Regional Bond Strength of Four Self-etching Primer/Adhesive Systems to Root Canal Dentin

Juthatip AKSORNMUANG1, Masatoshi NAKAJIMA1, Richard M. FOXTON2 and Junji TAGAMI1,3

1Cariology and Operative Dentistry, Department of Restorative Science, Tokyo Medical and Dental University, 1-5-45 Yushima, Bunkyo-ku, Tokyo 113-8549, Japan
2Department of Conservative Dentistry, Floor 25, Guy’s, King’s and St. Thomas’ Dental Institute, London Bridge, London, SE1-9RT, UK
3Center of Excellence Program for Frontier Research on Molecular Destruction and Reconstruction of Tooth and Bone, Tokyo Medical and Dental University, 1-5-45 Yushima, Bunkyo-ku, Tokyo 113-8549, Japan

Corresponding author, E-mail:juthatip.ope@tdm.ac.jp

Received January 26, 2005/Accepted April 26, 2005

The purpose of this study was to evaluate the regional bond strength of a dual-cure resin core material to root canal dentin using four self-etching primer/adhesive systems. Post spaces were prepared in extracted premolars, and their root canal dentin was treated with one of the following self-etching primer/adhesive systems: ED Primer II and Clearfil Photobond, photo-cure/dual-cure systems of Clearfil Liner Bond 2V, or Clearfil SE Bond. Post spaces were filled with the dual-cure resin core material, and microtensile bond strength (μTBS) at the coronal and apical regions was measured after 24-hour storage. There were no regional differences in μTBS of the photo-cure and dual-cure systems of Clearfil Liner Bond 2V, while μTBS at the coronal region of Photobond and SE Bond groups were higher than those at the apical region. At the apical region, photo-cured Clearfil Liner Bond 2V exhibited significantly higher bond strength than those of the other systems.

Key words: Microtensile bond strength, Root canal dentin, Dual-cure resin core material

INTRODUCTION

Recently, it has become increasingly popular to utilize fiber-reinforced composite posts in combination with adhesive resin cement to restore endodontically treated teeth. The major advantage of fiber posts is their similar elastic modulus to dentin, thereby producing a stress field that is similar to that of natural teeth and resulting in reduced root fractures — which is in sharp contrast to metal posts that exhibit high stress concentration at the post-dentin interface. However, in a flared canal, which might result from various injury, trauma, pulpal pathosis, iatrogenic misadventure, or idiopathic causes, there may exist an excessively thick layer of resin cement in the coronal region of post spaces which may not be strong enough to resist occlusal loading. To overcome this problem, a dual-cure composite resin core material has been introduced as a luting medium because it is stronger than resin cement and has a modulus of elasticity close to dentin and fiber posts.

Laboratory and clinical studies have found that failures of fiber post and core restorations often occurred following decementation between the fiber post-resin and/or resin-root dentin interfaces. Therefore, good adhesion at these interfaces is an important factor for a successful restoration. In our previous studies, we evaluated bonding of dual-cure composite resin core materials to non-translucent glass and quartz fiber posts, as well as to translucent quartz fiber posts. It was found that applying a silane coupling bonding agent to the post surface was effective in achieving optimal bond strength between dual-cure composite resin and silica-based fiber posts. Various dentin bonding systems have been developed to improve bonding quality and simplify bonding procedures. Currently, self-etching primer/adhesive systems are widely used for bonding to dentin substrate because of higher dentin bond strength and reduced technique sensitivity. These systems simultaneously demineralize and infiltrate resin monomer into dentin. The two-pronged action helps to reduce insufficiently resin-infiltrated dentin matrix, which may render the resin-dentin interface vulnerable to premature degradation under clinical conditions. However, there is limited published research on the adhesion of dual-cure composite resin to root canal dentin using contemporary self-etching primer/adhesive systems, although some studies have investigated the adhesion of resin cement to root canal dentin.

Most clinicians generally use dual-cure adhesives for bonding to root canal dentin because of their ability to self-polymerize in the absence of light in deeper regions of the post cavity. However, an adverse chemical reaction was reported to occur between chemically activated resin composite and acidic resin monomers. Self-etching primer contains acidic monomer, and consequently a high concentra-
tion of uncured acidic monomers would be present within the primed dentin surface. Therefore, the compatibility of dual-cure adhesive with self-curing mode and self-etch primed dentin is questionable. On the other hand, very few studies have evaluated the bonding efficacy of self-etching primer/photo-cure adhesive to root canal dentin. Photo-cure adhesive resin is uncommonly used for bonding to root canal dentin because of the major concern in inadequate light energy to cure the bonding resin at the apical region. In the present study, we hypothesized that the current photo-cure self-etching primer/adhesive systems could be used for bonding to root canal dentin substrate without creating any difference in regional bond strength.

Therefore, the purpose of this study was to evaluate the regional bond strength of a dual-cure composite resin core material to root canal dentin using four self-etching primer/adhesive systems including both photo- and dual-cure type adhesives.

**MATERIALS AND METHODS**

**Preparation of bonded specimens**

Twelve single-rooted premolar human teeth, recently extracted from adolescents for orthodontic reasons and stored frozen, were decoronated at the cementoenamel junction using a low-speed diamond saw (Isomet, Buehler, Lake Bluff, IL, USA). Pulpal tissue was removed using endodontic files, and post spaces prepared using Gates-Glidden drills (Matsutani Seisakusho Co. Ltd., Takanezawa, Japan) and FiberKor drills (Pentron Corporation, Wallingford, CT, USA) in a low-speed handpiece under copious water cooling to a depth of 8 mm and a diameter of 1.5 mm. After preparation, the post spaces were rinsed with distilled water and dried with paper points. Prior to the bonding procedure, external surfaces of the roots were built up with Clearfil DC Core composite resin (Kuraray Medical Inc, Tokyo, Japan) to make grips for testing and to prevent the effect of external curing light — which can pass through the thin portion of dentin wall to the bonding agent during irradiation. The materials used in this study and their chemical compositions are presented in Table 1.

The roots were randomly divided into four groups, and their root canal dentin surfaces treated with one of the following self-etching primers/adhesives: 1) dual-cure system ED Primer II/Clearfil Photobond (PB); 2) photo-cure system Clearfil Liner Bond 2V Primer/Bond A (PLB); 3) dual-cure system Clearfil Liner Bond 2V Primer/Bond A+B (DLB); 4) photo-cure system Clearfil SE Bond Primer/Bond

<table>
<thead>
<tr>
<th>Table 1 Materials used in this study</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Material</strong></td>
</tr>
</tbody>
</table>
| ED Primer II | Kuraray Medical Inc., Tokyo, Japan | Liquid A: MDP, HEMA, 5-NMSA, water, accelerator  
Liquid B: 5-NMSA, water, chemical initiator |
| Clearfil Photobond | Kuraray Medical Inc., Tokyo, Japan | Catalyst: MDP, HEMA, dimethacrylates, chemical initiator/photoinitiator  
Universal: Accelerator, ethanol |
| Clearfil Liner Bond 2V | Kuraray Medical Inc., Tokyo, Japan | Primer  
Liquid A: MDP, HEMA, water, photoinitiator, accelerator  
Liquid B: HEMA, water, chemical initiator  
Bond  
Liquid A: MDP, HEMA, dimethacrylates, microfiller, photoinitiator, accelerator  
Liquid B: HEMA, dimethacrylates, microfiller, chemical initiator |
| Clearfil SE Bond | Kuraray Medical Inc., Tokyo, Japan | Primer: MDP, HEMA, water, hydrophilic dimethacrylates, photoinitiator, accelerator  
Bond: MDP, HEMA, hydrophobic dimethacrylates, microfiller, photoinitiator, accelerator |
| Clearfil DC Core | Kuraray Medical Inc., Tokyo, Japan | Catalyst: Bis-GMA, TEGDMA, fillers, chemical initiator/photoinitiator  
Universal: Bis-GMA, TEGDMA, fillers, accelerator |

MDP: 10-methacryloxydecyl dihydrogen phosphate;  
HEMA: 2-hydroxyethyl methacrylate;  
5-NMSA: N-Methacryloyl 5-aminosalicylic acid;  
Bis-GMA: bisphenol-A-glycidyldimethacrylate;  
TEGDMA: triethylene glycol dimethacrylate
Bonding procedures were performed according to manufacturers' instructions presented in Table 2. Post spaces were filled with a dual-cure composite resin core material (Clearfil DC Core, Kuraray Medical Inc., Tokyo, Japan). The coronal surface of the root was covered with a plastic strip and pressed gently with a glass slide to squeeze out any excess resin. Light exposure was performed for 60 seconds by placing the tip of the light source (Optilux 500, Demetron, Danbury, USA) at the top of the cavity. Prior to each bonding procedure, power density of light source was checked with a digital radiometer (Jetlite light tester, J. Morita, Mason Irvine, CA, USA) to ensure that it ranged between 640 and 660 mW/cm². All specimens were then stored in water for 24 hours at 37°C.

**Bond strength testing**

After 24-hour storage, each bonded specimen was attached to the arm of a low-speed diamond saw (Isomet, Buehler, Lake Bluff, IL, USA) whereby eight slabs were serially cut perpendicular to the bonded interface under water cooling. Each slab was then transversely sectioned at the middle part of the post cavity into approximately 0.6 x 0.6 mm thick beams. The cross-sectional area of each beam was measured using digital calipers (Mitutoyo CD15, Mitutoyo Co., Kawasaki, Japan). One of the two interfaces of each beam was randomly selected for testing. The ends of the beam and the remaining interface were glued onto a testing device in a table-top testing machine (EZ Test, Shimadzu Co., Kyoto, Japan) using cyanoacrylate glue (Zapit, DVA, Anaheim, CA, USA) and subjected to a tensile force at a cross-head speed of 1 mm/min (Fig. 1). Microtensile bond strength data of four coronal beams were considered to represent the coronal portion of the post space corresponding to the coronal third of root canal, while data of four apical beams were considered to represent the apical region corresponding to the middle third of root canal.

**Fracture analysis**

After specimens had fractured, both the resin and dentin sides of the fractured beams were mounted on brass tablets and gold sputter-coated. Fracture modes were then observed using a scanning electron microscope (JSM-5310, JEOL, Tokyo, Japan), and classified as one of the following: adhesive failure, mixed adhesive/cohesive failure at resin/dentin interface, cohesive failure within resin, and cohesive failure within dentin.

**Statistical analysis**

The μTBS data were analyzed using two-way ANOVA to test how bonding agent and dentin region affected bond strength. In addition, the interaction between these two factors was tested. Dunnett’s T3 was used as a post-hoc test for multiple comparisons (α = 0.05), since Levene’s test indicated significant non-homogeneity among the variances. Failure mode data were analyzed using the chi-squared test (α = 0.05).
Table 3 Microtensile bond strength (MPa) of dual-cure resin core material to root canal dentin

<table>
<thead>
<tr>
<th></th>
<th>PB</th>
<th>PLB</th>
<th>DLB</th>
<th>SE</th>
</tr>
</thead>
<tbody>
<tr>
<td>Coronal</td>
<td>33.3 (4.2)^A</td>
<td>51.0 (11.8)^B</td>
<td>43.7 (11.7)^A,B</td>
<td>52.2 (15.7)^B</td>
</tr>
<tr>
<td></td>
<td>p&lt;0.05</td>
<td>NS</td>
<td>NS</td>
<td>p&lt;0.05</td>
</tr>
<tr>
<td>Apical</td>
<td>18.4 (5.7)^a</td>
<td>46.4 (7.1)^b</td>
<td>31.2 (7.4)^c</td>
<td>26.7 (12.1)^a,c</td>
</tr>
</tbody>
</table>

n=12 for each group.
All values are given as mean (SD). NS and the same superscripts indicate no significant differences.

RESULTS

Regional microtensile bond strengths of dual-cure resin core material to root canal dentin in each experimental group are summarized in Table 3. Two-way ANOVA revealed that bond strength was influenced by both bonding agent (p<0.001) and dentin region (p<0.001). An interaction was also found between these two factors (p=0.007).

There were no regional differences in the μTBS of photo-cure and dual-cure systems of Clearfil Liner Bond 2V (p>0.05), while μTBS at the coronal region of Photobond and SE Bond groups were significantly higher than those at the apical region (p<0.05). At the coronal region, the μTBS of photo-cure systems – PLB and SE groups – were significantly higher than that of the dual-cure PB group, and were higher than that of the dual-cure DLB group although the difference was not significant. At the apical region, the μTBS of PLB group was significantly higher than those of DLB, SE, and PB groups (p<0.05).

Chi-squared analysis revealed that the failure modes for each bonding agent were not significantly different between the coronal and apical regions. Therefore, the data were pooled together and the proportional frequencies of failure mode in each experimental group were compared using the chi-squared test. Fig. 2 presents the percentage of fracture modes for each bonding system. Most specimens fractured at the interface between bonding agent and dentin, which was classified as adhesive failure (Fig. 3). Some specimens in PB group failed cohesively in the bonding resin (Fig. 4). However,

![Fig. 2 Percentage of failure mode for each bonding system. There were no significant differences in failure mode among the four experimental groups.](image1)

![Fig. 3 SEM photographs of adhesive failure surfaces in SE group. Exposed dentinal tubules were observed on the dentin side (A), whereas resin tags were pulled out and observed on the resin side (B).](image2)

![Fig. 4 SEM photograph of a cohesive failure surface within adhesive resin in PB group.](image3)
there were no significant differences in the proportional frequencies of failure modes among the four experimental groups.

DISCUSSION

It has been found that adhesive systems, when used with composite resin material to cement endodontic posts, benefit post retention and fracture resistance of teeth. Many factors can influence the success of a restored, endodontically treated tooth. Adhesion at the post-resin and resin-dentin interfaces is one factor, and in fact found to be a common cause of failure. Another factor is attributed to dentin mechanical properties and structure—which are found to be dependent on dentin region. It was reported that tubule density in the coronal region of root dentin is higher than that of apical region, and that the diameter of tubules decreases in an apical direction. However, Liu et al. found that dentin location did not affect the microtensile strength of bovine root dentin. Additionally, it was revealed that bond strength to root canal dentin was neither influenced by dentin depth nor tubule density when a self-etching adhesive system was used. We therefore speculated that in the present study, in which only self-etching adhesive systems were used, resin/dentin bond strength might not be affected by the density of dentinal tubules. Instead, it might be the mechanical properties of the bonding agent—which could be enhanced by adequate polymerization—that affected the bond strength to root canal dentin.

Results of the present study demonstrated that photo-cure Clearfil Liner Bond 2V system provided a higher bond strength than that of dual-cure Clearfil Liner Bond 2V system at both the coronal and apical regions, and statistically significant difference was detected at the apical region. It was thought that after irradiation, the mechanical properties of photo-cure adhesive resin might have been greater than those of dual-cure adhesive resin—whereby this finding is in agreement with previous results obtained by Foxton et al. On the other hand, for photo-cure SE Bond, although the TBS obtained from the coronal region was comparable to that of photo-cure Clearfil Liner Bond 2V, the bond strength significantly decreased and was lower than that of PLB group at the apical region. This could be due to the different irradiation times for Clearfil Liner Bond 2V and SE Bond (Table 2). The manufacturer of Clearfil Liner Bond 2V adhesive recommended 20 seconds for photo-cure, but only 10 seconds was recommended for SE Bond adhesive. The manufacturer claimed that if duration of light irradiation was less than the recommended light irradiation time, it would result in decreased bond strength. In this study, however, it was more likely to be due to limited light irradiation from the orifice of post cavity. As a result, adhesives applied to root canal wall were light-irradiated from the lateral side. In this vein, the longer irradiation time of 20 seconds prescribed for Clearfil Liner Bond 2V could have helped light energy to access more areas and more thoroughly in the post cavity, enabling it to achieve higher bond strength at the apical region. Moreover, light accessibility through the root canal is limited. In clinical situations, remaining coronal tooth structure or adjacent teeth might prevent light tip from being placed closely to the root orifice, thereby resulting in inadequate polymerization of the adhesive. Additionally, the different curing systems in self-etching primers—Clearfil Liner Bond 2V and SE Bond—might be another factor that affected bond strength. Clearfil Liner Bond 2V is a two-bottle, dual-cure type of self-etching primer, while SE Bond system is a single-bottle, photo-cure type of self-etching primer. Where there is an absence of light energy, a dual-cure self-etching primer might improve the polymerization of resin monomer at the interface as compared to a photo-cure primer. This might explain why the bond strength at the apical region of PLB group was higher than that of SE group, and also not significantly different from the bond strength at the coronal region.

On the other hand, in PB group, though ED Primer is a dual-cure type of self-etching primer, mean TBS was lower than the other groups at both regions, and TBS at the apical region was significantly lower than that at the coronal region. This was probably due to ethanol, included in the PB adhesive resin, not being able to completely evaporate at the apical portion and becoming entrapped in the bonding layer. An absence of filler content in the bonding resin could be another factor that led to a lower bond strength in PB group compared with LB and SE groups, which contain silanated colloidal silica as fillers. Moreover, PB adhesive resin at the apical region was probably polymerized mostly by chemical activation because of limited light accessibility and short irradiation time (10 seconds). An adverse chemical reaction between the acidic monomer of ED Primer II and chemically activated Photobond adhesive resin might have occurred, although PB adhesive system contains aromatic sulfinate salts as a ternary catalyst to ensure optimal chemical polymerization of bonding agent in an acidic environment. As a result, cohesive failure within the adhesive resin of some fractured beams in PB group might have occurred because the adhesive resin of PB might not have been adequately polymerized.

In this experiment, post spaces were prepared in root canals which had not been endodontically treated or treated with NaOCl in order not to alter the biomechanical behavior of root dentin. This is because it has been reported that bond strength to root canal dentin is affected by chemical irritant, the
extent of which is dependent on the type of bonding agent used [10,11,12]. Moreover, eugenol — which could remain in drilled post space — was found to influence bond strength too [13]. To curb the influence of these variables, root canals in this experiment were therefore not obturated and irrigated only with distilled water after drilling.

It can be concluded from this study that bonding strategy affected the adhesion between dual-cure composite resin and root canal dentin. Differences in regional bond strength could also be attributed to the type of bonding system used, whereby photo-cure adhesive resin seemed to be effective on root canal dentin. Nevertheless, there should be further studies concerning the effect of irradiation time on adhesion to root canal dentin using photo-cure adhesive resin.

ACKNOWLEDGEMENTS
The authors gratefully acknowledge the financial support received from Center of Excellence Program for Frontier Research on Molecular Destruction and Reconstruction of Tooth and Bone, Tokyo Medical and Dental University.

REFERENCES

26) Liu J, Hattori M, Hasegawa K, Yoshinari M, Kawada...


