Retention of zirconium oxide copings using different types of luting agents

Khalil Aleisa*, Khalid Alwazzan a, Ziad Nawaf Al-Dwairi b, Hani Almoharib c, Abdulrhman Alshabib d, Abdulaziz Aleid e, Edward Lynch f

a Department of Prosthetic Dental Sciences, College of Dentistry, King Saud University, Riyadh, Saudi Arabia
b Department of Prosthodontics, Faculty of Dentistry, Jordan University of Science and Technology, Irbid, Jordan
c Department of Periodontics and Community Dentistry, College of Dentistry, King Saud University, Riyadh, Saudi Arabia
d Department of Restorative Dental Sciences, College of Dentistry, King Saud University, Riyadh, Saudi Arabia
e College of Dentistry, King Saud University, Riyadh, Saudi Arabia
f Warwick Dentistry, Warwick Medical School, University of Warwick, Warwick, UK

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Abstract  Background/purpose: There is limited information in the literature regarding the effect of different luting agents on the bond strength of zirconium oxide (ZrO2) copings to prepared crown. The aim of this in vitro study was to evaluate the ability of different luting agents to retain ZrO2 copings on prepared crowns under laboratory conditions.

Materials and methods: Forty-two extracted human maxillary first premolars were prepared with a flat occlusal surface, and rounded line angles, a 5° taper and approximately 3-mm occluso-gingival height. ZrO2 copings were manufactured with buccal and lingual projections to assist removal of the crown after cementation. All copings were airborne-particle abraded with 50 μm aluminum oxide for 15 seconds. The specimens were randomly distributed into three equal groups (n = 14) and cemented with one of three luting agents: resin-modified glass–ionomer cement, self-adhesive resin cement, and adhesive resin cement. The cemented specimens were thermocycled (3000 cycles, 5–55°C), and then removed along the path of...
Introduction

The relatively high strength, improved mechanical properties,\(^1\)\(^2\) and biocompatibility\(^3\) of zirconium oxide (ZrO\(_2\)) ceramics allow their use as a material for all ceramic crowns and fixed partial dentures (FPDs). However, FPDs made from commercially available ZrO\(_2\) ceramic can only be fabricated by procedures that require scanning or computer-aided design/computer-assisted manufacturing procedures\(^4\) because the sintering behavior of this material does not allow the fabrication of FPDs frameworks by direct sintering on customized dies.\(^2\) The most utilized zirconia in dentistry is yttria-containing tetragonal zirconia polycrystalline.\(^5\)\(^\text{-}^7\) It has been found to withstand cyclic fatigue testing and posterior all-ceramic FPDs of up to five units were reported to have lifetimes comparable to that achieved with metal ceramic restorations.\(^10\)

ZrO\(_2\) is essentially an inert and nonpolar material, and, in spite of its superiority in terms of mechanical performance, there are some inherent problems, including the adhesion to a variety of substrates.\(^6\) For example, acid etchants such as hydrofluoric acid or hydrophosphoric acid do not adequately roughen the surface for micromechanical retention.\(^5\)\(^\text{-}^\text{12}\) Therefore, alternative methods have been explored to bond ZrO\(_2\) such as surface grinding using silicon carbide or aluminum oxide (Al\(_2\)O\(_3\)), particle air-abrasion using Al\(_2\)O\(_3\), or using a diamond bur. These methods create high surface energy, promote microretention, and are generally easy to apply.\(^5\)\(^\text{-}^\text{9}\) However, it has been reported that air particle abrasion might cause microcracks when used with ZrO\(_2\).\(^6\) In addition, the use of a silane-coupling agent that is recommended for glasses and porcelains, in order to improve bonding, did not improve the bond strength of zirconia ceramics because of absence of silica, which is necessary to eliminate the chemical bonding reaction for silanization process.\(^6\)\(^\text{-}^\text{9}\) Therefore, it is apparent that the composition and physical properties of high-strength ZrO\(_2\) differ substantially from silica-based ceramics\(^13\) and require alternative bonding techniques to achieve a strong, long-term, and durable resin bond.\(^14\)

A recent publication\(^15\) evaluated the retentive strength of ZrO\(_2\)-based crowns with several luting agents and different ceramic pretreatments, using a new in vitro model for connection to a crown during retention testing. However, the results may have been affected by the low \(^5\) of taper used.\(^15\) By contrast, some investigators have examined and measured the shear bond strength of different cements on ZrO\(_2\) ceramic surfaces after different pretreatments; these studies provided varying and controversial results.\(^6\)\(^\text{-}^\text{16}\)\(^\text{-}^\text{17}\)

Other studies of shear bond strength to zirconia ceramics have shown that a composite resin cement containing an adhesive phosphate monomer provided significantly increased bond strength values.\(^15\)\(^\text{-}^\text{18}\) The self-adhesive modified composite resin cement represents a new type of cement and was developed with the goal of combining the ease of handling and absence of required pretreatment steps, along with favorable esthetics and firm adhesion to tooth structure.\(^19\) This cement has also demonstrated high shear bond strength to ZrO\(_2\) ceramics under specific conditions.\(^20\)

The purpose of this study was to evaluate the ability of three commonly used different luting agents to retain a representative ZrO\(_2\)-based coping on a prepared tooth crown under clinically simulated conditions following airborne particle abrasion. The research hypothesis was that the retention of ZrO\(_2\) ceramic copings is similar after luting with a resin-modified glass ionomer cement, or a self-adhesive composite resin cement, or a composite resin cement with an adhesive agent.

Materials and methods

Selection of teeth

Forty-two extracted human maxillary first premolars of approximate the same size were collected. The teeth were cleaned of surface debris, placed in 0.5% sodium hypochlorite immediately following extraction for 5 minutes and then stored in tap water that was changed weekly.

Mounting of teeth

To retain the specimens in the acrylic blocks during testing, the root surfaces were notched with an inverted cone bur (Komet, Gebr, Lemgo, Germany) in a high-speed hand-piece. Also, a 0.7 mm diameter hard steel wire was looped through a transverse hole drilled near the apex of each root. The root was embedded into a polyvinyl chloride (PVC) matrix (25 mm in diameter and 40 mm in length) and filled with self polymerized resin (Ortho Resin; Dentsply DeTrey, Konstanz, Germany) up to 2 mm below the mid facial cementoenamel junction. A dental surveyor (J.M. Ney, Bloomfield, CT, USA) was used when mounting the
Preparation of teeth

The tooth with its PVC matrix was secured vertically in a custom-made jig held firmly in the dental surveyor stand base. For the tooth crown preparation, a straight turbine hand-piece with a water jet was fixed on a laboratory milling machine (Milling unit BF 2; Bredent GmbH & Co.KG, Senden, Germany) to ensure the same preparation angle for each specimen and connected with a pressurized water container, and the cylinder with the tooth was held securely vertically and firmly in a surveyor base. The occlusal surface of each mounted tooth was prepared flat 5 mm above the top of the cylinders, using a diamond wheel shape bur (Komet) in a high-speed hand-piece. Occlusal reduction was oriented in a direction perpendicular to the axis of the PVC tube. Using a cylindrical (protapered) shaped diamond bur with a round tip (3069 diamond bur; KG Sorensen, Sao Paulo, Brazil) mounted to the milling machine, the axial wall of the teeth were prepared to a depth of 1.5 mm, with a 5° taper angle from a vertical axis to create an angle of convergence of 10° (Fig. 1). A new rotary instrument was used for each tooth. The resultant preparation had an axial length (occlusogingivally) of 3 mm with a modified chamfer finish line. All the axio-occlusal line angles of each tooth were rounded. Using a digital caliper (Ultra-Cal Mark III; Fowler/Sylvac, Crissier, Switzerland), the prepared teeth were measured mesiodistally (MD) and buccolingually (BL) to serve as a guideline to distribute the specimens into two groups (n = 21 specimens for each group) so that each group could contain similar sizes of teeth. Group size 1 had an MD width of 2.5–4.0 mm, and a BL width of 4.0–5.5 mm. Group size 2 had an MD width of 4.5–6.0 mm and a BL width of 6.0–7.5 mm. To minimize the effect of variations in the preparation procedure, the same clinician prepared all specimens.

Fabrication of ZrO₂ copings

A customized special tray was made for each prepared tooth using a visible-light polymerized acrylic resin (Triad, Dentsply International Inc., York, PA, USA). A special tray adhesive (Pulpdent Corporation, Watertown, MA, USA) was applied to each custom tray. An impression of each tooth was made with polyvinylsiloxane impression material (Virtual; Ivoclar Vivadent AG, Schaan, Liechtenstein) using the respective custom tray. After the impression had set, the trays were removed and the impressions were then poured with type IV gypsum stone (Die Keen; Modern Materials Manufacturing, Inc., St Louis, MO, USA). The master die was recovered from the impression, sectioned and trimmed, and a die hardener material (PDQ die hardener; Whipmix, Louisville, KY, USA) was applied. ZrO₂ copings (Lava Zirconia; 3M ESPE, St Paul, MN, USA) were manufactured using computer-aided design/computer-assisted manufacturing (Lava CNC milling system; 3M ESPE) for all prepared teeth with buccal and lingual projections to assist removal of the crown after cementation (Fig. 2). The internal surfaces of all copings were airborne-particle abraded with 50-μm Al₂O₃ (Strahlmittel abrasives; Renfert GmbH, Hilzingen, Germany) for a maximum of 15 seconds at a pressure of 1.5-bar (Easy Blast; Bego, Bremen, Germany). The copings then were gently air-sprayed, and cleaned in an ultrasonic bath (Transsonic; TechSpan, New Lynn, Auckland, New Zealand) containing isopropyl alcohol (Minuten spray; Arabian Products Factory for medical disinfectant, Riyadh, Saudi Arabia) for 3 minutes. Fresh alcohol solution was used for each group.¹⁴

Cementation of the zirconium copings

The specimens in each group were further divided equally into three cementation groups of 14 each according to the luting agents used. Each cementation group had seven specimens from each size so that each group contained

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Figure 1  Tooth specimen preparation with milling machine.

Figure 2  Occlusal view of zirconium oxide coping with buccal and lingual projections.
relatively similar mean surface areas. Resin-modified glass-ionomer cement (RL: RelyX Luting 2; 3M ESPE AG, Seefeld, Germany), self-adhesive and self-polymerized resin cement (RU: RelyX U200; 3M ESPE AG), and dual polymerized resin cement (RA: RelyX ARC; 3M ESPE AG) were the three luting cements used (Table 1). The first two cements did not require any special treatment of dentin, whereas etchant and bonding were applied for RA specimens. The tooth surface preparation, mixing and handling of the cements were accurately carried out according to the manufacturers’ instructions. For RL cement, the prepared tooth was cleaned thoroughly with a pumice slurry, rinsed with a water spray, and lightly air-dried. The tooth surface was left moist before cementation. The clicker dispenser was depressed to dispense equal volumes of cement pastes on to the mixing pad. The pastes were mixed using a plastic cement spatula for 20 seconds until a uniform color was achieved. A thin layer of cement was applied to the inside surface of each coping. The coping was seated firmly. Finger pressure was maintained for 5 minutes and excess cement was removed using a hand scaler. For RU, and prior to cementation, the prepared tooth was cleaned thoroughly with a pumice slurry, rinsed with a water spray, and lightly air-dried. The tooth surface was again left moist. The clicker dispenser was depressed to dispense equal volumes of cement pastes on to the mixing pad. The pastes were mixed using a plastic cement spatula for 20 seconds until a uniform color was achieved. A thin layer of cement was applied to the inside surface of each coping. The coping was seated firmly. The margins were light polymerized (3M Elissor ESPE, Seefeld, Germany) for 2 seconds and excess cement was removed. Light polymerization was then applied for 40 seconds for each surface. In the case with RA and prior to cementation, the prepared tooth was cleaned thoroughly with a pumice slurry, rinsed with a water spray, and lightly air-dried. The tooth surface was also left moist. Scotchbond Etchant (35% phosphoric acid gel; 3M ESPE, St Paul, MN, USA) was applied to enamel and dentin for 15 seconds. Etchant was rinsed with water for 10 seconds. The excess water was blotted leaving the tooth moist. Immediately after blotting, three consecutive coats of Adper Single Bond Plus adhesive (3M ESPE, Seefeld, Germany) were applied to etched enamel and dentin for 15 seconds with gentle agitation using a fully saturated applicator. Gentle air thinning was used for 5 seconds to evaporate solvents. Light-polymerization was used for 10 seconds/bonding surface. The cement was dispensed from the clicker onto a mixing pad and mixed for 10 seconds. A thin layer of cement was applied to the bonding surface of the coping. The coping was slowly seated. The margins were light polymerized for 2 seconds and excess cement was removed. Light polymerization was then applied for 40 seconds for each surface.

The specimens were left undisturbed on the bench for another 15 minutes. Specimens were placed in a thermocycling machine (THE-1100 Thermocycler; SD Mechatronik GmbH, Feldkirchen-Westerham, Germany) in water baths for 3000 cycles of 5°C to 55°C with a dwelling time of 15 seconds in each bath to attempt to imitate changes in the oral environment. The specimens were then stored in distilled water with a pH of 7 at 37°C for 24 hours in a laboratory oven (Imperial IV; Lab Line Instruments Inc, Melrose Park, IL, USA).

**Retention test of the zirconium-oxide coping**

To test the retention of specimens, a universal testing machine (Instron Model 8500 Plus Dynamic Testing System; Instron, High Wycombe, Bucks, UK) was used. A specially customized chain was made to ensure even distribution of pulling tensile forces using a locking mechanism. The cemented crowns were pulled off along the path of insertion with a crosshead speed of 0.5 mm/minute. The forces required for dislodgment of the crowns were recorded in N.

Statistical analyses of the data were performed by using SPSS v16.0 (SPSS Corp., Chicago, IL, USA). A one-way analysis of variance (ANOVA) was applied to the mean retentive strengths of different cement materials. When a significant cross product interaction was found, a Tukey multiple comparison test was performed to determine which groups were significantly different. All statistical analyses were performed at a 0.05 level of significance ($\alpha = 0.05$).

**Results**

A summary of the dislodging forces for all groups is given in Table 2. Mean coping bond strengths were 440 N, 416 N, and

<table>
<thead>
<tr>
<th>Luting agents</th>
<th>Tooth pretreatment</th>
<th>Type of polymerization</th>
<th>Manufacturer</th>
</tr>
</thead>
<tbody>
<tr>
<td>Resin modified glass-ionomer cement</td>
<td>None</td>
<td>Chemical</td>
<td>3M ESPE AG</td>
</tr>
<tr>
<td>(RelyX Luting 2)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Self-adhesive resin cement (RelyX U200)</td>
<td>None</td>
<td>Dual</td>
<td>3M ESPE AG</td>
</tr>
<tr>
<td>Adhesive resin cement (RelyX ARC)</td>
<td>37% phosphoric acid gel; Primer: water, HEMA, amine, methacryl-magnesium-chelate, stabilizers; Bond: bismethacrylates, HEMA, MAM, amine-diol methacrylate, photoinitiators.</td>
<td>Dual</td>
<td>3M ESPE AG</td>
</tr>
</tbody>
</table>

HEMA = 2-hydroxyethyl methacrylate; MAM = more activated monomer.
360 N for RL, RU, and RA, respectively. Although the RA group had a lower mean value compared to the other groups, the one-way ANOVA showed no statistically significant difference in retention force strength between the three groups (P > 0.05; Table 3). The results for characterization of failure type are presented in Table 4. Overall, the predominant mode of failure was type 3, where the cement was found principally on the copings. This was followed by type 1, where cement was found on both the tooth and the coping. For the cement group RL, 50% of the specimens had cement in the copings followed by about 29% of the specimens with cement principally on the tooth and the coping. In contrast, failure modes for RU were 43% for cement principally on the coping and 43% with cement on both. The group of copings cemented with RA had 72% of the specimens with cement in the coping and 14% with cement on both.

**Discussion**

The results of this study verify the research hypothesis that ZrO2 coping retention was similar for the three commonly used luting agents tested. All three cements were capable of retaining the ZrO2 copings successfully, with no treatment other than airborne-particle abrasion of the internal surface of each coping.

Bond strength to ceramic material is influenced by the polymerization mode of the resin luting agent (visible light-, dual-, or auto-polymerizing), thermocycling and water storage, and the luting agent itself. Long-term water storage and thermocycling are the conditions most often used to test the durability of cements. It was found that water aging played an important role in degradation of resin–cement zirconia–ceramic bonds. Therefore, thermocycling was done to attempt to simulate temperature changes in the oral environment and help evaluate cement degradation. Resin cements are a major part of today’s clinical practice due to their high compressive and tensile strengths, low solubility, and favorable aesthetic qualities. Their major disadvantages include difficult excess cement removal, technique sensitivity, time consuming process, and they are relatively expensive. Resin luting agents are unique in that a polymer matrix forms to fill and seal the tooth-restoration gap whereas other luting choices are true cements derived from mixing a powder and liquid to form a hydrogel matrix. Etching and bonding of the tooth structure will form micromechanical resin tags that increase the retention to tooth tissue and takes advantage of the resin's high tensile strength. The zirconium surface is easily coated with a passive oxide film (ZrO2). As such, the hypothesis that adhesive monomers may react with the zirconia surface as a metal oxide on base metal or alloy has been proposed, which may enhance bonding between the resin luting agent and zirconia. Resin-modified glass–ionomer cement is a hybrid material derived from adding water soluble polymers or polymerizable resins to conventional glass–ionomer cement. These ‘hybrids’ were created in the 1980s in an attempt to overcome the two main drawbacks of conventional glass–ionomer cement: low early strength and high solubility. Overall, resin-modified glass–ionomer cements have superior physical and mechanical properties compared to conventional glass–ionomers.

Both RL and RU cements provide good bond strengths to tooth structure without any pretreatment or bonding agents. As a result, their application is very simple and can be accomplished in a single clinical step, similar to the application procedures of conventional luting agents. In contrast, RA cement manufacturer’s instructions require etching, washing, drying, bonding, and cementing. The aforementioned steps of use are time consuming and technique sensitive and can compromise an ideal application technique of RA cement, which could lead to a decrease in their physical properties.

For better simulation of the clinical environment, investigation of the retentive strength of luting agents should be studied using a pull-off test, with crowns luted to extracted teeth. This testing procedure is complex and technique-sensitive but provides information on the retentive performance of a material. One study showed lower bond strengths after 1 year, but they varied among surface treatments and materials. The fact that the cementation process was carried out using hand-loading

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**Table 2** Means and standard deviations (SD) of forces (Newton) required to dislodge zirconium oxide coping (n = 14).

<table>
<thead>
<tr>
<th>Groups</th>
<th>Mean</th>
<th>SD</th>
<th>95% Confidence Interval for Mean</th>
<th>Lower</th>
<th>Upper</th>
</tr>
</thead>
<tbody>
<tr>
<td>RL</td>
<td>440</td>
<td>105</td>
<td>379–500</td>
<td>276</td>
<td>675</td>
</tr>
<tr>
<td>RU</td>
<td>416</td>
<td>126</td>
<td>343–489</td>
<td>292</td>
<td>735</td>
</tr>
<tr>
<td>RA</td>
<td>360</td>
<td>61</td>
<td>325–396</td>
<td>241</td>
<td>499</td>
</tr>
</tbody>
</table>

* Mean values designated with the same superscript are not significantly different (P > 0.05).

**Table 3** One-way ANOVA.

<table>
<thead>
<tr>
<th>Groups</th>
<th>Sum of squares</th>
<th>df</th>
<th>MS</th>
<th>F</th>
<th>P</th>
</tr>
</thead>
<tbody>
<tr>
<td>Between</td>
<td>0.047</td>
<td>2</td>
<td>0.023</td>
<td>2.282</td>
<td>0.116</td>
</tr>
<tr>
<td>Within</td>
<td>0.399</td>
<td>39</td>
<td>0.010</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Total</td>
<td>0.446</td>
<td>41</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

df = Degrees of freedom; MS = mean square.

**Table 4** Categories for characterization of type of failure after coping removal.

<table>
<thead>
<tr>
<th>Group*</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
</tr>
</thead>
<tbody>
<tr>
<td>RL</td>
<td>4 (28.6%)</td>
<td>1 (7.1%)</td>
<td>7 (50%)</td>
<td>2 (14.3%)</td>
</tr>
<tr>
<td>RU</td>
<td>6 (42.9%)</td>
<td>1 (7.1%)</td>
<td>6 (42.9%)</td>
<td>1 (7.1%)</td>
</tr>
<tr>
<td>RA</td>
<td>2 (14.3%)</td>
<td>1 (7.1%)</td>
<td>10 (71.5%)</td>
<td>1 (7.1%)</td>
</tr>
</tbody>
</table>

* Group 1: cement on both tooth and coping (adhesive); Group 2: cement principally on tooth > 3/4 axial surface (adhesive); Group 3: cement on coping > 3/4 axial surface (adhesive); Group 4: fracture of the tooth without coping separation (cohesive).
pressure without any type of standardized device might have increased the overall standard deviations of the results. By contrast, this procedure was comparable to a true clinical situation in which the cementation pressure is generally, controlled manually.

Investigation of the retentive strength of luting agents should be studied using axial dislodgment forces with crowns cemented on extracted human teeth. Clinically, not only is the adhesive bond strength important, but also the frictional resistance between a prepared tooth and the ceramic crown. Using a taper angle of approximately 5°, where bond strength is the most important factor, may produce different results. Also, only a few significant differences were found between groups. This is primarily based on the high standard deviations of the results from this study.

The findings of the present study are in agreement with the study by Ernst et al., who found that the same resin-modified glass ionomer cement (RL) demonstrated a comparable retentive strength. The composite resin cement, Superbond C & B (Sun Medical, Shiga, Japan) demonstrated the highest median retentive strength, but was not significantly different from Panavia F, RL, Dyract Cem Plus (Dentsply, Konstanz, Germany), and RU. The mean crown removal strengths found in Ernst et al’s study is similar in range to the present investigation for the three types of cements assessed; however, Ernst et al. reported high standard deviations. In addition, a low 5° preparation taper was used in the aforementioned study similar to the taper degrees in the present study. The low angle of preparation taper may increase the retention resistance to crown removal regardless of the type of luting cement used. Also, a similar ZrO2 ceramic system (Lava; 3M ESPE) was used in the Ernst et al. study.

Another investigation by Derand et al. tested different surface treatments and composite resin cements and found that acid etching and airborne-particle abrasion had only a minor influence on bond strengths. The authors also found that the autopolymerizing composite resin cement Superbond C & B exhibited the highest bond strength. Finally, the shear bond strength study by Piwowarczyk et al. evaluated 11 luting cements to a ZrO2 ceramic (Lava) and found that after airborne-particle abrasion, the highest shear bond strength mean value was obtained for Rely X Unicem. The authors also concluded, in contrast with the results from the present study, that it was not possible to achieve a stable bond using resin-modified glass ionomer or with other cements such as zinc phosphate and standard glass ionomer. These aforementioned publications were shear bond strength studies, which do not simulate clinical conditions and the cementation process, a testing design that does not reflect the factors that may affect the performance of the cement. Furthermore, the testing methods and conditions used in these studies were dissimilar, making it difficult to compare the results.

The results of this study may have important clinical implications, since there has been a lack of clear information for standard procedures with cementation of ZrO2 crowns. Discussions were entertained with the manufacturers of the copings and cements to arrive at clear, acceptable procedures for cementation. The processes have been simplified to some extent, since it was apparent that special treatment of the internal surface was unnecessary for the three cements evaluated (other than airborne particle abrasion with ZrO2). It may be that the inherent roughness of ZrO2 ceramics without airborne particle abrasion may be adequate to provide the necessary micromechanical interlocking of the luting agents. However, the limitations of in vitro studies, which do not completely simulate in vivo performance, should be appreciated. Further research is needed to examine various surface treatments, long-term storage, thermocycling, various ZrO2 systems, and different luting cements. In addition, long-term prospective, randomized controlled clinical trials are needed to evaluate the benefits of certain clinical procedures, including for this innovative type of all-ceramic restoration.

During the characterization of failure type, only the axial surfaces were evaluated to determine the type of failure, since the smooth flat uniform occlusal surface did not retain cement in most specimens and most of the time the cement was found in the internal surface of the coping. This is a limitation of the method, and in the future, it may be preferable to create a roughened occlusal surface using the same diamond rotary cutting instrument used on the axial surface.

Limitations of this experiment included finger pressure variability at the time of cementation, although this procedure resembles the clinical situation and one operator cemented all specimens. The surface area of the teeth was not determined, however, efforts were made to categorize the specimens and then redistribute them equally into the three experimental groups. Therefore, these could be considered as relatively arbitrarily equal groups in surface area. Some previous studies did not consider the surface area of specimens and yet recorded the dislodging forces in Newtons.

Within the limitation of this study, it can be concluded that retention of zirconium oxide coping to prepared crown was not influenced by the types of luting agents.

Acknowledgments

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