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Effects of Light-Curing on the Immediate and Delayed Micro-Shear Bond Strength between Yttria-Tetragonal Zirconia Polycrystal Ceramics and Universal Adhesive

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Purpose: To evaluate the effect of light-curing on the immediate and delayed micro-shear bond strength (μSBS) between yttria-tetragonal zirconia polycrystal (Y-TZP) ceramics and RelyX Ultimate when using Single Bond Universal (SBU).
Materials and Methods: Y-TZP ceramic specimens were ground with #600-grit SiC paper. SBU was applied and RelyX Ultimate was mixed and placed on the Y-TZP surface. The specimens were divided into three groups depending on whether light curing was done after adhesive (SBU) and resin cement application: uncured after adhesive and uncured after resin cement application (UU); uncured after adhesive, but light cured after resin cement (UC); and light cured after adhesive and light cured resin cement (CC). The three groups were further divided depending on the timing of μSBS testing: immediate at 24 hours (UUI, UCI, CCI) and delayed at 4 weeks (UUD, UCD, CCD). μSBS was statistically analyzed using one-way ANOVA and Student-Newman-Keuls multiple comparison test ($P<0.05$). The surface of the fractured Y-TZP specimens was analyzed under a scanning electron microscope (SEM).
Result: At 24 hours, μSBS of UUI group (8.60±2.06 MPa) was significantly lower than UCI group (25.71±4.48 MPa) and CCI group (29.54±3.62 MPa) ($P<0.05$). There was not any significant difference between UCI and CCI group ($P>0.05$). At 4 weeks, μSBS of UUD group (24.43±2.88 MPa) had significantly increased over time compared to UUI group ($P<0.05$). The SEM results showed mixed failure in UCI and CCI group, while UUI group showed adhesive failure.
Conclusion: Light-curing of universal adhesive before or after application of RelyX Ultimate resin cement significantly improved the immediate μSBS of resin cement to air-abrasion treated Y-TZP surface. After 4 weeks, the delayed μSBS of the non-light curing group significantly improved to the level of light-cured groups.

Key Words: Delayed micro-shear bond strength; Immediate micro-shear bond strength; Light-curing; Resin cements; Universal adhesive; Yttria-stabilized tetragonal zirconia polycrystals ceramic

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Introduction

Due to their excellent physical strength and esthetics, restorations fabricated with all-ceramic materials are becoming popular in dentistry[1,2]. Yttria-tetragonal zirconia polycrystal (Y-TZP) is commonly used because of its high fracture strength and toughness, but it is difficult to create a durable bond compared to silica-based ceramics as it is resistant to hydrofluoric acid-etching[3].

A reliable and durable bond formation is critical to the clinical success of all dental restorations including Y-TZP restorations[4,5]. For ceramic restorations, the use of resin cements is currently recommended[6]. According to previous studies, when cementing Y-TZP restoration, air-abrasion contributes to micromechanical bonding, and 10-methacryloyloxydecyl dihydrogen phosphate (MDP) contributes to strong and durable bond[7-11]. MDP is a bifunctional organic hydrophobic chain molecule. One end has a hydrophilic phosphate ester group, which bonds strongly to Y-TZP[12,13]. At the other end, vinyl groups react with the resin cement monomers during copolymerization[12].

Recently, a new type of bonding agent has been introduced to simplify conditioning procedures for both the tooth and restoration surfaces. This new bonding agent has been named "universal", as it can be used as a total-etch, self-etch or selective-etch adhesive, and can bind to the tooth as well as different types of restoration such as ceramic, resin, and metal[14]. Evaluations of this bonding system on resin cement to zirconia have shown good results as it contains MDP[10,15].

In the past, when adhesives were applied after silane treatment on silica based ceramics, it was traditionally recommended not to light cure the adhesive applied to the restoration inner surface[16] because the thick cured adhesive layer may cause misfit and interfere with full seating of the restoration during cementation. However, there have been concerns whether such uncured adhesive layer may interfere with adequate adhesion.

Recently, new advanced resin cement has been introduced to complement the universal adhesives. According to the manufacturer, the new resin cement contains a dual-cure activator that allows chemical curing of the universal adhesives when used together.

Therefore, the aim of this study was to evaluate the effects of light-curing on the immediate and delayed micro-shear bond strength between Y-TZP ceramics and RelyX Ultimate (3M ESPE, St. Paul, MN, USA) when using Single Bond Universal (SBU; 3M ESPE). The null hypothesis tested was that light curing does not influence the bond strength to Y-TZP ceramic.

Materials and Methods

1. Specimen Preparation

Partially sintered Y-TZP blocks of 97% zirconium dioxide stabilized with a 3% Yttria-Lava Frame (3M ESPE) were sectioned into 4 mm thick ceramic disks of 19 mm diameter and 100 mm height using a low concentration diamond blade (Allied High Tech Products Inc., Compton, CA, USA). The surfaces were polished and ground with 600-git silicon carbide abrasives under running water. Before sintering according to the manufacturer's instructions, the Y-TZP ceramic blocks were cleaned ultrasonically in distilled water for three minutes. The specimens were embedded in polyethylene molds of 19 mm inner diameter, 21 mm outer diameter, and 12 mm height. One side was left exposed for the bonding procedure.

The specimens were divided into three groups depending on whether light curing was done after adhesive (SBU; 3M ESPE) and resin cement application: uncured after adhesive and uncured after resin cement application (UU); uncured after adhesive, but light cured after resin cement (UC); and light cured after adhesive and light cured resin cement (CC). The three groups were further

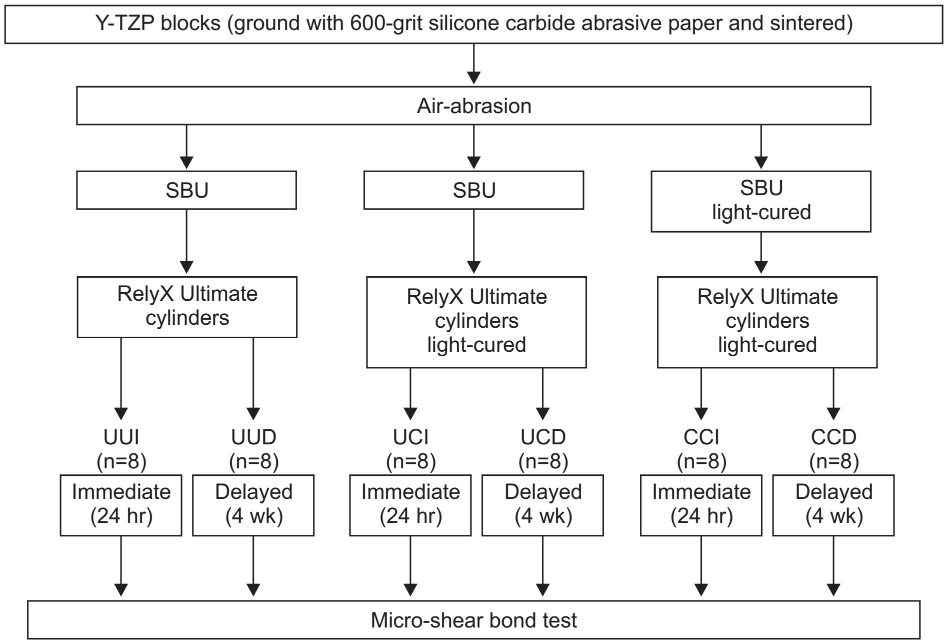

Fig. 1. Experimental design depending on whether light curing was done before or after resin cement application on yttria-tetragonal zirconia polycrystal (Y-TZP) specimens in this study. SBU: Single Bond Universal, UUI: uncured after adhesive and uncured after resin cement application (immediate testing at 24 hours), UUD: uncured after adhesive and uncured after resin cement application (delayed testing 4 weeks), UCI: uncured after adhesive, but light cured after resin cement (immediate testing at 24 hours), UCD: uncured after adhesive, but light cured after resin cement (delayed testing at 4 weeks), CCI: light cured after adhesive and light cured resin cement (immediate testing at 24 hours), CCD: light cured after adhesive and light cured resin cement (delayed testing at 4 weeks).

Table 1. Experimental materials and their characteristics

Materials	Brand	Composition	Manufacturer
Y-TZP	LAVA	97% zirconium dioxide stabilized with 3% Yttria-Lava Frame	3M ESPE, St. Paul, MN, USA
Universal Adhesive	Single Bond Universal	MDP, Bis-GMA, HEMA, decamethylene DAM, ethanol, water, silane treated silica; 2-propenoic acid, -methyl-, reaction products with 1,10-decanediol and phosphorous oxide; copolymer of acrylic and itaconic acid, dimethylaminobenzoat(-4), camphorquinone, (dimethylamino)ethyl methacrylate, methyl ethyl ketone	3M ESPE, St. Paul, MN, USA
Resin cement	RelyX Ultimate Clicker Adhesive Resin Cement	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	3M ESPE, St. Paul, MN, USA

Y-TZP: yttria-tetragonal zirconia polycrystal, MDP: 10-methacryloyloxydecyl dihydrogen phosphate, Bis-GMA: bisphenol A-glycidyl methacrylate, HEMA: hydroxyethyl methacrylate, DAM: diamine, DMA: dimethacrylate, TEGDMA: triethylene glycol, BPDM: biphenyl dimethacrylate.

divided into six groups of eight specimens each (n=48; 8 per group) depending on the timing of micro-shear bond strength testing: immediate at 24 hours (UUI, UCI, CCI) and delayed at 4 weeks (UUD, UCD, CCD). Fig. 1 and Table 1 show the experimental design and the materials used in this study, respectively. All groups were treated with air-abrasion at a standoff distance of 10 mm with a 3.5 bar press for 15 seconds using Al_2O_3 particles of 50 μm grain size. After air-abrasion treatment, the surface was rinsed for 30 seconds and then air-dried for 30 seconds. SBU was applied to the dried surface. RelyX Ultimate was mixed and placed in a Tygon tube (Saint-Gobain Performance Plastics, Akron, OH, USA) with 0.8 mm inner diameter. The Tygon tube was placed on the ceramic specimen. SBU and RelyX Ultimate were light cured using a light emitting diode (LED) curing light unit (Eliper S10; 3M ESPE) at 600 mW/cm^2 depending on the experimental condition for each group. The ceramic specimens were left to polymerize further for 1 hour at 23°C±1°C. The specimens were stored in distilled water at 37°C for 23 hours in the immediate groups and the delayed groups were stored in distilled water for 4 weeks. Afterwards, they were subjected to fracture test.

2. Bond Strength Test and Surface Analysis

The specimens were loaded with a universal testing machine (LF-plus; Ametek Inc., Largo, FL, USA) on the adhesive interface with a jig at a crosshead speed of 0.5 mm/min until failure. The stress (MPa) at failure was recorded.

3. Statistical Analysis

A one-way ANOVA and a Student-Newman-Keuls multiple comparison test were conducted using R programming language (R Foundation for Statistical Computing, Vienna, Austria). The mean difference was considered significant at the level of $P<0.05$.

4. Examination of the Fracture Surface

The fractured specimens of the immediate micro-shear bond strength groups were examined with a scanning electron microscope (SEM) at 600× magnification (S-4700 FESEM; Hitachi, Tokyo, Japan).

Result

The means and standard deviations for micro-shear bond strength of all groups are presented in Table 2. One-way ANOVA was used to calculate the statistical significance among the groups ($P<0.05$). At 24 hours, the micro-shear bond strength of UUI group was significantly lower than the UCI group and the CCI group ($P<0.05$). There was no significant difference between the UCI and the CCI group ($P>0.05$). At 4 weeks, there were not any significant differences among UUD, UCD, CCD groups ($P>0.05$) and the micro-shear bond strength of the UUD group significantly increased over time compared to UUI group ($P<0.05$).

Fig. 2 show the representative SEM images (×600) of fractured surfaces after micro-shear bond testing of immediate groups. Adhesive failure can be seen in Fig. 2A, whereas resin cement remnants are evident on the ceramic surface of Fig. 2B and C, indicating mixed failure.

Table 2. Micro-shear bond strength (MPa) depending on the timing of testing

Group	Immediate (24 hr)	Delayed (4 wk)
UU	8.60±2.06^a	24.43±2.88^{b,*}
UC	25.71±4.48^b	26.49±4.16^b
CC	29.54±3.62^b	28.30±4.76^b

UU: uncured after adhesive and uncured after resin cement application, UC: uncured after adhesive, but light cured after resin cement, CC: light cured after adhesive and light cured resin cement.
Values are presented as mean±standard deviation.
Within the same column, values with different superscript lower case letters are statistically significantly different ($P<0.05$).
*Significant increase in bond strength for each group after 4 weeks of water storage ($P<0.05$).

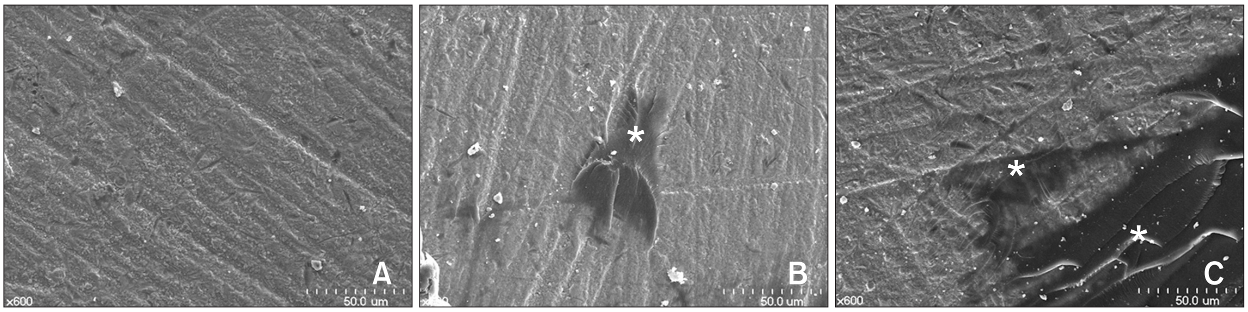

Fig. 2. Scanning electron micrograph images (×600) of fractured surface of yttria-tetragonal zirconia polycrystal ceramic specimen surfaces cemented with Single Bond Universal and RelyX Ultimate; uncured after adhesive and uncured after resin cement application (immediate testing at 24 hours) group (A), uncured after adhesive, but light cured after resin cement (immediate testing at 24 hours) group (B), and light cured after adhesive and light cured resin cement (immediate testing at 24 hours) group (C). Regions marked with asterisks indicate the resin cement remnants.

Discussion

This study investigated the effects of light-curing on the immediate micro-shear bond strength when SBU, a 10-MDP containing universal adhesive, and RelyX Ultimate were used on Y-TZP ceramic.

When SBU was not light cured both before and after resin cement application, immediate micro-shear bond strength was significantly lower compared to when light curing was done after resin cement application. While RelyX Ultimate is known to contain a dual-cure activator for SBU, it was not adequate to restore the micro-shear bond strength to the level of UCI or CCI group. Clinically, this would be a case where the curing light cannot penetrate the restoration, and SBU and RelyX Ultimate need to rely on dark curing. As shown in this study, the micro-shear bond strength of UUI group was too weak to be clinically acceptable as 10 to 13 MPa has been suggested as the minimum clinically acceptable shear bond strength[17].

However, when light curing was done after resin cement application, there was not any significant difference between the UCI group and CCI group. This result justifies the clinical practice of not light curing the universal adhesive on the ceramic surface for the risk of the thick adhesive layer interfering with the fit of the restoration as long as the light can penetrate through the restoration after resin cement application. RelyX Ultimate is known to contain co-initiators such as the sodium salt of aryl sulphinic acid that can overcome the incompatibility between acidic adhesives and self/dual-cured composite resin[18]. In addition, when comparing the delayed micro-shear bond strength, in the UU group, the micro-shear bond strength significantly improved after time, and this is probably related to the dual cure activator component in the resin cement. After 4 weeks, the micro-shear bond strength reached the clinically acceptable value of 24.43 MPa. However, one of the limitations of this experiment was that the specimens were not thermo-cycled during 4 weeks of storage. A previous study showed that the bond strength was significantly lower after thermocycling compared to water storage, and water storage did not have a negative effect on the bond strength[19]. This is consistent with the result of this study as the micro-shear bond strength did not change in the UC and the CC groups.

In the UCI and CCI group, the specimens showed mixed fracture patterns with the resin cement remnants showing on the Y-TZP surface. This is consistent with previous studies which showed strong and durable bond between Y-TZP and resin cement when the combination of air-abrasion treatment and MDP-containing primer was used and showed mixed failure patterns after micro-shear bond testing[20]. However, adhesive failure was seen in the UUI group, which indicates light curing is needed for SBU to be effective, and dark curing is not enough.

While different ceramic materials have different levels of translucency and opacity, zirconia is one of the more opaque materials[21,22]. In fact, in clinical practice, it is even used to mask discolored abutments[23]. Therefore, the curing light may not penetrate the zirconia restoration, and clinicians should keep in mind that the clinical situation may be similar to the UUI group rather than the UCI group, and the dual cure resin cement may not be sufficiently polymerized. Moreover, this study investigated the adhesion between zirconia restoration and the resin cement, but not between the resin cement and the tooth material. Therefore, the adhesion between the dual resin cement and the tooth using universal bonding when light-cured through opaque zirconia should also be considered in clinical practice.

Conclusion

Within the limitations of this study, light-curing of universal adhesives, SBU, before and after application of RelyX Ultimate significantly improved the immediate micro-shear bond strength of resin cement to air-abrasion treated Y-TZP surface. After 4 weeks, the delayed micro-shear bond strength of the non-light curing groups significantly improved to the level of light-cured groups.

The immediate micro shear bond strength was clinically acceptable as long as SBU applied on the inner surface of the restoration was light-cured through the restoration after resin cement application.

Conflict of Interest

No potential conflict of interest relevant to this article was reported.

Acknowledgement

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