

Influence of Acid Etching on Bond Strength Between Feldspathic Ceramics and Resin Cement

Ricardo Huver de Jesus,¹ Bianca Colombi Furlam,² Camila Maciel Martinusse,² Hiorrana Carla Vasconcelos Inácio,² Jefferson David Melo de Matos,³ Leonardo Jiro Nomura Nakano,³ Laura Patricia Nadal Ortiz,³ Marco Antonio Bottino,³ Lucas Campagnaro Maciel¹

¹Department of Dentistry, School of Dentistry of Multivix, Multivix College, Vitória, ES, Brazil

²Department of Dentistry, Escola Superior São Francisco de Assis (ESFA), Santa Teresa, ES, Brazil

³Department of Dental Materials and Prosthodontics, Institute of Science and Technology, São Paulo State University (Unesp), São José dos Campos, SP, Brazil

• **Conflicts of interest:** none declared.

ABSTRACT

Objective: analyze the influence of bond strength between resin cement and feldspathic ceramics using different concentrations and application times of hydrofluoric acid (HF). **Materials and Methods:** 18 feldspathic ceramic discs (Ø10mm - 2mm thickness) were made through the incremental technique, which were included in acrylic resin and regularized with silicon carbide sandpaper with granulations of 600, 1200 and 2000 through a polishing machine with a speed of 600 rpm. Soon after, they were immersed in distilled water for 10 minutes in an ultrasonic bath. All specimens were conditioned with HF and silanized. Four cement cylinders were made on each specimen (n=12), manufactured with Relyx ARC resin cement. After that, it was divided into six groups according to the surface treatment protocol: G1 (5% - 40s); G2 (5% - 60s); G3 (5% - 80s); G4 (10% - 40s); G5 (10% - 60s) e G6 (10% - 80s). The specimens were subjected to the Microshear Test, with vertical force at a speed of 0.5 mm min on the specimen. The microshear bond strength was calculated and expressed in Megapascal (MPa). The data were submitted to two-way analysis of variance (ANOVA) and Tukey's test for multiple comparisons, with a significance level of 5%. **Results:** when the concentration of HF was analyzed, there were no statistical differences (p>0.05). In the comparison of application times, G3 (5% - 80s) and G6 (10% - 80s) obtained higher results of microshear resistance than the other groups.

Conclusion: The different concentrations of HF did not influence the bond strength between feldspathic ceramics and resin cement. However, the increase in the time of HF conditioning affected in the bond strength.

Keywords: Ceramics; Resin Cements; Fluoridric Acid.

Introduction

Currently, dental ceramics are the restorative materials with the best optical properties¹. They have favorable properties and become able to resemble the dental element, offering a more natural treatment to the patient.² Other properties are desirable for this material as well, including chemical stability, compression resistance, biocompatibility and thermal expansion coefficient similar to dental structure.³

Campos *et al.*⁴ shows that, despite the numerous advantages of dental ceramics, there are some disadvantages, such as: friable and fragile material when subjected to traction. Therefore, the search to reduce failures has led to research to obtain a more resistant material, as well as surface treatments, to improve the union of ceramics and cement.⁵

It is not only the resistance of the interior of the prosthetic restoration that is relevant, the success of prosthetic rehabilitations with metal-free ceramics depends on numerous factors, including the cementing technique. This phase covers two bonding interfaces (dentin or enamel/cement and ceramic/cement).⁶ The adhesion of conventional resin cement to the dental element is improved when acid etching is used in enamel and dentin followed by hybridization. Likewise, ceramics should also be prepared to improve micromechanical retention to cement.⁷

Ceramics can be classified as vitreous ceramics, polycrystalline ceramics and hybrid ceramics. Just as there

are different compositions of ceramics and bonding agents, there are also some surface treatment protocols that differ according to the choice of these materials.⁸ Vitreous ceramics have a high silica content and have an excellent aesthetic performance (feldspathic ceramics, leucite and lithium disilicate), therefore, the surface treatment of choice for this type of ceramic is conditioning with 10% hydrofluoric acid (HF), followed by application of silane and a bonding agent, which is cement. This protocol is capable of producing good results.⁹

The absence of conditioning with hydrofluoric acid on the ceramic surface can reduce the bond strength of the material with the cement. Poor bonding quality at the cement/ceramic interface can decrease the ability of a ceramic to resist the beginning of a fracture.¹⁰

Most failures occurred in pure ceramic prostheses are caused by the bonding interface or internal ceramic surfaces. It is necessary to reduce marginal infiltration, improve retention and fracture resistance of fixed prostheses and identify a safe conditioning technique for the internal surface of ceramic systems available for cementation.¹¹

The aim of this study was to analyze the influence of different conditioning protocols of feldspathic ceramics with hydrofluoric acid on the bond strength of the interface between resin cement and restorative material.

Materials and Methods

Study Design

This study was an experimental research in which the factor under study was the time of acid application (in three levels) and the concentration of acid (in two levels). Having as a variable of response the microshear bond strength measured and expressed in Megapascal (MPa) of quantitative nature.

Specimen Preparation

Through the incremental technique, 18 discs (Figure 1) of feldspathic ceramics (Super porcelain EX-3, Noritake, Japan) were manufactured in the dimensions (Ø10 mm - 2 mm thick). The sintering process was done in a ceramic oven (Futura Brasil Premium Press, SP, BR) and temperature application according to the manufacturer's recommendations. The ceramic discs were embedded in a colorless, chemically activated acrylic resin (Articles Dental Classic, São Paulo, SP, Brazil) using a condensation silicone matrix (Speedex Putty Condensation Silicone, Coltene, Rio de Janeiro, RJ, BR) in a square format. All specimens were regularized with silicon carbide sandpaper (Silicon Carbide, 3M from Brazil, Sumaré, SP, BR) with granulations of 600, 1200 and 2000 through a polishing machine (Politriz PLO2E metallographic sander, Teclago, Vargem Grande Paulista, SP, BR) with a 600 rpm speed. Soon after they were immersed in distilled water for 10 minutes in ultrasonic bath (Soni Clean 6, Sanders do Brasil, Santa Rita de Sapucaí, MG, BR).



Figure 1. Specimen in ceramic disc format.

Hydrofluoric acid (Condporcelana, Dentscare LTDA – FGM, Joinville, SC, BR) was used at concentrations of 5% and 10% and arranged for different conditioning times of 40, 60 and 80 seconds on the ceramic surface. The specimens were randomly separated and divided into six groups according to the surface treatment protocol: Group 1 (G1), 5% hydrofluoric acid was used for 40 seconds; Group 2 (G2), 5% hydrofluoric acid for 60 seconds; Group 3 (G3), 5% hydrofluoric acid for 80 seconds; Group 4 (G4), 10% hydrofluoric acid for 40 seconds;

Group 5 (G5), 10% hydrofluoric acid for 60 seconds and Group 6 (G6), 10% hydrofluoric acid for 80 seconds. Right after, the washing was carried out under running water for 1 minute and the specimens were dried, a thin layer of silane agent was applied (Angelus Indústria de Produtos Odontológicas S/A, Londrina, PR, BR), and dried air after 1 minute.

On each ceramic disc, 4 cylinders of resin cement (Ø1.0 mm) were manufactured, using tubes for peristaltic pump (Tygon E-laboratory, Materflex, Vernon Hills, IL, USA) of 1.0 mm internal diameter and 1.0 mm high. The resin cement used to make the cylinders was the dual-cured resin cement (Relyx ARC, 3M ESPE, St Paul, MN, USA). The cementation protocol was standardized for the six groups. In making the cement tubes over the ceramic specimens, the cement used was spatulated on paper mixing pad with a spatula number 24 (Quinelato, Rio Claro, SP, BR) for 20 seconds. After manipulation, the cement was deposited inside the tube with a centrix syringe (Makeup Dental Products Industry S.A., Maringá, PR, BR) and insertion tips. Then, the tubes were placed on the ceramic blocks surface and photopolymerized (Bluephase N Polymerizer, Ivoclar Vivadent, Barueri, SP, BR) with light intensity of 1,200 mW/cm² for 20 seconds.

Microshear Test

All specimens presented were submitted to the microshear test (Figure 2), which was performed on a universal testing machine (EMIC DL-2000, São José dos Pinhais, PR, BR). A 10 cm high prefabricated vertical rod was used, with a 10 mm wide bevel-shaped tip. Then it was positioned as close as possible to the tested interface, making a vertical downward force on the fixed part to the base of the equipment. The micro-shear stress was applied at a speed of 0.5 mm/min until the failure of the resin cement cylinders. The bond strength to the micro shear was calculated and expressed in Megapascal (MPa).



Figure 2. Microshear test.

Failure mode check

After the micro shear tests, all specimens were checked with a stereoscopic magnifying glass (Aus Jena, Carl Zeiss, BW, DE) with a 40x magnification failure detection. The patterns were divided into: adhesive failure, when the rupture occurs at the interface between cement and ceramic; cohesive failure, when the rupture occurs in the restorative material (cement or ceramic); mixed failure, when the rupture occurs, part of the restorative material (cement or ceramic) and part of the cement/ceramic interface.

Statistical Analysis

The data were submitted to descriptive analysis and evaluated for compliance with the requirements of normality and homogeneity of variance. The aspects were met and the data were submitted to two-way analysis of variance (ANOVA), for multiple comparisons the Tukey test was used, at the significance level $p=0.05$.

Results

The results of the microshear testing are described in Table 1. When the variable concentration of hydrofluoric acid was tested, no statistical differences were observed for each application time ($p>0.05$). When comparing the application times, G3 obtained higher microshear resistance results than G1 and G2 ($p<0.05$) for a concentration of 5%. Likewise, G6 obtained higher resistance than G4 and G5 ($p<0.001$) when the concentration was 10%.

Table 1. Microshear bond strength (Means (MPa) ± standard deviation).

	Hydrofluoric Acid 5%	Hydrofluoric Acid 10%
40 seconds	9.32 ± 1.54 ^{Bc}	10.60 ± 1.68 ^{Bc}
60 seconds	10.18 ± 1.42 ^{Bb}	11.81 ± 2.32 ^{Bb}
90 seconds	13.92 ± 2.70 ^{Aa}	13.76 ± 1.55 ^{Aa}

Two-way ANOVA with Tukey. Different capital letters indicate difference between times, different lower letters indicate difference between concentrations ($p<0.05$).

The failure pattern of the specimens is described in Table 2. Adhesive failures (Figure 3) were observed in 65.27% of the specimens, mixed cement failures (Figure 4) in 33.33% of the specimens and mixed ceramic failures (Figure 5) in 1.38% of the specimens. No cohesive failures were observed, either for cement or ceramics.

Table 2. Failure Pattern.

	Hydrofluoric Acid 5%	Hydrofluoric Acid 10%
Adhesive	24	23
Predominantly Adhesive (Cement)	12	12
Predominantly Adhesive (Ceramic)	0	1
Cohesive	0	0

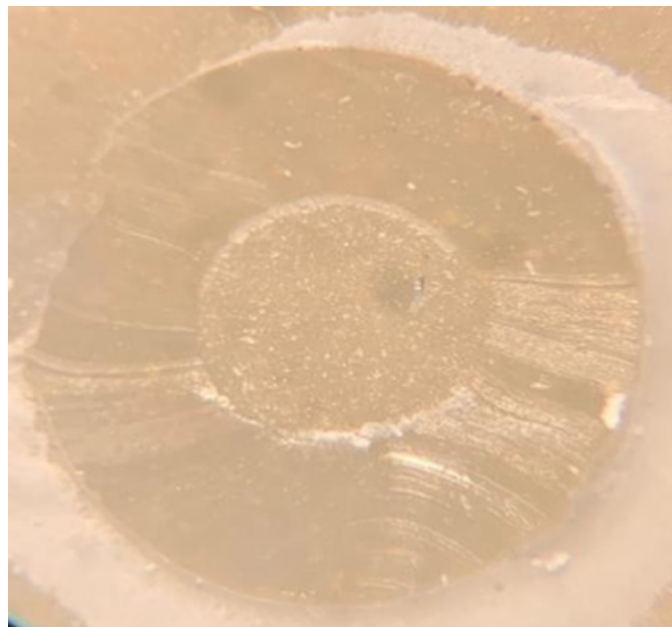


Figure 3. Adhesive Failure.



Figure 4. Predominantly Adhesive (cement).

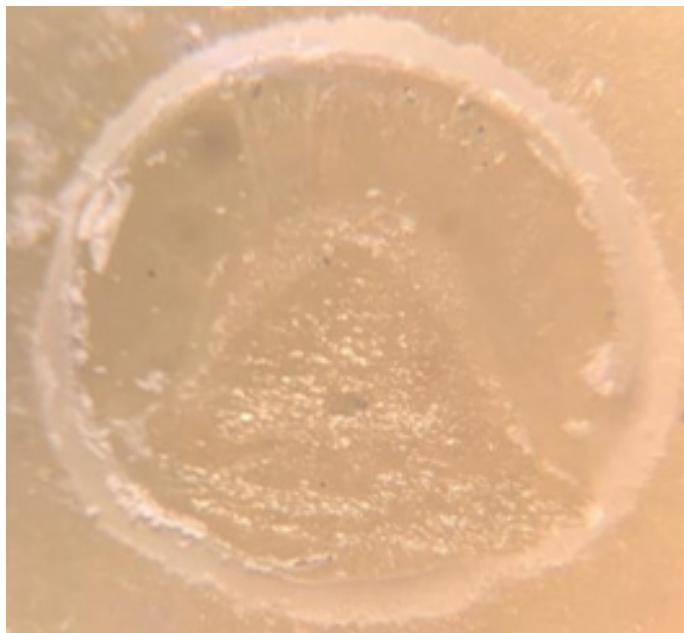


Figure 5. Predominantly Adhesive (ceramic).

Discussion

The present study observed that the result of bond strength between feldspathic ceramics and resin cement does not depend on the concentration of hydrofluoric acid, thus confirming the first null hypothesis.

Hydrofluoric acid reacts with silica in the glass matrix of feldspathic porcelain, forming free radicals called hexafluorosilicates, removed from the ceramic surface during washing under running water. Regardless of the acid concentration, irregularities on the ceramic surface occur due to the chemical reaction between the acid and silica, as shown by the study by Butze *et al.*³ and Guler *et al.*¹²

On the other hand, Sundfeld *et al.*,¹³ tested different hydrofluoric acid concentrations (1, 5 and 10%) on a vitreous ceramic, and shows that the 10% concentration caused greater removal of the vitreous matrix when compared to the concentrations of 1 and 5%. The ceramic used was lithium disilicate which, despite having a vitreous matrix, has a lower amount of silica in its composition than feldspar porcelain, thus requiring a higher concentration of HF to cause retention.

The results presented in the present study, the different concentrations obtained the same failure patterns within the same application time, suggesting that the bond strength between them was similar.

Regarding the variable time of application of hydrofluoric acid, this study showed that feldspathic ceramics, when submitted to application for a time of 80 seconds obtained higher bond strength than the times of 40 and 60 seconds, rejecting, therefore, the second null hypothesis.

Hydrofluoric acid reacts preferably with ceramic silica particles, creating retentive microchannels. Over time,

the acid reacts with the vitreous matrix, causing a partial dissolution, increasing the formation of retentive channels, providing bond strength. In the present study, feldspathic ceramics was chosen, which has in its composition very high vitreous matrix, being more sensitive to the use of conditioning with hydrofluoric acid, therefore that when the conditioning time was increased, there was higher bond strength between resin cement and feldspathic ceramics.

Corroborating this study, Guler *et al.*¹² evaluated the effect of different times (30s, 60s, 120s and 180s) of conditioning with hydrofluoric acid with a concentration of 9.6% in acid-sensitive porcelain. The authors concluded that conditioning with HF for 120s provided adequate bond strength between porcelain and cement.

In different circumstances, Zogheib *et al.*¹⁴ evaluated the conditioning with hydrofluoric acid with a concentration of 4.9% for four different times: 20s, 60s, 90s and 180s. The highest values were found in the 90s and 180s groups. Showing that the increase in the conditioning time affected the resistance of the lithium disilicate-based vitreous ceramic.

However, in Campos *et al.*⁴ study, when conditioning with HF at a concentration of 10% over 30 seconds and 2 minutes in a fluorapatite and feldspar-free leucite vitreous ceramic, it was found that the result of the bond strength between ceramic and resin cement was similar. This opposition is therefore given the ceramic used has quantity, size and type of inorganic molecules different from feldspathic ceramics, used in our research.

It is significant to mention that the excess of acid etching can generate a structural fragility in the ceramic piece, causing mechanical failures. Hence, it cannot be said that the solution for increasing the bond strength between resin cement and acid-sensitive ceramics is to increase the conditioning time without caution.¹⁵ Assessing the consequences of damage to the ceramic restoration is less harmful to obtain a failure in cementation than fracture in the porcelain, thus to lose the piece completely.

It is known that the use of HF in vitreous matrix porcelain provides the necessary surface roughness for micromechanical bonding. Although there was no significant difference using acid of different concentrations, according to the results, there was a difference in bond strength when the conditioning time was increased.^{16,17}

Under the failure pattern analysis, 2/3 of the specimens had adhesive-type failure and 1/3 of the specimens had mixed-type failure. The specimens that showed mixed cement and mixed ceramic failures prove better bonding properties than specimens with adhesive failures, as stated by Lodi, Marks e Borba¹⁸ in their study, which had similarity in the values of bond resistance between the groups in the shear test, but there was a difference in failure mode. The group that presented higher cement/ceramic resistance obtained more frequency

of mixed and cohesive failures.

Restorations with this type of ceramic have multifactorial failure means and the evaluation methods presented provide partial data, since the oral cavity is a complex environment, being difficult to reproduce all its variables in a mechanical test with an in vitro research. It is important to emphasize that the technique for microshear testing is very sensitive and failures resulting from this can happen. From this study it becomes evident the need to maintain the precision of the time of acid application and further studies are needed to

analyze with fractography tests to determine acceptable limits for the resistance of feldspathic ceramic through acid etching.

Conclusions

It can be concluded from this study that the different concentrations of hydrofluoric acid did not significantly influence the bond strength between ceramics/cement. In addition, the increase in conditioning time with hydrofluoric acid significantly affected the bond strength between ceramics and cement.

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Mini Curriculum and Author's Contribution

- Ricardo Huver de Jesus - DDS;Phd student. Contribution: Design of the study, data interpretation, writing of case report and critical review and final approval. ORCID: 0000-0002-2905-8068
- Bianca Colombi Furlam - DDS. Contribution: Data interpretation, writing of case report and critical review and final approval. ORCID: 0000-0002-9470-0512
- Camila Maciel Martinusse - DDS. Contribution: Data interpretation, writing of case report and critical review and final approval. ORCID: 0000-0002-1466-3548
- Hiorrana Carla Vasconcelos Inácio - DDS. Contribution: Data interpretation, writing of case report and critical review and final approval. ORCID: 0000-0002-4729-3360
- Jefferson David Melo de Matos - DDS;Phd student. Contribution: Design of the study, data interpretation, writing of case report and critical review and final approval. ORCID: 0000-0003-4507-0785
- Leonardo Jiro Nomura Nakano - DDS;Phd student. Contribution: Data interpretation, writing of case report and critical review and final approval. ORCID: 0000-0001-6786-862X
- Laura Patrícia Nadal Ortiz - DDS;MSc student. Contribution: Data interpretation, writing of case report and critical review and final approval. ORCID: 0000-0002-3577-0019
- Marco Antonio Bottino - DDS;Phd. Contribution: Writing of case report and critical review and final approval. ORCID: 0000-0003-0077-3161
- Lucas Campagnaro Maciel - DDS;Phd. Contribution: Design of the study, data interpretation, writing of case report and critical review and final approval. ORCID: 0000-0002-7687-4535

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Corresponding author:

Jefferson David Melo de Matos

E-mail: matosjefferson19@gmail.com

