CU Dent J. 2019;42:11-22

Original Article

Microtensile bond strength between zirconia ceramics to resin composite using different resin cements

Received : June 10, 2019
Revised : June 24, 2019
Accepted : August 3, 2019

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Abstract

Background/Objective: This study aimed to evaluate the microtensile bond strength (MTBS) of three zirconia ceramics from different manufacturers bonded to resin composite using three different resin cements.

Materials and methods: Three fully-sintered zirconia ceramic blocks (5x5x10 mm) from Katana (Nuvodent, Japan), Lava (3M ESPE, Germany) and Cercon (Degudent, Germany) were fabricated and bonded to resin composite blocks (Filtek Z250, 3M ESPE, USA) with the same size using one of the three resin cements : Panavia F 2.0 (Kuraray, USA), Superbond C&B (Sun Medical, Japan) and RelyX Unicem (3M ESPE, USA). After 24 h, each block was cut under water coolant to produce microbar specimens, with bonding area 1 ± 0.1 mm^2. The MTBS was tested with universal testing machine at a crosshead speed of 0.5 mm/min. The interface failures were examined using a scanning electron microscope. Two-way ANOVA and Tukey's tests were analyzed at 95% confidential interval.

Results: The MTBS value range from 43.3 - 53.9 N/mm^2. Lava/Superbond showed the highest value, while Cercon/Panavia showed the lowest value. Statistical analysis showed that types of zirconium-oxide ceramic (P=0.043) and types of resin cement (P=0.047) had an effect on MTBS, while the interaction between zirconia ceramic and types of resin cement (P=0.056) was not significant. Panavia F 2.0 and RelyX Unicem demonstrated resin cement cohesive failure. Superbond C&B showed mixed of adhesive and cohesive failure. No adhesive failure was observed.

Conclusion: The MTBS of zirconia ceramic bond to composite using resin cement depended on brands of zirconia and types of resin cement.

(CU Dent J. 2019;42:11-22)

Keywords: microtensile bond strength, resin cement, zirconia

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Introduction

Zirconia ceramic has become more popular as a restorative dental material since it has a favorable esthetic appearance and mechanical properties. These include high flexural strength, high fracture toughness, low thermal conductivity, high chemical resistance, together with good biocompatibility (Miyazaki et al., 2013). It has been proved to be used successfully in a clinical trial with high success rate for crown and bridge even for implant restorations (Denry and Kelly, 2008; Håff et al., 2015). The fabrication of zirconia restoration uses a computer-aided design/computer-aided manufacturing (CAD/CAM) process. The restorations can be completed as veneered zirconia for more esthetics, or monolithic zirconia to avoid the problems of porcelain chipping, while fracture of zirconia is rarely reported (Håff et al., 2015; Komine et al., 2010; Miura et al., 2018).

Bonding zirconia restoration in clinical practice is problematic. Since the structure of zirconia does not contain glass matrix which limited its bonding ability to resin cements. Hence, both conventional or resin cements can be used to bond zirconia restorations to the tooth structure or other substrates. Literally, resin cements showed higher bond strength, less microleakage, while they can improve the retention in CAD/CAM-milled ceramic restorations by sealing minor internal surface flaws created by the milling process (Luthra and Kaur, 2016). Sandblast or airborne-particle abrasion on the surface of zirconia can produce a mild roughness surface and can effectively promote a better bonding ability for luting cement to zirconia. Some early studies claimed that it can reduce the strength of zirconia restorations because the creation of multiple microcracks in the ceramic surface could propagate and reduce the fracture strength (Zhang et al., 2004). However, by using small particles of 50μ aluminum oxide with suitable pressure and distance, it can increase mechanical retention and this surface topography enhanced the bonding by increasing surface energy and wettability to promote micromechanical interlocking with luting agents. The luting agents can also heal minor cracks produced by airborne-particle abrasion which the flowability of resin cement can penetrate into the substrate micro-retentions, inducing interlocking formation at the interfaces (Borges et al., 2003; Ferrari et al., 2015). To date, air-particle abrasion is a prerequisite for achieving sufficient bond strength between the resin cement to zirconia (Blatz et al., 2003; Tsukakoshi et al., 2008).

To ensure more success in the clinical longevity, bonding of zirconia to resin cement still needs improvement. Massive studies had been proposed to improve the bonding ability of zirconia in many aspects. These included the surface modification by increasing its micro-mechanical retention, attaching silica particles, application of primer before bonding, varied types of resin cement used (Özcan and Bernasconi, 2015). The minor surface treatment by applying zirconia primer or MDP containing liquid had also been investigated, but the results were controversial (Bömicke et al., 2016). Chemical bonding had been developed such as silane coupling agents, tribochemical coating system (Rocatec), selective infiltration-etching (SIE) technique, molecular vapor deposition (MVD) laser treatments (Blatz et al,. 2018). However, these surface treatments to develop chemical bonding for zirconia are clinically impractical due to complicate equipment and causative materials.

Oyagüe et al. (2009) showed that the bonding of resin-ceramic longevity depended on cement selection rather than on surface pre-treatments. Saryazdi et al. (2009) investigated the retention of zirconia crowns and by various types of cement and surface treatments and also confirmed that the retention was dependent on the adhesive cement used and not on the internal surface treatment. While Ernst et al. (2005) determined the retentive strength of zirconia crowns with Superbond C&B, Panavia, Dyract Cem Plus, RelyX Luting, and RelyX Unicem and found that each cement system showed the highest median retentive strength values, which were not significantly different. The use of resin cements containing 10-methacryloyloxydecyl dihydrogenphosphate (MDP) after airborne particle abrasion on zirconia is recommended for an optimal bond strength and good bond durability after thermocycling and long term water storage (Kern and Wegner, 1998; Aboushelib et al., 2007). Lüthy et al. (2006) and Özcan et al. (2008) showed that Panavia F created greater bond strength than Superbond C&B. On contrarily, Derand and Derand (2000) showed that Superbond C&B had higher bond strength than Panavia 21, and explained that the anhydride group in 4-META can bond effectively with zirconia and the tribochemical coating. While Thompson et al. (2011) showed that RelyX Unicem, exhibited statistically comparable bond strength to MDP-containing resin cements.

Most studies have been performed the bond strength of zirconia using a specific brand. There are many manufacturers produce zirconia which might be vary from each other in compositions, procedure and process of fabrication (Table 1). The mechanical properties of zirconia depend largely on the starting powder, fabrication technique, pressing condition, sintering process and sintering time to allow the material to reach satisfactory characteristics (Casucci et al., 2010). The powder can have different grain sizes, distributions of the various grain sizes, and additives such as binder. These can affect the homogeneity and the density of zirconia, thus, might affect and the ability in the bonding of zirconia to resin cements. The aim of this study was to evaluate the MTBS between three brands of sandblasted zirconia ceramic and resin composite block using three resin cements. The null hypotheses were there was no significant difference of MTBS among brands of zirconia, types of resin cement or an interaction between brands of zirconia and types resin cement.

Table 1: Differences of each brand of zirconium-oxide ceramic (Data obtained from manufacturers)

	Cercon	**Lava**	**Katana**
%Zirconium oxide	92.3%	97%	94.4%
%Yttrium oxide	5%	3%	5.4%
Binder	HfO_2 < 2%	No	No
Grain size of powder	0.5 µm	0.07-0.3 µm	N/A
Sintering technique	Non-HIP (Pre-sintered)	Non-HIP (Pre-sintered)	Non-HIP (Non-presintered)
Final sintering temperature	> 1,350°C	1,360°C-1,530°C	1,400°C -1,500°C

Material and method

The specimens of MTBS in this study were prepared following the method of Sano et al. in 1994 and adapted for bonding zirconia to resin composite block as described by Oyagüe et al. (2009).

Specimen preparation

Three fully sintered rectangular blocks (5 x 5 x 10 mm^3) of three brands of zirconia ceramics: Katana (Nuvodent, Japan), Lava (3M ESPE, Germany) and Cercon (Degudent GmbH, Germany), were fabricated according to the manufacturer's instructions. (Table 1) Ceramic surfaces were finished with 100, 220, 400, 800, 1200 and 2000-grit silicon carbide abrasive in a polishing machine (Grinder-polisher Ecomet®250, Buehler, IL, USA) and sandblasted (Sandblasting machine P-G400; Harnisch & Rieth, Winterbach, Germany) with 50 µm aluminum-oxide particles at 0.4-0.5 MPa for 10 seconds at distance of 10 mm and cleaned for 10 minutes in an ultrasonic bath (Ultrasonic cleaner 5210, Bransonic, Germany).

Three resin composite blocks (Filtek Z250, shade A2; 3M ESPE, St. Paul, MN, USA) with the same size, were prepared by mold made from silicon impression material. The resin composite were filled up and condensed 2 mm incrementally into the mold with a clean plastic filling instrument to avoid any contamination. Each layer was light polymerized for 40 sec (Light curing unit XL 3000, 3M ESPE). The last increment of the resin composite was covered with a Mylar strip and glass slide. The resin composite surfaces were finished with the same grit silicon carbide abrasive and cleaned for 10 min in an ultrasonic bath.

Three resin cement systems: Panavia F 2.0 (Kuraray, USA), Superbond C&B (Sun Medical, Japan) and RelyX Unicem (3M ESPE) were used to lute the composite resin blocks to the sandblasted zirconia block, according to their manufacturers' instructions. The cement was mixed and applied on the resin composite block surface which was seated on top of the zirconia block and loaded with 1 kg (Durameter stand, Pacific transducer corp, CA, USA) for 60 sec and the excess cement was removed (Figure 1A), resulting in 9 groups of ceramic-composite blocks. The light polymerization was done 40 s on each side of the block using Panavia F2.0 and RelyX Unicem which are dual-curing cements to ensure initial polymerization. After cementation, the blocks were kept in a container for 24 h at room temperature for complete cement setting. The blocks were then bonded with Model Repair II Blue (Dentsply-Sankin, Ohtawara, Japan) to a metal base that was coupled to a cutting machine (High speed cutting machine, Model Isomet 4000 Linear precision saw, Buehler, IL, USA). Each bonded specimen was vertically sectioned under running coolant into 1 mm thick slabs and consequently into beams with cross-sectioned areas of 1 ± 0.1 mm^2 into at least 15 microbars (Figure 1B). The specimens were randomly selected for obtaining 10 specimens (n=10) for MTBS testing.

Microtensile bond strength test, Statistical analysis and Analysis of failure mode

The microbar specimens were attached to the flat grip with Model Repair II Blue. The MTBS was measured by applying tensile load to the bonded interface using a universal testing machine SHIMADZU EZ S (Shimadzu Corporation, Japan) at a crosshead speed of 0.5 mm/min until failure occurred.

Statistical analysis was performed using two-way analysis of variance (ANOVA) to study the contributions of the resin cement types, the brands of zirconia ceramic and their interaction on MTBS. Multiple comparisons were conducted using Tukey's tests at P-value = 0.05. Statistical analysis was carried out using computer software (SPSS Version 22.0, Inc., Chicago, USA).

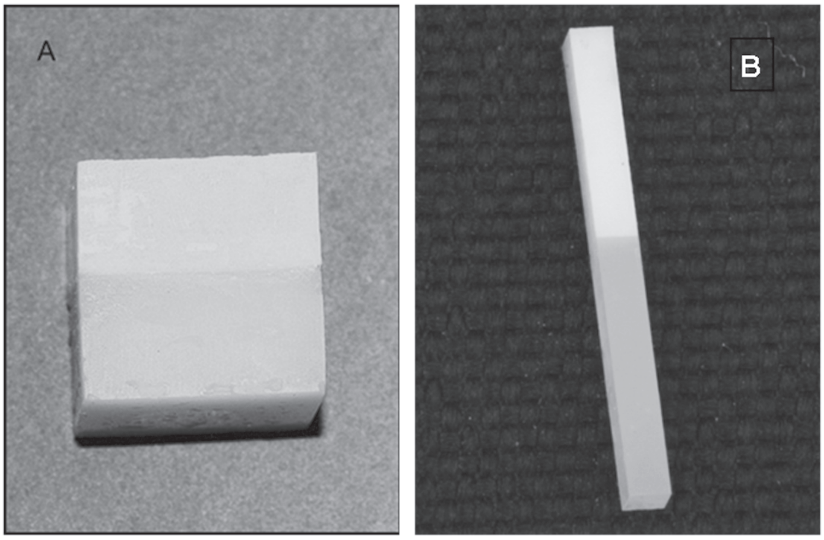

Figure 1: A: The bonded zirconia-resin specimens,
B: Microbars with cross-sectional areas of 1 ± 0.1mm^2.

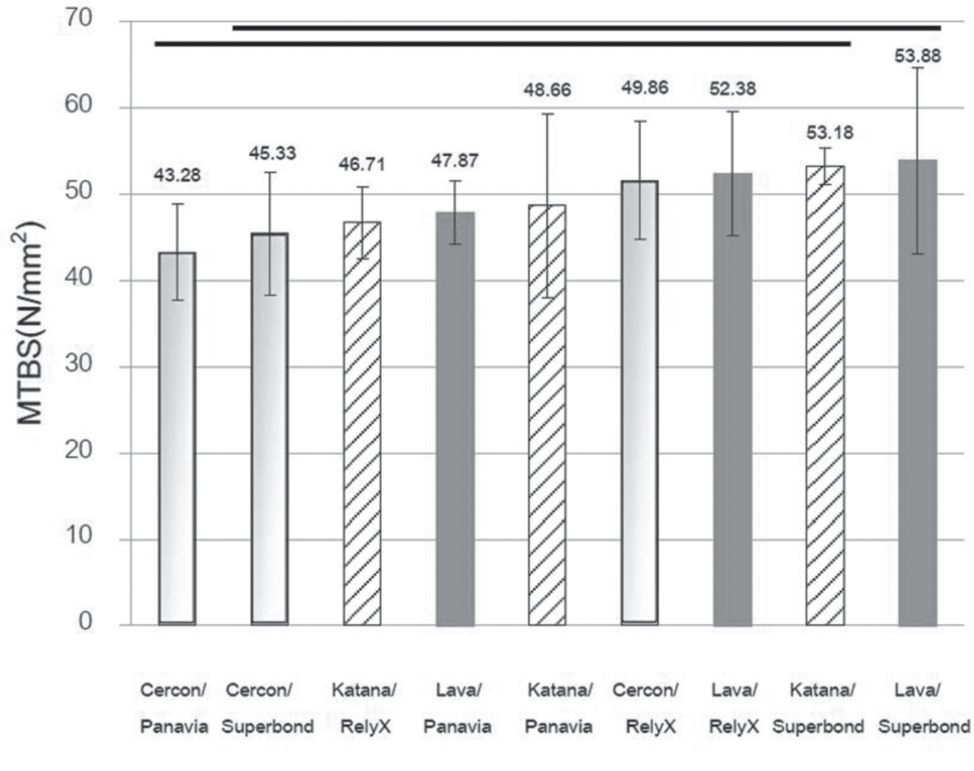

Figure 2: Mean and SD of MTBS values of experiment groups. ——— means no significant difference (P >0.05) .

The fractured specimens were evaluated under a stereomicroscope (Olympus SZ-CTV, Olympus Co., Tokyo, Japan) at 40x magnification to determine the failure mode. Then the fractured interface were examined for interface failure under a scanning electron microscope (SEM, XL 20; Philips, Eindhoven, The Netherlands) at 100x and 2000x magnification. The mode were classified as cohesive (within the cement or ceramic), adhesive (between the composite and the cement or at the cement/zirconia level) or mixed (adhesive and cohesive fractures occurred simultaneously).

Result

The mean and standard deviations of MTBS of each tested group are shown in Figure 2. The MTBS value range from 43.28 - 53.88 N/mm^2. The Lava/Superbond group (53.88 N/mm^2) produced the highest value, while Cercon/Panavia group (43.28 N/mm^2) produced the lowest value. The two-way ANOVA and Tukey's posthoc test revealed that type of resin cement (F=3.165, P=0.047) and the brand of zirconia ceramic (F=3.270, P=0.043) significantly affected the MTBS, while the interaction between brands of zirconia ceramic and types of resin cement (F=2.410, P=0.056) did not affect the MTBS.

Table 2: Failure types in each tested group.

Resin cement	Brand of zirconia ceramic	Failure mode of MTBS
Panavia F 2.0	Cercon	100% Cohesive failure in resin cement
	Lava	100% Cohesive failure in resin cement
	Katana	100% Cohesive failure in resin cement
Superbond C&B	Cercon	70% Cohesive failure in resin cement
		30% Mixed failure*
	Lava	70% Mixed failure*
		30% Cohesive failure in resin cement
	Katana	60% Mixed failure*
		40% Cohesive failure in resin cement
RelyX Unicem	Cercon	100% Cohesive failure in resin cement
	Lava	100% Cohesive failure in resin cement
	Katana	100% Cohesive failure in resin cement

*Mixed failure= Cohesive failure + Adhesive failure.

The types of cement had an effect on MTBS as RelyX Unicem had significantly higher MTBS than PanaviaF 2.0 in Cercon, while not significant in Katana and Lava. The types of zirconia ceramic also had an effect on MTBS as Lava had significantly higher MTBS than Cercon when bonded with Superbond, while three types of zirconia ceramic had no significant difference when bonded with Panavia F 2.0 and RelyX Unicem.

SEM analysis at 100X magnification (Figure 3) revealed that Panavia F 2.0 and RelyX Unicem demonstrated predominantly cohesive failure in resin cement as the surface of zirconia was covered by a layer of resin cement. On both zirconia and resin composite site, Superbond C&B showed a mix of adhesive and cohesive failure on the debond surfaces. No adhesive failure was observed along the ceramic-cement interface or the resin composite-cement interface in all groups. The different percentages in the pattern of failure are shown in Table 2.

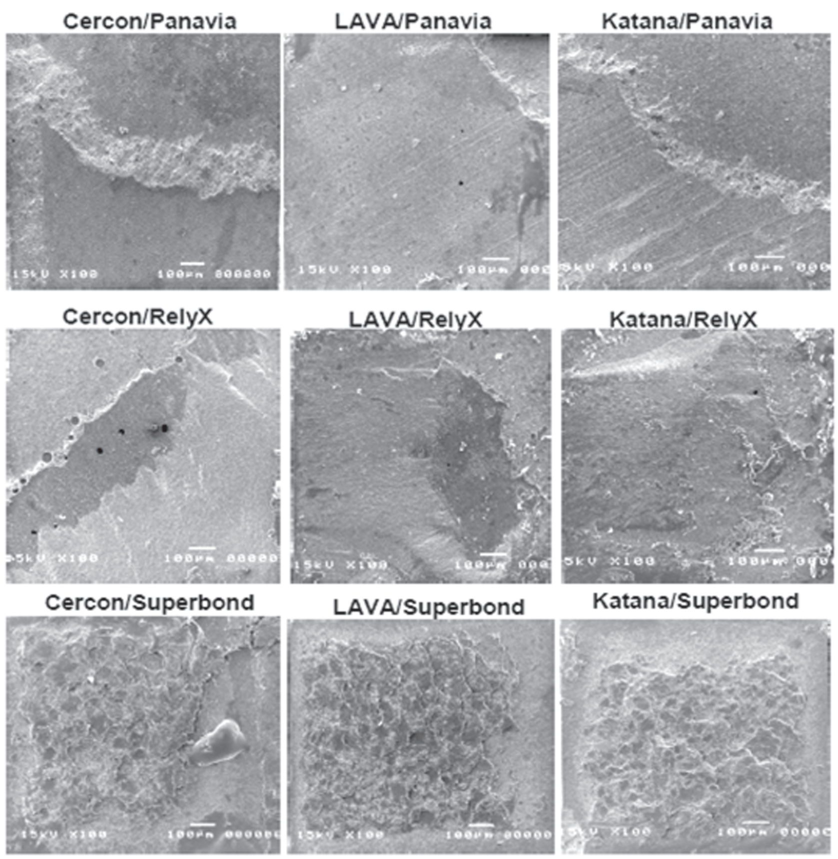

Figure 3: SEM image at 100X magnification, demonstrating the failure observed at the interface on zirconia surface in all groups. Panavia F 2.0 and RelyX Unicem showed predominantly cohesive failure as a layer of cement covered the surface of zirconia, while Superbond C&B showed a mix of adhesive and cohesive failure.

Discussion

The mechanism of bonding the zirconia to each resin cement used in this study were discussed in previous studies. In Panavia F2.0, the MDP and the phosphate ester group of this monomer bonds chemically to zirconium oxides (Kern and Wagner, 1998). Same as in RelyX Unicem, an adhesive phosphate monomer enhanced self-bonding to zirconia ceramics (Kern and Wagner, 1998; Lüthy et al, 2006; Mirmohammadi et al., 2010). In Superbond C&B, the bond strength was due to the anhydride group in 4-META/MMA-TBB resin which formed a cross-linked and strong adhesive (Derand and Derand, 2000).

The first null hypothesis was rejected since statistical analysis showed the significant difference of MTBS among the brands of zirconia ceramic. It can be noted that percent of zirconium-oxide in Lava (97%) is more than in Katana (94.4%) and Cercon (92.3%). The hydroxyl groups that present on the zirconia surface creates a bond to resin cement since the surface of zirconia ceramic is coated with a passive film of zirconium oxide. (Aleisa et al., 2013; Çakırbay Tanış et al., 2019) The higher percent of zirconia in Lava might enhance better bonding ability. Lucas et al. (2015) stated that grain size did not influence the amount of increased surface roughness of zirconia. Casucci et al. (2010) studied the surface of Cercon, Aadva Zr, and Lava and found that these three zirconias which did not receive a surface treatment had a uniform grain microstructure, only slight differences in grain organization, size and density. But after airborne particle abrasion was applied, the surface roughness were increased on Cercon and Aadva Zr ceramics, while no significant effect were produced on Lava. Hence, it seemed that the surface of Lava was more durable than Cercon. In contrast with the results of this study, higher bond strength were obtained when bonded Lava than Cercon with the three resin cements, which meant that the rougher surface of Cercon did not increase the MTBS. Each brand of zirconia ceramic might have differences in many aspects, such as powder properties: composition, impurities, particle size, crystalline size, fabrication sintering process, and etc., which affect the final properties of the zirconia in a complex relationship (Casucci et al., 2010) which further study in detail should investigated.

The second null hypothesis was rejected since statistical analysis showed the significant difference of MTBS among the types of cement. Superbond C&B showed two highest bond strength bonded with Lava and Katana, while bond strength to Cercon was lower, insignificantly. RelyX Unicem showed the third and the fourth highest MTBS to Lava and Cercon, while Panavia F2.0 seemed to show lower values of MTBS among the three cements which the lowest was found in bonding to Cercon with significantly lowest MTBS value. Other properties of cement such as fluidity or film thickness may affect the results. The thicker film thickness resulted in significantly decreased bond strength. Superbond C&B consists of linear polymers of MMA without inorganic fillers, displayed high plastic deformation and gives a significant advantage as a low film thickness adhesive cements (Taira and Imai, 2014). While in Panavia F 2.0 and RelyX Unicem, the cements form three-dimensional networks of polymerized bi-functional monomers, combined with inorganic fillers and create a rigid structure with high values of mechanical properties after curing (Radovic et al., 2008).The film thickness of RelyX Unicem is less than that of Panavia F 2.0. The difference in the amount of filler content may response this effect as RelyX Unicem has less reactive glass weight (72%wt) than in Panavia F 2.0 (78% wt) (Kious et al., 2009; White and Yu, 1993). Fluidity of cement also seems to play an important role in bonding zirconia. In Superbond C&B, the initial mixing stage has low viscosity which can easily luting the zirconia surface. In Relyx Unicem, water produced from the polymerization processes between phosphoric acid groups and alkaline filler made the cement's initial hydrophilicity which improved adaptation to the bonding structure (Radovic et al., 2008). This might cause better results of these two cements than Panavia F 2.0.

The results from SEM showed that Panavia F 2.0 and RelyX Unicem demonstrated predominantly cohesive failure while Superbond C&B showed mix of adhesive and cohesive failure. No complete adhesive failure was observed along the ceramic-cement interface or the resin composite-cement interface in all groups. Cekic-Nagas et al. (2010) showed that the fracture modes in a cement thickness of 50 μm were predominantly adhesive between the resin cement and ceramic, while the failures were mostly cohesive mode with the thickness of 100 μ. The thicker cement layer might be related to a reduced degree of conversion and incomplete polymerization of the cement, which induce the cement weakness to cause a cohesive failure. Furthermore, the MDP presented in Panavia F 2.0 and RelyX Unicem, contained phosphoric acid and methacrylate monomers, where the phosphate ester group has a chemical bond to the zirconium-oxides (Mirmohammadi et al., 2010). The performance of this bond should affect the mode of failure found in both Panavia F 2.0 and RelyX Unicem, which showed a cohesive failure of resin cement. In this study, film thickness of the resin cement might vary among types of cement as a constant load of 1 kg (98 N) was applied to control the luting procedures as performed in previous studies (Mirmohammadi et al., 2010; Smith et al., 2011; Oyagüe et al. 2009, Casuccia et al. 2011), thus, the film thickness of each cement might be different due to the viscosity and the amount and size of filler content.

The third null hypothesis was accepted since statistical analysis showed no significant difference of MTBS. The outcome of this study indicated that the MTBS did not depend on a combination of the types of resin cement and the brands of zirconia ceramics. Ones can try any types of resin cement with any brand of zirconia ceramics will provides nearly the same results. Clinical trials are needed to refine these conclusions since the cement-ceramic adhesion is susceptible to thermal, chemical and mechanical influences under intraoral conditions.

MTBS test was introduced to develop bond tests of small areas which the values are related to the bonded surface area (Sano et al., 1994; Pashly et al., 1999). Higher bond strength values were measured compared to other tests, though, its advantage is that most failures occurred at the interface. Other advantages are that it can be applied to small-sized specimens as a focusing area of a tooth substrate such as carious region, sclerotic dentin or irregular surfaces. MTBS test had been adapted to investigate the bonding of zirconia in several studies (Ikonishi et al., 2014). The fabrication of specimen for MTBS is rather labor-intensive, and requires careful handling of the fragile specimens (Ferrari et al., 2002) as also occurred in this study which many of the specimens failed during preparation. Besides, the samples which are small can dehydrate rapidly.

The limitation of this study might be the lack of thermocycling condition. It can be noted that long-term water storage and thermocycling significantly affect the bond strength of resin-based luting cement to zirconia ceramic (Kern and Wagner , 1998, Lüthy et al., 2006) especially in MDP-free resin cements (Yoshida et al., 2006; Saryazdi et al., 2014). Water storage or thermocycling procedure can deteriorate the bonded specimen and can cause a pre-test failure. Amaral et al. (2014) reported 100% and of pre-test failure of zirconia without any surface treatment bonded to lithium-disilicate glass-ceramic using Variolink II, and 50 % of pre-test failure with zirconia received 35 μm alumina air abrasion. Therefore, in order to emphasize the effect of various brands of zirconia and resin cement, the thermocycling was omitted, same as the study of Casuccia et al. (2011).

The latest generation of high-translucent, more cubic zirconia has significantly different properties and lower flexural strength (Blatz et al., 2018). The factors that affect the light-polymerization such as degree of conversion of the dual-curing resin cements may differ the results between cements and zirconia brands as well.

Conclusion

The MTBS of zirconia ceramic bond to composite using resin cement depended on the brands of zirconia and also types of resin cement. Lava tended to show the highest MTBS when bonded to all resin cements used in this study and Superbond C&B tended to show the highest MTBS when bonded to the zirconia ceramic.

Acknowledgement

The authors would like to thank In-House Dental Laboratory Co., Ltd., 3M ESPE Thailand and Sainamtip Dental Laboratory Co., Ltd. for providing some of materials used and specimen fabrication process in this study.

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