



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

Influência da cimentação e da termociclagem na resistência de união da cerâmica de silicato de lítio reforçada com zircônia

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ABSTRACT

Objective: To assess the influence of 5% hydrofluoric acid etching time (ET), cementation protocol (CP), and thermal cycling (TC) aging on the microshear bond strength (μ SBS) of zirconia-reinforced lithium silicate ceramic (ZLS) to adhesive resin cement. **Material and Methods:** Ten VITA Suprinity® ceramic blocks were cut in 120 slices (1.4 mm thickness) and randomly assigned to 12 groups (n = 10) according to the combination of factors (2x3x2 design): etching time (20 or 30 s), cementation protocol (silane + universal adhesive + resin cement; universal adhesive + resin cement; silane + resin cement) and thermal cycling (cycled or no-cycled). RelyX Ceramic Primer and Scotchbond™ Universal Adhesive were used respectively as silane (S) and universal adhesive (Ua). Ceramic surface was etched, and the cementation protocol performed on the delimited bonding area. Then, resin cement (RelyX™ Ultimate Cement [Rc]) cylinders were bonded and light cured. After, specimens were stored in deionized water at 37°C for 7 days and subjected to the μ SBS test. **Results:** Data passed the normality test and three-way ANOVA analysis showed statistical difference (p < 0.01) for isolated; double (ET/TC) (p < 0.05), and triple (p < 0.05) factor interactions. **Conclusion:** The combination 30s etching-Ua-Rc presented higher adhesive bond strength after thermal aging.

KEYWORDS

Adhesive; Ceramics; Hydrofluoric acid; Resin cements.

RESUMO

Objetivo: Avaliar a influência do tempo de condicionamento com ácido fluorídrico 5% (ET), protocolo de cimentação (CP), e envelhecimento por termociclagem (TC) na resistência de união ao microcisalhamento (μ SBS) da cerâmica de silicato de lítio reforçada com zircônia (ZLS) ao cimento resinoso adesivo. **Material e Método:** Dez blocos da cerâmica VITA Suprinity® foram cortados e 120 fatias (com espessura de 1,4 mm) e distribuídos aleatoriamente em 12 grupos (n=10) de acordo com o fator de combinação (2x3x2): tempo de condicionamento (20 ou 30 s), protocolo de cimentação (silano + adesivo universal + cimento resinoso; adesivo universal + cimento resinoso; silano + cimento resinoso) e ciclagem térmica (ciclado ou não-ciclado). RelyX Ceramic Primer e adesivo Scotchbond™ Universal foram utilizados respectivamente como silano (S) e adesivo universal (Ua). A superfície cerâmica foi condicionada e o protocolo de cimentação aplicado sobre área de cimentação delimitada. Portanto, cilindros de cimento resinoso (cimento RelyX™ Ultimate [Rc]) foram cimentados e fotopolimerizados. Após, os espécimes foram armazenados em água deionizada a 37°C por 7 dias e submetidos ao teste de μ SBS. **Resultados:** Após teste de normalidade, os dados foram analisados pelo teste ANOVA três critérios mostrando diferença estatística (p < 0.01) para a análise do fator isolado; para a interação entre dois fatores (ET/TC) (p < 0.05), e interação entre os três fatores (p < 0.05). **Conclusão:** A combinação 30s de condicionamento-Ua-Rc apresentou a maior resistência de união adesiva após a ciclagem térmica.

PALAVRAS-CHAVE

Adesivo; Cerâmica; Ácido fluorídrico; Cimento resinoso.

INTRODUCTION

Dental ceramics are widely used as aesthetic restorative material given the ability to simulate the natural teeth appearance [1] and their clinical success depends on the proper mechanical-chemical bond adhesion among resin cement (Rc), ceramic (C) and tooth substrate [2]. Moreover, the hydrofluoric acid (HF) etching is a necessary step to improve the Rc/glass-ceramic bond [1], allowed only for silica-based (SiO₂) or glass-ceramics [3]. The HF selectively dissolves the glass matrix, exposes the crystalline content and makes the etched surface rough. As result, micromechanical retention is obtained [4].

After etching, the silane (S) application step [5] increases the chemical bond. By improving infiltration and cementing agent-C surface porosities contact [6], the polymer interconnections are favored [7]. It happens because S-bifunctional molecules allow the bonding between organic (Rc monomers) and inorganic (glass matrix ceramic silica content) compounds [8].

Along with the process of incorporating crystals to improve the mechanical properties of ceramics, a new generation of glass-ceramics was developed: Suprinity zirconia-reinforced lithium silicate (ZLS) ceramic. Furthermore, zirconia does not allow mechanical/chemical bond to Rc using HF and silane. To this end, a bifunctional organic molecule MDP (10-Methacryloyloxydecyl dihydrogen phosphate) monomer [9-11] is required: ester phosphate monomer bonds to the oxides (such as zirconia oxide) [12] and the other groups copolymerize with the resin matrix of cements. However, the chemical reactions formed between hydroxyl groups of the MDP monomer and those groups on the zirconia ceramic surface can be weakened after thermal cycling [10]. Besides, the different HF etching time protocols indicated by ZLS ceramic manufacturers (Vita Zahnfabrik; CELTRA Duo ceramic, Dentisply) brings up some uncertainties since it may directly influence the C-Rc bond strength [4,13,14].

Recently, a new universal adhesive-resin cement (Ua+Rc) combination (resin cement RelyX™ Ultimate 3M ESPE; Scotchbond™ Universal 3M ESPE) recommended for glass-ceramic cementation presented good results for bond strength to zirconia after aging [11]. All the primers for indirect restorations, such as MDP, silane, and adhesive, compose that combination. Furthermore, the manufacturer assertion regarding possible protocols [(S+Rc); (S+Ua+Rc); (Ua+Rc)] to be applied is still under question.

The lack of evidence about the relationship among of 20 or 30 s HF etching, distincts cementation protocols as well thermal cycling on the microshear bond strength (μ SBS) of Suprinity ZLS ceramic prompted this study. Therefore, to elucidate such effects, we hypothesized that (1) different etching times, (2) cementation protocol, and (3) thermal cycling affect the μ SBS.

MATERIAL AND METHODS

Ten rectangular ZLS ceramic (Suprinity; Vita Zahnfabrik, Bad Säckingen, Germany, shade HT 0M1) blocks (18 x 14 x 12 mm) were vertically cut into 120 slices (12 x 7 x 1.4 mm) using a low-speed cutting saw (Labcut 1010 extec, Enfield, USA) with diamond disc 3" x 0.006" x 0.5" (Lapmaster Wolters, Mt Prospect, IL, USA) under water irrigation. They were wet-polished with a 600-grit silicon carbide paper for 1 min with a rotational polishing device (Arotec PL4, São Paulo, Brazil) and crystallized according to manufacturer's instructions in a Vita Vacumat oven (Vita Zahnfabrik, Bad Säckingen, Germany). Then, the specimens were randomly assigned to twelve groups (n=10) according to the combination of factors (2x3x2 design): etching time (20 or 30 seconds), cementation protocol (S+Ua+Rc; Ua+Rc; S+Rc), and thermal cycling (cycled, no-cycled) (Table 1). The materials used are shown on Table 2. A sample size of ten specimens was calculated considering minimum detectable difference in mean μ SBS of 5.0, standard deviation of 3.1, α -error level of

5% and 95% statistical power. The specimens were etched with 5% HF (Condac porcelana, FGM, Joinville, SC, Brazil), rinsed with water (60 s) and dried with oil free compressed air (30 s). The delimitation of the ceramic surface bonding area was performed using an adhesive tape (Imperial, 3M, Sumare, Brazil) with four holes (1 mm-diameter) positioned over it. Then, they received the following post etching surface treatments, according to the groups:

- Silane: two layer of a silane coupling agent (RelyX Ceramic Primer, 3M ESPE, St Paul, MN, USA) which were left in contact for 60 seconds and air-dried for 60 seconds.
- Universal adhesive: a thin layer of photo-activated universal adhesive (SBU-Scotchbond™ Universal 3M ESPE, 3M ESPE, St Paul, MN, USA) applied for 5 seconds with microbrush and the excess air-removed for 10 s.

The resin cement (RXU- RelyX Ultimate, 3M ESPE, St. Paul, MN, USA) was carefully inserted by spatule into translucent Teflon tubes (Solidor Haryana, India) matrices to obtain cylinders (2.0 mm height x 0.76 mm internal diameter). Then, each tube was positioned over the tape, ensuring the lumen coincided with the circular ceramic area exposed by the perfurtation [2] and light-cured (through the ceramic) for 40 s using a light-emitting diode (LED) device (Valo Cordless, standard mode; Ultradent Inc, South Jordan, UT, USA) with an output of 1,000 mW/cm².

The ceramic specimens were fixed with epoxy resin (Durepoxi, Loctite, Itapevi, SP, Brazil) to acrylic resin filled PVC rings and stored in deionized water for 7 days at 37°C. After 24 h, the teflon tubes and tape were carefully removed to expose the four resin cement cylinders and specimens subjected to 10,000 thermal cycles (ERIOS 3700, São Paulo, SP, Brazil) with water (5 °C to 55 °C) for 30 s dwell and 10 s transfer times.

For the microshear bond strength (μ SBS) test, using a universal mechanical testing machine (EMIC DL2000, São José dos Pinhais, PR, Brazil), a thin steel round wire (0.2 mm

diameter) was looped around each cylinder, aligned with the bonding interface. The shear load was applied at a crosshead speed of 1 mm/min until failure. The mean values of the four cylinders was recorded as the bond strength for each specimen, expressed in MPa. The mean bond strength values of each group represented the mean of the 10 specimens. After confirmation of the normal (Shapiro-Wilk) and homogeneous (Cochran) distributions, the data from μ SBS were submitted to three-way ANOVA, followed by the Tukey post hoc test ($\alpha=0.05$) for multiple comparisons.

The failure mode of debonded specimens was determined at 100% magnification using an optical microscopy (Dino-Lite Premier, Anmo Electronics Corporation, New Taipei City, Taiwan), and recorded as adhesive (interfacial failure), cohesive (within ceramic or within resin cement), or mixed (involving resin cement and ceramic).

Two representative specimens (A and B) of each etched group, after gold spraying via sputtering, were submitted to scanning electron microscopy (SEM) (JSM 5600 LV; JEOL, Pleasanton, CA, USA) analysis (500 x and 2,000x magnification and operated at 15 kV) to observe the ceramic surface morphology.

Table 1 - Group description

Group	Etching time	Cementation protocol	Thermal cycling	
G1	20SRc/C	silane + resin cement	cycled	
G2	20SRc/no-C	silane + resin cement	no-cycled	
G3	20SUaRc/C	20 seconds	silane + universal adhesive + resin cement	cycled
G4	20SUaRc/no-C	silane + universal adhesive + resin cement	no-cycled	
G5	20URc/C	universal adhesive + resin cement	cycled	
G6	20URc/no-C	universal adhesive + resin cement	no-cycled	
G7	30SRc/C	silane + resin cement	cycled	
G8	30SRc/no-C	silane + resin cement	no-cycled	
G9	30SUaRc/C	30 seconds	silane + universal adhesive + resin cement	cycled
G10	30SUaRc/no-C	silane + universal adhesive + resin cement	no-cycled	
G11	30UaRc/C	universal adhesive + resin cement	cycled	
G12	30UaRc/no-C	universal adhesive + resin cement	no-cycled	

(S) silane; (Ua) universal adhesive; (Rc) resin cement; (C) cycled; (no-C) no cycled.

Table 2 - 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.

RESULTS

The μ SBS results are shown in Tables 3 and 4. The statistical analysis showed significant differences for the isolated factor ($p < 0.01$). When etching times were compared, 30 seconds was significantly higher than the other one ($p < 0.01$). For the post etching treatment, no statistical difference was found among silanized, silanized/universal adhesive, and universal adhesive ($p > 0.05$). The no-cycled groups showed significantly higher μ SBS mean values than those cycled ($p < 0.01$). Statistical difference was found ($p < 0.05$) for the etching time/thermal cycling interaction. The 20 sec/cycled group showed the lowest μ SBS mean value. For the triple interaction, significant differences were found ($p < 0.05$). The 20 sec/cycled/silanized group showed the lowest μ SBS mean value. The greater μ SBS mean value was observed for the 30 sec/no-cycled/universal adhesive.

Figure 1 shows the descriptive analysis of the failure modes. An adhesive failure predominance was detected for all groups, regardless the etching times or thermal cycling. Conversely, mixed failures (cohesive within resin cement) were found in groups (20SRc/C, 20SRc/no-C, 30SRc/no-C, 30SUaRc/no-C) but with lower incidence.

Figure 2, 3 and 4, SEM photomicrographs shows the ceramic surface morphology etched for two different etching times (20 and 30 s).

Table 3 - Means of μ SBS (MPa) (\pm SD) for the isolated factors and double interaction

Thermal cycling	Etching time	
	20 seconds	30 seconds
	21.3 \pm 3.2b	24.9 \pm 3.5a
cycled (26.24 \pm 3.43)a	26.1 \pm 3.1aA	26.4 \pm 3.8 aA
no-cycled (19.97 \pm 3.17)b	16.5 \pm 3.2bB	23.4 \pm 3.1bA

Superscript different letters indicate statistically significant differences for the isolated factor ($p < 0.01$). Different letters indicate statistically significant differences for the double interaction between the factors of etching time and thermal cycling ($p < 0.05$), considering lowercase letters for columns and capital letters for rows.

Table 4 - Means of μ SBS (MPa) (\pm SD) for all groups

Etching time /Thermal cycling	Cementation Protocol		
	Silane	Silane + universal adhesive	Universal adhesive
20 / no-cycled	25.6 \pm 2.6 aA	26.6 \pm 2.7 aA	26.2 \pm 3.9 abA
30 / no-cycled	23.5 \pm 4.5 aB	26.7 \pm 2.7 aAB	28.8 \pm 4.1 aA
20 / cycled	13.8 \pm 3.8 bB	13.5 \pm 2.2 bB	22.2 \pm 3.7 bA
30 / cycled	22.2 \pm 4.5 aA	23.4 \pm 2.2 aA	24.7 \pm 2.7 abA

Means followed by different lowercase letters in each column and upper capital letters in each rows differ statistically by Tukey's test at 5%.



Figure 1 - Failure Mode results of debonded resin cement specimens.

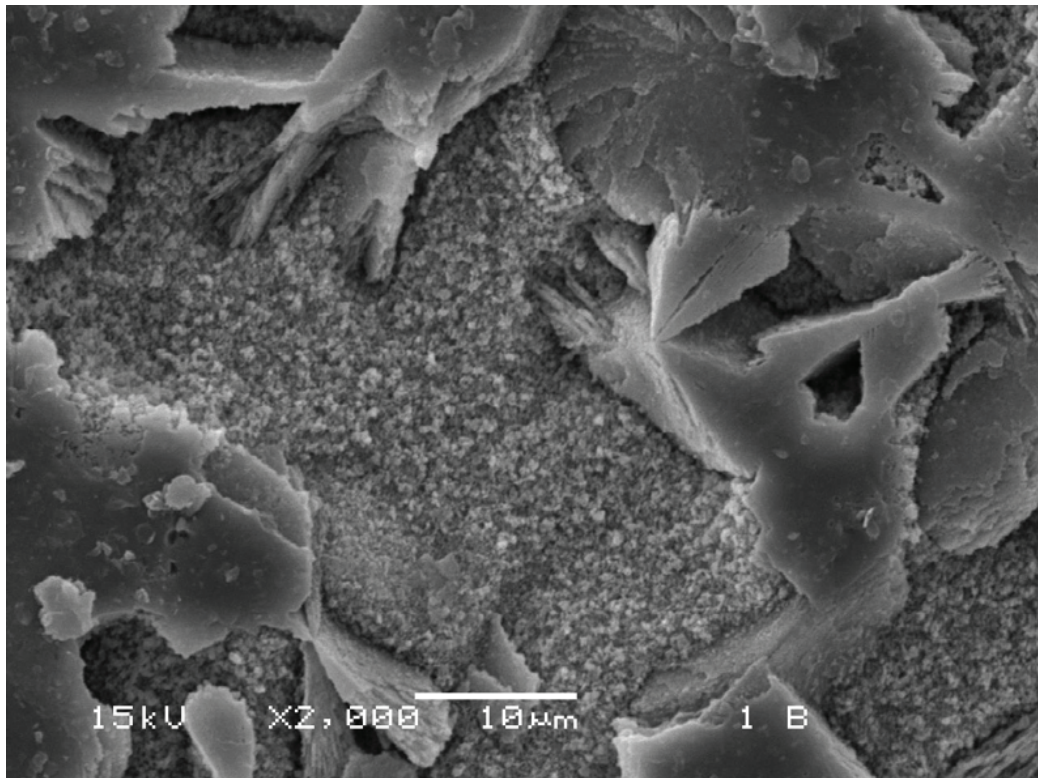


Figure 2 - SEM photomicrographs showing microstructure of ceramics etched for 5% HF during 20 s. Original magnification 2000x.

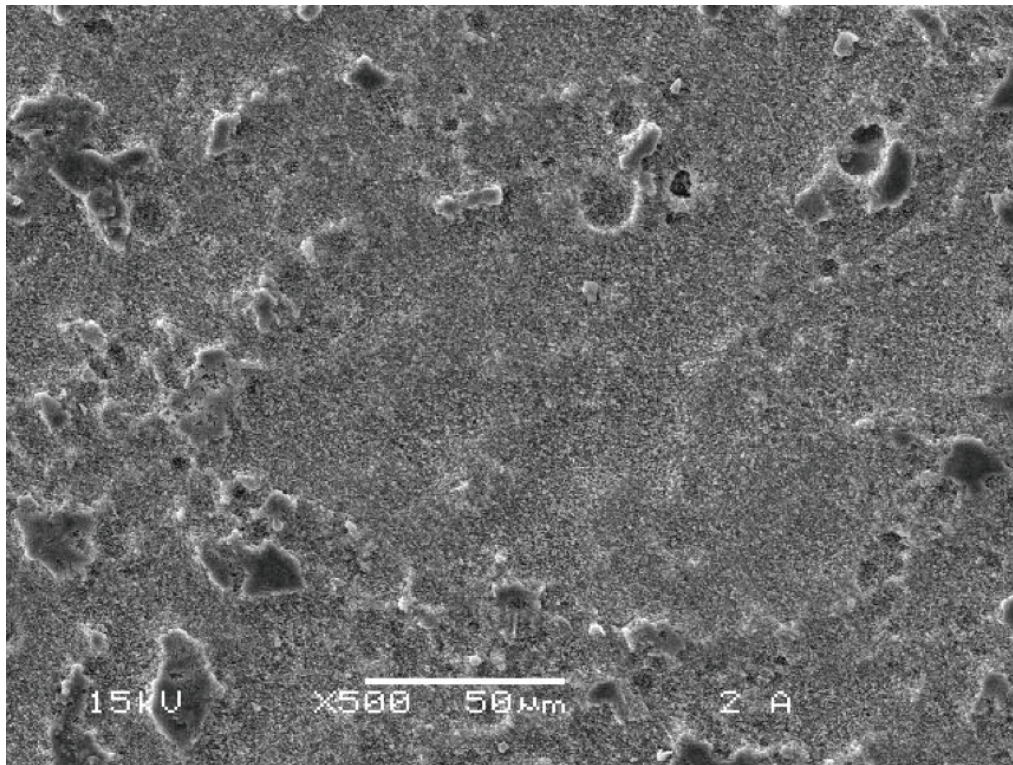


Figure 3 - SEM photomicrographs showing microstructure of ceramics etched for 5% HF during 30 s. Original magnification 500x.

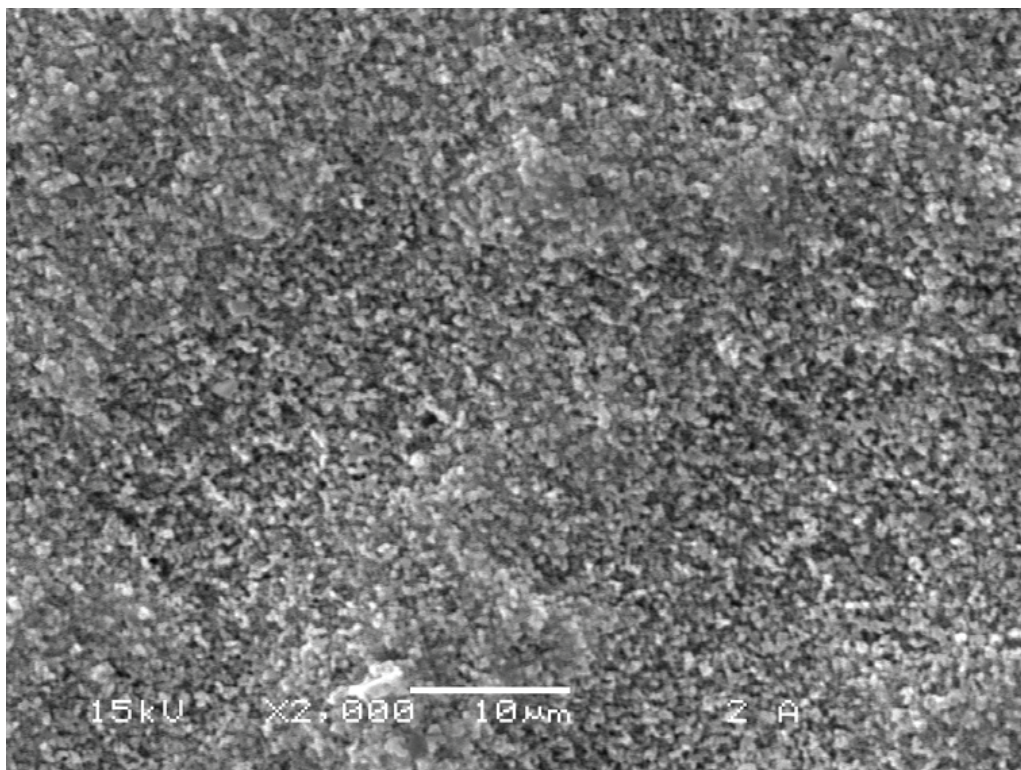


Figure 4 - SEM photomicrographs showing microstructure of ceramics etched for 5% HF during 30 s. Original magnification 2000x.

DISCUSSION

This study assessed the μ SBS of ZLS ceramic etched for 20 or 30 s, cemented with three different cementation protocols, and thermocycled or no. The null hypothesis was rejected since the three factors affected bond strength.

Given the high glassy phase solubility and quicker dissolution (compared to crystalline phase), HF etching dissolves the matrix and exposures crystals. As result, the created microroughness promoted micromechanical retention to the Rc [14]. Despite the time and acid concentration usually recommended by ceramic manufacturer, 30 s 5% HF etching resulted in a higher bond strength after thermocycled (Table 4). The aging process promoted an average decrease in bond strength of \approx 37% and 11% for groups etched for 20 and 30 seconds, respectively. This was probably due to the longer etching time, that increased the surface roughness, as well surface free energy [15] and mechanical interlocking and wettability [13,14].

The lower the mechanical interlocking by decreased etching time, the higher the dependence of chemical bond between ceramic and resin cement. Thus, as observed in 20 s etched groups, the lower bond strength was probably due to the deleterious effect of storage and thermal cycling of adhesive bonding [16,17] among resin cement, silane, and ceramic (Table 4). In turn, the silane-coupling agent interacts with silica matrix [8] promoting chemical bond between ceramic and resin cement. It was expected that silane-resin cement bond would suffer hydrolysis over time, leading to stress and formation of microcracks [8].

As cited above, RXU resin cement and SBU adhesive may be applied in different cementation protocols. SBU is composed by two bifunctional agents: MDP and silane.

The hydroxyl group (-OH) of MDP monomer can chemically react with the hydroxyl group of zirconia oxides [10]. That promotes a stable chemical bond resistant to hydrolytic degradation [18], which, however, may be weakened after thermal cycling [10] but still effective to promote zirconia bonding [19,20]. In the 30 s no-cycled groups (30SRc/no-C, 30SUaRc/no-C and 30UaRc/no-C), the use of universal adhesive promoted higher bond strength values than silane. It is known that both agents promote chemical bond between ceramic and resin cement. However, monomers with -OH groups (like MDP) might have hydrolyzed silane, producing silanols and low pH (water-containing SBU, pH = 2.7) facilitates the silanol condensation [21]. Thus, the higher bond strength value for 20UaRc/C group may be explained by "silane absence".

Contrary to our results, studies assessing RXU bond strength to a leucite-reinforced ceramic [22] or lithium disilicate ceramic (LDS) [23,24] showed lower bond strength values for SBU compared to the silane separate adhesive application. But differently from LDS, ZLS has zirconia and other metal oxides (like cerium) added to its composition [25]. Thus, MDP would also promote chemical bond to other metal oxides [26] present in the composition and this assumption prompts the investigation of MDP effectiveness. For 30 s etched no-cycled groups, the post etching SUa treatment was not different from the other two, which lead us to infer that isolated silane hydrolysis may be compensated by the presence of MDP incorporated to SBU adhesive.

The specimens subjected to 10,000 thermal cycles aging showed a significant decrease of μ SBS for the three post etching treatments etched for 20 sec. This finding is in accordance to Liu et al. (2011), who demonstrated a decrease in bond strength between 0 and 10,000 cycles, although no significant difference has been observed

between 10,000 and 30,000 cycles for conventional resin cements to self-adhesive cements. Thus, the 10,000 cycles used in our study were enough to evaluate the resin cement [27].

In the present study, adhesive failure was predominant for all groups. Mixed failures were found (20SRc/no-C, 30SRc/no-C, 30SUaRc/no-C), but with lower incidence. The differences in the failure-mode distribution can be explained by the reduced bond strengths after thermocycling.

Despite the limitation of this *in vitro* study, findings can be considered as indicators of material clinical capacity. However, some questions remain unanswered and should be assessed. Would MDP monomer presents in SBU adhesive composition able to bond on zirconia scattered in the glass matrix? Would MDP monomer able to bond on other metal oxides in the ceramic composition?

CONCLUSION

Based on our results we concluded that the bond strength of resin cement to ZLS ceramic was higher when 30 s of 5% HF etching and treatment with universal adhesive, without thermal cycling. Thus, 5% HF etching for 30 s and the use of universal adhesive for ZLS ceramic cementation should be recommend.

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