The Effect of Resin Cement Type and Thermomechanical Aging on the Retentive Strength of Custom Zirconia Abutments Bonded to Titanium Inserts

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Purpose: The purpose of this study was to investigate the effect of resin cement type on the retentive strength of custom zirconia abutments bonded to titanium inserts. Materials and Methods: Sixty implant (4.3 mm diameter and 11.5 mm length) and custom zirconia abutment (15 mm height and 2 mm wall thickness) pairs were used to form six groups (n = 10 each). Three different resin cements were used to bond the zirconia abutments and titanium inserts: Panavia F 2.0, Zirconite, and Multilink Hybrid Abutment. Control groups and thermomechanically aged groups were formed. Specimens were subjected to tensile test to failure, and the retention values were analyzed by two-way analysis of variance (ANOVA). Results: Two-way ANOVA showed a significant effect of the cement type and thermomechanical aging (P < .05). Regardless of the cement, control groups showed significantly (P < .05) higher retentive strength than the thermomechanically aged groups. Comparing the resin cements, the retentive strength of Zirconite, aged or not, was higher than that of Panavia F 2.0 and Multilink Hybrid Abutment; no significant differences between the latter two cements were found. In the Zirconite group, adhesive failure occurred mostly between the titanium and the resin cement. In the Panavia F 2.0 and Multilink Hybrid Abutment groups, adhesive failure between the zirconia and the resin cement was observed. Conclusion: Resin cement type had an effect on the retentive strength of custom zirconia abutments bonded to titanium inserts. Thermomechanical aging had a negative effect on the retentive strength. Zirconite adhesively bonded to the zirconia surface of the custom abutment. Differences were noted in adhesive failure types among the cements. Int J Oral Maxillofac Implants 2018;33:523–529. doi: 10.11607/jomi.5920

Keywords: dental abutment, tensile strength, zirconium

Titanium and its alloys have been widely used as abutment materials in implant dentistry and have well-documented biocompatibility¹ and mechanical properties.² High success rates of titanium abutments have also been reported in long-term clinical studies.³,⁴ However, the metallic color of titanium abutments can reflect through soft tissue, especially in thin soft tissue (2 mm or less in thickness) and/or translucent phenotypes.⁵ To achieve optimal esthetics, especially in the anterior region, all-ceramic abutments have been introduced due to their natural color and biologic advantages.⁶ Zirconia, with its superior esthetic properties, excellent biocompatibility, and high mechanical strength, has become a favorable abutment material.⁷⁻⁹ Zirconia abutments can be in the form of standardized (prefabricated or stock) components or customized by computer-aided design/computer-aided manufacturing (CAD/CAM) technology.¹⁰ Standardized zirconia abutments have some disadvantages compared with customized zirconia abutments, including a lower load-bearing capacity and limited ability for individualization.¹¹ CAD/CAM zirconia custom abutments provide optimal design of finish lines, emergence profile, and size and shape of the abutment; reduce the amount of time needed by the dentist; and eliminate abutment selection and preparation.

Customized zirconia abutments can be classified into two categories namely one-piece and two-piece zirconia abutments. One-piece zirconia abutments, made entirely of zirconia, are connected to the implant by the abutment itself. Two-piece zirconia abutments, consisting of a titanium insert and a transmucosal zirconia part, connect to the dental implant via the titanium insert.
In two-piece zirconia abutments, the individually fabricated zirconia part is connected to the titanium insert by way of resin cement, screw, or friction fit. This zirconia abutment design provides a titanium-titaniu

thermomechanical aging

strength of custom zirconia abutments bonded to titanium inserts. The null hypotheses were:

1) there is no difference in the retentive strength of custom zirconia abutments that are bonded to titanium inserts with different cements, and

2) thermomechanical aging has no effect on the retentive strength.

MATERIALS AND METHODS

Test specimens were composed of titanium inserts (Ti-Base, Sirona Dental Systems) that were prefabricated for the selected implant and zirconia abutments that were custom designed for tensile testing.

Sixty implants, 4.3 mm in diameter and 11.5 mm in length (NobelReplace; Nobel Biocare), were embedded in autopolymerizing acrylic resin by using a custom-made positioning device to standardize the implant position within the acrylic resin. The suitable ScanPost (Sirona Dental Systems) was inserted with 15 Ncm of tightening torque on the implant, and Scanbody was mounted on the top of the post. The ScanPost and the Scanbody were scanned using an intraoral camera (CEREC Omnicam, Sirona Dental Systems). The virtual model was calculated by the CAD/CAM software (inLab SW 4.2; Sirona Dental Systems). A custom zirconia abutment was specially designed for tensile testing to failure in the form of a tube with a height of 15 mm and wall thickness of 2 mm (Fig 1). Using this design, 60 identical zirconia parts were milled from a presintered Y-TZP material (incorisZI mesoblocks, Sirona Dental Systems) in the milling unit (CEREC MC XL, Sirona Dental Systems). After the milling process, zirconia abutments were dried and sintered in a calibrated sintering furnace (inFire HTC, Sirona Dental Systems) for 3.5 hours.

After the fabrication process, 60 zirconia part/titanium insert pairs were randomly divided into 6 groups (n = 10 each). Three different resin cements were used to bond the zirconia abutments and titanium inserts: Panavia F 2.0, Zirconite, and Multilink Hybrid Abutment. Luting space was directly determined by the software. Manufacturers and main compositions of the resin cements used in the study are presented in Table 1. Prior to cementation, titanium inserts were screwed on the implants with a torque of 35 Ncm. Screw holes of the titanium inserts were sealed with heavy-body impression material (Hydorise Maxi Heavy; Zhermack). Bonding surfaces of the titanium inserts were air particle abraded with 50-µm aluminum oxide particles at 2.0 bar pressure for 20 seconds at a distance of 10 mm. The titanium inserts and zirconia abutments were cleaned in an ultrasonic bath of distilled water and dried prior to the cementation. After cleaning, the bonding surfaces were protected from contamination. A metal primer (Alloy Primer, Kuraray Europe), recommended by the manufacturer of Panavia F 2.0 to increase the bond strength of resin-based materials to dental metals, was applied on the bonding surfaces of the titanium inserts. The cements were mixed according to the manufacturers’ instructions and applied to the titanium inserts. The abutments were carefully inserted on the titanium inserts considering the rotation and position stops. The two components were seated and pressed together by hand, using a constant pressure. Excess cement was removed. Then, as recommended by the manufacturers, the specimens of the Panavia F 2.0 and Zirconite groups were light cured for 20 seconds from each margin to initiate self-curing, and the specimens of the Multilink Hybrid Abutment group were left to self-cure for 7 minutes. The specimens were stored in a humidifier at room temperature for 24 hours.

Each luting cement group was divided into control and thermomechanically aged groups (n = 10 each). Specimens of the control group were subjected to tensile test to failure using a universal testing machine (Compression/Tension Device, Esetron Smart Robotechnologies) at a crosshead speed of 5 mm/min. Specimens of the thermomechanically aged group underwent mechanical loading (500,000 × 100 N) and thermal cycling (2,000 × 5°C/55°C) in a chewing simulator (Chewing Simulator, Esetron Smart Systems).
Robotechnologies) followed by the tensile test to failure (Fig 2). The load required to dislodge components of each two-piece zirconia abutment was recorded in Newtons, and in previous studies, this was presented as a retention between zirconia abutments and titanium bases.10,12,14 In these types of test specimens, the force applied per area cannot be accurately calculated. After the tensile test to failure, the location of luting cement residue was examined for each specimen using a magnifying glass (Loupe opt-on; Orange Dental) and the type of failure was categorized as follows:

- **Type 1**: cement remained on the titanium insert (adhesive failure between zirconia and resin)
- **Type 2**: cement remained on the zirconia surface (adhesive failure between titanium and resin)
- **Type 3**: cement remained on both components (cohesive failure)

### Table 1 Resin Cements Tested in this Study

<table>
<thead>
<tr>
<th>Product name</th>
<th>Type</th>
<th>Indications</th>
<th>Manufacturer</th>
<th>Chemical composition</th>
</tr>
</thead>
<tbody>
<tr>
<td>Zirconite</td>
<td>Dual-cure</td>
<td>Permanent cementation of zirconia crowns, bridges, inlays and onlays</td>
<td>BJM Lab Ltd, Yehuda, Israel</td>
<td>Bis-GMA, TEGDMA, UDMA oligomer, methacrylated phosphoric acid esters, 4-methacryloxyethyltrimellitic acid; (3-[methacryloyloxy]propyl) trimethoxysilane, photoinitiator, co-initiator, benzoyl peroxide, Ba-Al-borosilicate glass, fumed silica</td>
</tr>
<tr>
<td>Multilink Hybrid Abutment</td>
<td>Self-cure</td>
<td>Extraoral, permanent cementation of ceramic structures made of lithium disilicate glass-ceramic or zirconia on titanium/titanium alloy or zirconia bases</td>
<td>Ivoclar Vivadent AG, Schaan, Liechtenstein</td>
<td>Monomer matrix: dimethacrylate, HEMA Inorganic fillers (approx 36%): barium glass, ytterbium trifluoride, spheroid mixed oxide, titanium oxide</td>
</tr>
<tr>
<td>Panavia F 2.0</td>
<td>Dual-cure</td>
<td>Permanent cementation of metal, ceramic, and zirconia restorations</td>
<td>Kuraray-Noritake Dental Inc, Kurashiki, Japan</td>
<td>Base paste: hydrophobic aromatic and aliphatic dimethacrylate, sodium aromatic sulphinate, N,N-diehtanol-p-toluidine, functionalized sodium fluoride, silanized barium glass Catalyst paste: 10-MDP, hydrophobic aromatic and aliphatic dimethacrylate, hydrophilic dimethacrylate, silanized silica, photoinitiator, dibenzoyl peroxide</td>
</tr>
</tbody>
</table>

Bis-GMA = 2,2-bis(p-(2'-hydroxy-3'methacryloxypropoxy)phenyl)propane; TEGDMA = triethylene glycol dimethacrylate; UEDMA = 1,6-bis(methacryloxy-2-ethoxycarbonylamino)-2,4,4-trimethylhexane; HEMA = 2-hydroxyethyl methacrylate; 10-MDP = 10-methacryloyloxydecyl dihydrogen phosphate.

### Statistical Analyses

Data were analyzed with statistical software (SPSS version 20.0, SPSS Inc). The retention values obtained from the tensile test to failure were statistically analyzed by two-way analysis of variance (ANOVA), with the resin cement type and thermomechanical aging as the independent variables. P values less than .05 were considered to be statistically significant.

### RESULTS

During thermomechanical aging, no decementation, screw loosening or fracture, abutment fracture, or implant fracture were observed in the specimens.

Two-way ANOVA showed that the factors cement type and thermomechanical aging had a significant influence (P < .05), and an interaction between the two factors was found (Table 2). The mean retention...
force values and respective standard deviations of the mean are presented in Table 3. The retention forces of both the Zirconite control and thermomechanically aged groups were the highest (650.73 ± 80.30 N and 524.96 ± 76.60 N, respectively). Regardless of the cement, the control groups showed significantly \( P < .05 \) higher retentive strength than the thermomechanically aged groups. Comparing the resin cements, the retentive strength values of Zirconite, aged or not, were significantly higher than those of Panavia F 2.0 and Multilink Hybrid Abutment. No significant differences in retentive strength were found between Panavia F 2.0 and Multilink Hybrid Abutment.

The distribution of failure types for each group are shown in Table 4. In the Zirconite group, adhesive failures between the titanium and the resin cement mostly occurred (type 2). In the Panavia F 2.0 and Multilink Hybrid Abutment groups, adhesive failure between the zirconia and the resin cement was observed (type 1).

### DISCUSSION

The first null hypothesis of this study, that resin cement type would not have a significant influence on retentive strength of custom zirconia abutments bonded to titanium inserts, was rejected. Significant differences in the retentive strength of the two-piece zirconia abutments cemented with different resin cements were found. Thermomechanical aging resulted in significantly lower retentive strength compared with control groups for all resin cements; thus the second null hypothesis was also rejected.

Limited studies reporting on the retention of the zirconia part and titanium insert of CAD/CAM zirconia abutments are currently available in the literature.\(^{10,12,14}\) These studies reported that the resin cement type had no significant influence on the retentive strength of zirconia and titanium components of two-piece abutments, which is in contrast to the

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**Table 2** Summary of Two-Way Analysis of Variance

<table>
<thead>
<tr>
<th>Source of Variation</th>
<th>Sum of squares</th>
<th>df</th>
<th>Mean square</th>
<th>F</th>
<th>P</th>
</tr>
</thead>
<tbody>
<tr>
<td>Corrected model</td>
<td>886,724.179(^a)</td>
<td>5</td>
<td>177,344.836</td>
<td>17.808</td>
<td>.000</td>
</tr>
<tr>
<td>Intercept</td>
<td>12,942,345.127</td>
<td>1</td>
<td>12,942,345.127</td>
<td>1,299.627</td>
<td>.000</td>
</tr>
<tr>
<td>Resin cement</td>
<td>457,050.648</td>
<td>2</td>
<td>228,525.324</td>
<td>22.948</td>
<td>.000</td>
</tr>
<tr>
<td>Aging</td>
<td>360,365.700</td>
<td>1</td>
<td>360,365.700</td>
<td>36.187</td>
<td>.000</td>
</tr>
<tr>
<td>Resin cement * Aging</td>
<td>69,307.831</td>
<td>2</td>
<td>34,653.915</td>
<td>3.480</td>
<td>.038</td>
</tr>
<tr>
<td>Error</td>
<td>537,759.607</td>
<td>54</td>
<td>9,958.511</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Total</td>
<td>14,366,828.912</td>
<td>59</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Corrected total</td>
<td>1,424,483.786</td>
<td>59</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

\(^{a}R^2 = 0.622\) (Adjusted \(R^2 = 0.588\)).

**Table 3** Comparison of Retentive Strength (N) of Resin Cements

<table>
<thead>
<tr>
<th>Resin cement</th>
<th>Control, mean (SD)</th>
<th>Thermomechanically aged, mean (SD)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Zirconite</td>
<td>650.73 (80.30) Aa</td>
<td>524.96 (76.60) Ab</td>
</tr>
<tr>
<td>Panavia F 2.0</td>
<td>445.59 (108.18) Ba</td>
<td>355.29 (106.55) Bb</td>
</tr>
<tr>
<td>Multilink Hybrid Abutment</td>
<td>529.50 (135.42) Ba</td>
<td>280.58 (77.73) Bb</td>
</tr>
</tbody>
</table>

SD = standard deviation.
The same lowercase letters horizontally indicate that the retentive strength values of the control and thermomechanically aged groups were not statistically significant within the same resin cement group \( P > .05 \).
The same uppercase letter vertically indicate that the retentive strength values of the resin cement groups were not statistically significant within the control and thermomechanically aged groups \( P > .05 \).

**Table 4** Failure Types of the Experimental Groups

<table>
<thead>
<tr>
<th>Resin cement</th>
<th>Control (n)</th>
<th>Thermomechanically aged (n)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Cement residue on the abutment</td>
<td>Cement residue on the Ti base</td>
</tr>
<tr>
<td>Zirconite</td>
<td>8</td>
<td>2</td>
</tr>
<tr>
<td>Panavia F 2.0</td>
<td>–</td>
<td>10</td>
</tr>
<tr>
<td>Multilink Hybrid Abutment</td>
<td>–</td>
<td>10</td>
</tr>
</tbody>
</table>
results of the present study.\textsuperscript{10,14} Modification of surfaces has been shown to significantly influence the retentive strength of two-piece zirconia abutments.\textsuperscript{10,12,14} However, it should be noted that different metal primers and numbers of thermal cycles were used in these studies. Gathering additional information from studies on the retentive strength between titanium abutments and zirconia restorations would be beneficial for understanding the bond between components of two-piece abutments.\textsuperscript{12,15–21} These studies reported that the type of cement, mechanical and chemical modification of surfaces, geometry of the abutment, and luting gap may have a significant effect on retention between titanium and zirconia components of an implant-supported restoration.

In the present study, the effect of resin cement type on the retention between two-piece zirconia abutment components was investigated before and after artificial aging by standardizing remaining factors. The three resin cements investigated were Panavia F 2.0, Zirconite, and Multilink Hybrid Abutment. Panavia F 2.0 is a 10-methacryloyloxyethyl dihydrogen phosphate (10-MDP)-containing universal resin cement for use with metal, ceramic, and zirconia restorations. Previous studies showed a good and stable retentive strength of this resin cement to either titanium or zirconia surfaces.\textsuperscript{12,14,15,17,22} Multilink Hybrid Abutment and Zirconite are recently produced resin cements for particular clinical indications. Multilink Hybrid Abutment is a self-curing luting resin for cementation of ceramic structures made of lithium disilicate glass-ceramic or zirconia on titanium inserts in the fabrication of two-piece abutments. Zirconite is a dual-curing resin cement recommended for cementation of zirconia restorations. To the authors’ knowledge, there are limited data on the bond strength of these resin cements to various surfaces, such as prepared teeth, metals, zirconia, and glass ceramics. Comparison of the initial retentive strength of the cements used in the present study revealed that of Zirconite to be significantly higher followed by Multilink Hybrid Abutment. However, it should be noted that all cements showed comparable retentive strength values to those of previous research.\textsuperscript{10,12,14} Previous studies also demonstrated that resin cements specifically formulated for implant-supported restorations would provide high retentive strength values.\textsuperscript{10,23} Compositional differences between the tested materials (including monomer formulation; concentrations of sensitizer, initiator, and inhibitor; and different ratios of light/chemical catalysts) may lead to different resistances of the materials under tensile forces. Retentive strength may also be affected by processing variations of tested cements. Manual mixing of two components of the resin cement may cause air bubbles and voids in the material that may compromise retentive strength, while automix syringes can provide a uniform and bubble-free mixture.\textsuperscript{10} Luting gap has been reported to be an important factor influencing the retentive strength of zirconia copings.\textsuperscript{12,15} Ebert et al\textsuperscript{12} reported that zirconia copings with a 30-μm luting gap showed significantly greater retention than those bonded with a 60-μm luting gap. However, film thickness of the resin cement should be considered when using smaller luting gaps to improve retention of zirconia copings.

In addition to initial performance, durability is an essential property of dental restorations under clinical conditions. Therefore, in vitro studies investigating the retentive strength of resin cements commonly use water storage at a constant temperature and thermal cycling to simulate aging of resin-bonded test specimens.\textsuperscript{10,12,14,17,24} These aging conditions are crucial to test the long-term durability of the retention of specimens bonded with resin cement because water absorption and volume expansion may lead to damage of the bonding interfaces.\textsuperscript{10} Studies have shown that different bonding systems are influenced differently by aging in a wet environment.\textsuperscript{22,25,26} However, no study has evaluated the effect of repetitive masticatory forces on the retention of resin cements. Dynamic loading may change the mechanical behavior and long-term durability of resin cements.\textsuperscript{18} Therefore, an artificial aging protocol including simultaneous cyclic mechanical loading and thermal cycling in a chewing simulator was applied in the present study. This simultaneous mechanical and thermal aging process limited the number of the thermal cycles. For resin cements, retentive strength was significantly decreased after aging. In contrast to the findings of the present study, Ebert et al\textsuperscript{12} reported a significant increase of the retentive strength of cemented two-piece zirconia abutments up to 150 days. They explained this retention increase as postpolymerization effects of water storage, including stress relaxation, resin matrix plasticization, and hygroscopic expansion. However, the absence of chewing loads should be considered when interpreting this result. Two rigid components, the titanium insert and zirconia part, of a two-piece abutment are bonded with a relatively weaker resin cement layer, and repetitive stresses may compromise the integrity of this layer by introducing flaws and cracks.

Characteristics of the retentive surfaces of titanium and zirconia components have been reported as critical factors influencing the retentive strength between the two components. Several studies have reported that air particle abrading the surfaces with aluminum oxide particles is one of the most effective methods to increase retentive strength.\textsuperscript{10,12,14,17,24} The results of these studies showed that it is possible to achieve good and durable retention between zirconia and titanium using resin cements when air particle
abrating is used as a pretreatment. In the air particle abrading procedure, 50-µm \(^{10,15,24}\) or 110-µm \(^{14,16-18}\) aluminum oxide particles were used. Although there is no consensus on particle size, air particle abrading of the bonding surfaces with aluminum oxide has been used as a standard method for enhancing retentive strength in two-piece abutments. In the present study, the bonding surfaces of titanium inserts were also chemically conditioned with a metal primer. For enhanced adhesion of the resin cements to titanium surfaces, air particle abrading with 50-µm aluminum oxide particles and coating with metal primer is a recommended protocol.\(^{27}\)

Air particle abrasion of the bonding surface of zirconia abutments has been reported to be an effective method to increase retentive strength for two-piece abutments.\(^{12,14,17,24}\) However, studies have shown that air particle abrading with aluminum oxide particles can cause damage to the zirconia surface, such as flaws, microcracks, or tetragonal to monoclinic phase transformation.\(^{28,29}\) These surface alterations may compromise the mechanical strength and reliability of zirconia.\(^{28,30,31}\) Moreover, the long-term stability of zirconia is susceptible under intraoral conditions—hydrothermal, mechanical, and chemical.\(^{29,32-34}\) Therefore, in the present study the zirconia abutment bonding surface was not air particle abraded as in other studies on retention between the components of two-piece zirconia abutments.\(^{10,12,14}\) No surface treatment after sinterization was also recommended by the manufacturer of the zirconia abutment. Wegner and Kern\(^{25}\) reported that the resin cements may seal the roughened zirconia surface, thus preventing any negative effects of surface treatment; however, to date the effect of thermomechanical cycling on zirconia surface defects generated by air particle abrading has not been studied.

The modes of failure of Zirconite were predominantly adhesive failure between the titanium insert and resin cement, as cement remained on the zirconia abutment, which may indicate high adhesion between the cement and zirconia even though the zirconia surface was untreated. As the cement properties are determined by the degree of monomer conversion,\(^{35}\) high retentive strength and good bond of the Zirconite to zirconia surface may be associated with high degree of conversion. In the present study, the cement remained on the titanium surface of all Panavia and Multilink specimens, indicating adhesion to the zirconia ceramic was the weakest link for these cements. This finding is in accordance with previous studies.\(^{12,14}\) Adhesion of resin cements to the zirconia surface was improved by mechanical and chemical surface modification techniques.

This in vitro study has some limitations. Tensile test to failure was used, which allows functional evaluation of various factors on the retentive strength of zirconia restorations.\(^{36}\) However, this uniaxial tensile test to failure is limited in adequately simulating clinical situations because, intraorally, multidirectional forces are applied on restorations and implants during mastication or bruxism. Therefore, extrapolation of these in vitro test results to in vivo situations must be done with caution.\(^{16}\) The crosshead speed of the tensile test device was selected to be 5 mm/min, as no speed value has been specified for the test.\(^{12,14}\) The thermomechanical aging performed in the study, which simulates approximately 2.5 years of clinical service, is useful for evaluating the performance of different resin cements.\(^{37}\) However, further prolonged cycling experiments and prospective clinical studies are required to evaluate the reliability of two-piece zirconia abutments in clinical use.

CONCLUSIONS

Resin cement type has an effect on the retentive strength of the custom zirconia abutments bonded to titanium inserts. Zirconite provided the highest retentive strength between the zirconia abutment and titanium insert and was adhesively bonded to the zirconia part. Panavia F 2.0 and Multilink Hybrid Abutment were adhesively bonded to the titanium insert part of the custom abutment.

Thermomechanical aging has a negative effect on the retentive strength of the custom zirconia abutments bonded to titanium inserts.

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