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# EFFECT OF SURFACE TREATMENT ON FLEXURAL STRENGTH AND SUBCRITICAL CRACK GROWTH OF LITHIUM DISILICATE: AN IN VITRO STUDY

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## Abstract
Alternative surface treatments have been proposed for the cementation of lithium disilicate ceramics aiming to improve adhesive and flexural strength under fatigue. This study aimed to evaluate the slow crack growth (SCG) parameters of the lithium disilicate ceramic after hydrofluoric acid (HF) etching or air abrasion (AB) as surface treatments. Ceramic discs were treated with HF (5%, 20 s) or AB (30 µm silica-modified alumina particles, 2.8 bar, 10 mm distance, 15 s), and received a layer of resin cement. The surface roughness after surface treatment was evaluated (n = 5). Samples were tested in a piston-on-three-ball assembly to evaluate the flexural strength (n = 20), inert strength (n = 25), and to determine SCG parameters *n* and D (n = 35). The highest roughness ($p < 0.01$) was observed in the AB group, with the highest reliability according to the Weibull analysis, but the lowest SCG susceptibility. Flexural ($p = 0.03$) and inert strength ($p < 0.01$) were the greatest in the HF group. Despite exhibiting lower strength than 5% HF, air abrasion may be an alternative for the surface treatment of lithium disilicate surfaces, indicating the best prognosis over time.

**Keywords**: Ceramic. Fatigue. Flexural strength. Surface properties.

## 1. Introduction

Lithium disilicate reinforced glass ceramic has a wide range of clinical indications, combining mechanical strength (Weitzel et al. 2020) and esthetics (Malchiodi et al. 2019). Recent studies have reported clinical success rates of 98.3% after 3 years (Van den Breemer et al. 2019) and 83.5% after 10 years (Rauch et al. 2018) for lithium disilicate restorations, except in cases of fixed partial dentures (FPD) in the posterior region, which exhibit a clinical survival rate of only 22% after 15 years (Becker et al. 2019). Two clinical trials have reported the loss of retention of single crowns as failure (Rauch et al. 2018; Van den Breemer et al. 2019).

The surface treatment of glass ceramics prior to cementation is performed by hydrofluoric acid (HF) etching (Gracis et al. 2015), but the concentration and etching time may negatively affect the mechanical properties of the lithium disilicate, such as a decrease in the flexural strength of CAD/CAM materials

(Kurtulmus-Yilmaz et al. 2019; Tribst et al. 2019). Prolonged acid etching time is associated with low strength (Ren and Luo 2013). For these reasons, alternative surface treatments have been suggested, such as air abrasion under low air pressure (Meness et al. 2014), etching with 1.23% acidulated phosphate fluoride (Brentel et al. 2007), and a system of simultaneous acid etching and priming, which show enhances lithium disilicate properties because of reduced damage of the material surface than HF etching (Tribst et al. 2019). In addition, alternative surface treatments aim to reduce the clinical risk of HF, as HF has extremely caustic characteristics, is harmful to soft tissues, and vaporizes rapidly (inhalation risk).

Air abrasion (AB) with alumina particles or silica-modified alumina particles is the standard surface treatment for polycrystalline ceramics (Gracis et al. 2015). This treatment modifies the ceramic surface, permitting micro retention, and in the case of silica-modified particles, impregnation of the material surface with a silica layer, which shows greater adhesion with silane and composite materials. Silica-modified alumina particles are smaller, softer, and more spherical than alumina particles, which are considered hard and sharp (Zhang et al. 2006; Özdoğan et al. 2018). Therefore, the coverage of alumina particles by silica decreases the potential for introducing defects when compared to alumina-only particles (Cadore-Rodrigues et al. 2019).

After surface treatment with acid etching or air abrasion, a bonding agent composed of silane (bonding to silica/glass) and/or phosphate monomers (MDP, bonding to metallic oxides) is applied before the resin cement. The bonding agent promotes chemical linkage between the ceramic surface and the resin cement. The resin cement will not only bond to the ceramic surface, but will also seal small surface defects, thereby enhancing the flexural strength of ceramic materials, such as lithium disilicate (Ren and Luo 2013; Barchetta et al. 2019).

*Clinically, brittle materials such as ceramics are prone to fatigue* (Zhang et al. 2013). The micro-retention promoted by the surface treatment may create crack initiation sites. These cracks may propagate in a slow and stable manner, with the application of loads below the critical value ($K_{Ic}$) (Quinn 2006), thereby characterizing the slow crack growth (SCG) process. When dealing with glass-matrix ceramics and considering the SCG process, water molecules attack the silicate/oxide bonds at the crack tip, leading to their rupture and the extension of the crack (Quinn 2006). However, when the crack reaches a certain size (critical for that specific ceramic piece), the ceramic fractures even with the application of a relatively low stress. The SCG process is represented by the n parameter (susceptibility coefficient of crack growth), which is a material constant. A high n value indicates a low crack growth susceptibility. The n parameter helps simulate clinical scenarios reproducing clinical failures (Anusavice 2012), which are important in understanding the clinical behavior of a material in the medium-to-long term. Another parameter estimated by SCG testing is D, which represents the maximum strength at a stress rate of 1 MPa/s.

Thus, this study aimed to evaluate the mechanical behavior of a lithium disilicate ceramic after surface treatment with HF etching or AB with silica-modified alumina particles in terms of flexural strength, inert strength, and SCG analysis. The null hypothesis is that different surface treatments do not influence the (1) slow crack growth, (2) flexural, and (3) inert strength of lithium disilicate.

## 2. Material and Methods

### Specimen fabrication

Partially crystallized lithium disilicate blocks (IPS e.maxCAD, LTA3/ C14, Ivoclar Vivadent, Schaan, Liechtenstein), in their lithium metasilicate phase, were sectioned with a diamond trephine drill to obtain cylinders of 12 mm diameter. The cylinders were sectioned with a diamond disc (Extec HighConcentration, Extec, Enfield, CT, EUA) into 160 discs of 1.2 ± 0.2 mm thickness, using a precision saw machine (Isomet 1000, Buehler, Plymouth, Minessota, EUA), according to ISO 6872/2013 recommendations. All disks were sequentially polished (Ecomet 250 Grinder Polisher, Buehler; LakeBluff, Illinois, USA) with #400, #800, and #1200 granulation silicon carbide sandpaper (Norton, Guarulhos, SP, Brazil) and cleaned in an ultrasonic bath with isopropyl alcohol for five minutes. The ceramic discs were then subjected to crystallization firing according to the manufacturer's instructions. Discs were divided into two main groups according to the surface treatment (5% HF etching, or AB with silica-modified alumina particles), and subjected to three different mechanical tests: biaxial flexural strength (n = 20), inert strength (n = 25), and SCG (n = 35) test.

HF was performed by acid etching with 5% hydrofluoric acid (Fórmula & Ação, São Paulo, Brazil) for 20 s, washed and subjected to ultrasonic bath for 4 min, and finally dried it with an air blower. AB was performed using 30 µm silica-modified alumina particles (Rocatec Soft, 3M-ESPE, Campinas, Brazil) with an air abrasion device, under 2.8 bar pressure, at 10 mm distance for 15 s (Cristoforides et al. 2012).

Five randomly chosen samples from each surface treatment group had their roughness measured in three parallel lines (ʎc 0.25 mm) by a surface roughness tester (Surftest SJ 310, Mitutoyo). The values obtained for each sample (Ra) were recorded, and statistical analysis was performed using the t-test ($\alpha$ = 0.05).

After surface treatment, all sample surfaces were treated with a silane bonding agent (Monobond S, Ivoclar Vivadent, Schaan, Liechtenstein) for 60 s, followed by thermal treatment at 100°C for 2 min (F-1800 furnace, EDG, São Paulo, Brazil). A layer of dual-cure resin cement (Panavia F, Kuraray Medical Inc., Okayama, Japan) was applied to the disc surface, covered with a polyester strip, and a load of 750 g was applied to uniformly distribute the cement throughout the ceramic surface. After removal of the excess cement, the material was light-cured for 40 s on the surface of the ceramic and an additional 40 s on each side of the adhesive interface (Radii-Cal LED, SDI [North America]) at an intensity of 1200 mW/cm$^2$. The polyester strip was removed, and the samples were stored in distilled water at 37°C for 7 days.

**Mechanical Tests**

Tests were performed using a universal testing machine (EMIC DL-1000, Instron, Brazil). To determine the flexural strength of each group (HF or AB), the biaxial flexural test was performed according to ISO 6872:2013 (piston-on-three-ball design). The surfaces of the ceramic discs covered with resin cement were placed facing down on the testing device. Discs (n = 20) were immersed in distilled water and their centers were loaded with a flat piston (3 mm radius) at a crosshead speed of 1 mm/min until fracture. The maximum load reached (N) was recorded, and the flexural strength was calculated as:

$$\sigma=-0.2387P\frac{(X-Y)}{b^2}$$

where P is the maximum load at fracture (N), X and Y are parameters related to the elastic properties of the material (Poisson's ratio and elastic modulus), and b is the specimen thickness at the fracture origin (mm). Data were evaluated using Student's t-test ($\alpha$ = 0.05).

A constant stress rate test was performed to determine the SCG parameters *n* and D. The flexural strength test design described earlier was used and the samples were immersed in water; the load was applied at five stress rates: $10^{-2}$ (n = 10), $10^{-1}$ (n = 5), $10^{0}$ (n = 5), $10^{1}$ (n = 5), and $10^{2}$ (n=10) MPa/s. *n* was calculated by linear regression analysis according to the equation (ASTM 1368–00):

$$\log\sigma_f=\frac{1}{n+1}\log\dot{\sigma}+\log D$$

where $\sigma_f$ is the mean strength (MPa), and $\dot{\sigma}$ is the stress rate (MPa/s). At the lowest stress rate, pre-loading was performed with 50% of the flexural strength to reduce the testing time. Inert strength (n = 25) was measured with samples immersed in an inert environment (mineral oil) at a stress rate of $10^2$ MPa/s. Data were evaluated using Student's t-test ($\alpha$ = 0.05). The results from both inert strength and SCG testing were subjected to Weibull statistical analysis using the maximum likelihood method.

**Fractographic analysis**

All tested samples underwent fractographic analysis using a stereo microscope (Discovery V20, Carl Zeiss Microscopy) to determine the origin of the failure. Representative specimens were sputter-coated with gold and evaluated using scanning electron microscopy (SEM; Inspect S50, FEI) to examine the fractographic surface.

## 3. Results

The values obtained in the mechanical tests are listed in Table 1. The surface roughness was highest for the AB group. The flexural strength and inert strength were the highest for the HF-treated samples, which is the characteristic strength for inert strength evaluation. However, the SCG parameter ***n*** as well as the reliability (*m* value) in the inert strength test were the highest for the AB samples. The parameter **D** was the highest for the HF samples.

**Table 1.** Results obtained in mechanical tests according to the surface treatment performed.

| Test performed | Data obtained | Surface treatment | | p-value |
|---|---|---|---|---|
| | | HF | AB | |
| Ra Roughness | µm | 0.23 (0.03) | 0.57 (0.19) | < 0.01 |
| Flexural strength | Mean (SD) (MPa) | 279.44 (31.42) | 257.53 (21.22) | 0.03 |
| Inert strength | Mean (SD) (MPa) | 343.30 (47.93) | 235.70 (28.63) | < 0.001 |
| | Reliability (*m*-value) | 8.48 | 10.46 | |
| | $\sigma_c$ (MPa)* | 362.72 | 246.72 | |
| | 95% CI | 345.38–380.93 | 236.86–256.99 | |
| SCG testing | *n* parameter | 15 (1.5) | 18 (1.7) | |
| | D parameter | 222.88 (0.02) | 205.38 (0.02) | |

*Statistical difference is given with no overlapping of respective confidence intervals; SCG, slow crack growth, HF, hydrofluoric acid, AB, air abrasion; ***n*** parameter: susceptibility coefficient to subcritical crack growth; **D** parameter: subcritical crack growth parameter

Figure 1 shows data distribution according to Weibull analysis for inert strength data. Figure 2 represents the mean strength at each stress rate and also the mean inert strength. Steeper regression lines demonstrate higher susceptibility to SCG. Failure origins were all located at the tensile side of the tested samples (Figure 3).

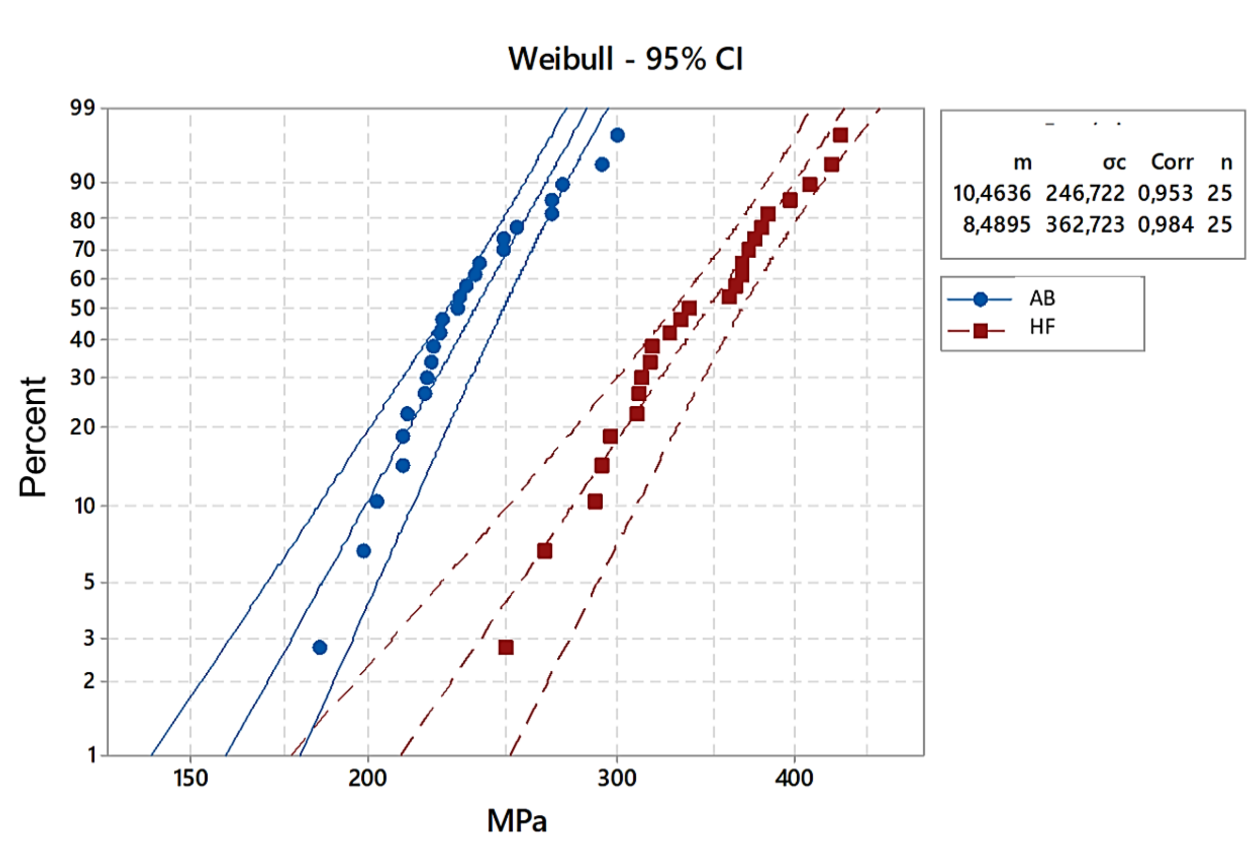
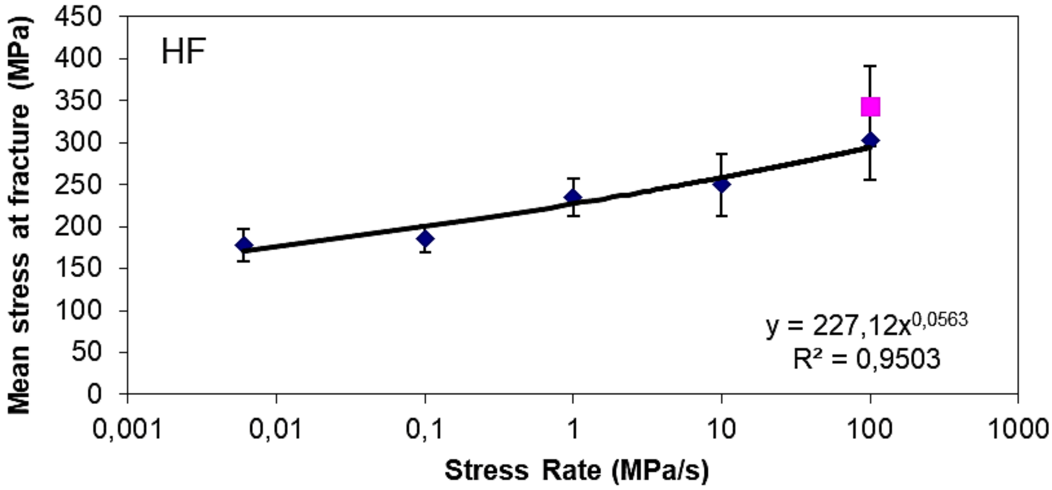



**Figure 1.** Representative graphic of Weibull analysis – the inclination of the main graphical line represents the reliability (*m* value) for each group.



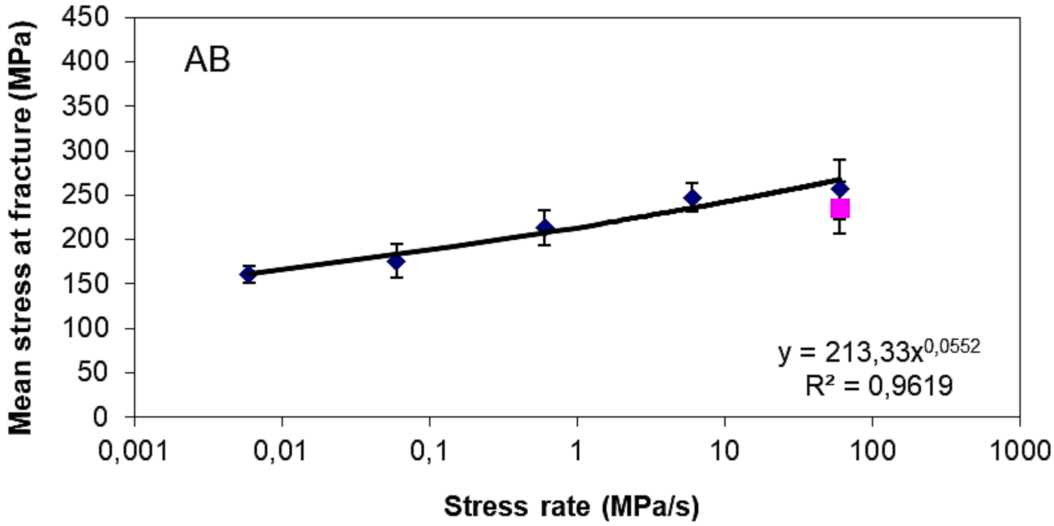




**Figure 2.** Mean strength at each stress rate and regression lines and mean inert strength of each surface treatment evaluated.

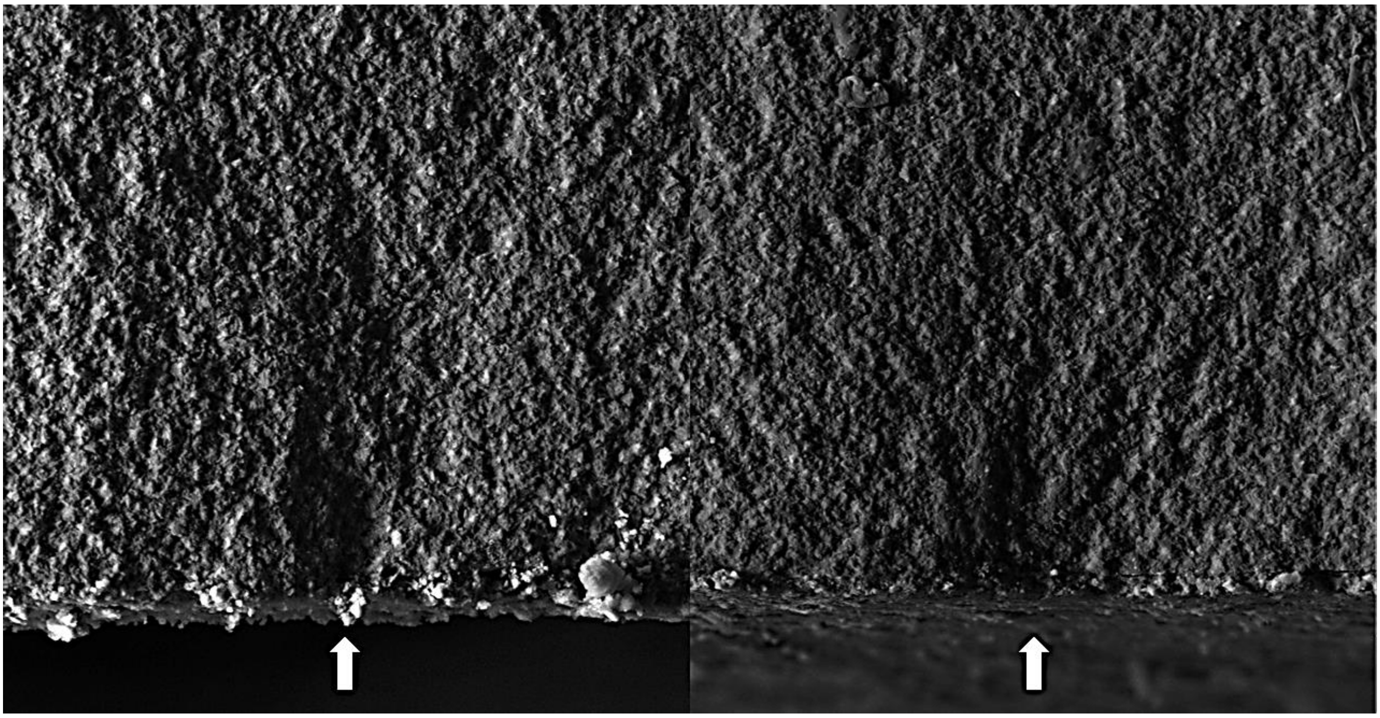


**Figure 3.** SEM evaluation of fractured surfaces from the HF-treated sample (left) and AB-treated sample (right) (×1000 magnification). SEM, scanning electron microscopy; HF, hydrofluoric acid; AB, air abrasion.

## 4. Discussion

The present study evaluated AB with silica-modified alumina particles as an alternative treatment to HF etching for the cementation of lithium disilicate ceramic. HF-treated lithium disilicate presented higher values for flexural strength, inert strength, and respective characteristic strength; thus, the second and third hypotheses were rejected. However, the AB lithium disilicate presented favorable results for reliability and slow crack growth parameter *n*, rejecting the first null hypothesis.

Lithium disilicate is a high-strength ceramic compared to other glass ceramics (Ramos et al. 2016; Weitzel et al. 2020) and has excellent clinical survival rates (Rauch et al. 2018; Van den Breemer et al. 2019). *In vitro* studies showed that, when compared to other CAD/CAM materials (feldspathic ceramic, polymer infiltrated ceramic, and zirconium reinforced lithium silicate), besides exhibiting the highest strength, lithium disilicate had the lowest *n* values and consequently the highest SCG susceptibility (Ramos et al. 2016). They also presented the worst fatigue behavior with respect to crack formation on the cementation surface compared to feldspathic ceramic (Weitzel et al. 2020), probably resulting in a high clinical failure rate of posterior fixed partial dentures (Becker et al. 2019).

The basic mechanical properties of dental materials can be described well using the flexural strength tests. The inert strength may also be a reference of strength, ignoring the effects of water on strength, representing the effect of preexisting defects in the material. The strength was the lowest for AB-treated lithium disilicate, showing that the surface defects created by AB may be less favorable than HF defects. HF etching after AB led to a reduction in surface mean roughness in a previous study (Uwalaka et al. 2018), possibly indicating gentle surface modification, since flexural strength in ceramic is highly dependent on the surface characteristics (Barchetta et al. 2019). However, strength data have a limitation in predicting clinical performance, since clinical failures occur after repeated occlusal loading (Homaei et al. 2016), following the crack growth under loads below the fracture strength (Kelly et al. 2017).

Low ***n*** values presented by HF surface-treated lithium disilicate may represent the degradation of the ceramic surface by acid etching, favoring SCG. Lithium disilicate has already been reported to be highly susceptible to SCG (Ramos et al. 2016), but the increase in susceptibility caused by acid etching has never been discussed. Without a cement layer, the effects of acid etching are also reported for flexural strength (Barchetta et al. 2019). HF attacks the interface between crystals and the glassy matrix phase, removing the glass matrix and creating irregularities in the lithium disilicate crystals, described as a hostile effect on the ceramic structure (Hooshmand et al. 2008). The resin cement provides bonding to the tooth structure, but also conceals the defects on the ceramic surface (Barchetta et al. 2019), thereby decreasing crack length and blunting of the crack tip (Öztürk et al. 2012).

The resin cement application proposed in the literature (Addison and Fleming 2008) is that the resin fills the surface flaws and contracts during load application, increasing the stiffness of the resin; then, the resin begins to behave like the ceramic, and owing to the optimal interface produced between the resin and glass ceramic, there is a strengthening of the assembly. Without the cement layer, it is possible that the effects caused by HF were deeper than those caused by AB. Due to the fact that strength relies more on surface characteristics, it was not affected by HF, but ***n*** values were, due to a more harmful growth of defects than in AB samples.

Currently, several studies (Prochnow et al. 2018; Veríssimo et al. 2019) showed that the 5% and 10% HF etching demonstrate a similar bond strength to lithium disilicate ceramic, however, in case of application accidents, the 5% is safer to the human tissues than 10%. Regardless of concentration, it is known that adhesive cementation tends to attenuate the weakness caused by HF etching (Miranda et al. 2020), but when Levartovsky et al. (2021) analyzed the possibility of adhesive failure, they concluded that this occur more frequently to 10% HF etching, when compared to 5% HF samples.

Although AB led to a decrease in strength, it increased reliability (*m* value) and decreased SCG susceptibility (*n* value) (Table 1). From a clinical standpoint, both parameters may provide a better estimation of survival, since the *m* value represents the structural reliability of the material with greater precision of strength values (Weibull 1939), and the *n* value may indicate susceptibility to clinical failures (Anusavice 2012). Thus, AB with silica-modified alumina particles promoted the most reliable strength results for lithium disilicate and the lowest risk of fracture under stresses below the critical value, and SCG occurs before the fast fracture. Under repeated and low load levels, lithium disilicate covered by cement after air abrasion as a surface treatment may present better results.

The best performance of AB in the ***n*** parameter and reliability may be associated with the highest roughness values found for the AB group (Table 1), as also reported by other studies (Dilber et al. 2012; Sudré et al. 2020). It is known that the micromechanical retention created by a surface treatment can increase the retention, favoring adhesion and the final ceramic strength (Gundogdu and Aladag 2017; Miranda et al. 2020). Therefore, in our study, the highest roughness values presented by AB may have increased the micromechanical retention between the ceramic and resin cement, decreasing the slow crack growth parameter for this group. Since this is an *in vitro* study, these affirmations are just speculations based on the results obtained. Well-conducted clinical trials will provide more reliable results.

Other factors, such as luting agents, may also influence the chipping and fracture of lithium disilicate veneers under accelerated fatigue and load-to-failure tests (Gresnigt et al. 2017), in addition to the previously mentioned before-concentration and etching time (Ren and Luo 2013; Kurtulmus-Yilmaz et al. 2019; Tribst et al. 2019). These reports, in addition to the results of the present study, demonstrate how the cementation process affects the mechanical properties of lithium disilicate (Ren and Luo 2013; Barchetta et al. 2019), and such factors must be clinically considered, especially for high-load areas and multiple FPDs.

## 5. Conclusions

Air abrasion with 30 µm silica-modified alumina particles promoted lower values of strength in different tests when compared to 5% hydrofluoric acid etching for the surface treatment of lithium disilicate ceramic. However, air abrasion promoted the highest values of structural reliability and the lowest slow crack growth susceptibility, which could indicate the best prognosis over time.

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**Ethics Approval:** Not applicable.

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