

A COMPARISON OF THE FILM THICKNESS OF TWO ADHESIVE LUTING AGENTS AND THE EFFECT OF THERMOCYCLING ON THEIR μ TBS TO FELDSPATHIC CERAMIC

Priscilla C. Pereira¹, Anderson A. Castilho¹, Rodrigo O. A. Souza², Sheila P. Passos¹, Fernando E. Takahashi¹, Marco A. Bottino¹

¹São José dos Campos Dental School, São Paulo State University, São José dos Campos, Brazil

²Federal University of Paraíba (UFPB), Department of Restorative Dentistry, Division of Prosthodontics, João Pessoa, Brazil.

ABSTRACT

The aim of the present study was to evaluate the effect of thermocycling (TC) on the microtensile bond strength (μ TBS) of two luting agents to feldspathic ceramic and to measure their film thickness (FT). For the μ TBS test, sixteen blocks (6.4 x 6.4 x 4.8 mm) were fabricated using a feldspathic ceramic, etched with 10% hydrofluoric acid, rinsed and treated with the silane agent. The ceramic blocks were divided into two groups (n= 8): Gr1: dual-cured resin cement and Gr2: flowable resin. The luting agents were applied on the treated surfaces. Microsticks (1 \pm 0.1mm²) were prepared and stored under two conditions: dry, specimens immediately submitted to the μ TBS test, and TC (6,000 cycles; 5°C-55°C). The μ TBS was evaluated using a universal testing machine (1 mm/min). The μ TBS data (MPa) were submitted to two-way ANOVA and Tukey's test (5%). For the FT

test (ISO 4049), 0.05 ml of each luting agent (n=8) was pressed between two Mylar-covered glass plates (150 N) for 180 seconds and light polymerized. FT was measured using a digital paquimeter (Model 727-2001). The data (mm) were submitted to one-way ANOVA. The luting cement did not influence the μ TBS results (p=0.4467). Higher microtensile bond values were found after TC (20.5 \pm 8.6 MPa) compared to the dry condition (13.9 \pm 4.7 MPa), for both luting agents. The luting agents presented similar film thicknesses: Gr1- 0.052 \pm 0.016 mm; Gr2- 0.041 \pm 0.003 mm. The luting agents presented similar film thickness and μ TBS values, in dry and TC conditions and TC increased the bond strength regardless of the luting agent.

Key words: adhesion, surface treatment, thermocycling, film thickness, luting agents, ceramic.

COMPARAÇÃO DA ESPESSURA DA PELÍCULA DE DOIS AGENTES CIMENTANTES E O EFEITO DA CICLAGEM TÉRMICA NA RESISTÊNCIA DE UNIÃO À MICROTRAÇÃO A UMA CERÂMICA FELDSPÁTICA

RESUMO

Avaliar o efeito da termociclagem (TC) na resistência de união à microtração (μ TBS) de dois agentes cimentantes a uma cerâmica feldspática e mensurar a espessura da película (FT) desses agentes cimentantes. Para o ensaio de μ TBS, dezesseis blocos (6,4 x 6,4 x 4,8 mm) foram fabricados utilizando uma cerâmica feldspática, condicionados com ácido fluorídrico 10%, lavados, secos e aplicado o agente silano na superfície. Os blocos cerâmicos foram divididos em dois grupos (n=8): Gr1: cimento resinoso de polimerização dual e Gr2: resina flow. Os dois agentes cimentantes foram então aplicados sobre as superfícies tratadas. Os palitos (1 \pm 0.1mm²) foram confeccionados e divididos de acordo com duas condições de armazenagem: seco, as amostras foram imediatamente submetidas ao teste de μ TBS, e TC (6.000 ciclos; 5°C-55°C). The μ TBS was evaluated using a universal testing machine (1 mm/min). Os dados de μ TBS (MPa) foram submetidos à Análise de Variância (ANOVA) dois fatores e ao teste de Tukey (5%). Para o teste FT (ISO 4049),

0,05 ml de cada agente cimentante (n=8) foi prensado entre duas placas de vidro (Mylar) (15kg) durante 180 segundos, fotopolimerizadas e a FT foi mensurada utilizando um paquímetro digital (Modelo 727-2001). Os dados (mm) foram submetidos à Análise de Variância (ANOVA) um fator. O agente cimentante não influenciou os resultados de resistência de união à microtração (p=0,4467). Os maiores valores de resistência de união foram encontrados após a TC (20,5 \pm 8,6 MPa) comparado com a condição sem TC (13,9 \pm 4,7 MPa), para ambos os agentes cimentantes. Os agentes cimentantes apresentaram espessuras de película similares: Gr1- 0,052 \pm 0,016 mm; Gr2- 0,041 \pm 0,003 mm). Os agentes cimentantes apresentaram espessura de película e valores de μ TBS, em condição com e sem TC, e que a TC aumentou a resistência de união, independente do agente cimentante utilizado.

Palavras chave: adesão, tratamento de superfície, termociclagem, espessura da película, agente cimentante, cerâmica.

INTRODUCTION

All-ceramic restorations have been used for a long time, mainly due to their excellent esthetic appearance and the excellent mechanical strength of the ceramic systems. However, many factors can affect the longevity of ceramic restorations; such as the type of ceramic¹, sur-

face treatment^{2,3}, luting agent, quality and amount of the remaining dental tissue⁴, adhesive system, thermocycling^{5,6} and mechanical cycling. The main failure found in this type of restoring treatment was debonding. The attachment of ceramic materials to dental tissue is obtained by a chemical and/or mechanical union

of these structures to the luting agents and is considered an important factor for the clinical success of these restorations. Depending on the microstructure of the ceramic material used, bonding of these restorations can be done using the adhesive technique associated with diverse surface treatments^{7,8}. However, glass ceramics (feldspathic, fluorapatite and lithium disilicate) have been widely used for producing partial veneer crowns (inlay, onlay, overlay and veneers) and complete crowns, either using the stratification, pressed, or CAD/CAM techniques^{9,10}. The surface treatment of glass ceramic for adhesive bonding is well established in the literature. Hydrofluoric acid is used to attack the glass phase, producing a retentive surface for micromechanical bonding, maximizing the bond strength between etched ceramic and resin cements. The etched ceramic surface must be coated with a suitable silane^{1,7,12}, which promotes the chemical bond between the silica of the ceramic and the methacrylate groups of the resin luting cements^{13,14}. This process also promotes the wettability of the ceramic surface, enhancing the contact with resin cements¹⁵. Flowable composites have been available for a relatively short time, yet many possible uses have been recommended by the manufacturers. However, there has been limited testing in laboratory or clinical settings of the efficacy of these suggested uses. These composites have lower viscosity, increased wettability, and when polymerized, have increased elasticity when compared to traditional composite resins¹⁷. They are often used in direct resin restorations^{18,19}, adhesive restorations associated with fibers²⁰, marginal repair and sealants²¹. Additionally, flowable composites have been proposed for luting ceramic restorations as inlays

and veneers in a few studies¹¹; this material also has a variety of shades and a lower price than resin cements, which represents an advantage in clinical practice. However, the bond strength durability of the flowable resin/ceramic interface is not well established.

Considering that the bond strength test has crucial importance when evaluating the behavior and longevity of the resin/ceramic interface, the aim of the current study was to evaluate the film thickness and bond durability of two luting agents (resin cement and flowable resin) to a feldspathic ceramic. The null hypotheses were that (1) the microtensile bond strength is not influenced by the thermocycling and (2) the different luting cements do not have different film thicknesses.

MATERIALS AND METHODS

The brand names, material types, main compositions, manufacturers and batch numbers of the products used in the current study are presented in Table 1.

With the assistance of a silicon mold, sixteen blocks (6.4 x 6.4 x 4.8 mm) of feldspathic ceramic (VITA VM7, Dentin 2M1, VITA Zanhfabrik) were produced, according to the manufacturer's instructions. The cementation surface of each ceramic block was leveled and polished in a machine using silicone carbide papers in sequence (600, 800 and 1200 grit) under water cooling (3M ESPE, St. Paul, USA). Impressions were made from each ceramic block after polishing using addition silicone putty (Elite HD, Zhermach, Badia Polesine, Italia, Batch#: 18443). The block was pushed into the silicone to leave 3 mm between the upper portion of the mold and the surface of the block. Thereafter, the cement was injected into this space (thickness: 3 mm) (Figures 1a-b).



Fig. 1a: Ceramic block after polishing.

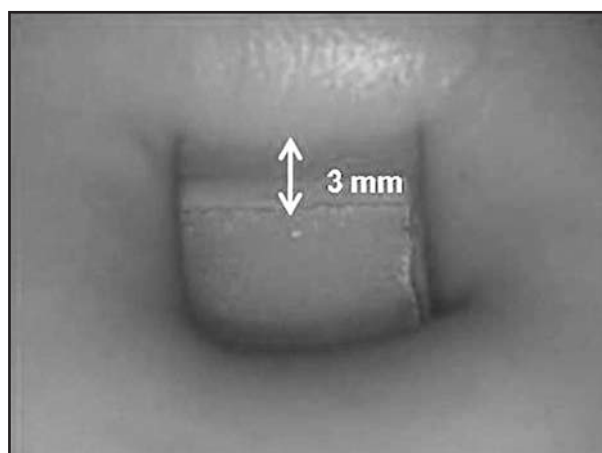


Fig. 1b: Ceramic block positioned inside the silicon mold prior to insertion of the luting agent.

Table 1: Brand names, material types, main components, manufacturers and batch numbers of the products used for the experiments.

Brand Name	Material Type	Main Composition	Manufacture	Batch number
VITA VM7	Feldspathic Ceramic	Si: 19,6%; Al: 4,9%; K: 4,0%; Na: 2,4%; Ca: 0,7%; C: 25,7% e O: 42,2%.	Vita Zanhfabrik, Bad Sachingen, Germany	23920
Ceramic etching gel	Hydrofluoric acid 10%	Fluoridric Acid, water, thickening and stain	Dentsply, Petrópolis, RJ, Brazil	L595588
Monobond-S	Silane agent	Ethanol, water, silane, acetic acid.	Ivoclar Vivadent, Schaan, Leichtenstein	H24764
Filtek Z350 Flow	Flowable composite resin	Bis-GMA, Bis-HEMA, TEGDMA, silica and zircon particles. Inorganic fillers: 55% vol.	3M ESPE, Irvine, CA, USA	5AP
Variolink II	Resin cement	Base: Bis-GMA, UDMA, TEGDMA, inorganic filler, ytterbium trifluoride, initiator, stabilizer. Inorganic fillers: 46.7% vol. Catalyst: Bis-GMA, UDMA, TEGDMA, inorganic filler, ytterbium trifluoride, benzoyl peroxide, stabilizer. Inorganic fillers: 43.6% vol.	Ivoclar Vivadent, Schaan, Leichtenstein	Base: G24884 Catalyst: J09824

Bis-GMA=Bis-phenol-A-glycidylmethacrylate, UDMA=Urethane dimethacrylate, TEGMA=Triethyleneglycol methacrylate and HEMA=2-hydroxyethyl methacrylate.

Prior to surface conditioning, all blocks were ultrasonically cleaned (Vitasonic, Vita Zanhfabrik, Germany) for 5 minutes using distilled water. The ceramic surfaces were etched with 10% HF acid gel (Dentsply, Petropolis, Brazil) for 60 seconds, rinsed with air-water spray for 60 seconds and air-dried. The ceramics were ultrasonically cleaned in distilled water for 5 minutes. The silane coupling agent was then applied (Monobond-S; Ivoclar Vivadent) with a clean brush in one layer and allowed to sit for 5 minutes.

The ceramic blocks were then randomly divided into two groups (n=8) according to the luting agent to be used: Gr1- Variolink II (Ivoclar-Vivadent) and Gr2- Filtek Z350 Flow (3M ESPE). Each block was placed in its silicone mold with the treated surface exposed. The luting agents were bonded to the ceramic surface, following the manufacturer's instructions, and injected into the mold on the treated surface of the ceramic block, using a centrix

syringe (DFL, Rio de Janeiro, Brazil). The cement in the mold was light-activated (XL 3000, 3M ESPE, MN, USA; light output: 500 mW/cm²) for 40 seconds on the upper side of the specimen (incremental technique: 1.5 mm of thickness from each increment). The intensity of the light was verified to be no lower than 500 mW/cm² using a radiometer (Demetron LC, Kerr) before starting light polymerization in each group.

After 10 minutes, the ceramic block-resin cement assembly was removed from the mold and the cement was submitted to light polymerization from the five aspects of the block (upper and laterals) for 40 seconds per side.

Specimen Preparation for the Microtensile Bond Strength Test (μ TBS)

Ceramic-cement blocks were sectioned using a diamond disc (Microdont, São Paulo, Brazil, n°. 34570) at low-speed, under water cooling, in a sec-

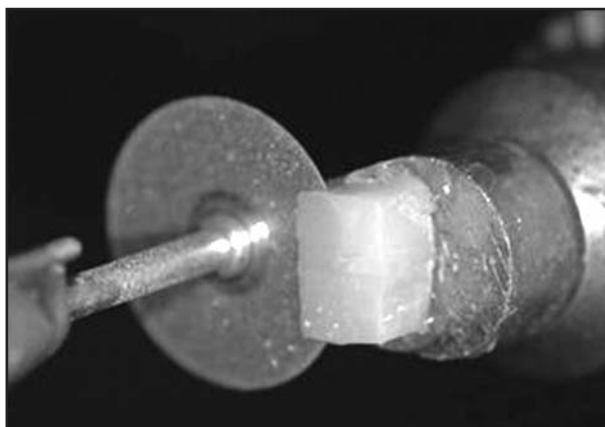


Fig. 2a: Luting agent applied on ceramic block and attached to the metal base for producing the sections.

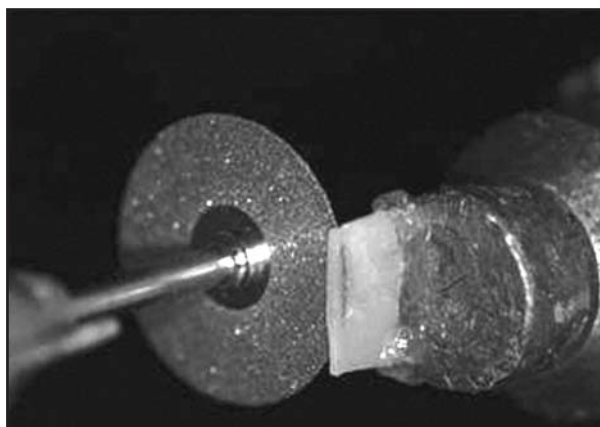


Fig. 2b: Production of microsticks from the sections.

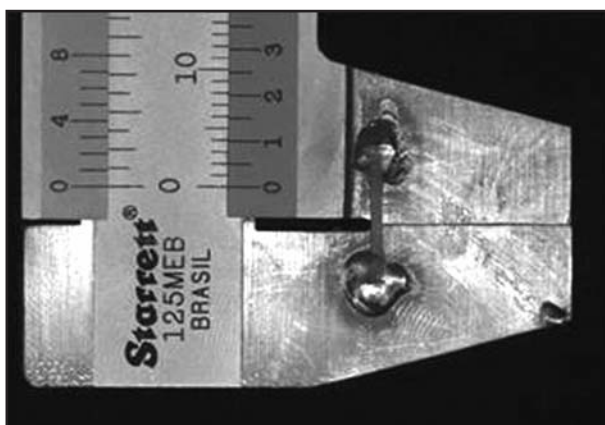


Fig. 2c: Sample positioned on the paquimeter prior to the μ TBST.

tioning machine (LabCut 1010, Extec, Enfield, CT, USA). Initially, the cemented blocks were attached to a metallic base that was attached to the sectioning machine using cyanoacrylate adhesive gel (Super Bonder Gel, Loctite Ltd, São Paulo, Brazil). The blocks were positioned as perpendicularly as possible in relation to the diamond disc of the machine. The first section, measuring approximately 1 mm, was discarded due to the possibility of an excess or absence of cement at the interface that might alter the results. Thereafter, three sections measuring 1.0 ± 0.1 mm in thickness were prepared. Each section was rotated 90° and once again attached to the metallic base. The first microstick was discarded (1 ± 0.1 mm) due to the aforementioned reasons. Subsequently, four additional microsticks were prepared, also measuring 1.0 ± 0.1 mm in thickness. This process was followed for the

other two sections; therefore, only the central microsticks were used for the experiments²². About ten specimens were obtained from each block. The beam specimens had non-machined (non-trimmed) bonding areas with a bonded area measuring approximately 1.0 ± 0.1 mm² and a length of 8 mm. The 10 specimens obtained from each ceramic block were randomly divided into 2 testing conditions. In the dry condition (Dry), specimens were immediately submitted to microtensile testing after sectioning. In the aged condition (TC), specimens were submitted to thermal cycling (6,000 cycles; 5°C - 55°C , dwelling time: 30 s, transfer time: 2 s) (Nova Etica, São Paulo, Brazil) and then submitted to testing. Thus, 4 groups were obtained, considering the “luting agent” (2 levels) and “thermocycling” (2 levels).

Microtensile Bond Strength Test (μ TBST)

Each specimen was attached with cyanoacrylate gel (Super Bonder Gel, Loctite Ltd, São Paulo, Brazil) to the rods of a device adapted for this test, keeping the adhesive zone free. The specimens were positioned parallel to the long axis of the device, in order to reduce the bending stresses. The device was used in a universal testing machine (EMIC DL-1000, EMIC, São José dos Pinhais, Brazil) and testing was performed at a cross-head speed of 1 mm/min²² (Figures 2a-c).

The bond strength was calculated using the formula: $R=F/A$, where “R” is the strength (MPa), “F” is the load required for rupture of the specimen (N) and “A” is the interface area of the specimen (mm²), measured with a digital caliper before the test.

Table 2: Results of 2-way analysis of variance for thermocycling, luting agent and the interaction terms for μ TBS data (* $p < 0.05$).

Effect	DF	SS	MS	F	P
Luting Agent	1	26.72	26.72	0.58	0.4467
Thermocycling	1	874.33	874.33	19.12	0.0001*
Interaction	1	8.31	8.31	0.18	0.6709
Residual	87	3978.10	45.72		
Total	90				

Table 3: Mean (\pm SD) of μ TBST values (MPa) for luting agent/ceramic combinations with and without thermo cycling conditions. *The same superscript letters indicate no significant differences (Tukey's test, $\alpha = 0.05$).

Experimental Groups	Thermocycling		Mean (SD)
	Without	With	
Variolink II (Gr1)	14.98 \pm 4.34 ^{bc}	21.20 \pm 9.36 ^a	17.21 \pm 7.55
Filtek Flow (Gr2)	13.13 \pm 5.55 ^c	20.70 \pm 7.20 ^{a,b}	15.70 \pm 7.23
Mean (SD)	14.41 \pm 4.77	21.06 \pm 8.72	

Fracture analysis

The specimens were analyzed under a stereomicroscope (Stemi 2000-C, Carl Zeiss, Gottingen, Germany) at a magnification of 30x, and the image was digitally recorded with a camera (Cybershot, Model DSC S85, Sony, Tokyo, Japan) connected to the microscope to characterize the ceramic surfaces and the failure modes.

The failure types were classified according to the following criteria: A) adhesive fracture along the interfacial region between the luting agent and ceramic, B) cohesive fracture along the luting agent C) cohesive fracture along the ceramic and D) mixed fracture (adhesive failure between the luting agent and ceramic coupled to cohesive fracture of the luting agent).

Film Thickness

Sample fabrication and thickness measurement

According to ISO Specification No. 4049²³, 0.05 ml of each luting agent (n=8) was extruded from 1.0-ml tuberculin syringes onto a Mylar sheet (Type D, 0.08 inch; DuPont, Wilmington, Del) and placed on the surface of a 3/4-inch-thick polished glass slab. Another piece of Mylar was placed on top of this composite resin, and a similar glass slab was placed on top. Immediately, a vertical load of 150 N was applied to the top glass plate for a period of 180 seconds in a Universal Testing-machine (EMIC DL-1000, EMIC, São José dos Pinhais, Brazil). The top glass plate was then removed and the composite

resin specimen was light polymerized (XL 3000, 3M ESPE, MN, USA; light output: 500 mW/cm²) for 40 seconds at 500 mW/cm² through the Mylar strip to provide a solid disc for thickness measurement.

The film thickness was measured with a digital paquimeter (Model 727-2001), precise to 0.1 mm. Three individual thickness measurements were made for each polymerized specimen and the mean was obtained from these values.

Statistical analysis

Statistical analysis of the film thickness was performed using one-way ANOVA. For the microtensile bond strength test, two-way ANOVA was used and multiple comparisons were made with Tukey's adjustment test, considering the luting agent and thermocycling. P values less than 0.05 were considered to be statistically significant in all tests.

RESULTS

Two-way ANOVA revealed that the microtensile bond strength was significantly affected by thermocycling ($p = 0.0001 < 0.05$) (Table 2). The luting agents presented similar μ TBS values in the dry (Gr1: 15.0 \pm 4.3 MPa; Gr2: 13.1 \pm 5.5 MPa) and aged conditions (Gr1: 21.2 \pm 9.3 MPa; Gr2: 20.7 \pm 7.2 MPa). However, when the dry and TC conditions were compared for each luting agent, the TC condition presented μ TBS values that were significantly higher than in the dry condition, regardless of the luting agent (Table 3). All groups

Table 4: Number (No) of specimens (sp) produced, and percentage (%) of pre-test failures (PTF) during cutting and thermocycling (TC) and total number of sp prior to μ TBST.

Groups	No of sp	No and % of PTF during cutting	No and % of spontaneous PTF during TC	Total no and % of PTF prior to μ TBST	No and % of tested sp in μ TBST
Variolink II	80	32 (40)	0	32 (40)	48 (60)
Filtek flow	80	46 (57.5)	0	46 (57.5)	34 (42.5)

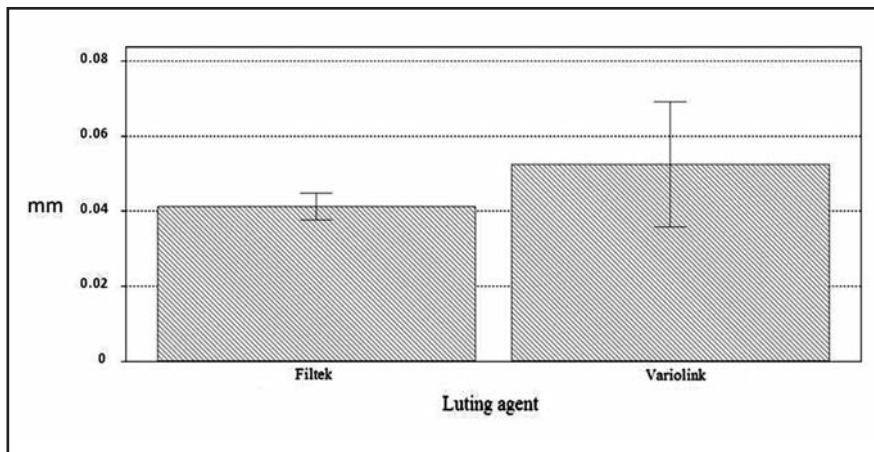


Fig. 3: Graphic representation (mean and SD) of film thickness results for the different luting agents tested.

presented premature debonding during specimen preparation, but the flowable resin presented a higher incidence than the resin cement (Table 4). The film thickness did not differ between the luting agents ($p=0.1203>0.05$): Gr1- 0.041 ± 0.003 mm and Gr2- 0.052 ± 0.016 mm (Figure 3).

The fracture analysis of the specimens revealed different fracture patterns: adhesive failure along the interfacial region between the luting agent and the feldspathic ceramic (Score A); cohesive fracture in the ceramic (Score B); cohesive fracture in the luting agent (Score C) and mixed failure (cohesive fracture of the luting agent combined with the adhesive failure) (Score D), with and without thermocycling. The percentages of the types of fractures are presented in Table 5 and a representative micrograph of a mixed fracture is presented in Figure 4. The pattern of failure was very similar in all groups and the failures were predominantly mixed (Score D).

The null hypotheses were partially accepted.

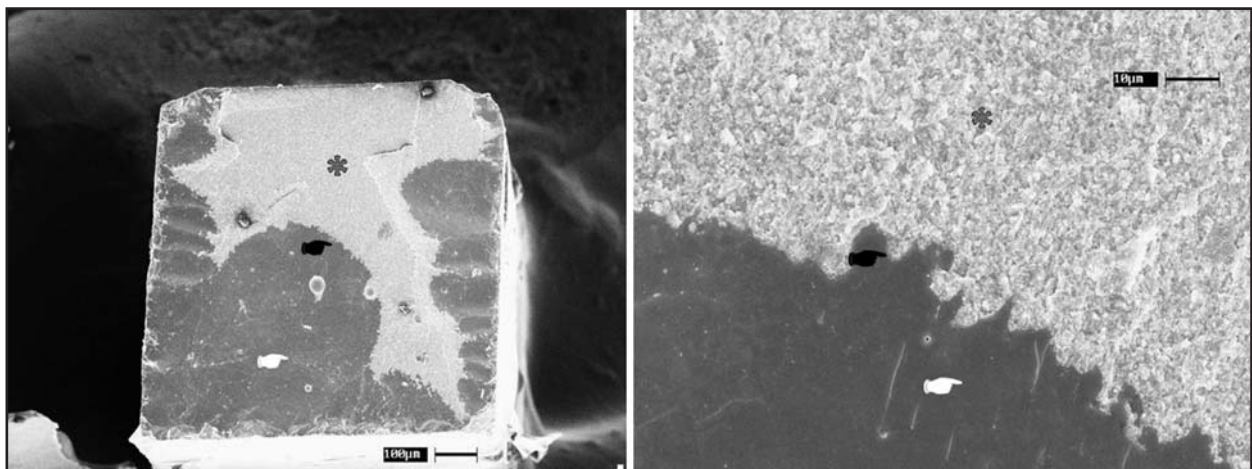


Fig. 4: Representative micrographs of a mixture fracture (Score D) between ceramic and cement layer of a specimen from group 2: a- 75x and b- x200. Asterisk * = resin cement; \blackrightarrow = rupture of the ceramic-resin cement interface; \blacklozenge = ceramic.

Table 5: Incidence of failure types (%) after the μ TBST. A: Adhesive failure along the interfacial region between the luting agent and the feldspathic ceramic; B: cohesive fracture in the feldspathic ceramic, C: cohesive fracture in the luting cement and D: mixed failure (cohesive fracture of the cement combined with the adhesive failure).

Experimental Groups	Thermocycling	
	Without Score A B C D	With Score A B C D
Variolink II	A(9) B(0) C(4) D(87)	A(35) B(0) C(5) D(60)
Filtek Flow	A(14) B(8) C(8) D(70)	A(15) B(8) C(0) D(77)

DISCUSSION

Several in vitro methods for measuring the resin-ceramic bond strength have been described, including many microtensile^{24,25} and shear bond strength tests²⁶. Della Bona et al.²⁷ found that the shear bond strength test created arch-shaped cohesive fractures in all samples. These failures occur because of the highly non-uniform tension distribution on the interface of the materials. Therefore, the microtensile bond strength test is considered appropriate for in vitro assessment of resin composites bonded to ceramic, as failure occurs at the adhesive interface and not in the substrate¹³. For this reason, this test was chosen for the current study.

This laboratory study was designed to investigate the influence of a dual-cure luting agent (Variolink II) and flowable composite resin (Filtek Z350 Flow) on the microtensile bond strength to feldspathic ceramic (VITA VM7). The bond strength was similar between the cements (mean range: 13.13 to 21.20 MPa), in dry and aged conditions. Similar results were found by Zohairy et al.²⁵, who observed that there were no significant differences in microtensile bond strength between a flowable resin (Tetric Flow) and resin cements (Nexus 2 and Rely X) when bonded to a ceramic or resin CAD/CAM block.

When the silica-based ceramic is etched with hydrofluoric acid (5-10%), the glassy matrix of the ceramic is dissolved from the surface to a depth of a few microns. This treatment changes significantly the surface morphology, where the pores created on the surface are the most important descriptive pattern for the ceramic treated with hydrofluoric acid. This increase of the surface area favors the infiltration and retention of adhesive materials and makes the ceramic surface more retentive^{1,8}. Saracoglu et al.⁴⁰ evaluated the influence of different etching protocols on a silica based ceramic and their effect on

the bond strength to resin cement. The scanning electron microscopy evaluation revealed that the hydrofluoric acid (5-10%) produced pores as the most important descriptive pattern for this treatment, whereas the orthophosphoric acid (40%) did not change the ceramic surface morphology. Furthermore, the hydrofluoric acid treatment increased the shear bond strength between ceramic and resin cement.

Another important aspect of resin bond to silica-based ceramics is the application of a silane coupling agent. The silane agent is a bifunctional molecule that promotes the chemical bonding with organic surfaces such as resin materials and polymers and inorganic surfaces, such as silica-based ceramics⁴¹. This agent reacts with the silica oxide present in feldspathic ceramics or with oxides artificially deposited on alumina and zirconia based ceramics, creating a favorable bonding^{2,42}. On the other hand, the silane agent increases the wettability of the luting agents on the ceramic surface, optimizing the bond strength results. Thus, hydrofluoric acid conditioning followed by the application of the silane agent on the ceramic promote conditions that favor the chemical bonding between the ceramic and flowable resin.

Barceleiro et al.²⁸ analyzed the shear bond strength of feldspathic ceramic to bovine enamel luted with dual-cured resin cement and light-cured flowable composite. They verified that both luting agents presented similar results and that flowable composites are a suitable alternative when used for porcelain laminate veneer bonding, since these veneers are generally slim (less than 2 mm) and light polymerized luting agents can be light cured, providing satisfactory bond strength to these substrates^{29,30}. Other authors also agree with the statement that a flowable composite can be used to lute indirect composite³¹ and ceramic restorations³⁰,

since this material has excellent characteristics, such as sufficient wear resistance¹⁷, greater fluidity and approximately 80% of the flexural strength of regular composites³², viscoelasticity³³ and thin film thickness²¹.

Thermocycling is a method frequently used for simulating intra-oral aging, combining hydrolytic and thermal degradation. In this study, half of the specimens from each group were subjected to 6,000 thermal cycles. The results revealed that thermocycling increased the μTBS for both luting agents. Although the cycle number used in this study was above the recommended number of cycles by ISO Specification No 10477³⁴ (500 cycles), one limitation of this study could still be the short-term water storage and reduced thermal cycling in comparison to other studies, making it difficult to predict the long-term durability of the tested bonding methods^{2,22,24}. Generally, thermocycling influences the bond strength results. The bond durability between the ceramic surface and the resin luting agent decreases with water storage and thermocycling². During thermocycling, the soluble components of the resinous materials are removed and the water is absorbed to replace them. Additionally, water causes hydrolysis of the interface matrix and can cause fissures in the polymer matrix, both of which contribute to the reduction of resin properties^{35,36}. This fact may also be responsible for the degree of degradation of the bond strength between the ceramic-resin interface during water storage, and this degradation is proportional to the time of storage^{37,38}. However, the results of the current study showed that the μTBS was significantly higher for all luting agents after thermocycling (Gr1: from 14.98 ± 4.34 MPa to 21.20 ± 9.36 MPa; Gr2: from 13.13 ± 5.55 MPa to 20.70 ± 7.20 MPa). These results can be explained by the short-term water storage associated with the temperature of thermocycling (55°C), which increased the degree of conversion for the luting agents, improving the bond strength of these materials to ceramic³⁹.

Pre-testing failures were experienced in this study (Table 4), similarly to other micro-tensile bond strength studies that showed debonding during the cutting procedure to make the microbars^{3,13,25}, but in contrast to the study of Peumans et al.⁶. The analysis of the fractured surfaces by light microscopy and SEM showed that most of the fail-

ures were mixed (Figs 4a-b), similarly to the studies of El Zohairy et al.²⁵ and Meyer Filho et al.³, who also evaluated the bond between glass ceramics and resin cement (Table 5).

The film thickness analysis showed that Filtek Z350 Flow (0.041 ± 0.003 mm) was similar to the resin cement, Variolink (0.052 ± 0.016 mm). This property is related with the percentage of inorganic filler of the materials. According to the manufacturers of these composites, they have a similar quantity of inorganic fillers: Filtek flow (55% vol.) and Variolink II (43.6% vol.), making them of similar viscosity. The film thickness revealed that the standard-deviation of flowable resin composite was smaller than for the resin cement. This can be explained by the single component of the flowable resin composite. Resin cements are composed of base and catalyst pastes, which have different compositions and might not be mixed exactly in the same amount. Similar studies^{21,32,43} observed thickness values ranging from 0.003 to 0.06 for different flowable composites and resin cements, which makes a flowable resin similar to the resin composite in terms of the marginal discrepancy of indirect restorations luted with these materials.

Finally, the findings of the current study seem to have an important clinical relevance for adhesive luting of indirect restorations. Thus, the use of a flowable resin to lute indirect ceramic or resin restorations can yield similar results when compared to the resin cement^{25,28}. However, this would occur only in those cases where the restorations have a thickness of less than 2 mm since the flowable resin needs a good intensity of light to provide satisfactory bond strength to the enamel/dentin/ceramic substrates^{29,30} and acceptable film thickness^{21,32,43}. So, according to the literature and to the findings of this study, the flowable resin is indicated for luting glass ceramic restorations and indirect resin restoration as veneer and inlay.

Further long-term in vitro and prospective clinical studies using mechanical fatigue tests must be conducted for confirmation of the clinical argument.

CONCLUSION

Based on the results, thermocycling increased the bond strength between ceramic-resin regardless of the luting agent. The flowable composite resin and the resin cement have similar film thicknesses.

CORRESPONDENCE

Dr. Rodrigo O. A. Souza
 Federal University of Paraíba
 Department of Restorative Dentistry
 Rua Praia de Guajirú, 9215
 Ponta Negra, Natal/RN
 Brazil, Zip Code: 59.092-220
 e-mail: roasouza@yahoo.com.br

REFERENCES

- Spohr AM, Sobrinho LC, Consani S, et al. Influence of surface conditions and silane agent on the bond of resin to IPS Empress 2 ceramic. *Int J Prosthodont* 2003;16:277-282.
- Özcan M, Vallittu PK. Effect of surface conditioning methods on the bond strength of luting cement to ceramics. *Dent Mater* 2003;19:725-731.
- Meyer Filho JA, Viera LCC, Araújo E, Monteiro Júnior S. Effect of different ceramic surface treatments on resin microtensile bond strength. *Int J Prosthodont* 2004;13:28-35.
- Hayashi M, Tsuchitani Y, Kawamura Y, Miura M, Takeshige F, Ebisu S. Eight-year clinical evaluation of fired ceramic inlays. *Oper Dent* 2000;25:473-481.
- Andreatta Filho OD, Galhano G, Bottino MA, Camargo FP, Valandro LF, Nishioka RS. Avaliação da resistência adesiva entre uma cerâmica aluminizada e um cimento resinoso submetidos à ciclagem térmica. *Cienc Odontol Bras* 2003 jul./set.;6:32-39.
- Peumans M, Hikita K, Munck J, Van Landuyt K, Poitevin A, Lambrechts P, Van Meerbeek B. Effects of ceramic surface treatments on the bond strength of an adhesive luting agent to CAD-CAM ceramic. *J Dent* 2007;35:282-288.
- Roulet JF, Soderholm KJ, Longmate J. Effects of treatment and storage conditions on ceramic/composite bond strength. *J Dent Res* 1995;74:381-7.
- Brentel A, Özcan M, Valandro LF, Alarça LG, Amaral R, Bottino MA. Microtensile bond strength of a resin cement to feldspathic ceramic after different etching and silanization regimens in dry and aged conditions. *Dent Mater* 2007;23:1323-1331.
- Chang JC, Hart DA, Estey AW. Tensile bond strengths of five luting agents to two CAD-CAM restorative materials and enamel. *J Prosthet Dent* 2003;90:18-23.
- Liu P, Isenberg BP, Leinfelder KF. Evaluating CAD-CAM generated ceramic veneers. *J Am Dent Assoc* 1993;124:59-63.
- Peumans M, Van Meerbeek B, Lambrechts P, Vanherle G. Porcelain veneers: a review of the literature. *J Dent* 2000;28:163-77.
- Aida M, Hayakawa T, Mizukawa K. Adhesion of composite to porcelain with various surface conditions. *J Prosthet Dent* 1995;73:464-470.
- Della Bona A, Anusavice KJ, Shen C. Microtensile strength of composite bonded to hot-pressed ceramics. *J Adhes Dent* 2000;2:305-313.
- Della Bona A, Shen C, Anusavice KJ. Work of adhesion of resin on treated lithia disilicate-based ceramic. *Dent Mater* 2004;20:338-344.
- Matinlinna JP, Lassila LVJ, Özcan M, Antti Y, Vallittu PK. An introduction to silanes and their clinical applications in dentistry. *Int J Prosthodont* 2004;17:155-164.
- Kern M, Thompson VP. Bonding to glass infiltrated alumina ceramic: adhesive methods and their durability. *J Prosthet Dent* 1995;73:240-249.
- Bayne SC, Thompson JY, Swift EJ Jr, Stamatiades P, Wilkerson M. A characterization of first-generation flowable composites. *J Am Dent Assoc* 1998;129:567-577.
- Erfes B, Dörter C, Gormez Y, Koray F. Two year clinical evaluation of ormocer and nanofill composite with and without a flowable liner. *J Adhes Dent* 2006;8:119-126.
- Korkmaz Y, Ozel E, Attar N. Effect of flowable composite lining on microleakage and internal voids in class II composite restorations. *J Adhes Dent* 2007;9:189-194.
- Belli S, Orucoglu H, Yildirim C, Gürçan E. The effect of fiber placement or a flowable resin lining on microleakage in class II adhesive restorations. *J Adhes Dent* 2007;9:185-181.
- Moon PC, Tabassian MS, Culbreath TE. Flow characteristics and film thickness of flowable resin composites. *Oper Dent* 2002;27:248-253.
- Foxton RM, Pereira PNR, Nakajima M, Junji T, Hiroyuki M. Durability of the dual-cure resin cement / ceramic bond with different curing strategies. *J Adhes Dent* 2002;4:49-59.
- International Organization for Standardization. ISO 4049. Dentistry - polymer-based filling, restorative and luting materials; 2000.
- Hooshmand T, van Noort R, Keshvad A. Bond durability of the resin-bonded and silane treated ceramic surface. *Dent Mater* 2002;18:179-188.
- El Zohairy AA, De Gee AJ, Mohsen MM, Feilzer AJ. Microtensile bond strength testing of luting cements to prefabricated CAD/CAM ceramic and composite blocks. *Dent Mater* 2003;19:575-583.
- Sorensen JA, Engelman MJ, Torres TJ, Avera SP. Shear bond strength of composite resin to porcelain. *Int J Prosthodont* 1991;4:17-23.
- Della Bona A, van Noort R. Shear vs. tensile bond strength of resin composite bonded to ceramic. *J Dent Res* 1995;74(9):1591-1596.
- Barcelheiro M de O, De Miranda MS, Dias KR, Sekito T Jr. Shear bond strength of porcelain laminate veneer bonded with flowable composite. *Oper Dent*, 2003;28(4):423-428.
- Akgungor G, Akkayan B, Gaucher H. Influence of ceramic thickness and polymerization mode of a resin luting agent on early bond strength and durability with a lithium disilicate-based ceramic system. *J Prosthet Dent* 2005;94:234-241.
- Bitter K, Paris S, Hartwig C, Neumann K, Kielbassa AM. Shear Bond strength of different substrates bonded to lithium disilicate ceramics. *Dent Mat J* 2006;23(3):493-502.
- Okuda M, Nikaido T, Maruoka R, Foxton RM, Junji T. Microtensile bond strengths to cavity floor dentin in indi-

- rect composite restorations using resin coating. *J Esthet Restor Dent* 2007;19(1):38-46; discussion 47-8.
32. Attar N, Tam L, McCom D. Flow, strength, stiffness and radiopacity of flowable resin composites. *J Can Dent Assoc* 2003;69(8):516-521.
33. Lee IB, Son HH, Um CM. Rheologic properties of flowable, conventional hybrid, and condensable composite resins. *Dent Mater* 2003 Jun;19(4):298-307.
34. International Organization for Standardization. ISO 10477 Polymer-based, Crown and Bridge Materials, Amendment 1; 1998.
35. Ferracane JL, Hopkin JK, Condon JR. Properties of heat-treated composites after aging in water. *Dent Mater* 1995; 11:354-358.
36. Umemoto K, Kurata S. Effects of mixed silane coupling agent on porcelain tooth material and various dental alloys. *Dent Mater* 1995;14:135-142.
37. Drummond JL, Novickas D, Lenke JW. Physiological aging of an all-ceramic restorative material. *Dent Mater* 1991; 7:133-137.
38. Ortengren U, Wellendorf H, Karlsson S, Ruyter IE. Water sorption and solubility of dental composites and identification of monomers released in an aqueous environment. *J Oral Rehabil* 2001;28:1106-1115.
39. Ferracane JL, Berge HX, Condon JR. In vitro aging of dental composites in water-effect of degree of conversion, filler volume, and filler/matrix coupling. *J Biomed Mater Res* 1998;42:465-472.
40. Saraçoglu A, Cura C, Çotert HS. Effect of various surface treatment methods on the bond strength of the heat-pressed ceramic samples. *J Oral Rehab* 2004;31:790-797.
41. Jardel V, Degrange M, Picard B, Derrien G: Correlation of topography to bond strength of etched ceramic. *Int J Prosthodont* 1999;12:59-64.
42. Soderholm KJM, Shang SW. Molecular orientation of silane at the surface of colloidal silica. *J Dent Res* 1993; 72:1050-1054.
43. Blalock J, Holmes R, Rueggeberg F. Effect of temperature on unpolymerized composite resin film thickness. *J Prosthet Dent* 2006;96:424-432.