Microtensile Bond Strength of Dual-cure Resin Cement to Root Canal Dentin with Different Curing Strategies

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The purpose of this study was to measure the bond strength of dual-cure resin cement at different regions of root canal dentin using three kinds of curing method. Thirty-six extracted bovine teeth were used. Each root was sectioned vertically into halves. Their pulpal dentin walls were polished flat and then applied with two dual-cure resin cements (Bistite II, Panavia F), and divided into three curing strategy groups: multi-direction light, one-direction light and no-light. The bonded specimens were sectioned perpendicularly to the long axis of the root into approximately 0.7mm thick slabs within two-third of the root from the coronal end, and prepared for microtensile bond strength (μTBS) test. Knoop hardness of the cements was also measured. Within each curing strategy for both dual-cure resin cements, there were no significant differences between the μTBS values at coronal third and mid third regions. The effect of curing method on bond strength and KHN was found to be dependent on the material.

Key words: Regional bond strength, Root canal dentin, Dual-cure resin cement

INTRODUCTION

Many post-core techniques have been developed for the restoration of endodontically treated teeth. However, clinically, vertical root fractures are often observed in endodontically treated teeth restored with metallic posts cemented with inorganic cement into the root canal⁶. Several studies have demonstrated that cast post-cores and prefabricated metallic posts are not able to reinforce endodontically treated teeth⁶⁰. Recently, carbon fiber posts were introduced because the elastic modulus of carbon fiber posts is close to that of root dentin, hence reducing the risk of root fracture⁶. Mannocci et al.⁵ reported that carbon fiber post-composite core restorations were less likely to produce root fractures than stainless steel post-composite cores. Moreover, the use of dentin adhesive resin cements is based on the premise that adhesive cements are used for bonding fiber posts⁶,⁰. The rationale for using post adhesively bonded to root canal dentin is to reinforce the tooth and help retain the post and coronal restoration⁶⁰.

Many researchers have evaluated the bond strength of adhesive resin cements to coronal dentin⁶⁰-¹¹. However, in coronal dentin, the bond strengths of dentin adhesive systems have been reported to be dependent on the adhesion region⁶²-⁴⁴. Furthermore, tensile strength of dentin varies according to the location and orientation of dentin tubules in the tooth¹⁵-¹⁷. Yoshiyama et al.¹⁸ evaluated the performance of adhesive resins to root versus coronal dentin. They reported that the bond strength to root dentin was dependent on the material¹⁰ because dentin structure (i.e., tubule density and orientation) and permeability varied with intra-tooth location¹⁹,²⁰. The tubular density of pulp corona dentin is significantly different from that of radicular dentin¹,². However, there have been few studies on bonding to root canal dentin²,²⁷.

Recently, dual-cure resin cement combined with a self-etching primer have been developed, which may offer a longer working time and an ability to self-polymerize in regions absent of light. Moreover, self-etching adhesive systems are generally less technique sensitive because they simultaneously demineralize and infiltrate resin monomer into dentin — hence eliminating the rinsing and drying steps²⁸, and they also reduce post-operative sensitivity²⁹. Self-etching primers contain an acidic resin monomer that is incompatible with the chemical polymerization of resin composites by basic amines³⁰. In order to improve the ability to self-polymerize in an acidic environment, a one-step, self-etching/dual-cure resin cement system — which contains sulphinate salts as ternary catalysts — has been developed (Panavia F, Kuraray Medical Inc., Tokyo, Japan). In addition, a
two-step, self-etching system — in which an additional primer is applied to tooth surface treated with self-etching primer prior to the application of resin cement — has been introduced (Bistite II, Tokuyama Dental Co., Tokyo, Japan). When photo-irradiating post cavity from a coronal direction, light energy through resin material would reduce towards the bottom of the post cavity. Therefore, at the bottom of the post cavity, resin polymerization is chiefly one by chemical activation. Okamoto et al.\(^{31}\) evaluated the correlation between the bond strength of dual-cure resin cement and porcelain thickness in porcelain veneer restoration. They reported that the bond strength of dual-cure resin cement was dependent on porcelain thickness, because of the reduction of light energy through porcelain\(^{31}\). An absence of light may result in poor mechanical properties and reduced bonding capacity of dual-cure resin cement to root canal dentin. However, little is known about the bonding performance of dual-cured adhesive cements applied under such conditions. Therefore, the purpose of this study was to measure the bond strength of dual-cure resin cements utilizing one- or two-step self-etching systems at different regions of root canal dentin and to evaluate the effect of light irradiation.

**MATERIALS AND METHODS**

*Specimens preparation*

Thirty-six extracted bovine teeth were used in this study. Their crowns were removed at the cementoenamel junction, and each root was sectioned vertically into halves. Their pulpal dentin walls were polished using wet 600-grit SiC paper and then 200 \(\mu\)m thick spacers were placed. The roots were then randomly divided into two groups: Bistite II (Tokuyama Dental Co., Tokyo, Japan) and Panavia F (Kuraray Medical Inc., Tokyo, Japan). The root canal dentin surface was applied with respective dual-cure resin cement according to manufacturers' instructions (Table 1) (Oxyguard II in Panavia F was not used in this study). After applying each material, the specimens were divided into three sub-groups (Fig. 1).

- **Multi-direction light group**
  The root halves bonded with resin cement without their matching halves were placed inside a laboratory light-curing unit (a-light II, Morita, Tokyo, Japan; conventional light source, 320 W halogen lamp) for 60 seconds in order to sufficiently light-polymerize the cement, and then covered with chemical-cure resin composite (Clearfil FII, Kuraray Medical Inc., Tokyo, Japan).

- **One-direction light group**
  The halves were bonded to their matching halves placed in darkness for 30 min.

**Specimens Preparation**

![Fig. 1 Schematic illustration of the curing strategy for each specimen.](image)

Table 1 Materials tested in this study

<table>
<thead>
<tr>
<th>Material</th>
<th>Manufacturer</th>
<th>Composition (according to manufacturer)</th>
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</table>
| Bistite II | Tokuyama Dental Co., Tokyo, Japan | Primer IA: Phosphoric acid monomer, Acetone  
Primer IB: Alcohol, Water, Initiator  
Primer 2: HEMA, Acetone, Initiator  
Paste A: Silica/Zirconia filler, NPDMMA, Bis-MPEPP  
Paste B: Silica/Zirconia filler, MAC-10, BPO, CQ |
| Panavia F | Kuraray Medical Inc., Tokyo, Japan | ED primer IIA: MDP, HEMA, Chemical initiator, Water, 5-NMSA  
ED primer IIB: 5-NMSA, Chemical initiator, Water  
A paste: Silanated silica, Microfiller, MDP, hydrophobic Dimethacrylates, hydrophilic Dimethacrylates, Photo/Chemical initiator  
B paste: Silanated barium glass, Surface treated NaF, hydrophobic Dimethacrylates, hydrophilic Dimethacrylates, Chemical initiator |
| Bonding procedure | Primer IA+IB  
Primer 2  
Paste A+B | Applied for 30 s, then dried gently  
Applied for 20 s, then dried gently  
Light-cured for 30 s |

Note: Table 1 contains some technical details about the materials used in the study, including their compositions and bonding procedures.
with resin cement and light-cured by a light-curing unit (Tokuso Power Lite, Tokuyama Dental Co., Tokyo, Japan; conventional light source, 80 W halogen lamp) for 60 seconds only from a coronal direction to simulate clinical situation.

**No-light group**

The halves were bonded to their matching halves with resin cement and placed in darkness for 30 minutes in order to polymerize the cement by chemical activation only.

**Microtensile bond strength (μTBS) test**

After water storage for 24 hours at 37°C, the cemented specimens were attached to the arm of a low-speed diamond saw (Isomet, Buehler Ltd., Lake Bluff, IL, USA) and sectioned perpendicularly to the long axis of the root into approximately 0.7 mm thick slabs within the two-third region of the root from the coronal end. The bonded areas were isolated using a superfine diamond bur (c16ff, GC Ltd., Tokyo, Japan) to create an hourglass shape with cross-sectional area of approximately 1 mm². The final width and thickness of the bonded area were measured using a digital caliper (Mitutoyo CD15, Mitutoyo Co., Kawasaki, Japan) to adjust the raw bonding data to an equalized bond/mm². Using cyanoacrylate glue, each specimen was carefully bonded to a testing device (Bencor-Multi-T, Danville Engineering Co., San Ramon, USA) mounted on a tabletop material testing machine (EZ Test, Shimadzu, Kyoto, Japan) and subjected to a tensile force at cross-head speed of 1 mm/min (Fig. 2). The data were divided into two groups: coronal third and mid third groups. All failed specimens were glued to brass tables, gold sputter coated, and their failure modes determined under a scanning electron microscope (JSM-5310, JEOL, Tokyo, Japan).

**Knoop hardness measurement of cement**

To measure the Knoop hardness of resin cements with each curing method, bonded specimens were prepared in a manner similar to those of μTBS test, and then sectioned perpendicularly to the resin cement layer. These halves were placed into acrylic rings, which were attached to adhesive tape and embedded in epoxy resin. After the epoxy resin had set, the acrylic resin blocks were polished using ascending grades of abrasive SiC paper under running water, and then polished using diamond paste (DP-paste P, Struers A/S, Denmark) down to 0.25 μm.

Each block was positioned on the traveling micrometer stage of a microindentation tester (Akashi MVK-E hardness tester, Omron Takei Electronics Co., Tokyo, Japan) fitted with a diamond Knoop indenter (Fig. 2). A load of 50 g was applied for a dwell period of 15 seconds. Indentations were made vertically, starting from the coronal end, in the upper left, mid and right third regions of the dual-cure composite resin at 1.0-mm intervals. Using ×400 magnification, the length of the long diagonal (μm) of each indentation was measured and the Knoop Hardness Number (KHN) calculated. The data were divided equally into two groups: coronal third and mid third regions.

**Statistical analysis**

The μTBS and KHN data (mean±SD, MPa) were analyzed by three-way ANOVA (curing method, region, material), two-way ANOVA (curing method, region) for each material, as well as by Fisher PLSD post-hoc test (p<0.05).

**RESULTS**

The μTBS data together with the statistical analysis results are presented in Table 2. Three-way ANOVA revealed that there were no interactions among the three factors (material, region, curing method) for

<table>
<thead>
<tr>
<th></th>
<th>Bistite II</th>
<th>Panavia F</th>
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<tbody>
<tr>
<td></td>
<td>Coronal third</td>
<td>Mid third</td>
</tr>
<tr>
<td>Multi-direction</td>
<td>20.1±13.6ab</td>
<td>18.0±9.7ab</td>
</tr>
<tr>
<td>One-direction</td>
<td>14.3±7.5abc</td>
<td>16.0±9.2abc</td>
</tr>
<tr>
<td>No-light</td>
<td>11.5±8.6abc</td>
<td>13.5±8.7abc</td>
</tr>
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All values are mean±SD. Number of specimens in each group=15
Groups with the same case superscript letters are not significantly different (p>0.05)

**Panavia F**

<table>
<thead>
<tr>
<th></th>
<th>Coronal third</th>
<th>Mid third</th>
</tr>
</thead>
<tbody>
<tr>
<td>Multi-direction</td>
<td>11.3±4.0a</td>
<td>14.1±5.3a</td>
</tr>
<tr>
<td>One-direction</td>
<td>11.3±4.3a</td>
<td>8.7±4.0a</td>
</tr>
<tr>
<td>No-light</td>
<td>11.6±7.0a</td>
<td>13.9±5.0a</td>
</tr>
</tbody>
</table>

All values are mean±SD. Number of specimens in each group=15
Groups with the same case superscript letters are not significantly different (p>0.05)
\( \mu \text{TBS} \) (\( p > 0.05 \)). The \( \mu \text{TBS} \) of Bistite II was significantly higher than that of Panavia F (\( p = 0.0014 \)), and there were significant differences between the curing methods (\( p = 0.0301 \)). The \( \mu \text{TBS} \) values were not significantly different between the coronal third and mid third regions (\( p = 0.5518 \)).

Two-way ANOVA revealed that for Bistite II, the \( \mu \text{TBS} \) values were significantly different between curing methods (\( p < 0.05 \)); while for Panavia F, there were no significant differences between curing methods (\( p > 0.05 \)). The multi-direction group of Bistite II was higher in bond strength than the no light group in each region of the root canal dentin (coronal third: 20.1±13.6 and 11.5±8.6 MPa respectively; mid third: 18.0±9.7 and 13.5±8.7 MPa respectively). Light irradiation from a coronal direction resulted in no significant differences in \( \mu \text{TBS} \) between the coronal third and mid third regions (14.3±7.5 and 16.0±9.2 MPa respectively) (\( p > 0.05 \)). On the other hand, for Panavia F, there were no significant differences in \( \mu \text{TBS} \) among all the groups. The \( \mu \text{TBS} \) values of the no-light group in the coronal third and mid third regions were 11.6±7.0 and 13.9±5.0 MPa respectively, similar to those of Bistite II.

Microhardness (KHN) data are shown in Table 3. Three-way ANOVA analysis revealed that there was an interaction between the material and curing method (\( p < 0.0001 \)). The KHN of Bistite II cement in the multi-direction group was significantly higher than that of the no-light group (\( p < 0.05 \)). In addition, cement hardness in one-direction group significantly decreased apically (\( p < 0.05 \)). For Panavia F,

<table>
<thead>
<tr>
<th>Curing Strategy</th>
<th>Bistite II</th>
<th>Panavia F</th>
</tr>
</thead>
<tbody>
<tr>
<td>Coronal third</td>
<td>80.2±9.6(^a)</td>
<td>84.4±10.4(^a)</td>
</tr>
<tr>
<td>Mid third</td>
<td>81.4±11.1(^a)</td>
<td>71.6±11.5(^f)</td>
</tr>
<tr>
<td>No-light</td>
<td>65.2±8.8(^b)</td>
<td>62.6±8.7(^b)</td>
</tr>
</tbody>
</table>

All values are mean±SD (n). Number of specimens in each group=15
Groups with the same case superscript letters are not significantly different (\( p > 0.05 \))

Fig. 3 SEM of a fractured specimen of Bistite II with multi-direction light exposure at the coronal third region. Adhesive failure at the interface is seen (\( \times 5000 \)).

Fig. 4 SEM of a fractured specimen of Bistite II with no-light at the mid third region. Mixed adhesive/cohesive failures at the resin-dentin interface is seen (\( \times 2000 \)).

Fig. 5 SEM of a fractured specimen of Panavia F with multi-direction light exposure at the coronal third region. Mixed adhesive/cohesive failures at the resin-dentin interface is seen (\( \times 5000 \)).
the KHN values were not significantly different between each region for all the curing methods (p > 0.05). With light irradiation, the hardness of Bistite II cement was greater than that of Panavia F cement (p < 0.05).

SEM observation of the fracture sites revealed that, in the case of Bistite II, most of the specimens of multi-direction group failed at the interface (Fig. 3). The one-direction and no-light groups showed mixed failure modes: adhesive failure at resin/dentin interface and cohesive failure within cement (Fig. 4). In the case of Panavia F, all specimens exhibited mixed failure modes at the interface and within the cement (Fig. 5).

DISCUSSION

Tubular density and diameter of the tubules have become important factors for bonding to dentin after acid etching. It has been reported that the bond strength to coronal dentin was dependent upon the tubular density of dentin. Ferrari et al. showed that the tubular density of pulpal coronal dentin was significantly greater than that of radicular dentin in all sites evaluated. Moreover, wide variations in tubular density between various locations within and between teeth of the same individual and between teeth of different individuals were found. Aging is known to cause morphological changes in dentin, such as the growth of peritubular dentin and occlusion of the tubules by minerals. With SEM observation of human anterior teeth aged around 59 years (range 55-68), it was reported that significant differences in the tubular density of root canal dentin existed, depending on location. However, Gaston et al. had different findings when they measured the regional tensile bond strength of chemically cured adhesive resin cements to the internal or endodontic surface of human radicular dentin. They reported that there were no statistically significant differences between the adhesive materials in any region (cervical, middle, apical), and that the apical bond strengths were significantly higher than those of cervical or middle dentin. These results are in agreement with the present study using dual-cure resin cement with self-etching primer. Pereira et al. investigated the effect of intrinsic wetness and regional difference on coronal dentin bond strength, and Akagawa et al. evaluated the bond strength to coronal dentin and pulp chamber floor dentin using self-etching primer adhesive system. They reported that the bond strengths of the tested self-etching primer adhesive systems were hardly influenced by regional differences in dentin. In this study, there were no findings of regionally led differences in μTBS in the multi-direction and no-light groups of both cements. This could be due to the mild etching effect of the self-etching primers. On the other hand, the absence of morphological changes in root dentin of young bovine teeth used in this study could have affected the μTBS results too.

Dual-cure resin cement contains both chemical initiators and photoinitiators. Therefore, it has the ability to polymerize in the absence of light. However, it was reported that chemical curing alone was not sufficient to achieve maximum hardening of the dual-cure resin cements. For dual-cure resin cement, a high degree of polymerization is necessary to ensure good mechanical properties and bonding capability. In the present study, for the Bistite II system, the μTBS of the multi-direction group was higher than that of the no-light group. Moreover, SEM observation of fracture sites revealed that most specimens of the multi-direction group failed at the interface, while the one-direction and no-light groups showed adhesive failure between cement and the interface or cohesive failure in cement. Furthermore, KHN measurement results indicated that the hardness of cement in the multi-direction group was higher than that of the no-light group, and that the hardness of cement in the one-direction group decreased apically — due to reduction in light irradiation. These results indicated that the mechanical properties of Bistite II and its μTBS to root canal dentin were dependent upon light irradiation.

On the other hand, for the Panavia F system, there were no significant differences in μTBS and KHN between the curing methods. Fractures of all the groups were mixed failures at the interface and within the cement. These results might indicate that the μTBS of Panavia F to root canal dentin and its mechanical properties were not influenced by light irradiation. According to the manufacturer’s instructions, ED primer II contained a promoter for chemical polymerization of the cement, hence facilitating cement polymerization without light irradiation. Therefore, the μTBS and KHN results of Panavia F might be due to the effect of chemical polymerization promoted by ED primer II.

Self-etching primer application decreases the pH value of dentin surface even if the dentin surface is air-dried after treatment. A high concentration of uncured acidic monomers in ED primer II is present within the primed dentin surface. Adverse reactions have been reported to occur between chemically-activated resin composite and acidic resin monomer. This could be the reason for its low bond strength to dentin when chemically cured Panavia F was used to lute indirect restorations, although Panavia F cement contains aromatic sulphinate salts as ternary catalysts to ensure optimal chemical polymerization of the cement in an acidic environment. Carvalho et al. has demonstrated that, when photo-irradiating along the resin cement margin of indirect restorations, the ED primer of Panavia F of one-step, self-etching system induced
interfacial structures of mushroom-shaped blisters and resin globules. These structures were formed because of water permeation through the thinner ED primer layer from the dentinal tubules. On the other hand, these interfacial structures were absent from the interface of Bistite II, a two-step, selfetching system. This was because a 6-8 μm thick primer layer was present between the cement and dentin⁴⁹. The thick primer layer of Bistite II could protect against the diffusion of acidic monomer within the self-etched, primed dentin to the setting resin cement, which might lead to an adverse chemical reaction. They speculated that these factors were responsible for the high bond strength of Bistite II compared with Panavia F. In this study, the hardness of Bistite II was dependent upon the curing method, which was not so with Panavia F. The hardness values were reported to be concerned with the degree of conversion of the resin matrix within the limitation of single resin material⁴⁰. Bistite II and Panavia F had similar filler loads (77 and 78 % respectively). Therefore, the μTBS and KHN results in this study might be due to differences in resin monomer components and/or self- or photo-polymerization process of the cements bonded to primed dentin.

When resin is light cured, shrinkage occurs so rapidly such that a polymerization force develops — which can pull the resin off the dentin⁴¹,⁴². The amount of resin flow that occurs during polymerization is determined by cavity configuration. The configuration factor, or C-factor, is the ratio of bonded surface area to unbonded surface area⁴¹. Only the unbonded, free surface can flow during polymerization, thereby relieving stresses. As more and more of the total surface area is bonded to the adhering surface, the polymerization stress applied to these bonded surface areas increases. An even worse C-factor is produced when resin is applied to a long, narrow root canal. Chemically cured cements have been reported to show the least incidence of premature failures during the trimming process when using post cavities, as compared with dual-cure resin cement⁴³. This might be a reason why chemically cured cements help to reduce stresses at the adhesive interface because their slower setting allows for the flow of material to relieve polymerization stress⁴⁴. Further, it has been reported that the unconfined root specimens showed a lower incidence of premature failures than the post cavity specimens due to less shrinkage stress during polymerization⁴⁵. Matched halves and unconfined specimens used in this study were not prepared in a clinically realistic manner. However, they were hardly influenced by the C-factor nor the relief of shrinkage stress. Indeed, in this study, the specimens of both dual-cure resin cements in all curing methods showed no failures before assembly for testing. Additionally, bonding to root canal dentin is affected by the endodontic procedures performed prior to post cementation. In this study, we did not use any endodontic irrigant such as sodium hypochlorite or RC Prep. On this note, further research is required to determine the effects of C-factor and endodontic treatment on composite-dentin bond strength.

CONCLUSION

Within each curing strategy for both dual-cure resin cements, there were no significant differences between the μTBS values at the coronal third and mid third regions. The effect of curing method on bond strength and KHN was dependent on the material.

REFERENCES