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Effect of the Etching Duration and Ultrasonic Cleaning on Microtensile Bond Strength Between Feldspathic Ceramic and Resin Cement

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This study assessed the effect of different etching durations of feldspathic ceramic with hydrofluoric acid (HF) and ultrasonic cleaning of the etched ceramic surface on the microtensile bond strength stability of resin to a feldspathic ceramic. The research hypotheses investigated were: (1) different etching times would not affect the adhesion resistance and (2) ultrasonic cleaning would improve the adhesion. Ceramic blocks (6 × 6 × 5 mm) (N = 48) were obtained. The cementations surfaces were duplicated in resin composite. The six study groups (n = 8) were: G1—Etching with 10% aqueous HF (30 s) + silane; G2—10% HF (1 min) + silane; G3—10% HF (2 min) + silane; G4—10% HF (30 s) + ultraic cleaning (4 min) in distilled water +silane; G5—10% HF (1 min) + ultrasonic cleaning + silane; G6—10% HF (2 min) ultrasonic

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cleaning + silane. The cemented blocks were sectioned into microbars for the microtensile test. The etching duration did not create significant difference among the groups ($p = .156$) but significant influence of ultrasonic cleaning was observed ($p = .001$) (Two-way ANOVA and Tukey's test, $p > 0.05$). All the groups after ultrasonic cleaning presented higher bond strength (19.38–20.08 MPa) when compared with the groups without ultrasonic cleaning (16.21–17.75 MPa). The bond strength between feldspathic ceramic and resin cement was not affected by different etching durations using HF. Ultrasonic cleaning increased the bond strength between ceramic surface and resin cement, regardless of the etching duration.

KEYWORDS Acid etching; Adhesion; Feldspathic ceramic; Hydrofluoric acid; Microtensile bond strength; Ultrasonic cleaning

INTRODUCTION

The use of metal-free fixed dental prosthesis (FDP) made of feldspathic ceramics (inlays, onlays, laminate veneers) utilizing adhesives requires minimal intervention that allows preservation of hard dental tissues to a great extent. Clinical durability of such restorations depends on the durable adhesion at different interfaces in the adhesive procedures [1,2]. Consequently, a precise conditioning method of the cementation surface of all-ceramic FDPs is essential [3–5].

One of the most commonly used dental ceramics is the feldspathic ceramic that consists of a vitreous structure basically composed of two minerals, namely, feldspar and quartz. The feldspar bonds to metallic oxides to form the vitreous phase of porcelain, while quartz composes the crystalline phase. More recently, a feldspathic ceramic classified as biphasic vitreous ceramic called VM7 (Vita Zahnfabrik, Bad Sackingen, Germany) has been introduced in the market. This ceramic material principally consists of Si: 19.6%; Al: 4.9%; K: 4.0%; Na: 2.4%; Ca: 0.7%; C: 25.7%; and O: 42.2% [3–5]. This material is indicated for inlay, onlay, partial restorations, laminate veneers, and also as a veneering material on some high-performance ceramic frameworks.

The cementation surface of all-ceramic restorations should be conditioned to allow for retention between the resin-based materials and the ceramic [4,5]. The kind of ceramic surface conditioning for adhesion is defined according to the type of ceramic involved [4]. Dental ceramics are classified according to their sensitivity to hydrofluoric acid etching as follows: a) acid-sensitive—ceramics that suffer surface degradation by aqueous hydrofluoric acid (feldspathic, leucite, and lithium disilicate ceramics), giving rise to a topographic pattern that favors the micromechanical retention and b) acid-resistant—ceramics resistant to etching by hydrofluoric acid (glass-infiltrated alumina or alumina/zirconia ceramics, containing densely sintered zirconia/yttrium and

alumina that are usually submitted to air-borne particle abrasion to optimize adhesion [4,5].

For acid-sensitive ceramics, such factors such as type, concentration, and time of hydrofluoric acid etching might influence the resin bond strength as a function of the sensibility for dissolution by acid attack of the glassy phase or leucite in these ceramics [6–9]. Thus, concentration and duration of acid etching should be established according to the type of feldspathic ceramic, in order to improve the bond between with the adhesive resin and the ceramic [7,8,10,11].

Acid etching with hydrofluoric acid on feldspathic ceramic significantly changes the surface morphology of the ceramics, creating irregularities on the ceramic surface due to selective dissolution of the vitreous phase, which is represented by retentive micropores. The number and size of these micropores have been associated with an increase in bond strength and their presence enhances penetration of the bonding agent [7,8,10,12]. Etchants such as hydrofluoric acid react with silica present in the ceramic and form acid precipitates, products of reaction of sodium fluorosilicate (Na), potassium (K), calcium (Ca), and aluminum (Al) located on the surface of micropores [13]. Their presence could damage the bond strength between ceramic and bonding agent and may cause clinical failure of the restorations [14]. Additional procedures such as elimination of excess acid and acid precipitates from the etched ceramic surface may improve adhesion [15]. One such procedure is ultrasonic cleaning in distilled water that effectively removes acid precipitates from ceramic restorations [13].

The comprehension of these factors displayed already has relevance, in view of the fact that the success and longevity of ceramic restorations are closely related to pre-cementation surface treatment and to cementation itself [1,2,7,8,16,17]. Thus, the duration of etching with hydrofluoric acid should be carefully followed, as well as cleaning of acid precipitates.

Hence, the objective of this study was to evaluate the impact of different durations of hydrofluoric acid and ultrasonic cleaning of the etched ceramic surface on the microtensile bond strength of resin cement to a feldspathic ceramic. The research hypotheses investigated were: a) different etching duration would not affect the adhesion and b) ultrasonic cleaning would improve the adhesion.

MATERIALS AND METHODS

Description of materials, brands, composition, and manufacturers of the products are presented in Table 1.

Production of Ceramic Blocks

An acetate template measuring $8 \times 8 \times 6$ mm was machined. Ceramic blocks ($N = 72$) (Vita VM7® Dentin 5M2, Vita Zahnfabrik, Bad Sackingen, Germany;

TABLE 1 Materials Used in This Study

Brand	Manufacturer	Composition
VITA VM7	VITA Zahnfabrik, Bad Säckingen, Germany	Si: 19.6%; Al: 4.9%; K: 4%; Na: 2.4%; Ca: 0.7%; C: 25.7%; O: 42.2%
W3D MASTER	Wilcos do Brasil Ltd, RJ, Brazil	Methacrylate monomers, pyrogenic silica, barium, and aluminum silicate
10% hydrofluoric acid	Dentsply, Petrópolis, RJ, Brazil	Hydrofluoric acid, water, thickener, and dye
Porcelain Primer	Bisco, Schaumburg, IL, USA	Hydrolyzed y-methacryloxypropyl trimethoxy silane
RelyX ARC	3M ESPE, St Paul, MN, USA	Paste A: bis-GMA, TEGDMA, particles of zirconia/silica, photoinitiator, pigments Paste B: bis-GMA, TEGDMA, particles of zirconia/silica, benzoyl peroxide

Batch # 7404) were prepared following the manufacturer's instructions. The bulk ceramic was inserted and packed into the template. The blocks were sintered in a furnace (Vacumat®, Vita) using the specific program indicated by the manufacturer. After sintering, volume shrinkage of the ceramic was nearly 20% and the blocks measured approximately $6.4 \times 6.4 \times 4.8$ mm.

The cementation surface (6.4×6.4 mm) was flattened and polished in a machine (Labpol™ 8–12, Extec, Enfield, CT, USA) using silicon carbide papers in a sequence of 600-, 800-, and 1200-grit (3 M™ ESPE, St. Paul MN, USA). Ceramic blocks were then ultrasonically cleaned with distilled water for 5 minutes (Vitasonic, Vita Zahnfabrik, Germany). Forty-eight ceramic blocks were used for adhesion testing, 12 blocks for micro-morphological evaluations, and 12 for energy dispersive spectrometry (EDS) analysis.

Production of Composite Blocks

Impressions were made from each ceramic block with putty addition silicone (Aquasil™, Dentsply, York, PA, USA), with the bonding surface turned downward, so as it was impressed in the material, as well as the entire ceramic block. After polymerization of the impression material, each ceramic block was removed from the impression material. Composite resin (W3D Master™, Wilcos, Petropolis, Brazil) was then inserted in the impression in 2-mm increments; each increment was photo-polymerized for 40 seconds (UltraLED™, Ultradent®, South Jordan, UT, USA) until the impression was completely filled. For each ceramic specimen a separate composite block was built up.

Experimental Design

The ceramic blocks were randomly divided into six groups ($n = 8$ per group), according to the duration of etching with 10% hydrofluoric acid. After rinsing

half of the specimens were randomly assigned for ultrasonic cleaning in distilled water for 4 minutes and the other half was not ultrasonically cleaned. The adhesion surface of the ceramic blocks from the groups 1, 2, and 3 were etched with 10% aqueous hydrofluoric acid for different durations of 20 seconds, 1 minute, and 2 minutes, respectively. After etching, the ceramic surfaces were rinsed with air-water spray for 60 seconds, and air-dried for 30 seconds. The adhesion surface of the ceramic blocks from the groups 4, 5, and 6 were acid etched for the same durations and submitted to ultrasonic cleaning with distilled water for 4 minutes, and air-dried for 30 seconds.

Cementation

All the adhesion surfaces of the ceramic blocks were silanized by a metacryloxypropyltrimethoxy MPS-based silane for 5 minutes. Each ceramic block was bonded to its corresponding composite resin block using resin cement (RelyX ARC™, 3 M ESPE, Minn, USA), prepared following the manufacturer's instructions, and applied with a plastic spatula on the treated surface of each ceramic block. The ceramic-composite assembly was placed on a surveyor adapted for cementation, with the cementation surface perpendicular to the loading jig. A vertical load of 750 g was employed throughout the cementation procedure for 10 minutes in order to control the cement film thickness [4,18].

After positioning the ceramic-cement-resin assembly, the excess cement was removed and adhesive interface at each side of the cement-ceramic assembly was photo-polymerized for 40 seconds with a polymerization unit (Ultra LED, Ultradent, South Jordau, UT, USA). The ceramic-cement-resin assemblies were stored in distilled water at 37°C for 7 days until preparation of specimens.

Production of Non-Trimmed Beam Samples

The ceramic-cement assembly was sectioned with steel diamond discs (no. 34570, Microdont™, Barueri, Brazil) at low speed under water cooling that was mounted on a handpiece (Kavo Ind. e Com. Ltda, Joenville, Brazil) connected to a modified mechanical lathe with calibration on the x- and y-axes, thus allowing sectioning in both directions [4,7,17,18].

Initially, each ceramic-cement-composite resin assembly was fixed on a cylindrical metallic base with cyanoacrylate adhesive (Super Bonder®, Henkel-Loctite®, Itapevi, Brazil). The metallic base was connected to a clamp in the sectioning machine. Each ceramic-cement-resin block was perpendicular to the diamond disc to allow sectioning as regularly as possible, to achieve sections of equivalent thickness. The first section was eliminated at each side of the specimen (±0.5 mm) that might have excess cement around the bonding interface, which, in turn, could directly influence the bond strength values. Thereafter, three sections were cut from the ceramic-cement-resin blocks, and

sections were obtained with approximately 1-mm thickness. Each section was then rotated 90° and once again fixed to the metallic base. The first section of the specimen (±0.5 mm) was eliminated for the same aforementioned reasons and a further three sections with ±1 mm in thickness were obtained. The same process was repeated for the other two sections, adding up to nine non-trimmed square cross–section (1 × 1 mm) beam-shaped specimens for each bonded ceramic-resin assembly. The beams had bonded area of ±1 mm^2 and length of ± 8 mm [4,7,17,18–21].

Microtensile Bond Strength Test

For the microtensile testing, each beam specimen was fixed with cyanoacrylate adhesive (Super Bonder, Henkel-Loctite, Itapevi, Brazil) to an adapted caliper that allowed parallel force application to avoid torsional stresses at the bonded area (Fig. 1). Only the end portions of the specimens were used for fixation as the bonded area was located between the caliper tips. The apparatus-specimen assembly was placed in a Universal Testing Machine (EMIC DL 1000™, EMIC, Sao Jose dos Pinhais, Brazil) and submitted to tension (1 mm/min, 10 kgf load cell) until debonding [4,7,17–21].

The interfacial cross-sectional area of all specimens was measured before testing with a digital caliper (Starret™, Itu, Brazil) to 0.01-mm precision. Measurement of the area and the load value required for debonding

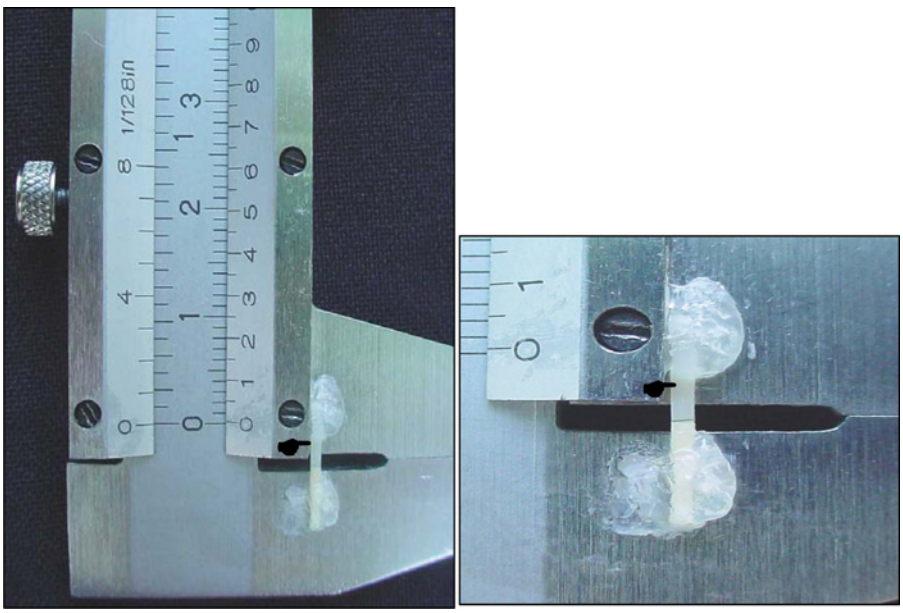

FIGURE 1 Microtensile testing apparatus. The beam specimen is indicated by (color figure available online).

allowed calculation of the bond strength (MPa) according to the following equation: $R_t = F/A$, in which R_t is the bond strength; F is the force applied for adhesion failure; and A is the adhered interfacial area.

After testing, the surfaces of beam specimens were examined under a stereomicroscope (Zeiss MC 80 DX™, Carl-Zeiss, Gottingen, Germany) at ×50 magnification to classify the failure pattern at the ceramic-cement interface. Failures were then classified as adhesive, cohesive, or mixed.

Representative pairs of tested specimens from each group were evaluated under scanning electron microscopy (SEM) (Jeol-JSM-T330A – Scanning Microscope, Tokyo, Japan) at ×150 magnification.

Micromorphological and EDS Analyses

Additional ceramic blocks were conditioned as described for each group and observed in the SEM at between ×500 and ×2,000 magnification to assess the topographic changes caused by different ceramic conditioning regimens.

Additional conditioned ceramic blocks were analyzed by energy dispersive spectrometry (EDS, Jeol-JSM-T330A, Scanning Microscope, Tokyo, Japan) to verify the chemical elements present on the ceramic surface. Mapping was performed per area to investigate the presence of precipitates of hydrofluoric acid on the ceramic surface.

Statistical Analysis

Microtensile data were submitted to two-way ANOVA and Tukey's post-hoc tests using MINITAN (Minitab, version 14.12, 2004; State College, PA, USA), STATISTICA (StatSoft®, version 5.5, 2000; Hamburg, Germany), and STATISTIX (Analytical Software, version 8.0, 2003; Tallahassee, FL, USA) (alpha = 0.05). The ceramic-composite blocks were considered as the statistical unit for the statistical analysis. Power analysis was performed using a statistical software package (Stata, StataCorp, Texas, USA).

RESULTS

Two-way ANOVA revealed that the duration of acid etching time did not significantly influence the bond strength results ($p = .156$) (Table 2) but the condition of ultrasonic cleaning significantly increased the bond results ($p = .001$) compared with non-cleaned groups regardless of the etching duration (Tukey's test) (Fig. 2, Table 3). The interaction terms were not significant ($p = .547$). The power of the study was calculated to be 80% (CI 95%).

SEM and optical microscope analysis of debonded specimens revealed exclusively mixed failure types between the adhesive and resin cement and the resin cement and ceramics.

TABLE 2 Analysis of Variance (ANOVA) of Bond Strength Data (MPa)

Source of variation	df	SS	MS	F	p
Ultrasonic cleaning	1	132.00	132.00	16.66	0.001*
Duration of acid etching	2	30.74	15.37	1.94	0.156
Interaction	2	9.71	4.86	0.61	0.547
Residue	42	332.80	7.92		
Total	47	505.25			

*$p < 0.05$.

Ceramic surfaces after treatment with 10% hydrofluoric acid at different etching durations and were not ultrasonically cleaned demonstrated pores, grooves, and deposition of precipitates resulting from etching of ceramic

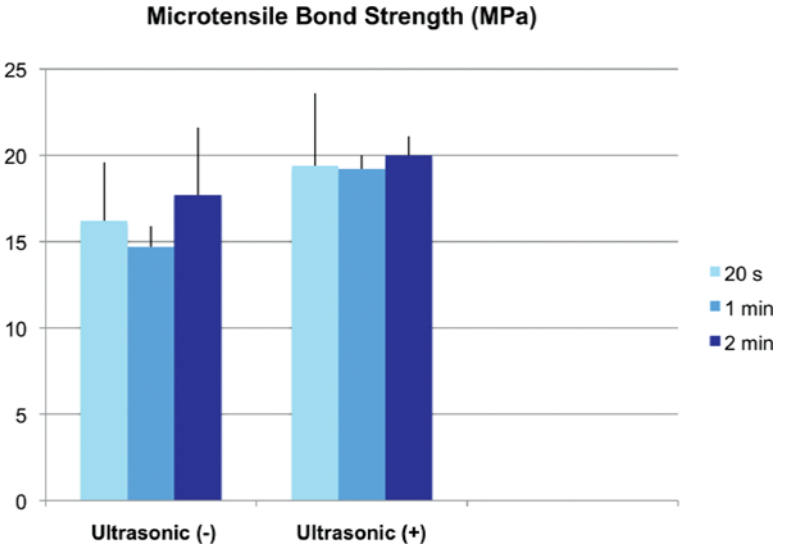

FIGURE 2 Mean microtensile bond results of the resin cement to feldspathic ceramic and standard deviations after various etching durations with and without ultrasonic cleaning conditions after etching (color figure available online).

TABLE 3 Mean Bond Strength (±Standard Deviation) (MPa) for the Groups and Post-hoc Tukey Test Results

	Ultrasonic cleaning		
Etching duration	Without	With	
20 s	16.2 ± 3.4^{b}	19.4 ± 4.2^{a}	17.8 ± 4
1 min	14.7 ± 1.2^{b}	19.2 ± 0.8^{a}	16.9 ± 2.5
2 min	17.7 ± 3.9^{b}	20 ± 1.1^{a}	18.9 ± 3
	16.2 ± 3.2^{A}	19.5 ± 2.5^{B}	

Same superscript letters in the same column indicates no statistically significant difference at the 5% level.

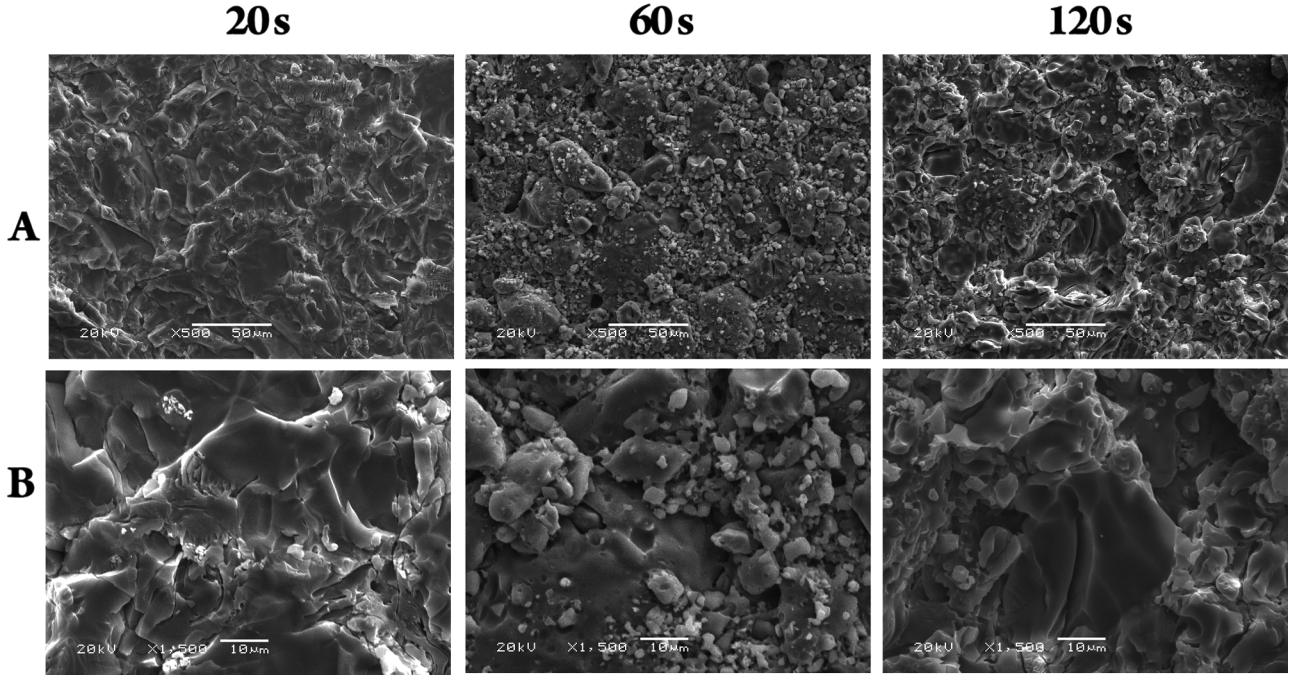

FIGURE 3 A-B. Micrographs (A- ×500, B- ×2.000) of the acid-etched ceramic surfaces after different etching durations (20 seconds, 1 minute, 2 minutes, from left to right, respectively), without ultrasonic cleaning.

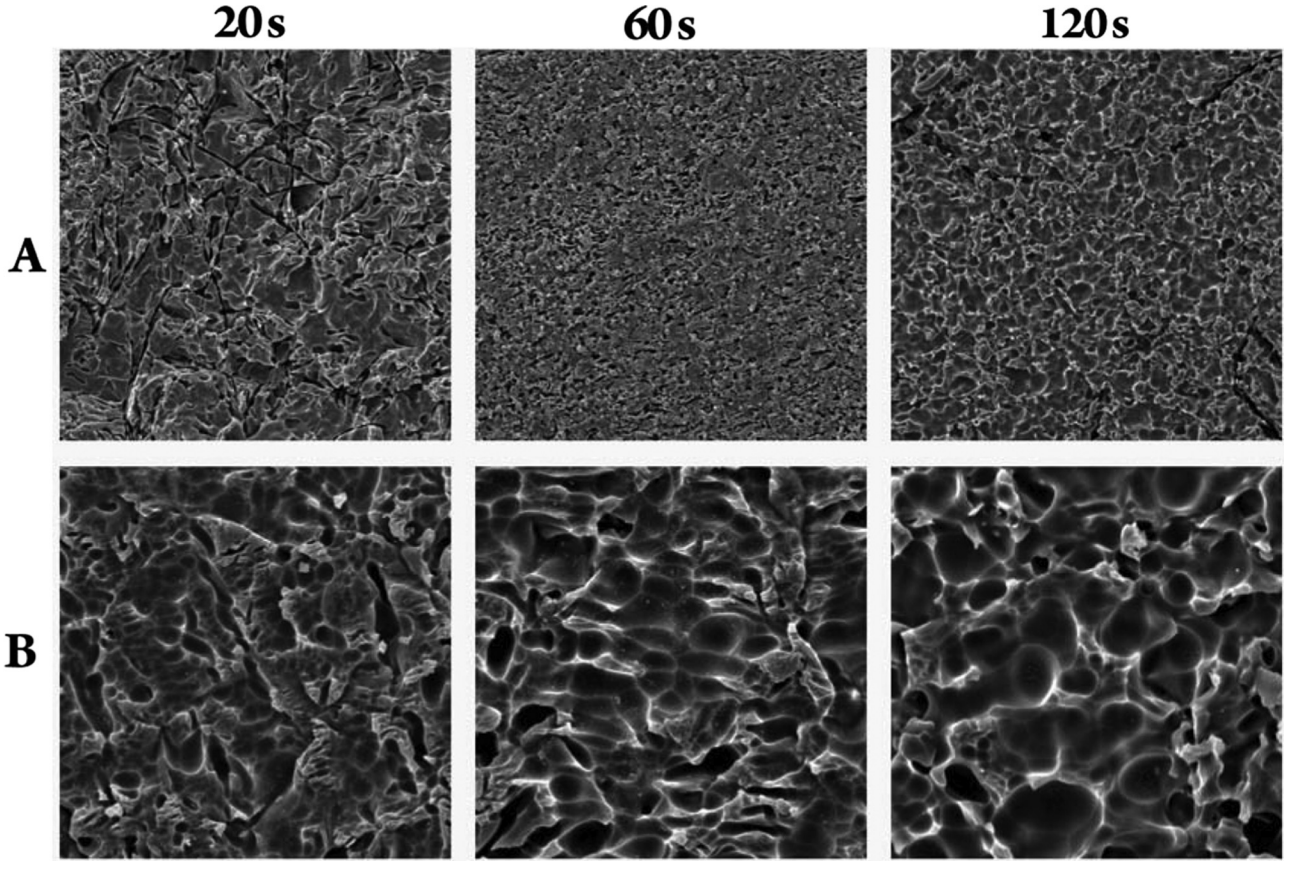

FIGURE 4 A-B. Micrographs (A- ×500, B- ×2.000) of the acid-etched ceramic surfaces after different etching durations (20 seconds, 1 minute, 2 minutes, from left to right, respectively), after ultrasonic cleaning.

surfaces on all specimens (×500 and × 1500) (Figs. 3A–B). The micropores and grooves quantitatively increased with the increase in etching time. The micropores became larger in form with the increase in etching time. On the other hand, ultrasonic cleaning was capable of removing acid precipitates, efficiently opening micropores and grooves (Figs. 4A–B).

Chemical elemental analysis of the ceramic surfaces using EDS revealed the presence of Si, Al, Na, K, and O, which characterize the microstructure of a vitreous ceramic, composing the network of silica (SiO_2) and potassium ($K_2O.Al_2O_3.6SiO_2$) or sodium ($Na_2O.Al_2O_3.6SiO_2$) feldspar, or both (Fig. 5A). The spectra of the specimens etched by hydrofluoric acid revealed the presence of fluorine, which is characteristic of the acid precipitate (reaction products of Na, K, Ca, and Al fluorosilicate) (Figs. 5B–D). On the contrary, EDS

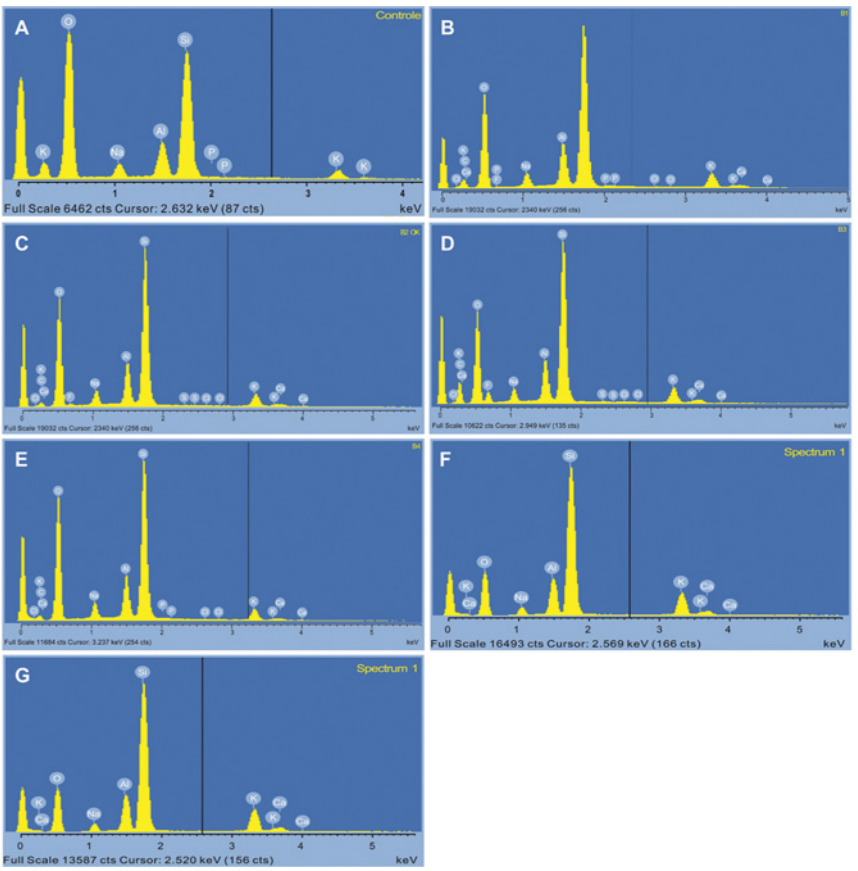

FIGURE 5 A-G. Spectra of the EDS analysis of the ceramic surface after different routes: A — non-etched surface; B, C, and D — ceramic surface etched with hydrofluoric acid for 20 seconds, 1 minute, and 2 minutes, respectively. Note that the F element has been detected; E, F, and G — ceramic surface etched with hydrofluoric acid for 20 seconds, 1 minute, and 2 minutes, respectively, and ultrasonically cleaned. Note the absence of F (color figure available online).

analysis of the acid-etched and ultrasonically cleaned specimens demonstrated the absence of fluorine, indicating the efficacy of the cleaning method (Figs. 5E–G).

DISCUSSION

Hydrofluoric acid etching followed by silanization is the most frequently employed surface conditioning method for feldspathic ceramics [14,16,22–25]. Hydrofluoric acid preferably reacts with the silica present in the ceramic microstructure, forming hexafluorosilicates. As a result of this reaction, the ceramic surface becomes porous, the irregular surface area is increased, and adhesive resin penetrates into the microretentions on the acid-etched ceramic surface [13].

Previous studies on etching of glassy-matrix ceramics [6,25–27] addressed the effect of different etching durations with hydrofluoric acid on the bond between adhesive resin and the feldspathic ceramics. The ceramic VM7 (Vita) is a type of feldspathic ceramic indicated for the fabrication of indirect FDPs and to veneer alumina ceramic (In-Ceram®) frameworks. However, no information is available on its adequate etching duration. Therefore, the first objective of this study was to characterize the effect of etching duration for this ceramic. The etching durations employed were based on previous studies [19,25–27]. The results revealed no statistically significant influence of etching time with hydrofluoric acid on the bond strength results, yielding to the acceptance of the first hypothesis. This does not corroborate the results of Chen et al. [26], who observed that the increase in etching duration led to an increase in bond strength. However, for this study a different ceramic was used. In fact, the ceramic microstructure and ceramic composition control the development of mechanical microretentions produced by hydrofluoric acid etching which might have influenced the variations in the results [23].

Ultrasonic cleaning of the etched ceramic surfaces removes the precipitates caused by hydrofluoric acid from the ceramic surface. These precipitates are insoluble fluorosilicate salts that remain on the surface of the micropores, possibly reducing the bond strength between cement and ceramic. Ultrasonic cleaning with distilled water was performed for 4 minutes, as suggested previously [13]. Ultrasonic cleaning of acid precipitates with distilled water significantly increased the bond strength results regardless of the etching time, yielding to acceptance of the second hypothesis. This may have occurred because ultrasonic cleaning removed the acid precipitates and consequently the resin cement could wet the surface more evenly and interact more efficiently with the etched ceramic surface. The bond strength values obtained after ultrasonic cleaning could not be compared with findings of other studies since no studies were found in the literature addressing this aspect.

Utilization of silane is an important step for adhesive cementation [20,22] since it is an organofunctional molecule that promotes chemical bonding between an inorganic substrate, herein represented by the vitreous matrix of feldspathic ceramic Vita VM7®, and organic polymers such as HEMA, which is found in the resin cement RelyX®. The application of silane on the hydrofluoric acid-etched ceramic surface may cause dissociation of fluorosilicate salts [13]. This occurs by hydrolysis and absorption of silane on the etched ceramic surface. Another important factor is the capacity of silane to promote better surface wettability, increasing the contact and infiltration of bonding agent into porosities created on the ceramic surface by hydrofluoric acid etching [29].

The analyses of failures of the beam specimens submitted to the microtensile testing displayed exclusively mixed types of failures in the iterfacial zone. These findings corroborated the information from the literature, where the percentage of failures in the interfacial zone was found to be higher in the microtensile test than in shear tests where in the latter cohesive failures in the ceramic substrate were more common [20,23,30–37].

In practice, there are two interfaces, namely, one to the ceramic and the other to the dentin. In this study, the ceramic blocks were bonded to composite resin because the main objective was to assess the adhesion of the resin to the ceramic substrate. Since the dentin substrate presents a highly heterogeneous composition, especially due to the variations in the orientation of the dentin tubules, the research question could not have been answered if the tooth would have been involved. If ceramic blocks had been bonded to dental substrate, the failure after testing could have happened at the dentin-cement interfaces and the bond to ceramic would then not be well evaluated. Nevertheless, this aspect could be considered one limitation of this study and it warrants future research. Further investigations focusing on the long-term aging conditions should also be studied to evaluate the durability of adhesion to feldspathic ceramic.

CONCLUSIONS

1. The adhesion of the resin cement to the feldspathic ceramic tested presented similar bond strength after 20 seconds, 1 minute, or 2 minutes of hydrofluoric acid etching. Clinical workflow could be reduced to 20 seconds for etching procedures of this ceramic.
2. Regardless of the etching duration, ultrasonic cleaning in distilled water for 4 minutes improved the adhesion of the resin cement to feldspathic ceramic significantly and removed the precipitates effectively. This procedure should be considered essential after etching the feldspathic ceramic.

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