Effect of Simplified Bonding on Shear bond strength between ceramic bracket and dental zirconia.

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Abstract: With the increase in adult orthodontic patients, it is common to bonding a ceramic bracket to the anterior zirconia restoration. In this study, we investigated whether the adhesion between a ceramic bracket and yttrium-stabilized tetragonal zirconia polycrystal (Y-TZP) can be simplified by using a 10-MDP-containing adhesive. In the control group, bracket bonded on maxillary central incisor. All Y-TZPs were polished and sandblasted; 10-MDP-containing primer and orthodontic primer were applied in group C, 10-MDP-containing adhesive was applied in group S, and both 10-MDP-containing primer and adhesive were applied in group CS. A ceramic bracket was bonded to every Y-TZP with orthodontic resin. SBS was measured after with or without thermocycling. The data were statistically analyzed by using the Kruskal–Wallis test. Without thermocycling (C, S, CS), the SBS was within the acceptable range and showed adhesive failure between resin and bracket in all groups. With aging (CT, ST, CST), group CT showed the highest and reliable SBS and exhibited adhesive failure between resin and bracket. This demonstrates that with a simplified bonding step (S), stable bond strength of Y-TZP and resin can be obtained, however, the long-term stable SBS were obtained in group CT.

Keywords: dental zirconia; orthodontic bracket; 10-MDP; surface treatment; shear bond strength; resin bonding

1. Introduction

The number of adult orthodontic patients is increasing as rising attention is paid to aesthetics [1, 2]. The use of bracket materials like ceramics instead of conventional metals is also increasing for the aesthetic appearance attached to teeth [2]. A ceramic bracket is usually attached to the anteriors for aesthetics. When the tooth is restored with monolithic zirconia (MZ), clinicians often experience difficulties in direct bonding between the materials [3].

In recent years, MZ has often been used for single crowns and fixed partial denture restoration, because it is a material that offers two advantages of aesthetics and strength [3,4]. In the past, patients usually chose conventional porcelain fused to the metal crown (PFM) as the anterior restoration material, but because of issues such as fracture of porcelain or darkening of gingival shade owing to the metal lower structure, the use of MZ is increasing [5,6]. Since MZ is a single body that has no distinction between upper and lower structures, partial chipping of the restoration seldom occurs;
the type of zirconia usually used in dentistry is yttrium-stabilized tetragonal zirconia polycrystal (Y-TZP), which remains in the tetragonal phase at room temperature with the addition of 2–3 mol% yttrium; the flexural strength is also very high because of zirconia’s unique resistance against crack propagation [4,7,8]. Crack propagation is blocked because when a stress-like crack is initiated, a transition to the monoclinic phase occurs and the volume increases at the same time in zirconia [9–11]. Furthermore, Y-TZP has another advantage that it can be fabricated quickly by using a computer-aided design and computer-aided manufacturing (CAD/CAM) system [12,13].

However, because Y-TZP has no silica phase, it cannot bond with resin through etching and silane treatment like that performed on conventional porcelain, and a different method is required [14,15]. Various methods have been considered for the adhesive surface treatment of Y-TZP, and they include mechanical methods of laser burning, sandblasting, and tribochemical treatment to create micro-ruggedness on the surface [14–17], and chemical methods that use a bonding agent, chlorosilane vapor, or fluorinated plasma to improve the bonding strength with Y-TZP [14,18–20].

Because the adhesive area of an orthodontic bracket is relatively small compared to that of a regular restoration, sufficient bonding strength for successful orthodontic treatment is required that can endure the shear stress during daily activities such as chewing. In general, the process of bonding an orthodontic bracket to the enamel of a tooth includes three stages: etching the tooth, applying the orthodontic adhesive primer, and then attaching the bracket with orthodontic resin. However, there is no established protocol regarding how the surface should be treated to bond the bracket to Y-TZP effectively. The aforementioned methods of surface treatment of Y-TZP to increase the bond strength with resin can be used independently or combined, and one of the combined methods can increase the bonding strength between the resin and Y-TZP by sandblasting the Y-TZP surface to create micro-ruggedness and then applying the functional monomer-containing product [8,18,21]. A functional monomer, 10-MDP (10-methacyrlolxydecyl dihydrogen phosphate) is known to improve the bonding strength between Y-TZP and resin by chemically combining with oxides on the Y-TZP surface [15,22]. Beside of various methods, an important point is that additional steps for surface treatment of Y-TZP should be simple and should guarantee stable long-term bonding strength.

Through experiments conducted by sandblasting, using functional monomers in different bonding steps, and performing thermocycling treatment, we attempted to examine a method to simplify the process of bonding a bracket to Y-TZP. In addition, we explored ways to maintain the bonding strength in the long term. The hypotheses tested in this study are as follows: (1) Applying 10-MDP-containing adhesive to the sandblasted Y-TZP surface does not maintain the stable SBS of resin and Y-TZP; (2) Applying 10-MDP-containing primer to the sandblasted Y-TZP surface does not maintain the stable SBS of resin and Y-TZP.

2. Materials and Methods

2.1. Specimen Preparation

2.1.1. Zirconia

A total of 60 rectangular monolithic zirconia specimens were prepared from a green-stage Y-TZP block (LAVA Plus, 3M ESPE, USA) that was sintered according to the manufacturer’s instructions.

Each specimen was embedded in polyester resin (EC-304, Aekyung, Korea), and its bonding surface was polished with a diamond disc of 500 grit (MD-Piano, Struers, Denmark) under constant water cooling. The samples were cleaned by ultrasonic vibration and then dried. The Y-TZP surfaces were then sandblasted with 50 µm alumina particles (SandStorm Expert, Vaniman, USA) at a distance of 20 mm and at a pressure of 0.4 MPa in the vertical direction for 20 s. The sandblasted samples were cleaned with distilled water in an ultrasonic bath for 2 min and dried.

2.1.2. Tooth
Extracted human teeth were immersed in a 0.1% thymol solution for a week and stored at 4 °C. Prior to the experiments, the teeth had been stored for less than 3 months, and they were used for the study with the approval of the School of Dentistry, Seoul National University Institutional Review Board (No. S-D20150041). After separating the dental root and crown of each extracted maxillary central incisor \( (n = 20) \), the crown was fixed to the acrylic resin so that the entire labial surface of the crown would be exposed and parallel to the base. It was cleaned with distilled water for 2 min by using an ultrasonic machine and then dried, and then the labial surfaces were treated by using 37% phosphoric acid etchant (Scotchbond Universal Etchant, 3M ESPE, USA) according to the manufacturers’ instructions.

As a control group (CON), the labial surface of right maxillary central incisors \( (n = 20) \) was treated with orthodontic primer \( (\text{XTp}; \text{Transbond XT adhesive primer, 3M Unitek, USA}) \) according to the manufacturers’ instructions.

### 2.2. Surface Treatment and Bracket Bonding

The sandblasted Y-TZP samples were divided into three groups (C, S, and CS) according to the 10-MDP-containing primer and adhesive \( (n = 20) \) used. The Clearfil ceramic primer \( (\text{CP}; \text{Kuraray, Japan}) \) as a 10-MDP-containing primer, Clearfil S\(^3\) bond \( (\text{SB}; \text{Kuraray, Japan}) \) as a 10-MDP-containing adhesive, and XTa as a general orthodontic primer were used according to the manufacturers’ recommendations. For group C samples, CP was first applied on the Y-TZP surface, followed by the application of XTa. For group S samples, only SB was applied and light-cured. For group CS samples, SB was applied after CP was applied to the Y-TZP surface. The primer- and adhesive-applied surfaces were dried by enough blowing with a three-way air syringe in every application stage.

A maxillary central incisor ceramic bracket (Perfect Clear II, Hubit, Korea) was used with orthodontic resin paste \( (\text{XTa}; \text{Transbond XT adhesive paste, 3M Unitek, USA}) \). After an appropriate amount of XTa was applied to the bracket base, the bracket was placed under gentle pressure until the margin of the bracket base reached the Y-TZP surface. Excessive resin was removed with a resin applicator, and the bracket was cured with a light-emitting diode (LED) curing unit (Elipar Free Light 2, 3M ESPE, USA) at each margin for 10 s (total curing period: 40 s). The overall flow according to the bonding step is shown in Table 1. The components of materials used in this experiment are shown in Table 2.

<table>
<thead>
<tr>
<th>Step 1. Pre-treatment</th>
<th>E</th>
<th>S</th>
<th>S</th>
<th>S</th>
<th>E</th>
<th>S</th>
<th>S</th>
<th>S</th>
</tr>
</thead>
<tbody>
<tr>
<td>Step 3. Adhesive</td>
<td>XTa</td>
<td>XTa</td>
<td>SB</td>
<td>SB</td>
<td>XTa</td>
<td>XTa</td>
<td>SB</td>
<td>SB</td>
</tr>
<tr>
<td>Bracket bonding</td>
<td>XTa</td>
<td>XTa</td>
<td>XTa</td>
<td>XTa</td>
<td>XTa</td>
<td>XTa</td>
<td>XTa</td>
<td>XTa</td>
</tr>
<tr>
<td>Thermocycling</td>
<td>N</td>
<td>N</td>
<td>N</td>
<td>N</td>
<td>Y</td>
<td>Y</td>
<td>Y</td>
<td>Y</td>
</tr>
<tr>
<td>Group</td>
<td>CON</td>
<td>C</td>
<td>S</td>
<td>CS</td>
<td>CONT</td>
<td>CT</td>
<td>ST</td>
<td>CST</td>
</tr>
</tbody>
</table>

Abbreviations: E, etching with 37% phosphoric acid on tooth; S, sandblasting with 50 µm alumina on Y-TZP; CP, Clearfil ceramic primer (10-MDP-containing primer); SB, Clearfil S\(^3\) bond (10-MDP-containing adhesive); XTa, Transbond XT adhesive primer; XTa, Transbond XT adhesive paste; N, no; Y, yes.
Abbreviations: *10-MDP, 10-methacryloyloxydecyl dihydrogen phosphate; TEGDMA, triethylene glycol dimethacrylate; bis-GMA, bisphenol-A-diglycidylether dimethacrylate; HEMA, hydroxyethyl methacrylate.

### Table 2. Materials used in this study.

<table>
<thead>
<tr>
<th>Material</th>
<th>Product name</th>
<th>Abbreviation</th>
<th>LOT number</th>
<th>Main component</th>
<th>Manufacturer</th>
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</thead>
<tbody>
<tr>
<td>Zirconia</td>
<td>LAVA Plus</td>
<td>MZ</td>
<td>515920</td>
<td>Tetragonal polycrystalline zirconia, 3 mol% yttria, alumina</td>
<td>3MESPE, USA</td>
</tr>
<tr>
<td>Primer</td>
<td>Clearfil ceramic primer</td>
<td>CP</td>
<td>240010</td>
<td>3-Methacryloyloxypropyl triethoxy silane, *10-MDP, ethanol</td>
<td>Kuraray, Japan</td>
</tr>
<tr>
<td></td>
<td>Transbond XT</td>
<td>XTpr</td>
<td>ER7BS</td>
<td>10-MDP, *bis-GMA, triethylene glycol dimethacrylate, dl-camphorquinone,</td>
<td>3M Unitek, USA</td>
</tr>
<tr>
<td></td>
<td>adhesive primer</td>
<td></td>
<td></td>
<td>ethyl alcohol, water, silanated colloidal silica</td>
<td></td>
</tr>
<tr>
<td>Adhesive</td>
<td>Clearfil S³ Bond</td>
<td>SB</td>
<td>170008</td>
<td>Silane treated quartz, *bis-GMA, bisphenol A bis (2- hydroxyethyl ether)</td>
<td>Kuraray, Japan</td>
</tr>
<tr>
<td></td>
<td>Transbond XT</td>
<td>XTPa</td>
<td>ER7BS</td>
<td>dimethacrylate, silane-treated silica</td>
<td>3M Unitek, USA</td>
</tr>
<tr>
<td></td>
<td>adhesive paste</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Etchant</td>
<td>Scotchbond Universal Etchant</td>
<td>–</td>
<td>577060</td>
<td>Water, phosphoric acid, synthetic amorphous silica, fumed, polyethylene glycol</td>
<td>3M ESPE, USA</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>aluminum oxide</td>
<td></td>
</tr>
</tbody>
</table>

126 Abbreviations: *10-MDP, 10-methacryloyloxydecyl dihydrogen phosphate; †TEGDMA, triethylene glycol dimethacrylate; ‡bis-GMA, bisphenol-A-diglycidylether dimethacrylate; ‡‡HEMA, hydroxyethyl methacrylate.
127
128 2.3. Observation of Microstructure of bracket
129 The topography of the bracket was observed by using topography analyzer (OLS 5000, Olympus, Japan).
130
131 2.4. Surface characteristics of Y-TZP
132 The surface topology of Y-TZP before and after sandblasting, the polished and sandblasted Y-TZP surface was observed by using FE-SEM at ×500 and ×5000 magnification. The roughness (Ra) of the surface before and after sandblasting, were determined by using a 3D surface contouring laser scanning microscope (CLSM; LSM 800-MAT, Carl Zeiss MicroImaging GmbH, Germany).
133 The contact angle of a water droplet was measured on each sample three times by using a contact-angle analyzer (Phoenix 150, SEO, Korea).
134
135 2.5. Shear Bond Testing and Observation of Failure Mode
136 After bracket bonding, all the samples were kept in a 37 °C and relative humidity 100% incubator for 24 h. Half of the samples were then randomly selected from each group (CON, C, S, and CS), and the shear bond strength (SBS) of these samples was measured with a universal testing machine (Instron 8848, Instron, USA) at a crosshead speed of 0.5 mm/min. The remaining samples were subjected to the aging process by thermocycling for 10,000 cycles at 5 and 55 °C. Hereafter, these aged samples are classified under groups CONT, CT, ST, and CST (see Table 1). The immersion time in water was 30 s, and the changing time was 20 s. The maximum load-at-failure was calculated in MPa.
by dividing the maximum load (N) by the area of the bracket base (12.24 mm² according to the manufacturer). The adhesive surfaces of Y-TZP and the bracket where bonding failure occurred were observed by FE-SEM at ×30 magnification. After the observation, the amount of residual resin on the surface of the Y-TZP and the tooth was classified according to the adhesive remnant index (ARI) score (Table 3).

Statistical analysis was performed using Kruskal–Wallis ANOVA and the Statistical Package for the Social Sciences 22.0 (SPSS, IBM, USA).

### Table 3. ARI score and criterion.

<table>
<thead>
<tr>
<th>ARI score</th>
<th>Criterion</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>No adhesive left on the tooth</td>
</tr>
<tr>
<td>1</td>
<td>Less than half of the adhesive left on the tooth</td>
</tr>
<tr>
<td>2</td>
<td>More than half of the adhesive left on the tooth</td>
</tr>
<tr>
<td>3</td>
<td>All adhesive left on the tooth, with a distinct impression of the bracket mesh</td>
</tr>
<tr>
<td>4</td>
<td>Enamel fracture</td>
</tr>
</tbody>
</table>

### 3. Results

#### 3.1. Microstructure of bracket

The strength, color, height, and map of the bracket were examined by analyzing the topography of bracket, as shown in Figure 1. The strength was higher at the part with attached beads than at the border of the bracket, and the height increased as the distance from the center to the outer edges on both sides increased.

![Figure 1](image.png)

**Figure 1.** (a) Topography, (b) strength, (c) color, (d) height, and (e) map of the ceramic bracket base used in this study.

#### 3.2. Surface Roughness of Y-TZP

When the polished Y-TZP sample was examined at ×500 magnification, a very even surface was found; when it was examined at ×5,000 magnification, some directionless scratches were observed (Figure 2a,b). Examination of the sandblasted Y-TZP sample at ×500 magnification revealed uneven and roughly dented traces scattered on the surface. Meanwhile, the ×5,000 image shows a much rougher surface than that of the polished sample (Figure 2c,d).
Figure 2. SEM images of Y-TZP surfaces after polishing with 500 grit diamond wheel: (a) ×500 magnification; (b) ×5000 magnification and after sandblasting with 50 μm alumina; (c) ×500 magnification; (d) ×5000 magnification.

As a result of CLSM observation, the Y-TZP surface that was polished with a 500 grit diamond disc appeared relatively even surface; the average Rₐ was 0.10 μm (Figure 3a,b). The sandblasted Y-TZP surface showed more irregular side with increasing height differences, and Rₐ was 0.70 μm (Figure 3c,d).

Figure 3. CLSM images of Y-TZP surfaces after grinding with 500 grit diamond disc: (a) three-dimensional images; (b) flat images with height difference in color and after air-abrasion with 50 μm alumina; (c) three-dimensional images; (d) flat images with height difference in color.
3.3. Contact Angle on Y-TZP

In case of polished Y-TZP surface, average contact angle was 64.65 ± 0.25 degree while the average contact angle was 50.48 ± 2.81 degree in the sandblasted Y-TZP surface.

3.4. Shear Bond Strength and Failure Mode

Table 4 shows the SBS between the natural tooth and ceramic bracket, and between the Y-TZP and ceramic bracket; the values were obtained before and after thermocycling of the samples. When thermocycling was not performed, there was no significant difference between the SBS of all groups (CON, C, S, CS; p > 0.05; Figure 4). After the thermocycling treatment, the group with the 10-MDP-containing-primer applied on the surface, i.e., group CT, showed the highest SBS. In every group except group CT, the SBS decreased significantly from that obtained before thermocycling (p < 0.05). There was no significant difference between the values of the group CONT, group ST, and group CST (p > 0.05), while the values of group CT showed significant difference from those of the other groups (CONT, CT and CST; p < 0.05; Figure 4).

Table 4. Comparison of shear bond strength of all groups.

<table>
<thead>
<tr>
<th>Group</th>
<th>Shear bond strength (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>CON</td>
<td>9.59 ± 1.77(8.32-10.86)</td>
</tr>
<tr>
<td>C</td>
<td>9.78 ± 1.94(8.40-11.18)</td>
</tr>
<tr>
<td>S</td>
<td>9.86 ± 1.33(8.90-10.81)</td>
</tr>
<tr>
<td>CS</td>
<td>9.16 ± 0.78(8.60-9.71)</td>
</tr>
<tr>
<td>CONT</td>
<td>5.65 ± 1.24(4.76-6.54)</td>
</tr>
<tr>
<td>CT</td>
<td>8.16 ± 1.78(6.88-9.43)</td>
</tr>
<tr>
<td>ST</td>
<td>4.99 ± 0.99(4.28-5.70)</td>
</tr>
<tr>
<td>CST</td>
<td>4.31 ± 1.02(3.58-5.03)</td>
</tr>
</tbody>
</table>

Shear bond strengths (SBS) of each group are presented as mean ± SD (range).

Figure 4. Shear bond strength (SBS) of orthodontic ceramic bracket bonded to Y-TZP; the values of before and after thermocycling were obtained for different groups of samples. The vertical bars indicate the standard deviations. There are significant differences in SBS between the groups marked with different letters A, a and b (p < 0.05).

The ARI scores, i.e., amount of resin remaining on the tooth's enamel and Y-TZP surface after debonding, of different sample groups are summarized in Table 5. Figure 5 shows FE-SEM images of the interface between Y-TZP and the bracket, and the enamel surface after debonding of a sample.
from each group. Before the thermocycling treatment was performed, an ARI score of 3 (Figure 5a–d) was obtained for all groups, indicating that the resin remained on the Y-TZP surface of all samples, and the small amount of resin that was stuck and fell off between the retentive beads of bracket was not counted. After the thermocycling treatment, all of the samples in group CT still showed an ARI score of 3 (Figure 5j), but the samples in group ST and group CST showed ARI scores of 1 (Figure 5k, l), with less than half of the resin remaining on the Y-TZP surface in 40% of the samples respectively.

Table 5. The percentage (%) of ARI scores in the non-thermocycling groups and thermocycling groups.

<table>
<thead>
<tr>
<th>Without aging</th>
<th>ARI score</th>
<th></th>
<th>With aging</th>
<th>ARI score</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>0</td>
<td>1</td>
<td>2</td>
<td>3</td>
</tr>
<tr>
<td>CON</td>
<td>–</td>
<td>20</td>
<td>–</td>
<td>80</td>
</tr>
<tr>
<td>C</td>
<td>–</td>
<td>–</td>
<td>–</td>
<td>100</td>
</tr>
<tr>
<td>S</td>
<td>–</td>
<td>–</td>
<td>–</td>
<td>100</td>
</tr>
<tr>
<td>CS</td>
<td>–</td>
<td>–</td>
<td>–</td>
<td>100</td>
</tr>
</tbody>
</table>

4. Discussion

This study evaluated the effectiveness of applying the 10-MDP-containing primer and 10-MDP-containing adhesive on sandblasted Y-TZP samples in order to simplify the steps of bonding between Y-TZP and the bracket. Moreover, the thermocycling treatment was performed to assess the long-term stability of bonding [23]. There are many different opinions among researchers regarding the number of thermocycling treatments, but considering that an orthodontic treatment is usually carried
out over a period of one year, the number of cycles was set to 10,000 in this experiment, which corresponds to the usage period of about one year in an oral cavity [24].

In this experiment, 50 μm alumina particles were sprayed in the same manner onto every Y-TZP specimen to only evaluate the effect of the 10-MDP-containing agents. This method created micro-mechanical ruggedness that could increase the bonding strength between Y-TZP and the resin, and it is the usual method of surface treatment in dentistry [25]. A previous study demonstrated that if alumina is sprayed on the Y-TZP surface, it generates defects on the surface and decreases the strength of Y-TZP [26]. However, Kosmac et al. [27] reported that grinding the Y-TZP surface with a diamond burr deteriorates the strength of Y-TZP, whereas sandblasting is a powerful method that strengthens Y-TZP by inducing a phase change. Moreover, Curtis et al. [28] and Karakoca and Yilmaz [29] also presented similar results of the bi-axial flexure strength increasing as a result of sandblasting.

In addition, Demir et al. [30] stated that sandblasting is more effective than laser irradiation for the surface treatment of zirconia. Qeblawi et al. [31] reported that if chemical conditioning is carried out after performing alumina blasting or silica blasting on the surface of Y-TZP, stable bonding can be obtained. Similarly, Kim et al. [32] also recommended the use of a 10-MDP-containing agent after alumina-blasting or silica-blasting. The work of Kern et al. [33] showed that when an adhesive primer was applied on the Y-TZP surface after sandblasting, the long-term bond strength was noticeably increased. Therefore, we carried out the alumina-blasting and used the functional monomer in parallel for every experimental group in our study.

Untreated Y-TZP is known to exhibit poor wettability [34]. However, if alumina particles are sprayed on Y-TZP, the surface area of Y-TZP will increase and the surface energy will also increase, thereby contributing to the bonding-strength improvement by improving the wettability [35,36]. This was confirmed by the results of obtained in our study (Figures 2 and 3). The increase in surface roughness from 0.10 μm to 0.70 μm indicates an increase in surface area that can be available for chemical reactions. The decrease in contact angle from 64.99 degree to 46.52 degree implies that the wettability increased. Based on these results, it can be said that by sandblasting the Y-TZP surface, a foundation was laid for effective resin bonding in a subsequent process.

For the control group of this study, the ceramic bracket was attached to the natural central incisor in three steps according to the routine bracket bonding protocol. The SBS was 9.59 MPa before the thermocycling treatment, and this result satisfies the bonding strength of 6–8 MPa required for bonding between a tooth and a bracket [37].

There was no significant difference in SBS before the thermocycling treatment between group C (9.78 ± 1.94 MPa), group S (9.86 ± 1.33 MPa), group CS (9.16 ± 0.78 MPa), and group CON (9.59 ± 1.77 MPa), and the bonding strengths generally required for the bonding of bracket were satisfied. The bonding steps can be simplified if the 10-MDP-containing adhesive is applied independently on the Y-TZP surface. Therefore, hypothesis 1 and 2 could not be validated. However, after the thermocycling treatment, since the SBS decreased significantly to 4.99 and 4.31 MPa in the 10-MDP-containing adhesive applied groups ST and CST, respectively, the bonding strength generally required for the bracket bonding was not satisfied. In contrast, group CT with 10-MDP-containing primer applied to zirconia retained stable SBS after thermocycling.

The thermocycling treatment is an artificial aging method that is widely used in general to evaluate the long-term bonding strength. Tsuo et al. [24] reported that when an adhesive primer was applied on sandblasted Y-TZP and bonded with the resin, there was no significant difference in the SBS before and after the thermocycling treatment. The result of our experiment is the same in this context. For group C with the 10-MDP-containing primer applied to the sandblasted Y-TZP, the SBS was 9.8 ± 1.8 MPa, and it was slightly reduced to 8.1 ± 1.6 MPa after thermocycling, which does not represent a significant difference. But the SBSs of groups ST and CST were significantly lower than that before aging.

In a single-step self-etching adhesive, an acidic monomer and water are added. However, water can ionize the acidic monomer and dissolve the teeth’s calcium and phosphate ions, giving the adhesive a hydrophilic characteristic by nature. Therefore, if a single-step self-etching adhesive is placed in a moist environment, it absorbs water and because of its solubility, the moisture weakens
the adhesive layer as time passes [38,39]. As the thickness of the bonding agent layer increases, the range of exposure to water increases and becomes more vulnerable to leakage. In case of the Clearfil ceramic primer used in this study, the thickness of the film is so thin that it is difficult to identify the traces after application. In contrast, S\(^6\) bonding agent adhesive forms a rather thick film. Therefore, the results of this study can be explained by the higher SBS reduction in Groups ST and CST (with the adhesive applied to the surface) when compared to group CT, which had the 10-MDP-containing primer applied to the surface. However, our results also contradicted reported findings. It was reported that when sandblasted Y-TZP was bonded with the resin after a self-etch adhesive (Scotchbond Universal Adhesive) was applied, high SBS was obtained even after the thermocycling treatment [40,41]. This was probably because the thermocycling was performed only 2,000–2,500 times, which was too low to be effective.

The ARI scores after bonding failure of sandblasted Y-TZP and the bracket are shown in Table 4, and the baselines of ARI scores are shown in Table 3 [42]. Without the thermocycling treatment, the samples of group C, group S, and group CS all showed an ARI score of 3, indicating that the resin remained on all of the Y-TZP surfaces. This result is also reflected by the stable SBS values (Figure 6). However, after the thermocycling treatment, only group CT had an ARI score of 3, and groups ST and CST showed that less than 50% of resin remained on the Y-TZP surface in 40% of the sample which corresponds to an ARI score of 1. This result is consistent with the decrease in SBSs of groups ST and CST after the thermocycling treatment.

To simplify the bonding steps between the Y-TZP and ceramic bracket and to obtain stable bonding strength, this study applied the 10-MDP-containing agents to the sandblasted Y-TZP and measured the SBS. The results indicate that when the 10-MDP-containing adhesive was used, one step was reduced in the bonding steps and stable SBS was obtained. However, after 10,000 rounds of thermocycling, the bonding strength decreased significantly in case of using the 10-MDP-containing adhesive. Therefore, considering that an orthodontic treatment is continued for a long period after the bracket bonding, it is preferable to apply the 10-MDP-containing primer to the sandblasted Y-TZP before applying the Transbond XT adhesive primer and bonding the bracket with Transbond XT paste, as in the conventional bracket-bonding method. Also, it will be necessary to conduct an additional study to focus on obtaining stable long-term bonding strength while reducing the number of bonding steps.

5. Conclusions

In this study, we applied 10-MDP-containing primer and adhesive to sandblasted Y-TZP to evaluate the effect of 10-MDP adhesive on the bonding of Y-TZP and ceramic brackets to simplify the bonding step. The following conclusions were drawn.

1. When the 10-MDP-containing adhesive was used, only acceptable SBS was obtained while simplifying the bonding step.
2. The long-term stable SBS values were obtained when the 10-MDP-containing primer followed by Transbond XT adhesive primer was applied to Y-TZP.

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