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Avaliação da resistência de união entre cimento e uma vitro cerâmica condicionada com diferentes tempos de ácido fluorídrico 5% e 10% - estudo *in vitro*

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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.

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Orientador: Prof[a]. Dr[a]. Fabiana Scarparo Naufel
Co-Orientador: Prof. Dr. Wagner Baseggio

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PARANÁ
GOVERNO DO ESTADO

CAROLINA BAUER GODOY VERONESE

Efeito do tempo de condicionamento com HF a 5% e 10% na resistência de união a uma cerâmica ZLS – estudo in vitro

Dissertação apresentada ao Programa de Pós-Graduação em Odontologia em cumprimento parcial aos requisitos para obtenção do título de Mestra em Odontologia, área de concentração Odontologia, linha de pesquisa Materiais Dentários Aplicados À Clínica Odontológica, APROVADO(A) pela seguinte banca examinadora:

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Cascavel, 23 de fevereiro de 2017

DEDICATÓRIA

Dedico esta dissertação de mestrado aos que mais amo nesta vida, minha mãezinha Zilnê, meu esposo Rodrigo, meus filhos Leonardo, Murilo, Isabelle e Emanuel e meu padrasto Tavares, obrigada por sempre estarem ao meu lado nas minhas escolhas, que muitas vezes também é pensada em colher bons frutos para vocês!

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Avaliação da resistência de união entre cimento e uma vitro cerâmica condicionada com diferentes tempos de ácido fluorídrico 5% e 10% - estudo *in vitro*

RESUMO

Objetivo: Avaliar o efeito de diferentes tempos de condicionamento com ácido fluorídrico (HF) nas concentrações de 5% e 10 % na resistência de união do cimento resinoso RelyX Ultimate a uma cerâmica de silicato de lítio reforçada com zircônia de nome comercial Vita Suprinity (VS), onde a indicação do fabricante é a utilização de HF a 5% durante um tempo de 20”. **Metodologia:** Estudo realizado com delineamento experimental inteiramente casualizado, com 8 tratamentos (n=8). Cada espécime de cerâmica foi condicionado com ácido fluorídrico (HF) em diferentes tempos e concentrações: 5% por 20, 30, 40 e 60 seg, e 10% por 10, 20, 30 e 40 seg; submetidas à análise da rugosidade (Ra), seguido pelo protocolo de cimentação empregando ácido fluorídrico, silano RelyX Ceramic Primer, adesivo Single Bond Universal e cimento resinoso RelyX Ultimate. A resistência de união foi avaliada pelo teste de microcisalhamento; seguido pela análise do padrão das fraturas. **Resultados:** O tratamento 5%/20” exibiu estatisticamente a menor resistência de união 21,96 MPa ($p<0.01$), diferindo do tratamento com HF a 10% nos quatro tempos estudados, 10” = 29,59 MPa, 20” = 29,77 Mpa, 30”= 29,68 Mpa e 40”= 30,78 Mpa ($p<0.01$); o tratamento 10%/40” exibiu a maior resistência de união (30,78 MPa), diferindo apenas do 5%/20” (21,96 MPa). O padrão de fraturas foi predominantemente adesivo. **Conclusão:** O condicionamento com ácido fluorídrico, em ambas concentrações e em seus diferentes tempos de exposição, aumenta a rugosidade da superfície da cerâmica Vita Suprinity, havendo diferenças significativas na resistência da união adesiva apenas quando a concentração e o tempo de condicionamento variam grandemente, e a maioria da ruptura foi adesiva, ocorrendo na interface do adesivo; com ácido fluorídrico na concentração de 5% durante 30” ou 10% por 10” ou 20” observou-se maior resistência de união do que o protocolo do fabricante, contudo sem observar porosidades amplas e rasas em MEV, sugerindo aumentarmos o tempo de exposição sugerido pelo fabricante de 20” para 30” ou utilizar ácido com concentração maior, como por exemplo 10% em 10” ou 20”, para resultados mais efetivos.

Palavras-chave: cerâmica, ácido fluorídrico, resistência ao cisalhamento

Assessment of bond strength between a cement and a glass-ceramic etched at different times with 5% and 10% hydrofluoric acid - *in vitro* study

ABSTRACT

Objective: to assess the effect of different hydrofluoric acid (HF) etching times at concentrations of 5% and 10% on the bond strength of RelyX Ultimate resin cement to a zirconia-reinforced lithium silicate ceramic of commercial brand Vita Suprinity (VS), for which the manufacturer recommends using 5% HF for 20". **Method:** experimental and completely randomized study with 8 treatments (n=8). Each ceramic sample was etched with hydrofluoric acid (HF) at different times and concentrations: 5% for 20, 30, 40, and 60 s, and 10% for 10, 20, 30, and 40 s; subjected to roughness (Ra) analysis, followed by the cementation protocol applying hydrofluoric acid, RelyX Ceramic Primer silane, Single Bond Universal adhesive, and RelyX Ultimate resin cement. Bond strength was assessed by the microshear test followed by fracture pattern analysis. **Results:** the 5%/20" treatment showed the statistically lowest bond strength of 21.96 MPa ($p<0.01$), differing from 10% HF treatment at the four times studied: 10" = 29.59 MPa, 20" = 29.77 MPa, 30" = 29.68 MPa, and 40" = 30.78 MPa ($p<0.01$). The 10%/40" treatment showed the highest bond strength (30.78 MPa), differing only from 5%/20" (21.96 MPa). The fracture pattern was predominantly adhesive. **Conclusion:** hydrofluoric acid etching increases surface roughness of the Vita Suprinity ceramic at both concentrations and their different times of exposure, showing significant differences in adhesive bond strength only when concentration and etching time vary widely, and with mostly adhesive rupture occurring in the adhesive interface. The concentration of 5% hydrofluoric acid for 30" or 10% for 10" or 20" showed higher bond strength than the manufacturer's protocol, but there were no wide and shallow porosities in SEM. This suggests the increase in exposure time recommended by the manufacturer from 20" to 30" or the use of higher concentrations of acid, such as 10% at 10" or 20", for more effective results.

Keywords: ceramic, hydrofluoric acid, shear bond strength.

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SUMÁRIO

Artigo: “Avaliação da resistência de união entre cimento e uma vitro cerâmica condicionada com diferentes tempos de ácido fluorídrico 5% e 10% - estudo *in vitro”*

Introduction

Ceramic restorations have been extensively used in aesthetic and functional rehabilitations because of characteristics such as biocompatibility, color stability, low thermal conduction, low plaque accumulation, and abrasion resistance, as well as for promoting excellent aesthetics [1, 2]. Recently, the Vita Suprinity zirconia-reinforced lithium silicate ceramic (ZLS) [3] was introduced in the market. It combines the mechanical properties of zirconia (ZrO_2) with glass-ceramic aesthetics and it is an acid-sensitive ceramic [3, 4, 5], because hydrofluoric acid (HF) can remove the glass matrix and the crystalline phase of the lithium orthophosphates, exposing lithium silicate crystals, which makes the surface irregular. It is also essential for good adhesion [6, 7].

The creation of a surface with optimum topography for a durable adhesion depends on ceramic composition, hydrofluoric acid concentration, and etching time [8]. Obtaining a properly porous surface is essential for the durable cementation of the indirect restoration and it may be obtained by short cycles of chemical attack on the surface; the etching time is mostly regulated by ceramic composition [7, 8, 9]. The changed topography results in increased surface area, favoring micromechanical retention with resin composites [8, 9]. Moreover, roughness also affects the wetting ability of the ceramic surface, and thus the subsequent application of bonding agents [7, 8, 9, 10, 11]. A positive correlation between roughness and bond strength has already been apparently observed up to a certain point [8, 11]. The hydrofluoric acid etching also cleans the surface by removing debris and impurities [7, 8, 9], and there is a positive relationship between etching time and wettability through the increase in free energy of the surface [8, 9, 11].

According to manufacturer Vita Zahnfabrik, the Vita Suprinity 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 [7, 8, 10].

The adhesive protocol for glass-ceramic involves the application of hydrofluoric acid, allowing the use of silane bifunctional agent and adhesive. Zirconia-reinforced lithium silicate ceramics may be bonded to dual resin cements, such as RelyX Ultimate, which have physical and mechanical properties such as bond strength, overtime stability, wear resistance, and compression resistance [14, 15]. These properties are superior to chemical or photopolymerizable cements, thus promoting more resistant and durable bonding between dental and ceramic structures [16].

Considering ceramic adhesion is strongly influenced by type of ceramic, HF concentration, and etching time, this study aimed to assess the effect of two HF concentrations (5% and 10%) at different etching times on the bond strength of a Vita Suprinity™ ZLS ceramic cemented with Relyx Ultimate™ resin cement (3M ESPE), also comparing the manufacturer's protocol of 5% HF for 20 s. The null hypothesis is that HF application at different concentrations and different times has no influence on the bond strength to a Vita Suprinity™ ZLS ceramic cemented with Relyx Ultimate™ resin cement, and the best option would be the recommendation of HF at the concentration and time suggested by the Vita Suprinity manufacturer.

Method

1. Research design

Experimental and completely randomized study with 8 treatments, involving two different concentrations of hydrofluoric acid and four different times (Figure 1) for each concentration (n=8), resulting in 64 samples. The sample was calculated at 5% α and 95% statistical power.

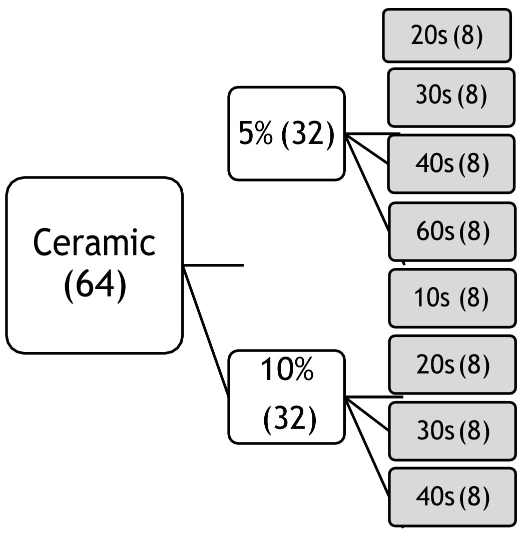


Figure 1. Illustrative scheme of the experimental design

2. Specimen preparation

The experimental units are ceramic slices (Vita Suprinity™) with 12x7 mm, which were bonded with epoxy resin to PVC tubes filled with acrylic resin for posterior setting in the testing machine. After the production of samples, they were hand-sanded for 30 s with #1200 grit papers, cleaned, and placed in an ultrasonic tank for 5 minutes, aiming to remove debris and standardize the surface.

3. Assessment of initial surface roughness

Initial roughness (Ra) was measured (Surfcorder SE 1700, Mitutoyo, Japan) with three readings in each sample in three different directions, going through 1.25 mm with 0.25-mm cut-off, meaning that for each 1.25-mm path 5 Ra measures were taken, one for each 0.25 mm. The Ra value of each sample is the mean of the three 1.25-mm readings. The samples were randomly divided into 8 groups according to HF concentration and etching time.

4. Surface treatment and roughness variation

Each sample was etched according to the experimental group, then abundantly washed for 1 min with air/water spray, and dried by air blast for 30". Next, a new Ra analysis was performed and the variation of roughness (ΔRa) was calculated per group studied.

5. Specimen production for the microshear test

Specimen production for the adhesive bond strength test through the microshear test (μSBS) refers to the production of 3 specimens in each sample with resin cement inserted in the Teflon mold. The adhesive bond strength value (MPa) of each sample was the mean of the 3 measurements.

The adhesive area of each specimen was defined by a duct tape fixed on the ceramic slice with 3 holes made with an Ainsworth perforator, with very close diameters to the external diameter of the Teflon mold. Consecutively, two layers of silane (Relyx Ceramic Primer - 3M ESPE - USA) were applied actively for 5 seconds and dried by air blast. The Single Bond Universal adhesive (3M ESPE - USA) was applied for 20 seconds, there was a mild air blast for 5 seconds, and no photopolymerization.

Each hole received a Teflon tube produced from catheters, making sure they were placed in the center of each adhesive area, resulting in 3 specimens per sample; they were horizontally exposed during the microshear test.

A portion of ¼ of the cement was manipulated by being inserted in the tubes with a spatula, preventing the formation of bubbles, and photopolymerized for 80 s (Valo Light Shield-Ultradent Products, USA). Then, the tubes were stored in a dark box, immersed in deionized water for 24 hours in a 37°C incubator.

Next, the surface of samples was cleaned, removing the molds carefully by manual traction, and the duct tape was cut with a scalpel blade from the holes and easily detached.

6. Microshear test (MPa)

Microshear test was performed in a universal testing machine EMIC DL2000 (São José dos Pinhais, PR, Brazil), applying load cell of 500 Kgf (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 1.0 mm/min until rupture.

7. Fracture mode analysis and SEM

The ceramic was analyzed with a digital microscope (Dino-Lite Premier, Anmo Electronics Corporation, New Taipei City, Taiwan) with 220x 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.

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.

8. Statistical analysis of the tests used

After testing data for normality with the Shapiro-Wilk test and variance homogeneity with the chi-square test, data were analyzed by ANOVA followed by Tukey's test (α=5%).

9.* Table 1.** ***Materials used

Ceramics	Vita Suprinity™ (Vita Zahnfabrik)
5% hydrofluoric acid	Condac (FGM)
10% hydrofluoric acid	Condac (FGM)
Adhesive	Single Bond Universal (3M ESPE)
Silane	RelyX Ceramic Primer (3M ESPE)
Dual Resin Cement	Relyx Ultimate (3M ESPE)

Results

Variation of surface roughness (Ra)

Table 2. Mean (standard deviation) of the variation of surface roughness - ΔRa (μm), according to the experimental groups

Acid	Etching time				
concentration	10''	20''	30''	40''	60''
5%	--	0.17(0.11)	0.29(0.12)	0.48(0.22)	0.60(0.15)
10%	0.32(0.15)	0.44(0.34)	0.57(0.23)	0.71(0.30)	--

Assessment of adhesive bond strength by the microshear test (MPa)

After verifying data normality and homogeneity, two-way ANOVA was performed, which evidenced significant differences among the experimental groups, therefore, Tukey's test was applied (α=5%). Table 3 presents these data.

There was statistical difference among groups 5%/20" and 10%/10", 10%/20", 10%/30", and 10%/40"; the remaining groups were not statistically different.

Table 3. Mean (standard deviation) of adhesive bond strength (MPa) to the microshear test of the eight experimental groups

Group	µSBS (MPa)
G.5/20	21.96 (6.21) b
G.5/30	25.00 (6.15) ab
G.5/40	28.30 (3.87) ab
G.5/60	29.04 (3.89) ab
G.10/10	29.59 (5.51) a
G.10/20	29.77 (3.91) a
G.10/30	29.68 (1.22) a
G.10/40	30.78 (2.61) a

Means followed by the same letter were not significantly different.

Fracture pattern analysis

After the microshear test, the fractures were assessed with 220x magnification through a digital microscope, and classified as adhesive, cohesive, and mixed. There was a prevalence of adhesive fractures (Table 4), except for G.5/40, in which adhesive and mixed fractures were equivalent **Table 4**. Rate of adhesive interface fracture pattern of the eight experimental groups.

	5%/20"	**5%/30"**	**5%/40"**	**5%/60"**
Adhesive	62.5%	62.5%	50%	75%
Mixed	37.5%	37.5%	50%	25%
	10%/10"	**10%/20"**	**10%/30"**	**10%/40"**
Adhesive	75%	75%	75%	62.5%
Mixed	25%	25%	25%	37.5%

Scanning Electron Microscopy (SEM) Analysis

Representative samples of each group were qualitatively assessed through scanning electron microscopy images (JSM 5600 Lv JEOL, Pleasanton, CA, USA) with 2000x magnification as shown in Figures 2 to 11B, and with 500x magnification in Figures 11A and 12. The exposure to hydrofluoric acid at 5% and 10% concentrations and different etching times shows the dissolution of the glassy phase, exposing larger microporosities and grooves.

After 5% hydrofluoric acid etching for 20", as suggested by the Vita Suprinity manufacturer, a higher amount of glassy phase could be observed, as well as small pores isolated with thin and irregular borders (Figure 2). The longer the acid etching time, the larger the pores and the more elongated the grooves, revealing areas described in the literature as honeycombs (Figure 11A) or beehives (Figure 12), potentially showing wider and shallower pores (Figure 12).

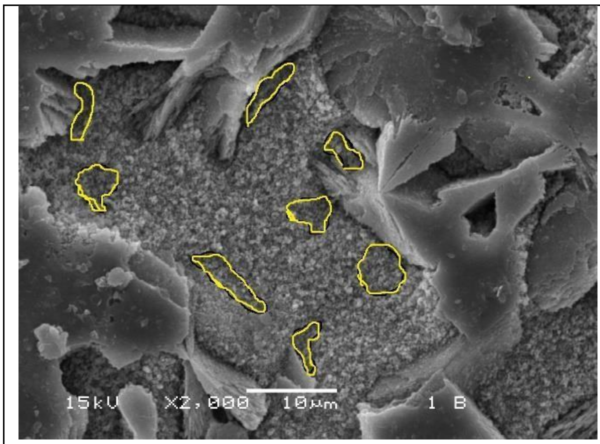


Figure 2. Photomicrograph of the ceramic surface after 5% HF etching for 20 s. Magnification 2000x; bar = 10 µm.

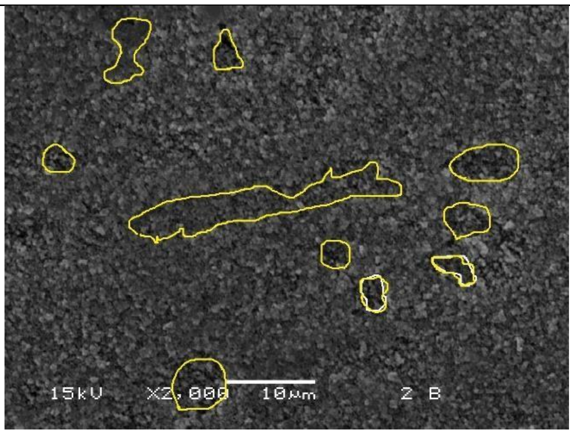


Figure 3. Photomicrograph of the ceramic surface after 5% HF etching for 30 s. Magnification 2000x; bar = 10 µm.

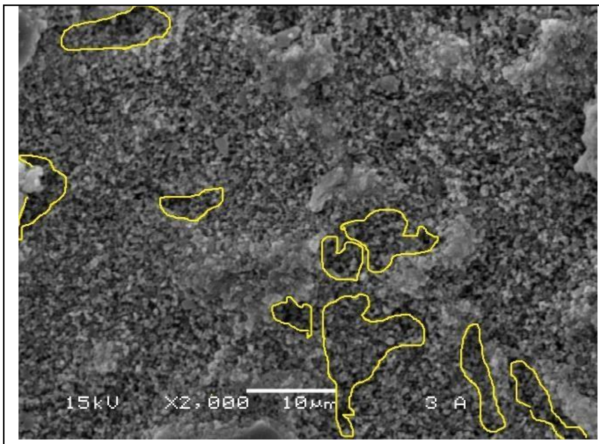


Figure 4. Photomicrograph of the ceramic surface after 5% HF etching for 40 s. Magnification 2000x; bar = 10 µm.

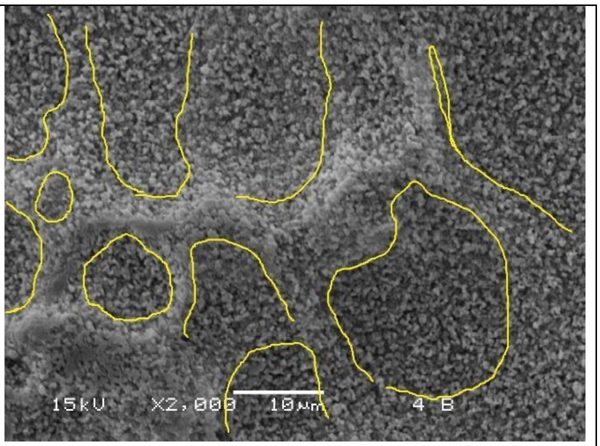


Figure 5. Photomicrograph of the ceramic surface after 5% HF etching for 60 s. Magnification 2000x; bar = 10 µm.

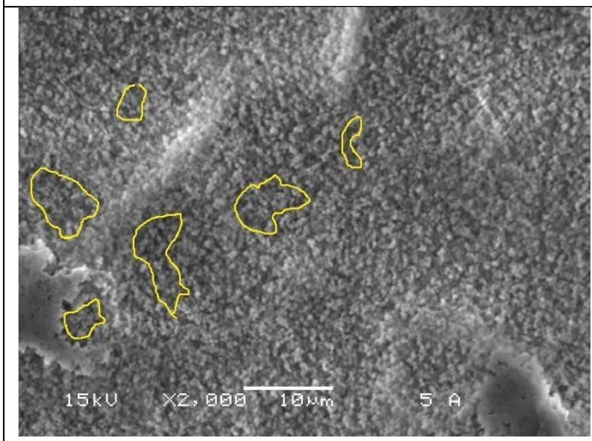


Figure 6. Photomicrograph of the ceramic surface after 10% HF etching for 10 s. Magnification 2000x; bar = 10 µm.

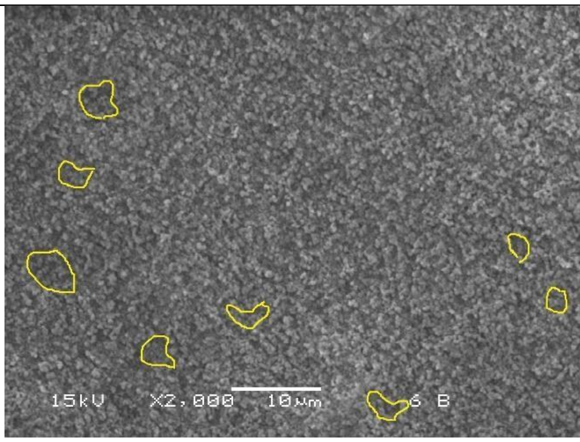


Figure 7. Photomicrograph of the ceramic surface after 10% HF etching for 20 s. Magnification 2000x; bar = 10 µm.

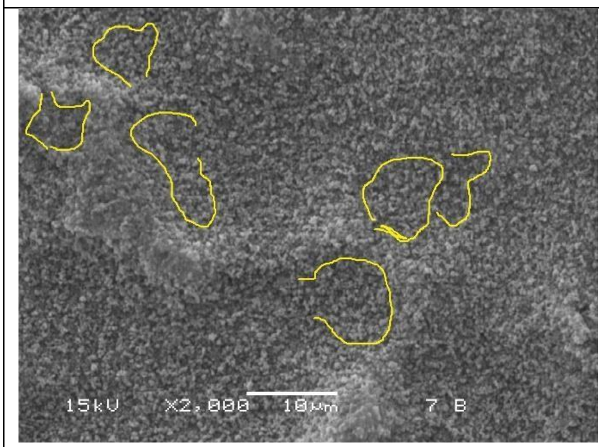


Figure 8. Photomicrograph of the ceramic

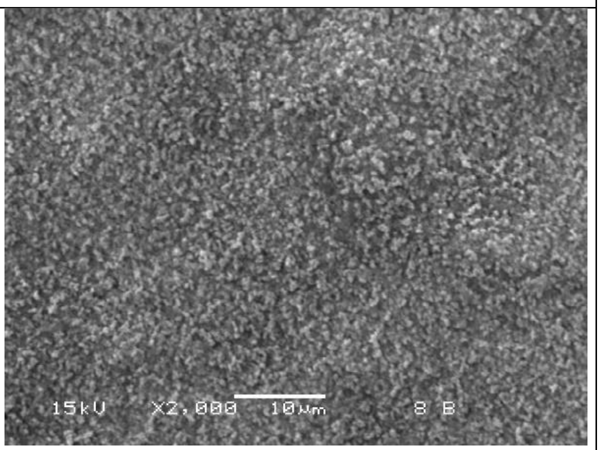
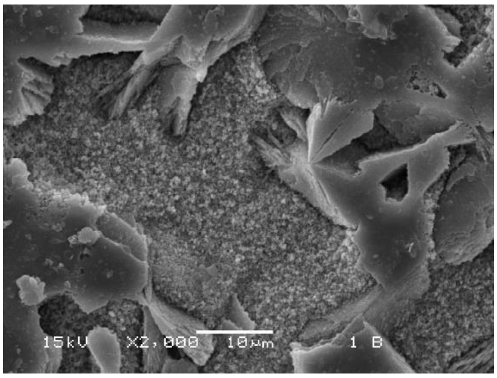
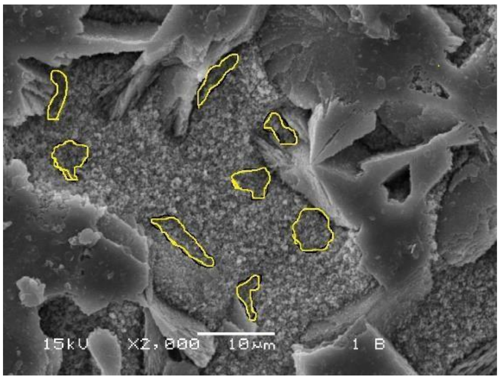


Figure 9. Photomicrograph of the ceramic

surface after 10% HF etching for 30 s. Magnification 2000x; bar = 10 µm.	surface after 10% HF etching for 40 s. Magnification 2000x; bar = 10 µm.

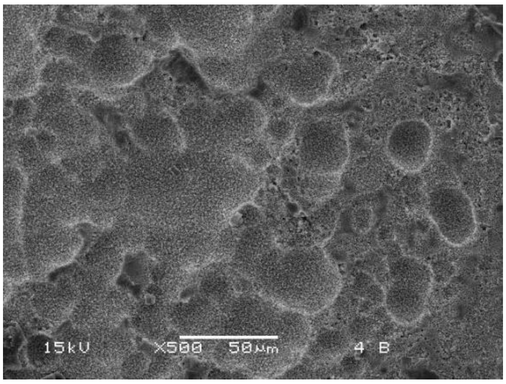


10a

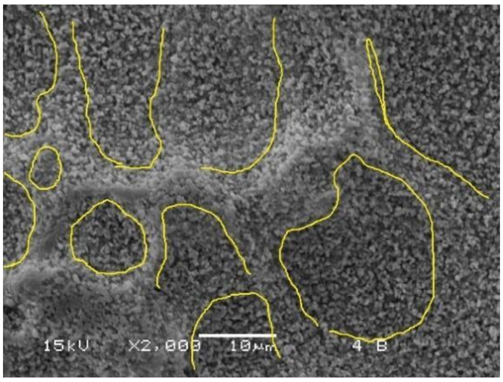


10b

Figure 10a e 10b. Photomicrograph of the ceramic surface after 5% HF etching for 20 s. Magnification 2000x; bar = 10 µm.

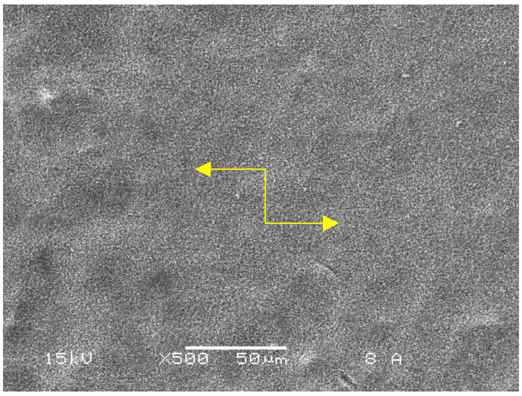


11a

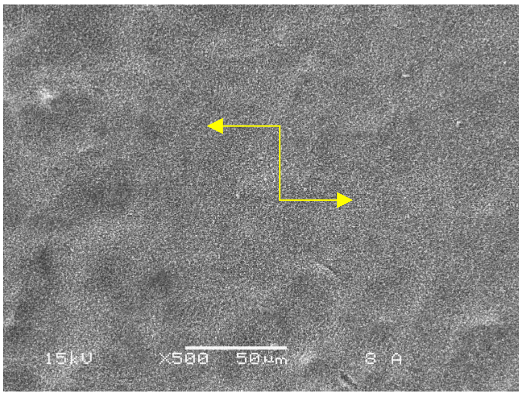


11b

Figure 11a e 11b. Photomicrograph of the ceramic surface after 5% HF etching for 60 s, showing the honeycomb aspect (arrow). 11a-Magnification 500x. 11b-Magnification 2000x. B-Shallow and wide pores.



12a



12b

Figure 12a e 12b. Photomicrograph of the ceramic surface after 10% HF etching for 40 s, showing the beehive aspect (arrows). Magnification 500x; bar = 50 µm.

Discussion

This study aimed to compare different concentrations of hydrofluoric acid applied on a ceramic surface at different times with the manufacturer's recommendation of using 5% hydrofluoric acid for 20". The results obtained showed there are differences in the adhesive bond strength between ceramic and dual resin cement depending on the selection of acid concentration and exposure time. Therefore, the null hypothesis tested was rejected, showing potential differences in adhesive bond strength at different concentrations of hydrofluoric acid and different times, using the Vita Suprinity ceramic after cementation with RelyX Ultimate resin cement.

This study assessed the adhesive bond strength to Vita Suprinity ceramic, which has a high amount of glass matrix, making this material vulnerable to acid corrosion [2, 7, 16] and consequently exposes elongated lithium silicate crystals, which favors the retention of cementation agents [9, 11, 16, 18, 20]. Acid corrosion is a dynamic process that depends on ceramic composition, surface topography, acid concentration, and etching time of the piece, whereas HF concentration is defined by the manufacturer, but etching time may be controlled by the operator [18].

The surface roughness of the etched ceramic may widely vary, reflecting the formation of micro- and nanoporosities and variable widths and depths [2, 7, 8, 18, 20, 22], due to the acid action on the glass matrix and exposure of the crystalline structure [7, 18, 19, 20]. Previous studies [19, 20, 21, 22] show there is no determining time for ideal roughness, considering retention depends on several factors such as the type of ceramic you are working with, whether it is a glass-ceramic or not, for instance; the hydrofluoric acid concentration to be applied on the ceramic surface, which is determined by the manufacturer but chosen by the operator; and the time this acid will stay on the ceramic surface, which is often suggested by the ceramic manufacturer but may be completely controlled by the operator [8]. This study found that increasing HF concentration and etching time promoted increased surface roughness, as presented in Table 2.

There is a positive relationship between ceramic roughness and wettability through the increase in free energy of the surface [7, 8, 18], which favors the mechanical interlocking of bonding agents and increases the retention of the piece [23, 24]. However, it will occur up to a certain point [7, 8, 9, 11], after which larger and wider, but shallower, porosities are observed [7, 8, 9, 22], corroborating findings of this study where SEM images (Figures 11A and 12), respectively for groups 5% 60" and 10% 40", showed larger porosities. Pore width increases because of the dissolution and disintegration of the weaker glassy phase to a faster rate than the crystalline phase, and this significant loss of the glassy phase weakens the ceramic and may affect bond strength [25, 26].

The bond strength observed in group 5% 20" was significantly lower than the four times (10", 20", 30", and 40") with 10% HF, but there were no increases as significant for bond strength. This suggests there is no need to extend hydrofluoric acid exposure for a long time, regardless of the acid concentration used, considering it will result in no gains for adhesive bond strength. Other researches [9, 12, 13, 18] show that applying silane and adhesive on ceramic surfaces with the presence of lithium disilicate may result in excellent bonding with lower HF acid concentration and within a shorter time. However, according to these authors, if the bifunctional agent is not applied, we should work with a higher acid concentration, 10% for instance, for a maximum of 60", in order to increase bond strength and seek less damage to the ceramic structure.

The assessment of fracture pattern showed prevalence of adhesive fractures, except for the group at 5% HF acid concentration and 40" of exposure time, in which adhesive and mixed fractures were equivalent. Studies show [12, 14] that non-homogeneous stress could be developed in the bonding interface and extend to the substrate and resin cement, leading to cohesive or mixed fractures. This occurs when mechanical and structural resistances of substrates represent the weaker bond in the system, so the adhesive bond strength exceeds the intrinsic ceramic resistance and causes cohesive fractures. On the other hand, in other studies [12, 14] as well as in this study, the Vita Suprinity ceramic, which is a glass-ceramic with reinforcing crystalline phase, promotes higher resistance to other ceramics, showing less cohesive and mixed fractures and consequently presenting higher values in the microshear test.

Bond strength increased with higher HF concentration and longer acid exposure time. The manufacturer of the Vita Suprinity ceramic recommends 5% HF etching for 20 s, but the SEM images show that the glassy phase is little dissolved, leaving surfaces with fewer irregularities. On the other hand, the 5% HF etching for 30 s and 10% for 10 and 20 s showed surfaces with more irregularities but no excessively wide and shallow pores, which favors micromechanical interlocking. Etching for more than 30 s did not result in significant bond strength increase and showed wider and shallower pores, which make the ceramic surface more fragile.

Therefore, it is possible to increase adhesive bond strength to Vita Suprinity ceramic by adding 10 seconds to the time suggested by the manufacturer (20 seconds) at 5% hydrofluoric acid concentration, meaning that when using such concentration the acid should be applied for

30 seconds. It is also possible to obtain a surface with higher adhesive bond strength to ceramic when using 10% hydrofluoric acid at exposure times of 10 seconds and 20 seconds.

These three options - 5% hydrofluoric acid for 30 seconds or 10% hydrofluoric acid for 10 and 20 seconds - resulted in surfaces more susceptible to mechanical interlocking, due to the pattern of pores and irregularities formed, than the suggestion of the Vita Suprinity manufacturer (5% HF for 20 seconds).

Conclusion

Considering the limitations of this study, it may be concluded that Vita Suprinity ceramic etched with 5% HF for 30 s or 10% HF for 10 and 20 s showed higher bond strength than the manufacturer's protocol, but without showing wide and shallow porosities.

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