Effect of Hydrofluoric Acid Concentration on Resin Adhesion to a Feldspathic Ceramic

Andressa Borin Venturini\textsuperscript{a} / Catina Prochnow\textsuperscript{b} / Dagma Rambo\textsuperscript{c} / Andre Gundel\textsuperscript{d} / Luiz Felipe Valandro\textsuperscript{e}

Purpose: To evaluate the effect of different concentrations of hydrofluoric acid (HF) on the contact angle and the resin bond strength durability to feldspathic ceramic.

Materials and Methods: To evaluate the contact angles of distilled water on etched feldspathic ceramic, 25 specimens (12 × 10 × 2.4 mm) of VitaBlocks Mark II were used, divided into 5 groups (n = 5): one unconditioned control (UC) group with no ceramic surface treatment, and 4 other groups that were etched for 60 s with different concentrations of HF: 1% (HF1), 3% (HF3), 5% (HF5) and 10% (HF10). The bond testing utilized 40 ceramic blocks (12 × 10 × 4 mm) that were fabricated and subjected to the same surface treatments as previously mentioned (excluding the control). The etched surfaces were silanized and resin cement was applied. After 24 h, the blocks were sectioned to produce bar specimens that were divided into two groups, non-aged (immediate testing) and aged (storage for 230 days + 12,000 thermocycles at 5°C and 55°C), and subjected to microtensile testing (μTBS). Micromorphogical analysis of the treated surfaces was also performed (atomic force and scanning electron microscopy). One-way ANOVA and Tukey’s tests were applied for data analysis.

Results: UC had the highest contact angle (61.4°), whereas HF10 showed the lowest contact angle (17.5°). In non-aged conditions, different acids promoted statistically similar bond strengths (14.2 to 15.7 MPa) (p > 0.05); in terms of bond durability, only the bond strength of the HF1 group presented a statistically significant decrease comparing before and after aging (14.5 to 10.2 MPa).

Conclusion: When etched with 3%, 5%, or 10% hydrofluoric acid, the ceramic tested showed stable resin adhesion after long-term aging.

Keywords: acid etching, porcelain, microtensile bond strength, topographical changes, wettability, contact angle.

J Adhes Dent 2015; 17: 313–320. Submitted for publication: 05.06.14; accepted for publication: 23.06.15
doi: 10.3290/j.jad.a34592

a PhD Student, Graduate Program in Oral Science (Prosthodontics Unit), Faculty of Odontology, Federal University of Santa Maria, Rio Grande do Sul State, Santa Maria, Brazil. Hypothesis, experimental design, performed the experiments and statistical evaluation, wrote the manuscript.

b PhD Student, Graduate Program in Oral Science (Prosthodontics Unit), Faculty of Odontology, Federal University of Santa Maria, Rio Grande do Sul State, Santa Maria, Brazil. Performed the experiments, wrote the manuscript.

c Dentist, Faculty of Odontology, Federal University of Santa Maria, Rio Grande do Sul State, Santa Maria, Brazil. Performed the experiments.

d Associate Professor, School of Physics, Federal University of Pampa, Rio Grande do Sul State, Bage, Brazil. Performed microtopographical analysis under atomic force microscopy, proofread the manuscript.

e Associate Professor, Head of MSciD/PhD Graduate Programs in Oral Science, Prosthodontic Unit, Faculty of Odontology, Federal University of Santa Maria, Rio Grande do Sul State, Santa Maria, Brazil. Idea, hypothesis, experimental design, performed statistical evaluation, contributed substantially to discussion, proofread the manuscript.

Correspondence: Professor Luiz Felipe Valandro, Federal University of Santa Maria, Faculty of Odontology, Prosthodontics Unit, R. Floriano Peixoto, 1184, 97015-372, Rio Grande do Sul State, Santa Maria, Brazil. Tel: +55-55-3220-9276, Fax: +55-55-3220-9272. e-mail: tfvalandro@hotmail.com

Continual technological advances in adhesive dentistry have enabled indirect restorations that use a minimally invasive approach, provide excellent mechanical properties, and produce satisfactory esthetic results. The clinical performance of indirect restorations made from silica-based ceramics (feldspathic ceramic) is based on a durable bond between resin cements, ceramic materials, and dental tissues.\textsuperscript{14,16} For adhesive cementation procedures, the enamel,\textsuperscript{24} dentin surfaces,\textsuperscript{24} and ceramic surfaces\textsuperscript{30} must be properly conditioned. Preparation of the ceramic surface for bonding requires etching with hydrofluoric acid, in which both concentration and duration of the etching influence the bond strength.\textsuperscript{2,3,7}

Silica-based ceramics, which are acid sensitive, undergo surface dissolution when exposed to hydrofluoric acid, which selectively attacks the glassy phase of these ceramics, exposing silicon dioxide (SiO\textsubscript{2}) and yielding topographic changes that contribute to micromechanical retention\textsuperscript{8} and chemical bonding when using a silane coupling agent and resin cement.\textsuperscript{29} The use of only one of these methods, either hydrofluoric acid etching or silanization, can be insufficient to promote high-strength, stable bonding.\textsuperscript{22,38,41}
Surface energy is a physical phenomenon that can be modified by acid etching and silanization of the ceramic surface, and which might influence the bond strength to resin cement. Both conditioning methods, etching and silanization, promote cement wettability on the ceramic surface, improving the interfacial contact with resin cements. An increase in the numbers and types of irregularities on the etched/silanized ceramic surfaces has been associated with enhancing the bond strength. Thus, increasing the area and the surface free energy reduces the contact angle, facilitates penetration of bonding agents and, consequently, increases the wettability and adhesive potential.

Another potential factor that affects adhesion to ceramics is the hydrolysis and degradation of the adhesive interface in the presence of moisture. Water storage and thermocycling have been described as detrimental to the silane-ceramic bond.

As previously mentioned, ceramic surface etching with hydrofluoric acid creates micromorphological alterations that promote mechanical interlocking; however, acid etching may also weaken the ceramic surface, with a progressive increase in weakening as a function of the concentration of the hydrofluoric acid used for etching. There is clear evidence detailing the modifications of surface defects as a function of acid concentration and etching time. The latter study found a significant weakening effect on flexural strength with increasing acid concentrations.

The use of hydrofluoric acid has been questioned due to its possible hazardous and caustic effects on soft tissues. Therefore, the use of low concentrations of this acid can be considered advantageous not only in terms of patients’ health but also especially that of clinicians, who often deal with adhesive cementation.

Although the adhesion protocol of resin cement to feldspathic ceramic is well established in the literature, controversy still exists regarding the optimal concentration of hydrofluoric acid necessary to promote durable bond strengths without weakening the ceramics. The question is: do different hydrofluoric acid concentrations promote different resin bond strengths?

Therefore, the present study 1) evaluated the effect of different hydrofluoric acid concentrations on the contact angle and the bond strength between resin cement and a feldspathic ceramic and 2) examined the influence of storage and thermocycling on the bond strength. The research hypotheses were: 1. Surface conditioning of feldspathic ceramic with hydrofluoric acid reduces the contact angle; 2. Different acid concentrations promote different bond strengths; 3. Thermocycling/storage reduces the mean bond strength values.

Table 1  Surface conditioning for contact angle and microtensile bond strength (μTBS)

<table>
<thead>
<tr>
<th>Codes</th>
<th>Surface conditioning for contact angle analysis</th>
<th>Surface conditioning for μTBS</th>
<th>Storage condition of the specimens for μTBS</th>
<th>Code for μTBS groups</th>
</tr>
</thead>
<tbody>
<tr>
<td>HF10</td>
<td>Etching with 10% hydrofluoric acid*</td>
<td>Without aging</td>
<td>HF10-baseline</td>
<td></td>
</tr>
<tr>
<td>HF1</td>
<td>Etching with 1% hydrofluoric acid**</td>
<td>Without aging</td>
<td>HF1-baseline</td>
<td></td>
</tr>
<tr>
<td>HF3</td>
<td>Etching with 3% hydrofluoric acid**</td>
<td>Without aging</td>
<td>HF3-baseline</td>
<td></td>
</tr>
<tr>
<td>HF5</td>
<td>Etching with 5% hydrofluoric acid**</td>
<td>Without aging</td>
<td>HF5-baseline</td>
<td></td>
</tr>
<tr>
<td>UC</td>
<td>Unconditioned control</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

* Condac Porcelana 10%, FGM; Santa Catarina, Brazil. ** Experimentally formulated, FGM.

Materials and Methods

Contact Angle Measurement

The contact angles were evaluated using 25 ceramic disks (12 x 10 x 2.4 mm) prepared from prefabricated ceramic blocks (VITA Mark II for Cerec/inLab, 2M2C/I12, Vita Zahnfabrik; Bad Säckingen, Germany). The ceramic blocks were sectioned using a diamond disk (Buehler-Series 15LC Diamond, Buehler; Lake Bluff, IL, USA) at low speed under water cooling in a sectioning machine (Isomet 1000, Buehler). The surface to be analyzed was ground sequentially using 400-, 600-, and 1200-grit silicon carbide papers (3M ESPE; St Paul, MN, USA). Then, all samples were cleaned in an ultrasonic device (Vitasonic, Vita Zahnfabrik) for 10 min using isopropyl alcohol.

The ceramic samples were randomly divided into 5 groups (n = 5) according to the surface conditioning method (Table 1). The experimental ceramic surfaces were etched using the different concentrations of hydrofluoric acid and the same procedures: etching for 60 s, rinsing with air-water spray for 30 s, drying, and ultrasonic cleaning in distilled water for 5 min.
The contact angle by the sessile drop technique was measured using a goniometer (Drop Shape analysis, model DSA 30S, Krüss; Hamburg, Germany), which was connected to a computer with dedicated software (DSA3, V1.0.3-08, Krüss) to assess the contact angles and surface energy. At room temperature (± 24°C), one drop (11 μl) of distilled water was placed at the center of the untreated and treated ceramic surfaces (Table 1) using a syringe. The contact angle was measured after 5 s.19

Microtopographical SEM and AFM Analysis
Two samples of each group were sputter coated with gold-palladium alloy prior to being examined in a scanning electron microscope (SEM; JEOL-JSM-T330A, JEOL; Tokyo, Japan) at 1000X and 3000X magnifications. Atomic force microscopy (AFM) (Agilent Technologies 5500 equipment; Chandler, AZ, USA) was used to obtain images. The images (40 μm × 40 μm) were collected using PPP-NCL probes (Nanosensors, Force constant = 48 N/m) in non-contact mode. AFM micrographs were analyzed using scanning probe microscopy data analysis software (Gwyddion version 2.33, GNU, Free Software Foundation; Boston, MA, USA).

Microtensile Bond Strength
Forty ceramics blocks (12 × 10 × 4.3 mm) were prepared as described above. Impressions were made from each ceramic block using addition silicone putty (Elite HD, Zhermack; Badia Polesine, Italy, batch # 122842) to fabricate a mold with a 3-mm gap between the upper portion of the mold and the surface of the ceramic block to allow for the controlled application of resin cement. Prior to etching and silanization, all blocks were ultrasonically cleaned for 5 min using isopropyl alcohol.

The ceramic blocks were randomly allocated into 4 groups (n = 10), according to the surface conditioning method (Table 1). The ceramic surfaces were etched with the appropriate concentration of hydrofluoric acid, using the same procedures as described previously. Then, a silane coupling agent (ESPE-Sil, 3M ESPE) was applied on all of the conditioned ceramic surfaces and allowed to sit for 5 min, as recommended by the manufacturer.

Each treated ceramic block was placed in its silicon mold, with the cementation face exposed and untouched. The resin cement (RelyX ARC, 3M ESPE) was mixed following the manufacturer’s instructions and injected onto the treated surface of the ceramic block using a centrix syringe (DFL; Rio de Janeiro, Brazil).26,29 The cement in the treated surface of the ceramic block using a centrix mold, with the cementation face exposed and untouched. The resin cement (RelyX ARC, 3M ESPE) was mixed following the manufacturer’s instructions and injected onto the treated surface of the ceramic block using a centrix syringe (DFL; Rio de Janeiro, Brazil).26,29 The cement in the treated surface of the ceramic block using a centrix mold, with the cementation face exposed and untouched. The resin cement (RelyX ARC, 3M ESPE) was mixed following the manufacturer’s instructions and injected onto the treated surface of the ceramic block using a centrix syringe (DFL; Rio de Janeiro, Brazil).26,29 The cement in the treated surface of the ceramic block using a centrix mold, with the cementation face exposed and untouched. The resin cement (RelyX ARC, 3M ESPE) was mixed following the manufacturer’s instructions and injected onto the treated surface of the ceramic block using a centrix syringe (DFL; Rio de Janeiro, Brazil).26,29

Each specimen was fixed with cyanoacrylate gel (SuperBonder Gel, Loctite; São Paulo, Brazil) to the rods of a device adapted for this test. The specimens were positioned parallel to the long axis of the device in order to reduce the bending stresses. The device was fixed in the universal testing machine (EMIC DL-1000; Santa Maria, Brazil) as parallel as possible to the application of the tensile load, and testing was performed at a crosshead speed of 1 mm/min.

The bond strength was calculated according to the 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 interface area of the specimen (mm²), as measured with a digital caliper before the test.

All specimens submitted to the microtensile test were analyzed under a light microscope (Discovery V20, Carl Zeiss; Gottingen, Germany) at 50X to 200X magnification. Some specimens were selected for analysis under a scanning electron microscope (JSM-6360, JEOL; Tokyo, Japan) at 90X, 1000X and 3000X magnification. Failures were classified into 3 types: 1) predominantly adhesive failure in the interfacial region between the resin cement and ceramic (ADHES); 2) cohesive failure in the cement (COHES-cem); 3) cohesive failure in the ceramic (COHES-cer).

Data Analysis
The block was used as the experimental unit in the microtensile data analysis (10 blocks per group). Thus, the mean values of the samples from the each block were taken for data analysis. Statistical analysis was performed using the Statistix 8.0 for Windows (Analytical Software; Tallahassee, FL, USA). The Anderson-Darling test was applied to test for nor-
mal distribution, and the Bonferroni test was used to verify homoscedasticity. Cohesive failures were excluded from the statistical analysis, since those failures were determined as not representing the real bond strength. Pre-test failures were also excluded from the statistical analysis.

One-way ANOVA and post-hoc Tukey’s test were used to evaluate the contact angle data \( (\alpha = 0.05) \). Bond strength data from the non-aged and aged conditions were separately submitted to one-way ANOVA and Tukey’s test \( (\alpha = 0.05) \). In addition, the groups were compared 2-2 to elucidate the isolated effect of storage for each surface treatment using Student’s t-test \( (p < 0.05) \).

**RESULTS**

**Contact Angle**

Different conditioning methods had a statistically significant influence \( (p < 0.0001) \) on the contact angle results. The unconditioned group (UC) achieved the highest contact angles. The lowest contact angle values were HF1, HF3, HF5, and HF10 (in descending order), which correspond to etching with hydrofluoric acid in 1%, 3%, 5% and 10% concentrations, respectively. Figure 1 presents the values and representative contact angle images for each different surface conditioning method.

**Microtopographical Analysis**

The SEM and AFM analyses (Fig 2) indicated that higher concentrations of HF acid promoted deeper and more evident craters and pits, as shown in Figs 2Q to 2T (10% HF). However, slight topographical changes were found for HF1% (Figs 2E to 2H) when compared to the untreated condition (Figs 2A to 2D).

**Bond Strength**

Means and standard deviations of the bond strengths are presented in Table 2. The factor “conditioning” \( (p = 0.8912) \) was not statistically significant for the non-aged condition, but it was statistically significant \( (p = 0.0033) \) after aging. For the aged groups, HF1 had the lowest mean bond strength values when compared to the other acid concentrations. Additionally, HF1 was the only group that presented a statistically significant decrease in the bond strength after aging when compared to the results of the similarly treated non-aged group.

Table 3 presents the number and percentages of specimens lost either during thermocycling or cutting prior to microtensile bond strength testing for the 8 experimental groups.

The numbers and percentages of the failure types for the specimens submitted to microtensile testing are presented in Table 4. Failure analysis demonstrated that all groups showed predominantly adhesive failures (96.2%), ie, the majority of fractured surfaces exhibited the presence of some resin cement on the ceramic. SEM micrographs representing the failure types of the debonded specimens are shown in Fig 3.

**DISCUSSION**

The present study found that hydrofluoric acid etching significantly reduced the contact angle values, therefore confirming the first hypothesis. Moreover, hydrofluoric acid concentrations of 3%, 5%, and 10% promoted similar and stable resin bond strengths to a feldspathic ceramic, while the 1% acid generated unstable bond strengths, meaning that the 2nd and 3rd hypotheses were partially accepted.

To promote effective adhesion between resin cements and ceramics based on silica (feldspathic ceramics), surface conditioning with hydrofluoric acid is necessary for a reaction with the ceramic glass matrix to promote a micromechanically retentive surface.\(^6\) This surface roughness obtained by acid etching increases the surface energy; the subsequent application of a silane coupling agent enhances the bonding potential of resin cement to feldspathic ceramic.\(^7,19\) However, adequate performance of silane is limited by water hydrolysis, which damages the adhesion.\(^18\) Thus, silane drying and post-silanization heat treatment have been performed to eliminate solvents and optimize silane’s effect on the adhesion resin to ceramic.\(^8,17\)
The contact angle analysis of a liquid on a substrate consists of using a medium to determine the surface energy and wettability of a given surface. An increase in surface area caused by HF etching induces an increase in wettability, which is associated with a lower contact angle and greater bonding potential.

In this study, HF etching decreased the contact angle in all groups, with the lowest mean contact angle values found for 10% hydrofluoric acid (Fig 1). The current results are in agreement with a study by Jardel et al., which evaluated the effect of surface modifications with 10% HF gel on the surface energy of two feldspathic ceramics. Those authors concluded that, after conditioning, ceramics showed smaller contact angles when compared to the groups that were only polished. However, despite the lower contact angle values for HF10 when compared to
HF1 and HF3, the current results did not find an effect on adhesion, since HF3, HF5, and HF10 promoted similar bond strengths. These results might indicate that resin adhesion to this type of ceramic is micromechanical in nature, ie, that microtopographical changes (Fig 2) play an important role in adhesion.

Only the group etched with 1% HF showed a significant decrease in bond strength after aging/thermocycling. Higher bond strengths were obtained when ceramic surfaces were conditioned with HF3, HF5, and HF10, without any statistically significant decrease after aging. Similar results were found by Amaral et al, who observed that, independent of the storage condition, bond strength values were significantly higher for groups etched with 9% and 4% hydrofluoric acid gels. An important aspect in the current investigation is that the tested acids had the same viscosity and were produced by the same manufacturer, which is different from the Amaral et al study, which assessed acids with different concentrations and viscosities. Thus, the present study prevented the possible effect of viscosity on the etching pattern of the ceramic surface, controlling the effects of different concentrations of acid on the contact angle and adhesion.

Representative micrographs clearly demonstrate the effect of increasing the HF acid concentration on the surface topography. However, no differences in the bond strength values were found between concentrations of 10%, 5%, and 3%. It is possible that bond strength is related to the efficacy of the silane coupling agent to promote chemical bonding between the resinous materials and ceramics in association with the topographical changes from etching. The silane coupling agents coat the silicon oxides present in the ceramic and bind to the organic matrix of resin cements via siloxane bonds.

Brentel et al evaluated the durability of the bond strength between resin cement and a feldspathic ceramic submitted to different etching protocols (10% HF acid gel and 1.23% acidulated phosphate fluoride), with and without silane application. They found that hydrofluoric acid treatment followed by silanization of the silica-based ceramic provided greater bond strength values.

HF3 and HF5, the current results did not find an effect on adhesion, since HF3, HF5, and HF10 promoted similar bond strengths. These results might indicate that resin adhesion to this type of ceramic is micromechanical in nature, ie, that microtopographical changes (Fig 2) play an important role in adhesion.

Table 2: Means and standard deviation of the bond strength data (MPa)

<table>
<thead>
<tr>
<th>Surface treatment</th>
<th>Storage condition before testing</th>
<th>p-value**</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Without aging*</td>
<td>With aging*</td>
</tr>
<tr>
<td>HF10</td>
<td>15.7 ± 2.8a</td>
<td>13.6 ± 2a</td>
</tr>
<tr>
<td>HF1</td>
<td>14.5 ± 3a</td>
<td>10.2 ± 1.7b</td>
</tr>
<tr>
<td>HF3</td>
<td>14.2 ± 3.3a</td>
<td>13 ± 1.5a</td>
</tr>
<tr>
<td>HF5</td>
<td>14.9 ± 2a</td>
<td>13 ± 2.2a</td>
</tr>
</tbody>
</table>

*Same superscript lowercase letters indicate no significant differences for comparison in the same columns (Tukey’s test, α = 5%). **Significant at p < 0.05. HF10: 10% hydrofluoric acid; HF1: 1% HF; HF3: 3% HF; HF5: 5% HF.

Table 3: Number and percentage (%) of pre-test failures (PTF) during cutting and thermocycling (TC) and final number of specimens submitted to the microtensile test (μTBS)

<table>
<thead>
<tr>
<th>Groups</th>
<th>Number and % of PTF during cutting</th>
<th>Number and % of PTF during TC</th>
<th>Number and % of tested specimens in μTBS</th>
</tr>
</thead>
<tbody>
<tr>
<td>HF10 baseline</td>
<td>69 (46)</td>
<td>0 (0)</td>
<td>81 (54)</td>
</tr>
<tr>
<td>HF10 aged</td>
<td>66 (44)</td>
<td>0 (0)</td>
<td>84 (56)</td>
</tr>
<tr>
<td>HF1 baseline</td>
<td>86 (57.3)</td>
<td>0 (0)</td>
<td>64 (42.7)</td>
</tr>
<tr>
<td>HF1 aged</td>
<td>85 (56.7)</td>
<td>0 (0)</td>
<td>65 (43.3)</td>
</tr>
<tr>
<td>HF3 baseline</td>
<td>82 (54.7)</td>
<td>0 (0)</td>
<td>68 (45.3)</td>
</tr>
<tr>
<td>HF3 aged</td>
<td>82 (45.3)</td>
<td>0 (0)</td>
<td>68 (45.3)</td>
</tr>
<tr>
<td>HF5 baseline</td>
<td>78 (52)</td>
<td>0 (0)</td>
<td>72 (48)</td>
</tr>
<tr>
<td>HF5 aged</td>
<td>75 (50)</td>
<td>0 (0)</td>
<td>75 (50)</td>
</tr>
</tbody>
</table>

For abbreviations, see Table 2.

Table 4: Number and percentages for type of fractures in the beam specimens submitted to the microtensile test

<table>
<thead>
<tr>
<th>Groups</th>
<th>Total number of tested samples</th>
<th>Type of Fracture</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>ADHES</td>
<td>COHEScem</td>
</tr>
<tr>
<td>HF10 baseline</td>
<td>81</td>
<td>77 (95.1%)</td>
</tr>
<tr>
<td>HF10 aged</td>
<td>84</td>
<td>79 (94%)</td>
</tr>
<tr>
<td>HF1 baseline</td>
<td>64</td>
<td>61 (95.3%)</td>
</tr>
<tr>
<td>HF1 aged</td>
<td>65</td>
<td>64 (98.5%)</td>
</tr>
<tr>
<td>HF3 baseline</td>
<td>68</td>
<td>65 (95.6%)</td>
</tr>
<tr>
<td>HF3 aged</td>
<td>68</td>
<td>67 (98.5%)</td>
</tr>
<tr>
<td>HF5 baseline</td>
<td>72</td>
<td>68 (94.4%)</td>
</tr>
<tr>
<td>HF5 aged</td>
<td>75</td>
<td>74 (98.7%)</td>
</tr>
<tr>
<td>Total</td>
<td>577</td>
<td>555 (96.2%)</td>
</tr>
</tbody>
</table>

ADHES: Adhesive fracture at cement/ceramic interface; COHEScem: cohesive fracture of the resin cement; COHEScer: cohesive fracture of the ceramic. For other abbreviations, see Table 2.
minimal change in topographical patterns after 1% HF acid etching promoted significantly lower bond strengths when aged compared to the other concentrations used for acid etching. When applied in the HF1 group, the silane was unable to improve the bond strength values due to insufficient micromechanical retention promoted by the minimal acid etching. Therefore, the current findings agree with those of other authors, who state that surface alterations are crucial for enhancing the bond strength. At the same time, even though the bond strength decreased statistically significantly, the HF1 group reached 10 MPa after aging, which could perhaps be sufficient from the clinical point of view. Thus, further research should be conducted to evaluate the in vitro performance of feldspathic ceramic restorations conditioned by hydrofluoric acid with different concentrations.

Regarding pre-test failures, these occurred only when cutting the ceramic-cement assemblies and can be seen as a limitation of the test protocol, as can the presence of cohesive failures. These can be explained by the stresses generated during specimen preparation, since the ceramic block has high strength and is resistant to cutting, inducing cracks in the ceramic during sectioning. Moreover, microtensile testing is only applicable when the bond strength values were higher than the approximately 5 MPa that is allowed for sectioning the specimens. These features might explain the number of pre-test failures observed in the present study and why the group without conditioning could not be tested, since in a pilot study, it was not possible to produce specimens after cutting the blocks of this group.

In terms of using mechanical testing to evaluate the real bond interaction between the materials/substrates/adherent, microtensile bond strength testing is more appropriate for evaluating the adhesive capabilities, as this kind of test generates more a homogeneous stress distribution at the interface than do other mechanical tests. Therefore, this “micro” test reduces the likelihood of intrinsic defects at the adhesive interface, thus affecting the results of the test. According to Griffith’s theory, the tensile strength of uniform materials decreases when the specimen size is increased, due to a higher probability of critical-sized defects than in smaller specimens. Hence, the failure analysis in the present study demonstrated that the most common failure mode of specimens involved the adhesive interface, indicating a more realistic assessment of the bond itself. Moreover, specimens that presented cohesive failures were excluded from the statistical analysis, since they do not represent actual values of adhesion and may be related to a non-uniform stress distribution or critical defect in the cement bulk.

The sample had a small area, so that thermocycling effects had a greater influence on its surface, which might have contributed to the decrease in the bond strength values. In vitro studies have inherent limitations, and some clinical conditions cannot be simulated. Thus, caution should be used when extrapolating the current findings to clinical situations. Further studies that utilize more realistic clinical situations should be conducted, such as evaluating the effect of hydrofluoric acid concentration on the adhesion to ceramic when acid etching is performed on the intaglio surface of inlay restorations used in posterior teeth and applying mechanical cycling.

CONCLUSIONS

Based on the present results, the following conclusions may be drawn:

- Of the four tested hydrofluoric acid concentrations for etching the feldspathic ceramic, 3%, 5%, and 10% promoted stable bond strengths.
• Greater concentrations of hydrofluoric acid produced more intense alterations of the surface topography of feldspathic ceramics, creating mechanical interlocking and smaller contact angles.

ACKNOWLEDGMENTS

CNPq (Brazil) and CAPES (Brazil) supported this study. We thank FGM for producing the different concentrations of hydrofluoric acid. This study was based on a dissertation submitted to the Graduate Program in Oral Sciences, Prostodontic Unit, Faculty of Odontology, Federal University of Santa Maria, Santa Maria, Brazil as part of the requirements for the M.Sci.D. degree (Dr. A. Venturini).

REFERENCES


Clinical relevance: When cementing feldspathic ce- ramic restorations, the inner surface of the res- torations can be etched with 3%, 5%, or 10% hydrofluoric acid for resin bond improvement.