Effects of calcium hydroxide reagent on the bond strength of resin cements to root dentin and the retention force of FRC posts

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The aim of this study was to determine the effects of calcium hydroxide (Ca(OH)₂) treatment on bond strength of resin cements to root dentin and retention force of fiber-reinforced composite (FRC) posts. Bovine root dentin was endodontically prepared and treated with Ca(OH)₂ for 7 days. Root dentin for bond strength test was adhered to resin-composite with resin cements. For pull-out test, posts consisting of FRC posts and resin-composites were fabricated and cemented to root. Shear bond and pull-out tests were performed using a universal testing machine. No significant differences in bond strength and post retention force were found between Ca(OH)₂ treated and untreated groups. Significant differences were found among the cements. A positive correlation was indicated between bond strength of cements and retention force of FRC posts. In conclusion, Ca(OH)₂ treatment on root dentin did not affect bond strength of resin cements and retention force of FRC posts.

Keywords: FRC post, Adhesive resin cement, Calcium hydroxide, Bond strength, Post retention

INTRODUCTION

Posts and cores are used as filling materials on endodontically treated tooth to ensure adequate retention of the final crown restoration. Metals have been conventionally used as post material due to their exceptional strength, however the probability of tooth fracture increases from stress generated between the post and dentin from the large difference in elastic modulus3.

Fiber reinforced composite resin (FRC) posts possess exceptional aesthetic properties and an elastic modulus which closely matches dentin which reduces the risk of severe tooth fractures compared to metal posts2,3. However inadequate adhesion between root dentin and posts have caused clinical complications such as post debonding4-6; therefore it is important to have sufficient adhesion between root dentin and post material for adequate post retention. Studies on the adhesive or retention force between dentin, post, and core materials have been reported7-8; however very few have investigated the relationship between bond strength of root dentin to resin composite and retention force of FRC posts.

Techniques for core build-up on endodontically treated teeth with FRC posts include the following: the direct method, where a bonding agent is used to directly build-up the core with a fiber post and resin composite, the indirect method, where the FRC post is fabricated outside the oral cavity and bonded later using adhesive resin cement, and the indirect-direct method, where the FRC post is fabricated in the patients mouth and bonded later. Higher retention force of posts and fracture resistance of teeth have been reported when adhesive resin cements were used, compared to non-resin cements, to bond posts to root dentin9. Post debonding often occurs interfacially between root dentin and adhesive resin cement however not as common between FRC post and resin composite10,11. Therefore it is important to determine any substances which may affect the bond strength and retention force at the interface between dentin and resin cements when FRC post are bonded. Root canal treatment is a complicated procedure where remains of root canal reagents can affect the adhesive system during post and core build-up. For example, sodium hypochlorite and hydrogen peroxide, used for root canal irrigation, are known to decrease bond strength of adhesive resin cements12,13. Calcium hydroxide reagents, which have been shown to be challenging to completely remove from root canals, are also commonly used on root canals as antibacterial medication14-17. Calcium hydroxide remains after endodontic treatment could possibly affect the bond strength of dentin to resin cements; however this subject has not yet been studied much.

The purpose of this study was to determine the effects of calcium hydroxide reagent on the bond strength of resin cement to root dentin and the retention force of FRC posts. In addition, the relationship between bond strength of resin cement to root dentin and retention force of FRC posts was also discussed. The null hypothesis was that the application of calcium hydroxide reagent on root dentin decreases both the bond strength of resin cements and retention force of FRC posts.

MATERIALS AND METHODS

Preparation of bovine tooth root canals
Two types of specimens were fabricated for the shear bond strength test and the pull-out test. Seventy bovine anterior teeth were prepared for each test. Previously frozen extracted bovine teeth were thawed and the crown
was cut above the cement enamel junction using a fine cutter (HS-100, Heiwa Technica, Kanagawa, Japan). Soft tissues surrounding the root and the tooth pulp were removed using hand scalers and endodontic files.

Preparation of specimens for shear bond strength test
Specimen design for shear bond test is shown in Fig. 1(a). Tooth roots were cut in half vertically and embedded perpendicular to the longitudinal axis with epoxy resin (Scandiplex, Fritsch Japan, Yokohama, Japan) in epoxy rings. Waterproof abrasive papers 120 grit were used on the root surface until dentin exposure then sequentially polished to 600 grit. Root canal treatment was performed on the dentin surface by applying 1 mL of 3% sodium hypochlorite (NaOCl; ChlorCid® J, Ultradent Japan, Tokyo, Japan) for 20 s, drying with paper points, then applying 2 mL of 18% ethylenediaminetetraacetic acid (EDTA; Ultradent® EDTA18%, Ultradent Japan) for 20 s, and irrigating with distilled water. After air drying, calcium hydroxide reagent (Calvital®, Neo dental chemical products, Tokyo, Japan), mixed at a powder to liquid ratio of 2:1 (P/L), was applied on the dentin surface of half of the specimens. All specimens were stored in a sealed container with a small amount of water and incubated at 37°C and 95% humidity for 7 days. After 7 days, calcium hydroxide treated specimens were ultrasonically cleaned for 1 min with distilled water to remove the reagent.

Cylindrical specimens were made with resin composites and used as adhesive substrates. Acrylic rings with an inner diameter of 6 mm and thickness of 2 mm were filled with resin composite (Clearfil DCcore Automix® ONE, Kuraray Noritake Dental, Tokyo, Japan). The surface was pressed with glass plates and light cured for 30 s. The cylindrical substrate was bonded to dentin surface after root canal treatment using various resin cements shown in Table 1. Two types of adhesives requiring pretreatment, Panavia® F2.0 (PAF, Kuraray Noritake Dental) and RelyX™ Ultimate Adhesive Resin Cement (ULR, 3M ESPE, St. Paul, MN, USA) and 3 types of self-adhesive cements RelyX™ Unicem2 AutoMix (UNA, 3M ESPE), Clearfil® SA Luting (CSA, Kuraray Noritake Dental), and G-CEM (GCM, GC, Tokyo, Japan) were used. Three millimeter holes were punched on double sided tape (Teraoka Tokyo, Japan). Post space 4.0 mm deep were prepared on tooth using a 3.0 mm drill, then rinsed with EDTA and air dried, calcium hydroxide reagent (Calvital®, Neo dental chemical products, Tokyo, Japan) for 20 s, and irrigating with distilled water. After air drying, root canal was obturated with gutta percha (Gutta-percha obturator, Morita, Tokyo, Japan). Post space 4.0 mm deep were prepared on tooth using a 3.0 mm drill, then rinsed with EDTA and distilled water.

Specimens design for pull-out test
Specimens design for pull-out test is shown in Fig. 1(b). Tooth with root canals less than 3 mm in diameter were selected. Specimens were fixed in acrylic rings with chemically activated resin (Tray Resin, Shofu, Kyoto, Japan) with the root canal perpendicular to the ring floor. Root canal treatment was performed on the dentin surface by applying 1 mL of NaOCl (ChlorCid® J, Ultradent Japan) for 20 s, drying with paper points, then applying 2 mL of EDTA (Ultradent® EDTA18%, Ultradent Japan) for 20 s, and irrigating with distilled water. After air drying, root canal was obturated with gutta percha (Gutta-percha obturator, Morita, Tokyo, Japan). Post space 4.0 mm deep were prepared on tooth using a 3.0 mm drill, then rinsed with EDTA and distilled water.

Post was fabricated from FRC post 1.6 mm diameter (Fiber post, GC) and resin composite (Clearfil® DC core automix® ONE, Kuraray Noritake Dental). The surface of FRC post was cleaned with ethanol then treated with silane coupling agent (Clearfil® CERAMIC PRIMER PLUS, Kuraray Noritake Dental). The post space was first filled with resin composite, then FRC post was placed into the post space until contact with canal floor at the root center and light cured for 10 s. The post was then removed and light cured again from 4 angles for 10 s each and stored in a sealed container until use. For half of the specimens, dentin surfaces were treated with
Table 1  Adhesives used in this study

| Adhesive resin cement                  | Main component                              | Pretreatment agent                              | Code |
|---------------------------------------|---------------------------------------------|-------------------------------------------------|------|
| Panavia® F2.0 (Kuraray Noritake Dental, Tokyo, Japan) Lot No: 6L0069 (A paste) Lot No: 6L0033 (B paste) | A paste methacrylate monomer, MDP, filler, photo initiator, chemical initiator B paste methacrylate monomer, sodium fluoride, photo initiator, chemical initiator | ED primer II A Liquid: HEMA, MDP, water, chemical initiator (Lot No: 6C0014) B Liquid: methacrylate monomer, water, chemical initiator (Lot No: 6G0014) | PAF  |
| RelyX™ Ultimate Adhesive Resin Cement (3M ESPE, MN, St. Paul, USA) Lot No: 593236 | Base paste glass powder, methacrylate, silica, initiator Catalyst paste glass powder, methacrylate, silica, initiator | Scotch BondTM Universal Adhesive (Lot No: 587884) phosphoric ester acid monomer, methacrylate, initiator, ethanol | ULR  |
| RelyX™ Unicem2 AutoMix (3M ESPE) Lot No: 612923 | Base paste glass powder, phosphoric ester monomer, TEGDMA, silica, initiator Catalyst paste glass powder, methacrylate, silica, initiator | none | UNA  |
| Clearfil® SA Luting (Kuraray Noritake Dental) Lot No: 3J0063 | Paste A Bis-GMA, TEGDMA, MDP, methacrylate monomer, filler, photo initiator, chemical initiator Paste B Bis-GMA, methacrylate monomer, sodium fluoride, filler, accelerator | none | CSA  |
| G-CEM (GC, Tokyo, Japan) Lot No: 1507161 | Powder fluoro aluminosilicate glass, polymerization initiator, colorant Liquid methacrylic ester, 4-AET, phosphoric acid ester monomer, water, silica, initiator | none | GCM  |

calcium hydroxide reagent, mixed at a powder to liquid ratio of 2:1 (P:L). All specimens were stored at 37°C and 95% humidity. After 7 days, calcium hydroxide treated specimens were ultrasonically cleaned for 1 min with distilled water to remove the reagent. Fabricated post was luted to respective dentin specimen using adhesive resin cements shown in Table 1. Excessive cement was removed and light cured from 4 directions for 20 s each. After placing post, specimens were stored in a closed container with a small amount of water and incubated at 37°C and 95% humidity for 7 days. Seven specimens were prepared for each type of cement for calcium hydroxide treated and non-treated groups.

**Pull-out test**
After 7 days, specimens were taken out of storage and pull-out test was performed using a universal testing machine (EZ graph, Shimadzu) at a crosshead speed of 0.5 mm/min. Retention force was considered the maximum pull-out force. After testing, fractured surfaces were investigated under the stereo microscope digital camera system (×1.25 magnification).

**SEM observations**
The surface of resin composite specimens after shear bond strength test, and dentin specimens after polishing, post space preparation, EDTA treatment, calcium hydroxide treatment and ultrasonic cleaning, and shear bond strength tests was observed under the field emission scanning electron microscopy (FE-SEM: SU6600, HITACHI, Tokyo, Japan).

**Statistical analysis**
All statistical testing was performed using software (Ekuseru-Toukei 2015, Social Survey Research Information, Tokyo, Japan). The bond strength and the retention force of posts with and without calcium hydroxide treatment and with various resin adhesives were statistically analyzed using two-way analysis of variance (ANOVA) and Tukey’s multiple comparison test. The Spearman’s rank correlation coefficient was used to investigate the relationship between shear bond
strength and retention force of posts. Significance level was set at 5% ($\alpha=0.05$).

RESULTS

SEM observation of treated surface
Figure 2 shows the root dentin surface after polishing, post preparation, EDTA treatment, and ultrasonic cleaning. The surface of specimens after polishing (Fig. 2(a)) and post space preparation (Fig. 2(b)) had a smear layer covering the dentinal tubules. After EDTA treatment (Fig. 2(c)) the smear layer was removed and dentinal tubules were exposed. After 7 days of calcium hydroxide treatment (Fig. 2(d)), images showed unidentifiable deposits on the dentinal surface and tubules even after ultrasonic cleaning.

Shear bond strength and fracture mode
Figure 3 demonstrates shear bond strength of various adhesives when bonded to root dentin. Shear bond strength of adhesive resin cements ranged from 4 to

![Graph showing shear bond strengths of resin composites bonded to root dentin.](image)

Fig. 3 Shear bond strengths of resin composites bonded to root dentin. The same letter in the same case indicates no significant differences. Large case letters indicates no calcium hydroxide treatment and small case letters indicates calcium hydroxide treatment.

| Table 2 | Results of two-way ANOVA for shear bond strength |
|---------|-----------------------------------------------|
|          | Sum of squares | df | Mean square | $F$  | $p$     |
| A: Cement       | 1,442.705 | 4  | 360.676      | 43.365 | 0.000  |
| B: Calcium hydroxide reagent | 14.846   | 1  | 14.846        | 1.785  | 0.187  |
| Interaction A*B | 52.896    | 4  | 13.224        | 1.590  | 0.189  |
| Error           | 499.036   | 60 | 8.317         | —      | —      |
| Total           | 2,009.483 | 69 | —            | —      | —      |
15 MPa when treated with calcium hydroxide reagent. Table 2 shows results of two-way ANOVA for shear bond strength. No significant differences were found between the calcium hydroxide treated and non-treated groups for all types of cements ($p>0.05$); however significant differences were found between the various adhesive resin cements ($p<0.05$). The interaction between calcium hydroxide treatment and adhesive cements did not significantly affect shear bond strength ($p>0.05$). ULR possessed the greatest shear bond strength with 15±3 MPa; CSA and GCM possessed lower shear bond strength at approximately 5 MPa.

Figure 4 shows the fracture mode of specimens after shear bond strength test. Most of CSA and GCM specimens showed adhesive failure at the interface between root dentin and resin composite regardless of calcium hydroxide treatment. PAF, ULR, and UNA demonstrated cohesive failure in 42–57% of specimens. Figures 5(a)–(j) displays SEM images of dentinal surface with and without calcium hydroxide treatment. Specimens without treatment displayed remains of adhesive resin cement in the dentinal tubules. Specimens with treatment revealed unidentifiable deposits in addition to adhesive resin cement remains in the dentinal tubules.

Figure 6 displays the resin composite surface after

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Fig. 4  Failure modes of specimens that underwent shear bond strength test.

Fig. 5  SEM observation of dentin surface after shear bond strength test. (a) and (f) PAF, (b) and (g) ULR, (c) and (h) UNA, (d) and (i) CSA, (e) and (j) GCM (a)–(e): dentinal surface without calcium hydroxide treatment (f)–(j): dentinal surface with calcium hydroxide treatment

Fig. 6  Typical resin composite surfaces after shear bond strength test. Both composites were bonded to root dentin after calcium hydroxide treatment. (a) ULR, (b) CSA
shear bond strength test with root dentin that was treated with calcium hydroxide reagent. Resin tags of adhesives were observed on some of the specimens.

**Retention force of posts and fracture mode**
Figure 7 displays the retention force of posts bonded with adhesive resin cements. Table 3 shows result of two-way ANOVA for retention force of FRC posts. Results were similar to that of bond strength in that the retention force of posts with and without calcium hydroxide reagent showed no significant differences \((p>0.05)\). Significant differences were found among the adhesive resin cements \((p<0.05)\). The interaction between calcium hydroxide treatment and adhesive cements did not significantly affect retention force \((p>0.05)\). ULR demonstrated the largest retention force with \(400\pm144 \text{ N}\); CSA and GCM demonstrated lower at approximately \(150 \text{ N}\).

Figure 8 shows the fracture mode of specimens after pull-out test. UNA, CSA, GCM specimens all showed adhesive failure between root dentin and post. PAF and ULR specimens showed either adhesive failure between resin composite and FRC post or cohesive failure of resin composite.

**DISCUSSION**
This study investigated the effects of calcium hydroxide reagent on the bond strength and retention force of FRC post on root dentin. Calcium hydroxide reagents are used during root canal treatments before post preparation. The application of calcium hydroxide reagent on root canals may alter the microstructure of root dentin by reacting with salivary components to produce calcium carbonate and calcium phosphate. In this paper, structural changes due to calcification cannot be expected since the specimens were placed in solution without mineral elements after the application of calcium hydroxide. Root canal treatment itself is a complicated procedure and many steps are involved. However shear bond strength test and pull-out test were used as a simplify method in order to investigate the effects of calcium hydroxide remaining on root dentin which could often occur in a clinical setting.\(^\text{18}\).

**Calcium hydroxide reagent treatment**
Calcium hydroxide reagents are effective for root canal treatments due to their high pH which leads to antibacterial effects\(^\text{14}\). Lee et al. studied the bond
strength of FRC post on root canal dentin via push-out method and reported that calcium hydroxide treatment decreases bond strength of adhesive resin cement\(^\text{18}\). However other studies using shear bond strength test and micro tensile test indicated that calcium hydroxide treatment on root dentin does not affect bond strength to resin cement\(^\text{19,20}\). These studies indicate that there is still no consensus on the effects of calcium hydroxide reagents on bond strength to resin cement. In our study, calcium hydroxide treatment on root dentin had no effect on the bond strength of resin cement to root dentin, and on the retention force of post made with FRC post and resin composite. Therefore the null hypothesis that calcium hydroxide decreases bond strength and retention force of post bonded to root dentin via adhesive resin cement can be rejected.

After root canal treatment, the remains of medicinal reagents can affect the adhesive system during post and core build-up. The general root canal procedure involves treatment with sodium hypochlorite and EDTA to remove the smear layer and expose the dentinal tubules (Fig. 2(c)). When calcium hydroxide was applied to dentin surface and ultrasonically cleaned, SEM images showed that dentinal tubules were partially exposed; however some calcium hydroxide still remained in the tubules (Fig. 2(d)). The fracture surface of dentin after shear bond strength test showed damaged resin tags as well as exposed dentinal tubules in both calcium hydroxide treated groups and non-treated groups (Fig. 5). In addition, the fracture surface of resin composite specimens had structures which appeared to be detached resin tags (Fig. 6). Studies up until now indicated that there is no effective way to completely remove calcium hydroxide from root canals\(^\text{15-17}\). Since calcium hydroxide is alkaline, the condition may neutralize etching agents and acidic monomers in bonding agents, thereby decreasing the bond strength. However, our results indicated that calcium hydroxide had no effect on the bond strength and retention force of posts on root dentin. The reason for this is that ultrasonic cleaning after calcium hydroxide treatment most likely washed off a majority of the remaining reagent for it to affect the adhesive system.

Shear bond strength and post retention force

Evaluation methods for bond strength of dental materials in dentistry usually include the micro-tensile and shear bond strength test\(^\text{8,21-26}\). Root dentin has lower potential for bonding with less exposure of dentinal tubules compared to coronal dentin\(^\text{7}\); reagents used during root canal treatment can affect bond strength even more\(^\text{13,22}\). In this study, we investigated the effects of calcium hydroxide on the bond strength of root dentin by bonding to resin composite, a core build-up material, with adhesive resin cement. Bond strength significantly varied among the types of adhesive resin cements used. A number of studies have been undertaken to compare the bond strength of various resin cements to dentin\(^\text{8,21,23-26,28}\). Most of these studies reported that adhesive resin cements, that require pretreatment, have higher bond strength compared to self-adhesive resin cements\(^\text{23,24,26}\). These results are consistent with our study where adhesive resin cements, ULR and PAF, which require pretreatment demonstrated greater bond strength.

Post retention is usually evaluated through push-out or pull-out tests. The push-out test is effective for evaluating bond strength of root dentin at different sections\(^\text{10}\). This study used the pull-out test to evaluate the adhesion resistance of post in root canals to consider post debonding which is a frequent clinical complication\(^\text{29}\). As previously discussed, posts bonded with PAF and ULR requiring pretreatment, displayed higher retention force compared to self-adhesive resin cements UNA, CSA and GCM (Fig. 7). The explanation for this observation can be the fact that self-adhesive cements take time to completely cure, indicated in the study by Soejima \textit{et al.} where the mechanical strength of self-adhesive resin cements increased after 14 days\(^\text{7}\). In addition, self-adhesive cements possess low retention force of posts due to insufficient formation of resin tags\(^\text{7}\). In our study, bond strength and retention force of posts was most likely influenced by the time of before testing.

Figure 9 displays the correlation between retention force of post and shear bond strength. The correlation coefficient (R) was 0.91 indicating a strong correlation (\(p<0.05\)). The y-intercept value was a positive value indicating that even without cement for adhesion, factors other than retention force had an influence on bond strength. This other factor is most likely friction which exists between the root dentin and cement\(^\text{28}\). In this study, a cylindrical post space was prepared where FRC post and resin composite was used to make a fitted core which could be sufficiently cured before placement. In this situation where the post-and-core build-up resin composite is fully cured, the bond strength of resin cement would be considerably large resulting in
high retention force. As study has shown that the post shape is related to its fracture resistance39; therefore further investigation is required to understand the relationship between post shape and retention force on endodontically treated tooth.

CONCLUSIONS
In this study, the effects of calcium hydroxide treatment on shear bond strength and retention force of posts of adhesive resin cements to root dentin was investigated. Within the limitation of this study, the following was concluded:
1. Calcium hydroxide treatment on root dentin did not affect both the shear-bond strength of adhesive resin cements and the retention of posts.
2. There was a correlation between shear bond strength and retention force of posts of adhesive resin cements; specimens with high shear bond strength generally demonstrated high retention force of posts.

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