

Influence of Heated Hydrofluoric Acid Surface Treatment on Surface Roughness and Bond Strength to Feldspathic Ceramics and Lithium-Disilicate Glass-Ceramics

Diana Leyva del Rio^a / Emmanuel Sandoval-Sanchez^b / Nadia E. Campos-Villegas^c / Francisco X. Azpiazú-Flores^d / Norma-Veronica Zavala-Alonso^e

Purpose: To evaluate the effect of heated and room-temperature hydrofluoric (HF) acid on surface roughness parameters (Ra and Rq) and microtensile bond strength (μ TBS) on feldspathic ceramic and lithium-disilicate glass-ceramics.

Materials and Methods: Disk-shaped samples made from both ceramics were divided into groups according to surface treatment: feldspathic ceramic polished surface (FP), feldspathic ceramic + 60 s of 9% HF acid etching at room temperature (FC), feldspathic ceramic + 60 s of 9% HF acid etching heated to 70°C (F70), lithium-disilicate polished surface only (LP), lithium disilicate + 20 s of 9% HF acid etching at room temperature (LC), and lithium disilicate + 20 s of 9% HF acid etching heated to 70°C (L70). To evaluate Ra and Rq, non-overlapping readings were taken on the surface of each sample with a contact stylus profilometer. To measure microtensile bond strength (μ TBS), samples of groups FC, F70, LC and L70 received their corresponding surface treatment, were silanized and then bonded using a dual-cure composite cement to resin composite disks. After 24 h, samples were sectioned to obtain specimens for μ TBS. Representative samples from each group were examined using scanning electron microscopy (SEM) to analyze the morphology of the etched surface. The data were analyzed for statistical significance using Welch's ANOVA with the Games-Howell multiple-comparison post-hoc test.

Results: For both surface roughness parameters and HF acid etching at room temperature (FC and LC) showed a significant increase ($p < 0.001$) in surface roughness when compared to polished surfaces (FP and LP). Furthermore, the use of heated HF acid etching significantly increased ($p < 0.001$) the surface roughness of the ceramic when compared to their counterpart sample of HF acid etching at room temperature. Group L70 obtained the highest μ TBS of all groups (29.11 ± 8.26 MPa) and was significantly higher ($p < 0.001$) than that of the other experimental groups. There were no statistical differences ($p > 0.05$) between groups FC (19.94 ± 4.14), F70 (18.24 ± 5.29), and LC (17.87 ± 6.96).

Conclusion: The use of 9% HF acid etching heated to 70°C resulted in significantly higher surface roughness and improved bond strength onto lithium-disilicate glass-ceramic compared to surface HF acid etching at room temperature.

Keywords: lithium disilicate, feldspathic ceramic, microtensile bond strength, heated hydrofluoric acid, surface roughness.

*J Adhes Dent 2021; 23: 549-555.
doi: 10.3290/j.ad.b2288275*

Submitted for publication: 17.05.21; accepted for publication: 15.07.21

Improvement in the optical and mechanical properties in addition to better production and bonding techniques have resulted in an increase in the use of dental ceramics

in the past decade.²⁶ The longevity and clinical success of these ceramic restorations is based on a durable physical and chemical bond between the glass ceramic and the com-

^a PhD Candidate, Division of Restorative and Prosthetic Dentistry, College of Dentistry, Ohio State University, Columbus, Ohio, USA. Study design, statistical analysis, wrote the manuscript.

^b Graduate Student, Graduate Program in Esthetic, Cosmetic, Restorative, and Implant Dentistry, College of Dentistry, Autonomous University of San Luis Potosí, San Luis Potosí, Mexico. Study design, performed experiments, wrote the manuscript.

^c Graduate Student, Graduate Program in Esthetic, Cosmetic, Restorative, and Implant Dentistry, College of Dentistry, Autonomous University of San Luis Potosí, San Luis Potosí, Mexico. Study design, performed experiments, manuscript editing.

^d Graduate Student, Advanced Prosthodontics Program, Division of Restorative and Prosthetic Dentistry, College of Dentistry, Ohio State University, Columbus, Ohio, USA. Manuscript editing, substantial contribution to the discussion.

^e Associate Professor, College of Dentistry, Autonomous University of San Luis Potosí, San Luis Potosí, Mexico. Study design, image acquisition, manuscript editing.

Correspondence: Dr. Diana Leyva del Rio, Division of Restorative and Prosthetic Dentistry, College of Dentistry, Ohio State University, 305 W. 12th Ave, Columbus, OH, USA 43210. Tel: +1-614-680-6214; e-mail: leyvadel@gmail.com

Table 1 Test groups according to the type of ceramic and surface conditioning

Group name	Ceramic/surface conditioning with hydrofluoric acid
FP	Feldspathic ceramic polished surface only
FC	Feldspathic ceramic + 60 s of 9% hydrofluoric acid at room temperature
F70	Feldspathic ceramic + 60 s of 9% hydrofluoric acid heated to 70°C
LP	Lithium-disilicate glass-ceramic polished surface only
LC	Lithium-disilicate glass-ceramic + 20 s of 9% hydrofluoric acid at room temperature
L70	Lithium-disilicate glass-ceramic + 20 s of 9% hydrofluoric acid heated to 70°C

posite cement³⁰ that prevents the initiation and progression of fractures, marginal discoloration and the development of secondary caries around the restoration. To achieve an optimal bond, feldspathic ceramic and lithium-disilicate glass-ceramic restorations require surface conditioning with hydrofluoric (HF) acid, followed by application of a silane coupling agent on the intaglio surface of the ceramic restoration.^{15,21} Silica-based ceramics undergo a selective dissolution of the glassy phase when HF acid etching is applied, exposing silicon dioxide (SiO₂) crystals, which results in a change of the ceramic's surface topography. In the case of lithium disilicate, acid treatment dissolves the glassy matrix and exposes lithium-disilicate crystals.^{6,13} This surface conditioning with HF acid etching results in a distinct micromorphological pattern on the ceramic surface when silicon dioxide and lithium disilicate crystals are exposed. Surface conditioning is essential to the formation and maintenance of both mechanical and chemical bonds, due to the creation of microporosities with which the composite cement interlocks upon polymerization. In addition to surface acid conditioning, the application of silane to the etched surface is fundamental, because it creates a chemical bond between the SiO₂ of the ceramic and the organic matrix of the composite cement through siloxane bonds, which promote a stronger, more stable adhesion onto indirect restoration.^{5,17,18}

Current research is focused on improving the bonding capabilities and resulting mechanical properties of ceramics by modifying etching protocols. For example, novel etching protocols have been proposed, which use a heated chemical etching solution based on HCl on the surface of zirconia ceramics. This protocol produced deeper retentions, resulting in overall higher surface roughness values and increased bond strength, regardless of the application time.⁷⁻⁹ Although zirconia ceramics are inert to HF acid etching due to the lack of a glassy matrix, the use of a heated HF acid etching solution at 100°C in an *in vitro* study resulted in significantly higher surface roughness and enhanced the reliability of the resin-zirconia bonding.¹⁶ This proves the positive influence of a heated acid treatment on the surface morphology and the bonding properties onto zirconia ceramic. The use of heated HF acid etching on

glassy ceramics such as lithium disilicate has also been evaluated in recent literature. Sundfeld et al^{28,29} observed that the use of different concentrations of heated HF acid etching on a lithium-disilicate glass-ceramic improved bond strength of composite cement. However, the literature lacks an evaluation of the effect of heated HF acid etching on the bonding ability onto other ceramics, such as feldspathic ceramic.

Surface conditioning with HF acid etching selectively etches and exposes the glass phase of ceramics, inducing topographic changes on the surface, such as surface roughness. This increase in roughness contributes not only to the micromechanical retention of the composite cement upon polymerization, but also increases the surface area and surface energy. The latter are important contributing factors for the wettability and penetration of the composite cement, thus increasing the adhesive potential.^{4,14} In the case of feldspathic ceramics, the size and shape of microretentions can be influenced by the concentration of HF acid etching and also by the application time, which directly affects the bond strength to the substrate.^{1,2} In the case of lithium disilicate, the glass matrix is selectively dissolved, exposing the lithium disilicate crystals, which results in a roughened surface ideal for micromechanical retention with the resin adhesive upon polymerization.³⁰

To date, the effect of heated 9% HF acid etching on the surface roughness and bond strength on feldspathic ceramic is unknown. Furthermore, the resulting surface roughness and surface morphology of heated 9% HF acid etching on both feldspathic ceramic and lithium-disilicate glass-ceramics have yet to be investigated. Therefore, the aim of this study was to evaluate the effect of heated and non-heated HF acid etching on the surface roughness parameters (Ra and Rq) and microtensile bond strength (μ TBS) and observe the surface morphology using scanning electron microscopy (SEM) of two glass ceramics: feldspathic ceramic and lithium-disilicate glass-ceramic. The hypotheses were: 1. the use of heated HF acid etching does not affect surface roughness parameters and surface morphology of the ceramics tested; 2. the use of heated HF acid etching does not affect the microtensile bond strength onto the ceramics tested.

MATERIALS AND METHODS

Surface Roughness

Feldspathic ceramic samples (EX-3, shade A2, Kuraray Noritake; Tokyo, Japan) were fabricated following the application and firing protocol recommended by the manufacturer. The ceramic paste was extensively vibrated, placed in circular plastic molds, and gently blot dried to remove any excess liquid. The samples ($n = 15$) were removed from the plastic molds and immediately placed in a ceramic furnace. The final dimensions of the samples were 10 mm in diameter and 2 mm thickness. Lithium-disilicate samples ($n = 15$) were fabricated from e.max Press ingots (IPS-e.max Press MO 3, Ivoclar Vivadent; Schaan, Liechtenstein), with the same dimensions as the feldspathic ceramic samples. The surface was polished to obtain a standardized flat surface using silicon carbide papers (180, 400, 600, 1500 and 2000 grit, 3M Oral Care; St Paul, MN, USA) under water cooling. Subsequently, the samples were cleaned in an ultrasonic bath with distilled water for 20 min. The samples of the two materials were divided into three groups ($n = 5$) according to surface treatment (Table 1).

For groups FP and LP, the surface was only polished according to the manufacturer's instructions. The ceramic surfaces of groups FC and LC were etched with 9% HF acid etching (Porcelain Etch, Ultradent; South Jordan, UT, USA) for 60 s and 20 s, respectively. For the etching application of groups F70 and L70, a de-waxing furnace (model 434-HD, CAISA; Mexico City, México) was heated to $70 \pm 5^\circ\text{C}$. Once the furnace reached the desired temperature, a small quantity of HF acid etching was placed in an Eppendorf tube and placed inside the furnace. The Eppendorf tube remained in the furnace for 1 min. The temperature in the furnace was monitored continuously. After removing the heated HF acid etching from the furnace, it was quickly applied in an even layer to the sample's polished surface of groups F70 and L70 for 60 s and 20 s, respectively. The process was repeated with a new Eppendorf tube and additional HF acid etching for each sample. The treated surfaces of all groups were later rinsed with air-water spray for 60 s, then rinsed with sodium carbonate for 60 s to neutralize the HF acid etching, and finally air dried. Subsequently, 37% phosphoric acid (3M Oral Care) was applied for 60 s, and the samples were cleaned in an ultrasonic bath (Quantrex Q310; NJ, USA) with distilled water for 5 min. After preparation and cleaning, the samples were fixed on a flat surface and a contact stylus profilometer (Surftest SJ-301, Mitutoyo; Tokyo, Japan) was used to evaluate the surface roughness of the disks after exposure to the acid treatment. Ten non-overlapping readings per sample were obtained over the entire surface of the disk.

Microtensile Bond Strength

Additional 5-mm-thick samples for groups FC, F70, LC and L70 were fabricated following the methodology mentioned above. After acid treatment and sample cleaning, two layers of silane (Ultradent) were applied for 60 s and dried with hot air for 30 s with a heat gun (Bosch; Gerlingen, Germany). The ceramic disks were bonded using a dual-cure composite cement

(Clearfil SA Luting, Kuraray Noritake) to resin composite disks (10 x 5 mm) (Tetric n-ceram, Ivoclar Vivadent). One surface of each resin disk was previously sandblasted with 50- μm aluminum oxide, cleaned with 37% phosphoric acid, dried, and coated with a layer of adhesive (Tetric N Bond, Ivoclar Vivadent). All surfaces of the ceramic disk/composite cement/resin composite disk assembly were light cured for 40 s with a light-curing unit (Bluephase N MC, Ivoclar Vivadent) at an irradiance of 800 mW/cm², and then stored dry at room temperature for 24 h. The samples were sectioned using a low-speed diamond saw (Isomet Low Speed Saw, Buehler; Lake Bluff, IL, USA) under copious water along the x- and y-axes to obtain stick-shaped specimens with a cross-sectional surface area of approximately $1 \pm 0.1 \text{ mm}^2$. The specimens obtained from the periphery of the sample were discarded, due to the possibility of absence or excess of composite cement that might alter the final results. A total of 25 specimens per group were tested to measure the bond strength. The area of the specimens was measured using a digital caliper and was recorded. Subsequently, the specimens were fixed with cyanoacrylate glue (Zapit, Dental Ventures of America; Corona, CA, USA) in a Geraldelli jig, placed in a universal testing machine (Shimadzu AG-I; Tokyo, Japan), and loaded in tensile force at a crosshead speed of 1 mm/min until fracture. The microtensile bond strength was calculated using the following formula: $R = F/A$, where R is the strength (MPa), F is the load required for failure of the specimen (N) and A is the surface area of the specimen (mm²).

Surface Morphology Analysis

One representative sample from each group was examined using scanning electron microscopy (SEM) to analyze the morphology of the etched surface. Samples were mounted on aluminum stubs and sputter-coated with gold. The images were taken at 1000X and 3000X magnification with a scanning electron microscope (JSM 6510 LV, JEOL; Tokyo, Japan).

Statistical Analysis

The normality of the data distribution was verified using the Kolmogorov-Smirnov test ($\alpha = 0.05$), and the homogeneity of group variances was verified with Levene's test ($\alpha = 0.05$). All data were analyzed for statistical significance using Welch's ANOVA with the Games-Howell multiple comparison post-hoc test to find differences between groups ($\alpha = 0.05$) for μTBS and surface roughness. The statistical analyses were performed using SPSS software (version 21, IBM; Armonk, NY, USA).

RESULTS

Surface Roughness

Welch's ANOVA showed that surface roughness was significantly influenced by the use of heated acid treatment on the surface of the ceramics ($p < 0.001$) (Fig 1). Similar findings were observed for both Ra and Rq parameters, where polished surfaces yielded the lowest roughness values within each material. Between the feldspathic ceramic groups, the

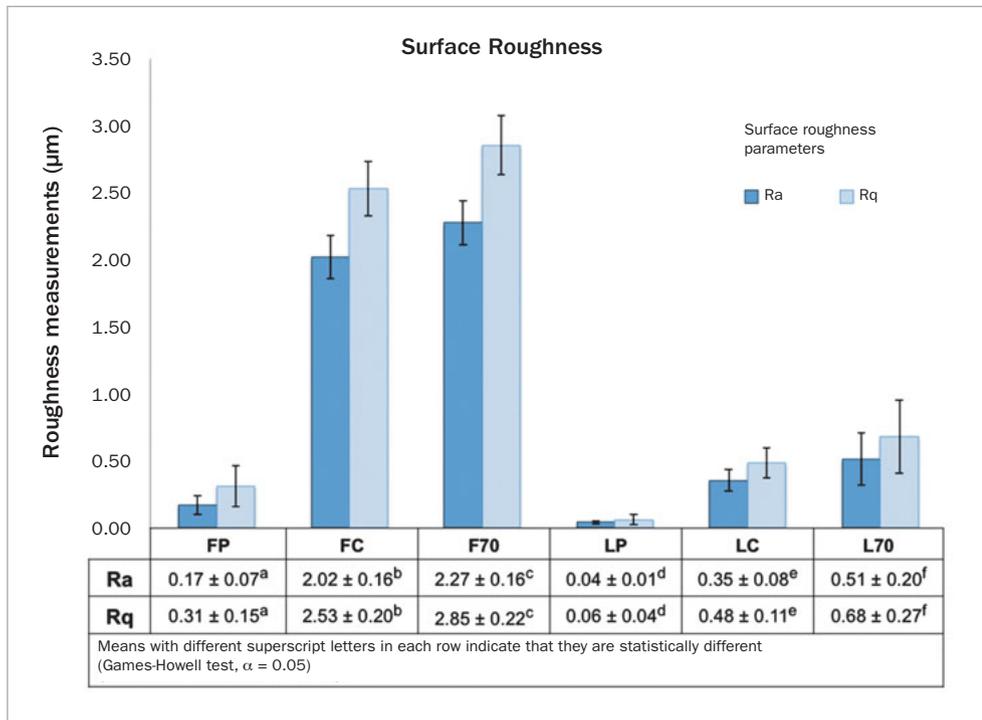


Fig 1 Bar graph of means and standard deviations for both surface roughness parameters tested: Ra and Rq (in μm). Means with different superscript letters in each row are statistically significantly different (Games-Howell test, $\alpha = 0.05$).

highest values were obtained in the heated acid etching group (F70) and were statistically significantly different ($p < 0.001$) from the feldspathic ceramic group treated with room-temperature acid (FC). Similarly, the highest value was obtained by the lithium-disilicate group treated with heated acid (L70) and was significantly different ($p < 0.001$) from the group subjected to acid treatment at room temperature (LC).

Microtensile Bond Strength

Welch's ANOVA revealed that the microtensile bond strength was influenced by the heated acid treatment of the ceramic surfaces ($p < 0.001$) (Table 2). Lithium-disilicate glass-ceramic with heated 9% HF acid etching (L70) resulted in the highest μTBS value of all groups (29.11 ± 8.26 MPa) and was significantly higher ($p < 0.001$) than that recorded for group LC and the two feldspathic ceramic groups (FC and F70). There were no statistically significant differences ($p > 0.05$) between groups FC, F70 and LC.

Surface Morphology Analysis

Scanning electron micrographs of the samples treated with different surface treatments are presented in Figs 2 and 3. The use of heated HF acid etching on feldspathic ceramic created more extensive, deeper craters in comparison with the use of HF acid etching at room temperature. Heated HF acid etching used on the surface of lithium disilicate produced greater glassy matrix dissolution and exposed a greater volume of lithium-disilicate crystals. It can be confirmed that the use of heated HF resulted in morphological changes in both ceramics.

DISCUSSION

The present study evaluated two surface roughness parameters and the bond strength of feldspathic ceramic and lithium-disilicate glass-ceramic using a novel surface treatment involving etching with 9% hydrofluoric acid heated to 70°C. It was found that ceramic surfaces treated with heated HF acid etching had higher surface roughness values than surfaces treated with HF acid etching at room temperature for both of the roughness parameters (Ra and Rq) evaluated; therefore, the first null hypothesis was rejected. Regarding μTBS , there was no statistically significant difference between the two surface treatments for feldspathic ceramic. However, for lithium-disilicate glass-ceramic, the surface treatment with heated HF acid etching showed statistically significantly higher MPa compared to the surface treatment with room temperature HF acid etching. Thus, the second null hypothesis was partially rejected.

A reliable adhesive bond of the ceramic restoration to the dental substrate with the use of a resin-based cement is fundamental for the clinical success and longevity of the dental restoration.¹⁹ A myriad of HF acid etching times and acid concentrations have been evaluated in the literature with the objective of improving the current etching protocol and maximizing the bonding ability and mechanical properties of the ceramics. Chen et al¹⁰ evaluated 5% HF acid etching on a feldspathic ceramic at different etching times ranging from 0 s to 180 s, finding the highest bond strength with 120 s of surface etching. Moura et al²⁰ found that etching time (60 or 120 s) did not influence the bond

strength to a feldspathic ceramic, and that 10% HF yielded higher bond strengths than did 5% HF. In terms of the latter, other literature has also focused on the effect on bonding properties and mechanical properties of HF acid etching at different concentrations on feldspathic ceramic. However, there is still much controversy about the ideal concentration for improving the bonding properties. Amaral et al³ found no statistically significant difference in bond strength between 4%, 5%, and 9% HF acid etching concentrations before thermocycling a feldspathic ceramic. Similar results were found by Venturini et al:³³ there was no statistically significant difference in bond strength for HF acid etching concentrations that ranged from 1% to 10% in non-aged samples, whereas on aged samples, stable resin adhesion after long-term aging was only found with concentrations of 3%, 5% and 10% of HF acid etching. Considering that the oral environment is a complex system which physically alters dental restorative materials through continuous and dynamic masticatory forces, some authors have analyzed the influence of different HF acid etching concentrations on the mechanical properties of feldspathic ceramic. According to Venturini et al,^{31,32} HF acid etching at 10% obtained the highest fatigue values when compared to 1% and 5% HF acid etching.

Alternative etching times and HF acid etching concentrations have also been studied on lithium-disilicate glass-ceramics in order to improve the adhesive strength and mechanical properties of the material. Puppini-Rontani et al²⁴ evaluated HF acid etching at 1%, 2.5%, 5%, 7.5% and 10% at different etching times, and observed that HF acid etching at 5%, 7.5%, and 10% provided significantly higher bond strengths irrespective of etching times. Fonzar et al¹² evaluated 4 different etching times (20 s, 40 s, 60 s and 120 s) at two HF acid etching concentrations (4.9% and 9.5%) and concluded that although etching time did not influence bonding strength, acid concentration did, showing that 4.9% HF acid etching resulted in significantly higher bond strengths than 9.5% HF acid etching concentration. On the other hand, Colombo et al¹¹ evaluated 2 different etching times (20 s and 60 s) at 2 different HF acid etching concentrations (5% and 10%) and observed higher bond strengths in the experimental groups with HF acid etching at 10% when compared to their counterparts at 5%. Prochnow et al²³ evaluated cyclic fatigue on lithium disilicate crowns, where the intaglio surface of the restoration was conditioned with HF acid etching at different concentrations (3%, 5%, and 10%), finding that although it was not statistically significant, HF acid etching at 10% resulted in the highest cyclic fatigue value.

In this study, we assessed the etching protocol described in the search for an alternative surface treatment that promotes better bonding to feldspathic ceramic and lithium-disilicate glass-ceramics. The use of a heated acidic solution to condition ceramic surfaces was first tested on zirconia⁹ in an effort to create a more retentive surface that would promote easy composite-cement flow into the ceramic and, after composite-cement polymerization, better interlocking. Currently, the literature contains only two studies on the use of heated HF acid etching on glass ceramics,

Table 2 μ TBS means and SD (in MPa)

Group name	Means \pm SD
FC	19.9 \pm 4.1 ^a
F70	18.2 \pm 5.3 ^a
LC	17.9 \pm 7.0 ^a
L70	29.1 \pm 8.3 ^b

Means with the same superscript letters are not statistically significantly different (Games-Howell test, $\alpha = 0.05$).

specifically lithium disilicate. Sundfeld et al²⁸ reported the use of heated HF acid etching at 5 concentrations (1%, 2.5%, 5%, 7.5%, and 10%) to etch the surface of lithium-disilicate glass-ceramic samples. It was observed that the use of heated HF acid etching yielded higher bond strengths when compared to its control group, at all HF acid etching concentrations. It is also important to notice the progressive increase of bond strengths as the HF acid etching concentration increases, showing very similar values for 7.5% and 10% acid concentration between the heated HF acid etching and its control. Similarly, in another study by the same authors,²⁹ where only two concentrations of HF acid etching were used (5% and 10%), a significantly higher bond strength was observed for the group treated with heated acid compared to the control at 5% concentration, whereas for HF acid etching at 10% concentration, the control and the heated experimental groups showed very similar values. In our study, we applied 9% heated HF acid etching to both lithium-disilicate and feldspathic ceramic surfaces. We observed significantly higher μ TBS between the heated HF acid etching in the lithium disilicate group (L70: 29.1 \pm 8.3) vs its counterpart group (LC: 17.9 \pm 7.0). On the other hand, there was no statistically significant difference between the feldspathic ceramic group treated with room-temperature HF acid etching (FC: 19.9 \pm 4.1) and the heated HF acid etching experimental group (F70: 18.2 \pm 5.3).

HF acid etching is used on the surface of ceramics in order to selectively dissolve the glassy phase and create a porous surface ideal for the composite cement to penetrate and create micromechanical retention upon polymerization. The surface morphology created by the etching process increases the surface area, which promotes wettability²⁵ and surface energy, which in turn enhances chemical bonding between the ceramic and the composite cement. It could be assumed that greater surface roughness is associated with higher bond strength onto dental ceramics; however, the literature that analyzed the effect of HF acid etching on surface roughness and bond strength has not yet provided a consensus on whether these two factors can be directly correlated to each other. Sudré et al²⁷ observed that the application of 10% HF acid etching on lithium disilicate achieved the greatest surface roughness, but not the highest bond strength. Those authors suggested that 10% HF acid etching and tap water led to dissolution and leaching

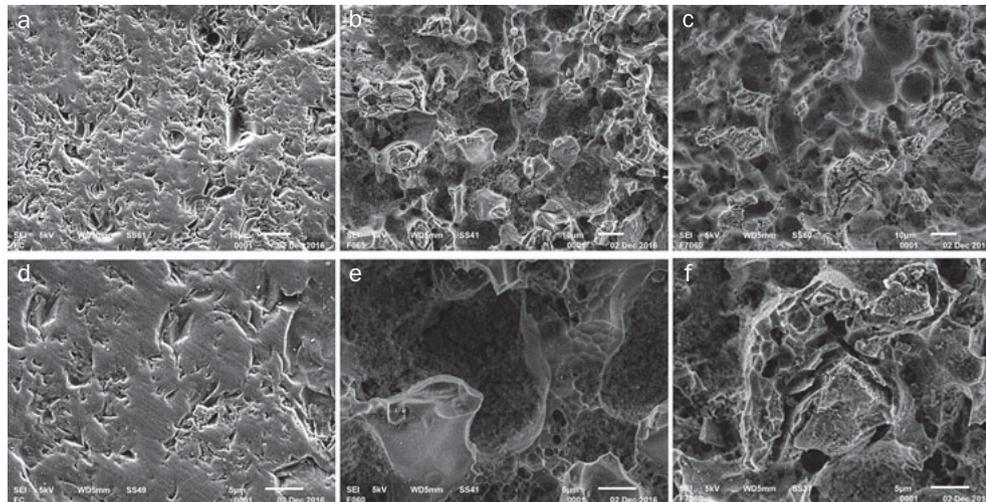


Fig 2 Representative SEM micrographs of the different surface treatments on feldspathic ceramic. a: polishing only (1000X); b: 60-s application of 9% hydrofluoric acid at room temperature (1000X); c: 60-s application of 9% hydrofluoric acid heated to 70°C (1000X); d: polishing only (3000X); e: 60-s application of 9% hydrofluoric acid at room temperature (3000X); f: 60-s application of 9% hydrofluoric acid heated to 70°C (at 3000X).

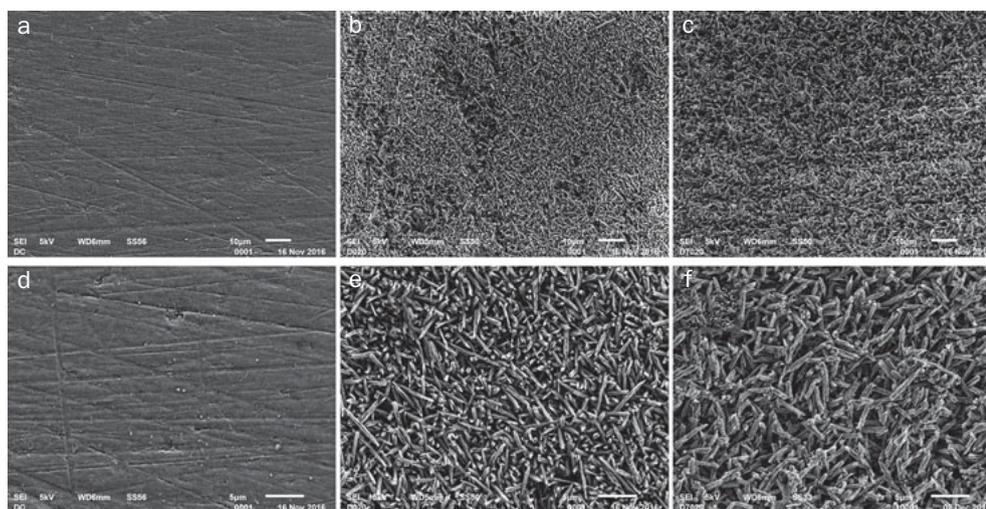


Fig 3 Representative SEM micrographs of the different surface treatments on lithium-disilicate glass-ceramic. a: polishing only (1000X); b: 20-s application of 9% hydrofluoric acid at room temperature (1000X); c: 20-s application of 9% hydrofluoric acid heated to 70°C (1000X); d: polishing only (3000X); e: 20-s application of 9% hydrofluoric acid at room temperature (3000X); f: 20-s application of 9% hydrofluoric acid heated to 70°C (3000X).

of the glassy matrix, leaving lithium-disilicate crystals clearly exposed. Conversely, Prochnow et al²² observed that lithium-disilicate surfaces conditioned with HF acid etching at 3%, 5% and 10% achieved statistically similar bond strengths, but that HF acid etching at 3% produced significantly lower surface roughness than did the 5% and 10% concentrations. In our study, group F70 obtained significantly higher surface roughness values compared to group FC. However, in this case, higher surface roughness values did not translate into higher bond strengths: these two groups obtained similar bond strengths. On the other hand, group L70 obtained the highest surface roughness values as well as the highest bond strength when compared to group LC. Representative SEM images (Fig 3) show that both etching protocols dissolved the glassy matrix and exposed lithium-disilicate crystals. The L70 images (Figs 3c and 3f) depict a rougher surface with more of the glassy matrix dissolved and thus more lithium disilicate needles exposed. A possible explanation for the improved bond strength in the L70 group is that the composite cement

flowed into an increased surface area caused by a higher number of micro- and nanoporosities in the etched ceramic, followed by interlocking upon polymerization.

The use of heated HF acid etching produced a significant increase in the surface roughness of the feldspathic ceramic; however, it did not yield an increase in bond strength when compared to the group that was surface conditioned with HF acid etching at room temperature. The use of HF acid etching also dissolved the glassy matrix and exposed silicon oxides (SiO_2), which produces the topographic changes needed for micromechanical retention when the composite cement is polymerized. The representative SEM images (Fig 2) reveal the presence of porosities with variable width and depth in the etched ceramic in both FC and F70 groups. However, larger pores and irregular grooves are evident in the F70 group vs the FC group. As stated by Colombo et al,¹¹ the presence of larger surface defects on the etched ceramic may favor micromechanical interlocking with the composite cement, as long as the composite cement can infiltrate those irregularities. We suggest that the presence of deeper

irregularities and narrow grooves caused the increase in the surface roughness in the F70 group. However, the etched surface may not have been completely filled, leaving voids at the interface between the composite cement and the ceramic surface, and thus bond strength did not increase.

Additional studies should be performed in order to fully understand the interaction between heated HF acid etching and the ceramics evaluated here. For example, it is unclear how aging in water affects the bond strength and other mechanical properties, such as flexural strength. Moreover, the use of different heated HF-acid concentrations and etching times on additional dental ceramics should be examined, considering that the only reports in the literature concern lithium-disilicate ceramic (and in our study also feldspathic ceramic). Finally, the analysis of cyclic fatigue resistance of bonded restorations etched with heated HF acid etching could yield additional information that is more relevant for the long-term success of the restoration.

CONCLUSION

The use of 9% HF acid etching heated to 70°C resulted in higher surface roughness and improved bond strength to lithium-disilicate glass-ceramic compared to surface conditioning with HF acid etching at room temperature. The application of heated HF acid etching on feldspathic ceramic surfaces did not show any improvement in the surface roughness or bond strength.

REFERENCES

- Addison O, Marquis PM, Fleming GJP. The impact of hydrofluoric acid surface treatments on the performance of a porcelain laminate restorative material. *Dent Mater* 2007;23:461–468.
- Aida M, Hayakawa T, Mizukawa K. Adhesion of composite to porcelain with various surface conditions. *J Prosthet Dent* 1995;73:464–470.
- Amaral R, Özcan M, Bottino MA, Valandro LF. Resin bonding to a feldspar ceramic after different ceramic surface conditioning methods: Evaluation of contact angle, surface pH, and microtensile bond strength durability. *J Adhes Dent* 2011;13:551–560.
- Ayad MF, Fahmy NZ, Rosenstiel SF. Effect of surface treatment on roughness and bond strength of a heat-pressed ceramic. *J Prosthet Dent* 2008;99:123–130.
- Della Bona A, Anusavice KJ, Hood JA. Effect of ceramic surface treatment on tensile bond strength to a resin cement. *Int J Prosthodont* 2002;15:248–253.
- Borges GA, Sophr AM, de Goes MF, Sobrinho LC, Chan DC. Effect of etching and airborne particle abrasion on the microstructure of different dental ceramics. *J Prosthet Dent* 2003;89:479–488.
- Casucci A, Mazzitelli C, Monticelli F, Toledano M, Osorio R, Osorio E, Papacchini F, Ferrari M. Morphological analysis of three zirconium oxide ceramics: Effect of surface treatments. *Dent Mater* 2010;26:751–760.
- Casucci A, Monticelli F, Goracci C, Mazzitelli C, Cantoro A, Papacchini F, Ferrari M. Effect of surface pre-treatments on the zirconia ceramic-resin cement microtensile bond strength. *Dent Mater* 2011;27:1024–1030.
- Casucci A, Osorio E, Osorio R, Monticelli F, Toledano M, Mazzitelli C, Ferrari M. Influence of different surface treatments on surface zirconia frameworks. *J Dent* 2009;37:891–897.
- Chen JH, Matsumura H, Atsuta M. Effect of different etching periods on the bond strength of a composite resin to a machinable porcelain. *J Dent* 1998;26:53–58.
- Colombo L de A, Murillo-Gómez F, De Goes MF. Bond Strength of CAD/CAM Restorative Materials Treated with Different Surface Etching Protocols. *J Adhes Dent* 2019;21:307–317.
- Fonzar RF, Goracci C, Carrabba M, Louca C, Ferrari M, Vichi A. Influence of acid concentration and etching time on composite cement adhesion to lithium-silicate glass ceramics. *J Adhes Dent* 2020;22:175–182.
- Holand W, Schweiger M, Frank M, Rheinberger V. A comparison of the microstructure and properties of the IPS Empress 2 and the IPS Empress glass-ceramics. *J Biomed Mater Res* 2000;53:297–303.
- Jardel V, Degrange M, Picard B, Derrien G. Correlation of topography to bond strength of etched ceramic. *Int J Prosthodont* 1999;12:59–64.
- Kalavacharla VK, Lawson NC, Ramp LC, Burgess JO. Influence of etching protocol and silane treatment with a universal adhesive on lithium disilicate bond strength. *Oper Dent* 2015;40:372–378.
- Liu JK, Tsoi J, Matinlinna JP, Wong H. Effects of some chemical surface modifications on resin zirconia adhesion. *J Mech Behav Biomed Mater* 2015;46:23–30.
- Lu R, Harcourt JK, Tyas MJ, Alexander B. An investigation of the composite resin/porcelain interface. *Aust Dent J* 1992;37:12–19.
- Matinlinna JP, Lung CYK, Tsoi JKH. Silane adhesion mechanism in dental applications and surface treatments: A review. *Dent Mater* 2018;34:13–28.
- McLean JW. Ceramics in clinical dentistry. *Br Dent J* 1988;164:187–194.
- Moura DMD, de Araújo AMM, de Souza KB, Verissimo AH, Tribst JPM, de Assunção e Souza RO. Hydrofluoric acid concentration, time and use of phosphoric acid on the bond strength of feldspathic ceramics. *Braz Oral Res* 2020;34.
- Pisani-Proença J, Erhardt MC, Valandro LF, Gutierrez-Aceves G, Bolanos-Carmona MV, Del Castillo-Salmeron R, Bottino MA. Influence of ceramic surface conditioning and resin cements on microtensile bond strength to a glass ceramic. *J Prosthet Dent* 2006;96:412–417.
- Prochnow C, Venturini AB, Grasel R, Gundel A, Bottino MC, Valandro LF. Adhesion to a lithium disilicate glass ceramic etched with hydrofluoric acid at distinct concentrations. *Braz Dent J* 2018;29:492–499.
- Prochnow C, Venturini AB, Guilardi LF, Pereira GKR, Burgo TAL, Bottino MC, Kleverlaan CJ, Valandro LF. Hydrofluoric acid concentrations: Effect on the cyclic load-to-failure of machined lithium disilicate restorations. *Dent Mater* 2018;34:e255–e263.
- Puppig-Rontani J, Sundfeld D, Costa AR, Correr AB, Puppig-Rontani RM, Borges GA, Sinhoreti M, Correr-Sobrinho L. Effect of hydrofluoric acid concentration and etching time on bond strength to lithium disilicate glass ceramic. *Oper Dent* 2017;42:606–615.
- Ramakrishnaiah R, Alkheraif AA, Divakar DD, Matinlinna JP, Vallittu PK. The effect of hydrofluoric acid etching duration on the surface micromorphology, roughness, and wettability of dental ceramics. *Int J Mol Sci* 2016;17.
- Seghi RR, Leyva del Rio D. Biomaterials: ceramic and adhesive technologies. *Dent Clin North Am* 2019;63.
- Sudré J, Salvio L, Baroudi K, Sotto-Maior B, Melo-Silva C, Assis N. Influence of surface treatment of lithium disilicate on roughness and bond strength. *Int J Prosthodont* 2020;33:212–216.
- Sundfeld D, Correr-Sobrinho L, Pini NIP, Costa AR, Sundfeld RH, Pfeifer CS, Martins LRM. Heat treatment-improved bond strength of resin cement to lithium disilicate dental glass-ceramic. *Ceram Int* 2016;42:10071–10078.
- Sundfeld D, Correr-Sobrinho L, Pini NIP, Costa AR, Sundfeld RH, Pfeifer CS, Martins LRM. The effect of hydrofluoric acid concentration and heat on the bonding to lithium disilicate glass ceramic. *Braz Dent J* 2016;27:727–733.
- Tian T, Tsoi JK, Matinlinna JP, Burrow MF. Aspects of bonding between resin luting cements and glass ceramic materials. *Dent Mater* 2014;30:e147–62.
- Venturini AB, Prochnow C, May LG, Kleverlaan CJ, Valandro LF. Fatigue failure load of feldspathic ceramic crowns after hydrofluoric acid etching at different concentrations. *J Prosthet Dent* 2018;119:278–285.
- Venturini AB, Prochnow C, Pereira GKR, Wernere A, Kleverlaan CJ, Valandro LF. The effect of hydrofluoric acid concentration on the fatigue failure load of adhesively cemented feldspathic ceramic discs. *Dent Mater* 2018;34:667–675.
- Venturini AB, Prochnow C, Rambo D, Gundel A, Valandro LF. Effect of hydrofluoric acid concentration on resin adhesion to a feldspathic ceramic. *J Adhes Dent* 2015;17:313–320.

Clinical relevance: Etching the surface of lithium disilicate glass ceramic with 9% HF acid at 70°C for 20 s improves the surface roughness and bond strength of composite cement. The application of heated HF acid on feldspathic ceramic does not have an effect on the adhesive properties.