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Original Article

Shear bond strength of dual-cured resin cements on zirconia: The light-blocking effect of a zirconia crown

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Abstract *Background/purpose:* The presence of restorative material between resin cement and the light-curing unit can reduce light transmission. This study aimed to evaluate the effect of the light-blocking effect of zirconia crown on shear bond strength (SBS) between three dual-cured resin cements and the zirconia surface.
Materials and methods: Sixty zirconia specimens were prepared and divided into three groups according to the type of resin cement [RXU (RelyX Ultimate); SC2 (SmartCem2); MEC (Maxcem Elite Chroma)]. Each group was further divided into two subgroups, with or without a 1-mm-thick zirconia crown (n = 10). The specimens were light-cured from five different directions for 20 s each. All specimens were thermocycled 5000 times and subjected to SBS testing, followed by scanning electron microscope examination.
Results: The presence of a 1-mm-thick zirconia crown had no significant effect on the SBS in all resin cements. However, the SBS was significantly affected by type of resin cement. RXU showed the highest SBS (8.35 MPa with crown; 8.57 MPa without crown), followed by SC2 (5.48 MPa with crown; 5.57 without crown) and then MEC (3.37 MPa with crown; 4.04 MPa without crown. Fractured surfaces exhibited varying degrees of mixed failure patterns.
Conclusion: A 1-mm-thick zirconia crown material between the light source and the dual-cured resin cement did not significantly influence the SBS of the resin cements on the zirconia

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substrates. RXU exhibited the highest SBS regardless of zirconia crown coverage. With sufficient light-curing, dual-cured resin cements can be a good choice for zirconia crown cementation.

Introduction

Zirconia has become one of the most commonly used dental restorative materials due to its esthetic and superior mechanical properties.[1] For clinical success of zirconia based restorations, a strong and stable interface between the zirconia and tooth structure should be established using resin based adhesive cement.[2,3] However, the acid-resistant and silica-free surface properties of zirconia hinder traditional surface treatment with hydrofluoric acid etching and silanization.[3,4] In addition to the micro-mechanical surface modification of zirconia surface, chemical modification using resin cements or primers containing 10-methacryloxydecyl dihydrogen phosphate monomer (MDP) has improved the adhesion between zirconia and resin cement. Therefore, phosphate-based primers or resin cements are expected to positively affect bond integrity between zirconia and resin cements, provided that sufficient polymerization is obtained.[5—8] However, light-curing of resin cements is impaired as light penetration significantly diminishes in the presence of overlying material, especially when the material is opaque such as zirconia.[9—17] To overcome the polymerization issue, many resin cements have been developed as dual-cured materials that can be polymerized both by chemical self-curing and light-curing. Previous studies that evaluated the influence of light-curing in dual-cured cements in terms of degree of conversion,[11,15,18-20] micro-mechanical properties,[19,21] and bond strength[16,19,22—24] reported that light-curing promotes superior properties of resin cements.

Zirconia is generally considered a semi-translucent restorative material that attenuates light transmission. Zirconia can possess varying degrees of translucency based on the yttria content. Zirconia with a higher yttria content comprises a greater cubic phase and has an isotropic light refractive index, increasing translucency by reducing light scattering.[11,14,25,26] Despite the increasing use of translucent zirconia due to the esthetic demands of patients without compromising strength, no study has been performed to evaluate whether translucent zirconia influences the bond strength between various dual-cured resin cements and the underlying zirconia substrates. Therefore, the aim of this study was to evaluate the effect of a simulated translucent zirconia crown on the shear bond strength (SBS) between three types of dual-cured resin cements and zirconia. The null hypotheses were that zirconia crown coverage would not affect the SBS and that the SBS would not differ between resin cements.

Materials and methods

Specimen preparation

Sixty zirconia discs (thickness, 2.5 mm; diameter, 14 mm) (Lava Esthetic Fluorescent Full-Contour Zirconia Disc; 3 M, St. Paul, MN, USA) were ground with 600-grit silicon-carbide abrasive paper and cleaned in distilled water for 3 min. After sintering, according to the manufacturer's instruction, each disc was embedded in resin (Bosworth Fastray; custom tray and acrylic base plate material) within an acrylic ring (outer diameter, 26 mm; inner diameter, 24 mm; height, 12 mm). The specimens were randomly divided into three groups based on type of resin cement (Table 1). The specimens were subjected to air-abrasion with 50-μm-grain size aluminum oxide (Al_2O_3) particles at a standoff distance of 10 mm with 3.5 bars of pressure for 15 s. The substrate surface was then rinsed for 30 s, ultrasonic cleaned with distilled water for 3 min, and dried with oil-free air. Each resin cement group was subdivided in half to be either covered by a zirconia crown or not (n = 10 per group).

Zirconia crown fabrication

Zirconia crown was fabricated by CAD/CAM on dental cast of a gelatin capsule (size #5) shaped as a cylinder with a dome on the top. The same type of zirconia used for disc specimen preparations was used for fabrication of 1-mm-thick crown with an internal space of 0.3 mm. The crown served as a standardized simulated clinical situation to evaluate the light-blocking effect of a zirconia crown on bond strength between zirconia discs and resin cements (Fig. 1).

Table 1 Experimental design for each surface treatment on the zirconia specimens.

Zirconia blocks (ground with 600-grit silicon carbide abrasive paper and sintered, n = 60)		
Air abrasion		
Scotchbond Universal + RelyX Ultimate	Prime & Bond Universal + SmartCem2	Optibond Universal + Maxcem Elite Chroma
Light curing with (3 groups, n = 10/each) or without a zirconia crown (3 groups, n = 10/each).		
5000 thermal cycles between 5 and 55 °C, shear bond strength test.		

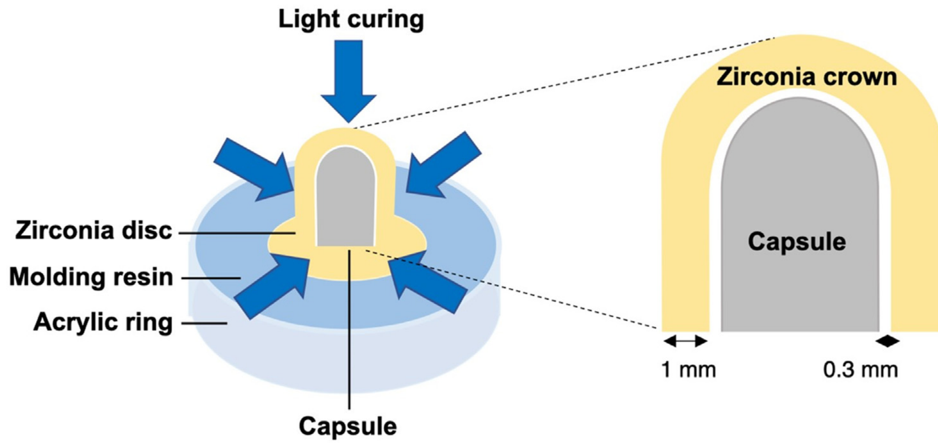

Figure 1 Schematic diagram of the experimental setup. A capsule containing resin cement was placed on a zirconia disc embedded in acrylic resin. Arrows indicate the directions of light irradiation. Dashed lines represent an enlarged view of the capsule with an overlying 1-mm-thick zirconia crown.

Cementation

Each resin cement was mixed according to the manufacturers' instructions (Table 2) and placed in a clear gelatin capsule of size #5 (4.91 mm diameter, 6.2 mm height, 0.089 mm thickness) (Fig. 1). The cement-filled capsule was positioned on the zirconia specimens pretreated with the corresponding adhesive system for each specific resin cement. Table 3 shows the composition of bonding and cement systems used in this study. After careful removal of excess cement with a microbrush, the cements were light-cured from five directions for 20 s per side with or without an overlying zirconia crown, as per manufacturers' recommendations. The five directions comprised one from each of four sides at 90° apart and one perpendicular to the zirconia disc facing the top of the capsule. An LED light-curing unit (Elipar S10, 3 M ESPE, St. Paul, MN, USA) was the light source (Fig. 1). The light-curing unit had a light irradiance of 1514 mW/cm2 and wavelength range of 430—480 nm quantified by a MARC® Resin Calibrator (BlueLight Analytics Inc., Halifax, Canada). After crown removal, the specimens were left at 23 °C ± 1 °C for 1 h and then stored in distilled water at 37 °C for 23 h. The specimens were subjected to 5000 thermal cycles between 5 °C and 55 °C. The dwelling time was 30 s at each temperature, and the transfer time between baths was 5 s.

Shear bond strength testing and surface analysis

The specimens were mounted in the jig of a universal testing machine, and a load was applied to the adhesive interface at a crosshead speed of 0.5 mm/min until failure occurred. The SBS (MPa) was calculated as the ratio of maximum load (in Newtons) required to produce failure to area of the bonded surface (in mm^2). After SBS testing, the debonded zirconia substrates were coated with gold in a vacuum sputter coater (SC7620 Sputter Coater, Polaron Range, Quorum Technologies) and subjected to scanning electron microscopy (SEM) (Apreo 2 SEM; Thermo Fisher Scientific, Foster City, CA USA) to evaluate the failure pattern at 60× magnification.

Table 2 Application of bonding and cement systems.

System	Manufacturer	Application
Scotchbond Universal RelyX Ultimate	3 M ESPE	Scotchbond Universal: A thin coat was applied to the pre-treated surfaces with a microbrush and left to react for 20 s. Then the excess was dispersed with a stream of oil-free air for 5 s. RelyX Utlimate: Applied directly from an Automix syringe into the capsules. Excess paste at the margin was removed with a microbrush.
Prime & Bond Universal SmartCem2	Dentsply Sirona	Prime & Bond: A thin coat was applied to the pre-treated surfaces with a microbrush. The sample was immediately air dried for 5 s. SmartCem2: Applied directly from an Automix syringe into the capsule. Excess paste at the margin was removed with a microbrush.
Optibond Universal Maxcem Elite Chroma	Kerr	Maxcem Elite Chroma: Gel-state color indicator that visually displays the optimal time to remove excess cement. Applied directly from an Automix syringe into the capsule. Remove all uncured, pink-colored excess cement using a microbrush.

Table 3 Compositions of bonding and cement systems.

Brand name	Composition	Manufacturer
Scotchbond Universal	10-MDP, DMA, HEMA, Vitrebond copolymer, fillers, ethanol, water, initiators, silane	3 M ESPE
Rely X Ultimate	Base: Silane-treated glass powder, 2-propenoic acid, 2-methyl-,1,1-[1-(hydroxymethyl) -1,2-ethanediyl] ester, reaction products with 2-hydroxy-1,3-propanediyl DMA and phosphorus oxide, TEGDMA, silane treated silica, oxide glass chemicals, sodium persulfate, tert-butyl peroxy-3,5, 5-trimethylhexanoate, copper (II) acetate monohydrate Catalyst: Silane-treated glass powder, substituted DMA, 1,12-dodecane DMA, silane-treated silica, 1-benzyl-5-phenyl-barbic-acid, calcium salt, sodium p-toluenesulfinate, 2-propenoic acid, 2-methyl-, [(3-metoxypropyl) imino]di-2,1-ethanediyl ester, calcium hydroxide, titanium dioxide	3 M ESPE
Prime & Bond Universal	Phosphoric acid-modified acrylate resin, PENTA, 10-MDP, Active Guard™ technology crosslinker, isopropanol, water, initiator (camphoroquinone, tertiary amine)	Dentsply Sirona
SmartCem2	Urethane DMA, urethane-modified bisphenol A diglycidyl ether, TEGDMA, DMA, PENTA, barium boron fluoroaluminosilicate glass amorphous silica	Dentsply Sirona
Optibond Universal	Glycerol phosphate DMA, HEMA, acetone, ethyl alcohol, disodium hexafluorosilicate	Kerr
Maxcem Elite Chroma	HEMA, 4-methoxyphenol, cumene hydroperoxide, uncured methacrylate monomers, titanium dioxide, pigments,barium-alumina silica glass, fluoroalumina silicate glass, nano-ytterbium fluoride	Kerr

MDP: Methacryloyloxydecyl dihydrogen phosphate; DMA: dimethacrylate; HEMA: hydroxyethylmethacrylate; TEGDMA: Triethylene glycol dimethacrylate; PENTA: pentaerythritol-pentaacrylate phosphate.

Statistical analysis

The Shapiro—Wilk test was conducted to verify the normality of each variable, and Levene's test was performed to assess the equality of variances before data analysis. Two-way analysis of variance (ANOVA) was conducted to examine the effects of zirconia crown and type of resin cement on bond strength. Tukey's post hoc test was performed for comparison of resin cements at a 5% significance level. Statistical analyses were performed using IBM SPSS Statistics, v25 (IBM Corp., Armonk, NY, USA).

Result

The SBS values ranged from 3.37 to 8.57 MPa (Tables 4 and 5, Fig. 2). There were significant differences among the investigated resin cements ($P < 0.001$), although the effect of a zirconia crown on bond strength was not significant ($P = 0.116$). There was no statistically significant interaction between the effects of resin cements and zirconia crown on bond strength ($P = 0.482$). Irrespective of the presence of a zirconia crown, RXU exhibited the highest SBS (8.35 MPa with crown; 8.57 MPa without crown), followed by SC2 (5.48 MPa with crown; 5.57 without crown) and then MEC (3.37 MPa with crown; 4.04 MPa without crown). Regarding the reduction percentage of SBS due to the presence of zirconia crown compared to the values without zirconia crown, SC2 showed the smallest reduction (1.6%), followed by RXU (2.6%) and MEC (16.6%).

Fig. 3 presents representative SEM images of the interfaces between the cements and zirconia specimens from each group (60× magnification). All examined surfaces exhibited varying degrees of mixed failure patterns, including adhesive failure at the interface between the zirconia and resin cement, and cohesive failure within the resin cement.

Discussion

The light-blocking effect of a zirconia crown on the SBS of three dual-cured resin cements on zirconia surfaces was evaluated in this study. The first null hypothesis that zirconia crown coverage would not alter the SBS was accepted because the presence of a zirconia crown failed to produce a significant difference in the tested resin cements. Dual-cured resin cements generally do not obtain sufficient polymerization without adequate light irradiation.[27—29] The amount of light absorbed into resin cement is attenuated by the brand, thickness, and shade of the zirconia restoration - the thicker and darker are the restoration, the greater will be the light attenuation.[9—16] The crown used in this study was made of translucent zirconia with a significantly larger

Table 4 Two-way ANOVA results.

	Sum of squares	df	Mean square	F	P
Cement	230.412	2	115.206	187.365	<0.001
Crown	1.571	1	1.571	2.556	0.116
Cement x Crown	0.910	2	0.455	0.740	0.482

df: degree of freedom; F: F-value; *P*: P-value.

Table 5 SBS values (MPa) of resin cements and SBS reduction (%) by crown coverage.

	With crown	Without crown	Reduction (%) by crown
RXU	8.35 (1.17)	8.57 (0.71)	2.6
SC2	5.48 (0.50)	5.57 (0.58)	1.6
MEC	3.37 (0.85)	4.04 (0.71)	16.6

Standard deviation is shown in parentheses.
SBS: shear bond strength; RXU: RelyX Ultimate; SC2: Smart-Cem2; MEC: Maxcem Elite Chroma.

yttria (Y_2O_3) content (approximately 5 mol%) compared to traditional zirconia. Increasing the yttria content creates more cubic phase; hence, incorporation of more cubic grains with isotropic optical properties reduces light scattering and improves translucency due to relatively fewer grain boundaries compared to the conventional tetragonal form.[11,14,25,26] Sulaiman et al.[11] reported that a more translucent type of zirconia allowed for easier light transmission, thereby achieving a higher degree of conversion.

Based on the findings of this study, light-curing through 1-mm-thick translucent zirconia crowns appears to be effective in terms of bond strength. The light-blocking effect of the interposed zirconia crown was almost negligible in RXU (2.6%) and SC2 (1.6%), while MEC exhibited a 16.6% nonsignificant reduction in mean SBS. These findings were in line with a previous study by Lee et al.[16] who found no significant difference in bond strength under 1-mm-thick zirconia (0 vs. 1 mm) compared with light-cured in the absence of overlying material. However, the bond strength of dual-cured resin cements to zirconia was reduced with an increase in the thickness of the material (1.5 mm, 2 mm).

It has been recommended to increase the light-curing period as the thickness of interposed material between the resin cement and light source increases in order to compensate for the impaired light transmittance.[30] In the present study, the resin cements were adequately light-cured for 20 s from each of five directions, in accordance with the manufacturer' recommendations, in order to test the light-blocking effect of zirconia crowns. The lack of such an effect of 1-mm-thick zirconia material on resin cements could be attributed to the thickness and translucency of the chosen zirconia, as well as light-curing time with light from multiple directions. After SBS testing, the fractured surfaces revealed a variety of mixed failure features, including adhesive failure at the zirconia-resin cement interface and cohesive failure within the resin cement, leaving resin residue on the zirconia substrate. There was no clear correlation between bond strength and failure presentation.

The SBS values of the three dual-cured resin cements were significantly different, regardless of interposition of a zirconia crown during light-curing. As a result, the second null hypothesis, that resin cement type has no effect on SBS was rejected. RXU exhibited the highest SBS irrespective of zirconia crown coverage, followed by SC2 and then MEC. This may be attributed to the difference in composition of the preconditioning adhesive systems and the corresponding resin cements. MDP is a versatile phosphate monomer that has become an essential chemical component of newly developed adhesive systems due to its durable chemical bond with zirconia.[5–8] However, MDP is not the only material incorporated in Scotchbond Universal used for preconditioning of the zirconia surface prior to cementation

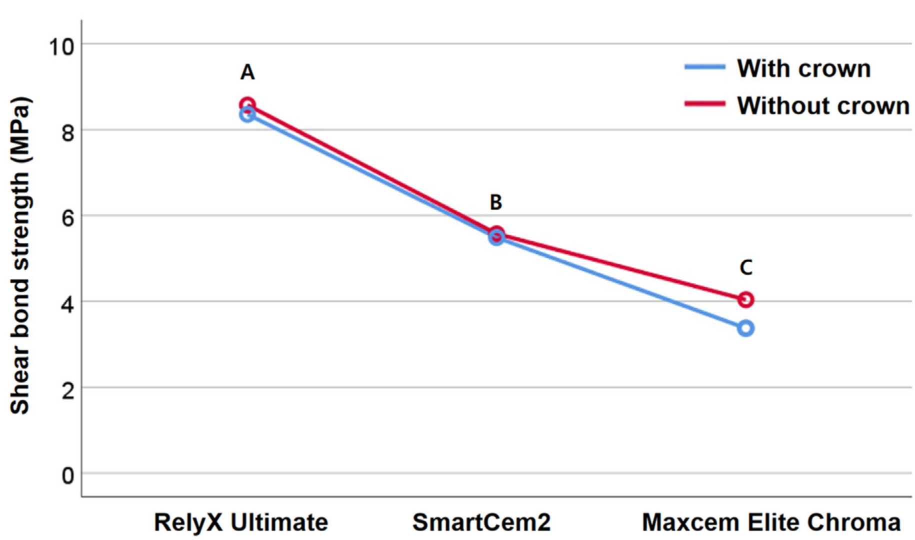

Figure 2 Shear bond strength values (MPa) of resin cements with and without zirconia crown. Different letters indicate a statistically significant difference between resin cements.

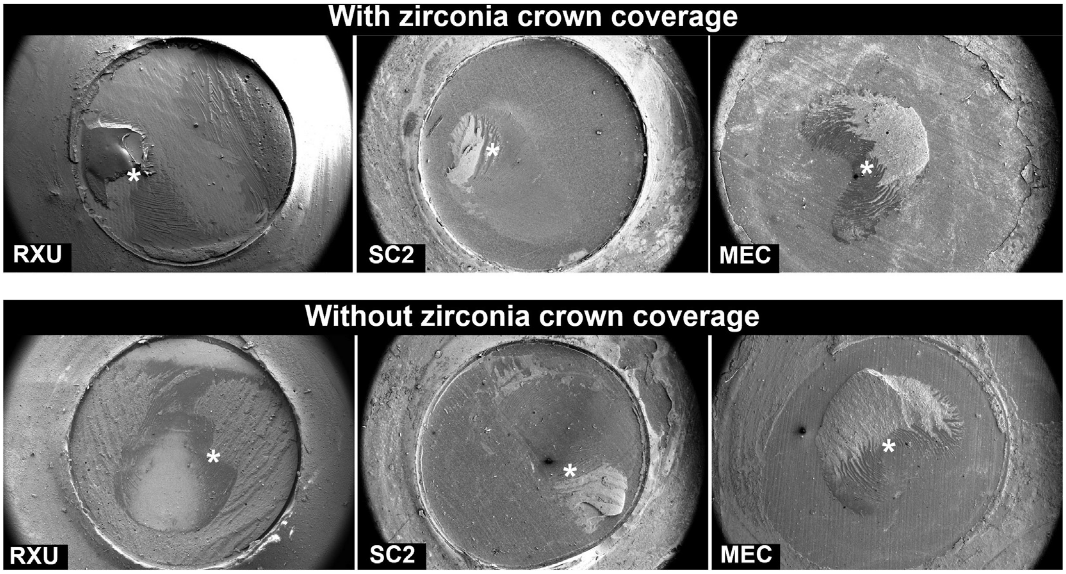

Figure 3 Representative SEM images of the fractured zirconia specimens with (top) and without (bottom) zirconia crown coverage. Asterisks indicate the junction between the resin cement and zirconia. Varying degrees of mixed failure pattern of adhesive failure at the interface between zirconia and resin cements, and cohesive failure within resin cement were observed in all examined surfaces.
RXU: RelyX Ultimate; SC2: SmartCem2; MEC: Maxcem Elite Chroma.

with RXU as the other adhesive systems (Prime & Bond Universal for SC2 and Optibond Universal for MEC) also contain phosphate monomers. Despite the comparable chemical function of phosphate monomers, similar but distinct versions of phosphate monomers in an adhesive system could have varied impacts on the zirconia-resin cement interface. For example, RXU has been shown to have a higher bond strength with zirconia in previous investigations using Single Bond Universal.[31—33]

The findings of this study should be carefully interpreted because the bond strength of dual-cured resin cements on zirconia was compared with and without a 1-mm thick translucent zirconia material. Although light curing of dual-cured resin cements is recommended to maximize polymerization and achieve optimal properties,[34] positioning of the curing light close to the resin cement is not always feasible in the limited space of the oral cavity. Furthermore, light attenuation is caused by the contours of the tooth structure, where the thickness of the crown is frequently greater than 1 mm towards the coronal surface, particularly around the cusp tips. Therefore, clinicians must provide adequate light curing time to compensate for impaired light transmittance since light intensity is inversely related to the thickness of restorative material and the distance between the curing unit and a material to be polymerized.[35] Dual-cured resin cements can be a good choice for zirconia crown cementation when exposed to adequate light, as demonstrated in this study. Further investigation is necessary with different types and thicknesses of zirconia and with different intensities of curing lights and curing times to provide in-depth clinical guidelines for ideal cementation of zirconia restorations using dual-cured resin cements.

Within the limitations of this study, the presence of a 1-mm-thick zirconia crown between the light source and the dual-cured resin cement did not significantly influence the SBS of the resin cements on the zirconia substrate. RXU exhibited the highest SBS, irrespective of the interposition of a zirconia crown. Dual-cured resin cements can be a good choice for zirconia crown cementation when adequate light-curing is provided.

Declaration of competing interest

The authors have no conflicts of interest relevant to this article.

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