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To cite this article: Diana Capelli Barca, Dayanne Monielle Duarte Moura, Giovanna de Fátima Alves da Costa, Carlos Augusto Pavanelli, Marco Antonio Bottino & Rodrigo Othávio Assunção Souza (2017) Influence of ceramic thickness and light-curing time on the long-term  $\mu$ TBS of silica-based ceramic to human dentin, Journal of Adhesion Science and Technology, 31:15, 1700-1710, DOI: [10.1080/01694243.2016.1274134](https://doi.org/10.1080/01694243.2016.1274134)

To link to this article: <https://doi.org/10.1080/01694243.2016.1274134>



Published online: 02 Jan 2017.



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## Influence of ceramic thickness and light-curing time on the long-term $\mu$ TBS of silica-based ceramic to human dentin

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### ABSTRACT

To evaluate the influence of ceramic thickness, light-curing time and thermal cycling (TC) on the  $\mu$ TBS of a glass ceramic cemented to human dentin. Ninety-six human molars were embedded in acrylic resin and the occlusal surface was sectioned to exposure dentin. Blocks of feldspathic ceramic (Vita PM9) with different thickness ( $6 \times 6 \times 1$  mm/2 mm/3 mm) were fabricated with wax pattern and sprue. The blocks and teeth were randomly distributed into 12 groups ( $n = 8$ ) according to the ceramic thickness (V1: 1 mm, V2: 2 mm and V3: 3 mm), light-curing time (40s; 80s), and TC (yes; no). Dentin was etched, washed and dried. The adhesive (Excite, Ivoclar) was applied onto the dentin surface and light-cured (20s), and the ceramic surface was etched with 10% HF, washed, dried and silanized. The ceramic blocks were cemented to dentin (Variolink II, Ivoclar). The assembly ceramic/dentin was stored in distilled water (37 °C; 24 h) and sectioned in X and Y axes to get the sticks. Half of the samples were submitted to TC while the other half underwent micro-tensile bond testing. The data (MPa) were analyzed by 3-way ANOVA and Tukey test (5%). ANOVA revealed significant interaction among the factors ( $p = 0.0001$ ). Tukey test showed significant higher bond strength for the 2 mm group (V280s =  $17.0 \pm 9.1$  MPa) in comparison to the other groups. V340s TC ( $2.7 \pm 6.3$  MPa) presented lower bond strength, which was similar to the groups V180s TC ( $4.6 \pm 4.9$  MPa) and V240s TC ( $5.9 \pm 4.4$  MPa). Light-curing for 80s promoted significant higher bond strength of thicker ceramic (3 mm) than light-curing for 40s.

### ARTICLE HISTORY

Received 30 May 2016

Revised 16 August 2016

Accepted 14 December 2016

### KEYWORDS

Resin cements; ceramic; dentin; light-curing; micro-tensile

## Introduction

Glass ceramics are well-known because of their advantages in esthetics [1,2], compression strength [3,4], translucency [4,5], and bonding to teeth [6]. Among the glass ceramics, the most commonly indicated are the feldspathic, lithium disilicate, and fluorapatite zirconia-reinforced lithium silicate [7]. These ceramics are used to fabricate inlays, onlays, veneers and anterior crowns [8]. In these situations, the adhesive cementation increases

the bond strength and fracture strength of silica-based ceramics, reducing the chances of debonding, adhesive failures and providing longevity for the restorations [9]. However, adhesion depends on the cementation protocol and bond durability at ceramic/cement/dentine interfaces [9–12].

High survival rates of glass ceramic restorations have been reported in several clinical situations: onlays (92–97% for 5 years) [13,14], inlays (92–80% for 7 and 8 years, respectively) [15,16] and veneers (96% for 21 years) [17]. Despite the high survival rates, some failures have been observed in clinical trials, such as crown fractures [18], endodontic problems [13], marginal staining [16], recurrent caries [18] and interface displacement [19,20]. Some factors are related to failures at dentine/ceramic/cement interface, including inappropriate treatment of ceramic surface [9], contamination of bonding interface [21], inappropriate treatment of substrate/dentine or enamel [22], insufficient light-curing of cement [23,24] and restoration thickness [21].

Many authors have studied the relation between those factors and degree of conversion, hardness and bond strength of resin cement. Cho et al. evaluated the effect of thickness (0.3, 0.6, 0.9 and 1.2 mm) on polymerization of two resin cements (light- and dual-cured) and found that the dual-cured resin cements promoted significantly lower degree of conversion and hardness for the groups with 1.2 mm [25]. Similarly, Passos et al. (2012) evaluated the bond strength of a feldspathic ceramic cemented to dentine using different cements (light- and dual-cured) and light-curing methods (halogen and LED) under thermal aging. The authors concluded that the light-curing methods influenced bond strength when light-cured cement was used (LED > halogen) [10].

Several studies [26–28] have evaluated the influence of ceramic thickness, cement type and light-curing time on degree of conversion and hardness of resin cement. To reduce clinical time and perform faster procedures, light-curing unit devices evolved over time showing higher irradiance and shorter time exposure while maintaining the effectiveness of the conversion of monomer [28]. However, few studies evaluated the effect of those factors on bond strength of resin cements to dentine [24]. In this sense, the aim of this study was to evaluate the influence of ceramic thickness and light-curing time on micro-tensile bond strength of a glass ceramic cemented to human dentine submitted or not submitted to thermal cycling (TC). The research hypotheses assumed that (1) ceramic thickness is a negative factor for bond strength, (2) light-curing time influences bond strength, and (3) thermal aging reduces bond strength.

## Material and method

Table 1 shows the materials, brands, manufacturers, and chemical compositions used in this study.

### *Selection and teeth preparation*

This research was approved by the Research Ethics Committee of the Dental School of São José dos Campos, under protocol number 079/2009-PH/CEP. A total of 96 human molars without caries and restorations were cleaned with curettes and stored in distilled water at 18 °C. Each tooth was embedded in self-curing acrylic resin using a silicone mold, and a

**Table 1.** Material, manufacturer, chemical composition and lot of the materials used in the study.

Material	Manufacturer	Chemical composition	Lot
VITA PM9	Vita Zanhfabrik, Bad Sachingen, Germany	Si: 19.6%; Al: 4.9%; K: 4.0%; Na: 2.4%; Ca: 0.7%; C: 25.7% and O: 42.2%	15800
Color: 2M1P-T	Dentsply, Petrópolis, RJ, Brazil	1.0 g contains: <5% hydrofluoric acid	11666
10% hydrofluoric acid	Ivoclar Vivadent, Schaan, Leichtenstein	Ethanol, water and silane	31371
Monobond	Ivoclar Vivadent, Schaan, Leichtenstein	37% phosphoric acid, thickening agent containing pyrogenic silica and surfactant	240909
37% phosphoric acid	Ivoclar Vivadent, Schaan, Leichtenstein	Paste of dimethacrylates (BisGMA, UEDMA, TEGDMA), benzoylperoxide, ytterbium trifluoride, inorganic fillers, initiators, stabilizers, pigment	L38887
Variolink II – color: clear	Ivoclar Vivadent, Schaan, Leichtenstein	Dimethacrylates, alcohol, phosphonic acid acrylate, HEMA, SiO <sub>2</sub> , initiators, stabilizers	M61177
Excite	Ivoclar Vivadent, Schaan, Leichtenstein		

surveyor was used to maintain the occlusal surface parallel to the resin base. The occlusal surface was then sectioned to expose dentine (Labcut 1010, Erios – equipamento técnicos e científicos LTDA, São Paulo, Brazil). The surface was planed and polished with sandpaper # 1200 (3 M, St. Paul, USA) in a polisher (Labpol 8-12, Extex, USA) to standardize the dentine substrate. The teeth were stored in water at 37 °C for 24 h before the cementation process was initiated.

**Fabrication of ceramic blocks**

Sixteen wax patterns were fabricated for each dimension: 6 × 6 × 1 mm, 6 × 6 × 2 mm and 6 × 6 × 3 mm; totaling 48 blocks. The sprues were fixed to the wax patterns and the assembly (pattern/sprue) was embedded in an investment ring. After wax evaporation, the ceramic ingots (VITA PM9, color 2M1P-T, VITA Zanhfabrik) were fused and injected according to the manufacturer’s instructions (EP 600, Ivoclar Vivadent Inc. Amherst, NY, USA).

Next, the ceramic blocks were divested and sandblasted with aluminum oxide in order to remove any investment residue. After sandblasting, the excess ceramic blocks were removed with diamond discs at low speed (KG- Sorensen) and the surface for cementation was planed and polished with sandpaper # 600, 800 and 1200 (3 M ESPE, St. Paul, USA). Thus, the sandblasted surfaces were completely removed and standardized after polishing. The blocks were submitted to different cementation protocols according to each experimental group (Table 2).

**Treatment of dentine surface**

The tooth surface was etched with 37% phosphoric acid (Ivoclar Vivadent, Schaan, Leichtenstein) for 15s, washed for 20s and dried with absorbent paper. Then, a light-cured adhesive system (Excite F, Ivoclar Vivadent) was applied with a disposable applicator according to the manufacturer’s instructions and light-cured for 20s (Radii-cal, SDI Limited, Australia, 1200 mW/cm<sup>2</sup>).

**Table 2.** Experimental groups based on ceramic thickness (3 levels), light-curing time (2 levels) and thermal cycling (TC) (2 levels).

Group	Ceramic thickness	Light-curing time	Thermal cycling
V1 <sub>40s</sub> TC	1 mm	40s	YES
V1 <sub>40s</sub>			NO
V2 <sub>40s</sub> TC			YES
V2 <sub>40s</sub>	NO		
V3 <sub>40s</sub> TC	3 mm		YES
V3 <sub>40s</sub>			NO
V1 <sub>80s</sub> TC		80s	YES
V1 <sub>80s</sub>	NO		
V2 <sub>80s</sub> TC	2 mm		YES
V2 <sub>80s</sub>			NO
V3 <sub>80s</sub> TC			YES
V3 <sub>80s</sub>	NO		

### ***Treatment of ceramic surface***

The ceramic blocks were previously cleaned in ultrasonic bath (Vitasonic, Vita Zahnfabrik, Germany) with distilled water for 5 min. Next, the surface for cementation was etched with 10% hydrofluoric acid (Ivoclar Vivadent, NY, USA) for 1 min, washed with water for 20s and cleaned in ultrasonic bath for 5 min. Finally, the blocks were air-dried for 30s and silanized (Monodond-S, Ivoclar, Vivadent, NY, USA) according to the manufacturer's instructions.

### ***Adhesive cementation***

After dentine etching, the teeth were positioned into a metallic device to standardize the bonding area and the compressive force applied on the ceramic block during cementation. Resin cement (Variolink II, Ivoclar Vivadent, NY, USA) was manipulated for 10s with equal amount of base and catalyst and then applied on the ceramic block surface. The blocks were positioned on dentine and the device was closed to maintain the lateral surfaces away from light. Subsequently, a load of 750 g was exerted on the ceramic block using a torque wrench for 1 min [29]. Light-curing of the cement (Radii-cal, SDI, 1200 mW/cm<sup>2</sup>) was conducted according to each experimental group (40s or 80s), perpendicular to the surface cementing. The samples were then washed with air-water spray and stored in distilled water at 37 °C for 24 h.

### ***Sectioning of the assembly and samples preparation***

The tooth/ceramic assembly was sectioned by a saw (Labcut 1010) under low speed and constant water refrigeration. The interface was positioned perpendicular to the diamond disc (Extex, Enfiel) in order to get rectangular sticks with non-trimmed interface and adhesive interface with area about 1 mm<sup>2</sup> [30,31]. All sticks of each tooth/ceramic assembly were stored in distilled water at 37 °C in an incubator for 24 h.

### ***Thermal cycling***

The sticks of each tooth/ceramic assembly were randomly distributed into two groups, according to the storage condition: no TC (dry) – stick submitted to micro-tensile bond

test 24 h after cutting; TC – stick submitted to TC (12,000 cycles, 5 °C/55 °C, 30s) and micro-tensile bond test immediately after TC.

### **Micro-tensile bond testing**

The area of all sticks was measured before testing using a digital caliper. Each stick was fixed with cyanoacrylate glue parallel to the long axis of the micro-tensile test device in order to minimize torsion at the adhesive zone. The device was fixed in a universal testing machine (EMMIC DL-1000, São José dos Pinhais, Brazil) and the test was conducted at 1 mm/min under 10 kg f.

Bond strength was calculated according to the formula  $R = F/A$ , where  $R$  is resistance,  $F$  is load to rupture (N) and  $A$  is interfacial area (mm<sup>2</sup>).

### **Fracture analysis**

All the tested sticks were analyzed under optical microscopy (Mitutoyo, Japan) with an increase of 50% to determine the pattern of failure at the ceramic/cement/tooth interface.

The failures were classified in four types: (1) adhesive (A): fracture at the interface between cement and ceramic or between cement and dentin; (2) dentine cohesive fracture (CD); (3) ceramic cohesive fracture (CC); (4) Combined (M): combined adhesive fracture with dentine or ceramic cohesive fracture.

### **Statistical analysis**

Data of bond strength was analyzed by 3-way analysis of variance and Tukey test at 5% level of significance using the software Statistix 8.0 for Windows (Analytical Software Inc., Tallahassee, FL, USA).

## **Results**

Bond strength of 2 MPa was assumed for those samples exhibiting failure previous to the test since the micro-tensile bond test does not detect values lower than 4 MPa [32]. Thus, the samples lost during cutting and TC were included in the statistical analysis for comparison between the materials. Table 3 shows a high number of pre-test failures in the V3<sub>40s</sub> TC group, while the V2<sub>80s</sub> group presented less frequent occurrence.

ANOVA revealed that bond strength was significantly influenced by light-curing time ( $p = 0.0001$ ; 40s: 6.2; 80s: 8.7), ceramic thickness ( $p = 0.0001$ ) and TC ( $p = 0.0001$ ; 5.7). Significant interaction was also found between thickness and TC ( $p = 0.0001$ ); thickness and light-curing time ( $p = 0.0001$ ); TC and light-curing time ( $p = 0.0003$ ); and thickness, TC and light-curing time ( $p = 0.0001$ ) (Table 4).

According to the Tukey test, the V280s group ( $17 \pm 9.1$  MPa) showed significantly higher bond strength than the other groups, ranging from 2.7 to 9.4 MPa. The V3<sub>40s</sub> TC group ( $2.7 \pm 6.3$  MPa) presented the lowest bond strength, which was similar to the V1<sub>80s</sub> TC ( $4.6 \pm 4.9$  MPa) and V2<sub>40s</sub> TC groups ( $5.9 \pm 4.4$  MPa) (Table 5).

**Table 3.** Number (*N*) and percentage (%) of samples that exhibited pre-test failures (PTF) during cutting and/or thermal cycling (TC) and total number (*N*) of samples submitted to micro-tensile bond test (MTBs).

Groups	<i>N</i>	<i>N</i> and % of PTF during cutting and TC	Total <i>N</i> and % of samples tested
V1 <sub>40s</sub> TC	80	36 (45)	44 (55)
V1 <sub>40s</sub>	80	40 (60)	40 (50)
V2 <sub>40s</sub> TC	80	41 (59)	39 (48.8)
V2 <sub>40s</sub>	80	27 (73)	53 (66.25)
V3 <sub>40s</sub> TC	80	73 (27)	7 (8.75)
V3 <sub>40s</sub>	80	52 (48)	28 (35)
V1 <sub>80s</sub> TC	80	60 (40)	20 (25)
V1 <sub>80s</sub>	80	29 (71)	51 (63.75)
V2 <sub>80s</sub> TC	80	53 (55)	27 (33.75)
V2 <sub>80s</sub>	80	8 (92)	72 (90)
V3 <sub>80s</sub> TC	80	29 (71)	51 (63.75)
V3 <sub>80s</sub>	80	45 (55)	25 (31.25)

**Table 4.** Analysis of variance (3 factors) for bond strength data (MPa).

Effect	DF	SS	MS	<i>F</i>	<i>p</i>
Thermal cycling	1	2917.7	2917.68	60.67	0.0001*
Thickness	2	2177.5	1088.75	22.64	0.0001*
Light-curing time	1	1530.4	1530.43	31.82	0.0001*
Thickness × Thermal cycling	2	1258.1	629.04	13.08	0.0001*
Thermal cycling × Time	1	639.3	639.34	13.29	0.0003*
Thickness × Time	2	954.2	477.12	9.92	0.0001*
Thickness × Thermal cycling × Time	2	1224.8	612.42	12.73	0.0001*
Residue	948	45,589.8	48.09		
Total	959	56,291.9			

\*statistical significance ( $p < 0.05$ ).

**Table 5.** Mean and standard deviation of bond strength (MPa) for each group according to the factors: thermal cycling (TC), light-curing time and ceramic thickness. (Tukey test,  $\alpha = 0.05$ ).

Thickness	Time			
	40s		80s	
	DRY	TC	DRY	TC
1 mm	7.1 ( $\pm 7.6$ ) <sup>BCD</sup>	7.4 ( $\pm 6.9$ ) <sup>BCD</sup>	9.4 ( $\pm 6.9$ ) <sup>B</sup>	4.6 ( $\pm 4.9$ ) <sup>DE</sup>
2 mm	8.8 ( $\pm 8.1547$ ) <sup>BC</sup>	5.9 ( $\pm 4.4$ ) <sup>BCDE</sup>	17 ( $\pm 9.1$ ) <sup>A</sup>	6.5 ( $\pm 7.9$ ) <sup>BCD</sup>
3 mm	5.7 ( $\pm 6.3624$ ) <sup>CD</sup>	2.7 ( $\pm 6.3$ ) <sup>E</sup>	7.6 ( $\pm 8$ ) <sup>BCD</sup>	7.6 ( $\pm 6.3$ ) <sup>BCD</sup>

( $p < 0.05$ ).

According to the analysis of fractured surfaces of the tested specimens, it can be seen that the predominant failure mode is the mixed type. V1<sub>40s</sub> TC and V1<sub>40s</sub> groups had lower adhesion resistance values and a large amount of cohesive ceramic failure.

## Discussion

Adhesive cementation of metal-free prostheses can be influenced by inappropriate polymerization of cement [33,34], which depends on color, opacity and thickness of the ceramic [35,36], as well as polymerization method and time [37]. The association of those factors with clinical conditions such as occlusal stress [22] and thermal variation [38], tends to reduce bond strength at dentine/ceramic/cement interface, leading to restoration displacement.



In this sense, several methods have been suggested to assess bond strength at adhesive interfaces including shear [39], micro-shear [40], tensile [41], and micro-tensile [42] testing. Although shear testing is commonly used to evaluate adhesion between several substrates because of its simplicity and ease, cohesive failures are frequently observed with this method. Also, data is influenced by non-uniform distribution of stress which is driven towards the material base instead of an adhesive interface as a result of adhesive areas being larger than  $2.0 \text{ mm}^2$  ( $7\text{--}11 \text{ mm}^2$ ) [43]. As a consequence, micro-tensile testing has been widely used to demonstrate the real adhesive force existing between different substrates, while less superficial failures and defects at the adhesive zone are observed [30,44]. Sano et al. [44] demonstrated the inversely proportional relation between adhesive area and bond strength when a reduction in testing area ( $<2.0 \text{ mm}^2$ ) resulted in higher bond strength. In addition, adhesive failure was the most common fracture pattern observed, which means that no cohesive fracture occurred in the substrates. Thus, micro-tensile bond test was selected as an appropriate and advantageous method to evaluate the adhesive interfaces in this study.

According to the results, the hypothesis about a negative influence of ceramic thickness on bond strength was accepted. The groups with thicker ceramics ( $V3_{40s}$ ,  $V3_{40s}T$ ,  $V3_{80s}$  and  $V3_{80s}TC$ ) exhibited lower bond strength at cement/dentine interface. A thicker ceramic probably reduced light intensity at the adhesive interface and affected cement polymerization [45]. Cho et al. stated that appropriate polymerization of resin cements is limited to a maximum thickness of 1.2 mm, so light-curing time or intensity should be increased for ceramics thicker than 0.9 mm. On the other hand, the present study showed worse performance for the 1 mm/80s group in comparison to the 2 mm/80s group. However, all groups with 1 mm-ceramic presented cohesive failure within the ceramic. This fact that can be explained by irregular and porous ceramic surfaces created by etching with hydrofluoric acid [22]. Furthermore, the fragility of 1 mm-samples probably caused cracks in the ceramic during the stick cutting and affected the bond strength of this group [46].

When the fractured surfaces of the tested samples were analyzed, it was observed that the predominant failure mode was the mixed type. However, the 1 mm groups showed a greater number of cohesive ceramic failures due to the small tile thickness (1 mm). This occurred during cutting due to the cutting saw vibration causing cracks in the ceramic structure, making it more friable.

The hypothesis assuming that light-curing time influences bond strength was accepted. In the present study, the highest bond strength was found in all groups submitted to longer polymerization ( $V1_{80s}$ ,  $V2_{80s}$  and  $V3_{80s}$ ), even after TC. Some studies have suggested longer polymerization [47] and high intensity light sources for cementation of thicker ceramic restorations [10] to manage the negative effect of ceramic thickness. This occurs because increasing the time of exposure to light promotes a higher degree of polymerization, and a higher conversion degree of dual resin cement monomers [47]. According to Rasetto et al. [48], light-curing of resin cements for ceramic restorations can be incomplete when low intensity polymerization units are used [48]. In this sense, ISO recommends light intensity of  $300 \text{ mW/cm}^2$  and thickness of 1.5 mm [49]. The light intensity used for polymerization in this study was  $1000 \text{ mW/cm}^2$ .

Lopes et al. evaluated the mechanical properties of different resin cements light-cured for 120s ( $650 \text{ mW/cm}^2$ ) through ceramic discs (feldspathic ceramic, lithium disilicate and zirconia veneered with feldspathic ceramic) with 1.5 mm thickness. The feldspathic ceramics exhibited deeper light transmission, while the other ceramics showed loss of energy



ranging from 11 to 22%. The authors found that light-curing time (120s) can manage the energy density reduction of the ceramics, which affects cement toughness. Thus, longer light-curing can be conducted to enhance polymerization of a dual-cured cement when a thicker ceramic system exhibiting minor light transmission is used [50].

The hypothesis assuming that thermal aging reduces bond strength was accepted. Adhesion between ceramic and cement is susceptible to thermal, chemical and mechanical conditions within an oral environment, so storage in water at a constant temperature and/or TC is frequently used to simulate material adhesion aging [47]. All groups experiencing TC showed reduction in bond strength, most likely as a consequence of hydrolytic degradation of cement/ceramic interface and degradation of the resin cement due to failure between the matrix and inorganic particles [51]. In addition, the substrates (teeth, ceramic and cement) present different coefficients of linear thermal expansion which causes stress and fatigue at adhesive interface when clinical conditions (high and low temperatures during TC) are simulated [32,52]. According to Özcan et al. [22], resin materials absorb water during storage over time and a period of days or weeks are required to achieve maximum absorption. Previous studies evaluating the influence of water absorption on bond strength of resin cements to dentine also found that TC was a negative effect for the experimental conditions [9,10,47].

Thus, within the limitations of this study, further research is required to assess the bond strength of resin cements to dentine regarding ceramic thickness in order to clinically evaluate and validate the parameters used in this study.

## Conclusion

According to the methods, results and limitations of this study, it was concluded that:

- (1) Thermal cycling was a negative effect for bond strength in all light-curing times used in this study.
- (2) Light-curing for 80s increased the bond strength between cement/dentine/ceramic in comparison to light-curing for 40s.
- (3) Significant increase in bond strength was achieved with light-curing for 80s in comparison to 40s, when a thicker ceramic (3 mm) was used.

## Clinical relevance

Longer light-curing (80s) must be conducted for polymerization of dual-cured resin cements when glass ceramic restoration with 3 mm in thickness is used in order to manage the reduction of light intensity at the adhesive interface.

## Acknowledgements

This study was based on a Master Thesis submitted to the Dental School of São Paulo State University (UNESP), São José dos Campos, SP, Brazil as part of the requirements for a MSc degree.

## Disclosure statement

No potential conflict of interest was reported by the authors.

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