

Effect of phosphoric acid concentration used for etching on the microtensile bond strength to fluorotic teeth

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Abstract

To evaluate the effects of different etching concentrations of phosphoric acid on the microtensile bond strength of Adper Single Bond 2 to fluorotic teeth.

Deidentified extracted teeth were collected, including 30 sound teeth, 30 teeth with mild fluorosis, 30 teeth with moderate fluorosis, and 30 teeth with severe fluorosis. The teeth in each group were randomly divided into 3 subgroups (n=10) that were subjected to acid etching using 35%, 40%, or 45% phosphoric acid. Adper Single Bond 2 (3M, Saint Paul, MN) was used as the adhesive for bonding Z250 universal resin (3M) to the etched dental enamel. Microtensile testing was used to determine the bond strength. After the microtensile test, the fractured specimens were examined under scanning electron microscopy (SEM).

Both dental fluorosis and concentrations of phosphoric acid significantly affected the microshear bond strength of Adper Single Bond 2 to dental enamel. The maximum bond strength was achieved by using 40% phosphoric acid. Failure analysis showed that most failures occurred at the bonding interface. The rates of failures at the bonding interface decreased as the degree of fluorosis increased and as the concentration of phosphoric acid increased from 35% to 45%.

The bond strength of fluorosis tooth was lower than that of healthy tooth, the bond strength increased with the increasing concentration of phosphoric acid, but an excessively high acid concentration can conversely lead to an apparent decline in bond strength.

Abbreviations: SEM = scanning electron microscopy, TFI = Thylstrup and Fejerskov Fluorosis Index.

Keywords: fluorosis, fluorotic teeth, microtensile bond strength, phosphoric acid

1. Introduction

In modern times, fluoride has been made widely accessible in daily life through drinking water, dentifrices, and food due to its extensive use for control of dental caries.^[1,2] However, excess intake or ingestion of fluoride during tooth development contributes to dental fluorosis, which is the condition of hypomineralization in enamel. Clinically, dental fluorosis causes varying degrees of intrinsic tooth discoloration, depending on the dose, duration, and age of the individual during the excessive fluoride exposure. The severity of dental fluorosis is frequently measured using the Dean's index or Thylstrup and Fejerskov

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Received: 30 June 2017 / Accepted: 6 August 2018 http://dx.doi.org/10.1097/MD.000000000012093 Fluorosis Index (TFI).^[3] Although mild dental fluorosis might not be a concern affecting oral health-related quality of life, moderate and severe fluorosis have been consistently reported to have negative effects on oral health-related quality of life.^[2] Specifically, they negatively influence psychosocial aspects of patients beyond the awareness/concern, acceptability, and/or satisfaction regarding the dental fluorosis.

Various treatments and managements such as bleaching, microabrasion, composite restorations, veneering, crowning, or a combination of 2 approaches have been proposed to correct the effects of dental fluorosis. The use of these methods depends on the severity of dental fluorosis.^[4] Among these, aesthetic veneers are more suitable for use in patients with a TFI \geq 5. In addition, nonmetallic veneer aesthetic restorations are preferred for patients due to their lower toxicity and lower risk of allergic reactions compared with alloys. However, like orthodontic brackets bonded to fluorotic teeth, clinical porcelain veneers bonded to fluorotic teeth have much higher failure rates due to loose bonding.^[5,6] Although great effort has been made to improve the bonding system, currently there is no consensus on the standard protocol and materials for bonding veneers to fluorotic enamel in order to improve the patients' dental aesthetics and oral health-related quality of life.

We hypothesize that the concentration of etching acid used to prepare the enamel might directly affect the bonding strength of veneers to fluorotic teeth. In the present study, we used different concentrations of phosphoric acid to etch fluorotic enamel and investigated their effects on the microtensile bond strength and failure mode. Specifically, microbeam specimens were prepared

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and the microtensile bond strength was measured using a WDW-100 electronic universal testing machine (Jinan, Jinan Henry Gold Testing Machine, China). The broken specimens from the microtensile bond strength test were further examined using a stereo microscope for their failure modes. The results suggest that the concentration of phosphoric acid used for etching significantly affects the bond strength for teeth with all levels of fluorosis.

2. Materials and methods

2.1. Materials

Adper Single Bond 2 and Z250 universal resin were purchased from 3M ESPE (Saint Paul, MN). Analytical grade phosphoric acid was obtained from West Long Chemical Co., Ltd (Guangzhou, Guangdong, China).

This study was approved by the Human Ethics Committee of our University, and complied with the ethical guidelines of the Helsinki Declaration, and written informed consent was obtained from individual human subject. Noncarious molars were selected from a pool of deidentified extracted teeth in our hospital. Oral practicing physicians with more than 5 years' experience randomly selected extracted teeth and assigned them to 4 groups (n = 30 teeth/group) according to the severity of dental fluorosis based on the TFI score standards: healthy teeth (Sound group), teeth with mild fluorosis (Mild-F group), teeth with moderate fluorosis (Mod-F group), and teeth with severe fluorosis (Seve-F group). The selected teeth were cleaned to remove periodontal film and stored in 1% thymol solution at 4°C after washing with saline.

2.2. Sample preparation

The teeth were immobilized in gypsum casts and polished using a diamond chip with running water to remove dirt and a small layer of enamel at the buccal section in order to generate a smooth flat enamel surface. The exposed enamel surfaces were further ground using 400 grit silicon carbide sandpaper for 30 seconds. The ground tooth samples in each group were randomly divided into 3 subgroups (n=10 per subgroup) and subjected to acid etching for 30 seconds using the different concentrations of phosphoric acid. The acid etched enamel surfaces were rinsed with water for 10 seconds and then blotted with a cotton swab or sponges to remove the water adsorbed on the enamel surface. Adper Single Bond 2 was incrementally applied onto the acid etched enamel in 2 layers, which were cured separately according to the manufacturer's instructions. Z250 universal resin was used to build a 5-mm-thick crown on the enamel in 4 increments (2 mm for the first increment and 1 mm the subsequent 3 increments). Each increment was cured using curing light (Dentsply International, York, PA) for 10 seconds according to the manufacturer's instructions. Teeth were sectioned perpendicularly to the bonded surface using a low-speed diamond saw under water irrigation to produce several slices and then sectioned further into 4 smaller beam shape pieces $(1.0 \times 1.0 \times 8.0 \text{ mm})$ consisting of tooth and bonding resin. Beam specimens were measured using calipers and stored in distilled water at 37°C for 1 day before microtensile bond strength testing.

2.3. SEM examination of specimens for microtensile testing

Some samples were first coated with a thin layer of gold via sputtering and examined using a SU1510 scanning electron microscope (JEOL, Ltd, Tokyo, Japan) at a magnification of 750×.

2.4. Microtensile strength test

The beam-shaped specimens were fixed onto a universal testing machine using super glue 502. The specimens were stressed at 0.5 mm/min on the universal testing machine until failure under tension. The microtensile bond strength was calculated by dividing the maximum test load by the bonding area for each specimen.

2.5. Fracture mode

After the microtensile test, the fractured specimens were observed under a stereo microscope (Motic, Carlsbad, CA). The factures were classified into 3 modes: adhesive surface fracture (Mode A), cohesive failure within the composite resin or tooth structure (Mode B), and mixed fracture (Mode C).

2.6. Statistical analysis

Numerical data were analyzed using SPSS 19.0 statistical software. Two-way analysis of variance (ANOVA) was used to identify statistically significant differences among the 4 groups or 3 subgroups. The least significant difference (LSD) test was used to analyze the differences between 2 subgroups. The level of statistical significance was set at 0.05.

3. Results

3.1. Appearance of bonded surfaces on SEM

Figure 1 shows the interfaces of acid-etched enamel and bound resin on teeth from the Sound and Mild-F groups under scanning electron microscopy (SEM). Sound enamel etched with 35% phosphoric acid had irregular shallow pits and formed a continuous bonding interface with the resin without any fractures/cracks (Fig. 1A). The enamel of the Mild-F group etched with 35% phosphoric acid showed no obvious dimples and formed an irregular bonding interface with the resin (Fig. 1B). The enamel of the Mild-F group etched with 40% phosphoric acid had a continuous bonding interface with the resin as well as a layer penetrated by resin (Fig. 1C). The enamel of the Mild-F group etched and the resin with visible cracks like parched and without a layer penetrated by resin (Fig. 1D).

3.2. Microtensile bond strength and fractures

Figure 2 shows the microtensile bond strengths determined from the microtensile testing for all 12 subgroups. The degree of fluorosis had a significant effect on the microtensile bond strength for any concentration of phosphoric acid used (all P < .05 for the different groups treated with 35%, 40%, or 45% phosphoric acid). The bond strength decreased with increasing severity of fluorosis. In addition, the concentration of phosphoric acid had a significant effect on the microtensile bond strength for all 4 groups of teeth (P < .05 for the different concentrations in all 4 groups). For the Sound group, the bond strength decreased as the concentration of phosphoric acid increased (P < .05). However, for the 3 groups with fluorosis, treatment of the enamel with 40% phosphoric acid resulted in the highest bonding strength compared with the other treatment concentrations (P < .05).

In each subgroup, the majority of specimens fractured in Mode A (Fig. 3). However, the incidence of facture in Mode B increased as the concentration of phosphoric acid increased for each group



Figure 1. Representative SEM images of cross-sections of the bonded teeth from the: (A) Sound group treated with 35% phosphoric acid, (B) the Mild-F group treated with 35% phosphoric acid, (C) the Mild-F group treated with 40% phosphoric acid, and (D) the Mild-F group treated with 45% phosphoric acid. Scale bars: 50 µm.

of teeth. When the same concentration of phosphoric acid was used for etching, the incidence of factures in Mode B increased as the severity degree of fluorosis increased.

4. Discussion

In this study, we obtained deidentified extracted fluorotic teeth and sound teeth from Gulin, which has a high incidence of endemic fluorosis due to coal burning. To eliminate potential bias, extracted teeth were randomly selected by oral practicing physicians with more than 5 years' experience and assigned to 4 groups based on the severity degree of fluorosis according to the TFI score standards.^[7] All teeth selected had no dental caries.



Figure 2. Microtensile bond strength for teeth in the various groups etched using different concentrations of phosphoric acid (35%, 40%, or 45%) for 30 seconds.

The bonding durability determines whether veneer restorations achieve long-term success for esthetic treatment of fluorotic teeth. Thus, the veneer must be bound to the fluorotic enamel with sufficient bond strength. The importance of preparation of the enamel surface for the successful bonding has been well established.^[8-13] To bond dental restorations or correction devices such as veneer, orthodontic brackets, and resin composite restorations to enamel, various methods to prepare enamel surfaces have been applied, including acid etching, laser treatment, and sandblasting.^[12] Among these, phosphoric acid etching has been used as the gold standard in esthetic dentistry due to its proven effectiveness in producing microporosity in healthy enamel,^[14] and therefore, we used phosphoric acid for acid etching of enamel in the present study.

Dental fluorosis is caused by the excessive intake of fluoride during tooth development. The highly available fluoride promotes hypomineralization of the enamel, which results in highly disordered enamel crystals. The high resistance of fluoritic enamel to acid etching and the porous nature of the affected enamel region make bonding to this enamel a significant challenge for oral care providers. Thus, much research has been devoted to finding solutions for improving the adhesive strength on fluorotic enamel.^[6,15–17] However, previous studies have reported conflicting results regarding the optimal etching time when using phosphoric acid to treat fluorotic teeth. Some studies have recommended that extended enamel conditioning with phosphoric acid should be performed for bonding onto fluorotic enamel.^[9,16] One study found that prolonged etching time (i.e., 120 seconds) could significantly improve the bond strength to fluorotic teeth,^[18] and another study found that prolonging the etching time increased the shear bond strength of composite resin on mildly and moderately fluorotic teeth in patients less than 40 years old.^[19] However, other studies have reported that varying



Figure 3. Failure modes in microtensile bond strength test for various groups subjected to enamel surface etching with 35% (A), 40% (B), or 45% (C) phosphoric acid for 30 seconds.

the etching time does not significantly affect the bond strengths on fluorotic and healthy enamel.^[15,20] Moreover, Waidyasekera et al found the total-etch system had greater adhesion strength than the self-etch system, and meanwhile, the 2-step self-etching system was found to achieve greater adhesive strength than 1-step self-etching systems.^[15] However, to our best knowledge, no studies have investigated the influence of the acid concentration used for etching on bond strength for fluorotic teeth. Therefore, in the present study, we examined the effects of the etching acid concentration on the bonding properties of a total-etch bonding

system Adper Single Bond 2 to teeth having various severity degrees of fluorosis. We used different concentrations of 35%, 40%, and 45% phosphoric acid to etch the enamel for 30 seconds. We then used microtensile strength testing to evaluate the bond strength onto the enamel, because this technique has been extensively accepted as an efficient method to measure the bond strength in dentistry.

Consistent with the results of previous reports,^[16] our results showed that the bonding to sound teeth was significantly better than bonding achieved with all fluorotic teeth in terms of microtensile bond strength (Fig. 2). This could be attributed to the fact that fluorotic teeth have an acid-resistant superficial layer in their enamel. Moreover, the bonding to fluorotic teeth decreased as the severity of fluorosis increased (Fig. 2). This finding was also consistent with previous studies examining the application of other adhesives to fluorotic teeth.^[16] Our results revealed that a maximum bond strength of 20.48 ± 1.5 MPa was achieved when 35% phosphoric acid was used to etch the enamel of sound teeth, and the bond strength decreased as the concentration of phosphoric acid used for etching increased. However, for all the 3 groups of fluorotic teeth, maximum bond strengths of 14.95 ± 0.75 , 11.38 ± 1.23 , and 9.23 ± 0.87 MPa were achieved when 40% phosphoric acid was used. This observation might be attributed to the fact that a layer penetrated by resin was formed during the generation of a continuous bonding interface (Fig. 1) when the fluorotic enamel in the different groups was etched with 40% phosphoric acid.

5. Conclusion

The results of the present study demonstrate that the concentration of etching phosphoric acid had a significant effect on the microtensile bond strength of Adper Single Bond 2 to fluorotic teeth. Further investigation is warranted to confirm whether our findings hold true for other commercial adhesives.

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Author contributions

Funding acquisition: Ling Guo. Investigation: Linhu Lv. Project administration: Mengqin Gu. Software: Wangyang Li. Supervision: Xiaoping He. Writing – original draft: Mengqin Gu. Writing – review & editing: Mengqin Gu.

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