Microshear Bond Strength of Zirconia-Reinforced Lithium Silicate Ceramic Bonded to Dentin

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Abstract

To examine microshear bond strength (μSBS) of zirconia-reinforced lithium silicate ceramics (ZLS) bonded to dentin, Celtra Duo (CD) blocks were sectioned into 24 microbars (1×1×3 mm^3). Half of the CD were additionally fired and defined as fired-Celtra Duo (fired-CD) while the rest was defined as unfired-Celtra Duo (unfired-CD). Each microbar was cemented to each flat occlusal dentin surface of human premolar using Single Bond Universal (SU) combined with RelyX Ultimate (n=12 per group). 24-hour μSBS was then determined, and data were analyzed using independent T-test (α = .05). Failure modes were analyzed under a stereomicroscope at 40×. Independent T-test revealed that a type of ZLS had no influence on μSBS (*P*=.159). Failure mode was predominantly cohesive failure in luting cement for fired-CD (66.7%) and adhesive failure between cement and dentin for unfired-CD (58.3%). The finding can conclude that unfired-CD and fired-CD comparably achieved μSBS when they were bonded to dentin using universal adhesive resin luting cement.

Keywords**:** *Microshear bond strength, Zirconia-reinforced lithium silicate ceramics*

1. Introduction

The ceramics classification proposed by Valandro et al. (2005) was based on the existence of ceramic surface degradation by hydrofluoric acid (HF). Ceramics with high glass content in their composition, such as feldspar-, leucite-, and lithium disilicate-based ceramics, can be etched by hydrofluoric acid, resulting in a micromechanical retentive surface and they are called acid sensitive or glass ceramics (Valandroet al., 2005). Nowadays, glass ceramics are mainly lithium disilicate-based pressable ingots or CAD/CAM blocks (Guess et al., 2013). These ceramics exhibit translucency and aesthetic appearance superior to those high strength polycrystalline ceramics (Raptis, Michalakis, & Hirayama, 2006).

Ceramics based on glass infiltrated alumina or zirconia, densely sintered alumina, and yttria-tetragonal zirconia polycrystal (Y-TZP) cannot be degraded by hydrofluoric acid, do not present micromechanical retention, and are referred to as acid resistant or polycrystalline ceramics (Valandro et al., 2005). Y-TZP has excellent mechanical properties, with a broad range of indications from frameworks for bridges to frameworks for single crowns (Sailer et al., 2015). Although in the recent time, new high translucency stabilized zirconia has been introduced for monolithic full contour restorations, they still remain opaque (Zhang, 2014). This aspect limits their use as monolithic restorations in the posterior region only (Traini et al., 2014). On the other hand, veneered zirconia restorations showed a considerable clinical rate of chipping and delamination of the veneering glass-ceramic (Larsson & Wennerberg, 2014).

Recently, a new material, zirconia-reinforced lithium silicate ceramic (ZLS), was launched under the argument that zirconia could act as a crystal phase that can reinforce the material; which can avoid crack propagation. Moreover, this material can perform esthetic excellence because of the glass-ceramics composition in the form of monolithic restoration so that ZLS could be etched by hydrofluoric acid and cemented with adhesive systems (Preis et al., 2015; Sato et al., 2016).

Nowadays, there are two brands of ZLS in the market. The ZLS Vita Suprinity (Vita Zahnfabrik) is a pre-crystallized ceramic material. Therefore, it needs a crystallization firing after milling to achieve the final density. However, the ZLS Celtra Duo (CD, Dentsply Sirona) is a fully crystallized ceramic, which can be delivered directly after finishing and polishing. The milled restoration (unfired-Celtra Duo, unfired-

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CD) has a flexural strength of 210 MPa. Alternatively, an additional stain and glaze firing (fired-Celtra Duo, fired-CD) will increase the material's flexural strength to 370 MPa (Qeblawi et al., 2010).

Adhesive cementation is one of the most important steps for ceramic restorations. It can cause sealing the margin of the restorations (Rosentritt et al., 2007), reinforce the ceramic structure (Jensen, Sheth, & Tolliver, 1989), have good adhesion to the tooth structure, and provide an opportunity to modify the color of the final restorations (Touati & Miara, 1993). However, the technique to be used is very sensitive and demands a careful implementation of a series of steps. The clinical success and longevity of the bonded esthetic restorations depend on both the adhesive and the resin luting cement forming an optimal attachment to tooth structure (Coelho Santos et al., 2005).

A new family of adhesive systems called a universal adhesive was introduced into the market. Some universal adhesives contain silane and a functional monomer like 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP). These adhesives can be used for direct or indirect restorations and are capable of bonding with various substrates including resin composite, ceramics, zirconia, and metal alloys with no need for additional primers (Seabra, Arantes-Oliveira, & Portugal, 2014). The adhesives have a variety of application methods to dental tissues, either by using etch-and-rinse or self-etch bonding approaches. Although self-etching adhesives are easier to apply and commonly less technique-sensitive than etch-and-rinse versions (Van Meerbeek et al., 2011), it has been shown that both techniques led to acceptable dental bonding (Pashley et al., 2011; Van Meerbeek et al., 2011).

Some studies have investigated the efficacy of adhesive resin luting cement to bond dentin with zirconia and glass ceramic (Lee et al., 2015; Shin et al., 2014; Yi et al., 2015). However, the effectiveness of universal adhesive resin luting cement on unfired-CD and fired-CD has not been thoroughly investigated.

2. Objectives

The objective of this study was to examine microshear bond strength (μSBS) of fired-CD and unfired-CD bonded to tooth structure using a universal adhesive resin luting cement.

3. Materials and Methods

3.1 Tooth Selection

24 extracted human first premolars, stored in 0.1% thymol solution at 4°C in the refrigerator no longer than 2 months after extraction, were selected for this study (Aydin et al., 2015). Teeth were analyzed at 4× magnification using a stereomicroscope (ML 9300 MEIJI, Japan) and following selection criteria: no caries or previous restorations, no cracks, and the presence of completely formed apexes. After the selection process, residual soft tissue was removed by hand scaling.

3.2 Tooth preparation

The roots of all teeth were embedded in polyvinyl chloride tube, left the cementoenamel junction of each tooth at the same level of top surface of acrylic resin bases (Trey Resin II, Shofu, Kyoto, Japan) and placed in tap water to reduce the temperature rise caused by the exothermic polymerization reaction of acrylic resin. One clinician prepared all teeth. The 2.0 mm thickness from the central pit of occlusal surfaces of all teeth were removed using a water-cooled slow-speed diamond saw (Isomet 1000 Precision Saw, Buehler, Lake Bluff, IL, USA) under water cooling to expose flat deep dentin surfaces (Figure 1). Dentin surfaces were controlled for the absence of enamel and pulp tissue using a stereomicroscope.

Standardization of smear layer was achieved by grinding the dentin surfaces with 600 grit silicon carbide paper at 100 rpm for 30s to produce standard smear layer, which was comparable to dentin grinding with the bur-cut surface (Finger, 1988; Pashley et al., 1988; Tao, Pashely, & Boyd, 1988). The grit silicon carbide paper was changed after grinding of 10 dentin specimens. After that, all teeth were randomly divided into 2 groups (n=12 per group).

The cementation area on the dentin specimen was defined and isolated using perforated Teflon tape. The dimension of the perforation was equal to 1×1 mm^2.

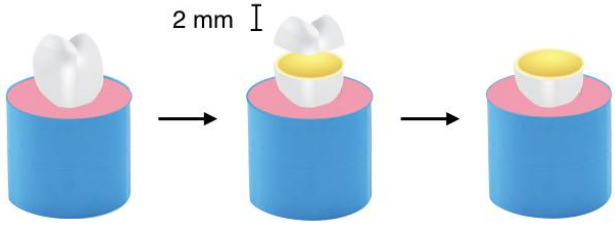

Figure 1 Preparation of dentin specimen

3.3 Ceramic microbar preparation

The CD in the form of CAD/CAM ceramic ingots were cut into 24 microbars in the dimension of 1×1×3 mm^3, using the diamond saw (Figure 2). Half of them were additionally fired in a ceramic furnace (Programat P700, Ivoclar Vivadent, Schaan, Liechtenstein) according to the manufacturer's instruction to increase the material's flexural strength to 370 MPa. The starting temperature was 500°C, and the heating rate was 55°C/minute to reach the final temperature of 820°C. After that, the temperature was held for 1 minute and 30 seconds and cooled for 3 minutes. The CD underwent additional firing were defined as fired-CD while the rest was defined as unfired-CD. Later on, the bonding area of each microbar was precisely measured using a stereomicroscope (MEIJI, Japan); width × length (mm^2).

The top face of each ceramic microbar was polished with 120-, 240-, 400-, 600- grit silicon carbide paper respectively (Weibao, China) at 100 rpm under running water for 10 seconds per each. This step simulated the preparation of ceramic surface with a medium-coarse diamond bur following with fine diamond (Song, Ren, & Yin, 2016).

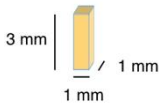

Figure 2 Preparation of CD microbar

3.4 Surface pre-treatment and cementation procedures

According to the manufacturer's instruction of Dentsply Sirona, the polished surfaces of unfired-CD and fired-CD microbars were etched with a 4.5 % hydrofluoric acid (IPS ceramic etching; Ivoclar Vivadent, Schaan, Liechtenstein) for 30 s. Etched surfaces were then thoroughly rinsed with water for 60 seconds. Subsequently, cleaned in the ultrasonic bath with 98 % alcohol for 3 mins, and air-dried. Single Bond Universal (SU, 3M ESPE, USA) was applied at etched ceramics and rubbed in for 20 s. Then, a gentle stream of air was blown over the liquid for about 5 s until it no longer moves and the solvent had evaporated completely. At the dentin surface, SU was applied at prepared dentin which was moist and did not have any puddles on it and rubbed in for 20 s. Then, a gentle stream of air was blown over the liquid for about 5 s. Light-curing for 20 s to dentin. Next, the resin cement RelyX Ultimate (RXU, 3M ESPE, USA) was applied copiously to the etched ceramics using the auto-mix syringe. After loading of the luting cement in all groups, the ceramic was positioned on the prepared dentin surface under a constant load of 1 kg placed on the top of the ceramic using a custom-made loading device (Durometer, ASTM D 2240 Type A, PTC Instrument, USA) (Souza et al., 2016). Excess material was removed with a brush tip. This study utilized a LED light-curing system (Demi Plus, Kerr Corporation, Orange, CA, USA) with 1,100 mW/cm^2 intensity. The light guide was held perpendicularly within 1 mm away from ceramic slabs for 20 s per surface. Then, the load was removed, and the specimens were additionally light-cured from the top during

20 s (100s light-curing in total). After that, the specimens were left for 10 mins (Luhrs et al., 2014), and the perforated Teflon tape was removed. Each specimen was examined under a stereomicroscope (ML 9300 MEIJI, Japan) at 25× magnification to verify that no bonding defects, air bubble inclusions, interfacial gaps, and any excess cement. If the excess cement was found, Blade No. 11 was used to remove it and changed every specimen. All specimens were stored in distilled water at 37°C in the incubator (Contherm 160M, Contherm Scientific Ltd., New Zealand) for 24 hours.

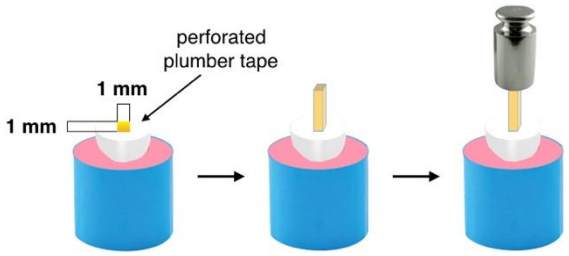

Figure 3 Cementation of the ceramic block to dentin specimen

Table 1 Materials used in this study

Adhesive systems	Manufacturers/Batch number	Composition
Single Bond Universal	3M ESPE/ 651936	10-MDP, Bis-GMA, phosphate monomer, dimethacrylate resins, HEMA, methacrylate-modified polyalkenoic acid copolymer, filler, ethanol, water, initiators, silane-treated silica
RelyX Ultimate	3M ESPE/ 662726	Base paste: methacrylate monomers, radiopaque silanated fillers, initiator, stabilizer, rheological additives Catalyst paste: methacrylate monomers, radiopaque alkaline (basic) fillers, initiator, stabilizer, pigments, rheological additives, fluorescence dye, dark cure activator for Scotchbond Universal
Celtra Duo	Dentsply Sirona	SiO_2, Li_2O, K_2O, P_2O_5, Al_2O_3, ZrO_2, CeO_2, pigments

3.5 Microshear bond strength testing (μSBS)

All specimens were subjected to μSBS testing. Each polyvinyl chloride tube with a ceramic microbar was placed horizontally on a support base so that the ceramic microbar was sticking out unsupported. The adhesive interface was parallel to shearing force. Subsequently, the axial load with a 5-N load cell at a crosshead speed of 0.5 mm/minute was applied using a chisel-shaped rod at the dentin/adhesive interface as close as possible to the surface of the tooth until fracture of the specimen (Moro et al., 2017; International Organization for Standardization [ISO], 2013). The maximum force (Fmax (N)) was recorded. The μSBS values (MPa) were calculated by (Fmax (N) / bonding area (mm^2), resulting in 12 μSBS values per group for statistical analysis.

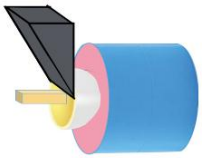

Figure 4 Microshear bond strength test

3.6 Data Collection and Analysis

All data were collected and analyzed using statistical software (IBM® SPSS® 20, SPSS, Chicago, IL). The normality of the data was determined using the Kolmogorov-Smirnov test (K-S test). Independent T-test was used to statistically analyze μSBS between groups. Significance level was set at $P \leq .05$.

3.7 Failure Mode Analysis

After debonding, the specimens were examined under a stereomicroscope at a magnification of 40× to verify failure type. Failure types were classified as shown in Table 2 (Flury et al., 2016; Kitasako et al., 1995; Manso et al., 2011).

Table 2 Types of failure

Type	Character
Adhesive failure between cement and ceramic	Where resin cement completely remained on top of dentin surface
Adhesive failure between cement and dentin	Where resin cement completely remained on ceramic surface
Cohesive failure in luting cement	Where remnants of resin cement partially remained on both dentin and ceramic surface
Cohesive failure in dentin	The failure was within dentin
Cohesive failure in ceramic	The failure was within the ceramic
Mixed failure	Failure at the cement and adhesive interface including cohesive failure of the neighboring substrates

4. Results and Discussion

There was no pre-test failure before or during the microshear bond strength testing. The K-S test indicated that the data were normally distributed (P=.200). The Mean and standard deviation of fired-CD and unfired-CD were equal to 31.63 ± 9.17 MPa and 26.40 ± 8.40 MPa, respectively (Figure 5). Independent T-test revealed that type of ZLS did not have a statistically significant influence on μSBS values of ZLS bonded to dentin (P=.159) (Table 3). It was observed that failure mode was a predominantly cohesive failure in luting cement for fired-CD (66.7%); meanwhile, an adhesive failure between cement and dentin was noticed to be a major finding in unfired-CD (58.3%). Failure type frequencies were given by group in Figure 6.

Table 3 Independent T-test used to statistically analyze μSBS between groups

	t	df	Sig. (2-tailed)
Microshear bond	1.459	22	.159

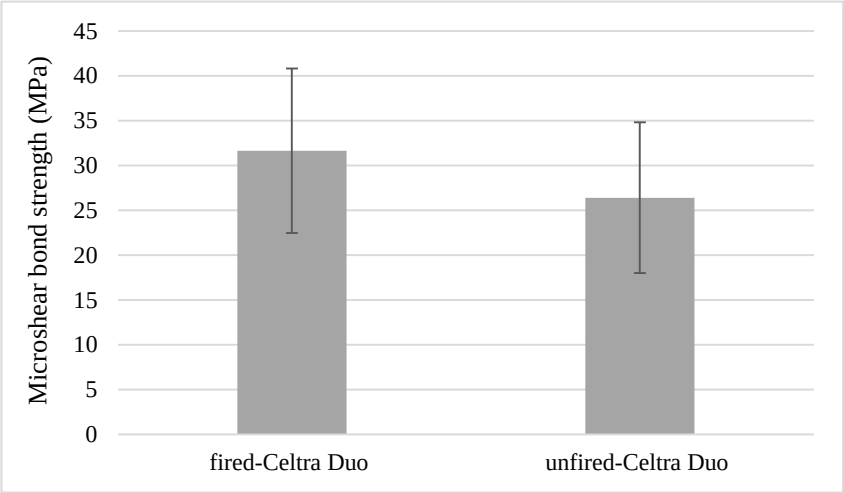

Figure 5 Microshear bond strength values (mean [MPa] ± SD) of different groups.

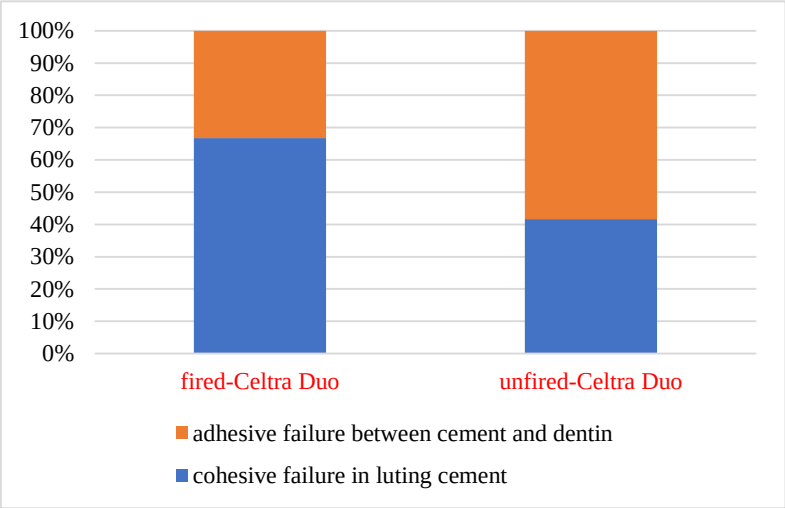

Figure 6 Failure mode percentages of all groups

In this present study**,** distinct forms of CD (fired- or unfired-CD) had no significant effect on μSBS of this ceramic cemented to dentin using universal adhesive resin luting agent. The ceramic comprised round and slightly elongated lithium-metasilicate (Li_2SiO_3) crystals, round lithium orthophosphate (Li_3PO_4) granules in glassy matrix and were reinforced with about 10% zirconium dioxide (ZrO_2) so that HF could pretreat the ceramics, silanized then bonded by adhesive resin luting cement **(**Belli et al., 2017; Ramos Nde et al., 2016; Schwindling, Rues, & Schmitter, 2017). Moreover, the firing process had a positive impact on the healing of natural flaws and zirconia crystallization, consequently improving mechanical properties but still etchable by HF (Qeblawi et al., 2010). Since the field emission scanning electron micrographs of fired-

and unfired-CD after pretreatment showed round defects inside the glassy matrix (Riquieri et al., 2018), both forms of CD comparably achieved μSBS. The final crystallized ZLS variations give advantages of short processing times and high stability. These properties especially allow the chairside fabrication for 1-day treatment of all-ceramic restorations.

Single Bond Universal, utilizing self-etch mode, comprised a various composition that mixed different functional components, including water, ethanol, and silane into the solution (Stape et al., 2018). The dihydrogen phosphate group of 10-MDP in this adhesive was responsible for reaction that led to the creation of ionic bond with calcium ions of hydroxyapatite, and formation a nano-layering structure of MDP-Ca salt at the adhesive interface (Tian et al., 2016; Y. Yoshida et al., 2012). In a previous investigation, it was hypothesized that without removing the smear layer and incorporating it into the adhesive interface, penetration of resin monomers and bonding effectiveness of this class of adhesive could be compromised (Scaminaci Russo et al., 2014). Meanwhile, the long hydrophobic carboxyl chain of this functional monomer copolymerized with resin monomers of the resin cement and provided hydrolytic stability of the bonding interface (K. Yoshida, Tsuo, & Atsuta, 2006). Moreover, 10-MDP was proven to offer a bond-mediating capacity to zirconia (Oyague et al., 2009; Samimi et al., 2015). Hydrophilic phosphate terminal end of this functional monomer has been claimed to interact chemically with oxide of zirconia in ZLS resulting in high bond strength. These may be the reasons why the failure type at the adhesive interface between cement and ceramic was not found.

In the previous study on comparing wire loop and blade method for μSBS testing, it was found that if the force was loaded at a contact point between the blade and interface, it created a less even uniformity of shear force (DeHoff, Anusavice, & Wang, 1995; Foong et al., 2006). That was the reason why the cross-section of ceramic microbar was prepared as a rectangular area in this study. When the blade touched the interface, the shear force was loaded at the contact area, consequently creating more uniform loading stress. In this present investigation, all of the failure modes were observed at the adhesive interface between cement and dentin and in luting cement; therefore, the value measured when specimen cracked represented a more reliable μSBS. Furthermore, no pretest failure was found because the ceramic microbars were prepared before cementation. Thus, the occurrence of structural defects from trimming after the adhesive procedure in specimen preparation was reduced.

The outcome of this present study may provide clinician useful information regarding the selection of either form of fully crystallized zirconia-reinforced lithium silicate ceramic to fabricate the indirect restoration. Further study should be carried out to test the bond strength of ZLS under different aging conditions and adhesive situations.

5. Conclusion

The different forms of ZLS comparably achieved μSBS when they were bonded to dentin using a universal adhesive resin luting cement. However, the clinician should know other mechanical properties of ZLS, such as flexural strength and fracture toughness, before making a decision on selecting an appropriate form of ZLS to restore the tooth.

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