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Resistência de união à cerâmica ZLS em diferentes tempos de condicionamento ácido e protocolos de cimentação, após envelhecimento

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Dissertação apresentada ao Programa de Pós-Graduação em Odontologia, Centro de Ciências Biológicas e da Saúde, Universidade Estadual do Oeste do Paraná, como requisito parcial para obtenção do título de Mestre em Odontologia

Área de concentração: Odontologia

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DEDICATÓRIA

“ Dedico este trabalho ao meu pequeno prodígio Theo, para que ele possa ver o resultado e entender que tudo na vida é uma somatória de muito esforço, persistência e dedicação.”

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Resistência de união à cerâmica ZLS em diferentes tempos de condicionamento ácido e protocolos de cimentação, após envelhecimento

RESUMO

Objetivo. Avaliar a influência de, dois tempos de condicionamento ácido, três diferentes protocolos de cimentação e do envelhecimento por ciclagem térmica, na resistência de união entre a cerâmica de silicato de lítio reforçada com zircônia e um cimento resinoso adesivo.

Método. Blocos cerâmicos foram seccionados em 120 fatias com 1,4 mm de espessura e distribuídos aleatoriamente em 12 grupos (n = 10). Após o condicionamento com ácido fluorídrico à 5% por 20 ou 30s, a área adesiva foi delimitada com fita isolante e silanizada. Cilindros de cimento resinoso foram cimentados a partir de uma matriz de teflon, seguindo três diferentes combinações: Silano (Si) + Cimento (Ci), Si + Adesivo (Ad) + Ci e Ad + Ci. Após a fotopolimerização através da cerâmica, as amostras foram armazenadas em água deionizada a 37º C durante 7 dias e então submetidas ao teste de resistência de união ao microcisalhamento.

Resultados. Após teste de normalidade, os dados foram analizados pelo teste ANOVA mostrando que houve diferença estatística ($p<0.01$) para os três fatores isolados; para a interação dupla entre os fatores tempo de condicionamento ácido e termociclagem ($p<0.05$), e também para a interação tripla dos fatores ($p<0.05$).

Significância. O condicionamento com ácido fluorídrico a 5% durante 30 s e a utilização do protocolo de cimentação Adesivo Scotchbond™ Universal + Cimento RelyX™ Ultimate, apresentou maior resistência de união adesiva após o envelhecimento térmico, para a cerâmica Suprinity®.

Palavras-chave: Resistência ao cisalhamento, Cimentos de resina, Cerâmica

Bond strength to ZLS ceramic at different etching times and cementation protocols after aging

ABSTRACT

Objective. To assess the influence of two etching times, three different cementation protocols, and thermal cycling aging on the bond strength between a zirconia-reinforced lithium silicate ceramic and an adhesive resin cement.

Method. Ceramic blocks were sectioned in 120 slices with thickness of 1.4 mm and randomly distributed in 12 groups (n = 10). After 5% hydrofluoric acid etching for 20 or 30 s, the adhesive area was delimited with a duct tape and silanized. Resin cement cylinders were cemented from a Teflon mold, following three different combinations: Silane (Si) + Cement (Ci), Si + Adhesive (Ad) + Ci, and Ad + Ci. After photopolymerization through the ceramic, the samples were stored in deionized water at 37°C for 7 days, and then subjected to the microshear bond strength test.

Results. After the normality test, data were analyzed by ANOVA, showing statistical difference ($p<0.01$) for the three factors isolated, the double interaction between the factors of etching time and thermal cycling ($p<0.05$), and the triple interaction of the factors ($p<0.05$).

Significance. The 5% hydrofluoric acid etching for 30 s and the use of the cementation protocol Scotchbond™ Universal Adhesive + RelyX™ Ultimate Cement presented higher adhesive bond strength after thermal aging, for Suprinity™ ceramics.

***Keywords*:** Shear bond strength, Resin composite cement, Ceramic

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SUMÁRIO

Artigo: "Bond strength to ZLS ceramic at different etching times and cementation protocols after aging"

1. Introduction

Dental ceramics are widely used as aesthetic restorative material for their ability to simulate the natural appearance of teeth [1]. Among the ceramic materials available in the market are silica-based ceramics, which are glassy and may be reinforced with leucite crystals, lithium disilicate, fluorapatite, and zirconia; ceramics with high crystalline content and low glass matrix such as zirconia and alumina; and ceramics with organic matrix highly filled with ceramic particles [2].

For clinical success, these restorations depend on adequate adhesion [3] from the chemical and/or mechanical bond of resin cement to ceramic and tooth substrate. The bond between resin cement and glass-ceramics depends on hydrofluoric acid (HF) etching [1], which is only possible in silica-based ceramics (SiO_2) or glass-ceramics [4, 5]. On the etched surface, the acid reacts by selectively dissolving the glass matrix and exposing the crystalline content. As a result, the surface becomes rough, allowing micromechanical retention [6].

The application of silane is required after etching [5, 7, 8], promoting the chemical bond between ceramic and resin cement for having bifunctional molecules that allow bonding organic compounds (monomers of resin cements) and inorganic compounds (silica present in the glass matrix of ceramics) [7]. It has the property of increasing ceramic surface wettability, improving the ability of the resin cement to bond to the surface [7] for providing more infiltration and contact of the cementing agent on ceramic surface porosities [5], thus facilitating polymer interconnections [9].

Along with the process of incorporating crystals to improve the mechanical properties of ceramics, a new generation of glass-ceramics was developed, resulting in the Suprinity zirconia-reinforced lithium silicate (ZLS) ceramic. In the pre-crystallized state, its microstructure presents two crystalline phases: lithium metasilicates, which are round and elongated submicrometer crystallites; and lithium orthophosphates, which are round nanometer granules. After crystallization, the crystalline phase is presented as lithium disilicate. Zirconia cannot be identified in its particle form because of the low concentration, but it is suggested to be dissolved in the glassy phase [10, 11]. It presents a homogeneous and thin microstructure [12] with high glass matrix content [13], excellent mechanical properties [14], and good visual and polishing qualities [15].

Zirconia is a compound that does not allow mechanical and/or chemical bond to resin cement using hydrofluoric acid and silane. Therefore, this bonding requires the MDP (10-Methacryloyloxydecyl dihydrogen phosphate) phosphate monomer [16, 17, 18], which is a bifunctional organic molecule where one of its edges bonds to the oxides and the other presents groups that copolymerize with the resin matrix of cements. The MDP ester phosphate monomer promoted direct bond to oxides, such as zirconia oxide [19]. However, the chemical reactions formed between hydroxyl groups of the MDP monomer and hydroxyl groups on the zirconia ceramic surface weaken after thermal cycling [17].

According to the manufacturer Vita Zahnfabrik, the ZLS ceramic should be etched with 5% hydrofluoric acid for 20 s, but Dentsply, the manufacturer of CELTRA Duo ceramic, which is also ZLS, indicates an etching time of 30 s, considering that etching time may directly influence bond strength between ceramic and resin cement [6, 13, 20].

The RXU adhesive resin cement (RelyX™ Ultimate 3M ESPE) combined with the SBU adhesive (Scotchbond™ Universal 3M ESPE) is a cement recommended for glass-ceramic cementation and has presented good results for bond strength to zirconia after aging [18]. It is composed by all the primers for indirect restorations, such as MDP, silane, and adhesive [21, 22]; hence, according to the manufacturer, it may be applied to three different cementation protocols: silane + cement (Si+Ci), silane + adhesive + cement (Si+Ad+Ci), or adhesive + cement (Ad+Ci).

In order to simulate the aging process in the oral cavity, water storage [19, 23, 24], autoclave [25], and thermal cycling [17, 21, 26, 27, 29, 30, and 31] may be used among others, considering these procedures influence the bond strength of cemented materials [18].

This study aimed to assess the effect of two different 5% hydrofluoric acid etching times, three different cementation protocols, and thermal cycling aging on the bond strength of Relyx™ Ultimate (3M ESPE) resin cement to Suprinity ZLS ceramic.

The null hypothesis is that etching at different times with different cementation protocols, and thermal cycling aging do not affect the bond strength of RXU resin cement to Suprinity ZLS ceramic.

2. Method

Suprinity ceramic blocks in HT 0M1 shade were sliced (120 slices) with dimensions of 12 x 7 x 1.4 mm by a precision cutting machine with water cooling (Labcut 1010 extec, Enfield, USA). Each slice was polished with a #600 sandpaper for 1 min in the polisher (Arotec PL4, São Paulo, Brazil). Then, the slices were crystallized according to manufacturer's instructions in a Vita Vacumat oven (Vita Zahnfabrik, Bad Säckingen, Germany). The sample was calculated at 5% α and 95% statistical power.

2.1. Ceramic surface treatment

The 120 ceramic slices were randomly distributed in 6 groups according to etching times (20 and 30 s) and cementation protocols (Si + Ci, Si + Ad + Ci, and Ad + Ci), and were then subdivided (n=10) for thermal cycling aging (with or without), as Figure 1 shows.

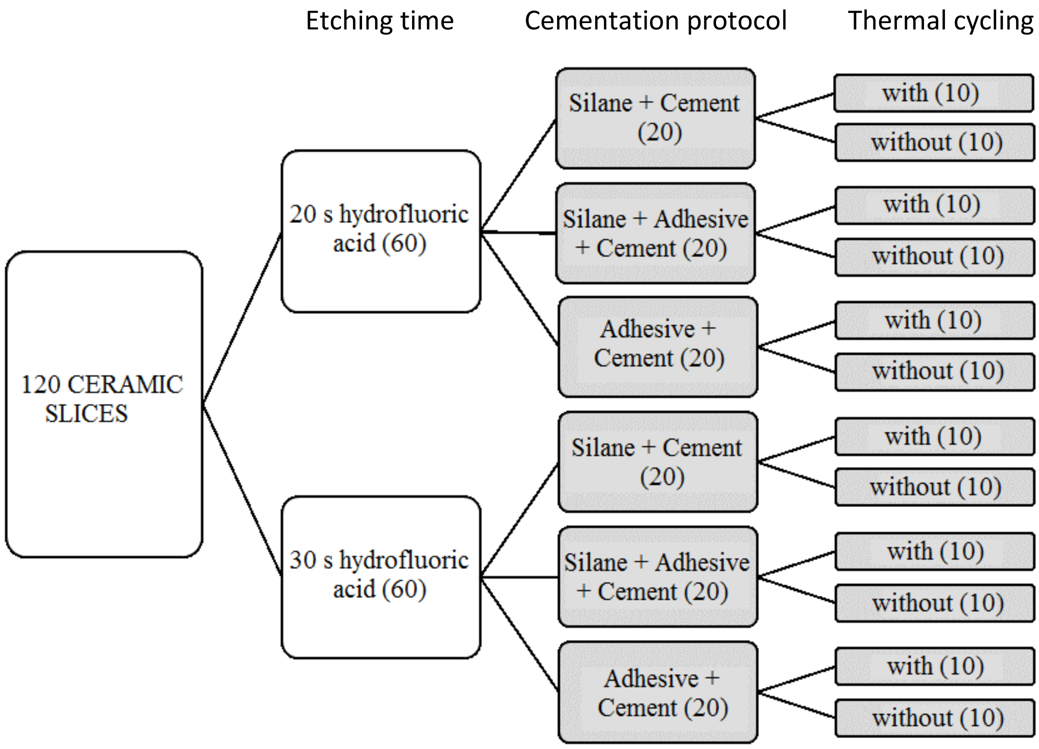


Figure 1 – Schematic drawing of the experimental design

The ceramic slices were etched with 5% HF (Condac porcelana, FGM, Joinville, Brazil) for 20 or 30 s, according to the group. They were washed in abundant water for 1 min and dried by air blast. A piece of duct tape (Imperial, 3M, Sumare, Brazil) with 4 holes aligned and prepared with an Ainsworth perforator was fixed on each ceramic slice to delimit the bonding area. Each cementation protocol was determined by a tape color to avoid confusion: red for Si + Ci protocol, yellow for Si + Ad + Ci protocol, and blue for Ad + Ci protocol.

2.2. Cementation procedure

Protocol Si + Ci - Two consecutive applications of silane (RelyX Ceramic Primer, 3M ESPE, St Paul, MN, USA) activated for 5 s with a microbrush, followed by air blast until drying. Approximately 1 mm of RXU resin cement (RelyX Ultimate 3M ESPE, St Paul, MN, USA) filled the transparent Teflon tube (Solidor, Haryana, India) with internal diameter of 0.76 mm, placed in a ceramic surface (perforated area), and photopolymerized for 100 s through the ceramic with 1000 mW/cm^2 (Valo™ Cordless, Ultradent, South Jordan, UT, USA).

Protocol Si + Ad + Ci - Two consecutive applications of silane activated for 5 s with a microbrush, followed by air blast until drying. Application of SBU adhesive (Scotchbond™ Universal adhesive, 3M ESPE, St Paul, MN, USA) activated for 5 s with a microbrush, excess removal, and solvent evaporation by air blast. Approximately 1 mm of resin cement RXU filled the transparent Teflon tube with internal diameter of 0.76 mm, placed on a ceramic surface with no tape (perforation), and photopolymerized for 100 s through the ceramic.

Protocol Ad + Ci - Application of SBU adhesive activated for 5 s with a microbrush, excess removal, and solvent evaporation by air blast. Approximately 1 mm of RXU resin cement filled the transparent Teflon tube with internal diameter of 0.76 mm, placed on a ceramic surface with no tape (perforation), and photopolymerized for 100 s through the ceramic.

The ceramic slices were fixed with epoxy resin (Durepoxi, Loctite, Itapevi, Brazil) to PVC cylinders filled with acrylic resin, and were stored in deionized water at 37°C for 7 days. Table 1 describes the composition of materials used in these studies.

Table 1 - Manufacturers and composition of materials used in this study

Material	**Batch**	**Composition**
Suprinity; Vita Zahnfabrik, Bad Säckingen, Germany	51590	Silicon dioxide 56-64% in weight, lithium oxide 15-21% in weight, zirconia 8-12% in weight, and others > 10% in weight.
Condac porcelana, FGM, Joinville, Brazil		5% hydrofluoric acid, water, thickener, tensioactive and coloring agents.
RelyX Ceramic Primer, 3M ESPE, St Paul, MN, USA	N662908	Ethyl alcohol, water, methacryloxypropyltrimethoxysilane
Scotchbond™ Universal, 3M ESPE, St Paul, MN, USA	577056	MDP phosphate monomer, dimethacrylate, HEMA, Vitrebond™ copolymer, alcohol, water, primers, and silane

Relyx™ Ultimate, 3M ESPE, St Paul, MN, USA	579623	Base paste - methacrylate monomers, radiopaque silane loads, primers, stabilizers, and rheological additives. Catalyst paste - methacrylate monomers, radiopaque alkaline fillers, primers, stabilizers, pigments, rheological additives, fluorescent coloring agents, dual Scotchbond™ Universal adhesive activator.

Acronyms: MDP - 10-Methacryloyloxydecyl dihydrogen phosphate; HEMA - Hydroxyethyl methacrylate

2.3. Thermal cycling

After storage, the Teflon tubes were carefully removed with just a pull, and the tape was removed with the help of a #15 scalpel blade. Thus, the resin cement cylinder bonded to the ceramic surface was exposed.

Ten ceramic slices of each group were subjected to thermal cycling; they were immersed in water, performing 10,000 cycles (TRIOS 37000, São Paulo, SP, Brazil), and alternating baths at 5 and 55°C with 30 s of dwell time at each temperature.

2.4. Shear bond strength test - µSBS

Microshear test was performed in a universal testing machine (EMIC DL2000, São José dos Pinhais, Brazil), applying load cell of 50 N with a specific device, where a ring-shaped orthodontic wire with approximate diameter of 0.20 mm was fixed and allocated around each resin cylinder for shear strength application, which occurred at speed of 0.5 mm/min until rupture.

2.5. Fracture mode analysis

The ceramic was analyzed with a digital microscope (Dino-Lite Premier, Anmo Electronics Corporation, New Taipei City, Taiwan) with 240x magnification and fracture mode was classified in adhesive (fracture between resin cement and ceramic), cohesive (complete fracture on ceramic or resin cement), and mixed (partially adhesive and partially cohesive fracture).

Representative samples of the fracture pattern from each group were selected for surface morphology analysis by SEM.

2.6. Scanning electron microscopy - SEM

The surface morphology of the ZLS ceramic after etching for 20 and 30 s was described after observation on the scanning electron microscope (JSM 5600 Lv JEOL, Pleasanton, CA, USA) with 2000x magnification, after gold spraying.

2.7. Data analysis

After testing data for normality with the Shapiro-Wilk test, they were analyzed by 3-way ANOVA followed by Tukey's test (5%).

3. Results

3.1. Microshear bond strength - μSBS

According to Table 2, the statistical analysis showed significant differences for the three factors isolated ($p<0.01$), the double interaction between the factors of etching time and thermal cycling ($p<0.05$), and the triple interaction of the factors ($p<0.05$).

Table 2 - Mean MPa and standard deviation of the interaction among thermal cycling, etching time, and cementation protocol.

Thermal cycling aging	Etching time	Cementation protocol		
		⊗ Si + Ci (21.28 ± 3.85)	⊗ Si + Ad + Ci (22.56 ± 2.44)	√ Ad + Ci (25.47 ± 3.61)
¯WITHOUT (26.24 ± 3.43)	∧ 20 s (21.32 ± 3.14)	25.59 ± 2.62 aA	26.61 ± 2.67 aA	26.17 ± 3.88 abA
	ʃ 30 s	23.53 ± 4.53 aB	26.72 ± 2.73 aAB	28.82 ± 4.14 aA
ϕWITH (19.97 ± 3.17)	∧ 20 s	13.82 ± 3.76 bB	13.53 ± 2.18 bB	22.20 ± 3.73 bA
	ʃ 30 s (24.89 ± 3.46)	22.20 ± 4.48 aA	23.38 ± 2.17 aA	24.70 ± 2.68 abA

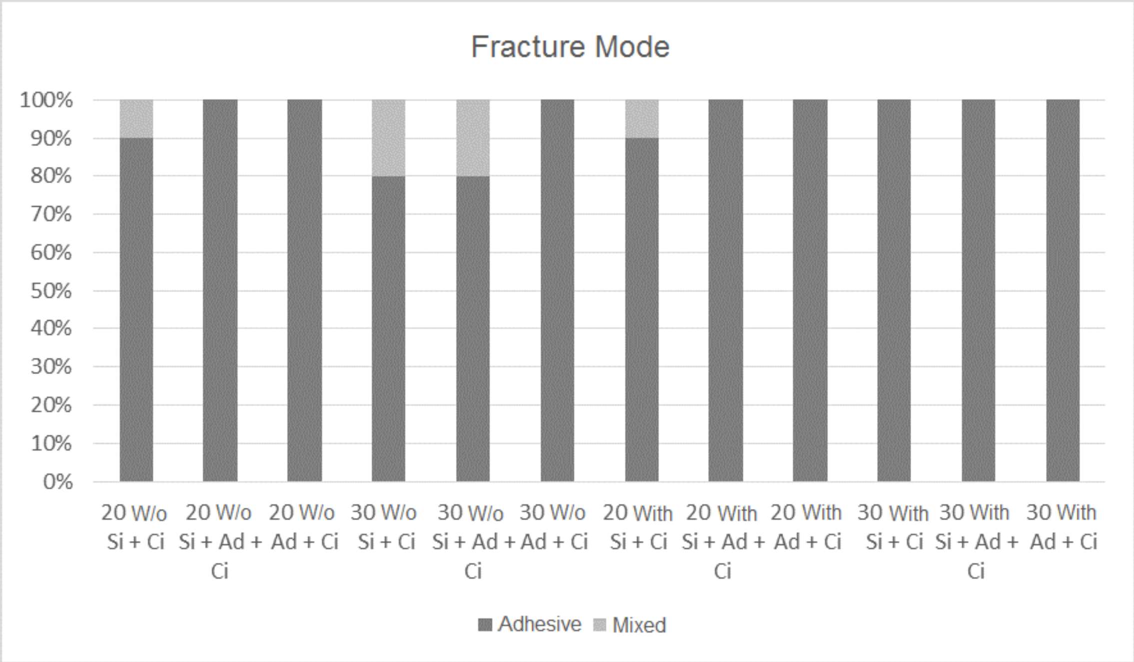
Analysis of factors isolated: different letters indicate statistically significant differences, considering lowercase letters for columns and capital letters for rows.
Triple interaction analysis: symbols
¯and ϕ – Thermal cycling factor
∧ and ʃ – Etching time factor
⊗ and √ – Cementation protocol factor

The analysis of factors isolated shows that for factor of cementation protocol, there was an equivalence between protocols Si + Ci (21.28 ± 3.85) and Si + Ad + Ci (22.56 ± 2.44), but they were statistically different from group Ad + Ci (25.47 ± 3.61), which presented higher bond strength values. For factor of etching time, 20 s (21.32 ± 3.14) resulted in lower bond strength values than 30 s (24.89 ± 3.46). For factor of thermal cycling, higher bond strength values were verified without thermal cycling (26.24 ± 3.43) when compared to groups subjected to thermal cycling (19.97 ± 3.17).

The analysis of triple interaction among factors (Table 2) allows observing the results of each experimental group separately and shows the exceptionalities of adhesive performance. These will be exposed next, but only the differences from those already described for each studied isolated factor: i) Cementation protocols - Ad + Ci was higher than the others after etching for 20 s and thermal cycling aging, but after etching for 20 s without thermal cycling and etching for 30 s with thermal cycling, the three protocols were equivalent; also after etching for 30 s without thermal cycling, Ad + Ci was only higher than protocol Si + Ci, while Si + Ad + Ci presented intermediate values with no difference from the other two protocols; ii) Etching time - without thermal cycling aging, etching for 20 and 30 s was equivalent for the three protocols studied, but after thermal cycling, etching for 30 s showed higher µSBS values than 20 s for protocols Si + Ci and Si + Ad + Ci, and protocol Ad + Ci was equivalent between the times studied; and iii)Thermal cycling aging - there was significant µSBS decrease in the three cementation protocols etched for 20 s, while etching for 30 s showed µSBS similarity for the three protocols, with or without aging.

3.2. Fracture mode

The fracture mode analysis showed that the type of prevalent fracture was adhesive. Groups with cementation protocol Si + Ci that were etched with HF for 20 s with and without thermal cycling presented 10% of mixed fracture. Groups with cementation protocol Si + Ci and Si + Ad + Ci that were etched for 20 s without thermal cycling presented 20% of mixed fracture, according to Graph 1.



Graph 1 - Mean rate of fracture mode observed in the groups studied.

3.3. Scanning electron microscopy - SEM

The SEM images with 2000x magnification showed that specimens etched for 20 s presented a predominant glassy phase, small pores, and isolation with irregular borders, as seen in Figure 2. Specimens etched for 30 s showed increased pore size appearing as elongated grooves, as seen in Figure 3.

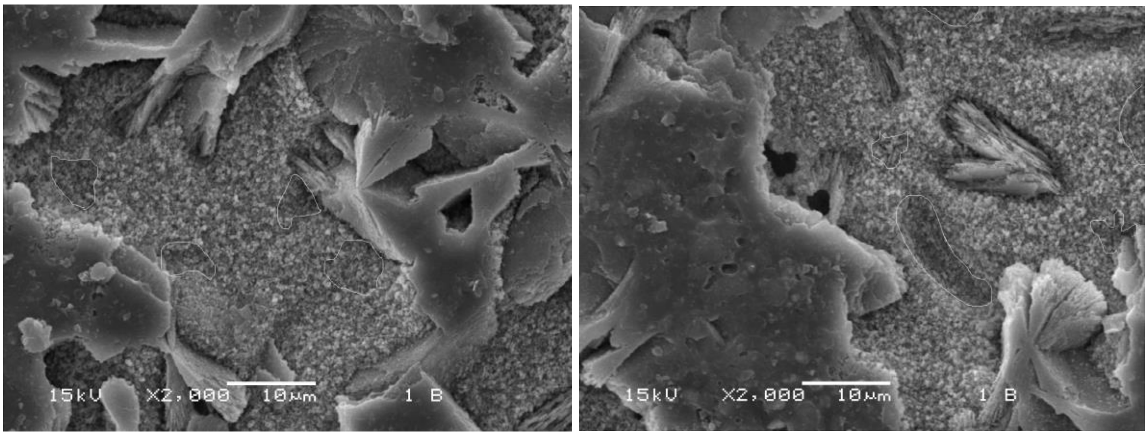


Figure 2 - Photomicrograph of the ZLS ceramic surface etched for 20 s. Original magnification: 2000x; bar = 10 µm

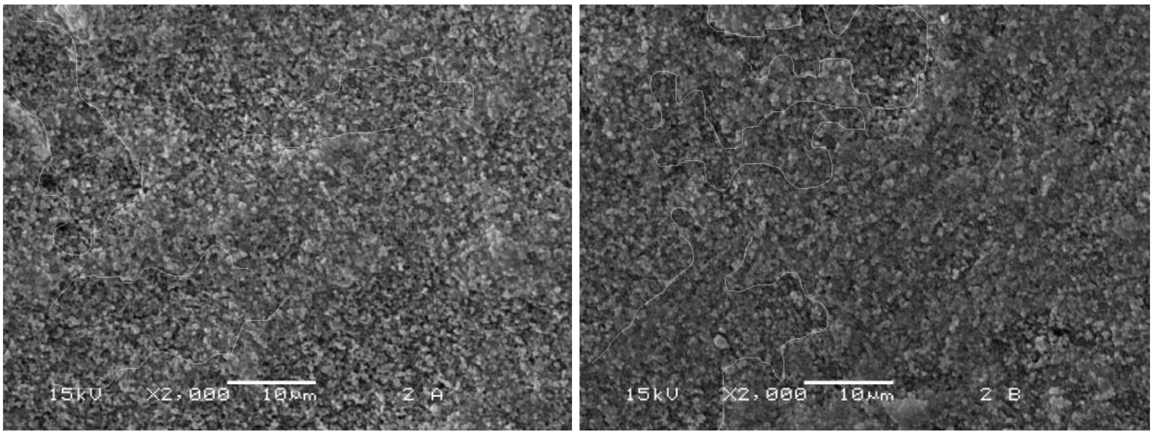


Figure 3 - Photomicrograph of the ZLS ceramic surface etched for 30 s. Original magnification: 2000x; bar = 10 μm

4. Discussion

The cementation of glass-ceramics requires hydrofluoric acid etching to dissolve the matrix and expose crystals, considering the glassy phase has high solubility to acid action and dissolves quicker than the crystalline phase, forming microroughness that allow promoting micromechanical retention to the resin cement selected [20, 32]. This study assessed the μSBS of ZLS ceramic etched for 20 or 30 s, cemented with three different cementation protocols, and thermocycled. The results of this study rejected the null hypothesis, in which etching time, cementation protocol, and thermal cycling influenced bond strength.

Usually, the ceramic manufacturer already indicates the time and acid concentration to be applied, but the recommended time is not always the best one [33]. In this study, the indication for etching time for the ZLS ceramic was 20 s by the manufacturer of ZLS Suprinity [34], and 30 s by the manufacturer of a similar ZLS ceramic [35]. It was observed that etching for 30 s resulted in higher bond strength, especially after thermal cycling. The accelerated aging process resulted in an average decrease in bond strength of approximately 37 and 11%, respectively for groups etched for 20 and 30 s. This is probably because the longer the etching time, the higher the surface roughness, the surface free energy [36], the mechanical interlocking, and the wettability [13, 20]. The SEM images obtained showed that ceramics etched for 20 s presented a predominant glassy phase and small isolated pores (Fig. 2), while etching for 30 s resulted in larger pores appearing as elongated grooves (Fig. 3). There are evidences of high dissolution of the glassy phase after 90 s [32, 33], presenting irregular crystals, slit-like gaps, and removal of crystals because of the extensive loss of the glassy phase [13], which may result in decreased bond strength [6].

The analysis of data from Table 2 shows decreased bond strength after thermal cycling in groups etched for 20 s, which is probably because of the deleterious effect of water storage and thermal cycling for adhesive bonding [26, 30, 37] among resin cement [19], silane, and ceramic. The cementation protocol that did not include silane (Ad + Ci) presented higher bond strength value within groups etched for 20 s and thermocycled, possibly because silane incorporated to adhesive suffers less degradation than silane applied separately, considering that silanized interfaces deteriorate in humid conditions and resins are water-permeable [38]. Thus, it is expected that the bond between silane and resin cement suffers hydrolysis over time, leading to stress and formation of microcracks [7].

The SBU adhesive is composed by two bifunctional agents: silane and MDP [7, 17]. The hydroxyl group of the MDP monomer can chemically react with the hydroxyl group of the zirconia oxides [17] scattered in the glass matrix, promoting a stable chemical bond resistant to hydrolytic degradation [39], which may be weakened after thermal cycling [17] and still be effective in promoting zirconia bonding [27]. In turn, the silane-coupling agent interacts with the silica matrix [7]. Both agents can promote the chemical bond between ceramic and resin cement. Thus, the lower the mechanical interlocking by decreased etching time, the higher the dependence of chemical bond between ceramic and resin cement. However, this chemical bond among silane, MDP, and ceramic is susceptible to degradation after thermal cycling [7, 17]. In the groups etched for 30 s that were not subjected to thermal cycling, the cementation protocol Ad + Ci presented higher bond strength values than protocol Si + Ci. Besides the incorporated silane, this adhesive presents MDP in its composition, which may be a factor that favors bond strength for ZLS ceramics because of the presence of zirconia scattered in the glass matrix [10, 11].

Opposite to that observed in the present study, researchers [31] assessing bond strength of the RXU cement to a leucite-reinforced ceramic, as well as other researchers [23, 28] assessing lithium disilicate ceramic, observed that SBU adhesive presented lower bond strength values when compared to the application of silane separate from the adhesive, which also contained MDP. The ZLS ceramic has zirconia metal oxides and cerium added to its composition when compared to lithium disilicate ceramic [14]. Perhaps MDP promotes the chemical bond to other metal oxides present in the composition, which indicates the need for studies assessing the effectiveness of MDP present in adhesive and whether it allows the chemical bond to other metal oxides of the ZLS ceramic. For groups etched for 30 s without thermal cycling, the cementation protocol Si + Ad + Ci was not different from the other two, which leads us to infer that isolated silane hydrolysis may be compensated with the presence of silane and MDP incorporated to SBU adhesive.

A study [40] assessed the behavior of conventional resin cements to self-adhesive cements on thermal cycling aging conditions, in 3 cycle quantities - 0; 10,000; and 30,000, and observed a decrease in bond strength between 0 and 10,000 cycles, but there was no significant difference between 10,000 and 30,000 cycles. In this study, specimens were subjected to thermal cycling aging in 10,000 cycles, showing a significant decrease of μSBS in the three cementation protocols etched for 20 s. Each thermal cycle consists in bathing the specimen for 30 s in water at approximately 5°C, 30 s in water at approximately 37°C, and lastly 30 s in water at approximately 50°C. This temperature variation promotes expansion and contraction of the elements involved, which usually present different thermal expansion coefficients (TEC). According to the manufacturer, the ZLS ceramic has a TEC of approximately 12.3 10^{-6}, while enamel and dentin have 16.96 19^{-6} and 10.59 10^{-6}, respectively [41]. Regarding the SBU adhesive and RXU cement, the values of these properties were not reported in the literature, but the TEC of composite resins were normally described as being higher than enamel and dentin [42], as for instance 22.5 10^{-6} and 32.6 10^{-6} [43]. Thus, we may infer that the difference in behavior of contraction and expansion among ceramics, adhesive, and resin cement may trigger fractures in a microscopic level, which leads to intermolecular fatigue and weakens chemical bond. However, after thermal cycling, the groups etched for 30 s with the 3 different cementation protocols maintained similar bond strength values, suggesting that thermal cycling degradation may also depend on surface etching [3, 30].

The *in vitro* findings do not directly correspond to *in vivo* conditions, but are valuable indicators of the clinical capacity of the material. Despite the limitations of our study, we may recommend 5% HF etching for 30 s for ZLS ceramic and the use of cementation protocol Ad + Ci, which presented the best characteristics regarding thermal aging, leading us to believe that this is the option with greater clinical longevity.

5. Conclusion

Considering the limits of this study, it may be concluded that the bond strength of Relyx™ Ultimate resin cement to Suprinity ceramic is higher after 30 s of 5% HF etching, using cementation protocol Adhesive + Cement and before thermal cycling aging.

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