Effects of photocuring strategy on bonding of dual-cure one-step self-etch adhesive to root canal dentin

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This study evaluated the effects of light power density and light exposure time on regional bond strength of Clearfil DC Bond to root canal dentin. Post spaces were prepared in extracted premolars. Root canal dentin was treated with a dual-cure bonding system, Clearfil DC Bond, and light-cured for 10, 20, or 30 seconds using two halogen light curing units: Optilux 501 (830 mW/cm²) and Hyperlight (1350 mW/cm²). Following which, all post spaces were filled with a dual-cure resin composite. After 24-hour storage, microtensile bond strengths (µTBS) at the coronal and apical regions were measured. At the coronal region, µTBS values were similar among all the experimental groups. At the apical region, bond strength improved when the curing time was extended to 30 seconds with Optilux 501, and likewise with Hyperlight when curing time was extended to 20 or 30 seconds. In addition, significant differences in µTBS between the coronal and apical regions disappeared with prolonged curing times.

Key words: Microtensile bond strength, Dual-cure self-etch adhesive, Root canal dentin

INTRODUCTION

Endodontically treated teeth often require posts and cores to provide retention for the final restoration. Presently, esthetic non-metal fiber posts are commonly used for radicular restoration because their moduli of elasticity are comparable to dentin, thereby producing a stress field similar to that of a natural tooth and resulting in reduced root fracture incidences¹⁻⁵. Resin cement or dual-cure resin core material in combination with an adhesive system are generally recommended for bonding fiber posts to the root canal wall⁶⁻⁹. It is noteworthy that in post spaces, light penetration is limited in the deeper region than at the upper region. Therefore, some manufacturers have recently developed a dual-cure version of the one-step self-etch adhesive to be used with resin core materials. Twofold benefits are hence reaped with these dual-cure one-step self-etch adhesives: (1) simplified application procedures for fiber post luting and core build-up; and (2) polymerization by self-cure mode in the deeper region when resin core material was filled into the post space.

One-step self-etch adhesives contain acidic resin monomers, organic solvent, and water. A major concern is the incompatibility between the acidic monomers of one-step self-etch adhesives and the self- or dual-cure composite resins. Many researchers have reported on adverse acid-base reactions between the acidic resin monomers and the tertiary amines used in the self-cure initiator systems, thereby causing the amines to lose their effectiveness as reducing agents and resulting in poor polymerization¹⁰⁻¹¹. In other words, this incompatibility would pose a barrier to good bonding. In the deeper region of post spaces, this incompatibility would cause the uncured acidic resin monomers of one-step self-etch adhesives to adversely affect the self-polymerization of dual-cure resin core materials. Therefore, conversely, if the acidic adhesive were to be completely cured, the adverse impact arising from the incompatibility issue and the self-polymerization of resin core materials would be reduced. For this reason, it is important to irradiate dual-cure adhesives with sufficient light energy — through the post space — to minimize the adverse effects of uncured acidic resin monomers of one-step self-etch adhesives.

Besides, the mechanical properties of dual-cure adhesives were also found to be affected by curing strategies. Bond strength to the tooth substrate (enamel or dentin) depends on the mechanical properties of the adhesive resin, which means that dual-cure adhesives should be exposed to light — versus chemical activation alone — to ensure good bond strength. However, within the post space, high attenuation of light passing through the canal may jeopardize bonding quality — especially at the
deeper region. To circumvent this problem, previous studies have recommended using a high-intensity light curing unit to enhance the curing efficiency of adhesive resins, especially for deep cavities or under tooth-colored restorations. Likewise, some studies have suggested extending the exposure time to light- or dual-cure adhesive resins to ensure optimal polymerization in the deep region of post spaces. In light of these suggestions and recommendations, it can be presumed that using a high-intensity light curing unit and/or prolonging the light exposure time to adhesive resins may improve the adhesion of dual-cure one-step self-etch adhesives to root canal dentin.

This study was conducted to evaluate the effects of light curing unit (which provided different power densities) and exposure time on the regional bond strength of a dual-cure one-step self-etch adhesive to root canal dentin. The null hypothesis was that the light curing unit, exposure time, and dentin region did not affect bond strength.

MATERIALS AND METHODS

Preparation of bonded specimens

Eighteen single-root human premolar 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 Corp., 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 a composite resin (Clearfil DC Core, Kuraray Medical Inc., Tokyo, Japan) to make grips for testing and to prevent the effect of external curing light — which could 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.

A dual-cure one-step self-etch adhesive system, Clearfil DC Bond (Kuraray Medical Inc., Tokyo, Japan), was used according to the manufacturer’s instructions for bonding to root canal dentin. Equal amounts of liquids A and B were mixed for five seconds, and the mixture was applied to root canal dentin in the post space for 20 seconds using a microbrush disposable applicator (Pentron Clinical Technologies, LLC, USA). The adhesive was dried with a high-pressure airflow for 5 seconds. Excess adhesive resin at the bottom of the canal was removed using a paper point, and the adhesive was dried again with high-pressure airflow for a further 5 seconds.

The adhesives were then light-cured for 10, 20, or 30 seconds using two quartz-tungsten-halogen light curing units, Optilux 501 (OP) (Demetron, Danbury, USA) and Hyperlightel (HL) (Kuraray Medical Inc., Tokyo, Japan). Their power densities, measured with a digital radiometer (Jetlite light tester, J. Morita, Mason Irvine, CA, USA), were 830 and 1350 mW/cm² respectively. The teeth were divided into six experimental groups (three teeth for each group) according to the light curing unit and exposure time: OP 10s, OP 20s, OP 30s, HL 10s, HL 20s, and HL 30s. All post spaces were filled with a

Table 1 Materials used in this study

<table>
<thead>
<tr>
<th>Materials</th>
<th>Manufacturer</th>
<th>Composition</th>
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<tbody>
<tr>
<td>Clearfil DC Bond</td>
<td>Kuraray Medical Inc., Japan</td>
<td>A liquid: MDP, hydrophilic dimethacrylates, HEMA, photoinitiator, chemical catalyst nanofiller</td>
</tr>
<tr>
<td></td>
<td></td>
<td>B liquid: Water, ethanol, chemical catalyst</td>
</tr>
<tr>
<td>DC Core Automix</td>
<td>Kuraray Medical Inc., Japan</td>
<td>Catalyst: Bis-GMA, TEGDMA, silanized glass fillers, silica microfillers, chemical/photoinitiator Universal: TEGDMA, methacrylate monomers, silanized glass fillers, silica microfillers, chemical/photoinitiator</td>
</tr>
</tbody>
</table>

MDP: 10-methacryloxydecyl dihydrogen phosphate; HEMA: 2-hydroxyethyl methacrylate; Bis-GMA: bisphenol-Aglycidylidimethacrylate; TEGDMA: triethylene glycol dimethacrylate
dual-cure resin composite core material (Clearfil DC Core Automix, 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 using Optilux 501 by placing the light source tip at the top of the cavity. 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 space into approximately 0.6 × 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 cyanacrylate glue (Zapit, DVA, Anaheim, CA, USA) and subjected to a tensile force at a crosshead speed of 1 mm/min (Fig. 1).  

**Fracture analysis**

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

**Statistical analysis**

The microtensile bond strength (µTBS) values of four coronal beams were considered to represent the coronal portion of the post space corresponding to the coronal third of root canal, while those of four apical beams were considered to represent the apical region corresponding to the middle third of root canal. The specimens that failed during cutting were excluded from average bond strength calculation, but the numbers of intact tested specimens were reported.

The µTBS data were analyzed using three-way ANOVA to test the effects of light curing unit, exposure time, and dentin region on bond strength. In addition, the interaction between these three factors was tested. Tukey’s HSD was used as a post hoc test to compare 12 groups of means attributable to the presence of interactions between tested variables. The proportional frequencies of failure modes in each experimental group were compared using the chi-square test. All statistical testing was performed at a 95% level of confidence.

**RESULTS**

Table 2 shows the regional microtensile bond strengths of dual-cure one-step self-etch adhesive to root canal dentin in each experimental group. Three-way ANOVA revealed that differences in light curing unit (p=0.005), exposure time (p<0.0001), and dentin region (p<0.0001) had a significant effect on µTBS. Interactions between the light curing unit and

![Fig. 1](image_url) Schematic illustration of specimen preparation method for microtensile bond strength test.
Table 2 \( \mu TBS \) values of DC Core Automix to root canal dentin treated with Clearfil DC Bond (MPa)

<table>
<thead>
<tr>
<th></th>
<th>Optilux 501</th>
<th>Hyperlighted</th>
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</thead>
<tbody>
<tr>
<td></td>
<td>10 s</td>
<td>20 s</td>
</tr>
<tr>
<td>Coronal</td>
<td>38.9(13.7)(^{AB})</td>
<td>38.8(12.5)(^{AB})</td>
</tr>
<tr>
<td></td>
<td>(11/12)</td>
<td>(12/12)</td>
</tr>
<tr>
<td>p&lt;0.05</td>
<td>p&lt;0.05</td>
<td>NS</td>
</tr>
<tr>
<td>Apical</td>
<td>11.1(2.1)(^{a})</td>
<td>20.4(8.3)(^{ab})</td>
</tr>
<tr>
<td></td>
<td>(8/12)</td>
<td>(12/12)</td>
</tr>
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</table>

Fig. 2 Percentages of failure modes for each light curing unit and exposure time. There were no significant differences in failure mode among the experimental groups except for HL 10s group.

exposure time (p=0.011), and between the light curing unit and dentin region (p=0.048) were also present. Although post hoc comparison was performed to compare 12 groups of means, significant differences in the data were only partially indicated, as shown in Table 2, to present the differences between curing methods at each dentin region, and between regions for each curing method.

At the coronal region, the HL 20s group achieved the highest average bond strength. However, there were no significant differences among the experimental groups (p>0.05) except between the HL 10s and HL 20s groups (p=0.023). At the apical region, high bond strengths were obtained in OP 30s, HL 20s, and HL 30s groups, and that no significant differences were found among these three groups (p>0.05). The lowest bond strength occurred when the adhesive resin was cured with Optilux 501 for only 10 seconds. As for the presence of differences between the coronal and apical regions, no significant differences were observed when the adhesive resin was cured with Optilux 501 for 30 seconds, or with Hyperlighted for 20 and 30 seconds (p>0.05).

Figure 2 shows the fracture mode percentages of the debonded specimens. There were no cohesive failures within the dentin substrate. Chi-square testing revealed no significant differences in failure mode among the experimental groups (p>0.05) except for HL 10s group, of which approximately 60% of the specimens failed at the interface between the dentin and the adhesive resin.

Figure 3 shows the representative SEM micrographs of the fracture surfaces. Some of the deboned specimens revealed abundant blisters at the adhesive resin surface and composite surface as shown in Fig. 4, which depicts fracture surfaces at the apical region of the OP 10s group.
**Adhesive failure**

**Cohesive failure in resin**

**Mixed adhesive failure/cohesive failure in resin**

Fig. 3  Representative SEM micrographs of the fractured surfaces of both dentin and adhesive resin in each failure mode. A=Adhesive, D=Dentin.
DISCUSSION

For dual-cure adhesive resins, it is of paramount importance that the resin surface is irradiated with sufficient light energy to ensure a high degree of polymerization, as the latter produces good adhesion. Light energy is a unit that encompasses two factors: intensity of the light curing unit, also referred to as power density, and the duration of light application over a given area. Therefore, an increase in either the intensity or light exposure time would produce a higher energy for the polymerization process. In this study, the effects of these two factors were investigated. Although several studies have investigated the effects of energy density and exposure time on the degree of cure, depth of cure, mechanical properties of resin composites, and dentin bond strength, most of these studies employed flat resin surfaces with an intimate curing distance. This study was different in that testing was carried out in deep and narrow root canals. Further, the adhesive surface was not perpendicularly exposed to the light source, which meant that light energy tended to decrease with distance.

It was reported that dentin mechanical properties and structure are dependent on the dentin region. Tubule density in the coronal region of root dentin was found to be higher than that in the apical region, and the diameter of the tubules decreases in an apical direction. However, Liu et al. reported that dentin region did not affect the microtensile strength of bovine root dentin. Additionally, it was found that bond strength to root canal dentin was neither influenced by dentin depth nor tubule density when a self-etch adhesive system was used. In the present study, it was therefore speculated that if only self-etch adhesive systems were used, resin-dentin bond strength would not be influenced by the density of the dentinal tubules. This also meant that the most important factor that would affect the bond strength to root canal dentin would be the mechanical properties of the bonding agent, which could be enhanced by adequate polymerization from the light source.

At the coronal region, no significant differences in bond strength values were found among the experimental groups. At this region, there was probably sufficient light energy for adequate curing of the adhesive resin although the curing time was only 10 seconds according to manufacturer’s instructions. In a study by Ye et al. which investigated the polymerization conversion of dental adhesives including one-step self-etch adhesive, it was found that each adhesive required a specific curing time to reach the conversion plateau — and thereafter the conversion rate was fairly constant. According to this principle, prolonging the light exposure time to 20 or 30 seconds would therefore have no positive effect on bond strength. However, effective curing with only 10 seconds of exposure time might be limitedly applicable to the upper half of the post space because significant reduction in bond strength was observed at the apical region.

At the apical region, the bond strength of the adhesive cured by OP for 30 seconds was nearly three times higher than that after 10 seconds’ curing. With HL, μTBS was doubled when light exposure time was extended from 10 to 20 seconds. In addition, the regional differences in bond strength disappeared with prolonged curing time, whereas significant reductions in bond strength at the apical region for OP 10s, OP 20s, and HL 10s groups emerged. According to the Beer – Lambert law, light is attenuated with increasing cross-sectional distance from the irradiated surface as a result of
light absorption. Based on this law, light energy in the deep region of the root canal would therefore be limited. This meant that the light curing time recommended by the manufacturer might not be enough for optimal curing of the adhesive resin in the deep region of the post space. Moreover, the adhesive surface at root canal dentin was parallel to the light direction — which meant that it was not perpendicularly exposed to the light source as with a flat dentin surface. These reasons might thus account for the decrease in bond strength at the apical region for OP 10s, OP 20s, and HL 10s groups.

From an alternative perspective, higher light energy by virtue of a prolonged curing time might be enough to initiate the polymerization process at the deeper region. It should also be mentioned that light was directly transmitted to the adhesive surface along root canal dentin without passing through any restorative materials. This meant that adhesive resin even in the deep region was probably wholly exposed to the curing light, thereby resulting in improved bond strengths for OP 30s, HL 20s, and HL 30s groups.

Some specimens failed prior to testing, especially at the apical region of the OP 10s group which exhibited the lowest average bond strength. However, in the other groups, none or only one or two beams failed during the cutting phase. The problem with microtensile technique is that the small-sized specimens may fail during sectioning if the bond strength is too low. In the present study, pre-testing failed specimens were excluded from average bond strength calculation, but the numbers of intact tested beams were reported. For the pre-testing failed specimens, their bond strengths were not treated as zero bond strength because it was not true zero bond strength. If zero bond strength were reported for these pre-testing failed specimens, it would adversely lower the average bond strength and widen the standard deviation of some groups of data. Consequently, this might have an undesirable impact on statistical test results.

On the failures that occurred after microtensile testing, three types of failure were found in this study as shown in Fig. 3: adhesive failure at the resin-dentin interface, cohesive failure in the resin including failure within the adhesive or at the interface between the adhesive and resin composite, or mixed failure of both types. In the present study, failure within the adhesive resin was probably a result of the undesirable properties of one-step self-etch adhesives. In previous studies conducted in the same manner as this study but using two-step self-etch adhesives, only a small number of specimens failed cohesively within the adhesive resin. For the one-step self-etch adhesive used in this study, the liquid B component of Clearfil DC Bond component contained water, and which was mixed with liquid A component and then applied within one single step. As for two-step self-etch adhesives, the hydrophobic resin is generally separated from the first-step solvent-included primer.

It must be highlighted that any water which remains within the adhesive layer after evaporation may weaken the mechanical properties of the resin. Additionally, water trapped at the surface of the adhesive resin might interfere with the bonding between the adhesive and resin composite. Further on absorbed water, Tay et al. found that reticular mode of nanoleakage could occur due to incomplete water removal from the adhesive layer. To date, studies regarding the bond strength of single-step adhesives have demonstrated significantly inferior μTBS of resin-dentin bonds after long-term water storage, and which are characterized by a typical deterioration at the adhesive-composite interface.

Indeed, as shown in Fig. 4, honeycomb structures formed by numerous blisters were seen all over the fractured surfaces of both the adhesive and resin composite. Similarly, these structures were observed on fractured microtensile specimens bonded with an all-in-one adhesive in a recent study. In light of these findings, air blowing of the adhesive during bonding procedures should be performed carefully to ensure absolute evaporation of the solvent within the adhesive. However, in practical dentistry, this step may be difficult to achieve at the deep region of post spaces.

This study evaluated the initial microtensile bond strength achieved with a dual-cure one-step self-etch adhesive. It demonstrated that the durability of one-step self-etch adhesives posed a problem to bonding because the hydrophilicity of this adhesive system could weaken long-term bond strength. This effect might be minimal in the post space due to crown coverage. Nonetheless, further studies should be conducted to investigate the bonding durability of this adhesive system with radicular dentin.

CONCLUSIONS

Within the limitations of this study, it was concluded that μTBS was affected by both light power density and curing time — especially at the apical region of the post space. The optimal curing time for dual-cure one-step self-etch adhesive applied to 8-mm-deep root canal dentin surface was 30 seconds with Optilux 501 and 20 seconds with the high-intensity curing unit, Hyperlightel.

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