Effect of Leucite Content of Porcelain, Types of Etchant and the Etching Time on Porcelain-Composite Bond

Nasser Barghi, DDS, MA*, Dan E. Fischer, DDS+, Ladan Vatani, DDS++

*Professor, Department of Restorative Dentistry, University of Texas Health Science Center-San Antonio, San Antonio, Texas
+Adjunct Clinical Professor, Department of Restorative Dentistry, University of Texas Health Science Center at San Antonio, San Antonio, Texas.
++ Private Practice, San Antonio, Texas.

Nasser Barghi, DDS, Department of Restorative Dentistry, University of Texas Health Science Center-San Antonio (UTHSCSA), 7703 Floyd Curl Drive, San Antonio, TX 78284-7890 • Tel: (210) 567-3665 • Fax (210) 567-6356 • Email: barghi@uthscsa.edu

Abstract
Objective: This study investigates the optimal etching time of high-leucite (HL) and medium-leucite (ML) content porcelain with 10% hydrofluoric acid (HF) in gel and liquid form. **Method**: Square shaped specimens of ML porcelain (Ceramco II, Ceramco Inc., Burlington, NJ, USA) and HL porcelain (Fortress, Chameleon Dental Prod. Inc., Kansas City, KS, USA) were fabricated, placed in 10 subgroups, etched with a 10% HF liquid (SuperEtch, Chameleon) and HF gel (Porcelain Etch, Ultradent Product, Inc. South Jordan, UT, USA) for a time period of 60 sec, 90 sec, 120 sec, 150 sec, and 180 sec. All specimens were bonded to a hybrid composite resin (Z100, 3M ESPE, Minneapolis, MN, USA) stored for seven days and subjected to shear bond strength testing. **Results**: The mean shear bond strength (Mpa) for the ML subgroups etched with liquid HF were: 6.6 ± 2.7, 10.9 ± 3.2, 11.5 ± 4.4, 16.1 ± 3.1, and 10.6 ± 6.2. The mean shear bond strength for HL subgroups etched with gel HF were: 17.0 ± 4.5, 10.7 ± 1.5, 12.0 ± 3.1, 12.2 ± 1.9, 12.1 ± 3.5. Resulting mean bond strengths for HL porcelain etched with liquid HF were: 7.3 ± 1.5, 7.1 ± 3.3, 8.4 ± .88, 13.2 ± 4.5 and 13.1 ± 2.5. For the same porcelain etched with gel, mean bond strengths were: 11.8 ± 4.8, 11.6 ± 3.8, 10.9 ± 2.1, 17.1 ± 7.7 and 18.8 ± 5.6. **Conclusion**: Optimal etching time of porcelain depends on the type of etchant (gel vs. liquid) used and the amount of leucite in the porcelain.

<u>Clinical Significance</u>

Types of etchant, etching time and the leucite concentration of porcelain are critical factors in achieving optimal porcelain-composite bonds. The gel is more effective in etching porcelain than the liquid etchant.

Article

The concept of etched porcelain bonded restorations (Figure 1, 2) was originally formulated based on etching of porcelain with hydrofluoric acid (HF) to generate the micromechanical retention necessary for the porcelain-composite bond.[i] The bond strength was further advanced by the combination of the mechanical bond and the chemical bond using silane coupling agents. Silane provides chemical covalent as well as hydrogen bonding and improves the wetability. [ii-iii] Early studies reported a bond strength of 7.5 MPa between etched porcelain and composite resin.[iv–v] Further developments in porcelain etchants and improvement in etching times have resulted in significantly higher bond strengths between etched porcelain and composite resin. Chen et al studied the importance of the etching time of porcelain in obtaining higher bond strengths.[vi] They reported the highest shear-bond strength when the porcelain surface was etched for 120 seconds with 10% HF. Additional etching time resulted in reduction of the bond strength. Similar observations have been reported by others.[vii-viii] Yen et al examined the flexural strength of a feldspathic and a cast glass ceramic. [ix] They reported that the adverse effect of over etching of porcelain on the flexural strength is eliminated if over-etched porcelain is properly silanated.

In conceptualizing the mechanism of the etching of porcelain versus the etching of glass, one must differentiate between the glassy matrix and the crystalline phases of dental porcelain.[6, x] Inclusion of leucite crystals into dental porcelain allows porcelain to be more thermally compatible with dental alloys. Leucite crystals grow as the fired porcelain cools off.[8] The function of leucite contributing to the formation of micromechanical retention for bonding is secondary to its original purpose. Studies have shown that the bond strength of composite to etched porcelain is dependant on the leucite concentration within the porcelain.[xi, xii, xiii] In general, low-fusing porcelain contains very little or no crystalline phase. High-leucite-content porcelain (49%- 51%) has been introduced in an attempt to improve some mechanical properties and to increase the bond strength of composite resin to porcelain. Etching of high-leucite-content porcelain allows for the formation of more and smaller microporosities and perhaps provides greater bond strength to resin composite (Figure 3-4). The content of leucite crystals in conventional feldspathic porcelain varies from 20% to 30%. It also varies between enamel porcelain and dentin porcelain. The size of leucite crystals in feldspathic porcelain ranges from approximately 5µm to approximately 20µm. This variation in size of crystals results in the amorphous appearance of etched porcelain as seen in photomicrographs. [13]

Studies have shown that different phases of porcelain react preferentially to different concentrations of etchants as well as to different etching times. [6,7,10] Hydrofluoric acid reacts with silica in feldspathic porcelain to form hexa-fluorasilicates. Stangel et al [7] demonstrated that etching of leucite content feldspathic porcelain with 52% concentration of HF for 90 seconds preferentially dissolves the glassy matrix leaving the crystalline phase intact. They also demonstrated that etching porcelain with a lower concentration of HF (20%) for 20 minutes dissolves the glassy matrix as well as and the crystalline phase of porcelain. Dissolving the crystalline phase may contribute to the reduction of interlocking porosities and micromechanical retention as evident from scanning electron microscopic examination reported by Barghi et al in 1998.[xiv]

In general, porcelain etchants are in the form of gel or liquid. The concentration and recommended etching times vary among various etchants. Liquid HF is a weak acid. Its effect is enhanced by the addition of other acids such as nitric ($HNO3$) and hydrochloric (HCl).[xv] There are three notable disadvantages with using liquid hydrofluoric acid for etching porcelain. These are:

1) rapid vaporization and the danger of inhalation;
2) volume control (liquid does not have adequate surface tension to maintain thickness of acid and a thin layer of acid is rapidly neutralized, which reduces its effectiveness);
3) caustic effects (the presence of stronger acids such as HNO3 and HCl make liquid HF more caustic with regard to tissue contact).

In contrast most gel products are buffered, user-friendly, do not vaporize, and maintain a heavy volume on the surface of porcelain for adequate etching. Therefore shorter etching times may be required for the gel etchants (Figure 5). Recommended etching times for porcelain range from 60 seconds to 3 minutes. These recommendations do not take into consideration variables such as the concentration of etchant, the type of etchant (gel vs. liquid), or the leucite content of porcelain. The objective of this study was to examine the effect of the leucite concentration in porcelain and the type of etchant on porcelain-composite bond.

Materials and Methods:

Two hundred square shaped porcelain specimens (12mm x 12mm x 2mm) were fabricated from the following two porcelains that have different leucite content.

1) Ceramco II, (Ceramco), medium-leucite-content porcelain (approximately 27%);
2) Fortress (Chameleon Dental), high-leucite-content porcelain (approximately 49% to 51%).

As a point of reference for this study, low-leucite-content porcelain refers to porcelain with less than 10% leucite crystals, medium-leucite porcelain contains 20%-30% and high-leucite porcelain contains nearly 50% crystal leucite.

Two commercially-available porcelain etchants were used. They were:

1) a 10% liquid HF etchant (Superetch by Mirage);
2) a 9.5% buffered HF gel (Porcelain etchant by Ultradent).

Porcelain samples were sandblasted with AL_2O_3 at 35 p.s.i. They were steam cleaned, placed in three main groups, twenty subgroups, and treated as follows:

<u>Group A:</u> Samples of the two porcelains in this group were placed in five subgroups and etched with 10% hydrofluoric acid liquid for time periods of 60 seconds, 90 seconds, 120 seconds, 150 seconds, and 180 seconds.

<u>Group B:</u> Samples of the two porcelains in this group were placed in subgroups and etched with a 9.5% concentration of buffered hydrofluoric acid gel for the same periods of time as for Group A.

<u>Group C:</u> Samples in this group were not etched. This group served as the control.

Samples of all subgroups including the control group were bonded to a cylindrically-shaped composite resin using the Ultradent bonding jig (Figure 3). A high-modulus hybrid composite resin was used (Z100, 3M ESPE). A Demetron 501 Curing Light, Kerr (tested to assure output of greater than 400 mw/cm^2) was used for curing the bonded composite.

All samples were stored in room-temperature water for seven days before bond strength testing. Shear bond strength testing was performed using an Instron Testing Machine with a crosshead speed of 1mm per minute. Data were analyzed using ANOVA ($p < .05$). Fractured specimens were visually examined to determine the mode of fracture. The mode of fracture was defined as cohesive (within composite or porcelain), adhesive (at the interface), or mixed (cohesive/adhesive).

RESULTS:
Resulting shear bond strengths (Mpa), and standard deviations (SD), are shown in Table I and Figure 6. The mean shear-bond strength, for five subgroups of medium leucite content conventional porcelain etched with liquid etchant for 60 to 180 seconds were: 6.6 ± 2.7, 10.9 ± 3.2, 11.5 ± 3.1 16.1 ± 3.2 and 10.6 ± 6.2. Samples etched for 150 seconds recorded significantly higher mean bond strengths than samples in the four subgroups etched for shorter etching times. The bond strength dropped significantly when samples were etched for an additional 30 seconds for a total of 180 seconds.

In the subgroups of medium-leucite content porcelain etched with HF gel, the 60 second group produced the highest mean bond strength (17.0 ± 4.5). Longer etching time resulted in the reduction of mean bond strength of porcelain-composite. However, no significant difference was recorded for samples of these subgroups etched for a longer period of time.

In the subgroup of high leucite content porcelain etched with liquid HF, samples etched for 150 & 180 seconds produced the highest mean bond strength. Shorter etching times (< 150 seconds) produced significantly lower bond strengths in these subgroups. No statistical difference was recorded among bond strengths of samples etched for 60, 90 and 120 seconds. Similar observations were recorded for the high-leucite content porcelain etched with HF gel. The highest bond was reached with both etchants after 150 and 180 seconds of etching time. However, HF gel etchant produced significantly higher bond strength with high-leucite content porcelain than the liquid etchant. Again, no differences in bond strength were recorded among samples of high-leucite-content porcelain etched for under 150 seconds with gel HF. Accumulative bond strengths of subgroups of both porcelains etched with gel HF were significantly higher than those for subgroups etched with the liquid etchant.

The mean bond strengths of composite resin bonded to the control group specimens were 3.9 ± 1.9 & 3.8 ± 1.6 for medium- and high-leucite content porcelain. In all specimens, the mode of fracture was generally adhesive when the bond strengths were below 12 MPa. When bond strengths exceeded this level, it became mixed (adhesive/cohesive) fracture and as bond strengths reached 17 Mpa, the mode of fracture was primarily cohesive within the porcelain. When the mode of fracture is cohesive, the exact bond strength remains unknown.

Discussion
Etching of porcelain with hydrofluoric acid results in creation of porosities necessary for micromechanical retention and subsequent bonding to composite resin. Unlike glass, dental porcelain consists of two phases known as the glassy phase and the crystalline phase. Proper etching of porcelain readily removes the glassy phase leaving the crystalline phase intact.

Improper etching affects both phases of porcelain and leads to reduced bond strength to composite resin. A previous study has reported significantly higher bond strengths with shorter etching times.[6] Since the amount and distribution of leucite crystals seems to be a factor in the formation of microporosities and the micromechanical retention, the main objective of this investigation was to examine the possible correlation between the leucite concentration, etching time, and type of etchant. Results indicate that the optimal etching time of porcelain not only depends on the type of etchant but also on the leucite concentration of porcelain. Medium-leucite-content porcelain etched for 150 seconds with the liquid HF produced the highest mean bond strength. Similar bond strength was reached when the same porcelain was etched with the gel etchant for 60 seconds. Additional etching time of this porcelain resulted in significantly lower bond strength with both etchants. The adverse affect of over-etching seems self-limited based on the findings of this study. While 60-second etching time of medium-leucite content porcelain with HF gel produced the highest porcelain composite bond, the adverse affect remained relatively the same when etching times were extended up to 3 minutes.

The need for longer etching time with significantly lower bond strength for the liquid etchant may demonstrate that the presence of additional leucite crystals make the porcelain more resistant to etching and etchants. Three times longer etching time was required to achieve the highest bond strength with the gel etchant and high leucite content porcelain as compared with the same etchant and the medium leucite content porcelain. Resistance of the high-leucite-content porcelain to etching was also noted for the liquid etchant. The adverse affect of over etching high leucite porcelain is not addressed in this study since the duration of etching was limited to 3 minutes.

Previous studies have reported that etching porcelain with 2.5 % to 10% solution of HF for 2-3 minutes provides a sufficient porcelain-composite bond.[6, xvi, xvii] Results of this study demonstrate that the most effective etching times for porcelain fall into a more narrow range than previously assumed. Effective etching depends on the percentage of leucite crystals of the porcelain and the type of etchant (gel vs. liquid).

In this study the gel was a more effective etchant than the liquid regardless of the leucite concentration of porcelain. As to why gel etchant is more effective in producing higher bond strength, one may theorize that gel etchants maintain their volume on the surface where as, liquid etchants readily vaporize and do not keep sufficient volume on the surface for adequate etching. The safety consideration of etchants in areas without adequate ventilation is another factor for recommending gel for etching dental porcelain.

Conclusions

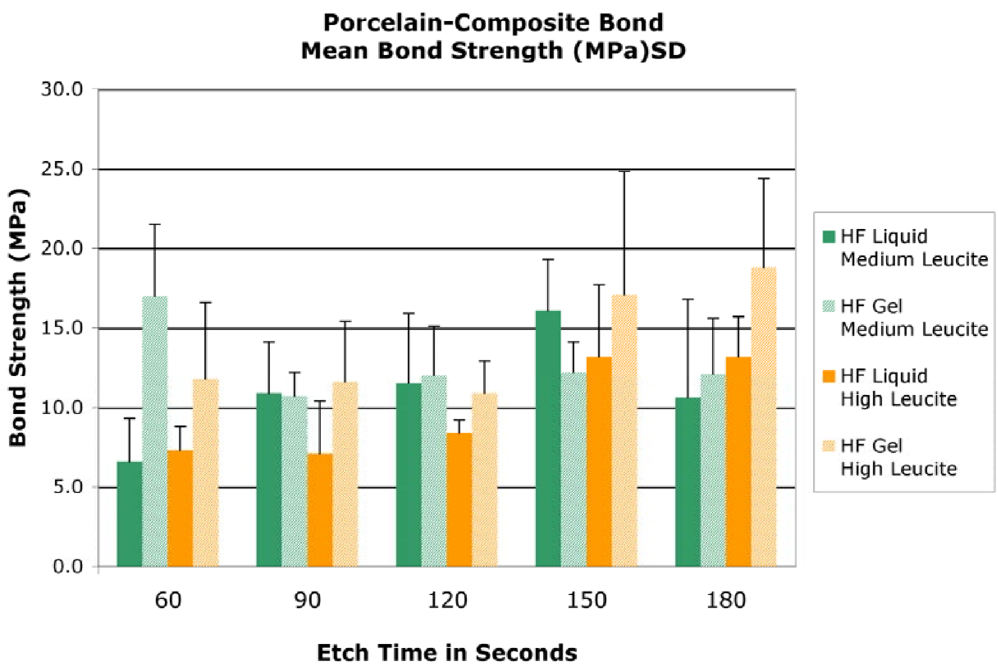
Within the limitations of this study, the following conclusions are made:

1) The gel HF etchant provided higher porcelain-composite bond strength than the liquid etchant.
2) Proper etching of porcelain for bonding depends on the leucite content of the porcelain as well as the type of etchant used.
3) The presence of additional leucite crystals makes porcelain more resistant to etching.

Table 1
Mean Bond Strength (Mpa) & SD Porcelain-Composite Bond
Time (seconds)

Materials	**60**	**90**	**120**	**150**	**180**
ML, HFL	6.6 (2.7)	10.9 (3.2)	11.5 (4.4)	16.1 (3.2)	10.6 (6.2)
ML, HFG	17.0 (4.5)	10.7 (1.5)	12.0 (3.1)	12.2 (1.9)	12.1 (3.5)
HL, HFL	7.3 (1.5)	7.1 (3.3)	8.4 (0.8)	13.2 (4.5)	13.2 (2.5)
HL, HFG	11.8 (4.8)	11.6 (3.8)	10.9 (2.0)	17.1 (7.7)	18.8 (5.6)

ML = Medium-Leucite HL = High-Leucite
HFL = HF Liquid HFG = HF Gel


Porcelain-Composite Bond
Mean Bond Strength (MPa)SD
Bond Strength (MPa)
30.0
25.0
20.0
15.0
10.0
5.0
0.0
60
90
120
150
180
Etch Time in Seconds
HF Liquid Medium Leucite
HF Gel Medium Leucite
HF Liquid High Leucite
HF Gel High Leucite

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