Evaluation of the Influence of Acid Etching Technique on Surface Roughness and Fracture Strength of Heat-Pressed Lithium-disilicate Veneers

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Abstract

Background  Metal-free ceramic veneers exhibit many desirable properties. However, low fracture strength is a major disadvantage. The success of the veneers depends on optimal tooth preparation and intaglio surface preparation techniques for bonding porcelain substrates to the tooth to ensure clinical success. This study was conducted to evaluate the influence of acid etching technique on surface roughness and fracture strength of veneers.

Methods  The in vitro study was conducted on maxillary central incisors that were prepared to receive the veneers; the specimens were divided into two groups—group A and group B. The intaglio surface of veneer in both groups was etched with 4.6 % of the hydrofluoric acid solution for 30 seconds, in group B specimens were further treated with 33% sodium hypochlorite solution treatment following which the surface roughness was measured and calculated with scanning electron microscope at a magnification of 3000X and profilometer, respectively. The fracture strength of the specimens in both groups was determined by subjecting the tooth with veneer to a universal testing machine; the force was applied until the fracture occurred.

Results  There was a significant difference observed in the surface roughness, fracture strength, and bond strength of the heat-pressed lithium-disilicate veneers treated solely with hydrofluoric acid and samples treated with hydrofluoric acid and phosphoric acid.

Conclusion  Optimizing porcelain surfaces before treatment with various adhesives and luting resins requires an understanding of the involved substrates and materials, as well as a logical and systematic methodology in their manipulation. The intaglio surface of the veneers treated with hydrofluoric acid followed with phosphoric acid has no significant difference observed in the properties of the veneers but has better bonding with the tooth structure.

Keywords
► hydrofluoric acid
► phosphoric acid
► veneers
► scanning electron microscope
► profilometer
► universal testing machine.

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**Introduction**

Heat-pressed lithium disilicate veneer materials exhibit many desirable properties, including biocompatibility, aesthetics, diminished plaque accumulation, low thermal conductivity, abrasion resistance, and color stability. However, brittleness and low tensile strength are weak points of heat-pressed lithium-disilicate veneer materials. Therefore, the clinical successes of heat-pressed lithium-disilicate veneers have been disappointing when compared with metal-ceramic restorations. Available data suggest that conventional metal-ceramic shows a survival rate of approximately 90% at 10 years. Until recently, only small fixed partial dentures made of glass-infiltrated alumina porcelain were recommended for the anterior region. Available data from clinical studies on all-ceramic anterior crowns indicate a success rate of 93 to 100% after 3 years.\(^1\) To achieve all-ceramic fixed partial dentures with appropriate fracture strength, new ceramic core materials were recently introduced into the dental market. Apart from lithium-disilicate glass-ceramic, new zirconia and alumina-based ceramic materials are now available. The mechanical properties of high-performance alumina and zirconia ceramics make them interesting as potential candidates for all-ceramic restorations in high stress-bearing areas. These ceramics are manufactured under optimized industrial conditions, and they are designed to be processed by computer-aided design/manufacturing technologies.\(^2,3\) The purpose of this study was to evaluate the influence of the acid etching technique on surface roughness and fracture strength of heat-pressed lithium-disilicate veneers, with the objectives to determine the influence of hydrofluoric acid and the influence of combined hydrofluoric acid and phosphoric acid solution on the surface roughness, fracture strength of heat-pressed lithium-disilicate.

**Materials and Methods**

**Source of Data Collection**

The in vitro study was conducted on 22 central incisors that were prepared to receive the heat-pressed lithium-disilicate veneers. The 22 specimens were further divided into two groups—group A and group B. The sample selection was made where only extracted maxillary central incisors were used in the study excluding the carious and fractured maxillary central incisors.

**Study Design**

The samples were sent to laboratory for scanning purpose; the scanning electron microscopic was used to capture the image following which profilometer was used to determine the depth and width of acid etched layer.

Samples from each group were then cemented onto the prepared tooth with resin cement following which the samples were subjected to universal testing machine to determine fracture strength.

**Methodology**

The extracted maxillary central incisors were mounted on plaster blocks. Further, the teeth were prepared to receive the heat-pressed lithium-disilicate veneer. The wax pattern was fabricated onto the prepared tooth with the direct method. The wax pattern was subjected to the ceramic unit to obtain heat-pressed lithium-disilicate veneers. The lithium-disilicate ingot size of 3 grams was used. The heat-pressed lithium-disilicate veneers were divided into group A and group B. Group A specimens were subjected to 4.6% of hydrofluoric acid treatment solution for 30 seconds and washed with distilled water for 20 seconds; the surface roughness was examined under a scanning electron microscope for qualitative surface appearance, at a magnification of 3000X. Group B specimens were subjected to 4.6% hydrofluoric acid treatment following which they were treated with 33% phosphoric acid treatment. The surface roughness was calculated using a profilometer. To determine the fracture stress, the specimens in group A and group B were bonded to the tooth structure with a universal bonding agent, following which the fracture strength was determined by subjecting the veneers fixed to tooth to a universal testing machine. At least for 48 hours, all the specimens were stored in distilled water at a temperature of 37 degrees following which a load of 219N load was applied with a 6 mm diameter stainless steel ball placed on the specimen with a cross-head speed of 0.5mm/min. The force was applied at the angulations of 15 degrees relative to the long axis and finally loaded until fracture occurred.

**Statistical Analysis**

Descriptive statistics will be documented by using mean, standard deviation, and confidence intervals. An independent sample t-test was used to compare the surface roughness between heat-pressed ceramic treated with hydrofluoric acid and the combined effect of hydrofluoric acid and phosphoric acid on lithium-disilicate-based veneers.

**Results**

- Table 1 shows a comparison of surface roughness between the study groups. The mean difference between the group 1 and group 2 calculating Ra values was –0.35, with a statistically significant p-value of less than 0.001. The mean difference between the group 1 and group 2 calculating Rq values was –0.38, with statistically significant p-value less than 0.001. The mean difference between the group 1 and group 2 calculating Rz values was –1.11, with statistically significant p-value less than 0.001.

- Table 2 shows the evaluation of fracture resistance using a universal testing machine. The maximum force required to fracture the samples in group 1 and group 2 was 8.5N and 29.10N, respectively, with a compressive stress maximum force 0.13 Mpa group 1 and 0.45 Mpa group 2 followed by a Compressive displacement break standard at 1.57 mm with Compressive stress at break 0.02 Mpa in group 1 and 0.37 Mpa in group 2 with Compressive stress at break 0.09 Mpa. Hence, there is a significant difference in the mean surface roughness values between group 1 and group 2 when determined in a scanning electron microscope followed by calculation of the roughness with a profilometer. There was a significant difference value in
Tooth preparation done on fresh extracted central incisors

Impression of prepared tooth was made from elastomeric impression material (Dentsplyaquaasil soft putty/regular set light body by single putty wash impression technique)

Final impressions were poured in type IV die stone (Kalrock). Once casts were obtained, base formation carried out using type II die stone (Whitestone, Asian chemicals)

22 wax models were prepared following which 22 veneers were obtained

**Group A.** The intaglio surface of the heat pressed veneers were etched with 4.6% of the hydrofluoric acid solution for 30 seconds.

**Group B.** The intaglio surface of the heat pressed veneers were etched with 4.6% of hydrofluoric acid treatment following which they will be treated with 33% Phosphoric acid treatment

### Table 1  Comparison of surface roughness between the study groups

<table>
<thead>
<tr>
<th>Group</th>
<th>n</th>
<th>Mean</th>
<th>Standard deviation</th>
<th>Mean difference</th>
<th>95% Confidence interval of the difference</th>
<th>t</th>
<th>Df</th>
<th>p-Value</th>
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<td>-0.37</td>
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<td>-1.11</td>
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Independent sample t-test.

*p < 0.05 Statistically Significant.
fracture resistance. Where the resistance to fracture was high when treated with phosphoric acid treatment.

**Discussion**

To enhance the fracture strength of heat-pressed lithium-disilicate veneer, the bonding capacity of the restorations was improved by etching the intaglio surface of the veneers with concentrated acid solutions; it was believed that treating the intaglio surface of the veneers will not only improve the bonding capacity but may also increase the surface area that may indirectly give enough space for a luting agent to wet the surface thoroughly to magnify the bonding capacity of the heat-pressed veneer, further improving the fracture strength leading to long term good prognosis of the veneers. The literature review and numerous discussions with manufacturers, laboratory technicians, and researchers concluded that no single specific hydrofluoric acid concentration and their application time exists that is optimal for etching all types of porcelains. As a consequence, it is not surprising that currently recommended hydrofluoric acid concentrations and application times vary significantly. It also appears that there is great possibility the veneer may be under-etched and over-etched based on the time consumed for etching and concentration of the acidic solution. As Calamia suggested years ago, the hydrofluoric acid concentrations and their application times should, ideally, be adjusted depending on the specific nature of the porcelain being treated. In 1998, it was published in the field of medicine other than dentistry that hydrofluoric acid was acknowledged as a hazardous chemical that may be highly toxic in response/reaction. There are many schools of thought believed that the hydrofluoric acid used for etching the intaglio surface of veneer should be cleaned thoroughly before the application of the silane coupling agent and bonding agent; the cleansing protocol is must as the hydrofluoric acid has various health hazards that may be acute or chronic in nature that may lead to various local or systemic disorders. The data published in literature studies, scientific case reports, medical opus, industrial counsel, and manufacturer’s information guides has no reports claiming the incidence of the hazardous or harmful effects of hydrofluoric acid. While acute symptoms include skin rashes, nail burn injuries, and chronic symptoms that include systemic toxicity, eye injuries, inhalation, and ingestion-related symptoms that can be fatal in nature. Potential hazards of hydrofluoric acid known from other applications other than dentistry should be considered in dental applications. Especially clinicians, who often work with the hydrofluoric acid while etching, and cementation of heat-pressed lithium-disilicate veneers, should take necessary precautions to avoid possible hazards of hydrofluoric acid. The greatest advantage of the use of hydrofluoric acid on veneers is its easy manipulation with uncomplicated chair-side execution, for the reason being sophisticated conditioning and etching effect. Hence, flushing of the hydrofluoric acid post etching the veneers is must; the use of distilled water is a routine protocol in washing the veneers but studies have also claimed that washing the intaglio surface with distilled water spray for 30 seconds is not adequate nor it will remove the residue of hydrofluoric acid completely from the intaglio surface of the veneers. Many studies in the literature describe that 33% phosphoric acid also plays a major role in clearing the left-out residue of hydrofluoric acid after treating the intaglio surface of the veneers with hydrofluoric acid. It is then incumbent on dentists and ceramists to follow these protocols. In regard to etching the porcelain, one goal should be to determine the time frame required, at a very specific hydrofluoric acid concentration that will adequately etch various porcelains without excessively degrading and/or weakening the substrate. Hence, the study was conducted to determine the combined effect of hydrofluoric acid and phosphoric acid on the surface of lithium-disilicate veneers. In this study, the heat-pressed veneers were milled and sintered before bonding the veneers to the tooth structure. The total number of 22 samples were divided into two groups: 11 samples in group A and 11 samples in group B. In group A, the intaglio surface of the heat-pressed lithium-disilicate veneers was etched with 4.6% of the hydrofluoric acid solution for 30 seconds, following which the veneers were washed with distilled water for 20 seconds; the surface roughness was examined under a scanning electron microscope for qualitative surface appearance, at a magnification of 3000X. – Fig. 1 shows the microscopic roughness on to the heat-pressed lithium-disilicate veneers after etching with hydrofluoric acid the depth of acid treatment ranging from 1.00 to 1.23µm. In group B, the intaglio surface of the heat-pressed lithium-disilicate veneers was etched with 4.6% of hydrofluoric acid treatment for 30 seconds following which they were further treated with 33% phosphoric acid treatment for 10 seconds, then the veneers were washed with distilled water spray for 30 seconds, and the surface roughness was examined under a scanning electron microscope for qualitative surface appearance, at a magnification of 3000X. – Fig. 2 shows the microscopic...
roughness on to the heat-pressed lithium-disilicate veneers after etching with hydrofluoric acid followed with etching with phosphoric acid the depth of acid treatment ranging from 1.14 to 1.72 µm, Hence, the etching of the heat-pressed lithium-disilicate veneers with two acids shows greater depth of roughness. The surface roughness was calculated using a profilometer. The values were determined by nomenclatures commonly used to determine the surface roughness Ra—arithmetic average value, Rq—root mean square roughness, and Rz—ten point height. —Fig. 3 Shows the Mean Difference between the two groups. To determine the fracture strength the specimens in group A and group B were bonded to the tooth structure with a universal bonding agent. Following this, the fracture strength was determined by subjecting the tooth to a universal testing machine. At least for 48 hours, all the specimens were stored in distilled water at a temperature of 37 degrees following which a load of 219Nload was applied with a 6mm diameter stainless steel ball placed on the specimen with an across head speed of 0.5mm/min. The force was applied at the angulations of 15 degrees relative to the long axis and finally loaded until fracture occurred. There was a significant difference in the fracture resistance of heat-pressed lithium-disilicate veneer treated with phosphoric acid treatment. However, the in vitro study may help us evaluate the mechanical and chemical properties of the materials by etching with various acids to improve the bonding and the fracture
resistances; the laboratory tests may provide us with information on the material strength, potential risk of failure, and deformation of the material. However, they cannot sufficiently predict the long-term performance and stability of veneer in the intraoral environments that need to be considered for dental usage which may include the humidity, salivary pH, masticatory forces, and cyclic loading. In literature, several studies concluded that the occlusal force also reported that age and sex of the individual and loading capacity of the intraoral regions especially the anterior region and posterior regions resulted in considerably different cyclic loading process; hence, the fracture resistance determined using a universal testing machine did not mimic the lateral shearing forces and torsional forces that produced during normal chewing cycle. The load in this study was applied at a constant angle over a specific area on the artificial veneer. However, the chewing capacity and the masticatory load are multidirectional that is applied repeatedly to a larger surface area; hence, the study can be further improved by testing the fracture resistance of the veneer by subjecting it to various forces that may mimic the masticatory model and the chewing forces.

Conclusions

There heat-pressed lithium-disilicate veneers samples treated with combined hydrofluoric acid and phosphoric acid showed better surface roughness and fracture resistance that increased the bond strength of veneers to the tooth structure. Caution must be exercised when extrapolating the laboratory data to clinical situations because many in vivo variables, such as cyclic loading or stress corrosion, were excluded from this study. Therefore, further clinical studies must be performed to ensure that the in vitro results are transferable to clinical situations.

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Conflict of Interest:

None declared.

References