The effect of zirconia thickness and curing time on shear bond strength of dual-cure resin cement

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This study evaluated how zirconia thickness affects shear bond strength (SBS) between zirconia and dual-cure resin cement. Eighty specimens (40 blocks of 1 mm thickness zirconia and 20 of each 1.5 and 2 mm) were divided into 8 groups according to zirconia thickness and light-curing time. Group A, B, C, and D were light-polymerized during 20 s and group E, F, G, and H were light-cured during 40 s. Self-adhesive dual-cure resin cement was placed onto the zirconia surfaces and then light-polymerized. The mean SBS of the 40 s curing time group was statistically higher than that of the 20 s curing time group. However, curing time did not affect the SBS significantly in the 2 mm thickness groups. The mean SBS of the 1.5 and 2 mm groups were statistically lower than those 1 mm groups. Therefore, zirconia thickness significantly affected the SBS of self-adhesive dual-cure resin cement.

Keywords: Resin cements, Zirconium oxide, Shear strength, Curing lights, Zirconia thickness

INTRODUCTION

As all-ceramic restorations have the definite advantage of superior esthetic appearance over metal-ceramic restorations, they have been frequently used in recent years. All-ceramic restorations can be fabricated in many systems using different ceramic materials such as silica-based ceramics, aluminum-oxide ceramics and zirconium-oxide ceramics¹. In the past, silica-based or aluminum-oxide ceramics were popular, but Zirconium-oxide ceramics, using CAD-CAM, has been a great alternative to other ceramics with conventional techniques, due to the ability to block out the shade of metal substructure and discolored abutment tooth². Zirconium-oxide ceramics also have demonstrated flexural strength of 900 to 1,200 MPa and fracture toughness of 9 to 10 MPa·m½, these readings are almost double those of aluminum-oxide ceramics and nearly three times more than those of silica-based ceramics³, so it has been used increasingly for the framework of anterior multi-unit fixed partial dentures. Several studies have indicated that zirconia-based restorations may not require adhesive cementation⁴⁻⁵. Although conventional cements like glass ionomer have been successfully used⁶, a reliable resin bond to zirconia that has sufficient durability needs to be achieved in some clinical situations, such as partial veneers and short abutments⁷. In general, dual-cure resin cements show greater mechanical properties, but under chemical polymerization conditions, without light polymerization, the merit of the dual-cure cements is lessened considerably⁸. In addition, increasing the thickness of porcelains provides insufficient light transmission to the underlying resin cements and produces a significant decrease in the mechanical properties of light-cured resin cements⁸⁻⁹. Because zirconia is a semitranslucent ceramic material and less translucent than glass ceramics, its thickness⁹ and shade can affect the amount of light transmission and polymerizing efficiency of light-activated resin luting cements¹⁰⁻¹¹. Dual-polymerizing luting resins combine the desirable properties of chemical and light-polymerizing materials, providing an adequate degree of monomer conversion (DC) beneath various restorations. With dual polymerization, the portion of luting resin that initially received insufficient irradiance continues to polymerize after light exposure by means of a delayed chemical reaction. If this chemical polymerization cannot compensate sufficiently for the reduced photopolymerization caused by reduced light irradiance, insufficient hardening and a lower level of conversion of a dual-polymerizing resin cement can lead to the early failure of adhesively retained zirconia restorations. Of particular interest is how ceramic disc thickness affects the penetration of light during curing. Surface hardness of dual-cured cements is decreased with light-curing through ceramic discs¹²⁻¹³. It was shown that the degree of conversion at depths greater than 2 mm was drastically reduced¹⁴. Ilie and Stawarczyk¹⁵ evaluated the quantification of the amount of blue light passing through monolithic zirconia with respect to thickness polymerization conditions. The highest influence on the measured irradiance passing through translucent zirconia was exerted by ceramic thickness, closely followed by polymerizing mode. Lee
and Um15 used thermal analysis. They showed that the cure speed by light exposure was 5–20 times faster than that by chemical cure in dual-cured cements. Thicker ceramic discs resulted in reduced polymerization rates. Also, the resin cements differed in sensitivity to light- and chemical-curing. The purpose of this study was to evaluate the attenuating effect of curing light through various thicknesses of zirconia and the subsequent shear bond strength of dual-cure resin cement. The null-hypothesis considered that the difference of zirconia thickness would not affect the SBS between dual-cure resin cement and zirconia.

**MATERIALS AND METHODS**

Eighty blocks (7 mm in length and width, with different thicknesses) of zirconia (LAVA, 3M-ESPE, Saint Paul, MN, USA) were prepared. Forty were 1 mm thick, twenty were 1.5 mm thick and twenty were 2 mm thick. The thicknesses correspond with clinically relevant ones found in all-ceramic crowns, and bridges. All blocks received airborne-particle abrasion (Microetcher II Kit, Danville materials, San Ramon, CA, USA) with 50 µm aluminum-oxide (Al2O3) particles, 2.5 bar pressure in circular movements at a distance of 10 mm perpendicular to the surface for 10 s. The surface of each block was cleaned with 37% phosphoric acid for 10 s, water rinsed for 10 s and air-dried for 5 s. Self-adhesive dual-cure resin cement (Rely-X U200, TR shade, 3M-ESPE) was chosen. Putty (ExaFine Putty Type, GC, Tokyo, Japan) molds with a hole (3 mm in diameter and 4 mm thick) were prepared by using a silicone point. The metal shank of the silicone point is of uniform 3 mm diameter. The holes of all the putty molds were equal. The reason we chose putty as the mold material is that putty can be closely fitted on to the zirconia surface with gentle pressure, and the area of the resin bonding surface would remain constant while mixed resin cement is packed into the mold. Eighty blocks were placed on a clear glass panel. The putty molds were then positioned on the treated side of the blocks. Resin cement was mixed according to manufacturers’ instructions and packed into the putty mold incrementally using a hand instrument. The light source of polymerizing unit (Dr’s Light AT, Good Doctors, Incheon, Korea) was high power Light Emitting Diode (LED) with light intensity ±1,400 Mw/cm² (wave length range 440 to 490 nm) passing through the different ceramic thickness. The light tip was contact with the glass panel. The exposure distance was 3 mm which is the thickness of the glass panel. Group A was 10 blocks of 1 mm thickness which were exposed to curing light directly above the resin cement for 20 s (Fig. 1A). Group B was 10 blocks of 1 mm thickness which were exposed to curing light passing through the zirconia and the 3 mm glass panel below for 20 s. The polymerizing tip was placed directly contact on the glass panel’s surface. Group C was 10 blocks of 1.5 mm thickness which were exposed to curing light passing through of the zirconia and the glass panel below for 20 s. Group D was 10 blocks of 2 mm thickness which were exposed to curing light for 20 s as previously stated (Fig. 1B). Group E was 10 blocks of 1 mm thickness which were exposed to curing light directly above the putty molds for 40 s (Fig. 1A). Group F was 10 blocks of 1 mm thickness which were exposed to curing light passing through the zirconia and the glass panel below for 40 s. Group G was 10 blocks of 1.5 mm thickness which were exposed to curing light passing through the zirconia and the glass panel below for 40 s. Group H was 10 blocks of 2 mm thickness which were exposed to curing light for 40 s as previously stated (Fig. 1B). (N=80, n=10/group).

Group A and E were exposed to curing light directly above the resin cement to measure the bond strength between the zirconia surface and the resin cement regardless of zirconia thickness as positive control. All blocks were bonded onto polytetrafluoroethylene (PTFE) cylinders (18 mm in diameter, 10 mm in height) with superglue (Fig. 2), stored at room temperature for 24 h, to get enough self-polymerization, and submitted to
a shear bond strength test (Instron Model 3345, Instron Worldwide Headquarters, MA, USA). Load was applied in shear at a constant crosshead speed of 0.5 mm/min until failure occurred. The maximum load (MPa) to failure was recorded by corresponding software (Bluehill software, Instron Worldwide Headquarters). The Shapiro-Wilk test was applied to ensure the normal distribution of the data before the ANOVA and the t-test ($p>0.05$). All statistical tests were performed using log transformed data as follows; (1) a two-way ANOVA with interaction model was used to find out the factors which affect the bond strength and (2) a one-way ANOVA was performed for each length of curing time (20 and 40 s) to detect the difference among thicknesses (0, 1, 1.5, and 2 mm) and an independent samples t-test was used to detect differential effects between curing times for each thickness. All statistical analyses were performed using statistical software, SAS 9.2 (SAS Institute, Cary, NC, USA). A value of $p<0.05$ was regarded as statistically significant.

RESULTS

The mean shear bond strength values are illustrated in Table 1 and Fig. 3. According to the two-way ANOVA test, while thickness factor showed a significant difference ($p<0.05$), time factor did not affect bond strength significantly ($p>0.05$) and there is no interaction between the two factors ($p>0.05$) (Table 2). When light-polymerized for 20 s, group C and D showed lower mean SBS values than group A and B, and no statistically significant difference was found between group A and

Table 1 Shear bond strength according to time and thickness

<table>
<thead>
<tr>
<th>Groups</th>
<th>Time (s)</th>
<th>Thickness (mm)</th>
<th>Mean±SD (MPa)</th>
<th>$p$</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>0</td>
<td>0</td>
<td>7.86±2.13a</td>
<td></td>
</tr>
<tr>
<td>B</td>
<td>20</td>
<td>1</td>
<td>5.94±2.01a</td>
<td>&lt;0.0001</td>
</tr>
<tr>
<td>C</td>
<td>1</td>
<td>1.5</td>
<td>4.19±1.23b</td>
<td></td>
</tr>
<tr>
<td>D</td>
<td>2</td>
<td>2</td>
<td>3.10±1.03c</td>
<td></td>
</tr>
<tr>
<td>E</td>
<td>0</td>
<td>0</td>
<td>7.70±3.15a</td>
<td></td>
</tr>
<tr>
<td>F</td>
<td>40</td>
<td>1</td>
<td>6.09±1.81a</td>
<td>0.0003</td>
</tr>
<tr>
<td>G</td>
<td>1.5</td>
<td>1.5</td>
<td>4.16±1.00b</td>
<td></td>
</tr>
<tr>
<td>H</td>
<td>2</td>
<td>2</td>
<td>4.19±1.27b</td>
<td></td>
</tr>
</tbody>
</table>

SD: standard deviation
Different letters indicate significant differences between the thicknesses (two-way ANOVA $p<0.05$)

Table 2 Statistical analysis* of shear bond strengths of the 8 groups

<table>
<thead>
<tr>
<th>Factor</th>
<th>Type III SS</th>
<th>df</th>
<th>MS</th>
<th>F</th>
<th>$p$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Time</td>
<td>0.12</td>
<td>1</td>
<td>0.12</td>
<td>1.11</td>
<td>0.2947</td>
</tr>
<tr>
<td>Thickness</td>
<td>6.99</td>
<td>3</td>
<td>2.33</td>
<td>22.32</td>
<td>&lt;0.0001</td>
</tr>
<tr>
<td>Time* Thickness</td>
<td>0.39</td>
<td>3</td>
<td>0.13</td>
<td>1.24</td>
<td>0.3022</td>
</tr>
<tr>
<td>Error</td>
<td>7.51</td>
<td>72</td>
<td>0.13</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Total</td>
<td>15.01</td>
<td>79</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

*: Two-way analysis of variance with interaction.
when light-polymerized for 40 s, the mean SBS values of group G and H were significantly lower than group E and F with no significant difference between group G and H (*p=0.0003) (Table 1). According to an independent samples t-test between two groups of the same thickness but different curing times, only the 2 mm thickness groups (group D and H) showed a statistically significant difference between 20 and 40 s (*p<0.05) (Table 3).

DISCUSSION

To gain a reliable bond between ceramic materials and resin cements, it is necessary to get mechanical retention by surface roughening and additional retention by hydrofluoric acid etching and silane agent. However, unlike conventional feldspathic and lithium-disilicate ceramics, zirconium-oxide ceramics contain just below 1 wt% silica, and it is difficult to obtain the additional retention in this way. The mechanical retention of resin cement, which locks into the rough surface of the sandblasted zirconia, closely relates to the mechanical properties of the resin cement. The mechanical properties of dual-cure resin cement can be decreased when it is chemically polymerized without light polymerization. Therefore, the thickness of zirconia through which curing light passes can affect the mechanical properties of the resin cement as well as shear bond strength. In this experiment, the shear bond strength tends to lessen as the thickness increases (Table 1). It is likely that the thicker the zirconia, the weaker the intensity of curing light which passes through it, and that the mechanical properties of the resin cement decreases. To maximize bond strength between the zirconia and the resin cement, it would be valuable to avoid excessive tooth reduction.

When the 2 mm thickness group H is compared with the 1.5 mm thickness group C and G, group H is not as significantly decreased as group C or G even though the thickness of zirconia increased. It was reported that light intensity decreased exponentially with increasing ceramic disc thickness in the previous study. Increasing thickness, however, did not affect shear bond strength significantly no matter how much the intensity of curing light decreased. Between the two mechanisms of dual-cure resin cement, chemical- and light-polymerization, the chemical-polymerization might be a more effective polymerization mechanism than light-polymerization, when the thickness of zirconia is more than 1.5 mm. Choosing dual-cure resin cement rather than light-cured resin cement can be more favorable if we must set thick zirconia restorations of more than 1.5 mm.

Unlike thickness, curing time did not show a statistically significant difference in shear bond strength between the 20 and 40 s of light cure time in the independent t-test (Table 3). What is interesting is that while 40 s light polymerization did not increase the bond strength significantly in the case of the 0, 1 and 1.5 mm groups, it increased the bond strength significantly only in the 2 mm group although the difference was not notably large (Table 3). The attenuation of light intensity may be relatively small in the three thin groups and 20 s exposure to curing light may be enough to be polymerized. However, since the light attenuation was large in the 2 mm group, just 20 s exposure to curing light may be insufficient to achieve optimal light-polymerization. Insufficient polymerization can lead to adverse clinical consequences such as microleakage, postoperative sensitivity, marginal discoloration, secondary caries. Therefore it is necessary to apply sufficient light polymerization in the initial phase of setting. Longer exposure to curing light than recommended could help improve the mechanical properties of resin cement if the thickness of the zirconia restoration is over 2 mm.

Sandblasting increased the zirconia surface area, permitting substantial surface reaction and greater contact between the hydrophilic adhesive monomers and zirconia ceramics. Several techniques, especially the airborne particle abrasion with alumina, have been reported to facilitate "mechanically" the bond strength between resin cement and Y-TZP ceramic. In the present study, the zirconia surfaces were sandblasted for the microretentive grooves with the 50 μm aluminium-oxide microspheres which were commonly used in previous experiments. Gomes et al. observed that no differences were identified in the architecture of zirconia surfaces sandblasted with different-sized Al2O3 particles (25, 50 and 110 μm), that showed comparable microretentive grooves at the micrometre scale. Also, there were no significant differences between 2 mm thickness groups showed a significant difference between 20 and 40 s of light cure time (*p<0.05).

<table>
<thead>
<tr>
<th>Group</th>
<th>Thickness (mm)</th>
<th>p</th>
</tr>
</thead>
<tbody>
<tr>
<td>A, E</td>
<td>0</td>
<td>0.7317</td>
</tr>
<tr>
<td>B, F</td>
<td>1</td>
<td>0.7991</td>
</tr>
<tr>
<td>C, G</td>
<td>1.5</td>
<td>0.9211</td>
</tr>
<tr>
<td>D, H</td>
<td>2</td>
<td>0.0367**</td>
</tr>
</tbody>
</table>

*: Independent samples t-test
**: 2 mm thickness groups showed a significant difference between 20 and 40 s of light cure time (*p<0.05).
differences on the effect of sandblasting particle sizes of air-abrasion alumina particles on the bond strength between resin cements and zirconia dental ceramic. Phark et al., and Valentino et al. found similar results that no statistical difference regarding SBS between 50 and 110 μm Al₂O₃ particles after bonding Panavia F or Enforce to zirconia disks.

However, the bond strength between resin cements and zirconia dental ceramic should be further evaluated, taking into account the possible influence of different particle sizes of air-abrasion in the long-term.

Many authors have found that shear bond strengths to dentin were lower after self-curing than after dual-curing of dual-cured composite materials. Shear bond strength to composite is also enhanced by dual-curing a dual-cured cement. A possible explanation for the phenomenon is a lower degree of conversion present in the specimens that had to rely on chemical initiators alone, as in the self-cure groups, resulting in lower mechanical properties of the cements. The weaker shear bond strength of the 2 mm thickness groups in this study could also be explained by a difference in degree of cure of the specimens. The volumetric shrinkage of RelyX Unicem was significantly lower after self-curing compared to after dual-curing. This is representative for a lower degree of cure. It has been shown that dual-curing RelyX Unicem results in a degree of conversion that is twice as high as compared to self-curing. Higher degree of cure results in increased mechanical properties. This can explain the higher shear bond stresses of the 1 mm thickness groups.

This study was conducted at room temperature, however, if after being exposed to curing light the chemical polymerization was activated at the temperature of the oral cavity for 24 h, the differences between thicknesses might have been reduced, due to improved effect of the chemical polymerization. The temperature of the oral cavity should be reflected in future experiments.

CONCLUSION

Within the limitations of this study, the following conclusion was drawn: As the thickness of zirconia increases, the shear bond strength of dual-cure resin cement decreases. Compensation for decreased bond strength by an increased duration of light-polymerization of 40 s would be helpful to obtain a reliable bond between zirconia ceramics thicker than 2 mm and dual-cure resin cements.

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