Masters of Esthetic Dentistry

EFFECTS OF PORCELAIN LEUCITE CONTENT, TYPES OF ETCHANTS, AND ETCHING TIME ON PORCELAIN-COMPOSITE BOND

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PROFILE

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The concept of etched porcelain bonded restorations was originally formulated based on the etching of porcelain with hydrofluoric acid (HFA) to generate the micromechanical retention necessary for the porcelain-composite bond.[1] 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.[2,3] Early studies reported a bond strength of 7.5 MPa between etched porcelain and resin composite.[1,4]

Further developments in porcelain etchants and improvements in etching times have resulted in significantly higher bond strengths between etched porcelain and resin composite. Chen and colleagues studied the importance of the etching time of porcelain in obtaining higher bond strengths.[5] They reported the highest shear bond strength when the porcelain surface was etched for 120 seconds with 10% HFA. Additional etching time resulted in a reduction of the bond strength. Similar observations have been reported by others.[6,7] Yen and colleagues examined the flexural strength of a feldspathic and a cast glass ceramic.[8] They reported that the adverse effect of overetching porcelain on the flexural strength is eliminated if overetched porcelain is properly silanated. Using porcelain with a medium leucite content (MLC), Wolf and colleagues reported the highest composite-porcelain bond with 150 and 300 seconds etching of porcelain when a silane coupling agent was used.[9]

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.[5,10] The inclusion of leucite crystals into dental porcelain allows the porcelain to be more thermally compatible with dental alloys. Leucite crystals grow as the fired porcelain cools off.[7] The function of leucite contributing to the

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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.[11–13] In general, low-fusing porcelain contains little or no crystalline phase. Porcelain with a high leucite content (HLC; 49–51%) has been introduced in an attempt to improve some mechanical properties and to increase the bond strength of resin composite to porcelain. Etching of HLC porcelain allows for the formation of more and smaller microporosities and perhaps provides a greater bond strength to resin composite (Figures 1 and 2). 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 5 to 20 μm. This variation in size of crystals results in the amorphous appearance of etched porcelain, as seen in Figures 1 and 2.[13]

Studies have shown that different phases of porcelain react preferentially to different concentrations of etchants as well as to different etching times.[5,6,10] HFA reacts with silica in feldspathic porcelain to form hexa-fluorosilicates. Stangel and colleagues demonstrated that etching of leucite content feldspathic porcelain with a 52% concentration of HFA for 90 seconds preferentially dissolves the glassy matrix, leaving the crystalline phase intact.[6] They also demonstrated that etching porcelain with a lower concentration of HFA (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 is evident in scanning electron microscopic examination reported by Barghi and colleagues in 1998.[14]

In general, porcelain etchants are in the form of gel or liquid. The concentration and recommended etching times vary among the various etchants. Liquid HFA is a weak acid. Its effect is enhanced by the addition of other acids such as nitric (HNO_3) and hydrochloric (HCl) acids.[15] There are three notable disadvantages to using liquid HFA for etching porcelain:

1. Its rapid vaporization and the danger of inhalation
2. Volume control (liquid does not have an adequate surface tension to maintain the thickness of the

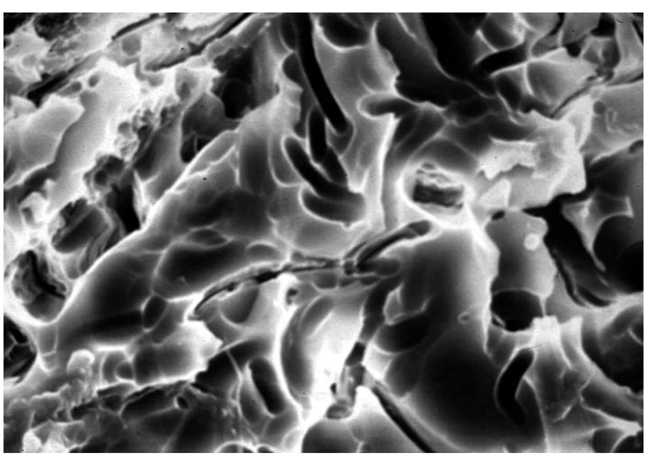

Figure 1. Photomicrograph of porcelain with a medium leucite content etched with hydrofluoric gel for 60 seconds (×2,000 original magnification).

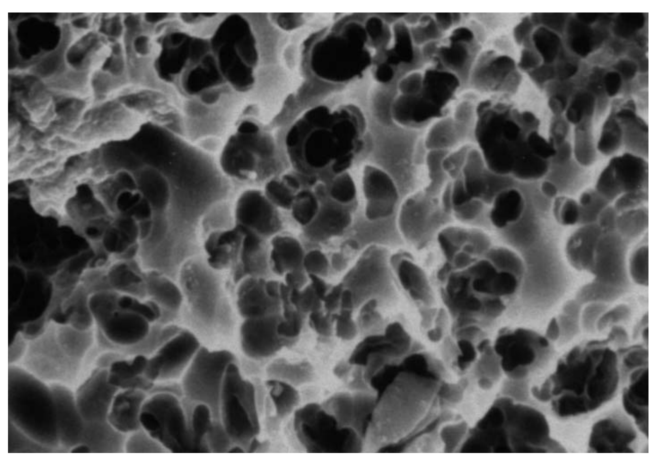

Figure 2. Photomicrograph of porcelain with a high leucite content etched with hydrofluoric gel for 150 seconds (×2,000 original magnification).

acid, and a thin layer of acid is rapidly neutralized, which reduces its effectiveness)
3. Caustic effects (the presence of stronger acids such as HNO_3 and HCl make liquid HFA more caustic with regard to tissue contact)

In contrast, most gel products are buffered, are 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. 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 the etchant, the type of etchant (gel vs liquid) used, 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 a porcelain-composite bond.

MATERIALS AND METHODS

Two hundred square-shaped porcelain specimens (12 mm × 12 mm × 2 mm) were fabricated from the following two porcelains that have different leucite content: Ceramco II MLC porcelain (approximately 27%; Ceramco II, Ceramco Inc., Burlington, NJ, USA) and Fortress HLC porcelain (approximately 49–51%; Fortress, Chameleon Dental Prod. Inc., Kansas City, KS, USA). As a point of reference for this study, porcelain with a low leucite content refers to porcelain with < 10% leucite crystals, MLC porcelain contains 20 to 30%, and HLC porcelain contains nearly 50%. Two commercially available porcelain etchants were used: a 10% liquid HFA etchant (Super-Etch, Chameleon) and a 9.5% buffered HFA gel (Porcelain Etch, Ultradent Products, Inc., South Jordan, UT, USA).

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

- Group A. Samples of the two porcelains in this group were placed in five subgroups and etched with 10% HFA liquid for 60, 90, 120, 150, and 180 seconds.
- Group B. Samples of the two porcelains in this group were placed in subgroups and etched with a 9.5% concentration of buffered HFA gel for the same periods of time as for group A.
- Group C. 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 resin composite using a bonding jig (Ultradent Products, Inc.). A high-modulus hybrid resin composite was used (Z100, 3M ESPE, St. Paul, MN, USA). A Demetron 501 Curing Light (Kerr Manufacturing Co., Orange, CA, USA), tested to ensure an output of greater than 400 mw/cm^2, was used to cure the bonded composite.

All samples were stored in room-temperature water for 7 days before bond strength testing. Shear bond strength testing was performed using an Instron Testing Machine (Instron Corporation, Canton, MA, USA) with a crosshead speed of 1 mm/min. Data were analyzed using two-way multiple-comparison analysis of variance ($p < .05$) and Fisher's PLSD. Fractured specimens were examined visually to determine the mode of fracture, which was defined as cohesive (within composite or porcelain), adhesive (at the interface), or mixed (cohesive/adhesive).

RESULTS

Resulting shear bond strengths (in megapascals) and SDs are shown in Table 1 and Figure 3. The mean shear bond strength for five subgroups of MLC conventional porcelain etched with liquid etchant for 60, 90, 120, 150, and 180 seconds were 6.6 ± 2.7, 10.9 ± 3.2, 11.5 ± 4.4, 16.1 ± 3.2, and 10.6 ± 6.2, respectively. Samples etched for 150 seconds recorded significantly higher mean bond strengths than did samples in the four subgroups etched for shorter etching times. The bond strength dropped significantly when samples

TABLE 1. MEAN BOND STRENGTHS AND SDS FOR PORCELAIN-COMPOSITE BOND TIMES.

	Mean Bond Strength in MPa (SD) for Bond Time*				
Materials	60 s	90 s	120 s	150 s	180 s
MLC, HFAL	6.6 (2.7)^{Aa}	10.9 (3.2)^{Ab}	11.5 (4.4)^{Ab}	16.1 (3.2)^{Ac}	10.6 (6.2)^{Ab}
MLC, HFAG	17.0 (4.5)^{Ba}	10.7 (1.5)^{Ab}	12.0 (3.1)^{Ab}	12.2 (1.9)^{Bb}	12.1 (3.5)^{ABb}
HLC, HFAL	7.3 (1.5)^{Aa}	7.1 (3.3)^{Ba}	8.4 (0.8)^{Ba}	13.2 (4.5)^{Bb}	13.2 (2.5)^{Bb}
HLC, HFAG	11.8 (4.8)^{Ca}	11.6 (3.8)^{Aa}	10.9 (2.0)^{Aa}	17.1 (7.7)^{Ab}	18.8 (5.6)^{Cb}

HFAG = hydrofluoric acid gel; HFAL = hydrofluoric acid liquid; HLC = high leucite content; MLC = medium leucite content.
*Values with different superscripts letters are different at $p < .05$, 95% CI. Lowercase superscript letters refer to within-group differences. Uppercase superscript letters refer to between-group differences with same time.

were etched for an additional 30 (total of 180 s).

In the subgroups of MLC porcelain etched with HFA 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 HLC porcelain etched with liquid HFA, samples etched for 150 and 180 seconds produced the highest mean bond strengths. Shorter etching times (< 150 s) 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 HLC porcelain etched with HFA gel. The highest bonds were reached with both etchants after 150 and 180 seconds of etching time. However, HFA gel etchant produced significantly higher bond strengths with HLC porcelain than did the liquid etchant. Again, no differences in bond strength were recorded among samples of HLC porcelain etched for under 150 seconds with gel HFA. Accumulative bond strengths of subgroups of both porcelains etched with gel HFA were significantly higher than those for subgroups etched with the liquid etchant.

The mean bond strengths of resin composite bonded to the control

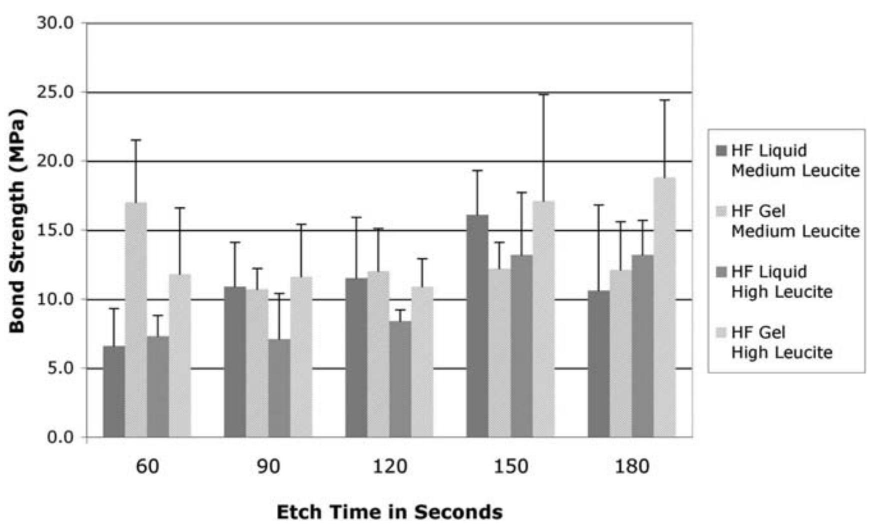

Figure 3. Mean shear bond strengths and SDs for 20 experimental subgroups. HF = hydrofluoric (acid).

group specimens were 3.9 ± 1.9 and 3.8 ± 1.6 for MLC and HLC 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 HFA results in the creation of porosities necessary for micromechanical retention and subsequent bonding to resin composite. 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 resin composite. A previous study has reported significantly higher bond strengths with shorter etching times.[5]

Since the amount and distribution of leucite crystals seem to be factors 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 depends not only on the type of etchant but also on the leucite concentration of porcelain. MLC porcelain etched for 150 seconds with the liquid HFA produced the highest mean bond strength. Similar bond strengths were 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 overetching seems self-limited based on the findings of this study. Whereas a 60-second etching time of MLC porcelain with HFA 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 a longer etching time with significantly lower bond strengths for the liquid etchant may demonstrate that the presence of additional leucite crystals make the porcelain more resistant to etching and etchants. The etching time required to achieve the highest bond strength with the gel etchant and HLC porcelain was three times as long as that for the same etchant and the MLC porcelain. Resistance of the HLC porcelain to etching was also noted for the liquid etchant. The adverse affect of overetching HLC 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 a 2.5 to 10% solution of HFA for 2 to 3 minutes provides a sufficient porcelain-composite bond.[5,16,17] 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). Based on the finding of this study, the optimal etching time for MLC porcelain is 60 seconds with 9.5% HFA gel and 150 seconds with 10% HFA liquid. Using the same etchants as for the MLC porcelain, the optimal etching time for HLC porcelain is 150 to 180 seconds for both the gel and 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 whereas liquid etchants readily vaporize and do not keep sufficient volume on the surface for adequate etching. The safety consideration of etchant use in areas without adequate ventilation is another factor for choosing gel for etching dental porcelain.

CONCLUSIONS

Within the limitations of this study, the following conclusions can be made:

1. The gel HFA etchant provided higher porcelain-composite bond strength than did 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 may affect the time required for proper etching of porcelain.

DISCLOSURE

Dan Fischer, DDS, is president and chief executive officer of Ultradent Products, Inc.

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