

Effect of the internal glaze on pull-off bond strength between Rely X Ultimate resin cement and zirconia crown

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Objectives: To evaluate the effect of the internal glaze of Ceramill ZI zirconia crown on pull-off bond strength using RelyX Ultimate resin cement.

Materials and Methods: Twenty-four human upper premolar teeth were obtained. The teeth were prepared to create a flat occlusal surface, 16° total occlusal convergence, 0.8 mm deep chamfer margin, and 4 mm height. All specimens were divided randomly into two groups as internal glaze (ING) (n=12) and control groups (n=12). Each specimen was scanned and designed to fabricate 2 mm thickness crown. Subsequently, zirconia discs were milled and sintered. For the ING group, the internal surface of crowns was glazed by VITA AKZENT Plus glazing spray. The glaze surfaces were etched with 10% HF for 20 seconds, rinsed, cleaned, and air-dried. Single Bond Universal was applied to the specimens. Then, each specimen was cemented with RelyX Ultimate resin cement and the 50 N (Newton) load was applied during cementation. The crown pull-off test was performed by using a universal testing machine. The mean pull-off bond strength between groups was compared using an independent t-test. The tooth surface and zirconia crowns from the crown pull-off test were examined by a light microscope and scanning electron microscope.

Results: Compared to the control group, the mean pull-off bond strength of internal glaze group groups did not increase the bond strength of resin cement to zirconia crown. Both groups show mixed failure mode (adhesive and cohesive).

Conclusion: Within the limitations of this study, using internal glazing under our study design did not improve the bond strength of resin cement to the zirconia crown.

Keywords: crown pull-off test, internal glaze, zirconia crown

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Introduction

All ceramic restorations have more frequently been used for tooth-colored restorations. Zirconia-based ceramics can be used as an alternative to conventional metal frameworks. Zirconia is gaining popularity in full contour crowns and fixed partial denture prostheses because of their superior mechanical properties, that exhibit high flexural strength and fracture toughness [1, 2].

Along with the strength of the material, the long-term success of restoration depends on the success of the cementation procedure [3]. However, the adhesive bond strength of resin cement to zirconia is not optimal. Because of its high crystalline contents, none of a glassy matrix, and the absence of SiO₂, using common hydrofluoric (HF) etching combined with silanization does not create a sufficiently roughened surface for enhanced micromechanical retention [4].

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Zirconium oxide crowns can be cemented using conventional methods recommended by the manufacturers. However, resin bonding between a tooth and the restoration is advocated for improving retention, marginal adaptation, and fracture resistance of the restorations [3]. Obtaining adhesion between the luting agent and ceramic surface requires surface pretreatment [5]. Different methods to promote adequate adhesion between the resin cement and zirconia have been proposed, for example, air-abrasion, selective infiltration etching (SIE), use of phosphate-modified monomer in resin cement, Si-coated aluminum particles (Rocatec, CoJet), silane coupling agent, Plasma spraying, Erbium-doped yttrium aluminum garnet (Er:YAG) or CO₂ laser, or ceramic coating [6] and internal coating technique [7].

The internal coating technique is the internal surface of the zirconia restoration being thinly coated by fusing silica-based ceramic. In this technique, the internal surface of zirconia framework is modified with silica-based ceramic; therefore, the bonding of resin cement to zirconia ceramic may be improved via silane coupling agents [7-11]. To create the coating layer, various techniques such as selective infiltration etching, glazing techniques, and ceramic liners can be used. However, the thickness of the coating layer may influence the fit of the restorations [12]. Unlike the previous studies, the crown appearance was used in this study instead of the zirconia disc. Clinically, not only the adhesive bond strength but also the other factors, such as the diameter of tooth abutment, is important. Although many factors such as occluso-cervical height, total occlusal convergence, and thickness of margin were controlled in this study, each natural tooth has its own characteristic that affects the retention of crown.

Shear bond strength test (SBS), flexural strength test (three-point bending or four-point bending), tensile bond strength test (TBS), micro-tensile bond strength test (MTBS), short-bar fracture toughness test, laboratory simulation of clinical behavior, and others were used in various studies [13]. The advantages of bond strength tests, when properly designed, include reproducibility of results within the same test lab and the ease of conducting the test. Although bond strength tests have different absolute values, they are ranked similarly. The bond strength test utilizing a specimen disc does not accurately reflect the clinical situation [14].

To simulate the clinical condition, crown pull-off tests were done on prepared natural teeth in this study. The glazing spray was chosen for the internal glaze because it was feasible, simple, and time-efficient. Superior bond strength was expected using this material and procedure due to the chemical bonding of the coating layer, primer, and resin used for cementation.

The objective of this study is to evaluate the effect of internal glaze of Ceramill ZI zirconia crown on pull-off bond strength using RelyX Ultimate resin cement.

Materials and Methods

Ethical exemption

Because of the use of non-identifiable human teeth, the research protocols were approved for ethical exemption by the Faculty of Dentistry/ Faculty of Pharmacy, Mahidol University, Institutional Review Board is in full compliance with International Guidelines for Human Research Protection such as the Declaration of Helsinki, the Belmont Report, COMS Guidelines and the International Conference on Harmonization in Good Clinical Practice (ICH-GCP). (COE.No. MU-DT/PY-IRB2020/052.1610)

Preparation of abutments

Twenty-four extracted caries-free and unrestored human upper premolars were cleaned all remnant soft tissue and stored in distilled water. All teeth were extracted from patients in need of extractions as part of their dental treatment plan. The axis of all teeth was adjusted to be perpendicular to the horizontal plane and cemento-enamel junction then roots were embedded in low-shrinkage resin (Figure 1a).

The crowns of all teeth were prepared for zirconia crown with a milling surveying machine (Schick Dentalgerate S master 3, Vacalon, OH, USA). Prepared teeth were resulted in flat occlusal surface, 16° total occlusal convergence (TOC), 0.8 mm deep chamfer margin, and an approximate height of 4 mm (Figure 1b). The preparation design was also in accordance with the other article's guideline for monolithic zirconia crowns [15]. All prepared teeth were randomly assigned into 2 groups for the luting procedure. After preparation, the teeth were stored in distilled water.

The specimens were randomly divided into 2 groups; the control group (n=12) and the internal glaze group (ING) (n=12).

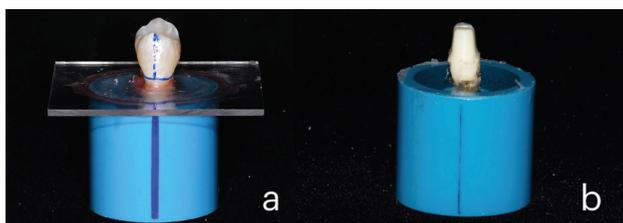


Figure 1 Preparation of abutments (a) The axis of tooth was adjusted to be perpendicular to the horizontal plane. (b) The prepared tooth resulted in a flat occlusal surface, 16° TOC, 0.8 mm deep chamfer margin, and an approximate height of 4 mm.

Fabrication of zirconia crowns

The prepared tooth abutments were scanned with a laboratory-scanning device (Ceramill Map 300, Amann Girrbach AG, Koblach, Austria) and crowns were designed on the system's CAD program (Ceramill Mind). The obtained scanning-data of each abutment was used to design a yttrium oxide partially stabilized zirconia crown (Ceramill ZI disc, Amann Girrbach AG, Koblach, Austria) with a thickness of 2 mm. The cement gap was set at 0.05 mm for the control group and 0.1 mm for ING group. The zirconia crowns were constructed with three outer macro-retention bars to obtain retention in a low-shrinkage resin material (Figure 2).

Twenty-four zirconia crowns were prepared by cutting CAD/CAM milling discs with a milling device (Ceramill Motion, Amann Girrbach AG, Koblach, Austria). Final sintering was performed according to the following program: heat-up phase: from room temperature to final temperature 1,450°C; heating rate 5-10 K/min, dwell time at final temperature for 2 hours, cooling phase: final temperature 1,450°C to room temperature (at least <200°C); cooling rate 5K/min (approximate 5 hours). Subsequently, the specimens were cleaned for 5 min using an ultrasonic bath with distilled water.

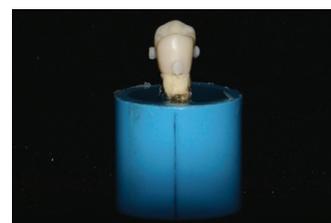


Figure 2 The zirconia crown with three outer macro-retention bars.

Internal glazing of zirconia crowns of ING group

In the ING group, the internal surface of the zirconia crown was sprayed with glassing spray (VITA AKZENT Plus glazing spray, VITA Zahnfabrik, Bad Säckingen, Germany) at the distance of approximately 10 centimeters between the spray head and the surface. Waited briefly until the glaze layer is a dry and uniform whitish layer. The crowns were fired following the ceramic manufacturer's instructions. After that, the glaze surface was etched with 10% HF for 20 seconds, rinsed with tap water for one minute, ultrasonically cleaned in a water bath for 10 minutes, and air-dried.

Determination of surface area

The surface area of each prepared tooth abutment was measured and calculated using AutoCAD software.

Bonding procedures

The adhesive was applied with Singlebond Universal (Single Bond Universal; 3M ESPE, St. Paul, MN, USA). Then, the adhesive was gently air-dried for approximately 5 seconds to evaporate the solvent. Also, the adhesive was applied to the inner surface zirconia crown for 20 seconds and air-dried for 5 seconds.

The specimens were cemented with RelyX Ultimate (RelyX Ultimate; 3M ESPE, St. Paul, MN, USA) and light-cured by an LED dental curing unit for 20 seconds. The 50 N load was applied during cementation. The specimens were stored in distilled water at 37°C for 24 hours. (ISO/TS 11405:2015)

Pull-off bond strength measurements

A crown pull-off test was performed with a universal testing machine (Intron 5566 Intron Ltd., Buckinghamshire, UK). The specimens were tested by pulling out with a crosshead speed of 1 mm/min until debonding of the crown. (ISO/TS 11405:2015) The force at dislodgment was recorded and divided

by the total surface area of each preparation to yield the pull-off bond strength (MPa) (Figures 3a, b).

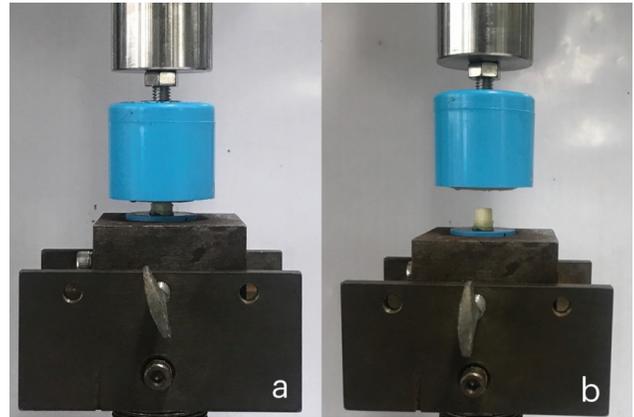


Figure 3 Crown pull-off test with a universal-testing machine (a) Specimen installing (b) The specimen was pulled out with a crosshead speed of 1 mm/min until debonding of the crown.

Fractographic analysis

The tooth abutments and zirconia crowns from the crown pull-off test were examined by a light microscope (Eclipse E400 POL, Nikon Inc., NY, USA). The failure patterns are classified as: adhesive failure (80-100% of failure occurred at the interface between zirconia/dentin and resin cement), cohesive failure (80-100% of failure occurred inside resin and/or zirconia/dentin), and mixed failure (mixed with adhesive and cohesive failure patterns in the same specimen). After that zirconia crowns were randomized selected and dissected for evaluated the glaze layer by scanning electron microscope (SEM).

Statistical analysis

The pull-off bond strength of resin luting cements was calculated by using the statistical software SPSS (Version 25, IBM Corp., Armonk, NY, USA). The normality of the data was analyzed by the Shapiro-wilk test. Independent samples t-test was performed to compare data from a different control group and ING ($\alpha = 0.05$).

Results

Pull-off bond strength

The force at dislodgment was recorded and divided by the total surface area of each preparation to yield the retention value (MPa). The means and standard deviations of force at dislodgment (N) and pull-off bond strength (MPa) of resin luting cement of control and ING groups were shown in Table 1.

According to Shapiro-Wilk were not significant ($p > .05$). There was no violation of the assumption of normality so independent groups t-test was

used for statistical analysis. Comparing between the control and ING groups, there was no statistically significant difference in both forces at dislodgment (N) ($p = 0.928$) and pull-off bond strength (MPa) ($p = 0.143$) of luting cement on an internal glaze of Ceramill ZI zirconia crown.

Fractographic analysis

The tooth surface and zirconia crowns from the crown pull-off test were examined by a light microscope at 10× magnification. Illustrations of the surfaces are shown in failure occur in mixed failure mode (Figures 4a-d).

Table 1 Means and standard deviations of force at dislodgment (N) and pull-off bond strength (MPa) of control and ING groups

	n	Force at dislodgment (N)	Pull-off bond strength (MPa)
Control	12	358.60 ± 27.032	3.72 ± 0.236
ING	12	256.68 ± 22.262	2.83 ± 0.370

Control = control group

ING = internal glazing group

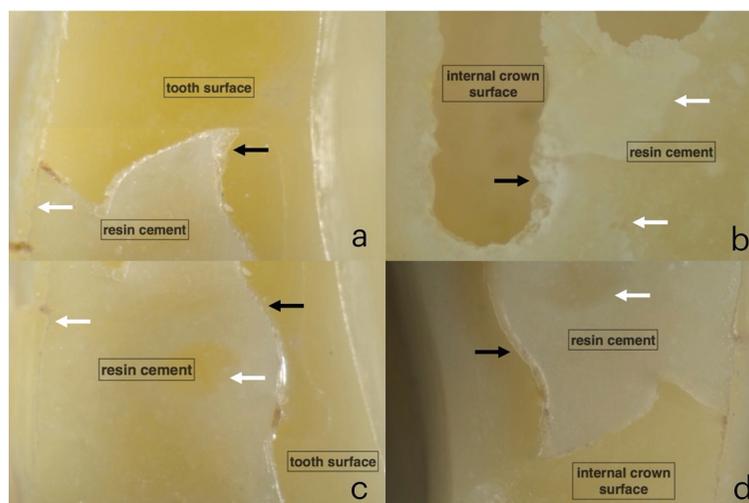


Figure 4 The tooth surface and zirconia crowns were examined by a light microscope at 10× magnification. Adhesive failure between dentin/zirconia and resin cement is shown by a black arrow. Cohesive failure in a resin cement layer is shown by the white arrow.

- (a) Mixed failure mode on tooth abutment surface in the control group
- (b) Mixed failure mode on an internal crown surface in the control group
- (c) Mixed failure mode on tooth abutment surface in the ING group
- (d) Mixed failure mode on an internal crown surface in the ING group

The dissected zirconia crowns were evaluated the glaze layer by scanning electron microscope (SEM). The pictures of the glaze and resin cement layer at a magnification of 50× and 1,000× are shown in the figures (Figure 5a-d).

Discussion

Bonding of resin cement to tetragonal stabilized zirconia ceramics requires surface treatment. Recommended methods often include the use of silica coating and silanization, or phosphate monomer-containing MDP composite resin. Both methods necessitate air-borne particle abrasion prior to cementation that not only need additional equipment but also may detrimentally affect the fatigue properties of zirconia [16]. For these reasons, this study was undertaken to examine the

effect of the application of an etchable, thin glazed layer on the internal surface of the zirconia crown.

For the method, applying glaze to the internal surface of the crown by spraying method can be easily implemented but it may not be a constant enough layer and not cover all internal surfaces. Because of its inner surface appearance, there is a curved angle and narrow opening. Unlike the straight surface such as a disc or block, it was more difficult to control.

Dissected zirconia crowns were examined by scanning electron microscope. Glaze surfaces were found only on the occlusal side that might be related to the spraying technique which caused glazed particles attached only on the occlusal side, not the axial side. These might be caused the cement gap at the axial wall to be thicker than those in the control group. The increasing cement thickness at the axial side results in the retention of the ING group lower than the control group. The studies of cement thickness on tensile bond strength of resin cement found that the decrease in bond strength at greater cement thicknesses with resin cement may be related to the increasing of voids which could have led to crack propagation at lower forces [17, 18].

The thickness of the glaze layer was found almost 100 microns that resemble the defined cement gap. The increasing thickness of the glaze layer resulted in an inadequate space for resin cement. Even though the differences were not statistically significant, the internal coating group had lower bond strength than the control group in this investigation. The findings were influenced by

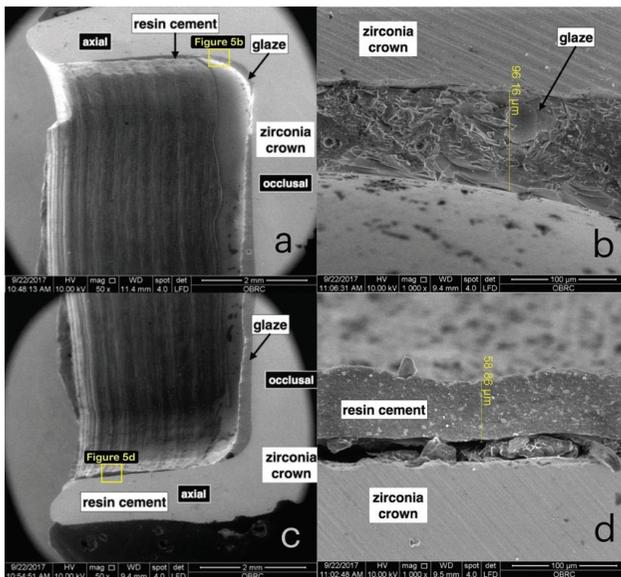


Figure 5 The dissected zirconia crowns were evaluated the glaze layer by SEM. The glazing layer is about 100 microns in some areas.

- (a) Dissected zirconia in the ING group at 50×
- (b) Dissected zirconia at an axial part in the ING group at 1,000×
- (c) Dissected zirconia in the ING group at 50×
- (d) Dissected zirconia at an axial part in the ING group at 1,000×

several factors, including the glazing technique used, which resulted in inconsistency and a thick coating, as well as the improper fit of the zirconia crown on the abutment teeth. In terms of the cement space designed in our protocol, for the ING group, the cement space was set at 100 microns, but the glazing thickness was nearly that value, possibly resulting in an improper fit of the restorations. This incorrect fit could cause adhesive failure. In a clinical situation, an internal crown adjustment may be required, and this may have a negative impact if it is close to the crown margin area. This improper restoration fit might be the explanation for the decreased bond strength of ING group when compared to the control group.

Tensile bond strength values of 1–10 MPa have been reported in retentive strength tests of final crowns luted on extracted permanent teeth with luting agents [14]. Furthermore, shear and tensile procedures used to evaluate adhesive systems with critical bond strengths greater than 15 MPa tend to produce non-uniform stresses during the debonding process, resulting in cohesive fractures in the dentin [19]. Because of the different testing conditions, bond strength values cannot be compared between studies, however, we can remark bond strength values and mode of failure of our study were within the normal limits both in the control and ING groups.

A similar method of the coating was applied in the internal of a zirconia crown in a study by Jo EH *et al* [20], however, instead of the natural tooth, a titanium abutment with a flat occlusal table and 40° TOC was used. A thin uniform coating layer was achieved by spraying for 2-3 seconds at 1.5-2 centimeters (about 15 microns). Another study by Lee ES *et al* [21], had used the dipping method to apply a coating layer on a zirconia crown. They also were able to make a thin layer of about 15-20 microns on the internal surface, however, the disadvantage of this method is the accumulation

of liner at the line angle from the occlusal to the axial surface. Regarding our results, the glazing method should be improved in terms of the technique of spraying. The 10-centimeter distance from the spray head and the restorations cannot make a precise coating in the internal surface of the crown of natural tooth abutment that has a specific shape and form.

Regarding the failure modes, mixed failure modes were observed in ING, indicating that a siloxane network might successfully form between the coating layer and the resin cement, similar to the bond between the resin cement and the tooth surface. When the load is distributed throughout the surface, failure can occur at the interface, as well as at the coating layer and tooth surface. This siloxane bond between the coated surface and the resin has been reported in many studies with a similar internal coating method [9, 20, 21].

One of the advantages of CAD/CAM systems is that restoration design can digitally factor in space for the glaze and cement layers and gradually reduce that space at the margins to ensure a good fit. This study used the spray technique for glazing, the glazing was not spread all over the internal surface. Further study using other methods of glazing is needed to improve the glazing layer. The number of samples should also be examined because various influencing factors must be controlled when performing the test using a crown on a natural abutment tooth to achieve a valid statistical analysis.

Conclusion

Within the limitations of this study, using internal glazing under our study design did not improve the bond strength of resin cement to the zirconia crown.

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