Effect of Phosphoric Acid Etching on Bond Strength of Resin according to the Type of Sealer for Root Canal Filling

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Abstract

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I. Introduction

The use of root canal sealers is indispensable for the obturation of root canals. Epoxy resin-based sealers such as AH Plus (Dentsply Sirona, DeTrey, Konstanz, Germany) are commonly used in endodontic practice, but calcium silicate-based sealers have been widely used in recent years because of their osteoinductive effect, long-term antibacterial effect,

biocompatibility, sealing properties. When the sealer used for root canal filling is not completely removed from dentin, it can interfere with the adhesion of dentin to the resin core restoration, resulting in interfacial microleakage. To avoid this, several methods have been suggested to remove root canal sealers from contaminated dentin. Phosphoric acid (PA) etching is considered useful for hydrophobic sealers such as AH Plus; however, researchers have not studied whether itis also effective for water-soluble calcium silicate-based sealers, which have been commonly used in recent years. This study evaluated the microtensile bond strength of calcium silicate-based sealers and AH Plus depending on the use of phosphoric acid etching before immediate resin restoration of proper filling methods in this situation.

II. Materials and method

Exposed dentin surfaces of extracted human third molars were randomly assigned to 3 groups depending on sealer type (AH Plus [Dentsply Sirona DeTrey], CeraSeal [Meta Biomed Co.], and EndoSeal MTA [Maruchi]). Half of the samples were treated with PA for 30 seconds and cleaned with distilled water for additional 30 seconds, and the other half were cleaned with only distilled water for 30 seconds. Untreated specimens were used as control. Self-etching adhesive (Clearfil SE Bond, Kuraray) was applied, and composite resin (Tetric N-Ceram, Ivoclar Vivadent) was used to resin core build-up. After 24 hours, the microtensile bond strength was measured (EZ-S Test, Shimadzu Co.). The failure mode

was determined by light microscopy and scanning electron microscopy. One-way analysis of variance with the Bonferroni correction was used to analyze the data ($p<0.05$).

III. Result

The bond strength of the water-washed dentin surfaces in the calcium silicate-based sealer groups did not differ significantly from those of the Control group, but the PA-pretreated surfaces exhibited relatively low bond strength. The AH Plus-treated group had lower bond strength than the Control group when no PA treatment was applied, but PA treatment restored the bond strength. The adhesive failure mode was most frequently found in the AH Plus group without PA etching.

IV. Conclusion

When a water-soluble calcium silicate-based sealer is used, sufficient bond strength can be obtained by washing with water alone, with no need for PA use. When a liposoluble AH Plus sealer is used, PA etching is effective to remove AH Plus sealer.

Keywords: Microtensile bond strength; Calcium silicate-based sealer; Immediate bonding; Phosphoric acid pretreatment; Core resin bond strength

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I. INTRODUCTION

Sealer is one of the root canal obturation materials and plays a critical role in sealing the root canal system [1]. Without a sealer, canal obturation exhibit greater leakage. The use of sealer is necessary to fill voids and gaps between the gutta-percha and the root canal walls. Coronal sealing using resin core restoration is critical for the success of endodontically treated teeth [2]. Because coronal seals generated by temporary restorative materials are less predictable, dentin adhesives have been advocated for creating additional orifice barriers and sealing pulp chambers to protect endodontically treated teeth. The amount of coronal microleakage should be considered a potential etiological factor for root canal failure.

Epoxy resin-based sealers such as AH Plus (Dentsply Sirona DeTrey, Konstanz, Germany) are commonly used in endodontic practice [3]. These advantages include antimicrobial action, adhesion to dentin, long working time, ease of mixing, and good sealing ability. However, when the root canal sealer is not completely removed from tooth surface for direct resin core restoration, it can interfere with the adhesion of the resin core restoration, resulting in bond deterioration [4-6]. To avoid this, several methods have been suggested to remove AH Plus from dentin surface. Some researchers recommended to remove the sealer with a dry cotton pellet or 95% ethanol [7-10] until the cavity surface around the orifice looks physically clean. However, this method was not entirely effective. The other paper suggests that it is effective to use an organic solvents such as Endosolv R (Septodont, Saint-Maur-des-Fosses, France) [4]. However, Endosolv R contains the potential inclusion of teratogenic components such as formamide [4-6]. AH Plus cleaner (Dentsply Sirona DeTrey) [11] is also used to remove the remaining sealer. However, this product requires the high cost of processes and the difficulty of storage.

Calcium silicate-based sealers have been widely used in recent years because of their osteoinductive effect, long-term antibacterial effect, biocompatibility, sealing properties, and expansion in the root canal [12-16]. Calcium silicate-based sealers have been reported to have hydrophilic properties and a bioactivity similar to that reported for mineral trioxide aggregate (MTA) type materials. It was initially used as a root-end filling material but is now used in a variety of challenging clinical situations such as pulp capping, pulpotomy, apexogenesis, apical barrier formation in teeth with open apexes, repair of root perforations, and as a root canal filling material. In gutta-percha cones tapered according to the final shape of canals prepared by using different mechanized instrumentation systems, the sealer-based obturation has been used increasingly. The advantages of this technique include easy handling, low cost, and short procedure time.

The obturation technique using calcium silicate-based sealers has become popular because it is less technique-sensitive and easier to use than warm vertical compaction with the epoxy resin-based sealer AH Plus [17]. Calcium silicate-based sealers are water-soluble and appear to differ from the liposoluble AH Plus sealer in their adhesion to dentin for the composite restoration after the root canal obturation. In a previous study, after the removal of a calcium silicate-based sealer with ethanol, no significant difference was found between immediate and delayed resin restoration [18]. In clinical practice, immediate resin core restoration is more time-efficient for the patient than delayed restoration, and it is also advantageous to prevent contamination in the root canal [5,19,20]. The sooner possible the restoration period after root canal treatment is better. This is because complete seal may not be achieved due to wear and loss of temporary restorative materials such as Caviton (GC Corporation, Tokyo, Japan).

The sealer AH Plus has been widely used in clinical practice for many years. However, when utilizing AH Plus, the use of phosphoric acid (PA) etching is also recommended to remove the sealer and facilitate bonding with the core resin. In a study by Rodríguez-Martínez et al., the microtensile bond strength of the AH Plus sealer to dentin was greater with a etch-and-rinse system than with a self-etching system [21]. In the etch-and-rinse system, PA can more effectively remove the hydrophobic AH Plus sealer. That is, PA etching is considered useful for hydrophobic sealers such as AH Plus; however, researchers have not studied whether PA is also effective for water-soluble calcium silicate-based sealers, which have been commonly used in recent years. Recently, due to its ease of use, self-etching system has been more widely used than the etch-and-rinse system as a dental adhesive. To date, research is scarce on immediate adhesion with the self-etching system for calcium silicate-based sealers.

Therefore, this study was conducted to evaluate the effect of the additional acid etching by PA on resin bonding when using a self-etching system with calcium silicate-based sealers as opposed to epoxy resin-based sealers such as AH Plus. The null hypotheses were that (1) the AH Plus sealer and calcium silicate-based sealers would not differ significantly in bond strength when removed with water alone (no PA), (2) the sealers would not differ significantly in bond strength when PA was used.

II. MATERIALS AND METHODS

1. Specimen Preparation

Twenty-one extracted human third molars were used in the study within 3 months after extraction. The teeth used in the experiment were sound and had no cracks or caries. This study was approved by the Institutional Review Board of Yonsei University (IRB No.: 3-2022-0113). The tested materials investigated in the present study are listed in Table 1.

The root portion of the tooth was embedded in MG Crystal Rock (Maruishi, Osaka, Japan), and the crown portion of the tooth was sectioned using a high-speed diamond bur under constant water cooling, exposing the coronal dentin. The flat dentin surface was wet-polished with 600-grit silicon carbide paper to create a standardized substrate for the bonding test. The exposed dentin surfaces of the prepared samples were assigned to 3 groups depending on sealer type, each of which was subdivided into 2 groups based on the additional PA procedure. The untreated specimens were used as the Control group.

Table 1. Tested materials

Brand	Material	Composition	Manufacturer
AH Plus	Epoxy resin -based sealer	Bisphenol epoxy resin, calcium tungstate, zirconium oxide, silica, iron oxide, silicone oil	Dentsply Sirona DeTrey, Konstanz, Germany
CeraSeal	Calcium silicate -based sealer	Calcium silicates, Zirconium oxide, thickening agents	Meta Biomed Co., Cheongju, Korea
EndoSeal MTA	Calcium silicate -based sealer	Calcium silicates, calcium aluminates, calcium aluminoferrite, calcium sulfates, radiopacifier, thickening agents	Maruchi, Wonju, Korea
Etch-37	Phosphoric acid etchant	37% phosphoric acid semi-gel etchant, benzalkonium chloride	Bisco, Schaumburg, IL, USA
Clearfil SE bond	Two-step self-etch dhesive	Primer:10-MDP, HEMA, hydrophilic aliphatic dimethacrylate, photoinitiator, water Bond:10-MDP, Bis-GMA, HEMA, hydrophobic aliphatic dimethacrylate, colloidal silica	Kuraray, Osaka, Japan
Tetric N-Ceram	Composite resin	Monomer matrix: dimethacrylates Filler: barium glass, Ytterbium trifluoride (YbF_3), mixed oxides Filler content: 80%-81% by wt.	Ivoclar Vivadent, Schaan, Liechtenstein

10-MDP, 10-methacryloyloxydecyl dihydrogen phosphate; HEMA, 2-hydroxyethyl methacrylate; Bis-GMA, bisphenol A-glycidyl methacrylate.

(1) Control: The dentin surface was left untreated with any sealer and was rinsed with distilled water for 30 seconds.

(2) AH Plus (AH): The dentin surface was treated with an epoxy resin-based sealer, left undisturbed for 10 minutes, and then rinsed with distilled water for 30 seconds.

(3) AH Plus+PA (AHPA): The dentin surface was treated with an epoxy resin-based sealer, left undisturbed for 10 minutes, and then hand-scrubbed using a dry cotton pellet with 37% phosphoric acid (Etch-37; Bisco, Schaumburg, IL, USA) for 30 seconds. The dentin surface was then rinsed with distilled water for 30 seconds.

(4) CeraSeal (CS): The dentin surface was treated with a calcium silicate-based sealer (CeraSeal), left undisturbed for 10 minutes, and then rinsed with distilled water for 30 seconds.

(5) CeraSeal+PA (CSPA): The dentin surface was treated with a calcium silicate-based sealer (CeraSeal), left undisturbed for 10 minutes, and then hand-scrubbed using a dry cotton pellet with 37% phosphoric acid for 30 seconds. The dentin surface was then rinsed with distilled water for 30 seconds.

(6) EndoSeal MTA (ES): The dentin surface was treated with a calcium silicate-based sealer (EndoSeal MTA), left undisturbed for 10 minutes, and then rinsed with distilled water for 30 seconds.

(7) EndoSeal MTA+PA (ESPA): The dentin surface was treated with a calcium silicate-based sealer (EndoSeal MTA), left undisturbed for 10 minutes, and then hand-scrubbed

using a dry cotton pellet with 37% phosphoric acid for 30 seconds. The dentin surface was then rinsed with distilled water for 30 seconds.

2. Bonding Procedure & Resin Build-Up

The rinsed specimens were dried using an air syringe for 60 seconds, and Clearfil SE Bond (Kuraray, Osaka, Japan) was applied according to the manufacturer's instructions and light-cured for 20 seconds using a light-emitting diode curing unit (Elipar S10; 3M ESPE, St. Paul, MN, USA) at 1200 mW/cm^2. After the bonding procedures, resin composite (Tetric N-Ceram; Ivoclar Vivadent, Schaan, Liechtenstein) were build up with a total thickness of 4.5 mm (3 times by 1.5 mm) and light-cured for 20 seconds for each layer.

3. Microtensile Bond Strength Testing

The prepared samples were stored at 37°C for 24 hours. The samples were sectioned longitudinally into approximately 1-mm-thick slabs, and the slabs were further sectioned into approximately 1 × 1 mm beams using a low-speed cutting machine (RB 205 METSAW LS; R&B, Daejeon, Korea) in Figure 1A. The dimensions of the adhesive surface of each beam were measured using a digital caliper. Each beam was attached with a cyanoacrylate adhesive (Zapit; DVA, Corona, CA, USA) to a microtensile testing jig (Bisco) to measure microtensile bond strength. The microtensile bond strength test was performed using a universal testing machine (EZ Test; Shimadzu Co., Kyoto, Japan) at a 1 mm/min cross-head speed in Figure 1B.

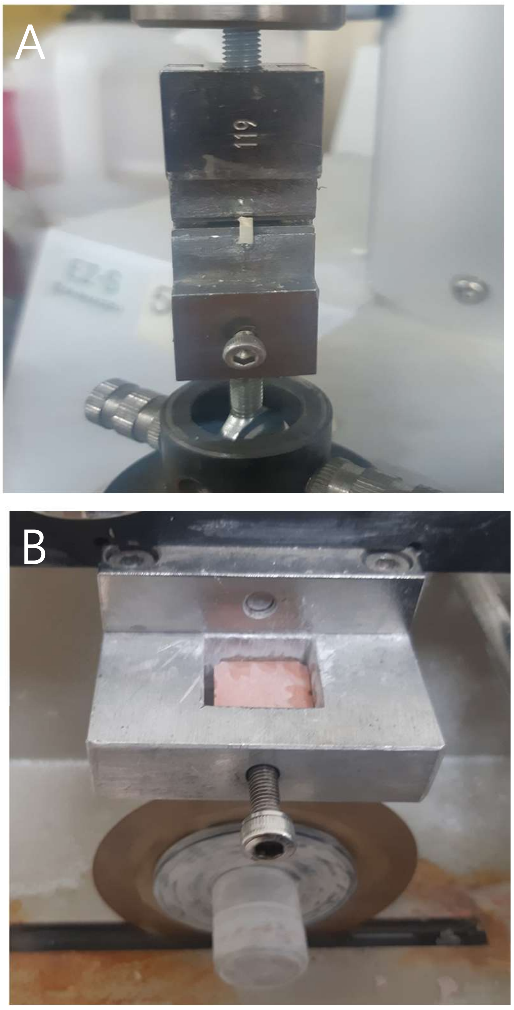

Figure 1. Sample preparations.

(A) The samples were sectioned by diamond saw in a low speed cutting machine

(B) The beam separated by the universal testing machine was shown.

4. Scanning Electron Microscopy (SEM)

After the microtensile bond strength test, the dentin surface of each beam was air-dried. The samples were sputter-coated with gold/palladium. A scanning electron microscope (SEM) (S-3000N; Hitachi, Tokyo, Japan) with an accelerating voltage of 15.0 kV was used to observe all coated samples. The dentin surface of each beam was evaluated using a stereomicroscope to determine the mode of failure and observed using SEM at ×100 and ×1000 magnification.

An additional experiment using SEM was conducted separately to observe the sealer distribution in each group. Before adhesives and the bonding procedure, the dentin surfaces were air-dried. The samples were sputter-coated with gold/palladium at a voltage of 15.0 kV. The dentin surface of each sample was examined under SEM at ×2000 magnification.

5. Statistical Analysis

Microtensile bond strength analyses were performed using data derived from the 3 groups categorized by sealer type (AH, CS, ES), the 2 subgroups for each (categorized by using PA), and the Control group. For the total of 7 groups (Control, AH, AHPA, CS, CSPA, ES, and ESPA), the normality and homoscedasticity assumptions were assessed using the Shapiro-Wilk test and QQ plots. If those assumptions were satisfied, the data sets were analyzed using one-way analysis of variance. If the assumptions were violated, the data were nonlinearly transformed to satisfy those assumptions prior to the use of the aforementioned parametric statistical procedures. In assessing whether normality was satisfied in each group, normality was found to be satisfied overall, so one-way analysis of variance (a parametric method) was utilized. Statistical analyses were performed using SAS software version 9.4 (SAS Institute Inc., Cary, NC, USA). *P*-values of less than 0.05 were considered to indicate statistical significance.

III. RESULTS

1. Microtensile Bond Strength

Pre-test failure rates were compared between each group using Fisher's exact test in Table 2. Because the p-value was greater than the significance level of 0.05, there was no statistically significant difference for each group. Table 3 and Figure 2 show the mean and standard deviation values of microtensile bond strength for each group except pre-test failure. CS and ES groups, the calcium silicate-based sealers, showed no significant difference from the Control group when they were washed with water alone. However, the CSPA and ESPA groups, in which the calcium silicate-based sealers were removed using PA, exhibited significantly lower microtensile bond strength than the Control group (marked +). In comparison, AH group had a significantly lower microtensile bond strength than the Control group when washed with only water (marked +). However, the AHPA group, in which AH Plus was removed using PA, the microtensile bond strength was greater and did not differ significantly from the Control group.

In addition, any group with a significant difference relative to AH group is marked with an O. Control, AHPA, CS, CSPA, ES, and ESPA groups had significantly greater microtensile bond strength than AH group (marked O). It meaned that all groups other than the reference AH group had significantly greater microtensile bond strength than AH group.

In addition, a # notation denotes groups treated with the same sealer that differed significantly in microtensile bond strength according to using PA. The AHPA group (in which PA was applied to the AH sealer) had significantly greater microtensile bond strength (marked #) than the AH group. However, the CSPA group, in which PA was applied to the CS sealer, exhibited slightly lower microtensile bond strength than the CS group, albeit not to a significant extent. The ESPA group, in which PA was applied to the ES sealer, exhibited slightly lower microtensile bond strength than the ES group (marked #), which constituted a significant difference.

Table 2. Pre-test failure using Fisher's exact test for the seven groups

Group	No = Success (%)	Yes = Failure (%)	*P*-value
Control	25(100)	0(0)	> 0.05
AH	20(80)	5(20)	
CS	23(92)	2(8)	
ES	24(96)	1(4)	
AHPA	22(88)	3(12)	
CSPA	24(96)	1(4)	
ESPA	23(92)	2(8)	

Table 3. Mean and standard deviation values of microtensile bond strength for the seven groups

Water (No PA)		**PA**		***P*-value**
Control	26.514 ± 6.835^{O}			
AH	$14.348 \pm 3.570^{+}$	**AHPA**	$22.208 \pm 6.675^{O\#}$	< 0.05
CS	23.239 ± 5.052^{O}	**CSPA**	$19.785 \pm 4.694^{+O}$	
ES	24.862 ± 5.378^{O}	**ESPA**	$20.024 \pm 6.286^{+O\#}$	

$^{+}$Significant difference compared to the Control group.

^{O}Significant difference compared to the AH group.

$^{\#}$Significant difference between water and PA subgroups among groups treated with the same sealer.

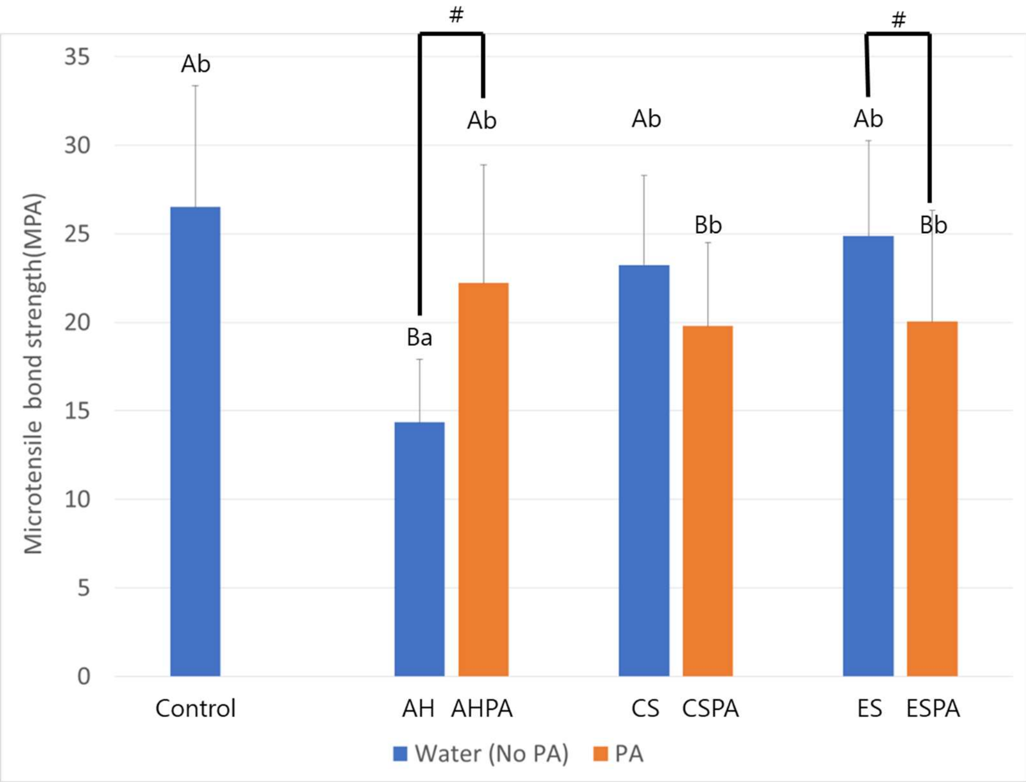

Figure 2. Microtensile bond strength of dentin to resin composite using different sealers with or without phosphoric acid (PA) etching. Groups with different upper case letter B on top of the data bars are significantly different data ($p < 0.05$) compared with Control group with upper case letter A on the top of the data bars. Groups with different lower case letter b on the top of the data bars are significantly different data ($p < 0.05$) compared with AH group with upper case letter a on the top of the data bars. # means significant difference between water and PA subgroups among groups treated with the same sealer.

2. SEM Analysis of Sealer-Contaminated Dentin With or Without PA

SEM images of sealer-contaminated dentin surfaces with or without PA are shown in Figure 3. When only AH Plus was used, the dentin surface was covered with sealer. Filler particles in the AH group were highly granular and prominent, while those in the CS and ES groups were more refined and smaller. Treatment of the AH Plus-contaminated dentin surface with PA reduced the sealer, and the dentinal tubule orifices were opened. Ceraseal was water-soluble and not visible with the naked eye, but small particles were observed to be spread over the surface on the SEM image. Treatment of the Ceraseal-contaminated dentin surface with PA reduced the sealer, and the opend dentinal tubules were observed. Finally, Endoseal was also water-soluble and not visible with the naked eye, although most dentinal tubule orifices were filled with Endoseal. Treatment of the Endoseal-contaminated dentin surface with PA reduced the visible sealer, and the dentinal tubules were opened.

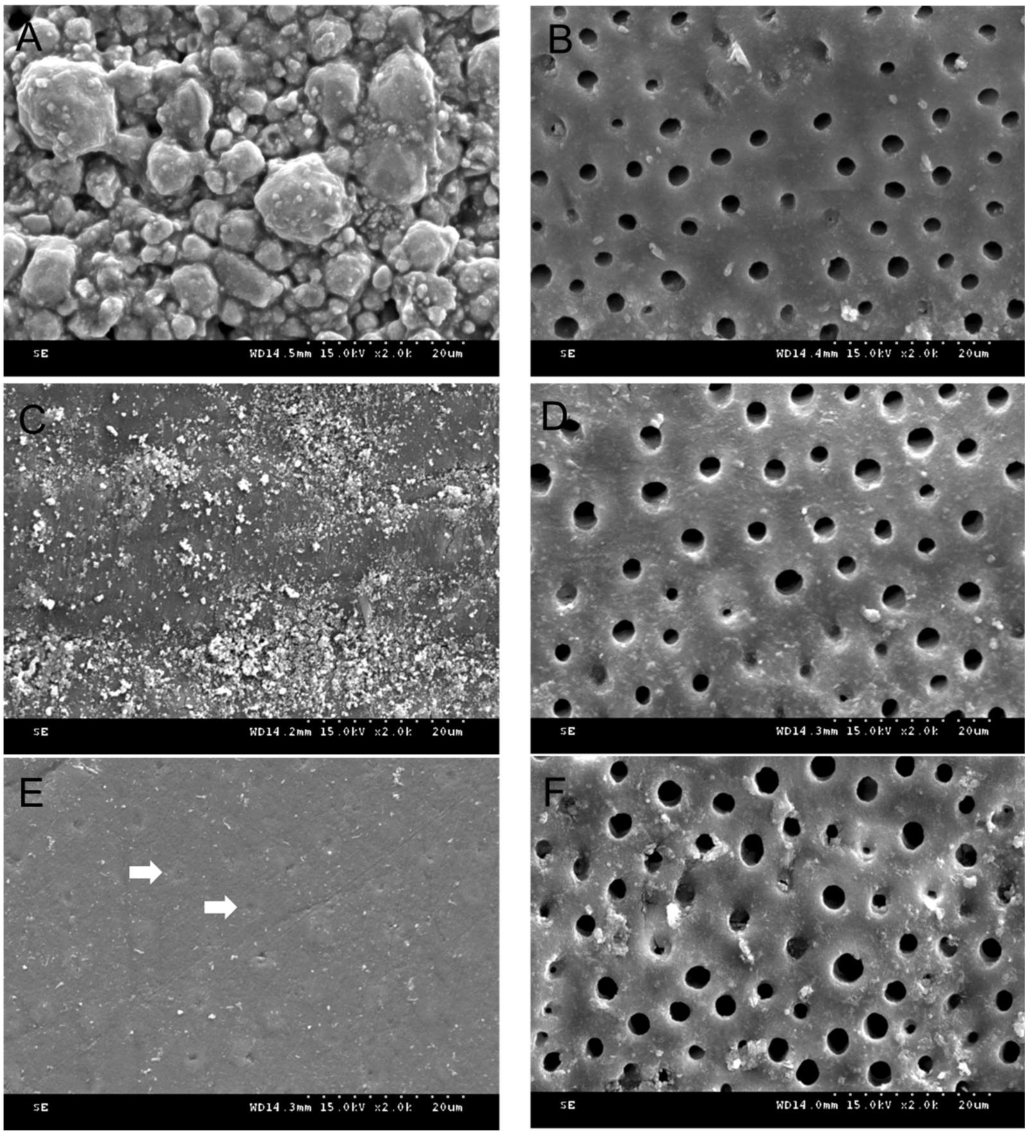

Figure 3. Representative scanning electron microscopy (SEM) images of the pulpal floor dentin of each group (×2000).

(A) Representative scanning electron microscopy (SEM) image of the pulpal floor dentin after AH Plus without PA. Filler particles in the AH group were highly granular and relatively prominent. (B) AH Plus with PA. PA etching of the AH Plus-contaminated dentin surface reduced the visible sealer, and the dentinal tubules were opened. (C) Ceraseal without PA. Filler particles in the CS group were more refined and smaller than AH Plus particles. (D) Ceraseal with PA. PA etching of the Ceraseal-contaminated dentin surface reduced the visible sealer, and the dentinal tubules were opened. (E) Endoseal without PA. Filler particles in the ES group were more refined and smaller than AH Plus particles. Most dentinal tubule orifices were filled with Endoseal (white arrows). (F) Endoseal with PA. PA etching of the Endoseal-contaminated dentin surface reduced the visible sealer, and the dentinal tubules were opened.

3. Failure mode

Figure 4 shows the ratios of failure modes in each group. Overall, the most common failure mode in all groups was adhesive failure. The AH group, in which the microtensile bond strength was the lowest, showed the highest rate of adhesive failure. The ratios of failure modes across the remaining groups (Control, AHPA, CS, CSPA, ES, and ESPA) were similar to each other. SEM images of the failure modes are shown in Figure 5.

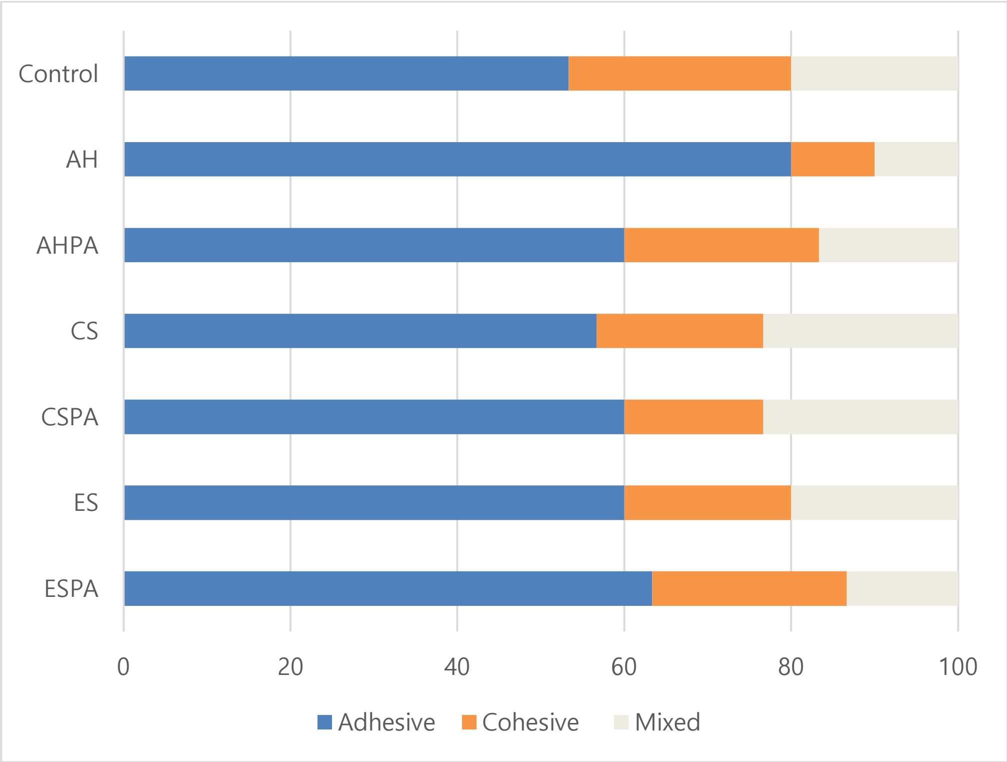

Figure 4. All groups except the AH group (Control, AHPA, CS, CSPA, ES, and ESPA) had similar ratios of failure modes. AH group showed higher adhesive failure than other groups.

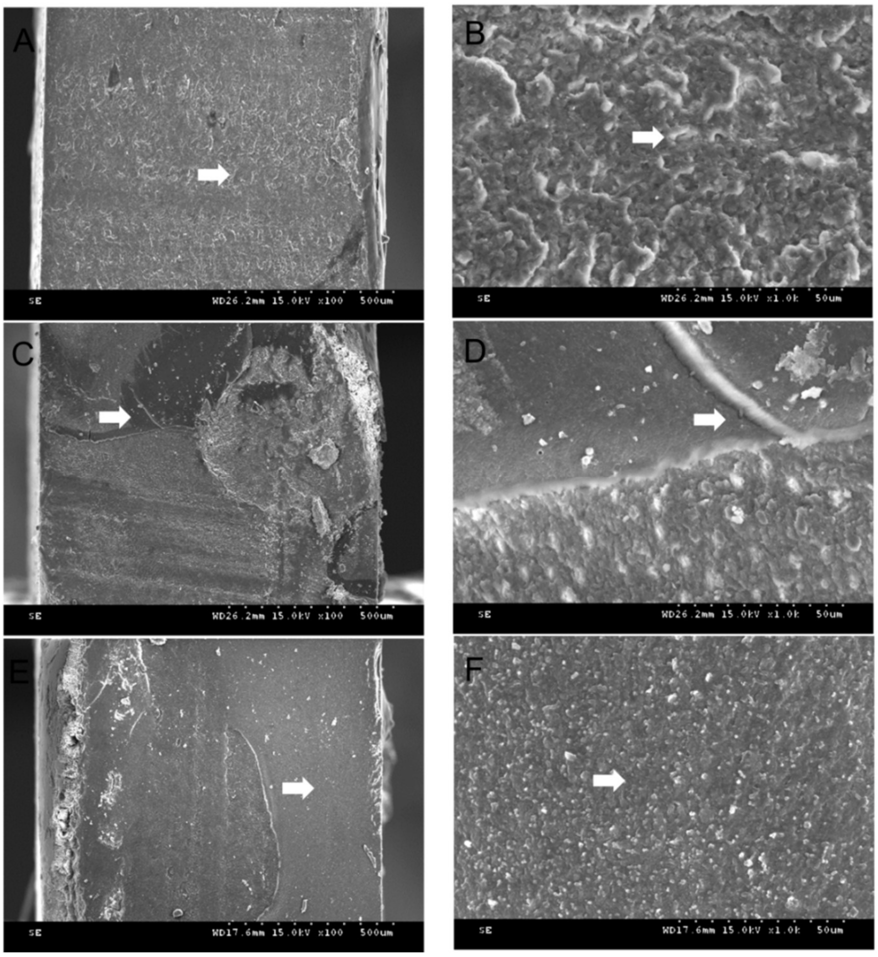

Figure 5. Representative scanning electron microscopy images of fractured surfaces. Areas corresponding to the same location are marked with white arrows.

(A) Adhesive failure at ×100 magnification.

(B) Adhesive failure at ×1000 magnification. (C) Mixed failure at ×100 magnification.

(D) Mixed failure at ×1000 magnification. (E) Cohesive failure at ×100 magnification.

(F) Cohesive failure at ×1000 magnification.

V. DISCUSSION

According to the results of this experiment, the null hypothesis (1) the two types of sealers would not differ significantly in bond strength when removed with water alone (no PA) was rejected. In this study, when a calcium silicate-based sealer was used (CS, ES groups), the microtensile bond strength did not differ significantly from that of the Control group, even when removal was performed with water alone without using PA. In contrast, the bond strength was significantly lower when AH Plus was used (AH group) than in the Control group. In a previous study, when using etch-and-rinse system after using calcium silicate-based sealer, it was shown that using ethanol was not affected by the time of core restoration and even when washed only with water alone, sufficient bond strength was recovered [18].

In this experiment, the experiment was performed using the self-etch system, which is much more convenient and more widely used than the etch-and-rinse system currently, after using calcium silicate-based sealer. Our results indicated that when a calcium silicate-based sealer was used, sufficient bond strength could be obtained by washing with water alone (no PA) in self-etching system. No additional use of PA was necessary to remove the calcium silicate-based sealer. This is thought to be the result of the calcium-silicate based sealer having a hydrophilic property, which enables it to be easily washed away by water. However, when AH Plus was used, sufficient bond strength could not be obtained by washing with water alone in self-etching system. This is thought to be the result of the AH Plus sealer having a liposoluble property, which is difficult to remove completely using water alone.

According to the results of this experiment, the null hypothesis (2) the sealers would not differ significantly in bond strength when removed with PA was rejected. In this study, when a calcium silicate-based sealer was used and removed with PA (CSPA, ESPA groups), the microtensile bond strength was decreased significantly from that of the Control group.

When AH Plus was used and removed with PA, the microtensile bond strength did not differ significantly from that of the Control group. That is, the microtensile bond strength of AHPA group was significantly higher than that of CSPA and ESPA groups when reference group is control group.

In addition, the bond strength would be affected by the use of PA for the calcium silicate-based sealer. This may be because washing with water alone was sufficient to remove the water-soluble calcium silicate-based sealer to make partially demineralized hybrid layer when applied the self-etching system. However, PA removed too much mineral content from the dentin surface, resulting in the absence of Ca ions that could react with MDP, resulting in a decrease in bond strength.

That is, when a water-soluble calcium silicate-based sealer was used, washing with water alone was sufficient to remove the calcium silicate-based sealer and additional PA excessively demineralized the dentin and exposed the hydroxyapatite-containing collagen matrix. The rubbing action with a cotton pellet, along with acid etching on the calcium silicate-based sealer-contaminated dentin surface, could destroy collagen fibers and hydroxyapatite crystals. Hashimoto et al. noted that excessive acid conditioning caused deeper demineralization of both intertubular and peritubular dentin; thus, those two kinds of dentin could not be entirely infiltrated by resin monomers, leading to decreased bond strength [22].

In other words, using PA with a water-soluble calcium silicate sealer decreases the bond strength. It can be surmised that the smear layer is completely removed by PA and the exposed dentin becomes fully, rather than partially, demineralized; therefore, using the self-etching system results in significantly reduced bond strength.

In summary, excessive acid etching in self-etching systems can have negative consequences on bond strength [23]. However, when using epoxy resin-based sealer, AH Plus, PA was found to be more effective and resulted in about 36% decrease in bond strength when PA was not used. In addition, AH group had lower microntensile bond strength significantly compared with other groups in Table 3.

The low bond strength for the AH group can be explained by its hydrophobic nature, meaning that the self-etch system alone did not sufficiently remove and demineralize the dentin for optical bonding. This could be confirmed when observing the fracture surface. In Figure 4, the other groups showed adhesive failure around 50-60%, whereas the AH group showed 80%, which showed a lower bond strength and is consistent with the failure mode observed in the AH group. Figure 5 showed adhesive, mixed and cohesive failure pattern and other groups except AH group had similar ratio of adhesive, mixed and cohesive failure pattern.

Our discussion on Figure 3 was related to the previous content in null hypothesis (1). Calcium-silicate based sealer had a hydrophilic property, which enables it to be easily washed away by water. although Figure 3C of CS group and Figure 3E of ES group have different patterns of remaining sealer, it can be observed that most of the sealer has been removed compared to Figure 3A of AH group. In the case of CS group, small particles showed a general spread pattern, whereas, with ES group, the dentinal tubule orifice was blocked rather than displaying small particles, which may be due to minute differences in components between the sealers. However, these differences did not seem to affect microtensile bond strength because neither CS nor ES groups differed significantly in microtensile bond strength from the Control group. That is, washing with water alone was sufficient to remove the water-soluble calcium silicate-based sealer. Water alone without using PA may be difficult to completely remove the hydrophobic AH Plus in Figure 3A. Therefore, when PA was not used, the exhibited AH Plus surfaces significantly lower microtensile bond strength values. Therefore, AH group had lower microntensile bond strength significantly in Table 3 and showed high adhesive failure pattern compared with other groups.

Our discussion on Figure 3 was also related to the previous content in null hypothesis (2). AHPA, CSPA and ESPA has similar pattern with the dentinal tubules open in Figure

3B, 3D, and 3F. However, strictly speaking, these were SEM images before applying the self-adhesive etching system. Because the self-adhesive etching system was applied after AH Plus was completely removed due to the additional PA process, the AHPA group had microtensile bond strength with no significant difference from the Control group. The CSPA group and ESPA group had lower microtensile bond strength significantly than the Control group, because the self-etching system was applied after dentin was fully demineralized due to the additional PA process.

In addition, AH vs AHPA, CS vs CSPA, and ES vs ESPA groups were compared, excluding the Control group, as a comparison between horizontal items in Table 3. Interestingly, when the dentin surface was treated with PA and water rinse, both calcium silicate-based sealers showed a decrease in tensile bond strength, and a statistically significant difference was found in the ES group. The dentinal tubule orifice was blocked by Endoseal sealer in Figure 3E of ES group, but it did not have a significant effect on the microtensile bond strength as the dentin might be partially demineralized normally after applying the self-etching system. ESPA group had lower microtensile bond strength by additional PA. Hence, statistically significant difference was found in the ES group. The small particles of Ceraseal sealer showed a general spread pattern in Figure 3C of CS group, so remaining small particles had a small but negative effect on the self-etching system. Additional PA in Figure 3D of CSPA group also had negative effect on the self-etching system. Hence, the CS group had microtensile bond strength with no significant difference from the CSPA group.

However, when looking at the CS and ES groups overall, the sealer was sufficiently removed by washing with water. Additional use of PA resulted in excessive opening of the dentinal tubules and a decrease in bond strength. In other words, since calcium silicate-based sealers are water-soluble, sufficient bond strength can be obtained by washing with water alone.

In contrast, The SEM pattern of the AH group in Figure 3A shows that AH Plus was not sufficiently removed by washing with water and instead remained in the dentin (6). However, after treatment with PA, the AH Plus was no longer present and the dentinal tubules were opened, indicating that PA can remove AH Plus in Figure 3B.

In summary, the additional use of PA can be useful for removing AH, but it can negatively impact the bond strength of the self-etching system for calcium silicate-based sealers.

The present in vitro study has some limitations. Although a variety of experimental groups were analyzed, the number of samples in each group was small. Furthermore, no experimental group was subjected to PA etching alone, so insufficient evidence was generated regarding whether the acid etching procedure actually reduced the microtensile bond strength in the calcium silicate groups. In addition, the action of rubbing with a cotton pellet in the acid etching procedure has been claimed to be technique-sensitive.

V. CONCLUSION

1. When a water-soluble calcium silicate-based sealer is used, sufficient bond strength can be obtained by washing with water alone, even without additional use of PA to remove the sealer.

2. When a liposoluble AH Plus sealer is used, additional use of PA is effective to remove AH Plus sealer.

References

1. Camilleri J. Sealers and warm gutta-percha obturation techniques. J Endod 2015;41:72-8.

2. Swanson K, Madison S. An evaluation of coronal microleakage in endodontically treated teeth. Part I. Time periods. J Endod 1987;13:56-9.

3. Heling I, Gorfil C, Slutzky H, Kopolovic K, Zalkind M, Slutzky-Goldberg I. Endodontic failure caused by inadequate restorative procedures: review and treatment recommendations. J Prosthet Dent 2002;87:674-8.

4. Roberts S, Kim JR, Gu LS, Kim YK, Mitchell QM, Pashley DH, et al. The efficacy of different sealer removal protocols on bonding of self-etching adhesives to AH plus-contaminated dentin. J Endod 2009;35:563-7.

5. Galvan RR, Jr., West LA, Liewehr FR, Pashley DH. Coronal microleakage of five materials used to create an intracoronal seal in endodontically treated teeth. J Endod 2002;28:59-61.

6. Tian F, Jett K, Flaugher R, Arora S, Bergeron B, Shen Y, et al. Effects of dentine surface cleaning on bonding of a self-etch adhesive to root canal sealer-contaminated dentine. J Dent 2021;112:103766.

7. Gonçalves Galoza MO, Fagundes Jordão-Basso KC, Escalante-Otárola WG, Victorino KR, Rached Dantas AA, Kuga MC. Effect of cleaning protocols on bond strength of etch-and-rinse adhesive system to dentin. J Conserv Dent 2018;21:602-6.

8. Devroey S, Calberson F, Meire M. The efficacy of different cleaning protocols for the sealer-contaminated access cavity. Clin Oral Investig 2020;24:4101-7.

9. Topçuoğlu HS, Demirbuga S, Pala K, Cayabatmaz M, Topçuoğlu G. The bond strength of adhesive resins to AH plus contaminated dentin cleaned by various gutta-percha solvents. Scanning 2015;37:138-44.

10. Bronzato JD, Cecchin D, Miyagaki DC, de Almeida JF, Ferraz CC. Effect of cleaning methods on bond strength of self-etching adhesive to dentin. J Conserv Dent 2016;19:26-30.

11. Peters OA, Teo MRX, Ooi JM, Foo ASW, Teoh YY, Moule AJ. The effect of different sealer removal protocols on the bond strength of AH plus-contaminated dentine to a bulk-fill composite. Aust Endod J 2020;46:5-10.

12. Du T, Wang Z, Shen Y, Ma J, Cao Y, Haapasalo M. Combined Antibacterial Effect of Sodium Hypochlorite and Root Canal Sealers against Enterococcus faecalis Biofilms in Dentin Canals. J Endod 2015;41:1294-8.

13. Zhang H, Shen Y, Ruse ND, Haapasalo M. Antibacterial activity of endodontic sealers by modified direct contact test against Enterococcus faecalis. J Endod 2009;35:1051-5.

14. Zhou HM, Du TF, Shen Y, Wang ZJ, Zheng YF, Haapasalo M. In vitro cytotoxicity of calcium silicate-containing endodontic sealers. J Endod 2015;41:56-61.

15. Zoufan K, Jiang J, Komabayashi T, Wang YH, Safavi KE, Zhu Q. Cytotoxicity evaluation of Gutta Flow and Endo Sequence BC sealers. Oral Surg Oral Med Oral Pathol Oral Radiol Endod 2011;112:657-61.

16. Zhou HM, Shen Y, Zheng W, Li L, Zheng YF, Haapasalo M. Physical properties of 5 root canal sealers. J Endod 2013;39:1281-6.

17. Kim JH, Cho SY, Choi Y, Kim DH, Shin SJ, Jung IY. Clinical Efficacy of Sealer-based Obturation Using Calcium Silicate Sealers: A Randomized Clinical Trial. J Endod 2022;48:144-51.

18. Morais JMP, Victorino KR, Escalante-Otárola WG, Jordão-Basso KCF, Palma-Dibb RG, Kuga MC. Effect of the calcium silicate-based sealer removal protocols and time-point of acid etching on the dentin adhesive interface. Microsc Res Tech 2018;81:914-20.

19. Belli S, Zhang Y, Pereira PN, Ozer F, Pashley DH. Regional bond strengths of adhesive resins to pulp chamber dentin. J Endod 2001;27:527-32.

20. Belli S, Zhang Y, Pereira PN, Pashley DH. Adhesive sealing of the pulp chamber. J Endod 2001;27:521-6.

21. Rodríguez-Martínez JB, González-Rodríguez MP, González-López S, Ferrer-Luque CM. Influence of adhesive systems on microtensile bond strength of resin-based endodontic sealers to the root dentin. J Clin Exp Dent 2014;6:e203-8.

22. Hashimoto M, Ohno H, Kaga M, Sano H, Tay FR, Oguchi H, et al. Over-etching effects on micro-tensile bond strength and failure patterns for two dentin bonding systems. J Dent 2002;30:99-105.

23. Van Landuyt KL, Kanumilli P, De Munck J, Peumans M, Lambrechts P, Van Meerbeek B. Bond strength of a mild self-etch adhesive with and without prior acid-etching. J Dent 2006;34:77-85.

국문 요약

근관충전용 실러 종류에 따른 인산 산부식이 레진의 결합강도에 미치는 영향

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I. 서론

근관 치료시 실러를 사용하는 것은 필수적이다. 통상적으로 AH Plus 실러를 사용하였지만 최근에는 골유도 효과, 장기간의 항균 효과, 생체 적합성, 밀폐성이 우수한 Calcium silicate-based 실러를 많이 사용하고 있다. 근관 충전에 사용되는 실러가 묻은 상아질에서 이 실러를 완전히 제거되지 않으면 레진 코어 수복에서 상아질의 접착을 방해하여 미세누출이 발생할 수 있다. 이를 방지하기 위해 AH Plus 가 묻은 상아질에서 AH Plus 를 제거하는 여러 가지 연구가 제안되었다. 그 중 인산 에칭은 AH Plus 와 같은 지용성 실러에 유용한 것으로

간주되었다. 그러나 최근 많이 사용되는 Calcium silicate-based 실러에도 인산 에칭이 효과적인지 여부에 대해서는 연구가 부족하였다. 따라서 이 연구의 목적은 즉각 레진 수복을 하기 전 근관충전용 실러인 Calcium silicate-based 실러와 AH Plus를 사용하였을 때 이를 제거하기 위한 phosphoric acid 유무에 따른 접착 강도를 비교 하기 위함이다.

II. 재료 및 방법

제 3대구치의 상아질을 편평하게 노출시켜 AH Plus, Ceraseal, Endoseal, 3 가지 그룹으로 나누었다. 각 그룹에서 sample의 절반은 phosphoric acid로 30초간 실러를 세척하고 추가로 물로 30초간 추가 세척하였고, 다른 절반은 물로만 30초간 세척하였다. 실러가 묻지 않고 물로만 30초간 세척한 그룹을 대조군으로 설정하였다. 이후 Clearfil SE Bond로 self-etching system을 적용한 후 composite resin으로 충전하였다. 24시간후 미세접착강도를 측정하였다. Failure mode는 광학 현미경과 주사 전자 현미경으로 결정되었다. Bonferroni 보정을 사용한 One-way anova로 데이터를 분석하였다. ($p < 0.05$)

III. 결과

칼슘 실리케이트 실러를 물만으로 세척한 그룹과 대조군간의 미세 접착강도는 유의차가 없었으나, 오히려 phosphoric acid로 세척한 그룹이 상대적으로 낮은 미세 접착 강도를 보였다. AH Plus 실러를 Phosphoric acid 없이 물만으로 세척한 그룹은 가장 낮은 미세 접착 강도를 보였지만, phosphoric acid로 세척하니 미세 접착 강도가 회복되는 양상을 보였다. AH Plus 실러를 Phosphoric acid 없이 물만으로 세척한 그룹에서 adhesive failure mode가 가장 흔하게 관찰되었다.

IV. 결론

수용성인 칼슘 실리케이트 실러를 사용시에는 추가적인 phosphoric acid처리 과정 없이 물만으로도 충분한 미세 접착강도를 얻을 수 있다. 반면에, 지용성인 AH Plus를 사용시에는 추가적인 phosphoric acid가 AH Plus제거하는데 효과적이다.

핵심되는 말: 미세 접착 강도, 칼슘 실리케이트 기반 실러, 인산 처치, 코어 레진 접착 강도