Effects of Dental Adhesive Cement and Surface Treatment on Bond Strength and Leakage of Zirconium Oxide Ceramics

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To evaluate the interactive influence of adhesive materials and surface treatments on bond strength of zirconium oxide ceramics, six types of adhesive resin cements (RelxX ARC (RA), Super-Bond C & B (SB), Linkmax (LM), Panavia Fluoro Cement (PF), Bistite II (BT), and Imperva Dual (ID)), three types of resin-reinforced glass ionomer cements (Xeno Cem Plus (XC), Vitremer Luting (VR), and Fuji Luting (FL)), as well as four types of surface treatments (#600 polishing, sandblasting, silane, and Rocatec system) were used in this study.

Results of this study indicated that all the tested adhesive materials treated with Rocatec system achieved the highest shear bond strength (31.9–67.1 MPa). In particular, the highest shear bond strength value of 67.1 MPa was found for Linkmax and Rocatec treatment combination, while the lowest shear bond strength value of 5.4 MPa was found for RelxX and #600 polishing combination. Furthermore, results showed that Rocatec treatment was an effective way to prevent marginal leakage.

Keywords: Zirconium oxide, Resin cement, Bond strength

INTRODUCTION

Since zirconium oxide ceramics¹¹ provide excellent mechanical properties such as high strength and high toughness, they have been clinically applied as implant body and core material for all ceramic crowns and bridge. Nonetheless, when fabricating CAD/CAM restorations using zirconium blocks, it is critical to achieve cost effectiveness, quality control, and improved machining accuracy. It is reported that zirconium oxide restorations, due to their high fracture resistance, can be cemented using conventional luting agents. However, microleakage caused by inadequate marginal seal often leads to poor prognosis including dislodgement of restorations, secondary caries, and discoloration of cervical area.

For restorations to function for a long time, it is very important to clarify the adhesive behaviors at the interface between the hybrid layer formed on the surface of dentin-pulp complex and resin luting agents, as well as between zirconium oxide ceramics and resin luting agents. Obtaining good adhesion between luting agents and the ceramic surface requires surface pretreatment such as sandblasting, silanization, and Rocatec treatment. Silanization is obtained by the bond of silanol group (Si-OH) on the surface of silica (SiO₂) and methoxy group of silane coupling agent, thereby forming siloxane bond (Si-O-Si) or hydrogen bond.

With zirconium oxide ceramics, little information is available because the adhesive behavior at zirconium-resin luting agent interface has not yet been fully clarified. In light of the paucity of information, findings about how microleakage can be prevented by increasing the bond strength and the clinical soundness of restorations improved by preventing microleakage will therefore shed new light on these important clinical issues.

Therefore, the aim of this study was to evaluate the effects of different surface treatments and different luting agents on bond strength, marginal leakage, and failure mode of zirconium oxide ceramics.

MATERIALS AND METHODS

Materials

Table 1 shows the materials used in this study. Six different adhesive resin cements, namely RelxX ARC (3M ESPE, St. Paul, MN, USA; RA), Super-Bond C&B (Sun Medical, Kyoto, Japan; SB), Linkmax (GC, Tokyo, Japan; LM), Panavia Fluoro Cement (Kuraray Medical, Okayama, Japan; PF), Bistite II (Tokuyama, Tokyo, Japan; BT), Imperva Dual (Shofu, Kyoto, Japan; ID), and three different resin-reinforced glass ionomer cements, namely Xeno Cem Plus (Dentsply-Sankin, Tokyo, Japan; XC), Vitremer Luting cement (3M ESPE, St. Paul, USA; VR), and Fuji Luting (GC, Tokyo, Japan; FL), were used in this research — making it a total of nine different resin luting agents.

For surface treatment methods, they were silani-
zation (Porcelain Bond, Kuraray Medical, Okayama, Japan) and Rocatac treatment (Rocatec™ Jr. Bonding System, 3M ESPE, St. Paul, USA) which consisted of two phases, as well as sandblasting with alumina particles (Rocatec™ Soft, 3M ESPE, St. Paul) and tribochemical treatment (ESPE™-Sil, 3M ESPE, St. Paul).

For the adherend used in this research, it was zirconium oxide ceramic (5.03wt%Y2O3-94.67wt% ZrO2; Nikkato, Tokyo, Japan).

Methods
1) Experimental method
The experiments were performed in accordance with two-way analysis of variance (ANOVA) with the combination of factor A, type of resin luting agent (nine levels: RA, SB, LM, PF, BT, ID, XC, VR, FL) and factor B, surface treatment (four levels: # 600 polishing, sandblasting, silanization, and Rocatec system), making it a total of 36 combinations. Experiments were repeated six times, such that 216 experiments in all were carried out randomly.

2) Specimen preparation
Two square-shaped zirconium blocks of A (10 × 10 × 20 mm) and B (10 × 10 × 10 mm) were fabricated. Bonding surfaces of A and B blocks were polished in one direction with #600 SiC polishing paper. Specimens were ultrasonically cleaned with phosphate gel (K-etchant, Kuraray Medical, Okayama, Japan) for 60 seconds, followed by acetone and refined water for 15 minutes each to remove factors that inhibit adhesion, then dried naturally in the atmosphere. Four different surface treatments were used in this study as follows: (1) Polishing with #600 SiC polishing paper (Buehler®, Sankei, Tokyo, Japan); (2) Sandblasting (Jelenko, NY, USA) with 50-μm

Table 1  Materials used in this study

<table>
<thead>
<tr>
<th>Materials</th>
<th>Code</th>
<th>Manufacturer</th>
<th>Lot number</th>
<th>Composition</th>
</tr>
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<tr>
<td>Adhesive Resin Cements</td>
<td></td>
<td></td>
<td></td>
<td></td>
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<tr>
<td>Rely X ARC</td>
<td>RA</td>
<td>3M ESPE</td>
<td>CWCX</td>
<td>TEGDMA, Bis-GMA</td>
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<td>SB</td>
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<td>polymer: GK1, monomer: GK1, catalyst: GF21</td>
<td>PMMA, MMA, 4-META, TBB</td>
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<td>GC</td>
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<tr>
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<td>KURARAY MEDICAL</td>
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<td>MDP</td>
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<td>BT</td>
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<td>MAC-10</td>
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<td>IMPERVA DUAL</td>
<td>ID</td>
<td>SHOFU</td>
<td>P: 050307, L: 060326</td>
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<td>Resin-reinforced glass ionomer cements</td>
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<td>DENTSPLY-Sankin</td>
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<td>Polyacrylic acid, HEMA</td>
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<td>Universal</td>
<td>00941A</td>
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<td>Rocatec TM Jr Bonding system</td>
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<td>Etching Agent</td>
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<tr>
<td>K-etchant</td>
<td>KURARAY MEDICAL</td>
<td>00308B</td>
<td>phosphate gel</td>
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alumina particles applied perpendicular to the surface at 3.0 bar for 10 seconds at a distance of 10 mm; (3) Silanization (after polishing with #600 SiC polishing paper, polished surface was ultrasonically cleaned in acetone for 15 minutes and dried naturally in the atmosphere), where a drop each of Clearfil New Bond (Kuraray Medical, Okayama, Japan) and activator were mixed, and this mixture applied and allowed to air-dry for 10 seconds; (4) Rocatec system, which entailed sandblasting with 110-μm, silica-coated alumina particles applied perpendicular to the surface at 2.8 bar for 10 seconds at a distance of 10 mm, followed by application of silane coupling agent (ESPE-Sil, 3M ESPE, St. Paul) to the silica-modified zirconium surface.

3) Bonding procedure
Bonding procedures were carried out as follows: (a) For paste-type bonding agents, equal amounts of pastes A and B were dispensed on the mixing paper supplied and mixed for 10 seconds (RA, LM, BT, FL) or 20 seconds (PF); (b) For luting agent consisting of monomer, catalyst, and polymer, four drops of monomer were pipetted into the dappen dish supplied, and to which one drop of catalyst was added and stirred. Subsequently, one cup of polymer was added and mixed for 10 seconds (SB); (c) For powder-liquid type of luting agents, a cup of powder and 1–2 drops of liquid were dispensed on the mixing paper supplied and mixed for 20 seconds (ID, XC) or 30 seconds (VR).

After applying luting agents to the bonding surfaces of both specimens A and B, these two specimens were luted together such that the polishing directions of both specimens A and B, as well as the loading direction, coincided with each other. Luted specimens were placed on a fixed loading device (Seiki Co. Ltd., Tokyo Japan) and loaded in compliance with Japanese Industrial Standards, JIS T6602-1993, under a fixed load of 15 kgf for 15 minutes. Excess luting agent, which exuded from the bonding surfaces after 1–2 minutes’ loading, was removed with a laboratory knife. The specimens were bonded according to manufacturers’ instructions.

Dual-polymerizing resin cements can be polymerized by light or by chemical polymerization. Specimens were not light-polymerized in this study. Bonding procedure was done in accordance with the standard temperature and Class II humidity.

Fig. 1 Compressive shear bond testing device used in this study.

![Compressive shear bond testing device](image)

Fig. 2 Stain infiltration method.

<table>
<thead>
<tr>
<th>Score</th>
<th>Description</th>
</tr>
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<tbody>
<tr>
<td>0</td>
<td>No stain infiltration</td>
</tr>
<tr>
<td>1</td>
<td>Stain infiltrated in less than 1/2</td>
</tr>
<tr>
<td>2</td>
<td>Stain infiltrated in more than 1/2</td>
</tr>
<tr>
<td>3</td>
<td>Stain infiltrated in whole</td>
</tr>
</tbody>
</table>
(23±2°C, 50±5% RH) based on JIS Z 8703. Luted specimens were stored in 0.5% fuchsin solution at 37°C for 24 hours.

4) Compressive shear bond test
Figure 1 shows the compressive shear bond testing machine used in this study. The test was done by connecting the compressive shear bond testing machine to a material testing machine (Servo Pulser EHF-FDI, Shimadzu Co. Ltd., Kyoto, Japan). Specimen A was inserted into the testing machine, and load was applied by means of a sliding block parallel to the bonding surface at a crosshead speed of 0.5 mm/min until failure. Ultimate load to failure of specimen B was recorded. Bond strength (MPa) was calculated by dividing the load (N) at which failure occurred by the bonding area (mm²). After confirming that XR control limit was equally dispersed, the significance of differences was assessed using two-way analysis of variance (ANOVA) and Tukey’s multiple comparison test (p<0.05).

5) Assessment of marginal leakage and failure mode
Marginal leakage was evaluated using Stain Infiltration Method and assessed as follows: Score 0 — No stain infiltration at any bonding surface; Score 1 — Stain infiltrated less than half of the bonding surface from margin; Score 2 — Stain infiltrated more than half of the bonding surface from margin; and Score 3 — Stain infiltrated the whole bonding surface (Fig. 2).

Fractured surfaces of specimens A and B were examined with the naked eye to assess the failure mode, and which was classified as follows: Type I — Interfacial failure; Type II — Cohesive failure or mixed failure of interfacial and cohesive failures; and Type III — Adherend failure.

6) SEM examination
Stain-infiltrated specimens and fractured surfaces were photographed (F5, Nikon Co. Ltd., Tokyo, Japan) and examined (×1). Experiments were repeated three times, and typical cases were used for illustration. A scanning electron microscope (SEM; S-400, Hitachi Co. Ltd., Tokyo, Japan) at ×2000 magnification and 5 kV accelerating voltage was used to examine the specimens of different surface treatments and the representative failure surfaces thereof.

RESULTS

Compressive shear bond strength
Table 2 presents the two-way ANOVA results for different factors and interactions. Significant differences were found for both main factors, factor A (type of luting agent) and factor B (surface treatment), as well as for the interaction between them (p<0.01). Figure 3 then shows the influence of dif-

<table>
<thead>
<tr>
<th>Table 2</th>
<th>Results of two-way ANOVA for adhesive resin and surface treatment</th>
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<td>Factor</td>
<td>s.s.</td>
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<tr>
<td>A: Adhesive Resin</td>
<td>5508</td>
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<tr>
<td>B: Surface Treatment</td>
<td>27783</td>
</tr>
<tr>
<td>A×B</td>
<td>12676</td>
</tr>
<tr>
<td>E</td>
<td>2423</td>
</tr>
<tr>
<td>T</td>
<td>48390</td>
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<tr>
<td>Qij=±2.94</td>
<td>**p&lt;0.01</td>
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</tbody>
</table>

Fig. 3 Effects of dental adhesive cements and surface treatments on bond strength of zirconia ceramics.
ferent luting agents and surface treatments on bond strength. 95% confidence interval was Qij=±2.94. On the overall, compressive shear bond strength values ranged between 5.4 and 67.1 MPa. Highest shear bond strength value of 67.1 MPa was obtained with LM specimen treated with Rocatec system, whereas the lowest value of 5.4 MPa was obtained with RA specimen treated with #600 polishing. In other words, the shear bond strength of the former was 12.4 times higher than the latter. Indeed, as seen in Fig. 3, Rocatec treatment enhanced the bond strength for all the luting agents tested. Apart from Rocatec treatment, the bond strength values yielded with other types of surface treatments differed significantly depending on the luting agent tested. For four luting agents, RA, SB, LM, and PF, their bond strengths in ascending order were #600 polishing < sandblasting < silanization. One luting agent (BT) obtained the same bond strength with both #600 polishing and sandblasting treatments. Further, the bond strengths of four luting agents (BT, ID, VR, FL) were lower following silanization.
Table 5  Assessment results of leakage and failure mode

<table>
<thead>
<tr>
<th></th>
<th>#600</th>
<th>Sand blast</th>
<th>Silane</th>
<th>Rocatec</th>
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<td></td>
<td>Failure mode</td>
<td>Leakage</td>
<td>Failure mode</td>
<td>Leakage</td>
</tr>
<tr>
<td>RA</td>
<td>3</td>
<td>I</td>
<td>0</td>
<td>II</td>
</tr>
<tr>
<td>SB</td>
<td>0</td>
<td>II</td>
<td>0</td>
<td>II</td>
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<td>LM</td>
<td>1</td>
<td>I</td>
<td>0</td>
<td>II</td>
</tr>
<tr>
<td>PF</td>
<td>0</td>
<td>II</td>
<td>0</td>
<td>II</td>
</tr>
<tr>
<td>BT</td>
<td>1</td>
<td>II</td>
<td>0</td>
<td>II</td>
</tr>
<tr>
<td>ID</td>
<td>0</td>
<td>I</td>
<td>0</td>
<td>I</td>
</tr>
<tr>
<td>XC</td>
<td>1</td>
<td>II</td>
<td>1</td>
<td>II</td>
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<tr>
<td>VR</td>
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<td>II</td>
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<td>II</td>
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<tr>
<td>FL</td>
<td>0</td>
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<td>0</td>
<td>II</td>
</tr>
</tbody>
</table>

Fig. 4a  Leakage surfaces and failure modes with respect to adhesive resin cement and surface treatment after insuccation.
than with the other surface treatments.

To compare the differences between each pair of luting agents and surface treatments, Tukey’s multiple comparisons test was performed and the comparison results are listed independently in Tables 3 and 4 respectively. LM and ID specimens showed significant differences in all combinations. SB specimen did not show significant differences between Rocatec treatment and silanization, silanization and sandblasting, as well as sandblasting and #600 polishing. RA, SB, PF, BT, XC, and VR specimens did not show significant differences between sandblasting and #600 polishing, whereas significant differences were found in other combinations. No significant differences were found in FL specimen except for Rocatec treatment and silanization.

Marginal leakage and failure mode
Table 5 shows the assessment results of marginal leakage and failure mode, while Figs. 4a–c show the representative microphotographs thereof. For marginal leakage, score 3 was found in RA specimen treated with #600 polishing, score 1 in LM, BT, XC, and VR specimens treated with #600 polishing, XC
and VR specimens treated with sandblasting, as well as XC specimen treated with silanization and Rocatec system. All other combinations of luting agents and surface treatments exhibited score 0.

For failure mode, type I interfacial failure was observed in RA, LM, and ID specimens treated with #600 polishing, ID specimen treated with sandblasting, as well as VR specimen treated with silanization. Mixed failure of type II was observed in all other combinations of luting agents and surface treatments. Adherend failure of type III was not observed in this study. All resin-reinforced glass ionomer cements showed mixed failure.

**SEM observation of specimens**

Figure 5 shows the scanning electron microscope images of zirconium oxide ceramic surfaces subjected to different surface treatments. Topographic patterns differed greatly among the specimens, and the following surface textures were noted: streaked surface caused by #600 polishing paper, uneven surface caused by sandblasting with aluminum particles, smooth surface caused by silanization, as well as infiltration of silane agent into silica-

![Fig. 4c](image)

Leakage surfaces and failure modes with respect to adhesive resin cement and surface treatment after insuccion.

![Fig. 5](image)

SEM images of different surface treatments.
roughened surface caused by Rocatec treatment.

Figure 6 shows the representative examples of fractured surfaces. For RA specimen treated with #600 polishing, a streaked surface with interfacial failure was shown and which yielded the lowest bond strength value of 5.4 MPa. For LM specimen treated with Rocatec system, it exhibited cohesive failure in luting agent and mixed failure, and whereby the highest bond strength value of 67.1 MPa was obtained.

**DISCUSSION**

**Material and surface treatment**

Tetragonal zirconia polycrystals (TZP) have been commercially available for a wide range of applications, such as optical fiber connectors, precision machine parts, and pulverizer beads. However, due to difficulty in processing, TZP has only recently achieved widespread clinical application in dentistry to bridge frameworks and post cores\(^7\). Unlike brittle materials like conventional dental porcelain, zirconium oxide ceramics provide excellent mechanical properties. In particular, its fracture toughness value is very high\(^7\).

As for luting agents in clinical practice, two types are commonly used—adhesive resin cement and resin-reinforced glass ionomer cement. This is chiefly because both materials exhibit good bonding to tooth structure and metals. However, for good adhesion between luting agents and a ceramic surface, surface pretreatment is required.

To evaluate the interactive influence of luting agents and surface treatments on bond strength of high-strength zirconium oxide ceramics, six different adhesive resin cements and three different resin-reinforced glass ionomer cements, totaling nine resin luting agents, were selected for this research.

In terms of surface pretreatment method, sandblasting and hydrofluoric acid etching produce irregular ceramic surfaces with increased surface area which are necessary for micromechanical bonding. However, with due consideration to the difficulty in handling at chairside and the presence of hazardous components, hydrofluoric acid etching was excluded from this study.

As for silane coupling agents, they are commonly used with binders in clinical practice to increase the bond strength between silica-based ceramics and resin luting agents. In dentistry, r-methacryloxypropyl (trimethylsiloxy)silane (r-MPTS) is used as a silane coupling agent\(^\text{10}\). When applied to the ceramic surface, methoxy group of r-MPTS reacts with silanol group on the ceramic surface to form chemically stable siloxane bond. Silanization has been reported to be an effective means to increase the bond strength not only for silica-based ceramics, but also for zirconium and alumina ceramics\(^\text{8,9}\).

Rocatec system was originally developed as a metal surface modification method for composite resin facings without the use of mechanical retention. However, it has been reported that Rocatec system (silica coating followed by silanization) also improves the bond strength of alumina-based ceramics by means of mechanical and chemical surface treatments simultaneously\(^\text{17}\).

In the present study, marginal leakage was evaluated by assessing stain-infiltrated areas with the naked eye, and then ranked from score 0 to score 3. In particular, this easy method is applicable to conservative dentistry when evaluating the degree of marginal leakage between a restoration and tooth structure.

**Evaluation of compressive shear bond strength, marginal leakage, and failure mode**

To preserve as much healthy tooth structure as possible, minimal intervention (MI) or minimally invasive preparation technique has become more widely accepted and practiced in dentistry. Marginal
leakage due to inadequate bonding often causes secondary caries and discoloration of cervical area, resulting in restoration dislodgement. In a crownbridge preparation with 1 mm cervical reduction, marginal leakage must be prevented with tenacious and durable bonding so as to endure a dynamic, intraoral environment.\textsuperscript{16}

Results of this study revealed that RA specimen treated with #600 polishing showed marginal leakage at the front part of bonding surface, yielded the lowest bond strength of 5.4 MPa, and exhibited interfacial failure. LM, BT, XC, and VR specimens treated with #600 polishing, XC and VR specimens treated with sandblasting, and XC specimen treated with silanization or Rocatec system showed marginal leakage at less than half of the bonding area. The bond strength of these specimens ranged from 5.5 to 41.1 MPa. LM specimen exhibited interfacial failure, while BT, XC, VR, and FL specimens exhibited mixed failure. No marginal leakage was observed in other specimens, and the bond strength of these specimens ranged from 9.8 to 67.1 MPa and exhibiting mixed failure.

No marginal leakage was found in SB, PF, and ID specimens. It is noteworthy that these adhesive resin cements contained the following adhesive monomers: 4-META for SB, MDP for PF, and 4-AET for ID. RA, which exhibited the highest degree of marginal leakage, contained no adhesive monomer. LM and BT contained adhesive monomers of 4-MET and MAC-10 respectively. As for all the three types of resin-reinforced glass ionomer cements tested, they contained the HEMA adhesive monomer.

When investigating the influence of surface treatments on bond strength, bond strength for #600 polishing was within the range of 5.4–28.3 MPa, whereas that for Rocatec treatment was 31.9–67.1 MPa, indicating a significant improvement of bond strength. Consequently, with significantly improved compressive shear bond strength, Rocatec treatment effectively prevented marginal leakage in eight luting agents, except for XC.

Rocatec treatment resulted in the highest compressive shear bond strength for all the resin luting agents tested. This result might be attributed to the formation of a silica coating layer on the zirconium oxide ceramic surface, which was processed with surface impaction of alumina particles modified by silica. This then enabled the silane coupling agent (ESPET\textsuperscript{TM}-Sil, 3M) used for silanization (tribochemical coating) to exhibit its chemical bonding ability.\textsuperscript{12,13} Takakuwa et al.\textsuperscript{21} evaluated the interactive effects of surface treatments (hydrofluoric acid etching, sandblasting, Rocatec System) and luting agents (Panavia Fluoro Cement, Super-Bond C&B) on bond strength of zirconium oxide ceramic surface. Similarly, they reported that the highest bond strength was obtained with Rocatec system, and that surface treatment had a significant influence on bond strength.

Scanning electron microscope images of the differently treated ceramic surfaces are shown in Fig. 4. Topographic patterns differed significantly among the specimens depending on treatment method, whereby each surface treatment demonstrated its own unique peculiarity. Due to polishing with #600 polishing paper, a streaked surface was obtained (#600 polishing, control). Due to abrasion with aluminum oxide particles, a rough irregular surface was obtained (sandblasting). Due to treatment with silane coupling agent, a fine smooth surface was obtained (silanization). With Rocatec treatment, silane infiltrated the roughened surface abraded with aluminum oxide particles modified by silica. In a previous study by Hinokiyama et al.,\textsuperscript{20} it was reported that a silica layer was found, by means of EPMA analysis, on the surface of zirconium oxide ceramics treated with Rocatec system. Similarly, in the present study, silica particles were found to adhere on the surface of zirconium ceramic.

When comparing the bond strengths of resin-reinforced glass ionomer cements with those of adhesive resin cements—with Rocatec treatment for both, resin-reinforced glass ionomer cements showed lower bond strength values than the adhesive resin cements. As seen in Fig. 3, the bond strengths of resin-reinforced glass ionomer cements did not exceed 40 MPa.\textsuperscript{27,29} It is noteworthy that bond strength values differed significantly between XC specimen and RA, LM, and ID specimens (resin-based cement), but not significantly between XC specimen and SB, PF, and BT specimens. As for VR and FL specimens, they showed significantly lower bond strength values than all the other resin luting agents tested. XC, which exhibited marginal leakage under all surface treatments, contained only 10 wt% HEMA. This was a relatively smaller HEMA content compared with 20–30 wt% for VR or 20 wt% for FL, thereby accounting for the decrease in bond strength. Therefore, future studies should include these issues for investigation.

The silane agent used for silanization contributes to the bond strength of silica-based ceramics by promoting chemical bonding.\textsuperscript{15,16} Although it is reported that silane treatment also enhances the bond strength of alumina and zirconium ceramics,\textsuperscript{20,21} the bond strength in this study did not differ significantly between silane treatment and sandblasting, and that the bond strength of silane treatment was lower than that of Rocatec treatment.

Silanization is achieved by the bonding of silanol group on the silica surface with the methoxy group of silane coupling agent, thereby forming siloxane bond or hydrogen bond. However, in the case of zirconium, the methoxy group of silane coupling agent
did not form any bond to the surface of zirconium. This might thus explain for the lack of improvement in bond strength of zirconium. Uo et al.\textsuperscript{30} reported that the bond strength of zirconium-based ceramic (Denzir) pretreated with silanization using phosphate resin cement was lower than that of silica-based ceramic (Empress), indicating that silanization did not improve the bond strength of zirconium oxide ceramics. Therefore, the results of this study were in agreement with those of Uo et al.\textsuperscript{30}. Further, Kajiwara et al.\textsuperscript{30} showed that sandblasting followed by silanization significantly increased the bond strength of zirconium oxide ceramics compared with #600 polishing or sandblasting, and that the silane agent improved the wettability of bonding surface.

Unlike other adhesive resin cements tested, SB specimen showed no significant difference between Rocatec treatment and silanization. Further, SB specimen yielded the highest bond strength among the adhesive resin cements when treated with #600 polishing. Other adhesive resin cements contained 60–80% fillers, whereas SB contained no fillers but 4-META adhesive monomer. The adhesive monomer enabled SB to bond to any pretreated surface, thereby rendering the latter as an easy-to-use material in clinical practice\textsuperscript{31}. In view of the results obtained in this study for SB, further investigations to evaluate the interactive influence of adhesive monomer and r-MPTS-treated surface are indicated.

Sandblasting is an effective technique to improve bond strength by producing rough irregular surfaces with increased surface area necessary for micromechanical bonding. Sandblasting a porcelain surface, however, is likely to induce crack initiation, thus resulting in deteriorated mechanical properties of the material. Phase transformation of zirconia from tetragonal phase to the monoclinic phase is associated with volume expansion. This volume expansion stemming from the transformation causes a crack sealing compressive stress, which is why zirconium exhibits relatively high fracture toughness and strength, as well as the ability to resist crack propagation. On this ground, sandblasting is thought to be effective for zirconium oxide ceramics\textsuperscript{29}.

With adhesive resin cements, results from this study revealed that bond strength values were higher for sandblasted specimens than for #600-polished specimens. On the other hand, with resin-reinforced glass ionomer cements, no significant differences were found between sandblasted specimens and #600-polished specimens, which was consistent with the results of Uo et al.\textsuperscript{30}.

With adhesive resin cements, sandblasting served to prevent marginal leakage—which was not achieved in the case of resin-reinforced glass ionomer cements. This could be due to the different adhesive monomers contained, in particular the action of HEMA.

In general, bond strength decreases with increase in the area of marginal leakage, eventually culminating in interfacial failure between ceramic and luting agent. For RA specimen treated with #600 polishing, it yielded the lowest bond strength of 5.4 MPa in conjunction with the following observations: stain infiltrated the whole bonding area, and that interfacial failure was exhibited with a streaked surface topography. For quite a number of specimens in this study, whereby stain infiltrated less than half of the bonding area, they obtained bond strengths in the range of 5.5 to 41.1 MPa. As for failure type, it was either interfacial failure or mixed failure. For LM specimen treated with Rocatec system, it yielded the highest bond strength in this study. As for failure type, it exhibited mixed failure of cohesive failure within cement and interfacial failure\textsuperscript{32,33}. Differences in bond strength might be explained by the compatibility between luting agent and surface treatment, as well as the interaction of adhesive monomer.

Clinical application
Zirconium oxide ceramics have been in clinical use as core materials for posts and frameworks for bridges. Alongside their excellent mechanical properties such as high strength and high toughness, zirconium oxide ceramics offer optimal biocompatibility and desirable optical properties. Against this lineup of advantages, it can be foreseen that zirconium will become an increasingly popular choice for metal-free esthetic restorations and patients with metal allergy. Although the Rocatec system has been traditionally used as surface treatment to bond metal frameworks or high-strength ceramics to composite facing materials, it has been proven to be an effective surface treatment for intaglio surface of metal frameworks\textsuperscript{35-36}.

Adhesive resin cements are used to prevent the dislodgement of restorations and to achieve a strong and durable bond between restorations and tooth structures. Therefore, it is highly critical to choose the best combination of resin luting agent and surface treatment to the end of avoiding marginal leakage and secondary caries formation. In this light, durability test of luting agents simulating an actual dynamic intraoral environment will help to establish a safe and sound clinical protocol for zirconium oxide ceramic restorations.

CONCLUSIONS
This study evaluated the interactive effects of luting agents and surface treatments on shear bond strength, marginal leakage, and failure mode of zirconium oxide ceramics, using six different adhesive resin cements, three different resin-reinforced
glass ionomer cements, and four different surface treatments (#600 polishing, sandblasting, silanization, and Rocatec system).

Within the limitations of this in vitro study, the following conclusions were drawn:

1) Compressive shear bond strength for zirconium oxide ceramic differed significantly depending on luting agents and surface treatments. The highest shear bond strength value of 67.1 MPa was achieved with Linkmax treated with Rocatec system, whereas the lowest value of 5.4 MPa was achieved with RelyX treated with #600 polishing.

2) Rocatec treatment caused all the tested resin luting agents to achieve high bond strength values of 31.9 – 67.1 MPa.

3) Marginal leakage was not found in Super-Bond C&B, Panavia Fluoro Cement, and Imperva Dual under all conditions. Interestingly, marginal leakage was found in Xeno Cem Plus treated with Rocatec system.

4) Type I interface failure was found in RelyX, Linkmax, and Imperva Dual treated with #600 polishing, Imperva Dual treated with sandblasting, and Vitremer Luting treated with silanization. Mixed failure of type II was found in all other combinations of resin luting agents and surface treatments. Adherend failure of type III was not found in this study.

Results of this study clarified that Rocatec treatment significantly improved the bond strength of all the resin luting agents tested. In addition, Rocatec system also prevented marginal leakage except for Xeno Cem Plus (resin-reinforced glass ionomer cements), thus indicating a need for careful selection of luting-agent-and-surface-treatment combination to achieve optimal bond strength.

REFERENCES


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