

Dissolution Depth and Surface Morphological Alterations in Ultrathin Glass Ceramic Etched with Different Hydrofluoric Acid-etching Protocols

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Purpose: To evaluate the effect of different HF-etching protocols on the dissolution depth and micromorphology of the etched and adjacent surfaces of ultrathin glass-ceramic specimens.

Materials and Methods: One hundred twenty specimens (6 x 6 x 0.3 mm) of leucite-reinforced glass-ceramic (LEU, IPS Empress, Ivoclar Vivadent) and lithium-disilicate-reinforced glass-ceramic (LD, IPS e.max, Ivoclar Vivadent) were prepared. Specimens were divided into 5 groups (n = 12) according to etching protocol: G1: control, untreated; G2: 5% hydrofluoric acid (HF) etching for 20 s (HF5%20s); G3: HF5%60s; G4: HF10%20s; and G5: HF10%60s. To analyze the dissolution depth, specimens were sectioned into two similarly sized halves using a chisel to create an internal surface (IS). Specimens were analyzed with scanning electron microscopy (SEM) on the following surfaces: HF application surface (AS), lateral surface (LS), internal surface (IS), and the surface opposite to the AS (OS). Dissolution patterns were identified. Data were submitted to one-way ANOVA and Bonferroni's test ($\alpha = 0.05$). Dissolution depth data were submitted to Kruskal-Wallis and Mann-Whitney U-tests ($\alpha = 0.05$). The prevalence of different dissolution patterns was analyzed using SEM.

Results: HF gel applied on the AS also affected the adjacent surfaces of all specimens. Different dissolution patterns were observed, which were dependent of HF-etching protocol and proportion of the glass phase in the ceramic. These patterns were categorized into four types for LEU (I-IV) and three for LD (I-III) according to the micropore size. The greater the micropore size, the more pronounced the etching pattern ($p < 0.001$). Higher HF times and concentrations showed prevalence of more severe etching patterns. HF10%60s produced greater dissolution depth in both materials when compared with other HF-etching groups ($p < 0.05$).

Conclusion: Hydrofluoric acid etching not only affects the surface upon which it is applied, but internal, lateral and even opposite edges of glass ceramic. Different dissolution patterns and depths can be formed which are dependent of hydrofluoric acid concentration, application time, and proportion of the glass phase in the ceramic.

Keywords: ceramics, hydrofluoric acid, acid etching, etching deepness, scanning electron microscopy, surface treatments.

*J Adhes Dent 2021; 23: 579–587.
doi: 10.3290/j.jad.b2287769*

Submitted for publication: 10.09.20; accepted for publication: 02.08.21

Hydrofluoric acid (HF) etching of glass-ceramic surfaces is essential for the micromechanical retention of composite cement.^{11,12} HF dissolves the glass phase of the ceramic by reacting with silicon dioxide, creating a micro-

porous structure that provides adhesive/composite cement retention and increases the ceramic surface energy.^{12,18} It is well known that the greater its concentration and the longer the application time, the rougher, deeper, and larger the glass-ceramic dissolution area will be.^{19,21}

The literature provides no consensus on the morphology of glass-ceramic dissolution, which has been variably described as “greater vitreous phase dissolution” or “slightly greater dissolution”²¹ or “weaker glassy phase”.¹⁶ Authors report different dissolution patterns with distinct degrees of glass-ceramic dissolution; however, no description of such patterns is provided.²¹ Other studies described glass-ceramic dissolution in feldspathic ceramics as having a honeycomb-like appearance and scratch-like gaps for lithium-disilicate glass-ceramic, without correlating such descriptions with a particular HF-etching protocol.^{1,4,7,16}

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Table 1 Materials used and their composition

Material/Manufacturer	Type/Lot No.	Composition
IPS Empress CAD (LEU) / Ivoclar Vivadent	Leucite-based glass-ceramic Lot V01131	SiO ₂ (60–65wt%), Al ₂ O ₃ (16–20wt%), K ₂ O (10–14wt%), Na ₂ O (3.5–6.5wt%), other oxides (0.5–7wt%), pigments (0.2–1wt%) Leucite crystal: KAlSi ₂ O ₆ (35–45vol%)
IPS e.max CAD (LD) / Ivoclar Vivadent	Lithium-disilicate-reinforced glass-ceramic Lot V13056	SiO ₂ (57–80wt%), Li ₂ O (11–19wt%), K ₂ O (0–13wt%), P ₂ O ₅ (0–11wt%), ZrO ₂ (0–8wt%), ZnO (0–8wt%), Al ₂ O ₃ (0–5wt%) MgO (0–5wt%), coloring oxides (0–8wt%) Lithium-disilicate glass-ceramic crystal: Li ₂ Si ₂ O ₅ (70 vol%)
Condac Porcelana, FGM	5% hydrofluoric acid	Hydrofluoric acid 5%, water, thickener, surfactant and colorant
Condac Porcelana, FGM	10% hydrofluoric acid	Hydrofluoric acid 10%, water, thickener, surfactant and colorant
Data were taken from material safety data sheets and/or technical data information provided by manufacturers.		

No study was found to investigate glass-ceramic dissolution in all the surfaces of the specimen. Only two studies were found that evaluated a surface (the internal one) other than that to which HF was applied; however, their results showed discrepancies.^{14,16} The authors reported the dissolution to be as deep as 0.6 mm¹⁴ and as shallow as 5 to 7 μm.¹⁶ Considering that in minimally invasive dentistry ultrathin restorations range from 0.3 to 0.5 mm in thickness,^{9,10} the HF might affect the integrity of these restorations. Therefore, the aim of this study was to evaluate the effect of different HF-etching protocols on the dissolution depth and micromorphology of the etched and adjacent surfaces of ultrathin glass-ceramic specimens. The experimental hypothesis was that variations in HF-etching protocols might influence the dissolution depth and micromorphology of the etched and adjacent surfaces of the glass-ceramics tested.

MATERIALS AND METHODS

Specimen Preparation

Specimens consist of two CAD/CAM materials (Table 1): leucite-reinforced glass-ceramic ([LEU] IPS Empress CAD, Ivoclar Vivadent; Schaan, Liechtenstein) and lithium-disilicate glass-ceramic ([LD] IPS e.max CAD, Ivoclar Vivadent). One hundred twenty square-shaped specimens (6 x 6 x 0.3 ± 0.05 mm) were prepared using a diamond saw (Isomet 1000, Buehler; Lake Bluff, IL, USA) at 100 rpm (weight: 100 g). All the specimens' surfaces were polished using 600-, 1200-, and 2000-grit silicon-carbide (SiC) abrasive paper (Norton AS; São Paulo, Brazil) in an automatic polisher (APL4, Arotec; Cotia, SP, Brazil) to standardize the thickness and surface of the specimens. Specimens were then ultrasonically cleaned for 10 min. The LD specimens were crystallized according to manufacturer's instructions. Specimens (LD and LEU) were assigned to 5 groups (n = 12) according to previously-reported etching protocols: G1: control (C), no treatment; G2: 5% hydrofluoric acid (HF) etching for 20 s (HF5%20s);^{14,15,23} G3: 5% HF etching for

60 s (HF5%60s);^{14,23} G4: 10% HF etching for 20 s (HF10%20s);^{4,14,15,23} G5: 10% HF etching for 60 s (HF10%60s).^{4,14,15,23}

After etching, specimens were rinsed with compressed air-water spray for 60 s and ultrasonically cleaned in distilled water for 10 min. The specimens were then sectioned approximately into halves (Fig 1) using a chisel placed on the surface opposite the etched one, a procedure that allowed measuring the dissolution depth (HF penetration through the etched surface) on the internal surface of each specimen. Before sectioning, for later tracking of the etched surface, a mark was made on each of the two halves (surface opposite the etched one) using a diamond bur. After sectioning, the surfaces were ultrasonically cleaned, dried for 30 s, and examined using scanning electron microscopy (SEM, JSM 5600 LV, JEOL; Tokyo, Japan). These surfaces were referred to as the application surface (AS: the surface to which HF acid was applied), the internal surface (IS: the surface created by sectioning), the lateral surface (LS: opposite IS); and opposite surface (OS: opposite AS) (Fig 1).

Surface Dissolution Analysis — SEM

The sectioned specimens were placed onto aluminum stubs, sputter-coated with gold/palladium (SCD 050; Balzer; Schaan, Liechtenstein), and then analyzed using an SEM operating at 15 kV at a working distance of 15 mm, involving different magnifications (from 330X to 5000X). For LS and IS, image scanning was from top (AS) to bottom (OS). For AS and OS, image scanning started from the surface opposite to the sectioned one.

Image analyses of each material revealed different dissolution patterns (LEU-I, LEU-II, LEU-III, LEU-IV and LD-I, LD-II, LD-III) in ascending order of the micropore size in μm², which was measured using a thresholding tool (adjust → threshold, analyze → analyze particles) of FIJI software (ImageJ, NIH; Bethesda, MD, USA). Images of 330X magnification were also submitted to a roughness/pseudo-color mode tool which distinguishes rougher regions (warm colors) from smoother regions (cold colors).

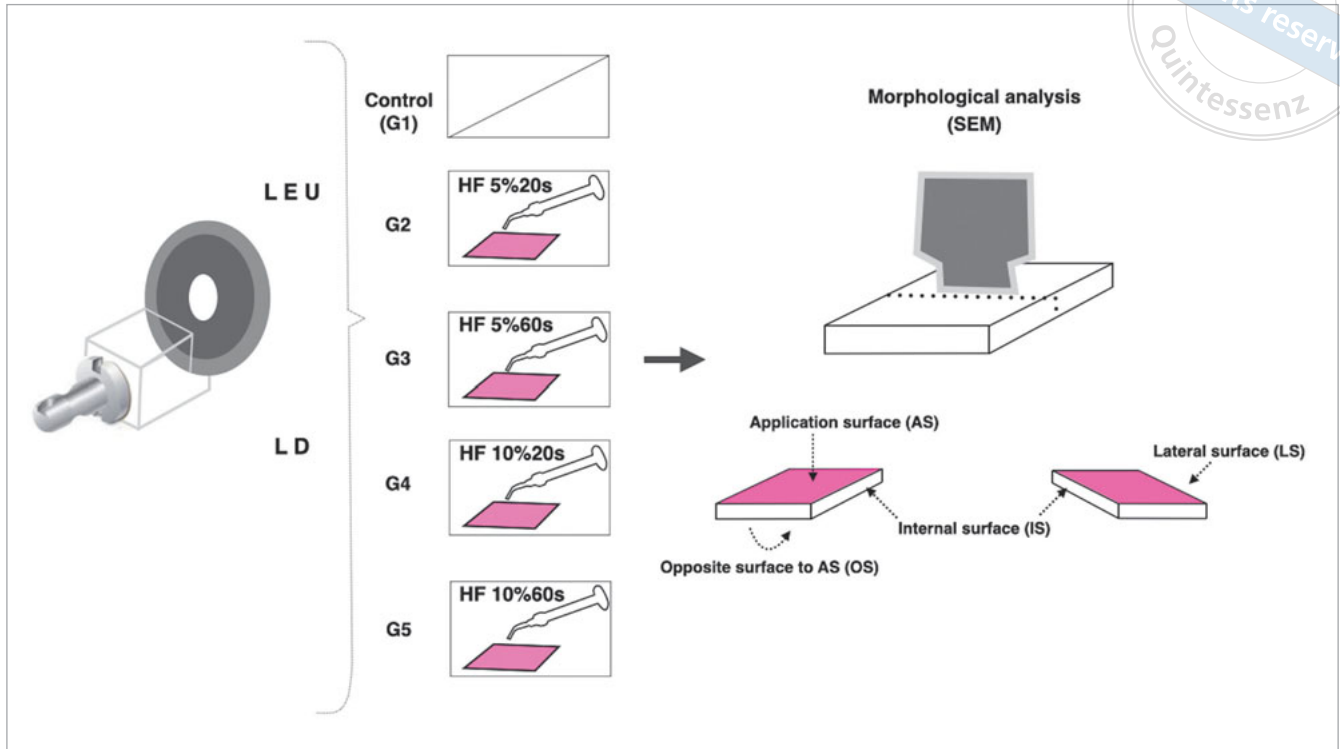


Fig 1 Schematic representation of the study set-up. LEU: leucite glass-ceramic; LD: lithium-disilicate glass-ceramic; HF: hydrofluoric acid.

Dissolution Extension Analysis – LS

Dissolution was measured by a single examiner using an SEM measurement tool and expressed in μm . The dissolution extension on the LS of each specimen was identified at 2000X and categorized according to the pattern (Fig 2). The prevalence of pattern types for each HF group was recorded.

Dissolution Depth Analysis – IS

Five dissolution regions per specimen were selected in the IS image, and the depth and width of the dissolution were measured. A mean value concerning both variables was calculated for each specimen. The measurement baseline, as illustrated in Fig 3, was the widest and deepest boundary of the dissolution region.

Statistical Analysis

Data concerning the micropore size (patterns) were tested for normality and homoscedasticity using the Kolmogorov-Smirnov and Levene tests, respectively ($\alpha = 0.05$). The normally distributed data were then submitted to one-way ANOVA, followed by a post-hoc Bonferroni test ($\alpha = 0.05$).

Data regarding IS were not normally distributed (Kolmogorov-Smirnov and Levene tests) and thus were submitted to non-parametric statistical analysis using the Kruskal-Wallis test followed by a pairwise Mann-Whitney U-test ($\alpha = 0.05$).

To define the reliability of the dissolution measurements, an intraclass correlation coefficient (ICC) was calculated, resulting in a reliability index of the correlation and agreement between measurements.¹⁷ To calculate the ICC, two-way mixed effects were applied, considering the mean of k measurements and an absolute agreement definition. SPSS v 25 software (IBM SPSS; Armonk, NY, USA) was used for all statistical analyses.

RESULTS

Surface Dissolution Analysis – SEM

Figure 2 illustrates different morphological characteristics for LEU and LD, including different dissolution patterns. SEM images revealed surfaces with gradually increasing mean micropore size: 0.2 (0.1) μm^2 (LEU-I); 1.1 (0.3) μm^2 (LEU-II); 3.1 (1.4) μm^2 (LEU-III); and 4.9 (1.3) μm^2 (LEU-IV). The images concerning patterns II, III, and IV showed interconnected micropores, with those in pattern III resembling a cauliflower. Pattern IV images revealed a surface full of defects, resembling a honeycomb.

With regard to LD, SEM revealed significantly different mean micropore sizes: 0.1 (0.04) μm^2 (LD-I); 0.8 (0.3) μm^2 (LD-II); and 1.6 (0.6) μm^2 (LD-III). Exposed LD crystals exhibited patterns II (resembling a bush) and III (sponge-like),

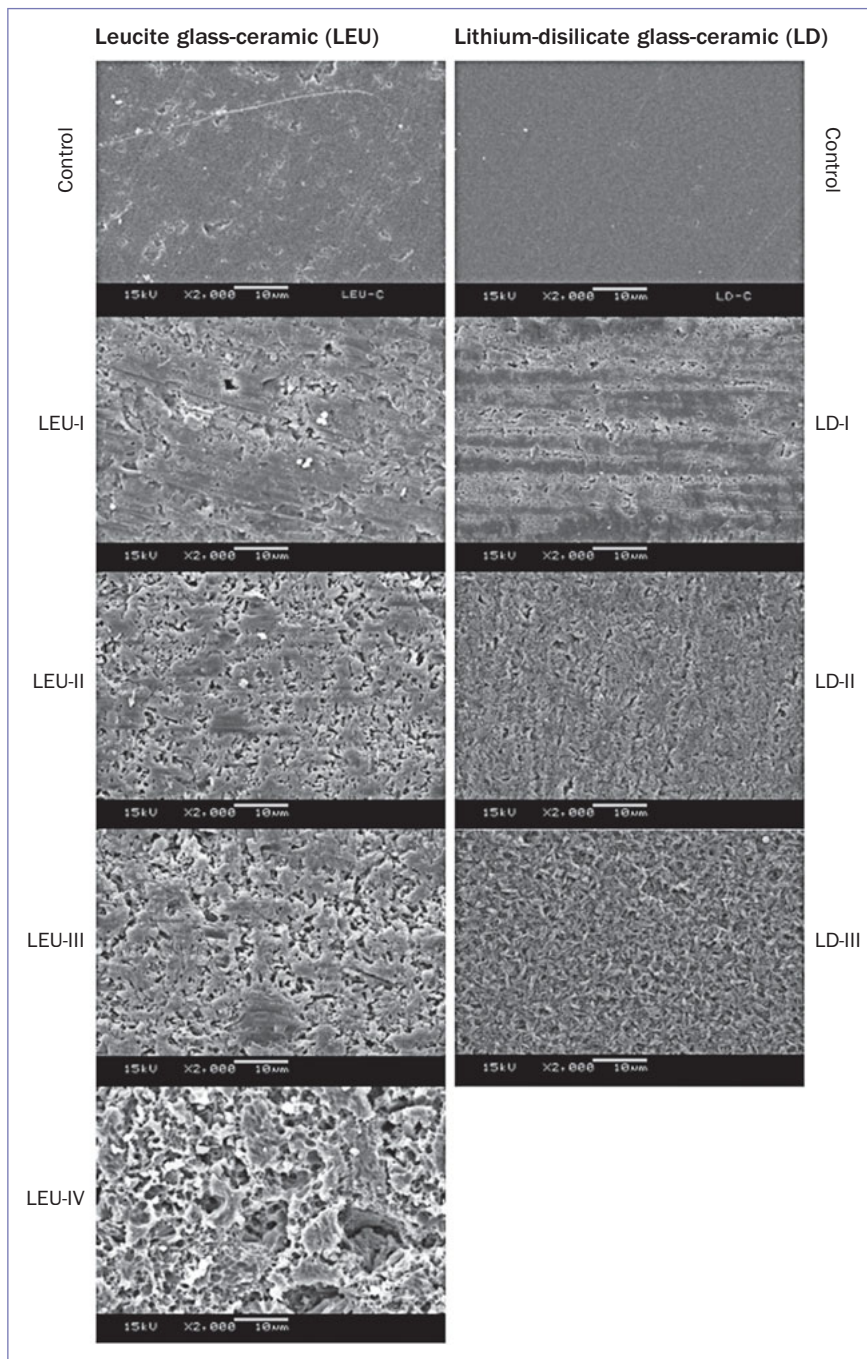


Fig 2 Hydrofluoric acid dissolution patterns according to glass content of the ceramic.

being more pronounced in the latter. The factor micropore size was significantly different ($p < 0.001$) between the patterns for both materials (Fig 6). Figures 4 (LEU) and 5 (LD) show the dissolution morphology of AS, LS, and IS, considering all the HF etching groups and the control.

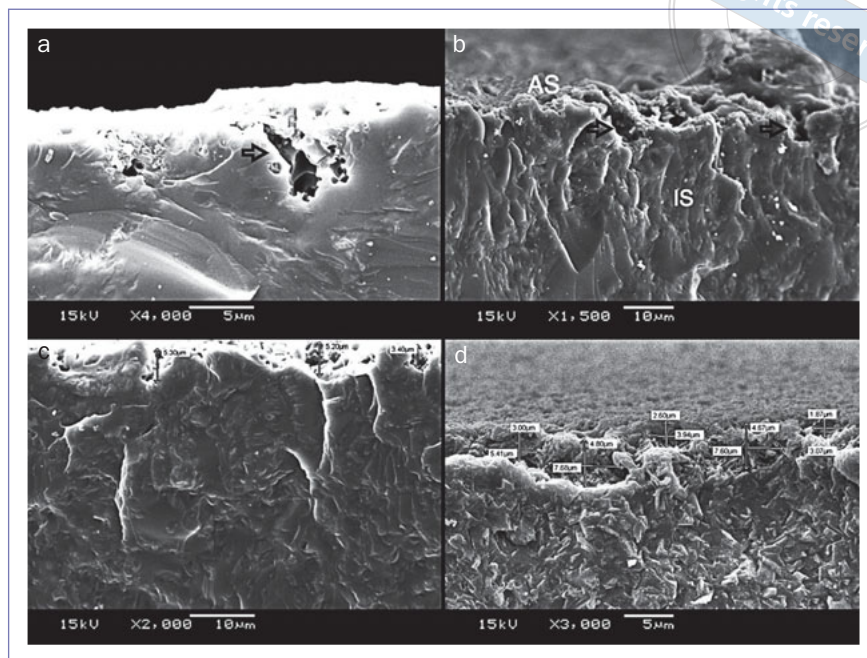
Dissolution Extension Analysis – LS

SEM analysis (2000X magnification) showed that the entire expanse (300 µm) of LS was etched, concerning both ce-

ramics and all etching protocols. OS was also affected by all HF-etching protocols (Fig 7), albeit only at its edges. The pattern LEU-IV was observed in all AS groups. In terms of AS, the pattern LD-III was observed for all HF-etching protocols except for HF5%20s, which showed only LD-II.

Different dissolution patterns were detected on the LS of both ceramics. Figure 8 shows the prevalence of all patterns according to each ceramic and HF-etching protocol. LEU-IV at HF10%60s was the most prevalent (58%) among

Fig 3 LEU internal dissolution regions. a. IS showing a region of dissolution (arrow) (4000X); b. sectioned specimen showing the AS, and IS with presence of dissolution (arrows) (1500X); c. LEU dissolution depths of a section of the specimen (2000X); d. LD dissolution depth and width (3000X). AS: application surface; IS: internal surface; LEU: leucite glass-ceramic; LD: lithium-disilicate glass-ceramic.



the groups. As the analysis moved from the top to the bottom of LS (Figs 4 and 5), the dissolution severity decreased according to the HF concentration and application time (Fig 8) for both ceramics.

Dissolution Depth Analysis – IS

The ICC value obtained for the IS was 0.987 (95% confidence interval 0.967–0.994), indicating excellent reliability in measuring dissolution in terms of micropores. Statistical analysis showed that the factor “HF protocol” significantly ($p < 0.05$) influenced the depth and width of the dissolution on IS for both ceramics (Table 2). HF10%60s showed the largest dissolution (depth and width) among the groups for both ceramics. HF5%20s, HF5%60s and HF10%20s showed no statistically significant difference between depth and width for either ceramic. HF10%60s showed a mean depth of 6.2 μm and a mean width of 9.6 μm for LEU, and 2.0 μm (depth) and 1.1 μm (width) for LD, revealing values greater than those obtained for the other LD-HF protocols. Widths were always greater than depths for both ceramics ($p < 0.05$).

DISCUSSION

This study showed that HF applied to glass ceramics can produce different dissolution patterns, which vary according to the concentration and application time of HF and the proportion of glass phase of the ceramic. Our hypothesis must be accepted, as the HF-etching protocols affected not only the application surface, but all of the other surfaces tested as well.

The LS showed a 300- μm extension of dissolution, which agreed with a previous study;¹⁴ in which the authors even protected and isolated the lateral surfaces with teflon tape to prevent the HF from flowing onto such surfaces. The authors assumed that the tape and the viscosity of HF would be enough to prevent it from flowing onto the lateral surfaces. To verify that, we used insulating tape to protect the lateral surfaces prior to HF application, after which the specimens were sectioned and analyzed. SEM images showed that the dissolution values of the lateral surface were much higher than those of the internal surface, indicating that the isolation procedures might have been ineffective in preventing dissolution in those surfaces.

In the present study, no protection was used because it is not applied in clinical settings. The lateral surfaces of all specimens were etched, considering all etching protocols. Two physicochemical phenomena might account for such HF overflow. First, HF gel has thixotropic and pseudoplastic behaviors; once it is applied, it loses its viscosity and starts flowing at a speed that depends on shear rate, pressure, temperature, pH, and electric field.^{3,13,22} As the HF gel is pushed out of the syringe, it receives a shear stress, and its temperature rises, its pressure drops, its viscosity decreases, and it starts to flow.³ Second, HF interacts with silica in two stages: hydration of silica surface and reaction between the hydrated silica and HF. When HF dissociates into fluoride (F^-) and hydronium (H_3O^+), hydrogen atoms will bond to the non-bridging oxygen of the silica (O-Si-O), creating a surface layer of hydroxyls, which can be replaced by fluoride ions to form $\text{SiF}_4 + 2\text{H}_2\text{O}$.^{13,20,22} Besides the dissolution of the glass phase of the ceramic, a capillary effect – possibly caused by the same intermolecular forces be-

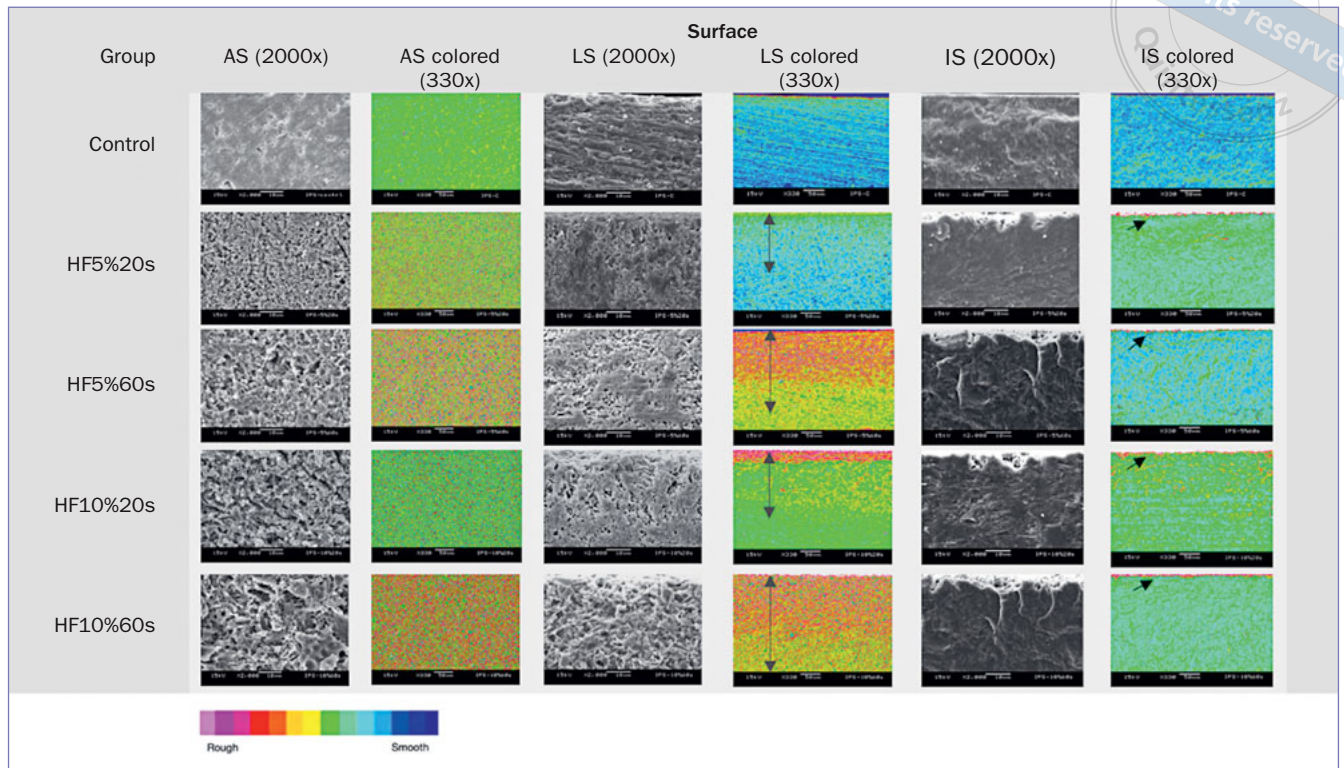
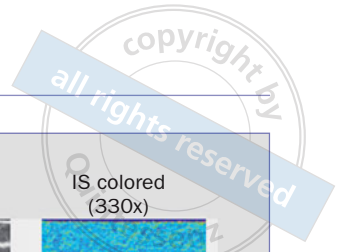


Fig 4 AS, LS, and IS morphology (2000X) of all LEU groups and its corresponding colored profile (330X). LS and IS (330X) show the lateral and internal surface, respectively. Arrows indicate lateral-surface and internal-surface dissolution.

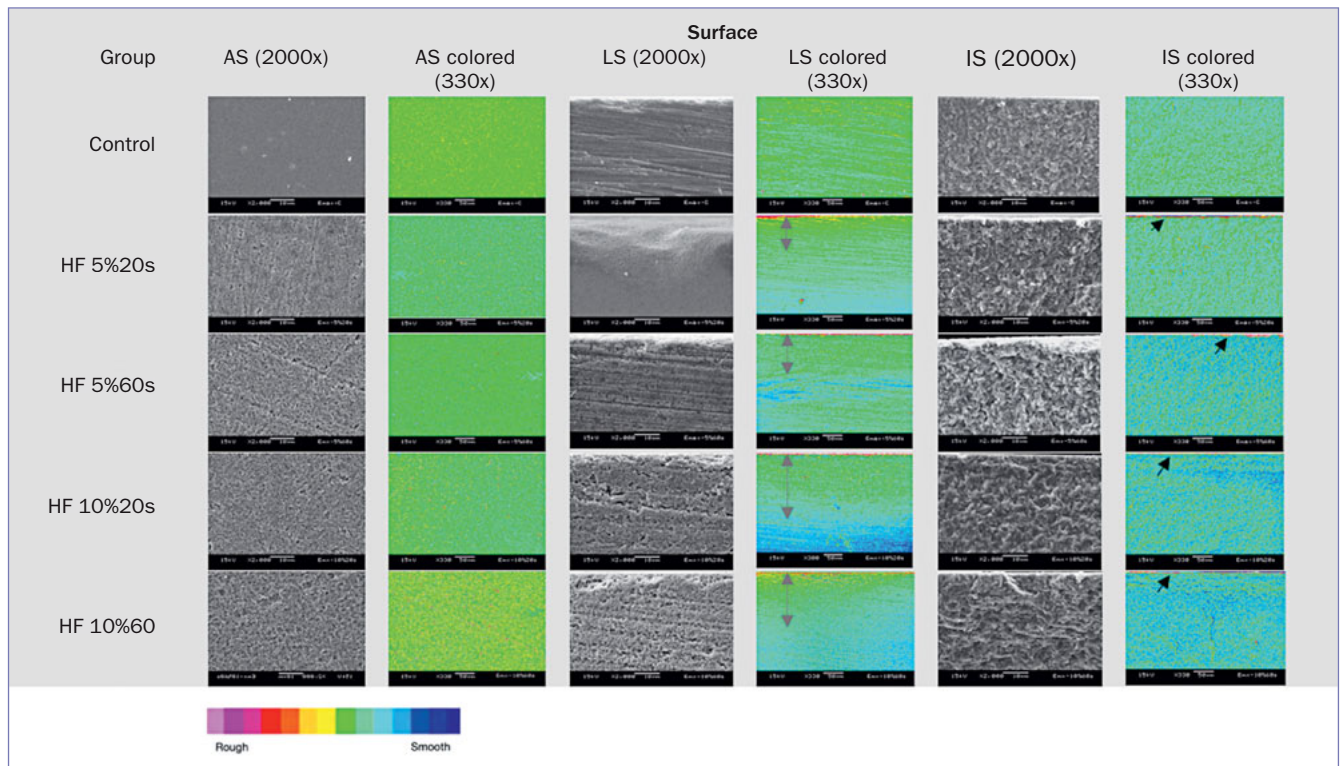


Fig 5 AS, LS, and IS morphology (2000X) of all LD groups and its corresponding colored profile (330X). LS and IS (330X) show the lateral and internal surface, respectively. Arrows indicate lateral- and internal-surface dissolution.

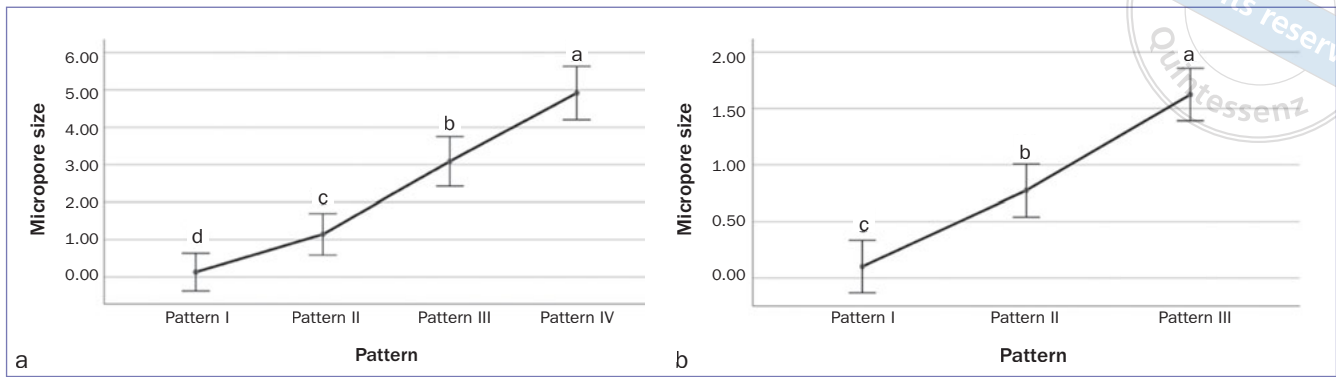


Fig 6 Different letters indicate statistical difference in micropore size in µm between patterns (p < 0.001). a. LEU: leucite glass-ceramic; b. LD: lithium-disilicate glass-ceramic.

Fig 7 Images of the OS (5000X) treated with HF10%60s, showing the etched edges of a. LEU (leucite glass-ceramic), and b. LD (lithium-disilicate glass-ceramic). Arrows indicate zones with evident dissolution. Circles show areas of partial crystal exposure.

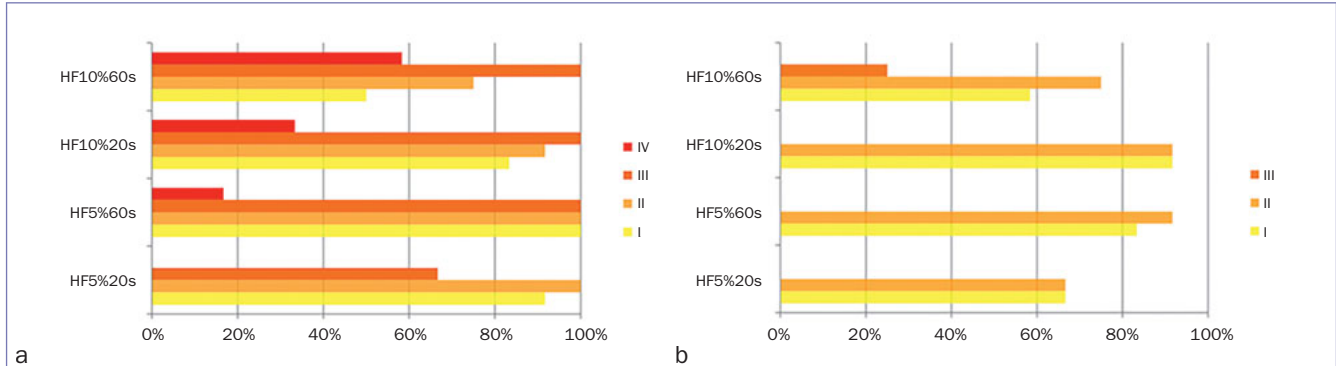
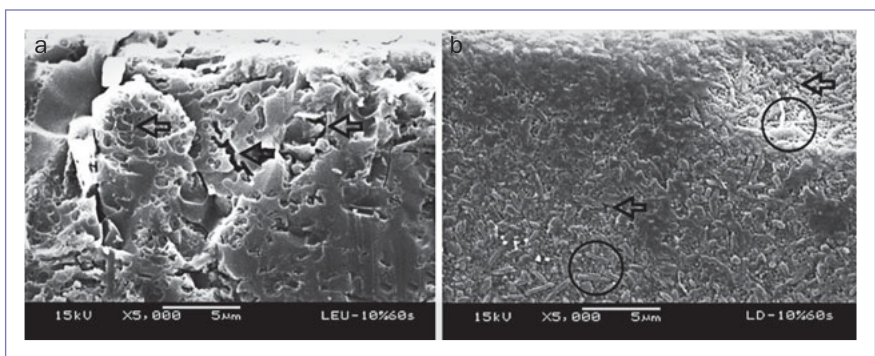


Fig 8 Dissolution pattern prevalence among the HF etching groups. LS. a. LEU (I-IV); b. LD (I-III).

tween HF and silica – might occur during this reaction, making the HF gel flow over the specimen’s lateral surfaces, as observed in the present study (Figs 4 and 5).

The SEM images regarding LS showed four different dissolution patterns for LEU and three for LD, with the patterns revealing statistically different micropore sizes (Fig 6). HF10%60s, HF10%20s, and HF5%60s groups showed LEU-IV

at the top portion of LS, followed by patterns LEU-III, II, and I, from the middle to bottom. In contrast, HF5%20s revealed only patterns LEU-III, II, and I. Regarding LD, which has a lower glass-phase content than LEU, patterns LD-III, II, and I were observed for HF10%60s, and patterns LD-II and I for the other HF protocols (Fig 8).

Table 2 Mean depth and width dissolution values on the IS (internal surface) followed by standard deviations (in parenthesis) of ceramics treated with different HF-etching protocols

Ceramic	HF protocol	Depth (μm)	Width (μm)
Leucite (LEU)	HF5%20s	3.72 (1.89) ^{Bb}	4.32 (1.7) ^{Ba}
	HF5%60s	3.63 (1.7) ^{Ba}	4.14 (1.9) ^{Ba}
	HF10%20s	4.76 (2.0) ^{ABb}	6.3 (3.7) ^{Ba}
	HF10%60s	6.19 (3.2) ^{Ab}	9.62 (4.2) ^{Aa}
Lithium-disilicate glass-ceramic (LD)	HF5%20s	0.36 (0.1) ^{Bb}	0.81 (0.3) ^{Ba}
	HF5%60s	0.49 (0.1) ^{Bb}	0.87 (0.4) ^{Ba}
	HF10%20s	0.68 (0.4) ^{Bb}	1.12 (0.6) ^{Ba}
	HF10%60s	1.14 (0.8) ^{Ab}	2.02 (1.3) ^{Aa}

Different capital letters indicate statistical differences between groups (columns) within the same ceramic ($p < 0.05$). Different lowercase letters indicate statistical differences between depth and width of the same group (rows) ($p < 0.05$). Comparisons between ceramics should not be made, as ceramic was not included as a factor. HF: hydrofluoric acid.

The patterns obtained for LEU were classified separately from those obtained for LD because the glass-ceramics tested have different compositions. LEU ($\text{SiO}_2\text{-Al}_2\text{O}_3\text{-K}_2\text{O-Na}_2\text{O}$) has an amorphous phase, which is basically composed of silicon and reinforced with leucite crystals ($\text{KAl-Si}_2\text{O}_6$; 35–45 vol%), which consist of metallic ions and great amounts of silicon. Accordingly, HF might corrode the glass and part of its crystalline phase. In contrast, LD ($\text{SiO}_2\text{-Li}_2\text{O-K}_2\text{O-Al}_2\text{O}_3\text{-P}_2\text{O}_5$) has twice the crystalline phase, approximately 70 vol% of lithium-disilicate glass-ceramic crystals, with a much lower amount of silica and a high degree of reticulation, conditions in which greater energy is required to dissolve the crystals.² Additionally, LD has fewer pores and surface defects than LEU, resulting in lower dissolution rates.^{3,13,20,22}

Previous studies may have observed only the dissolution patterns that were more evident (IV and III) or pronounced, and might have overlooked the other patterns (II and I), or they might have judged them irrelevant. To the knowledge of the present authors, the patterns assessed in this study have never been discussed or published before; their relevance relies on the possible deleterious effects of HF-etching protocols on dental restorations in clinical practice. If unaware of the impact of HF-etching protocols during ceramic surface treatment, especially those involving greater glass content (LEU), clinicians might create a more severe dissolution pattern that could affect the long-term performance and structure of the restoration, including areas that might be unintentionally etched.

Considering that the greatest dissolution depth found on IS was 9.62 μm and that dissolution was found only on the edge of OS next to LS for both ceramics (Fig 7), it is evident that HF cannot penetrate through the specimen. Instead, it flows over the lateral surfaces and reaches the edge of the specimen surface opposite the application surface. The

diffusion-controlled reaction between silica and HF – inhibiting the formation of deep pores – might account for the inability of the HF to penetrate the ceramic deeper than 9.62 μm . Therefore, the diffusion and the reaction rates of HF drop as its concentration gradient decreases.^{13,22}

Based on our findings, particular care should be taken when preparing a glass-ceramic surface for cementation, since dissolution may compromise the ceramic's color and adjacent surfaces, especially when using higher concentrations of HF, longer etching durations, and ultra-thin restorations, in which damage may be worse. Also, since previous studies reported similar or greater cement-ceramic bonding efficacy for HF5%20s,^{5,6,8,23} HF at a lower concentration and shorter application time could be considered for both LEU and LD.

The specimen shape might be a limitation of this in vitro study, since a real veneer or fragment involve different shapes. Regardless of shape, the physicochemical process described above, where the HF flows over surfaces other than the application surface, could also occur in ultrathin ceramic restorations. Therefore, further investigations correlating different dissolution patterns (LEU I–IV, LD I–III) and the internal dissolution of etched glass-ceramic restorations considering their mechanical properties, bond strength, and color stability would be of great relevance for establishing an ideal HF-etching protocol for ultrathin glass-ceramic restorations.

CONCLUSION

All the surfaces of ultrathin glass-ceramics can be affected by hydrofluoric acid gel, producing different patterns of dissolution. The hydrofluoric acid etching protocol with a higher HF concentration and longer application time revealed more severe patterns, especially on ceramics with greater glass

content. 5% hydrofluoric acid for 20 s showed less severe patterns for both ceramics; thus, it could be an ideal protocol to be applied at the center of the restoration's intaglio surface and carefully distributed to the rest of the intaglio surface, taking extra care at the margins.

ACKNOWLEDGMENTS

This study was conducted in partial fulfillment of the requirements for CG to obtain a MSc degree and made up part of her MSc thesis. This work was supported by the Brazilian Federal Government Agency, Ministry of Education, Coordination for the Improvement of Higher Education Personnel (CAPES No.0878/2018) and by the Brazilian National Council for Scientific and Technological Development (CNPq No.132709/2018-0)

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Clinical relevance: Since HF etching affects not only the surface on which it is applied, special care should be taken during its application. Lower concentrations and application times should be considered.