The Effect of Curing Mode in Universal Adhesive on Zirconia and Resin Cement Shear Bond Strength

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Abstract
This study examined the effect of curing mode in the universal adhesive on the shear bond strength of zirconia and resin cement interface. Universal adhesive (Singlebond universal, SBU) and dual-cure activator (Singlebond universal dual-cure activator, DCA) were used. Forty zirconia disks were prepared and embedded in dental gypsum. The samples were randomly divided into four groups (n = 10) according to curing mode: group 1 (No tx), group 2 (SBU+LC), group 3 (SBU+DCA+LC) and group 4 (SBU+DCA). Silicone mold was placed on top of the treated zirconia surface. Resin cement was filled into the mold and subsequently stored in a yellow box for 30 minutes. All bonded specimens were kept in distilled water at 37°C for 24 hours and they were subjected to bond strength measurement using a universal testing machine. The data were statistically analyzed using one-way ANOVA and Tukey’s test. The shear bond strengths of group 1 to group 4 were 6.13±2.45, 25.42±3.09, 24.26±1.98 and 17.38±2.76 MPa, respectively (P < 0.05). There was no statistical difference between group 2 and group 3 (P > 0.05). In conclusion, the light activation in the universal adhesive with/without DCA (dual-curing mode/light-curing mode) had a positive effect on zirconia and resin cement interface.

Keywords: Curing mode, resin cement, universal adhesive, zirconia.

Received date: 26 May 2021. <br>Accept date: 08 July 2021

Introduction
Zirconia has been recently introduced in restorative, prosthetic, and aesthetic dentistry for the fabrication of crowns and fixed partial dentures. Zirconia has appeared as a versatile and promising material among dental ceramics, due to its biocompatible characteristics and excellent mechanical properties owing to the transformation toughening mechanism.¹⁻³ Its mechanical properties are very close to those of metals and its color similar to tooth color.⁴ Retention of zirconia restorations depends on mechanical roughening of the zirconia surface⁵⁻⁶ and chemical bonding with the adhesive functional monomer in special primers, adhesives, and resin cements.⁷⁻⁹ An acidic adhesive functional monomer such as 10-methacryloyloxydecyldihydrogen phosphate (10-MDP) bonds to zirconia-based ceramics. The phosphate functional group of the acidic monomer results in chemical adhesion to metal oxides, such as zirconia-based ceramics. Kim et al. reported that the addition of 10-MDP-containing monomer in the universal adhesive to enhance the bond strength of zirconia and resin cement interface.⁹

In the past recent years, dental manufacturers have been developed a new trend of adhesive called “universal adhesive”. The term “universal” offers the creativity of use with a variety of direct and indirect dental restorative materials, capable of bonding to various restorative materials combined with appropriate surface treatments.¹⁰ All the universal adhesive products tested were based on the 10-MDP acidic functional monomer with a documented adhesion capacity with the tooth.¹¹ titanium, base
metal alloys and polycrystalline ceramics such as zirconia. One of the advantages of using universal adhesive in the clinical routine would be granting the dentist to choose the type of application protocol according to the clinical situation, improving the final result of the procedure. However, while the development of universal adhesive systems has been an innovation in adhesive dentistry, it is questionable whether or not they are appropriate for curing mode of universal adhesive in indirect restorative procedures. There is still a lack of literature about universal adhesives, with few studies in the curing mode of these materials.

The purpose of this study was to examine the effect of the curing mode in the universal adhesive on the shear bond strength of zirconia and resin cement interface. The null hypothesis of this study was that the curing mode in universal adhesive does not affect the shear bond strength of zirconia and resin cement interface.

Materials and methods

The descriptions of the universal adhesive, dual-cure activator, and resin cement investigated in this study are given in Table 1.

<table>
<thead>
<tr>
<th>Material</th>
<th>Composition</th>
</tr>
</thead>
<tbody>
<tr>
<td>Singlebond universal (3M, Deutschland GmbH, Neuss, Germany)</td>
<td>10-MDP, Bis-GMA, HEMA, DMA, methacrylate functional copolymer, silane, filler, initiators, ethanol, water</td>
</tr>
<tr>
<td>Lot: 483316</td>
<td></td>
</tr>
<tr>
<td>Singlebond universal dual-cure activator (3M, Deutschland GmbH, Neuss, Germany)</td>
<td>Sodium toluene sulfonate and ethanol curing agent</td>
</tr>
<tr>
<td>Lot: 472079</td>
<td></td>
</tr>
<tr>
<td>Multilink N (Ivoclar Vivadent, AG, FL-9494 Schaan, Liechtenstein)</td>
<td>Pastes of dimethacrylates, HEMA, inorganic fillers, ytterbium trifluoride, benzoylperoxide, initiators, stabilizers and pigments</td>
</tr>
<tr>
<td>Lot: W34404</td>
<td></td>
</tr>
</tbody>
</table>

Table 1. Materials used in the study.

Abbreviations: 10-MDP, 10-methacryloyloxydecyl dihydrogen phosphate; Bis-GMA, bisphenol A-glycidyl methacrylate; HEMA, 2-hydroxyethyl methacrylate; DMA, dimethacrylate.

Specimen preparation

Forty fully sintered zirconia disk specimens (VITA YZ HT, VITA Zahnfabrik, Germany) (6.0 mm in diameter, 4.0 mm in thickness) were supplied by the manufacturer. Each disk specimen was embedded in polyvinyl chloride (PVC) pipe with dental gypsum type IV. The surfaces of all bonding specimens were polished with 600 grid Silicon carbide paper (3M Wetordry abrasive sheet, 3M, Minnesota, USA) followed by ultrasonic cleaning for 10 minutes in distilled water and subsequent drying with triple syringe oil-free air for 10 seconds.

The specimens were randomly divided into 4 groups of 10 specimens each and zirconia surface treated with universal adhesive [Singlebond universal (SBU), 3M, Deutschland GmbH, Neuss, Germany] and with or without dual-cure activator [Singlebond universal dual-cure activator (DCA), 3M, Deutschland GmbH, Neuss, Germany] as follows:

Group 1, no zirconia surface treated, serves as a negative control.

Group 2 (SBU+LC), specimens were treated with single bond universal using a microbrush, and then dried with triple syringe oil-free air for 10 seconds and subsequent light cured 20 seconds, as a light-curing mode.

Group 3 (SBU+DCA+LC), specimens were treated with single bond universal + dual-cure activator (mixing) using a microbrush, and then dried with triple syringe oil-free air for 10 seconds and subsequent light-cured 20 seconds, as a dual-curing mode.

Group 4 (SBU+DCA), specimens were treated with single bond universal + dual-cure activator (mixing) using a microbrush, and then dried with triple syringe oil-free air for 10 seconds, as a self-curing mode.

Pieces of polyethylene adhesive tape (Scotch blue painter’s tape, 3M, Minnesota, USA) approximately 80 microns in thickness with 2.0 mm diameter circular holes were placed on the pretreated surfaces of the specimens to define the bonding area. A silicone mold (2.0 mm in diameter, and 4.0 mm in thickness) was placed on the adhesive tape top, and then filled with resin cement (Multilink N, Ivoclar Vivadent, AG, FL-9494 Schaan, Liechtenstein), and subsequently kept in a yellow box for 30 minutes. The bond specimens were stored in an incubator (Incubator; Contherm 160M, Contherm Scientific Ltd., Korokoro, Lower Hutt, New Zealand) at 37 °C in distilled water for 24 hours.

Shear bond strength testing and surface analysis

The shear bond strengths of the specimens were executed in a universal testing machine (AGS-X 500N, Shimadzu Corporation, Kyoto, Japan), with a load applied in the direction parallel to the bonding surface at a crosshead speed of 0.5 mm/minute. The peak of failure load and bonding surface area was used to calculate
the shear bond strength value (MPa).

The fractured zirconia surfaces were investigated under a stereomicroscope (Stereomicroscope; ML 9300, Meiji Techno Co. Ltd., Saitama, Japan) at a magnification of x40 to examine failure types.\textsuperscript{14,20,21} The failure modes were classified as A, an adhesive failure at the zirconia and resin cement interface; B, a mixed failure of an adhesive failure at the zirconia and resin cement interface and a cohesive failure of the resin cement; and C, a cohesive failure in the resin cement.

**Statistical analysis**

The data were statistically analyzed using one-way ANOVA and Tukey’s test. Results with $P < 0.05$ were considered statistically significant.

**Results**

The highest and least of shear bond strength was in a range as follows: (1) SBU+LC, (2) SBU+DCA+LC, (3) SBU+DCA, and (4) no treatment ($P < 0.05$). However, there was no statistical difference between SBU+LC and SBU+DCA+LC. In this study, no specimens showed cohesive failure. SBU+LC and SBU+DCA+LC had higher percentages of mixed failure than SBU+DCA and no treatment group.

Details were summarized in Table 2.

<table>
<thead>
<tr>
<th>Group</th>
<th>Mean bond strength ±SD (MPa)</th>
<th>Failure mode</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Adhesive</td>
<td>Mixed</td>
</tr>
<tr>
<td>No treatment</td>
<td>6.13±2.45a</td>
<td>0</td>
</tr>
<tr>
<td>SBU+LC</td>
<td>25.42±3.09b</td>
<td>60</td>
</tr>
<tr>
<td>SBU+DCA+LC</td>
<td>24.26±1.98b</td>
<td>60</td>
</tr>
<tr>
<td>SBU+DCA</td>
<td>17.38±2.76a</td>
<td>70</td>
</tr>
</tbody>
</table>

**Table 2.** Mean bond strength, standard deviation (Megapascal) and percentage of failure mode.

The value with the identical letters indicates no statistically significant difference.

**Discussion**

This study examined the effect of curing mode in the universal adhesive on zirconia and resin cement shear bond strength. The result shows significant differences between the shear bond strength of each group. Therefore, the null hypothesis was rejected.

One of the most current novelties, in adhesive and restorative dentistry, was the introduction of universal adhesives. Its composition is an important factor to be taken into account since most of the universal adhesive contain specific phosphate and/or carboxylate acidic functional monomers. For example, 10-MDP is an acidic functional monomer found in certain universal adhesives.\textsuperscript{22} This is a hydrophilic functional monomer with mild-etching properties, with a documented adhesion capacity with the tooth,\textsuperscript{11} titanium, base metal alloys\textsuperscript{12-14} and polycrystalline ceramics such as zirconia.\textsuperscript{15-18} Klaisiri et al. reported that the universal adhesive containing phosphate acidic functional monomer such as MDP might be an alternative to the primer containing MDP for zirconia surface modification.\textsuperscript{17}

In the present investigation, the negative control group showed the lowest shear bond strength as a universal adhesive was not used. All other groups exhibited significantly higher shear bond strength than the negative control group. However, the shear bond strength varied greatly among the curing modes. Many factors also may influence the shear bond strength of a material, for example, light activation, DCA, or wettability of materials.\textsuperscript{23,24}

Regarding the self-curing mode (SBU+DCA) had lower shear bond strength than the light-curing mode (SBU+LC) and dual-curing mode (SBU+DCA+LC). Universal adhesives have pH values ranged from 1.6 to 3.2,\textsuperscript{25} though the majority most of the universal adhesives have a pH value of more than 2.0.\textsuperscript{26} The incompatibility with dual-cured and self-cured resin composite materials has been shown in previous studies.\textsuperscript{26,27} Some manufacturers included DCA bottles separately with their adhesives. The use of DCA with the universal adhesives to bond dual-cured resin composite materials was found to be unacceptable, as adhesive and dual-cured resin composite material incompatibility was reported.\textsuperscript{28} Otherwise, the hydrophilicity of universal adhesives showed a role in the determination of the incompatibility between universal adhesives and dual-cured resin composite materials.\textsuperscript{29} Besides, DCA might dilute the concentration of monomer contents of the universal adhesive,\textsuperscript{23} which had lower bond strength on their adhesion to zirconia. On the other hand, Gutiérrez et al. reported that the addition of DCA might enhance the bond strength of the universal adhesive. The residual acidic monomer in the underlying oxygen inhibited layer of universal adhesive might be activated by DCA, resulting in higher bond strength.\textsuperscript{24}
For the light-curing mode (SBU+LC) and dual-curing mode (SBU+DCA+LC), there was no statistically significant difference between light-curing and dual-curing modes. The result of this investigation came in agreement with the previous studies. Gutiérrez et al. and Elsayed et al., found that the decrease in the hydrophilicity of universal adhesive was responsible for the insignificant difference in the shear bond strength of the universal adhesive between the different curing mode, and the low HEMA concentration within the universal adhesive (5%-10%) might improve the chemical adhesion of the universal adhesive.

Regarding the failure mode, the shear bond strength test can show predominantly adhesive and mixed failure modes. The adhesive failures were mainly obtained from all groups. The mixed failures were often correlated with high shear bond strength, as observed from group 2, group 3, and group 4 (Table 2).

Conclusions

Within the limitations of the present study, the light activation in the universal adhesive with/without DCA (dual-curing mode/light-curing mode) had a positive effect on zirconia and resin cement interface. Using the DCA only (self-curing mode) did not enhance shear bond strength for bonding dual-cured resin cement to zirconia.

Acknowledgements

This study was supported by the Thammasat University Research Unit in Restorative and Esthetic Dentistry, Thammasat University, Thailand. The English proofreading of this manuscript was kindly performed by Dr. Ahmed A. Abbas, Faculty of Dentistry, Thammasat University.

Declaration of Interest

The authors declare no potential conflicts of interest with the materials involved in the present study.

References