Effect of adhesive system on retention in posts comprising fiber post and core resin

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The purpose of this study was to compare the retention of fiber-reinforced composite (FRC) posts luted with either conventional or self-adhesive resin cement. The FRC posts and core resin were built up in bovine teeth. The posts were luted with standard etch-and-rinse cement, self-etch cement, or one of two self-adhesive cements. The samples were stored in water for 1 or 14 days or subjected to thermal cycling (TC). Retention value was measured with the pull-out test using a universal testing machine. Conventional adhesive resin cement yielded significantly greater retention than self-adhesive resin cement at 1 day. No significant difference was observed in retention among the adhesive systems tested at 14 days or after TC. During the early luting stage, self-adhesive resin cement yielded lower retention value than conventional resin cement. After 14 days storage or TC, retention was comparable to that with conventional resin cement.

Keywords: Fiber post, Adhesive, Pull-out test, Aging, Polymerization

INTRODUCTION

Fiber-reinforced composite (FRC) posts are used to build up the core in endodontically treated teeth. Due to the similarity between the elastic modulus of this particular material and that of the natural tooth, it is possible to achieve a low rate of root fracture while preserving good esthetics1-3). Some clinical studies, however, have reported that FRC posts are prone to debonding due to unstable adhesion between the post and root canal dentin2-6). Therefore, stable adhesion between the post and root canal dentin is critical for the long-term success of such procedures.

When luting posts with conventional adhesive resin cement, it is necessary to first etch and rinse the root canal dentin or use a self-etching primer7-12). An acid etchant or self-etching primer is first applied to the intraradicular dentin, after which resin cement is applied to the post and root canal dentin. Both methods result in higher bond strength on the coronal dentin. It should, however, be pointed out that poor moisture control or incomplete resin inter-diffusion can significantly decrease bond strength, and technical errors may easily occur due to the complex two- or three-step procedure involved10). Recently, several self-adhesive resin cements have been introduced that do not require any pretreatment of the tooth substrate; once the cement is mixed, the application is accomplished in a single clinical step13,14). The use of self-adhesive resin cement can decrease chair time for patients by simplifying and improving the operating technique15).

While several studies have indicated that the bond strength of self-adhesive cement is comparable to that of conventional adhesive resin cement16-18), others have reported that that of the latter is superior20-23). Self-adhesive resin cement involves a more complicated dual-cure mechanism than conventional resin cement24).

Moreover, Baena reported that post-curing increases the hardness of self-adhesive resin cement25). This suggests that factors such as temperature change and storage period affect bonding strength. Mazzoni et al. showed that the strength of conventional adhesive resin cement was more stable than that of self-adhesive resin cement following thermal cycling26). However, few studies have compared the features of self-adhesive resin cement with that of other multi-step luting agents.

The retention of posts and their integration with other materials on the tooth have been evaluated by the push-out and pull-out methods27,28). Post retention depends not only on the adhesive cement, but also on friction between the post and intraradicular dentin29). Therefore, determining retention requires the actual oral environment to be reproduced as closely as possible. Unfortunately, few studies have investigated fiber posts, and core resin-constructed posts are made based on overall retention and the effects of adhesive systems.

The goal of the present study was to compare the retention of FRC posts luted with either conventional or self-adhesive resin cement on endodontically treated dentin. The influence of storage time and thermocycling (TC) on retention was also investigated.

MATERIALS AND METHODS

Root preparation

Extracted bovine incisors in frozen storage were used in this study. Crowns were cut at the cemento-enamel junction and roots with pulp cavities less than 3 mm in diameter selected. Root canal preparation was performed by the crown-down method using Ni-Ti rotary instruments (ProTaper Universal, Dentsply Sankin, Tokyo, Japan). The working length for root canal treatment was set at 1 mm from the root apex. The root canal was irrigated with about each 3 mL of
Table 1  Adhesive resin luting systems used in this study

<table>
<thead>
<tr>
<th>Type</th>
<th>Luting agent (company)</th>
<th>Component</th>
<th>Etching agent</th>
<th>Code</th>
</tr>
</thead>
</table>
| Etch-and-rinse            | Super-Bond C&B (Sun Medical) | Monomer liquid: 4-META, MMA  
Powder: PMMA  
Paste A:  
MDP, Methacrylate monomer, filler, photo initiator, chemical initiator  
Paste B:  
Methacrylate monomer, sodium fluoride, filler, photo initiator, chemical initiator | Green Activator:  
10 wt% citric acid,  
3 wt% ferric | SB |
| Self-etch                 | Panavia F 2.0 (Kuraray Medical) | A paste:  
Bis-GMA, TEGDMA, MDP, methacrylate monomer, filler, photo initiator, chemical initiator  
B paste:  
Bis-GMA, methacrylate monomer, sodium fluoride, filler, accelerator | None | SA |
| Clearfil SA Luting (Kuraray Medical) | None | None | None | GL |
| Self-adhesive resin cement | G-luting (GC) | A paste:  
Fluor alumino silicate glass, methacrylate ester  
B paste:  
Silica filler, methacrylate ester, Phosphate ester monomer | None | GL |

Post-and-core fabrication

Obturated roots were randomly divided into 4 groups and root canal posts constructed using serrated FRC posts (Fiber post: FibreKleer®: 1.5 mm in diameter, Pentron Japan, Tokyo, Japan) and core resin (PostResin, Sun Medical Co., Shiga, Japan) due to adaptation to the root walls and reduces the thickness of the resin cement. The surface of the FRC post was treated with a saline coupling agent (Ceramic Primer, GC, Tokyo, Japan) and air-dried. The direct-indirect technique was used and separating agents (Super-bond SEP, Sun Medical Co.) applied to the root canal walls before filling the root canal with core resin. Post resin was injected into the root canal using a syringe. A fiber post was then inserted and light-cured (Griplight II, Shofu, Kyoto, Japan) for 10 s. After light-curing, the post was pulled out and light-cured for a further 10 s from 4 directions. After curing, the post and post space were rinsed with water and dried to remove the remains of the separating agent. The posts were luted with the 4 types of luting agent shown in Table 1. The following 4 types of conventional adhesive resin cement were used: the etch-and-rinse cement Superbond C&B (Sun Medical Co.: SB); the self-etch cement Panavia F2.0 (Kuraray Noritake Dental, Tokyo, Japan: PA); and the self-adhesive cements Clearfil SA luting (Kuraray Noritake Dental, Tokyo, Japan: SA) and G-luting (GC, Tokyo, Japan: GL). Each cement system was handled according to the manufacturer’s instructions. The luted samples were stored in water at 37°C for 1 or 14 days (hereafter, denoted as 1 day or 14 days) or subjected to TC (10,000 cycles) in water at 4°C and 60°C, with the dwelling time at each temperature at 30 s to give 1 cycle. The TC samples were initially placed in water at 37°C for 24 h prior to TC, after which they returned to 37°C water until day 14.

Evaluation of retention force

Figure 1 is a schema of the pull-out test. The luted samples were placed in acrylic rings (17 mm in diameter, 30 mm in height) and embedded in chemically activated resin (Tray Resin, Shofu, Kyoto, Japan). An extension pipe 4 mm in diameter and 8 mm in length was attached to the fiber post by filling the chemically activated resin.
in the space to allow it to be held. An instrument was used to hold the sample at the extension and the pull-out test performed using a universal testing machine (Autograph AG-I 20 kN, Shimadzu, Kyoto, Japan) at a crosshead speed of 0.5 mm/min. The maximum load recorded in the pull-out test was considered to indicate degree of retention. After the pull-out test, a digital microscope (VH-5000, Keyence, Osaka, Japan) was used to search for any fracturing. Fracture mode was classified as follows: fracture between the fiber post and core resin (P/R); fracture between the core resin and dentin (R/D); or a mixture of both P/R and R/D (Mix). When a specimen exhibited P/R mode, additional specimens were prepared in each group to allow a precise evaluation of the influence of the adhesive resin cement system on retention.

**SEM observation**
In order to observe the adhesive surface of the posts, the root canal was treated with EDTA and then cut using a diamond saw (ISOMET 1000, Buehler, Tokyo, Japan). Additional samples were prepared to observe the adhesive surface. Samples with luted posts were also cut using the diamond saw and the adhesive cross-section etched for 10 min with 6 mol/L hydrochloric acid. The samples were sputter-coated with gold and then observed under a scanning electron microscope (FE-SEM:SU6600, HITACHI, Tokyo, Japan). The root dentin and post surface of certain samples were observed under FE-SEM after the pull-out test.

**Statistical analysis**
Degree of retention was determined with a one-way analysis of variance (ANOVA) or two-way ANOVA with respect to type of adhesive resin cement, storage period, and TC. Statistical differences were also analyzed using the Tukey-Kramer multiple comparison test at a 5% level of significance.

**RESULTS**

**Post retention and fracture mode**
The average retention value and standard deviation of all posts luted with each adhesive system are shown in Table 2. Retention at 1 day was as follows: 305 N for SB, 282 N for PA, 139 N for SA, and 181 N for GL. Retention values for SB or PA was comparable, but significantly greater than that for SA or GL. Unlike at day 1, no clear difference was observed between cement types at 14 days, with retention value ranging from 217 N (GL) to 278 N (SB). With TC, retention values ranged from 198 N (SA) to 289 N (SB), similar to that at 14 days, with no significant difference being found between groups. In addition, no statistically significant difference was found in retention value between each adhesive system depending on storage period or TC.

Figure 2 shows the ratio and numbers of all different fracture modes. In posts luted with SB or PA, P/R was observed in 40% of cases, regardless of storage period or TC. Fewer than 17% of specimens luted with SA or GL showed P/R.

Figure 3 shows retention values in 10 samples under each condition according to the pull-out test, exclusive of the data on posts showing P/R. Retention value with SB and PA at 1 day was 313±109 N and

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**Table 2** Mean retention values and standard deviations (N)

<table>
<thead>
<tr>
<th></th>
<th>Luting system</th>
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<tbody>
<tr>
<td></td>
<td>SB</td>
</tr>
<tr>
<td>1 day</td>
<td>305 (113) b</td>
</tr>
<tr>
<td></td>
<td>(n=16)</td>
</tr>
<tr>
<td>14 days</td>
<td>278 (92) a</td>
</tr>
<tr>
<td></td>
<td>(n=19)</td>
</tr>
<tr>
<td>TC</td>
<td>289 (93) a</td>
</tr>
<tr>
<td></td>
<td>(n=15)</td>
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</tbody>
</table>

\(n\): number of samples

Same superscript small letters indicate no significant differences among different adhesive luting cement systems (reading horizontally). Same superscript capital letters indicate no significant differences among storage periods and thermal cycling (reading vertically).
Fig. 2 Outcome of fracture analysis in all samples.
Fraction of fracture modes after pull-out test of posts luted to endodontically treated root dentin with different systems and then stored in 37°C-water for 1 or 14 days or aged by thermal cycling (TC). Right side shows the number of samples.
P/R: Fracture between fiber post and core resin, R/D: Fracture between root dentin and core resin, Mix: Combination of P/R and R/D or fracture within core resin.

Fig. 3 Retention force of posts luted to endodontically treated dentin with different systems and then stored in 37°C water for 1 or 14 days or aged by thermal cycling (TC).
Results do not include retention in specimens fractured at interface between fiber post and core resin. Same superscript small letters indicate no significant differences among adhesive luting cement systems, storage periods, or thermal cycling (TC).

273±43 N, respectively, values significantly greater than those for SA (138±64 N) or GL (179±84 N). Unlike at day 1, retention value at 14 days was 211±49 N with GL and 250±68 N with SB, with no significant difference being observed among cements. With TC, retention value was 199 N±49 N for SA and 266±96 N for SB, with no significant difference being found, similar to at 14 days. In terms of storage period and TC, SA showed significantly greater retention at 14 days than at 1 day.

Figure 4 shows the ratio (%) of fracture modes after the pull-out test, excluding the data for P/R mode.
Regardless of storage period or TC, fracture type for SB was equally divided between R/D and Mix, and fracture type for PA was mostly Mix. At 1 day, R/D was 40–70% with SA or GL. However, at 14 days or TC, over 70% of fracture cases were Mix.

Observation of interface of root dentin and resin cement
Figures 5 (a) and (b) show the root dentin after the drilling a post hole and treatment with EDTA. A smear layer was observed during post hole preparation, as shown in Fig. 5 (a). Figure 5 (b) shows the removal of the smear layer from the root dentin after EDTA treatment, after which openings were observed in the dentinal tubules. Figures 5 (c)–(f) show SEM images of adhesive interfaces luted with different adhesive systems. Resin tags were observed infiltrating the dentinal tubules in the root dentin. A hybrid layer was also recognized in some areas on the interface of the root dentin and resin cement.
DISCUSSION

In this study, 4 different agents representing 3 kinds of adhesive system were used to lute fiber posts in endodontically treated bovine root canals in order to compare and evaluate retention with the pull-out test. The microtensile bond test, diametral tensile test, and shear bond test are often used to calculate tensile or shear stress based on adhesive area in order to determine the retention capability of an agent. Retention of luted posts in the hole depends on the degree of adhesion between the fiber post and the core resin, between the adhesive resin cement and the core resin, and between the adhesive resin cement and the root canal dentin. Some parameters also affect bonding within the root canal, such as the post-space preparation method, pretreatment of intraradicular dentin, and high C-factors. These parameters cannot be evaluated based purely on local adhesive strength. Therefore, in certain cases, the push-out and pull-out tests are used to evaluate retention. The push-out test is useful for evaluating bond strength at varying locations in the root canal, such as in the cervical or apical areas of the root20). The pull-out test involves pulling the root parallel to the vertical axis of the tooth in order to determine resistance against pull and/or shear force throughout the entire
The pull-out test was chosen for this study, as testing the post in an entire root canal rather than using root segments has greater clinical relevance.

In general, it is recommended that the post length be approximately 8 mm, which is around 2/3 of the root length. In this study, serrated fiber posts 1.5 mm in diameter were subjected to saline treatment and core resin used to fabricate posts for roots with post spaces of 3 mm in diameter and 4 mm in depth. Preliminary experiments revealed that when the post space depth was 8 mm, the adhesive systems could not be evaluated as the fiber posts broke either at the extension or between the core resins. Therefore, although contrary to what might be expected in a clinical setting, the post space was set at a depth of 4 mm. The results showed that P/R was around 40% for SB or PA, and less than 17% for SA or GL (Fig. 2).

Effect of adhesive system on retention

To determine the effect of adhesive resin cement system on retention, 10 samples were used under each condition (excluding the P/R fracture mode samples). Mean retention value in posts luted with conventional adhesive resin cement was 313±109 N for SB and 273±43 N for PA, and these values were greater than those for SA (138 N) or GL (179 N) at 1 day. Conventional adhesive resin cement requires an acidic etchant or self-etching primer to be applied to the root canal dentin. This allows it to infiltrate the dentin, thus increasing the final adhesive strength. On the other hand, self-adhesive resin cement has been reported to adhere, even if a smear layer is present. In this study, root canal treatment was initially performed with 3% NaOCl and 18% EDTA. After post cavity preparation, EDTA was used again to treat the canal before the posts were luted. This resulted in removal of the smear layer and exposure of the dentinal tubules, so resin tags and a hybrid layer were readily formed (Fig. 5). The surface of the root canal dentin thus allowed good adherence, regardless of the adhesive system used.

Consequently, differences in post retention at 1 day directly reflected the mechanical strength of the resin cement and/or bond strength.

Effect of storage period and thermal cycling

Storage and TC in water are useful in determining the deterioration of materials through water absorption and thermal stress. These factors are thought to affect the bond strength and mechanical strength of materials used for posts (fiber post, core resin, luting cement). For the purposes of this study, the effects of the fiber post and core resin were assumed to be equal under any condition. Therefore, any difference in retention force was considered due to the adhesive resin cement used. In this study, neither water storage or TC affected posts pretreated with conventional resin cement. However, with the self-adhesive cements, retention improved with increase in the length of storage. In addition, with use of self-adhesive cement, fracture between the tooth and core resin occurred in many cases at 1 day, and mixed fracture modes were seen in samples subjected to TC at 14 days. Posts luted with SA showed cement remains on the root dentin and post surfaces after the pull-out test at day 14, with fracture due to cohesive failure (Fig. 6 (a) and (b)). The 4-META/MMA-TBB-type adhesive resin cement cures by means of chemical polymerization, while other composite related adhesive resin cements are of the dual-cured type. Consequently self-adhesive resin cements are initially light cured, after which chemical polymerization continues until curing is complete. Baena et al. demonstrated that post-curing increases hardness in self-adhesive resin cement. In our preliminary studies, SA yielded compressive strength of 236 MPa at 1 day, with strength increasing to 280 MPa at 14 days. Thus, the post retention of self-adhesive resin cements increases after 14 days of storage due to post-curing, which leads to an increase in mechanical strength.

Goracci et al. reported that remnants of acidic monomers, a component of self-adhesive cement, decreased bond strength by hydrolysis. Cal et al.
reported that micro-leakage increased in self-adhesive cement as a result of TC\(^26\). This suggests that self-adhesive cement is sensitive to water absorption and thermal stress. In this study, the retention of posts subjected to TC was comparable with that of luted posts at 14 days, and no significant difference was observed among the various cements. This indicates that TC (10,000 cycles) has no effect on the retention capability of adhesive resin cements. Palacios et al. also reported that there was no difference in bond strength between conventional resin cements and self-adhesive cements subjected to TC\(^32\). This may be due to pretreatment, as previously described. That is, application of EDTA to the root dentin removes the smear layer, enabling resin tags to form; in addition, the post-curing of resin cement leads to an increase in mechanical strength, improving retention. This increase in mechanical strength makes up for the deterioration caused by TC and the differences in values became harder to detect.

Gale et al. suggests that approximately 10,000 cycles represents a service year\(^29\). The majority of earlier studies investigating the effects of TC confirm that the number of cycles performed in this study was appropriate. However, Mazzoni et al. reported decreased bond strength resulting in increased nano-leakage after 40,000 cycles\(^26\). Further study involving larger numbers of TC is needed to clarify the long-term durability of retention obtained with self-adhesive resin cements.

In this experiment, two types of self-adhesive cement with different components were used to evaluate the retention of luted posts. The retention of posts luted with these two types of self-adhesive resin cement was lower than that of posts luted with conventional adhesive resin cements during the early luting stage. However, while the results at 14 days and after TC showed that type of cement had no effect on retention, they do indicate that the curing mechanism of the cement does, with different mechanisms tend to show different results. The present results demonstrate that it is important to recognize that retention during the early luting stage will be relatively low when using self-adhesive cement to lute posts.

**CONCLUSION**

This study compared post retention between conventional and self-adhesive resin cements under different storage periods and thermal stress. Within the limitations of this laboratory study, the following conclusions can be drawn:

Conventional adhesive resin cements that utilize etch and rinse or self-etching primers yield significantly greater retention, and thermal stress has little effect on this type of cement.

During the early luting stage, self-adhesive resin cement yields a lower degree of retention than conventional resin cement. After 14 days storage or TC, retention is comparable to that of conventional adhesive resin cement.

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