Influence of Surface Treatment of Lithium Disilicate on Roughness and Bond Strength

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Purpose: To evaluate the effect of different concentrations of hydrofluoric acid (HF) on the surface roughness of a ceramic reinforced by lithium disilicate and on the bond strength formed between the ceramic and self-adhesive resin cement. Materials and Methods: A total of 100 disks of IPS e.max Press ceramic (Ivoclar Vivadent) reinforced by lithium disilicate were prepared as follows: group 1 remained untreated (control group); in groups 5-20, 5-40, and 5-60, the surfaces were etched with 5% HF for 20, 40, and 60 seconds, respectively; in groups 10-20, 10-40, and 10-60, the surfaces were etched with 10% HF for 20, 40, and 60 seconds, respectively; and in groups 10-20P, 10-40P, and 10-60P, the surfaces were etched with 10% HF for 20, 40, and 60 seconds, respectively, followed by treatment with 37% phosphoric acid for 5 seconds. Surface roughness and bond strength were analyzed with confocal microscopy and microshear testing, respectively. The values obtained were statistically analyzed using paired t test and two-way ANOVA followed by Tukey post hoc test at a 5% significance level. Results: Surface roughness was influenced by the concentration and exposure time of acid applied (P < .05) and by the combination of these two factors (P < .05). Treatment with 10% HF for 40 seconds (group 10-40) achieved the highest roughness value. In contrast, bond strength was affected only by the acid exposure time (P < .05). Conclusion: Conditioning of lithium disilicate ceramics can change the surface morphology, thereby affecting bond strength with resin cement. Int J Prosthodont 2020;33:212–216. doi: 10.11607/ijp.6453

Restorative dentistry combines both esthetic and conservative concepts. The introduction of acid-etchable ceramics in the early 1980s made it possible to provide greater clinical longevity and esthetic satisfaction for patients while also preserving tooth structure. Within this context, ceramic laminate veneers and ceramic crowns have been widely used for cosmetic procedures involving the anterior teeth due to the excellent esthetics, durability, and biocompatibility that characterize these materials.1-3

Lithium disilicate is a filler agent of acid-etchable ceramics, and 60% to 65% of its composition includes lithium oxide (Li2O) crystals. Consequently, this filler agent lithium disilicate is more resistant than feldspathic ceramic4; therefore, it is the material of choice for esthetic treatments with or without a framework.5,6 However, lithium disilicate remains sensitive to acid etching, which can alter the morphology of the ceramic and increase its capacity to bond with resin cements. Etching with hydrofluoric acid (HF) at different concentrations and with different application times has been...
The acid reacts with the glassy matrix that contains the silica to form hexafluorosilicates. As this glassy matrix is selectively removed, the crystal structure is exposed and the surface of the ceramic material becomes rough. Consequently, micromechanical retention on the surface of the ceramic is achieved.\(^1\)

This etched surface also provides a higher surface energy prior to the application of a silane agent, which enhances chemical bonding between the ceramic and a resin material, thereby increasing the functional characteristics of the ceramic.\(^1\) Furthermore, this process promotes greater wettability over a ceramic surface, and this increases the bonding capacity of the surface with resin cements.\(^1\)–\(^3\)

Currently, there is no consensus regarding the optimal acid concentration and etching time for the treatment of ceramic surfaces.\(^1\)\(^8\),\(^1\)\(^9\) Moreover, the potential for fractures to form is an important consideration, especially when acids are applied at high concentrations and/or for extended exposure times. The production of insoluble fluoride salts from silica can also interfere with and/or for extended exposure times. The production of insoluble fluoride salts from silica can also interfere with resin cements.\(^2\)\(^0\)

Therefore, the objective of this study was to evaluate the effect of different HF concentrations and application times, with and without an additional etching step with 37% phosphoric acid (H\(_3\)PO\(_4\)), on the surface roughness of a ceramic reinforced by lithium disilicate and also on the strength of the bond formed between the ceramic and self-adhesive resin cement.

## MATERIALS AND METHODS

### Preparation of the Ceramic Disks

A total of 100 ceramic disks of medium opacity (7.0-mm diameter, 2.0-mm thickness) of IPS e.max Press (Ivoclar Vivadent) lithium disilicate, color A2 on the VITA scale, were produced in accordance with the manufacturer’s instructions. Circular patterns in the wax were obtained by fixing them to an inclusion ring (IPS PressVEST Speed Powder, Ivoclar Vivadent) through the sprue. The wax patterns were embedded in an IPS PressVEST Speed Powder coating and, after solidifying, were brought to 850°C in a preheated oven for complete carbonization of the wax. The ceramic ingots were then pressed inside molds in an automatic oven (EP600, Ivoclar Vivadent) at 920°C. After cooling, the samples were cleaned and blasted with 50-μm aluminum oxide (Al\(_2\)O\(_3\)) particles (EDG Soluções) at a pressure of 1 bar. The surfaces of all the samples were standardized by subjecting them to wet polishing with silicon carbide sandpaper with grains of 300, 600, and 1,200 (Norton). The samples were then washed in tap water and placed in a neutralizing substance (IPS Ceramic Neutralizing Powder, Ivoclar Vivadent) for 5 minutes. Thereafter, the samples were divided into 10 groups of 10 samples each.

### Experimental Design

The 10 groups of prepared lithium disilicate disks (n = 10 per group) were treated as follows. The disks in group 1 remained untreated, thus serving as the control group. In groups 5-20, 5-40, and 5-60, the surfaces were etched with 5% HF for 20, 40, and 60 seconds, respectively. In groups 10-20, 10-40, and 10-60, the surfaces were etched with 10% HF for 20, 40, and 60 seconds, respectively. In groups 10-20P, 10-40P, and 10-60P, the surfaces were etched with 10% HF + 37% H\(_3\)PO\(_4\) for 20, 40, and 60 seconds, respectively, followed by treatment with H\(_3\)PO\(_4\) for 5 seconds (Table 1).

### Roughness Analysis

The mean surface roughness (Ra) of each sample was measured using a confocal optical microscope (Axion 700 CSM, Carl Zeiss). Three measurements were obtained at 45-degree intervals on the surfaces of each specimen, and the mean values (in μm) were analyzed. Samples from each group were randomly subjected to a qualitative analysis of surface morphology with a scanning electron microscope (EVO 10, Carl Zeiss).

### Microshear Bond Strength Test

In the microshear testing, a layer of silane bonding agent (RelyX Ceramic Primer, 3M ESPE) was applied inside silicone microtubes (tygons) (Sirona), self-adhesive resin cement (RelyX U200, 3M ESPE) was applied to each ceramic disk for 1 minute, followed by 5 seconds of drying time. Using a Centrix syringe (SDR, Dentply Sirona), self-adhesive resin cement (RelyX U200, 3M ESPE) was applied inside silicone microtubes (tygons) positioned perpendicularly over each ceramic surface. Three tygons were set up (3 mm in height, 0.8 mm in diameter) for each disk in each group (n = 30 per group). The cylinders were cured for 60 seconds with an LED curing light (Bluephase, Ivoclar Vivadent). Excess cement was subsequently removed, and the samples were stored in deionized water for 24 hours at 37°C before being subjected to a microshear test in a universal

## Table 1  The Various Treatments Applied to the Prepared Lithium Disilicate Disks

<table>
<thead>
<tr>
<th>Group</th>
<th>Acid treatment</th>
<th>Exposure time (s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Untreated</td>
<td>0</td>
</tr>
<tr>
<td>5-20</td>
<td>5% HF</td>
<td>20</td>
</tr>
<tr>
<td>5-40</td>
<td>5% HF</td>
<td>40</td>
</tr>
<tr>
<td>5-60</td>
<td>5% HF</td>
<td>60</td>
</tr>
<tr>
<td>10-20</td>
<td>10% HF</td>
<td>20</td>
</tr>
<tr>
<td>10-40</td>
<td>10% HF</td>
<td>40</td>
</tr>
<tr>
<td>10-60</td>
<td>10% HF</td>
<td>60</td>
</tr>
<tr>
<td>10-20P</td>
<td>10% HF + 37% H(_3)PO(_4)</td>
<td>20</td>
</tr>
<tr>
<td>10-40P</td>
<td>10% HF + 37% H(_3)PO(_4)</td>
<td>40</td>
</tr>
<tr>
<td>10-60P</td>
<td>10% HF + 37% H(_3)PO(_4)</td>
<td>60</td>
</tr>
</tbody>
</table>

HF = hydrofluoric acid; H\(_3\)PO\(_4\) = phosphoric acid.
In the images obtained with a scanning electron microscope, significant changes were observed across the surfaces of the disks that were etched with 5% or 10% HF for various exposure times. In particular, a greater number of depressions were observed on the surfaces of the disks exposed to 10% HF compared to the other treatments (Fig 2).

As shown in Fig 1, treatment with 5% HF for 40 seconds produced the highest bond strength value (22.75 ± 1.97 MPa), while the control group presented the lowest bond strength value (5.25 ± 1.29 MPa). In all of the control group specimens, the failures observed were adhesive and/or mixed (Fig 3).

**DISCUSSION**

The variations in the acid concentration and application time were found to influence surface roughness, indicating that these factors are essential aspects in achieving successful adhesion of glass-ceramic restorations. The application of 10% HF achieved the greatest surface roughness, but not the highest bond strength value. This result may be due to the exposure of lithium disilicate crystals following the solubilization and leaching of the glassy matrix by acid and tap water. Ten different surface treatments of ceramic lithium disilicate disks were performed and subsequently analyzed. It has been previously demonstrated that the alterations occurring on ceramic surfaces following treatment with HF represent a dynamic process, and this process is dependent on the constitution of the ceramic, the surface topography, the concentration of the applied acid, and the duration of the acid treatment.21,22

To a great extent, the clinical success of a ceramic restoration depends on the quality and durability of the bonding that occurs between the ceramic surface and resin
is in agreement with Puppin-Rontani et al. Correspondingly, the groups that exhibited the highest mean microshear bond strength were the ones that presented more cohesive cement failures. These results suggest that the bonding of the cement with the ceramic was very efficient. Moreover, these results are consistent with those reported by Fabianelli et al., in which higher bond strength values predominantly exhibited cohesive failure. In the present study, the groups with the lowest mean microshear bond strength presented both adhesive and mixed failures.

The surfaces etched with different concentrations of acid but the same exposure time (groups 5-40 and groups 10-40) exhibited the highest mean bond strength values. However, when the exposure time to HF was increased to 60 seconds, reduced bond strength and roughness were detected. It is possible that the increase in exposure time resulted in the partial destruction of the lithium disilicate crystals present, while the crystals that were exposed on the surfaces etched with HF for only 40 seconds remained intact. These findings are consistent with those previously reported by Kern et al.

In the scanning electron microscope images obtained, there were fewer peaks and valleys on the surfaces of the disks in group 10-60P compared to the disks in group 10-40P. The application of 37% H₃PO₄ furthered destruction of the peaks of the lithium disilicate crystals by HF, and either the redistribution of the displaced material into the valleys or phosphoric acid etching did not produce any phosphorus-containing compounds to be deposited on the surface, or these byproducts did not have a complex structure and could be easily washed off from the phosphoric acid-etched surface.

A limitation of this study is the fact that only one brand of self-adhesive resin cement was used to assess microshear bond strength of lithium disilicate ceramic, and when using self-adhesive resin cement for bonding with a ceramic substrate, it is recommended that the ceramic surface be treated with silane. Therefore, in the present study, all of the treated disks were subjected to an application of silane.
CONCLUSIONS

Surface treatment is essential for microsand bond strength between lithium disilicate ceramics and self-adhesive cement and for creating roughness of the lithium disilicate ceramic. However, acid concentration and exposure time are important factors in surface treatment. The 10% HF acid concentration and exposure time from 20 to 40 seconds showed the best results.

ACKNOWLEDGMENTS

The authors report no conflicts of interest.

REFERENCES


Literature Abstract

The Influence of Different Grafting Materials on Alveolar Ridge Preservation: A Systematic Review

The purpose of this review was to evaluate the effects of different bone substitutes used for alveolar ridge preservation on postextraction dimensional changes. An electronic literature search in MEDLINE (PubMed), Embase (Ovid), and Cochrane (CENTRAL) were performed, in addition to a manual search through all periodontics- and implantology-related journals, up to December 2018. Inverse variance–weighted means were calculated for all the treatment arms of the included trials for the quantitative analysis. A total of 40 randomized controlled trials were included in the quantitative analysis. Dimensional changes were obtained from clinical measurements and three-dimensional imaging. The mean amount of horizontal ridge resorption was 1.52 (standard deviation [SD] 1.29) mm (allograft), 1.47 (SD 0.92) mm (xenograft), 2.31 (SD 1.19) mm (alloplast), and 3.1 (SD 1.07) mm for unassisted healing. Similarly, for all the evaluated parameters, spontaneous healing of the socket led to a higher bone loss rate than with the use of a bone grafting material. Utilization of a bone grafting material for alveolar ridge preservation reduces the resorption process that occurs after tooth extraction. However, minimal differences in resorption rate were observed among allogeneic, xenogeneic, and alloplastic grafting materials.