Effect of Different Cleansing Agents on the Shear Bond Strength Between Resin Composite and Lithium Disilicate Ceramic after Contamination with a Hemostatic Agent

Sirichan Chiaraputt¹*, Tool Sriamporn², Sirirat Manopinives³

1. Department of Conservative Dentistry and Prosthodontics, Faculty of Dentistry Srinakharinwirot University, Bangkok, Thailand.
2. Department of Prosthodontics, Faculty of Dental Medicine, Rangsit University, Pathumthani, Thailand.
3. Postgraduate student, Department of Conservative Dentistry and Prosthodontics, Faculty of Dentistry Srinakharinwirot University, Bangkok, Thailand.

Abstract
The aim of this study was to evaluate the effect of cleansing agents on the shear bond strength of resin cement and lithium disilicate ceramic after being contaminated with the hemostatic agent. The specimens were randomly divided into six groups (n=10) according to different treatments on ceramic surfaces: Group 1 without any surface treatment, Group 2 only applied a silane coupling agent, Groups 3-6 also applied silane coupling agent, followed by a hemostatic agent (Viscostat® Clear) for 5 minutes and then cleaned with cleansing agents (Group 3 distilled water, Group 4 phosphoric acid, Group 5 chlorhexidine, Group 6 ethanol). All prepared specimens were bonded with resin cement (PanaviaTM V5). All bonded specimens were subjected to shear bond strength measurement using a universal testing machine. The data were statistically analyzed using one-way ANOVA and Tukey’s test (α=0.05). The results showed that the shear bond strength of Group 4-6 had no significant differences in shear bond strength between groups (p>0.05) and were lower than Group 2, which was the control group but higher than Group 1, the only group without surface treatment. In conclusion, cleaning hemostatic agent contaminated surfaces of ceramics can restore shear bond strength between lithium disilicate ceramic and resin cement. Cleaning with water showed the lowest shear bond strength among other cleansing agents in this experiment.

Keywords: Cleansing methods, Hemostatic agent.

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Introduction
Lithium disilicate ceramic is one of the most popular ceramic materials. The material is classified as a synthetic crystal ceramic, which also has the advantage of having good strength¹². The material is utilized in a wide range of dental applications, such as for inlays, onlays, veneers, and posterior fixed prostheses.

A rigid bond of the restoration to the tooth structure is one of the keys to achieving a successful restoration. To achieve a high-quality bond, all the substrates to be bonded must be clean, as having a fresh clean surface of the substrate increases its wettability, therefore allowing a firm bond to be created³⁴. However, the surfaces of substrates are prone to becoming contaminated during the bonding procedure. Previous study reported the interference of hemostatic agent to the bond which reduced the bond strength between dentin substrate and resin.⁵ Little is known about the contamination of ceramic substrate and the proper cleansing method. This study investigated the bond quality between lithium disilicate and resin cement after contamination and cleaning. It is shown that an effective cleaning protocol can aid forming stronger bonds and thus can support a higher endurance of the restoration.

The aim of the current study was to investigate the effect of different cleansing agents on the bonding efficacy of lithium disilicate ceramic. The null hypothesis tested was there would be no significant different of shear bond strength between lithium disilicate ceramic and resin composite when contaminated and cleaned with distilled water, chlorhexidine gluconate, phosphoric acid, and ethyl alcohol.
Materials and methods

The materials used in this study and their batch numbers are described in table 1.

<table>
<thead>
<tr>
<th>Product</th>
<th>Batch no.</th>
<th>Composition</th>
</tr>
</thead>
<tbody>
<tr>
<td>Lithium disilicate ceramic (IPS e.max Press, Ivoclar Vivadent AG, Liechtenstein)</td>
<td>W02583</td>
<td>Lithium disilicate crystals (approx. 70%), Li2SiO2Si5, embedded in a glassy matrix. Standard compositions: SiO2, Li2O, K2O, P2O5, ZrO2, ZnO, other oxides and ceramic pigments</td>
</tr>
<tr>
<td>Resin cement (Panavia VS, Kuraray Noritake Dental Inc., Japan)</td>
<td>AN0018</td>
<td>Base: bisphenol A diglycidylmethacrylate (Bis-GMA), triethylene glycol dimethacrylate (TEGDMA), silanated barium glass filler, silanated fluororaiminosilicate glass filler, colloidal silica, hydrophobic aromatic dimethacrylate, hydrophilic aliphatic dimethacrylate, initiators, accelerators Catalyst: bisphenol A diglycidylmethacrylate (Bis-GMA), hydrophobic aromatic dimethacrylate, hydrophilic aliphatic dimethacrylate, silanated barium glass filler, silanated aluminium oxide filler, di- Camphorquinone, accelerators, pigments</td>
</tr>
<tr>
<td>Hemostatic agent (ViscoStat Clear, Ultradent Products, Inc.)</td>
<td>BG7KB</td>
<td>25% m/m Aluminium chloride solution</td>
</tr>
<tr>
<td>Phosphoric acid (SDI Super Etch Bayswater, Victoria, Australia)</td>
<td>180402</td>
<td>37% Phosphoric acid</td>
</tr>
<tr>
<td>Chlorhexidine gluconate (C-20, Oocho Inter Laboratories Co., LTD, Thailand)</td>
<td>02390016</td>
<td>Chlorhexidine gluconate 0.12 g</td>
</tr>
<tr>
<td>Ethyl alcohol (Srinbuncha Co., LTD, Thailand)</td>
<td>0979343</td>
<td>Ethyl alcohol 70% V/V</td>
</tr>
</tbody>
</table>

Table 1. Materials and compositions.

Specimen preparation

Sixty lithium disilicate specimens were fabricated using IPS Emax ingot shade MO (IPS e.max Press, Ivoclar Vivadent AG, Liechtenstein), which is a lithium disilicate glass-ceramic ingot. The cylindrical specimens were prepared with a diameter of 5 mm and thickness of 3 mm. The specimens were polished in sequence with silicon carbide abrasive papers from 200 to 600 grits under water coolant (Nano 2000, Pace Technologies, USA). The specimens were then cleaned with an ultrasonic cleaner. The surfaces of the specimens were investigated with a light stereomicroscope (Olympus Co., Japan) with 40X magnification. This process was done to ensure the quality of the ceramic surfaces before the test. The ceramic specimens were then divided into 6 groups.

Group 1, no surface treatment
Group 2, lithium disilicate ceramic surface treated with silane.
Group 3, lithium disilicate ceramic surface treated with silane then contaminated with hemostatic agent and cleaned with distilled water.
Group 4, lithium disilicate ceramic surface treated with silane then contaminated with hemostatic agent and cleaned with 37% phosphoric acid.
Group 5, lithium disilicate ceramic surface treated with silane then contaminated with hemostatic agent and cleaned with 0.12% chlorhexidine.
Group 6, lithium disilicate ceramic surface treated with silane then contaminated with hemostatic agent and cleaned with 70% alcohol.

The surface of lithium disilicate specimens were then covered with adhesive tape with the 2mm diameter hole in the center. This surface was prepared for bonding with resin cement. The resin cement was delivered into the prepared surface area of each specimen. After resin cement bonding, the specimens were stored in distilled water for 24 hours. The materials used with batch numbers are shown in table1.

The resin cement was applied onto the prepared surface area of each specimen. After the resin cement had bonded, the specimens were then stored in distilled water for 24 hours.

Shear bond strength test

The bonded specimens underwent a shear bond strength test using a universal testing machine (EZ-S 500N, Shimadzu Corporation, Kyoto, Japan) with a crosshead speed of 0.5 mm/min. The shear bond strength is reported in megapascals (MPa).

Failure analysis

After the shear bond strength test, the fractured surfaces were investigated by light stereomicroscopy (SZ61, Olympus Co., Japan). At 40X magnification, the fracture surfaces were classified into 4 failure categories: adhesive failure, cohesive failure in the ceramic; cohesive failure in the resin cement; and mixed failure. The surface was assessed using ImageJ software.
Elemental chemical analysis

Another set of specimens was prepared and investigated by scanning electron microscopy—energy dispersive X-ray spectroscopy (SEM-EDS; JEOL, JSM-IT300, Japan and Oxford Instruments, X-MaxN20, Canada). The specimens underwent elemental chemical analysis by energy dispersive X-ray spectroscopy, while the micromorphology of all the samples were evaluated by SEM.

Results

The mean shear bond strength of each group and the failure modes are shown in Table 2. The elemental chemical analysis results are shown in Table 3.

<table>
<thead>
<tr>
<th>Group</th>
<th>Shear bond strength (MPa ± SD)</th>
<th>Number of pretest failures</th>
<th>Mode of failure (%)</th>
<th>Adhesive</th>
<th>Cohesive</th>
<th>Mixed</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 No treatment</td>
<td>2.13 (0.43)</td>
<td>3/10</td>
<td>100</td>
<td>0</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>2 Control group</td>
<td>18.04 (1.18)</td>
<td>0/10</td>
<td>100</td>
<td>0</td>
<td>90</td>
<td>0</td>
</tr>
<tr>
<td>3 Hemostatic agent + Distilled water</td>
<td>9.79 (0.86)</td>
<td>0/10</td>
<td>100</td>
<td>0</td>
<td>0</td>
<td>90</td>
</tr>
<tr>
<td>4 Hemostatic agent + 37% Phosphoric acid</td>
<td>14.31 (3.03)</td>
<td>0/10</td>
<td>100</td>
<td>0</td>
<td>0</td>
<td>90</td>
</tr>
<tr>
<td>5 Hemostatic agent + 0.12% Chlorhexidine</td>
<td>13.96 (3.27)</td>
<td>0/10</td>
<td>100</td>
<td>0</td>
<td>0</td>
<td>90</td>
</tr>
<tr>
<td>6 Hemostatic agent + 70% Ethyl alcohol</td>
<td>13.07 (3.47)</td>
<td>0/10</td>
<td>100</td>
<td>0</td>
<td>0</td>
<td>90</td>
</tr>
</tbody>
</table>

Table 2. Mean shear bond strength and modes of failure.

*Superscript letters following mean values: the same letter indicates not significantly different at p-value > 0.05.

<table>
<thead>
<tr>
<th>Element wt. (%)</th>
<th>No treatment</th>
<th>Control group</th>
<th>H</th>
<th>H + water</th>
<th>H + 37%H3PO4</th>
<th>H + 0.12% CHX</th>
<th>H + 70% Alc</th>
</tr>
</thead>
<tbody>
<tr>
<td>C</td>
<td>8.97</td>
<td>27.30</td>
<td>26.92</td>
<td>20.85</td>
<td>36.74</td>
<td>28.25</td>
<td>16.86</td>
</tr>
<tr>
<td>O</td>
<td>45.18</td>
<td>38.04</td>
<td>34.33</td>
<td>36.90</td>
<td>28.09</td>
<td>33.80</td>
<td>40.98</td>
</tr>
<tr>
<td>Mg</td>
<td>0.79</td>
<td>0.55</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>0.50</td>
<td>0.57</td>
</tr>
<tr>
<td>Al</td>
<td>1.00</td>
<td>0.62</td>
<td>-</td>
<td>-</td>
<td>0.64</td>
<td>-</td>
<td>1.09</td>
</tr>
<tr>
<td>Si</td>
<td>39.00</td>
<td>29.36</td>
<td>26.20</td>
<td>36.07</td>
<td>31.97</td>
<td>31.38</td>
<td>34.28</td>
</tr>
<tr>
<td>Cl</td>
<td>-</td>
<td>-</td>
<td>7.15</td>
<td>3.27</td>
<td>2.56</td>
<td>3.23</td>
<td>4.86</td>
</tr>
<tr>
<td>K</td>
<td>2.77</td>
<td>2.09</td>
<td>1.94</td>
<td>3.2</td>
<td>-</td>
<td>2.84</td>
<td>2.43</td>
</tr>
<tr>
<td>Zn</td>
<td>2.89</td>
<td>2.16</td>
<td>1.45</td>
<td>-</td>
<td>-</td>
<td>1.78</td>
<td>2.86</td>
</tr>
</tbody>
</table>

Table 3. Elemental chemical analysis.

H = Hemostatic agent, H3PO4 = Phosphoric acid, CHX = Chlorhexidine, Alc = Ethyl alcohol, C = Carbon, O = Oxygen, Mg = Magnesium, Al = Aluminium, Si = Silicon, Cl = Chloride, K = Potassium, Zn = Zinc.
Discussion

The results from this study showed the different shear bond strengths between lithium disilicate ceramic and resin cement after being contaminated with a hemostatic agent and cleaned with different cleansing agents. The water cleansing group showed the lowest shear bond strength, and therefore, the null hypothesis was rejected.

This study simulated the cementation of ceramic restoration with resin cement in a clinical situation. Aluminum chloride was used as the hemostatic agent, at a concentration of 25%, as it is the most used hemostatic agent and has been shown to be able to effectively control bleeding in clinical situations. 6,7 The duration of hemostatic application was 5 minutes as this has been reported in previous studies to be an effective duration. 8

Generally, the conditioning of lithium disilicate for bonding with resin cement involves two aspects: mechanical bonding, which can be enhanced by hydrofluoric acid etching, and chemical bonding, which is provided by silane treatment afterward. 4,6,9 However, this study aimed to study the chemical interaction with a hemostatic agent and therefore we excluded the effect of the micromechanical bonds from the hydrofluoric acid etching in this study. Moreover, recent study reported a significant increase in the bond strength of lithium disilicate treated with various type of silane without hydrofluoric acid. 10 Therefore, the effect of chemical bond from silane can be investigated in this study with the same protocol.

The effect of having a clean ceramic surface before bonding must be considered to achieve high-quality bonding. Previous studies have reported a reduction in bond strength when the substrates are contaminated before bonding. 11-13 In addition, our study also found a significant reduction in bond strength for the lithium disilicate in Groups 3 to 6 when contaminated with aluminum chloride hemostatic agent. This study also investigated the effectiveness of different cleansing agents. A study reported the lowest bond strength when contaminated lithium disilicate was cleaned with distilled water. 14 Our study also found that 37% phosphoric acid, 0.12% chlorhexidine, and 70% alcohol were all more effective cleansing agents than distilled water and enabled stronger bonds to be formed.
Yoshida et al. studied saliva-contaminated lithium disilicate and leucite-reinforced ceramic and reported that phosphoric acid could clean the ceramic surface effectively and was a suitable substrate for bonding.15 It has also been reported that phosphoric acid could form an aluminum phosphate compound with aluminum in the hemostatic agent, which could eventually be removed.16

Previous study found that ethyl alcohol could effectively clean the contaminated lithium disilicate as ethyl alcohol is a solvent that can dissolve both polar and non-polar agents.17 Therefore, aluminum chloride in the hemostatic agent can bond with OH groups, which could be the removal process by which alcohol functions in this study. Nikolous et al. reported that the bond strength could be regained when the saliva-contaminated ceramic was cleaned with ethanol.12

Chlorhexidine is an antimicrobial agent that can inhibit the MMPs (metrix metalloproteinase) in dentin.18 MMPs have been reported to be the initial factors involved in collagen degradation in dentin.19-20 Chlorhexidine was found to be effective in this study since it is also a detergent. Previous studies also reported that chlorhexidine could clean the remaining hemostatic agent19, although Sharafeddin et al. found that chlorhexidine had no effect on the bond strength after contamination.21

Araújo et al.22 reported a decreasing degree of conversion when resin cement was contaminated with aluminum chloride hemostatic agent. In addition, they found that the degree of conversion could affect the strength, elastic modulus, solubility, and hardness, which are the physical properties of resin cements. The degree of conversion also affects the mechanical properties of resin cements, such as the dimensional stability and the bonding quality.23

Recent studies have reported a reduction in the bond strength in self-etching resin cement when contaminated with aluminum chloride hemostatic agent.24-25 This is because the acidic functional monomer in the self-etching system cannot function on a contaminated surface. Kuphasuk et al. used EDX to study the joint interface and found aluminum residue from the aluminum chloride when using self-etching resin cement. However, there was no significant difference in bond strength from the control group when using total etch resin cement, as the phosphoric acid in the total etch resin cement could remove the aluminum chloride more that the functional monomer in the self-etching resin cement.7

Apart from the bond strength the bond quality was also evaluated together with the mode of failure. The most common failure mode was a mixed failure. There was no cohesive failure in the ceramic in this study since the hydrofluoric acid was not used to exclude the mechanical bond from the study.10

The EDX study indicated that aluminum chloride crystals remained on the ceramic surface after contaminating with hemostatic agent. After cleaning with the tested cleansing agents, we also found a reduction in chloride on the ceramic surface. Phosphoric acid and chlorhexidine were able to remove the chloride most effectively. This result conformed with the bond strength results. However, chloride residue on the ceramic surface was also found after cleaning with alcohol. This result was not related to the bond strength. However, the bond strength results were reported after 24 hours, and long-term study may be needed as any remaining hemostatic agent can affect the polymerization, color change, and bond durability.22

Conclusions

The contamination of a hemostatic agent on a ceramic surface can reduce the bond strength significantly. Cleansing agents can be used to clean ceramic surfaces. In testing different cleaning agents, we found that distilled water cleaned the ceramic surface less effectively than the other cleansing agents. Phosphoric acid, chlorhexidine, and ethyl alcohol as cleansing agents provided better bond strengths on the contaminated surface without significant differences among them. However, phosphoric acid cleaning showed a best surface with less contamination on the EDX and SEM study.

Declaration of Interest

The authors report no conflict of interest.

References

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