond strength test

The specimens were secured in the universal testing machine (Instron series IX, ITW, MA, USA) for the push-out bond strength test with a constant crosshead speed of 0.5mm/min until it reached the maximum load, which was sufficient to dislodge fiber post from the dentin (Figure 3). A plunger tip of 1mm diameter was used to apply vertical force over the fiber post. The maximum load was recorded in newtons(N). Using the following formula, push-out bond strength (MPa) was calculated: Maximum load (N)/area of fiber post (mm^2). The area of fiber post was calculated using $\pi(R+r)[(h^2+(R-r)^2]/2$ where R(mm), r(mm) and h(mm) are larger radius, smaller radius and the height of the root section, respectively. The radius and the thickness were measured by a digital caliper.

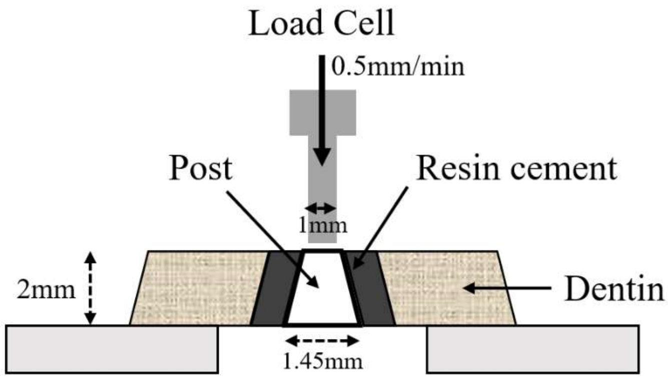

Figure 3. Scheme of the push-out bond strength test

6. Evaluation of failure mode

After the push-out bond strength test was done, a stereomicroscope (SZ 61, Olympus Ltd., Tokyo, Japan) was used to examine the surface of debonded posts at ×30 magnification and identify the type of failure: adhesion failure between fiber post and resin cement, adhesive failure between dentin and resin cement, cohesive failure of fiber post, resin cement or dentin and mixed failure, which is the combination of the adhesive and cohesive failure.

7. Scanning electronic microscopy (SEM) analysis

For specimens for the scanning electronic microscopy (SEM) analysis, four teeth in each subgroup were examined. The surfaces of the specimens were polished with #1200-

grit abrasive sandpaper with a grinding machine (R&B Inc., Daejeon, Korea). Next, the specimens were rinsed with distilled water and ultrasonic for 1 minute, dried at 37 °C for 24 hours, placed in a vacuum chamber, and sputter-coated with a gold layer. Each surface was examined by scanning electronic microscope (SEM, JEOL-7800F, JEOL Ltd., Tokyo, Japan) to evaluate the gap distance between resin cement and dentin at a magnification of ×500, ×1000, and ×2000. Gap distance was measured on three different points for each specimen.

8. Confocal laser scanning microscopy (CLSM) analysis

Two extra teeth in each subgroup were prepared for confocal laser scanning microscopy analysis. Each sealer was labeled by 0.1% sodium fluorescein (FNa; Daejung Chemicals, Seoul, Korea), bonding agent (Single bond universal, 3M ESPE, Germany) was labeled by rhodamine B (Sigma Aldrich, Steinheim, Germany), and resin cement (Rely X U200) was labeled by DAPI (Sigma Aldrich, Steinheim, Germany). The process, such as endodontic treatment and fiber post cementation, was performed the same way as mentioned above. Specimen sectioning was done within 1mm of the thickness of the specimen, which is an adequate sample thickness for CLSM analysis. CLSM analysis was performed with an LSM-700 microscope (Carl Zeiss, Oberkochen, Germany) at a magnification of ×20. The fluorescent materials of DAPI, sodium fluorescein, and rhodamine B were detected by the wavelength of 405nm, 488nm, and 555nm.

9. Statistical analysis

All statistical analyses were done by SPSS 26 software (IBM Corp., New York, NY, USA). According to the Shapiro-Wilk test, the data were normally distributed in all groups. The quantitative data were statistically analyzed with a two-way ANOVA test to evaluate the interaction between the type of sealer and post cementation time. If there was a statistical significance, a one-way ANOVA test followed by post-hoc Tukey's test was performed for multiple comparisons. Independent T-tests were performed to evaluate the difference according to the post cementation time in each sealer group. The significance level was set at $p=0.05$.

III. Results

1. Push-out bond strength test

Table 3 and Figure 4 show the bond strength values measured by the push-out bond strength test for all experimental groups. According to the two-way ANOVA test, there was a significant interaction between the type of sealer and post cementation time (p=0.037). When posts were cemented immediately after root canal filling, one-way ANOVA revealed significant differences between the type of sealers (p=0.006). AH-Plus showed higher bond strength than Ceraseal and Endoseal (p=0.022, 0.008, respectively). There was no significant difference between Ceraseal and Endoseal (p=0.819). However, when posts were cemented 48 hours after root canal filling, there was no significant difference among the three sealer groups (p=0.701).

For comparing the factor of post cementation time in each sealer group, an independent t-test was performed. The push-out bond strength in the AH-Plus group showed a significant difference (p=0.004) according to the post cementation time. The immediate post cementation group showed higher bond strength than the delayed post cementation. However, there was no significance in Ceraseal and Endoseal groups according to the cementation time (p=0.282, 0.39, respectively).

The statistically significant difference between AH-IM and AH-DE, AH-IM and CE-IM, and AH-IM and EN-IM are shown in Figure 4.

Group (n=9 each)		Push-out bond strength (MPa)		
Sealer	Timing	Mean(SD)	Median(Q3-Q1)	Min-Max
AH-plus	Immediate	19.52(±4.15) [A,a]	20.42(6.25)	12.66-25.13
	Delayed	12.44(±3.39) [B,c]	12.85(3.51)	5.44-15.66
Ceraseal	Immediate	10.57(±6.58) [C,b]	9.25(10.07)	3.25-20.45
	Delayed	14.89(±7.47) [C,c]	13.81(13.77)	4.72-24.39
Endoseal	Immediate	12.44(±3.39) [D,b]	12.42(5.46)	7.83-18.50
	Delayed	15.66(±9.84) [D,c]	14.95(11.05)	4.45-35.06

Table 3. Push-out bond strength for each group. Different superscript uppercase letters (A,B,C,D) indicate significant differences depending on the post cementation time in the same sealer (P<0.05). Different superscript lowercase letters (a,b,c) indicate significant differences according to the type of sealer at the same post cementation time. (P<0.05).

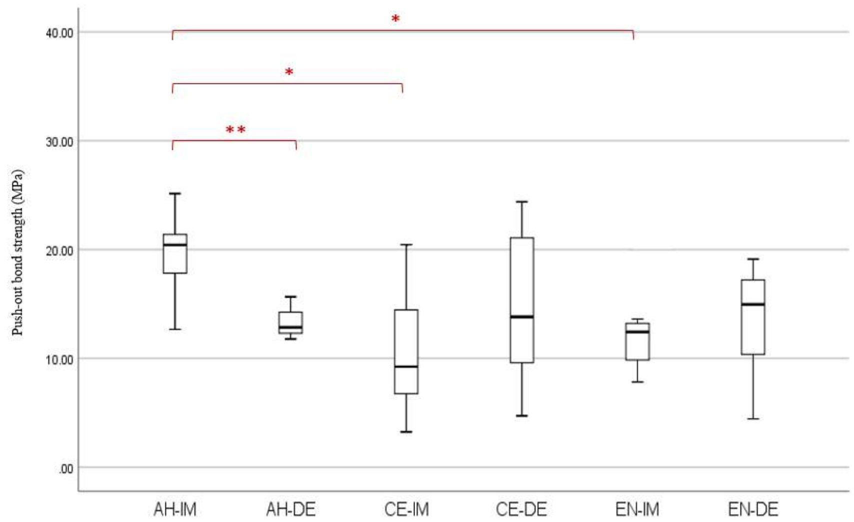

Figure 4. Box plot of the push-out bond strength. * indicates significant difference by post-hoc Tukey's test (p<0.05), ** indicates significant difference by Independent t-test (p<0.05).

2. Stereomicroscopy image

After the push-out bond strength test, the residual sealer and failure mode was observed in the cross-section using a stereoscopic microscope (Figure 5). In all sealer groups, adhesive failure between dentin and resin cement was observed (a-f). Mixed failure included cohesive failure in resin cement, adhesive failure between dentin and resin cement, and between fiber post and resin cement (a, e, f). Also, the fracture line starting from the separation between resin cement and fiber post, extended into the dentin in the specimens with mixed failure.

The results of observing the failure mode after the push-out bond strength test are shown in Table 4. Adhesive failure between dentin and cement was observed in all samples. There was a mixed failure in 33% of the AH-IM group and 11% of the CE-DE, EN-IM, and EN-DE groups. However, cohesive failure only did not occur.

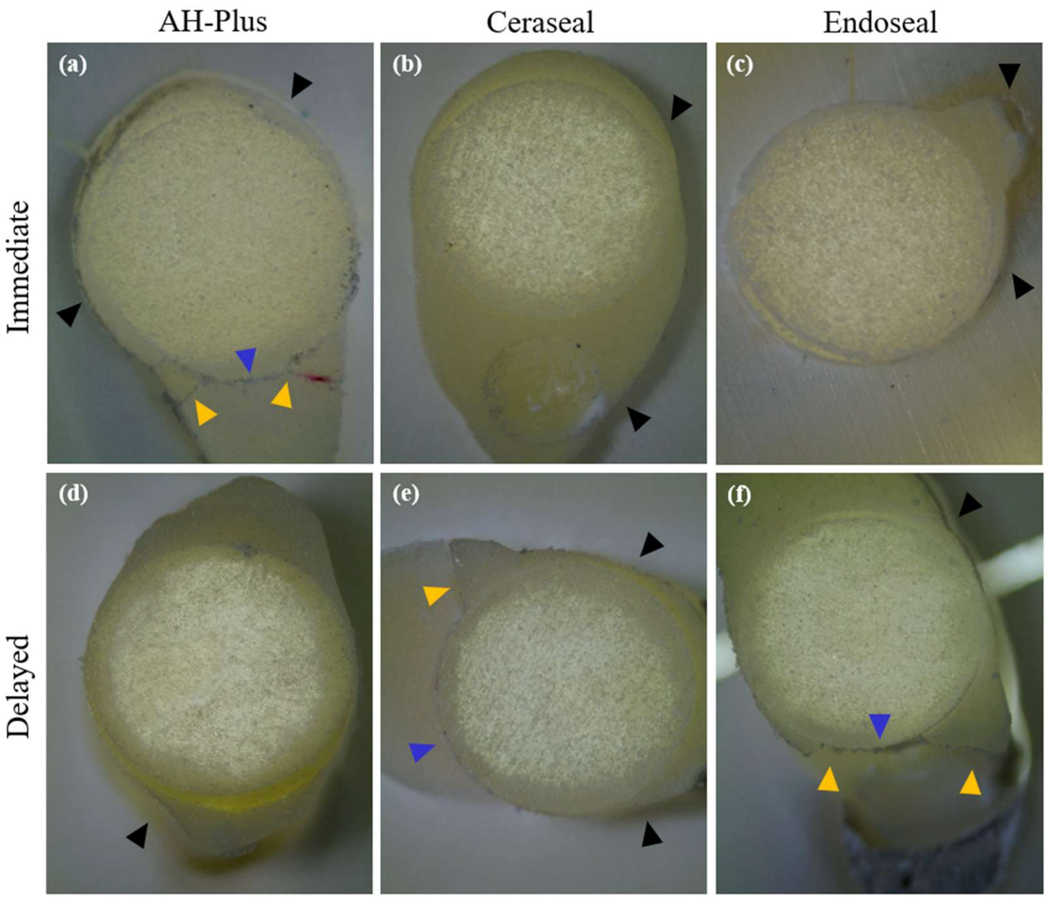

Figure 5. Stereomicroscopic images after push-out bond strength test. Adhesion failure between dentin and resin cement is marked with black arrow, adhesion failure between resin cement and fiber post is marked with blue arrow and cohesion failure in resin cement is marked with yellow arrow. (a) AH-IM group specimen with mixed failure. (b) CE-IM group specimen with adhesion failure. (c) EN-IM group specimen with adhesion failure. (d) AH-DE group specimen with adhesion failure. (e) CE-DE group specimen with mixed failure. (f) EN-DE group specimen with mixed failure.

Group	Failure mode		
(n=9 each)	Adhesive failure (Dentin-Cement)	Mixed failure	Cohesive failure
AH-IM	66%	33%	0%
AH-DE	100%	0%	0%
CE-IM	100%	0%	0%
CE-DE	88%	11%	0%
EN-IM	88%	11%	0%
EN-DE	88%	11%	0%

Table 4. Failure mode after push-out bond strength test for each group

3. Scanning electron microscopy analysis

Table 5 and Figure 6 show the values of gap distance measured at the interface between dentin and cement observed through a scanning electron microscope.

According to the two-way ANOVA test, there was no significant interaction between the two factors, type of sealer and post cementation time on gap distance (p=0.369). However, a one-way ANOVA test performed in each factor revealed that the type of sealer and post cementation time significantly influence the gap distance (p=0.001, <0.001, respectively). Overall, AH-plus (0.90±0.36μm) showed a significantly narrower gap distance than Ceraseal (1.29±0.61μm) and Endoseal (1.47±0.60μm) (p=0.001, 0.034, respectively), whereas there was no significant difference between Ceraseal and Endoseal (p=0.451). According to post cementation time, the gap distance was wider in the case of immediate post cementation (1.46 ±0.58μm) than in delayed post cementation (0.97 ±0.47μm) with a significant difference (p=0.001).

When posts were cemented immediately after root canal filling, there was no significant difference in gap distance among the three types of sealer (p=0.066). However, when posts were cemented 48 hours after root canal filling, a significant difference was shown (p=0.001), especially between AH-Plus and Endoseal (p=0.008). There was no significant difference between AH-Plus and Ceraseal (p=0.594) and Ceraseal and Endoseal (p=0.372).

For comparing the factor of post cementation time in each sealer group, an independent t-test was performed in each sealer group. Ceraseal showed a significant difference between immediate post cementation and delayed post cementation ($p=0.01$). At the same time, AH-Plus and Endoseal did not show a significant difference between the two different post cementation times ($p=0.097$, 0.656, respectively).

Group (n=12 each)		Gap distance between dentin and cement (㎛)		
Sealer	Timing	Mean(SD)	Median(Q1-Q3)	Min-Max
AH-plus	Immediate	1.15(±0.24) ^{A,a}	1.21(0.43)	0.76-1.49
	Delayed	0.61(±0.25) ^{A,b}	0.70(0.40)	0.20-1.08
Ceraseal	Immediate	1.63(±0.49) ^{B,a}	1.62(0.66)	0.59-2.40
	Delayed	0.94(±0.54) ^{C,b,c}	1.05(1.00)	0.20-1.71
Endoseal	Immediate	1.62(±0.78) ^{D,a}	1.76(1.60)	0.42-2.71
	Delayed	1.33(±0.28) ^{D,c}	1.33(0.35)	0.77-1.80

Table 5. Gap distance between dentin and cement for each group. Different superscript uppercase letters (A,B,C,D) indicate significant differences depending on the post cementation time in the same sealer ($P<0.05$). Different superscript lowercase letters (a,b,c) indicate significant differences according to the type of sealer at the same post cementation time. ($P<0.05$).

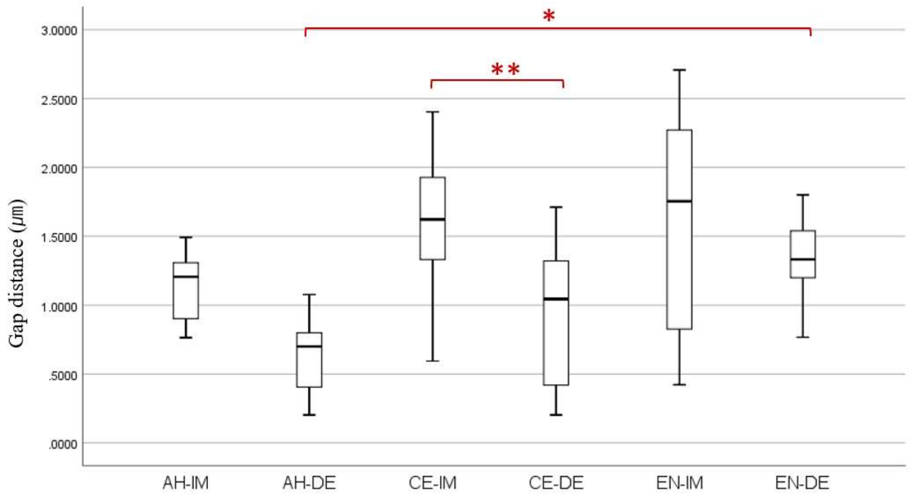

Figure 6. Box plot of gap distance between dentin and cement. * indicates significant difference by post-hoc Tukey's test ($p<0.05$), ** indicates significant difference by Independent t-test ($p<0.05$).

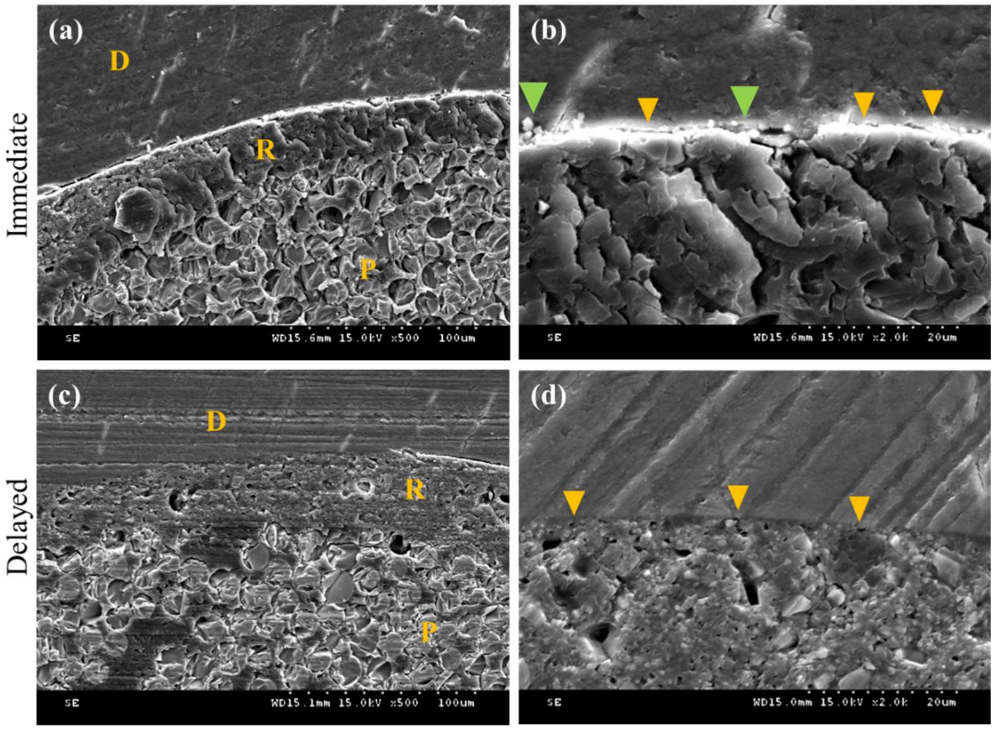

Figure 7. Scanning electron microscopic images of resin cement-adhesive layer-dentin interfaces of AH group. (a) Low magnification image of AH-IM group (×500). The gap is on the margin between resin cement and dentin. (b) High magnification image of AH-IM group (×2000). A slight residual sealer particle remained (green arrow), and a narrow gap of fewer than 1μm was observed between dentin and cement (yellow arrow). (c) Low magnification image of AH-DE group (×500). There was no gap in the margin. (d) High magnification image of AH-DE group (×2000). An adhesive layer penetrated with a dental tube was observed, without a gap in the margin (yellow arrow). D: Dentin, R: Resin cement, P: Fiber post

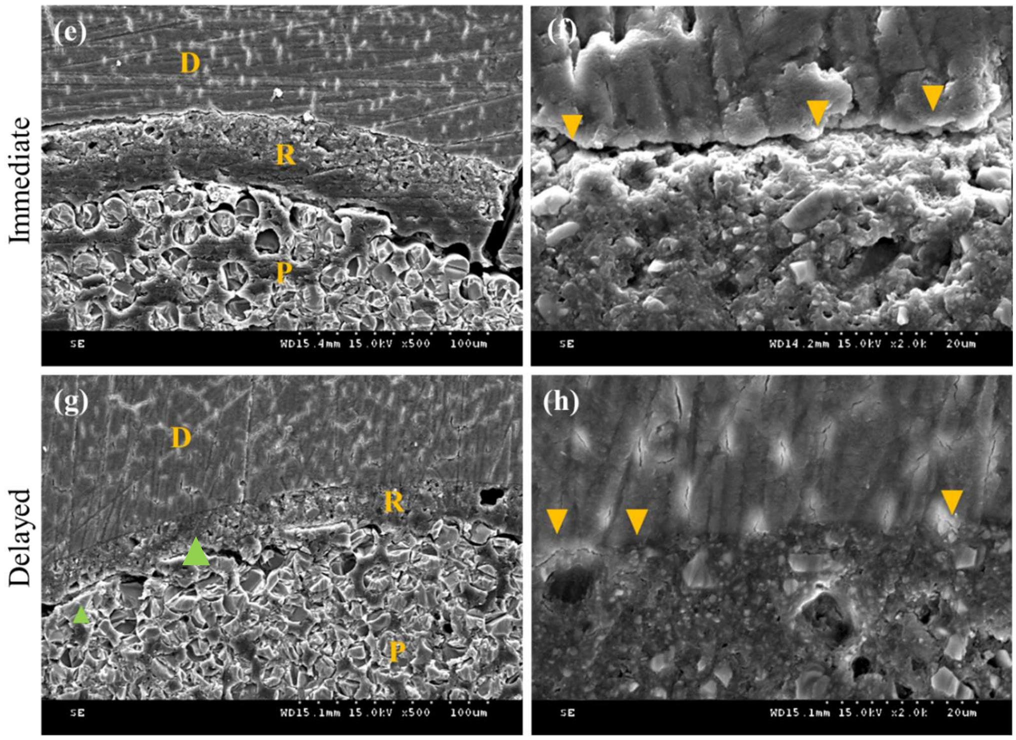

Figure 8. Scanning electron microscopic images of resin cement-adhesive layer-dentin interfaces of CE group. (e) Low magnification image of CE-IM group (×500). There was a gap between dentin and resin cement. (f) High magnification image of the CE-IM group (×2000). The gap was observed (yellow arrow) between dentin and resin cement without the remaining sealer particle. (g) Low magnification image of the CE-DE group (×500). There was almost no gap between dentin and cement, and a slight gap between cement and post was observed (green arrow). (h) High magnification image of CE-DE group (×2000). An adhesive layer penetrated the dentinal tube was observed. Also, very slight gap between dentin and resin cement was observed (yellow arrow). The remaining sealer particle was not observable. D: Dentin, R: Resin cement, P: Fiber post

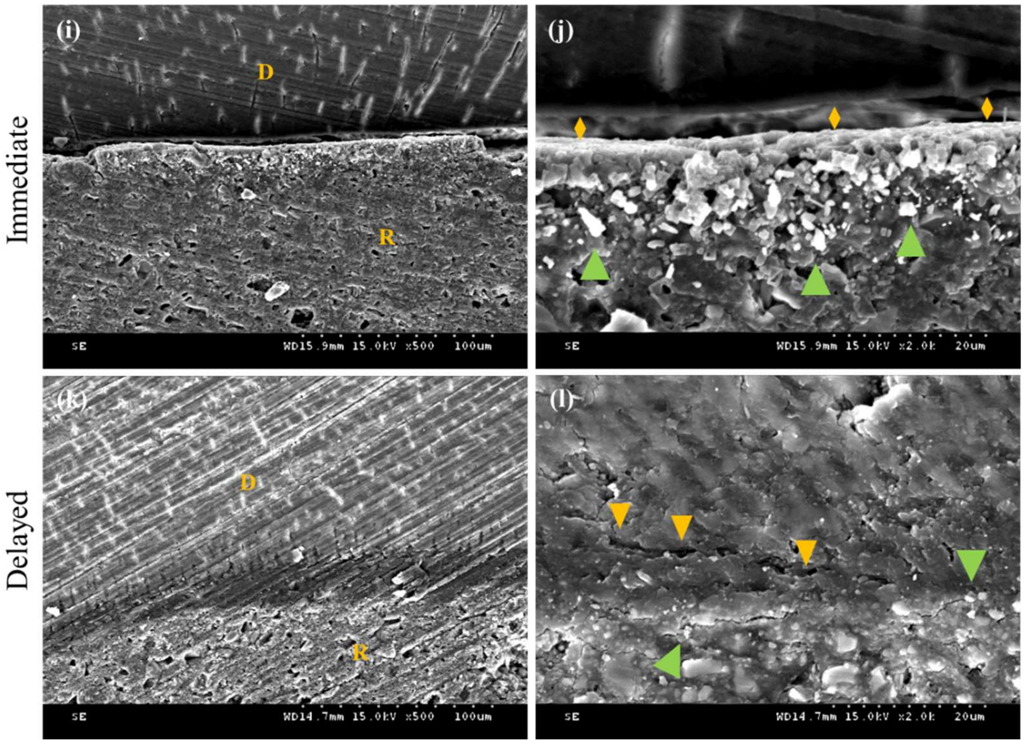

Figure 9. Scanning electron microscopic images of resin cement-adhesive layer-dentin interfaces of EN group. (i) Low magnification image of EN-IM group (×500). A significant gap between dentin and cement was observed. (j) High magnification image of EN-IM group (×2000). Many residual sealer particles were scattered on the surface of the cement (green arrow). A significant gap between dentin and resin cement was observed (yellow arrow). (k) Low magnification image of EN-DE group (×500). The gap between dentin and resin cement was not observed and showed a well-bonded pattern with an adhesive layer. (l) A high magnification photo of the EN-DE group (×2000). A slight gap between dentin and resin cement was observed (yellow arrow). A slight residual sealer particle was shown (green arrow), and an adhesive layer penetrated the dentinal tubule was observed. D: Dentin, R: Resin cement, P: Fiber post

4. Confocal laser scanning microscopy image

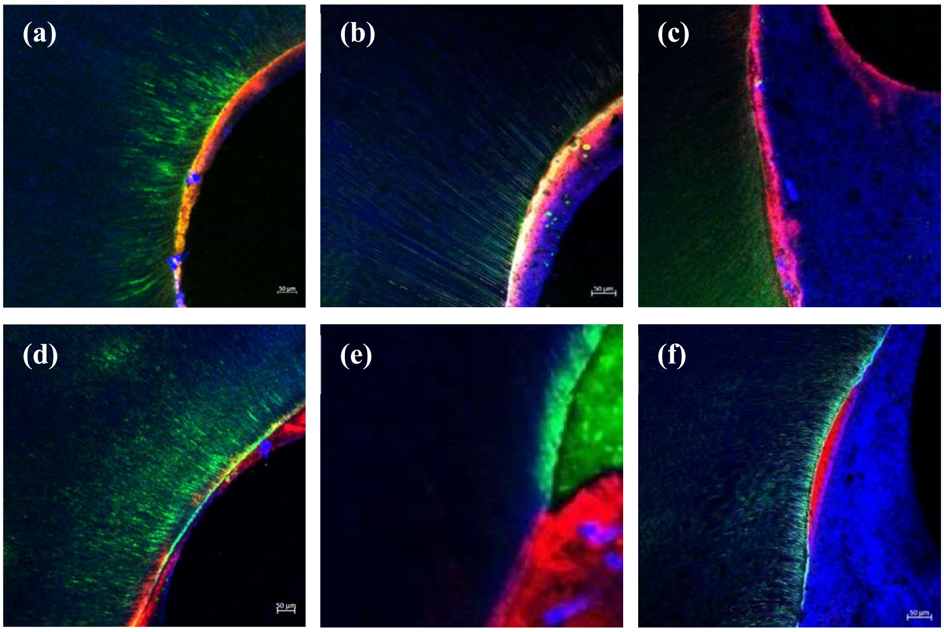

Figure 10. Confocal laser scanning microscopy (LSM 700) images for each group (×20). (a) AH-IM group, (b) CE-IM group, (c) EN-IM group, (d) AH-DE group, (e) CE-DE group, (f) EN-DE group. Green area indicates endodontic sealer, red area indicates adhesive (Single Bond Universal), and blue area indicates resin cement (Rely X U200).

Figure 10 is a cross-section of each sample observed with a confocal laser scanning microscope. The sealer is dyed with fluorescein and appears green, resin cement is dyed with DAPI, blue, and the adhesive is dyed with rhodamine and appears red. It is observed that the sealer and the adhesive penetrated the dental tube. Overall, the length of sealer penetration was longer in AH-plus groups than in calcium silicate-based sealer groups. There was a longer maximum length of sealer penetration in the immediate post cementation (362 μm) than in the delayed post cementation (220 μm) in the AH-plus group. In the calcium silicate-based sealer group, higher density was shown on delayed post cementation (g) than immediate post cementation (b).

IV. Discussion

For the long lifespan of the teeth, it is essential to seal the canal using gutta-percha and sealer to ensure proper canal filling during root canal treatment to prevent infection from the oral cavity and the infection caused by residual bacteria. On the other hand, the post and core should be performed to reinforce the teeth with severe damage to the crown so that the function and esthetic can be restored.

The main factor that affects the survival of fiber post is known as an appropriate bond between fiber post and radicular dentin. The most critical factor for the variation in the bond strength was the type of sealer (Soares et al., 2020; Vilas-Boas et al., 2018). Also, the bonding system of a self-adhesive cement includes micromechanical retention and chemical adhesion to hydroxyapatite. Surface cleanliness is essential to get proper micromechanical retention; the presence of residual endodontic sealer or gutta-percha and the occurrence of droplets may affect the retention (Zicari et al., 2008).

In the present study, the most common type of failure mode was adhesive failure between dentin and resin cement, which is in accordance with previous studies (Dibaji et al., 2017; Özcan et al., 2012). Mixed failure was also shown in the group AH-IM, CE-DE, EN-IM, and EN-DE, which showed relatively higher bond strength than the other groups. However, adhesion failure only between the fiber post and resin cement did not occur. As most of the failure occurred at the interface between resin cement and dentin, it may be expected that bonding between the fiber post and resin cement was adequate, which is in accordance with Vilas-Boas et al. (2018).

Many different results about push-out bond strength of fiber post after using resin-based or calcium silicate-based sealer have been reported. Some articles reported that resin-based sealer showed higher bond strength of fiber post than calcium silicate-based sealer (Dibaji et al., 2017; Mahardhini et al., 2021). However, other articles reported no significant difference between resin-based and calcium silicate-based sealers (Alsubait, 2021; Özcan et al., 2012; Vilas-Boas et al., 2018).

Also, since calcium silicate-based sealer has been newly introduced, there are few studies for appropriate post cementation time after using calcium silicate-based sealer for the root canal filling. Two studies reported no significant difference according to the post cementation time after root canal filling, with slightly higher bond strength for immediate post cementation (Alsubait, 2021; Vilas-Boas et al., 2018). However, Rosa et al. (2013) showed higher bond strength when fiber post was cemented 15 days after canal filling than immediate cementation on bovine teeth.

In the present study, a two-way ANOVA test revealed that the two variables, sealer type and post cementation time, did not individually affect push-out bond strength. However, the interaction between the two variables showed significance ($p=0.037$) which means post cementation time affected the push-out bond strength according to sealer type. AH-plus sealer showed higher bond strength than the other sealers, with a significant difference on the one-way ANOVA test (p=0.006) when posts were cemented immediately after root canal filling. No significant difference was observed when posts were cemented 48 hours after root canal filling. Comparing the different times for the same endodontic sealer, only AH-plus showed a significant difference (p=0.004), whereas Ceraseal and Endoseal did not show differences. Thus, the null hypotheses were partially rejected.

AH-IM group showed significantly higher push-out bond strength than the other groups with Ceraseal and Endoseal and also with delayed post cementation. In the case of AH-plus, the cases with immediate post cementation showed higher bond strength than those delayed post cementation with a significant difference. AH-plus has epoxy resin bisphenol as a component, which might have an affinity for components of Rely X U200, a resin cement that might lead to better bond strength (Cecchin et al., 2011; Vano et al., 2008). Also, epoxy resins in resin-based endodontic sealers did not interfere with the activation of free radicals in composite resins; thus, it may not have adverse effects on resin cement adhesion (Cohen et al., 2002). It can be expected that the unset resin-based sealer was co-polymerized with U200; This finding has shown similar results in previous articles (Mahardhini et al., 2021; Vilas-Boas et al., 2018).

Moreover, in the case of immediate post cementation after root canal filling, AH-plus showed higher bond strength than calcium silicate-based sealer, which is in accordance with Vilas-Boas et al. (2018). This can also be explained by the root canal filling technique as well as the affinity between the resin component of AH-plus and resin cement. In the past study which compared the retrievability between the teeth filled with AH-plus sealer in continuous-wave technique and with Endoseal sealer in sealer-based technique, canal filling material was removed faster and fewer residual materials were in the case of AH-plus sealer with continuous-wave technique than the other cases. This result was due to the higher ratio of gutta-percha to sealer in the continuous-wave technique than sealer-based technique, as gutta-percha is easier to remove than sealer (Kim et al., 2019). Also, the presence of a high amount of residual calcium silicate-based sealer left inside the tubules may interfere with the bonding process (Oltra et al., 2017).

In the case of delayed post cementation after root canal filling, AH-plus showed similar bond strength compared to Ceraseal and Endoseal, which is in accordance with Alsubait (2021) and Vilas-Boas et al. (2018). This result explains that the calcium silicate-based sealer is not inferior to AH-plus in fiber post cementation. Thus, calcium silicate-based sealer can be an alternative to AH-plus in endodontic treatment with fiber post cementation.

Regarding the root canals filled with calcium silicate-based sealer, the results showed that it does not matter whether the post cementation is performed immediately or 48 hours after the root canal filling, which is in accordance with Rosa et al. (2013). However, bond strength was slightly higher in the delayed post cementation group than in the immediate post cementation group. It may be due to the narrower gap distance in the delayed post cementation group.

Preparing a post space without any contamination is ideal and an essential factor for optimal adhesion of resin cement (Davis & O'CONNELL, 2007). It may contribute to gap formation between the adhesion surfaces if it is not well prepared. Even after proper mechanical removal and irrigation protocol, a smear layer produced by post preparation may contain dentin powder, endodontic sealer, resin cement residues, and residual gutta-percha, which may influence wettability, permeability, and reactivity of the dentin (Mahardhini et al., 2021). Thus, the endodontic sealer should be removed thoroughly to achieve an ideal surface condition for adhesion. If it is impossible, the dentinal surface with residual sealer should be in a condition with an affinity to resin cement.

A previous literature review article reported that the mechanical removal of a sealer is an important factor of retreatment in a canal filled especially with calcium silicate-based sealer (Komabayashi et al., 2020). Since calcium silicate-based sealer (BC sealer, Endoseal MTA), with hydrophilic property, showed superior wettability to dentin than AH-Plus (Ha et al., 2018), the more calcium silicate-based sealer remains, the more hydrophilic the dentin surface is, which leads to less wettability and integrity with resin cement and forms a gap in the interface. However, it is challenging to remove the sealer completely. Sealers or gutta-percha remnants may remain even if no debris is attached to the hand instrument during endodontic retreatment (Kim et al., 2019). Some factors may contribute to the mechanical removal of the sealer as follows.

First, calcium silicate-based sealer with unset conditions seemed to make the removal procedure difficult from the root canal. In contrast, the sealer becomes harder after being set, and it could be easier to remove mechanically by using instruments (Kim et al., 2019). Thus, it may be expected that a set calcium silicate-based sealer with higher hardness may be removed better than the unset condition.

Also, the type of canal shape may influence the removal of the sealer. One study reported that the percentage volume of root filling material removal after retreatment was lower in short-term storage after root canal filling than long-term storage in the oval-shaped canal filled with calcium silicate-based sealer (EndoSequence BC) by sealer-based technique (Zhang et al., 2022). The efficiency of retreatment was closely related to the storage time. This result may be attributed to the interplay between the mechanical action of the rotary instrument and fluid dynamics on the hard material after long-term storage. Thus, sufficient mechanical debridement is needed for the root canal with an asymmetry shape.

Single Bond Universal was used in the present study for adhesion of root dentin; however, the manufacturers' instruction did not mention the use of adhesive materials for cementation with Rely X U200. According to Lee et al. (2021), fiber post cementation with Rely X U200 and Single Bond Universal after root canal filling with AH-Plus showed higher push-out bond strength than cementation without adhesive bonding. However, post cementation with Rely X U200 only without adhesive bonding also showed clinically acceptable push-out bond strength. This study reflects the clinical usage of cementation with Rely X U200 combined with Single Bond Universal after using AH-Plus sealer. In addition, a previous in vitro study showed superior shear bond strength between calcium silicate-based material (Biodentine) and composite resin bonded with Single Bond Universal than without using the universal adhesive (Kudva et al., 2022). It is explained by the bifunctional silane molecule in the universal adhesive bonds chemically to silica-containing constituents and has the methacrylate functionality that allows chemical reactions with the adhesive substrate (Deepa et al., 2016).

Meanwhile, the result of the present study is different from the result of Alsubait (2021) and Vilas-Boas et al. (2018) that delayed post cementation after canal filling with calcium silicate-based sealer showed similar or less bond strength. If thorough removal of the endodontic sealer and the ideal surface condition was prepared, the adhesion into the intratubular area may affect the bond strength. A hybrid layer with resin tag formation, a reinforcing factor to the bond strength, may be interfered with by the set sealer in the intratubular area. Calcium silicate-based material form calcium hydroxide when reacted with water, and the high pH causes the phosphate ions in body fluids to precipitate hydroxyapatite at the dentin surface. These studies explained the potential of calcium silicate-based sealer to form tag-like structures consisting of the sealer itself or crystals, suggesting intratubular precipitation, which may be applied to the microscopic condition of the dentinal surface (Han & Okiji, 2013). Thus, the prepared adhesion surface condition and hydroxyapatite precipitation in the intratubular area may be the reason why the present study showed different results from Alsubait (2021) and Vilas-Boas et al. (2018).

Confocal laser scanning microscopy (CLSM) was used to observe the microscopic condition of the remaining materials in the canal space and sealer penetration into dentinal tubules. It is known that cross-sectional observation using a CLSM can effectively measure the degree of sealer penetration (Bitter et al., 2004; Kim et al., 2015). In the present study, the length of sealer penetration was longer in the case of immediate post cementation than in the delayed post cementation in AH-Plus groups. The difference in sealer penetration may be the cause of stronger push-out bond strength in delayed post cementation, even though there was a slightly wider gap between the surface. An affinity between the unset epoxy resin component in AH-Plus sealer and methacrylate resin monomers in resin cement with the deeper sealer penetration into the dentinal tubule can be the facilitating factor for strong bond strength in the case of immediate post cementation in the AH-Plus group. Meanwhile, low density of penetration of sealer into the dentinal tubule was shown in immediate post cementation than delayed post cementation in the calcium silicate-based sealer group. The amount of sealer penetration and the hydrophilic surface condition can both affect the bond strength of calcium silicate-based sealer, considering that there was no significant difference between the two different times. Since the CLSM analysis in this study did not include statistical analysis, further study is needed to quantitatively evaluate the interface between dentin, endodontic sealer, and resin cement.

V. Conclusion

Within the limitation of the present study, epoxy resin-based sealer (AH-Plus) can be a better choice for the root canal filling than calcium silicate-based sealer (Ceraseal, Endoseal) when immediate post cementation is planned. Sufficient setting time is needed for the root canal filling with calcium silicate-based sealer to get better retention of fiber post.

VI. References

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Abstract (In Korean)

파이버 포스트 접착에 대한 칼슘 실리케이트 및 에폭시 레진 계 근관 충전용 실러 간의 비교 연구

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본 연구의 목적은 자가접착형 레진 시멘트를 사용하여 근관 상아질에 시행한 글래스-파이버 포스트 접착에 대하여 근관 충전용 실러의 종류 및 파이버 포스트의 접착 시간이 미치는 영향을 비교하기 위한 연구이다.

66 개의 인간 하악 소구치를 대상으로 물리 화학적인 과정을 거쳐 근관 성형 및 세척을 시행하였다. 이후 근관 충전용 실러의 종류와 파이버 포스트 접착 시간에

따라 무작위하게 6 개의 군으로 다음과 같이 나눴다. (1) AH-Plus 실러를 사용하여 근관 충전한 직후 포스트 접착을 시행한 경우 (AH-IM), (2) AH-Plus 실러를 사용하여 근관 충전한지 48 시간 이후 포스트 접착을 시행한 경우 (AH-DE), (3) Ceraseal 실러를 사용하여 근관 충전한 직후 포스트 접착을 시행한 경우 (CE-IM), (4) Ceraseal 실러를 사용하여 근관 충전한지 48 시간 이후 포스트 접착을 시행한 경우 (CE-DE), (5) Endoseal 실러를 사용하여 근관 충전한 직후 포스트 접착을 시행한 경우 (EN-IM), (6) Endoseal 실러를 사용하여 근관 충전한지 48 시간 이후 포스트 접착을 시행한 경우 (EN-DE). 파이버 포스트 접착을 시행한 이후 2mm 두께의 시편을 형성한 후 push-out bond strength test 를 시행하였다. 이후 파절 양상은 광학현미경 하에서 관찰하였다. 시편의 각 표면은 주사전자현미경을 통해 관찰하였으며 레진 시멘트와 상아질 사이의 계면의 파절 양상을 분석하였다. 각 그룹 별로 2 개의 치아를 추가로 형광 표식을 진행하여 근관치료를 진행한 후, 공초점 레이저 주사 전자현미경을 통해 상아질 내부에서의 실러의 양상에 대해 관찰하였다. 통계학적 유의성은 일원 및 이원 분산 분석, Tukey’s test, independent t-test 로 확인하였으며, 유의 수준은 $p<0.05$ 로 설정했다.

근관 충전 직후 파이버 포스트 접착을 시행한 경우, AH-Plus 실러는 Ceraseal, Endoseal 실러에 비해 비해 높은 결합 강도를 나타냈다. 한편, 근관 충전한지 48 시간 이후 파이버 포스트 접착을 시행한 경우, 근관 충전 실러 종류에

따른 유의한 차이는 나타나지 않았다. 포스트 식립 시기에 따른 유의한 결합 강도 차이는 AH-plus 실러의 경우에서만 나타났다. 가장 많이 관찰된 파절 양상은 상아질과 레진 시멘트 사이의 접착 실패였다 (88%).

전반적으로 즉시 포스트 식립이 일어난 경우에서 지연 식립을 한 경우에 비해 더 넓은 레진 시멘트와 상아질 간 간격이 넓게 나타났다. 각 실러별로 포스트 식립 시기에 따른 레진 시멘트와 상아질 간 간격을 비교한 경우, Ceraseal 의 경우에서만 유의한 차이를 나타냈다. 근관 치료 후 즉시 포스트 식립을 시행한 경우 실러 종류에 따른 차이는 나타나지 않았으며, 지연된 포스트 식립을 시행한 군에서 AH-Plus 와 Endoseal 사이 에서만 유의한 간격의 차이를 나타냈다.

따라서, 근관충전 직후 포스트를 식립하는 경우, 칼슘 실리케이트 실러에 비해 에폭시 레진 실러를 사용하는 경우 더 우수한 파이버 포스트의 유지를 기대할 수 있다. 칼슘 실리케이트 실러를 사용하여 근관 충전을 시행하는 경우, 충분히 경화 시간을 확보한 후 포스트를 식립하는 것이 파이버 포스트의 유지에 도움을 줄 수 있을 것으로 생각된다.

핵심 되는 말 : 근관 충전 실러; 칼슘 실리케이트; 에폭시 레진; 자가접착형 레진 시멘트; 파이버 포스트; 결합 강도; 주사 전자 현미경; 공초점 레이저 주사 현미경