Revista ESTOMATOLÓGICA Herediana
E-ISSN 2225-7616

ORIGINAL ARTICLE
DOI: https://doi.org/10.20453/reh.v35i1.6324

Bond strength to micro-shearing in feldspathic ceramics conditioned with hydrofluoric acid and subjected to different removal times and methods*

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ABSTRACT

Objective: To evaluate the micro-shearing bond strength in feldspathic ceramics through various methods of hydrofluoric acid residue removal and different application times. **Materials and methods:** An *in vitro* study in which 9% hydrofluoric acid was applied for 1 minute as a surface treatment on feldspathic ceramic discs. The samples were divided into 4 groups with 3 different application times and subjected to different removal methods: water and air spray; ultrasonic bath with distilled water; active application of 37% phosphoric acid for 1, 2, and 4 minutes; and a combination of 37% phosphoric acid actively applied for 1, 2, and 4 minutes followed by an ultrasonic bath with distilled water for 4 minutes. Cylinders of flowable resin were prepared and placed on the ceramic discs. Samples were subjected to micro-shear tests on a semi-universal testing machine. **Results:** The two-way ANOVA test and Tukey›s *post-hoc* test (p < 0.05) revealed that the water spray group at 1 and 2 minutes, and the ultrasonic bath at 4 minutes showed the highest bond strength values with a statistically significant difference. **Conclusions:** The water spray removal method for 1 and 2 minutes showed the highest bond strength value, while the lowest was observed with the 37% phosphoric acid removal method for 2 minutes.

Keywords: hydrofluoric acid; ceramics; shear strength; contaminant removal; distilled water.

Received: November 13, 2023
Accepted: November 18, 2024
Online: March 31, 2025

Cite as:
Peralta FB, Niño MA, Chávez PA, Dominguez JA. Bond strength to micro-shearing in feldspathic ceramics conditioned with hydrofluoric acid and subjected to different removal times and methods. Rev Estomatol Herediana. 2025; 35(1): 7-12. DOI: 10.20453/reh.v35i1.6324

* This article was developed based on the thesis by María Angélica Niño Osorio and Fátima Brunella Peralta Mas to obtain the professional degree of Second Specialty in Oral Rehabilitation from Universidad Peruana Cayetano Heredia.

INTRODUCTION

In modern dentistry, patients' esthetic demands have increased, leading to the use of new materials that provide a natural appearance, esthetics, and comfort. Therefore, the use of ceramic materials has become one of the most requested treatment options, making it essential to understand the types available on the market and their properties (1). Gracis et al. (2) established a new classification of dental ceramics into three categories: glass-matrix, polycrystalline, and resin-matrix. Feldspathic ceramic belongs to the group of ceramics with a glass matrix and exhibits high translucency, which provides superior optical properties that translate into greater patient satisfaction.

In addition to optical properties, mechanical characteristics are also relevant. To achieve proper adhesion, micromechanical retention must be created on the surface of the feldspathic ceramic. This treatment is achieved through hydrofluoric acid etching, which selectively dissolves the glassy phase, generating waste products resulting from the reaction of sodium, potassium, calcium, and aluminum fluorosilicates (3, 4). Moreover, micropores are created that significantly influence the mechanical adhesion of resin materials and enable bonding with the adhesive system and subsequently with the low-viscosity polymers found in resin cements, while optimizing surface wettability to promote better contact between the resin and ceramic (3, 4). Additionally, the acid-conditioned areas decrease the surface tension, allowing the adhesive system to achieve greater surface contact and thus enhance bonding to the resin (4).

Acid etching, in addition to creating microroughness, produces precipitates on the ceramic surface. The amount of these residues is related to the type, time, and concentration of the acid, and may negatively affect bond strength by interfering with the penetration of the adhesive system (1). In light of this issue, Canay et al. (5) observed that the surface of feldspathic ceramics etched with 9.5% hydrofluoric acid for 1 minute exhibited adequate microroughness and fewer residual precipitates compared to those etched for 4 minutes. These residues remaining on the surface are silico-fluoride salts that interfere with the bonding process (6).

Given the importance of removing residual salts, various methods were developed for this purpose. Steinhauser et al. (7) tested the effectiveness of different procedures (phosphoric acid, ultrasonic bath, and water spray) on feldspathic ceramics after etching with 10% hydrofluoric acid, and found no significant differences in bond strength. However, residual salts were observed on the surfaces of the control and water spray groups, while more efficient results were obtained in the groups treated with an ultrasonic bath after the application of hydrofluoric acid. Similar results were reported by Martins et al. (8), who found that the use of an ultrasonic bath with distilled water for 4 minutes was the most effective removal method.

On the other hand, regarding the use of phosphoric acid, Chávez (9) concluded that its application at 37%, regardless of the type, method, or time of application, decreases the bond strength of feldspathic ceramics conditioned with hydrofluoric acid.

Therefore, this study aims to determine whether reducing the application time and simplifying the different cleaning methods of ceramic surfaces can achieve optimal results in the bonding of restorations. Accordingly, the objective is to evaluate *in vitro* the micro-shear bond strength of feldspathic ceramics using various hydrofluoric acid residue removal methods and application times.

MATERIALS AND METHODS

This *in vitro* study determined the sample size through a pilot test based on the thesis *Micro-shear bond strength of feldspathic ceramic conditioned with hydrofluoric acid and subjected to phosphoric acid application as a residue removal technique according to type, method, and time. In vitro study*, by Chávez (9). The statistical formula for the comparison of means was applied to this test using the Epidat software, version 4.0. Due to the characteristics of this *in vitro* research, evaluation by an Ethics Committee was not required.

Preparation of ceramics discs

A total of 48 feldspathic ceramic discs (IPS Classic®, Vivadent; Schaan, Liechtenstein) in dentin shade 210 were fabricated, with initial dimensions of 2.6 mm in height and 12.5 mm in diameter. After sintering in a porcelain furnace (Pro 200 Series Furnace, Whip Mix®; KY, USA), the discs underwent a 20% contraction, resulting in final dimensions of 2 mm in height and 10 mm in diameter. Finally, the surfaces were polished using new 10 × 10 cm wet sandpapers of different grits.

The feldspathic ceramic discs were placed and fixed inside a PVC tube (Matusita®, Tigre SA, Lima, Peru) measuring 10 mm in diameter and 20 mm in height. The samples were mounted using transparent self-curing acrylic resin (Vitacryl®, A. Tarrillo Barba SA; Lima, Peru) and secured with double-sided adhesive tape (Topex®; Lima, Peru). The samples were sanded by rubbing from end to end 10 times using new 10 × 10 cm wet sandpapers to remove any type of acrylic residue.

Ceramic conditioning

The feldspathic ceramic discs were conditioned with 9% hydrofluoric acid (Porcelain Etch™, Ultradent™; Utah, USA) for 1 minute. Immediately afterward, the acid was removed with a water spray for 20 seconds, and the discs were gently dried with an air jet for another 20 seconds.

Removal methods and times

The discs were divided into four groups with three different application times:

- *Group 1*, water spray (WS): A water and air spray was applied using a triple syringe at a distance of 10 mm and an angle of 90° for 1, 2, and 4 minutes.
- *Group 2*, ultrasonic bath (UB): The samples were subjected to an ultrasonic bath (Ultrasonic Cleaner, Codyson® CD-4800, China) with distilled water for 1, 2, and 4 minutes.
- *Group 3*, phosphoric acid (PA): 37% phosphoric acid (Ultra-Etch™, Ultradent™; Utah, USA) was actively agitated with a microbrush for 1, 2, and 4 minutes.
- *Group 4*, phosphoric acid + ultrasonic bath (PA+UB): 37% phosphoric acid (Ultra-Etch™, Ultradent™; Utah, USA) was actively agitated with a microbrush for 1, 2, and 4 minutes, followed by an ultrasonic bath with distilled water for 4 minutes.

After each removal method, the samples from Groups 2, 3, and 4 were rinsed with a water spray and dried with an air spray from the triple syringe at a distance of 10 mm and a 90° angle for 20 seconds each.

Application of silane and adhesive system to the ceramic

Silane (Silane, Ultradent™; Utah, USA) was applied to the surface of the discs with a microbrush and allowed to dry for 60 seconds. A stream of air from a triple syringe was then applied for 15 seconds, followed by a layer of adhesive (Adper Single Bond 2, 3M™ Espe™; St. Paul, MN, USA), which was actively agitated with a micro-brush. Finally, the adhesive was polymerized for 20 seconds using an LED curing unit with a power density of 1000 mW/cm² (VALO™ Cordless, Ultradent™; South Jordan, UT, USA).

Fabrication and placement of resin cylinders

Resin cylinders were fabricated using a Tygon tube (TYGON; USA) measuring 2 mm in height and 0.8 mm in diameter, and flowable resin (Filtek™ Z350 Flow, 3M™ Espe™; St. Paul, MN, USA). The cylinders were placed on the ceramic discs and polymerized for 20 seconds using an LED curing lamp of 1000 mW/cm² (VALO™ Cordless, Ultradent™; South Jordan, UT, USA). A distance of 2 mm was maintained between the cylinders; in addition, the cylinders were covered with aluminum foil, and a black cloth was placed beneath the PVC tubes to absorb light. Subsequently, the silicone coating of the Tygon tubes was removed by cutting with a No. 11 scalpel blade.

Evaluation of micro-shear bond strength (in MPa)

Samples were stored for 1 day in physiological saline solution at room temperature. The microshear bond strength test was performed at the dental materials laboratory of the Universidad Peruana Cayetano Heredia using a semi-universal testing machine (OM 100, Odeme® Dental Research; Brazil), for which an orthodontic wire No. 7 bent in the shape of an "8" was used (Morelli® Ortodontia; Brazil), with a 50 N load cell, at a crosshead speed of 0.75 mm/min, until failure occurs.

Analysis plan

A descriptive analysis was performed to obtain the mean and standard deviation of bond strength for each evaluated group. Data normality was assessed using the D'Agostino test. Bivariate analysis was conducted using two-way ANOVA followed by Tukey's *post hoc* test. A confidence level of 95% was assumed ($p < 0.05$).

RESULTS

This study aimed to determine the bond strength values after hydrofluoric acid cleaning at three different times (1, 2, and 4 minutes) using four methods (WS, UB, PA, and PA+UB).

The different cleaning methods were compared at each application time. At 1 minute, the highest micro-shear bond strength was observed in the WS group (11.79 ± 4.70 MPa); at 2 minutes, the highest values were found in the WS (11.63 ± 4.04 MPa) and PA+UB (8.82 ± 3.01 MPa) groups; and at 4 minutes, the UB group showed the highest bond strength (9.46 ± 4.38 MPa) ($p = 0.028$).

Considering each method individually, the highest bond strength values were observed at 1 minute (11.79 ± 4.70 MPa) and 2 minutes (11.63 ± 4.04 MPa) for WS, and at 4 minutes for UB (9.46 ± 4.38 MPa). The PA and PA+UB techniques did not yield higher micro-shear bond strength at any of the tested times. Therefore, the WS technique during 1 and 2 minutes, UB during 4 minutes, and PA+UB during 2 minutes achieved the highest and statistically significant values compared to the other groups and times evaluated (p = 0.015) (Table 1).

Table 1. Micro-shear bond strength values (in MPa) of feldspathic ceramics according to the type and application time of different hydrofluoric acid residue removal methods.

Groups	Time		
	1 min	2 min	4 min
WS	11.79±4.70^{Aa}	11.63±4.04^{Aa}	6.63±3.29^{Bb}
UB	7.18±2.76^{Bb}	7.66±2.31^{Bb}	9.46±4.38^{Aa}
PA	7.10±3.39^{Ab}	5.98±3.36^{Ab}	6.08±3.12^{Ab}
PA+UB	6.81±2.94^{Ab}	8.82±3.01^{Aa}	6.62±3.82^{Ab}

*Data normality: D'Agostino test; bivariate analysis: two-way ANOVA and Tukey's *post hoc* test; confidence level: 95% (p < 0.05).
Different uppercase letters indicate significant differences horizontally; different lowercase letters indicate significant differences vertically.
WS: water spray; UB: ultrasonic bath; PA: phosphoric acid.

DISCUSSION

Etching dental glass-ceramics with hydrofluoric acid is a crucial procedure to optimize the bond between the ceramic and the future restoration. Likewise, the acid concentration and etching time are key factors in creating a microroughened surface that enhances the substrate's bonding strength. However, hydrofluoric acid application produces a significant amount of residues on the ceramic surface, making their removal essential for improving the adhesion of ceramic materials (5, 10-13).

When comparing the results of the study by Steinhauser et al. (7) with the present research, it is evident that the values obtained in their study were higher, which may be related to the use of a longer etching time and higher hydrofluoric acid concentration (10% for 2 minutes), whereas, in our study, a 9% concentration for 1 minute was employed. Therefore, higher concentration and longer etching time would likely increase microroughness and enhance the adhesion of resin materials.

Another difference is that, for polishing the ceramics, Steinheuser et al. (7) used an electric polisher with water, obtaining a smoother, more uniform surface free of abrasive residues, unlike the present study, in which polishing was performed manually. They also concluded that there were no statistically significant differences between the groups analyzed, whereas in our study, four groups showed superior results (WS for 1 and 2 minutes, UB for 4 minutes, and PA+UB for 2 minutes) compared to the other methods at different application times. This is despite the fact that the aforementioned study also evaluated the micro-shear bond strength of feldspathic ceramics treated with the same removal methods: water and air spray, ultrasonic bath with distilled water, 37% phosphoric acid, and 37% phosphoric acid combined with an ultrasonic bath.

Sağlam et al. (14) evaluated micro-shear bond strength by comparing different surface treatments (hydrofluoric acid, aluminum oxide sandblasting, and silica coating) in three types of ceramics (feldspathic ceramic, lithium disilicate, and CAD-CAM reinforced zirconia). They obtained better results compared to the other methods applied when using 5% hydrofluoric acid for 1 minute on feldspathic ceramic and performing cleaning with an ultrasonic bath with distilled water for 5 minutes. These results are consistent with the present study, as the ultrasonic bath also achieved a high bond strength value.

Martins et al. (8) studied the effect of different cleaning methods after etching feldspathic ceramic blocks (Vita VM7®) with 10% hydrofluoric acid. The ceramic blocks were then cemented to composite resin blocks (W3D Master®) using resin cement (RelyX™ ARC). They found that ultrasonic cleaning with distilled water for 4 minutes resulted in the highest bond strength (18.8 ± 0.4 MPa). These results are comparable to the present study, where the ultrasonic bath removal method for 4 minutes achieved the highest value (9.46 ± 4.38 MPa), showing a statistically significant difference compared to 1 and 2 minutes. It is also noteworthy that the mean bond strength in Martins et al. (8) was higher than that obtained in our study, which may be attributed to their use of blocks instead of discs, and resin cement instead of flowable resin.

Belli et al. (3), Steinhauser et al. (7), and Moura et al. (15) compared different methods of hydrofluoric acid removal across various types of ceramics, all using 37% phosphoric acid applied for 1 minute. They concluded that surface cleaning with phosphoric acid did not affect the resin-feldspathic ceramic bond strength. Among them, Steinhauser et al. (7) reported that in the group treated with phosphoric acid only, dark spots were observed at low magnification, which could be residues of acid not removed with water, and at higher magnification, the surface appeared granular or sand-like, resembling over-etching; however, this did not affect the micro-shear bond strength values. These findings are similar to those of the present study, as phosphoric acid as a removal method yielded the lowest bond strength values compared to the other groups.

A study conducted by Magne & Cascione (16) showed the importance of using ultrasound as a complementary cleaning method, as optical microscopy revealed a significant amount of white residues on feldspathic porcelains resulting from hydrofluoric acid etching. Consequently, these were cleaned with 37.5% phosphoric acid for 1 minute, which removed the crystalline residues.

However, scanning electron microscopy analysis showed that these samples only revealed microscopic deposits that still contaminated the etched surface, which were efficiently removed after ultrasonic cleaning.

In 2010, Belli et al. (3) evaluated the bond strength of a resin to two types of ceramics (leucite-reinforced ceramic and lithium disilicate) using different cleaning techniques following etching with 10% hydrofluoric acid for 60 and 20 seconds. The groups were as follows: no cleaning; water spray for 30 seconds; 37% phosphoric acid for 1 minute (non-active) followed by a 30-second water rinse; ultrasonic bath with distilled water for 5 minutes; and 1-minute phosphoric acid etching followed by a 30-second water rinse and a 5-minute ultrasonic bath. They found that the group achieving the highest bond strength in leucite-reinforced ceramic was the phosphoric acid etching combined with the ultrasonic bath (74.1 ± 10.9 MPa); however, the other groups were not statistically different, except for the control group. These findings, along with our results, indicate that the combination of ultrasonic bath and phosphoric acid improves the removal of hydrofluoric acid residues.

Finally, Sriamporn et al. (17) evaluated the effect of neutralizing agents on the shear bond strength of feldspathic ceramic etched with 9% hydrofluoric acid under aged and non-aged conditions. They concluded that the shear bond strength values between hydrofluoric acid-etched porcelain and water spray (19.44 ± 3.54 MPa), hydrofluoric acid etching with neutralizing agents, and hydrofluoric acid etching followed by ultrasonic bath for 10 minutes (20.69 ± 3.17 MPa) were not significantly different under the conditions mentioned. Similar to our study, water spray and the ultrasonic bath were the removal methods that achieved the highest results in eliminating hydrofluoric acid residues.

A potential limitation of the present study was the type of water used, since other studies report the use of distilled water, whereas in this study, tap water from the dental unit was used, which may contain contaminants that could affect the adhesion process; nevertheless, it is still close to real clinical conditions.

CONCLUSION

The highest bond strength values in feldspathic ceramics were obtained using hydrofluoric acid residue removal methods such as water spray for 1 minute and ultrasonic bath for 4 minutes.

Conflict of interest:
The authors declare no conflict of interest.

Funding:
Self-funded.

Ethics approval:
Evaluation by an Ethics Committee was not required since this was an *in vitro* study.

Author contributions:
FBPM, MANO: conceptualization, research, resources, visualization, writing – original draft, writing – review & editing.
PACA: conceptualization, project administration, supervision, validation.
JAD: methodology, data curation, formal analysis, supervision, validation.

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