

## Intra-oral Repair of Porcelain

### Bonding to Enamel/Dentin Etched with Phosphoric and Hydrofluoric Acids

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#### **ABSTRACT**

**Objective:** To develop an order for using a dentin/enamel etchant and a porcelain etchant which yields the highest bond strength to resin composite. Intraoral repair of porcelain requires sequential etching of porcelain and tooth structure with different etchants. Etching of enamel and dentin with hydrofluoric acid has been shown to have an adverse effect on bonding to composite resin. **Method:** Human molars were sectioned longitudinally mesiodistally to provide two sections from each molar with enamel (E) on one side and dentin (D) on the other. Enamel was flattened to provide an enamel surface that was parallel to the dentin surface that resulted from sectioning. Sections were embedded in polymethylmethacrylate so that enamel and dentin surfaces were exposed. They were randomly sorted into four groups for testing bond to enamel and four groups for testing bond to dentin. The surfaces were etched as follows: (Group 1) 35% phosphoric acid for 30 seconds(Ultra-etch, Ultradent), (Group 2) 9.5% buffered hydrofluoric acid for 90 seconds(Porcelain Etch, Ultradent); (Group 3) phosphoric acid followed by hydrofluoric acid for 30 seconds; and (Group 4) hydrofluoric acid for 90 seconds followed by phosphoric acid for 30 seconds. After etching, a dentin bonding system (PQ1, Ultradent) and a hybrid resin composite (Z100, 3M) were applied according to the manufacturer's instructions. The bonds were tested using an Instron testing machine at a crosshead speed of 1 mm/min. Resulting shear bond strength (MPa) for enamel (E), dentin (D) and four etching treatments were: 1E = 28.2±7.1; 2E = 6.1 ± 1.8, 3E = 13.4 ± 3.5, 4E = 13.2 ± 4.1, 1D = 15.0 ± 4.3, 2D = 8.9 ± 2.4, 3D = 11.7 ± 6.5 and 4D = 11.01 ± 6.5. **Results:** ANOVA (P<0.05) revealed significant differences between groups. **Conclusion:** Combined use of hydrofluoric acid and phosphoric acid significantly reduced shear bond strength of resin bonded to enamel and dentin. The sequence of use of the two etchants had no effect on the bond of composite resin to enamel or dentin.

#### **INTRODUCTION**

Bonded ceramic restorations have become the restorations of choice when indicated.<sup>1-3</sup> Advances in bonding technology and the development of more effective dentinal adhesives allows dentists to bond etched ceramic restorations to both enamel and dentin with a certain degree of reliability and predictability.<sup>4</sup> Longitudinal studies of porcelain veneers bonded to enamel and dentin have shown a low frequency of debonding, fracture and microleakage.<sup>5-11</sup>

In spite of these controlled clinical studies, failures do occur. Several in vitro studies have investigated factors attributing to the fracture and debonding of porcelain veneers.<sup>12-14</sup> These factors include, but are not limited to, the thickness of porcelain, the thickness of die spacer, thickness of luting resin, improper silanization of porcelain, improper etching of porcelain and most importantly, improper bonding. Controlling these variables has significantly reduced, but not eliminated, the likelihood of fractures and undesirable failures.

The concept of the 1970's, that the best repair of ceramic restorations serves as a provisional restoration is not valid today. Product developments have provided the profession with techniques and materials that achieve a durable and lasting repair. Following the same protocol for bonding ceramic restorations to tooth structure, one may bond ceramic to ceramic or resin composite to ceramic achieving a bond strength in excess of the cohesive strength of ceramic materials.<sup>15-18</sup> Adding to these developments is the esthetic quality of resin composites. Some of the presently available direct resin restorations can clinically mimic the esthetic quality of ceramic veneers.

With the increased popularity in bonded ceramic restorations in restorative dentistry, one should expect intraoral repair of porcelain a clinical necessity and not an option.

Fractured bonded ceramic restorations may be placed in two categories.

**Category 1 (Simple):** Cohesive fracture of porcelain known as a simple fracture (Figure 1-4).

**Category 2 (Mixed):** Cohesive and adhesive fracture of porcelain known as a mixed fracture (Figure 5,6)

Category 1(Simple) is less complicated and easier to restore than Category 2 since it involves one etching followed by priming (silanization) and bonding (Figures 7-12).

Category 2 (Mixed) requires the etching of porcelain and the tooth structure (Figures 13-14).

Etching of porcelain with hydrofluoric acid (HF) by means of surface treatment was first reported by Calamia and Horn.<sup>19-20</sup> They reported a bond strength of resin to porcelain of 7.5 MPa. Subsequent improvements in porcelain etchants, duration of etching and leucite concentration of porcelain have resulted in bond strengths in excess of 15 MPa.<sup>21-25</sup> The resin composite-porcelain bond is further enhanced by the application of the silane coupling agents.<sup>17</sup>

Intraoral repair of bonded porcelain to tooth structure involves etching and priming of three separate components: porcelain, enamel and dentin. Etching of enamel and dentin is performed with phosphoric acid (H<sub>3</sub>PO<sub>4</sub>) while etching of porcelain is accomplished using hydrofluoric acid (HF) or hydrofluoric acid substitute such as acidulated phosphate fluoride(APF). Barghi et al reported that etching porcelain with 3% APF provided bond strengths of resin composite to porcelain similar to that obtained when porcelain was etched with a 10% solution of hydrofluoric acid.<sup>26</sup> Lacy et al investigated the effect of 1.23% APF on bonding resin composite to porcelain with and without using Silane; they demonstrated similar results for both HF and APF.<sup>27</sup>

Two major problems associated with intraoral use of hydrofluoric acid for etching fractured porcelain for repair using a resin system have been reported in the literature.<sup>28-32</sup> These are:

- a) the adverse effect of hydrofluoric acid and fluoride ions on bonding to enamel and dentin when the fracture involves tooth structure as well as porcelain, and

- b) the adverse clinical effect of intraoral use of hydrofluoric acid such as acid burns and toxicity.

The development of 9.5% buffered hydrofluoric acid has reduced and perhaps eliminated the problem associated with the direct contact of hydrofluoric acid with oral tissues. Nevertheless, the use of HF presents a major problem for bonding to adjacent enamel and dentin.

Szep and her co-workers demonstrated effective removal of the smear layer and enlarged orifices of dentinal tubules when dentinal surfaces were etched with 36% phosphoric acid and viewed under SEM; however, when surfaces were etched with 9.5% hydrofluoric acid, numerous cracks or star-like defects concentrated at the orifices of dentinal tubules were observed. These defects partially occluded the tubules; this could result in decreased bond strengths. When tooth structure was etched with  $H_3PO_4$  and followed by HF, surfaces appeared similar to  $H_3PO_4$  alone; however, when etched with HF and followed by  $H_3PO_4$ , dentinal surfaces again revealed star-like defects and partially covered orifices. This could presumably result in decreased bond strengths. Szep et al suggest that fluoride may react with calcium in dentinal tubular fluid to form  $CaF_2$  that then occludes dentinal tubule orifices and impedes bonding to intertubular dentin.<sup>33</sup>

Two possible methods for intraoral etching of fractured bonded porcelain involving enamel and dentin have been proposed.

1. Etching porcelain with 9.5% buffered hydrofluoric acid gel, which will inadvertently contact enamel and dentin. This will be followed by etching enamel and dentin with  $H_3PO_4$ .
2. Etching enamel and dentin with  $H_3PO_4$ , followed by etching porcelain with the buffered gel.

In both methods, enamel and dentin are inadvertently exposed to hydrofluoric acid, leaving behind an amorphous precipitant of fluoride. It is not known which sequence provides better bond of composite resin to enamel and dentin that have been exposed to hydrofluoric acid.

### **OBJECTIVE**

The objective of this research project was to better determine which sequence of enchants of enamel and dentin with HF and  $H_3PO_4$  will produce the strongest bond strength to resin composite.

### **MATERIALS AND METHODS**

Recently-extracted caries-free human molars were used for this study. Roots were sectioned at the level of the furcation and then crowns were sectioned longitudinally mesio-distally using a diamond disc under a constant flow of water focused on the cutting edge. Enamel of each section was flattened providing a flattened area of enamel and a flattened area of dentin on each section. Each section was then secured in a mold with chemical cure polymethylmethacrylate.

Using a custom parallel mandrel, mounted specimens were ground on both sides to produce parallel, flattened surfaces in enamel and in dentin. Exposed dentin and enamel were finished with 600 grit silicon carbide under copious water. Prepared specimens were stored in de-ionized water before bonding. Sections were randomly sorted into four groups and received the following surface treatments (Table I):

Group I was cleaned with pumice and rinsed and then etched with 35% phosphoric acid (Ultra-etch, Ultradent) for 30 seconds then rinsed for 10 second with air-water spray and air dried. For both enamel and dentin this group served as control group.

Group II was cleaned with pumice, rinsed and then etched with 9.5% buffered hydrofluoric acid (Porcelain Etch, Ultradent) for 90 seconds. The 90 seconds time is based on optimal required time to etch porcelain with 9.5% HF. After etching, each surface was rinsed for 10 second with air-water spray and air dried.

Group III was cleaned with pumice, rinsed and then etched with 35% phosphoric acid for 30 seconds rinsed for 10 second with air-water spray, etched with 9.5% buffered hydrofluoric acid for 90 seconds and again rinsed for 10 second with air-water spray and air dried.

Group IV was cleaned with pumice, rinsed and then etched with 9.5% buffered hydrofluoric acid for 90 seconds and followed by etching with 35% phosphoric acid for 30 seconds.

#### BONDING

An ethanol-based dentinal adhesive (PQ1, Ultradent) was used for bonding resin composite to the treated specimens of each group. The adhesive was applied and agitated with the applicator brush for 20 seconds, excess was removed with a gentle flow of air and then light cured with an Optilux 501 curing light for 20 seconds.

Each section was then placed into the jig bonding clamp. The device is designed by Ultradent and has been used in various bond-strength testing. The bonding site was chosen and then the specimens were tightened into position. A hybrid resin composite, Z100, (3M-ESPE) was used and placed into a 2.4 mm diameter mold with a smaller diameter packing instrument. The specimen was then light cured for 30 seconds, using an Optilux 501 curing light. The light was calibrated prior to the bonding of each group. For each specimen, surface treatment and bonding were done individually and not in a group. After bonding, specimens were labeled and stored in 37° C water. An Instron testing machine with crosshead speed of 1 mm/min was used for shear bond strength testing. Data were analyzed using ANOVA (P<0.5).

#### RESULTS

Resulting shear bond strengths and standard deviations for resin composite bonded to enamel are shown in Table 2. Etching enamel with HF produced lowest mean bond strength, as expected. HF followed by H<sub>3</sub>PO<sub>4</sub> produced a mean bond strength of 13.2 MPa, whereas H<sub>3</sub>PO<sub>4</sub> followed by HF produced a mean bond strength of 13.4 MPa. No significant differences in bond strengths were recorded between these two groups.

Table 3 shows resulting bond strengths of resin composite to dentin for all treatments. As with specimens bonded to enamel, etching dentin with HF alone produced the lowest mean bond strength. The two sequences of HF - H<sub>3</sub>PO<sub>4</sub> and H<sub>3</sub>PO<sub>4</sub> – HF resulted in nearly identical bond strengths of resin composite to dentin.

#### DISCUSSION

This study was designed to examine the bond strength of resin composite to enamel and dentin following treatment with HF and H<sub>3</sub>PO<sub>4</sub>. Individually or in sequential combinations, it was an effort to determine which sequence of etching would result in the greatest bond strengths. Intraoral repair of bonded porcelain involving porcelain and tooth structure requires two separate etchings. In most clinical instances, it is almost impossible to apply etchant to the porcelain without also applying it to the tooth structure and vice versa. It has been demonstrated that treatment of dentin with HF does not remove the smear layer. Szep et al

demonstrated a fluoride-rich layer as the result of etching dentin with HF. High fluoride concentration of dentin to which APF had been applied was also reported by Ellingsen and Rolla.<sup>34</sup> Treatment of dentin with H<sub>3</sub>PO<sub>4</sub> before application of HF has been shown to produce open dentinal tubules.<sup>33</sup> However, this combined treatment did not improve the bond strength of composite resin to dentin. Using energy dispersive radiographs, Szep et al demonstrated the presence of fluoride when hydrofluoric acid was involved. Based on our result, and other studies involving etching enamel and dentin with HF or HF substitute, it appears that bond strengths of resin composite to enamel and dentin are highly compromised by exposure to HF. The sequences of etching treatment with H<sub>3</sub>PO<sub>4</sub> and HF had no effect on bonding composite resin to enamel and dentin.

Results of this and other studies clearly suggest that exposure of the enamel and dentin to HF has an adverse effect on bonding to resin composite. In light of this information, two possible solutions for repairing complex fracture of bonded porcelain are proposed:

1. Isolating the tooth structure with a barrier such as a liquid rubber dam (opal dam, Ultradent Products, Inc.) prior to the etching of porcelain. The use of a barrier prevents HF from reaching the tooth structure.
2. Avoid etching the porcelain with HF and rely on silane for bonding resin composite to porcelain. Studies have shown that the bond strength of resin composite to properly silanated porcelain exceeds the cohesive strength of porcelain.

Although both suggestions may provide an adequate and a durable bond, the authors recommend isolation of tooth structure and the etching of the porcelain with HF.

#### CONCLUSION

1. Combined treatment of enamel and dentin with H<sub>3</sub>PO<sub>4</sub> and HF has adverse effect on bond strength to resin composite.
2. The sequence of etching with H<sub>3</sub>PO<sub>4</sub> and HF has no effect on the bond strength outcome.

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TABLE 1  
Materials and Methods

Group	Etchant
1	35% H <sub>3</sub> PO <sub>4</sub> for 30 seconds
2	9.5% HF for 90 seconds
3	35% H <sub>3</sub> PO <sub>4</sub> for 30 seconds – 9.5% HF for 90 seconds
4	9.5% HF for 90 seconds – 35% H <sub>3</sub> PO <sub>4</sub> for 30 seconds

TABLE II  
Bond Strength to Enamel (MPa)

	Count	Mean Shear Bond Strength	Standard Deviation	Standard Error
<b>9.5% HF</b>	12	6.175	1.874	.541
<b>35% H<sub>3</sub>PO<sub>4</sub></b>	12	28.267	7.105	2.051
<b>9.5% HF-35% H<sub>3</sub>PO<sub>4</sub></b>	12	13.225	4.102	1.184
<b>35% H<sub>3</sub>PO<sub>4</sub> - 9.5% HF</b>	12	13.408	3.557	1.027

TABLE III  
Dentin

	Count	Mean Shear Bond Strength	Standard Deviation	Standard Error
<b>9.5% HF</b>	11	8.936	2.469	.744
<b>35% H<sub>3</sub>PO<sub>4</sub></b>	11	25.000	4.352	1.312
<b>9.5% HF-35% H<sub>3</sub>PO<sub>4</sub></b>	10	11.070	6.557	2.073

<b>35% H<sub>3</sub>PO<sub>4</sub>-9.5% HF</b>	10	11.010	6.534	2.066
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