Micro-tensile bond strength of a self-etching primer bonding system to fluorosed enamel

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Purpose: The aim of the study was to evaluate micro-tensile bond strength and the conditioning patterns of a self-etching primer bonding system to un-etched (with extended priming) and etched ground fluorosed enamel.

Materials and Methods: Extracted premolars were classified according to severity of fluorosis using Thylstrup and Fejerskov index (TFI: 0, normal; 1-3, mild fluorosis; and 4-6, moderate fluorosis) into three groups, which were again divided into four sub groups to receive different enamel conditioning. The ground enamel surfaces of the teeth in first and second sub groups were primed with a self-etching primer (SE primer, Clearfil SE Bond) for 60 and 90 s respectively prior to application of the adhesive. The teeth in the remaining sub groups were etched with 37% phosphoric acid (H_3PO_4) for 60 or 90 s prior to application of the self-etching primer bonding system according to the manufacturer's instructions. All the teeth were then restored with resin composite, stored for 24 hours in water and prepared for micro-tensile bond strengths. The data were analyzed with analysis of variance (ANOVA) and Fisher's PLSD test ($p<0.05$).

Results: Significantly higher micro-tensile bond strengths were observed when the self-etching primer bonding system was applied to etched ground fluorosed enamel ($p<0.05$) compared to un-etched enamel with extended priming. The bond strengths were significantly stronger in normal and mildly fluorosed enamel compared to moderately fluorosed enamel when etched for 60 s ($p<0.05$). Predominantly Type II etching pattern was observed in H_3PO_4 conditioned fluorosed enamel while relatively shallow etching pattern was seen in un-etched fluorosed enamel with extended priming.

Conclusion: Severity of fluorosis, etching time and pattern influence the bond strength of the self-etching primer bonding system to fluorosed enamel. **(Int Chin J Dent 2002; 2: 107-115.)**

Clinical Significance: Before application of self-etching primer bonding systems, H_3PO_4 conditioning is necessary to obtain high bond strength for fluorosed enamel.

Key words: bond strength, enamel fluorosis, self-etching primer.

INTRODUCTION

Enamel fluorosis is characterized by surface hypermineralyzation and sub surface porosity. Clinically, moderate/severe fluorosis appears as irregular opacities with staining and pitting resulting in loss of surface enamel.[1] Therefore, these patients usually require tooth colored restorations such as composite/porcelain veneers.[2]

Compared to normal enamel, fluorosed enamel is difficult to etch, as fluoroapatite is more resistant to acid dissolution than hydroxyapatite.[3] Moreover, the fluoroapatite content of the fluorosed enamel varies with severity.[4] Therefore, previous investigators[5,6] recommended extended enamel conditioning with phosphoric acid when bonding fluorosed enamel. In contrast, Ng'ang'a et al.[7] and Ateyah et al.[8] reported no significant differences in the bond strengths in fluorosed and normal enamel at different etching times. Therefore, these controversies make it difficult to determine the most suitable etching time with phosphoric acid for varying degrees of fluorosis.

Self-etching primer bonding systems are effective for bonding resin composite to dentin.[9,10] However, controversies exist as to use of these bonding systems in enamel.[11-14] Moreover, these primers have shown less etching ability in enamel compared to phosphoric acid because of their high pH.[13] Therefore, with self-etching primer-bonding systems extended priming or additional phosphoric acid etching may be necessary to obtain adequate bonding to fluorosed enamel. However, there are no reports on bond strengths or conditioning patterns produced by self-etching primers on fluorosed enamel.

The aim of this study therefore, was to evaluate micro-tensile bond strength (μTBS) and the conditioning patterns of a self-etching primer bonding system to fluorosed enamel of varying severity using primer of the bonding system or 37% phosphoric acid at different etching and priming times.

MATERIALS AND METHODS

The materials used in this study are listed in Table 1 with their abbreviation code. Thirty non-carious premolars extracted for orthodontic reasons from patients in areas endemic for fluorosis in Sri-Lanka were used for the study. The teeth were obtained from patients who consented verbally to use their teeth for research and educational purposes. The extracted teeth were cleaned and stored in water in a refrigerator at 4˚C. Prior to use, the teeth were classified according to the severity, using Thylstrup and Fejerskov index[1] (TFI: 0, normal; 1-3, mild fluorosis; and 4-6, moderate fluorosis) into three groups. Teeth with severe fluorosis (TFI: 7-9) were not available for the present study. Eight teeth from each group were randomly divided into four subgroups to receive different enamel conditioning as described below in preparation for μ-TBS.

Specimen preparation for μ-TBS

Both buccal and lingual surfaces of each tooth were used for the bond strength measurement as previous studies have shown that there are no significant differences in the fluoride concentration between buccal and lingual aspects of the same tooth.[7] In order to standardize the enamel reduction, 0.5 mm grooves were prepared and superficial enamel was removed initially using a superfine diamond bur (SF #145, Shofu Inc., Kyoto, Japan) on a high-speed hand piece followed by grinding with #600 grit SiC paper under water coolant. The teeth were then ultrasonically cleaned in distilled water for 5 minutes prior to the bonding procedure.

Table 1. Materials used.

Product		Batch number	Components
Self-etching primer bonding system			
Clearfil SE Bond	Primer (SE primer)	003A	MDP, HEMA, Hydrophilic dimethacrylates, Photo initiator, Water
	Bond (SE bond)		MDP, Bis-GMA, HEMA, Photo initiator, Water
Conditioner			
K-etchant		184	37% Phosphoric acid gel
Restorative material			
Clearfil AP-X		0482	Silanated barium glass, Silica, Colloidal silica, Bis-GMA, TEGDMA, Photo initiator

All materials were manufactured by Kuraray Medical, Tokyo, Japan.
Bis-GMA: bisphenol A-diglycidyl methacrylate, HEMA: 2-hydroxyethyl methacrylate, MDP: 10-methacryloyloxydecyl dihydrogen phosphate, TEGDMA: triethyleneglycol dimethacrylate.

The buccal and lingual enamel surfaces of teeth in first and second sub-groups were primed with SE primer for 60 and 90 s and gently dried. The adhesive of SE Bond was then applied and light cured for 20 s. Thereafter, both surfaces were built up with the resin composite to a height of 5 mm in three increments, light curing each increment for 20 s, at 400 mW/cm^2 (New Light VL-II, GC Corp., Tokyo, Japan).

The teeth in the remaining sub groups were etched with 37% phosphoric acid (H_3PO_4, K-etchant) for 60 and 90 s, rinsed with water for 20 s, and dried for 20 s. Thereafter, SE Bond was applied according to the manufacturer's instructions as follows; SE primer was applied for 20 s and gently dried. Then the adhesive was applied and light cured for 20 s. The resin composite was bonded in a manner similar to the primed groups. Thereafter, all the specimens were stored in water at 37˚C for 24 hours.

The following day, each tooth was sectioned perpendicular to the bonded interface to obtain three to four 0.7 mm thick slabs. Then each slab was longitudinally sectioned at the center to separate the buccal and lingual surfaces. Thereafter, each section was trimmed with a superfine diamond bur (v16ff, GC Corp.) to obtain an hourglass shape so that the narrowest portion at the adhesive interface has a surface area of 1.0±0.2 mm^2 in preparation for μ-TBS test.[15] Each specimen was attached to a Bencore-Multi T testing apparatus (Danville Engineering Co., San Ramon, CA, USA) with Zap-It cyano-acrylate adhesive (Dental

Ventures of America, Corona, CA, USA) and placed in a universal testing machine (EZ test, Shimadzu Corp., Kyoto, Japan) for μ-TBS testing at a crosshead speed of 1 mm/minute (Fig. 1). The data were analyzed with ANOVA and Fisher's PLSD test at 5% level of significance.

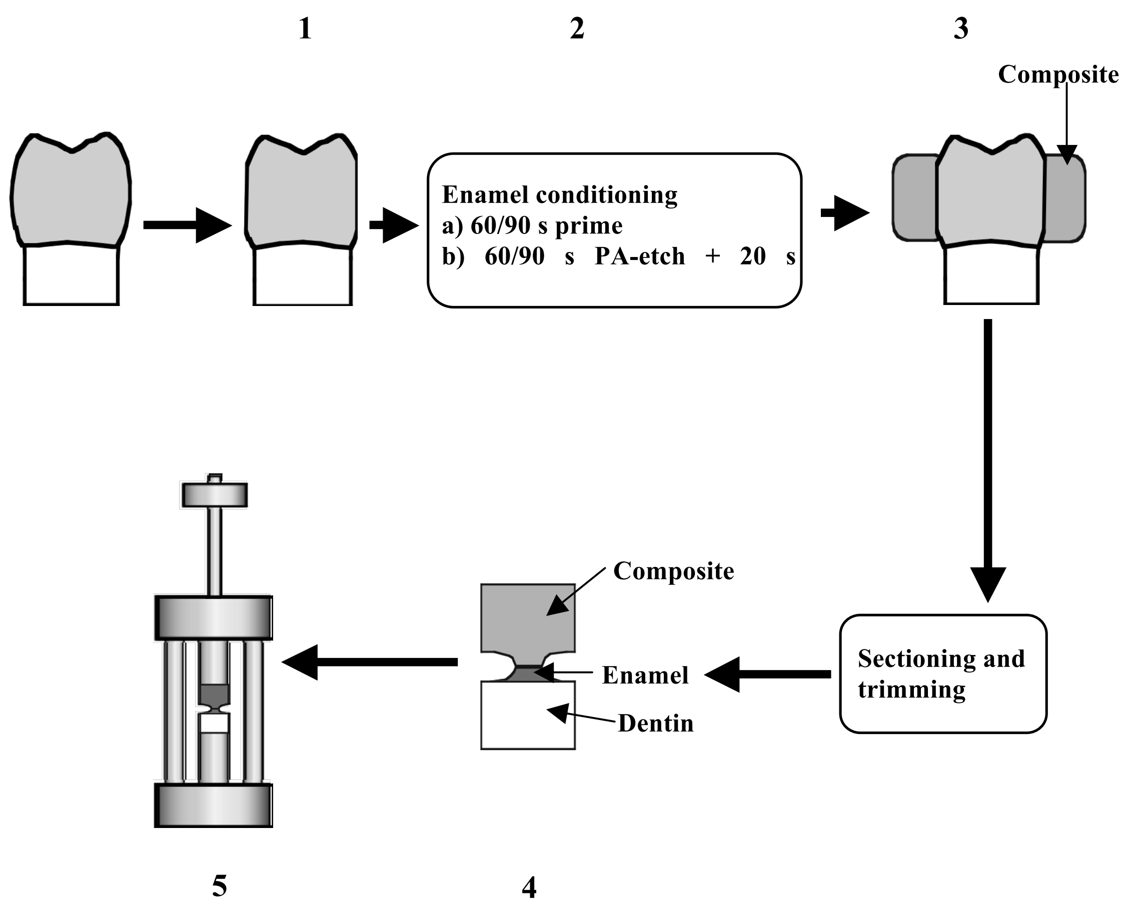


Fig. 1. Schematic illustration of specimen preparation. 1) ground buccal and palatal enamel, 2) enamel conditioning, 3) restoration with resin composite, 4) specimen tested with μ-TBS, and 5) assembly for tensile test.

The fracture modes

The fracture patterns after μ-TBS testing were observed visually, and were classified into two groups as follows; A, cohesive failure in fluorosed enamel, and B, adhesive failure at the interface between bonding resin and enamel.

Specimen preparation for microscopic observation of conditioning patterns of fluorosed enamel

Two teeth from each group were used to observe the effect of extended priming and conditioning with 37% H_3PO_4 on ground fluorosed enamel. Ground palatal and buccal enamel was sectioned with a diamond saw (Isomet, Buheler Co., Lake Bluff, IL, USA) to obtain eight to ten 1 mm thick slabs from each tooth.

Alternate slabs from one tooth were either primed or etched for 60 s while the alternate slabs of the other tooth were primed or etched for 90 s. Each primed slab was then rinsed with water and acetone and dried while etched slabs were rinsed with water only. All the slabs were mounted on stubs and gold coated for observation under a scanning electron microscope (SEM, JSM-5310LV, JEOL, Tokyo, Japan).

RESULTS

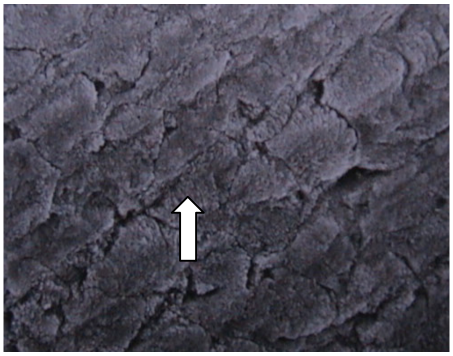
The μ-TBS of the self-etching primer bonding system to fluorosed enamel is summarized in Table 2. Extended priming did not significantly improve the bond strengths of un-etched fluorosed enamel ($p>0.05$). However, phosphoric acid etching prior to application of the self-etching primer bonding system resulted in significantly higher bond strengths in fluorosed enamel ($p<0.05$). The bond strengths were significantly stronger in normal and mildly fluorosed enamel compared to moderately fluorosed enamel when etched for 60 s ($p<0.05$). The bond strengths of moderately fluorosed enamel significantly improved to a level comparable to other groups when etched for 90 s ($p<0.05$). Therefore, 60 s phosphoric acid etching was sufficient to improve the bond strengths of the self-etching primer bonding system to mildly fluorosed enamel while moderately fluorosed enamel required longer etching time (90 s) to improve the bond strength.

Table 2. The micro-tensile bond strength results.

Surface conditioning	Normal			Mild fluorosis			Moderate fluorosis		
Bond strength (MPa)	Mean	SD	Category	Mean	SD	Category	Mean	SD	Category
20 s prime (Control)	16.3	4.3	a,b	12.4	5.6	c	12.0	2.5	c
60 s prime	15.9	3.2	b	13.8	3.8	b,c	11.6	2.5	c
90 s prime	16.6	6.0	a,b	13.5	6.9	b,c	12.1	3.8	c
60 s etch + 20 s prime	20.1	6.6	a	19.7	4.0	a	15.5	4.5	b
90 s etch + 20 s prime	19.1	4.2	a	18.8	7.3	a	18.4	3.5	a

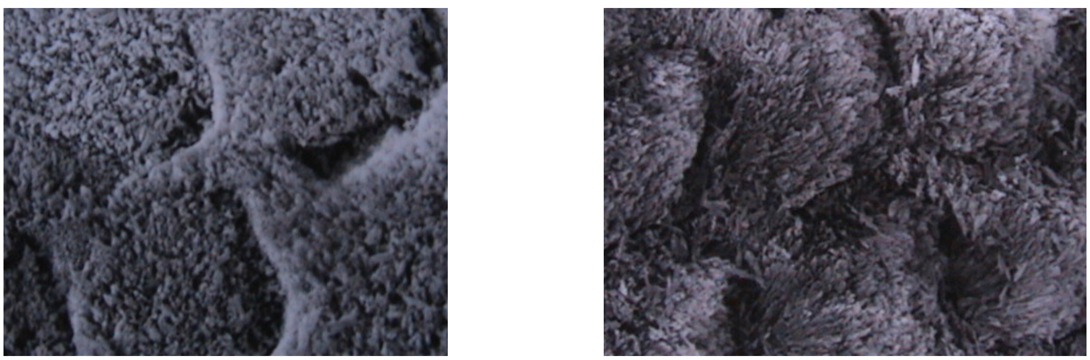
SD: standard deviation. Sample size: 12-16. Classification of fluorosis according to the Thylstrup and Fejerskov index (TFI: 0, normal; 1-3, mild fluorosis; and 4-6, moderate fluorosis).[1] Identical letters among the surface conditioning groups represents no statistically significant difference ($p>0.05$).

Visual observation of fracture modes revealed cohesive fractures involving enamel (A) in 5-10% of the specimens in each group. The remaining 90-95% of the specimens showed a mixed fracture pattern (B). There were no significant differences in fracture patterns observed between groups. A relatively shallow etching pattern was seen in un-etched fluorosed enamel with extended priming (Fig. 2). Moreover, scratch marks created during tooth preparation were also observed (Fig. 2, arrow). All three etching patterns previously described for normal enamel with phosphoric acid conditioning[16] was also observed with fluorosed enamel. Namely, Type I pattern in which hollowing of the prism centers were observed with relatively intact prism peripheries (Fig. 3a); Type II pattern where prism peripheries appeared to be removed (Fig. 3b); and a Type III pattern with generalized roughening which did not confirm to any prism structure (not shown).



10 µm

Fig. 2. Scanning electron micrograph of the etched enamel surface with extended priming (90 s) on ground fluorosed enamel.



a 5 µm b 5 µm

Fig. 3. Scanning electron micrographs of the etching patterns of ground mild fluorosed enamel following H_3PO_4 conditioning for 60 s; a) Type I and III etching patterns, and b) Type II etching pattern.

DISCUSSION

Though there are controversies regarding the bonding performance of self-etching primer bonding systems in normal uncut enamel, there is sufficient evidence to indicate that these bonding systems provide bond strengths comparable to bonding systems that use phosphoric acid in ground/cut enamel.[12,13] However, the pilot study revealed low bond strengths of ground fluorosed enamel when the self-etching primer was applied according to the manufacturer's instructions (results-control). Therefore, the effect of extended priming or prior enamel conditioning with phosphoric acid on the bond strength of the self-etching primer bonding system to ground fluorosed enamel was evaluated in the present study. The conditioning time of 60 and 90 s with phosphoric acid was chosen according to the previous studies by Al-Sugair[3] and Ateyah's[8] recommendations.

Results revealed significantly higher μ-TBS of Clearfil SE Bond when the self-etching primer bonding system was applied to etched ground fluorosed enamel ($p<0.05$) compared to un-etched enamel with extended priming. SEM observation of the conditioned surfaces revealed relatively shallow etch pattern with extended priming. Therefore, insufficient etching-abilities of the self-etching primer may result in lack of resin tag formation resulting in low bond strengths.[17]

No statistically significant differences were observed in the μ-TBS of teeth with the normal and mild groups etched for 60 s with H_3PO_4. This is similar to the previous reports by N'gang'a et al.[7] and Ateyah et al.[8] However, teeth with the moderate group etched for 60 s resulted in significantly lower bond strengths, though it improved sufficiently to levels comparable to other groups when etched for 90 s. Al-Sugair et al.[3] reported the presence of a subsurface insoluble organic network in fluorosed teeth (TFI: 4-5) when etched for 45 s. They also reported the reappearance of the typical etching patterns (Type I, II, and III) after treating the enamel with H_3PO_4 for 90 s. This insoluble organic network may have contributed to the significantly low bond strengths reported for the moderate group etched for 60 s in our study and the reappearance of the typical etch patterns for the improvement of the bond strengths when etched for 90 s. However, in contrast, Ateyah et al.[8] reported similar bond strengths for all groups of fluorosis in young patients. They attributed this similarity to the loss of hypermineralyzed surface layer because of using ground enamel as bonding substrate. Moreover, they also reported that bonds tend to fail predominantly cohesively in enamel when the fluorosis was severe. This difference in the fracture pattern was not observed in our study as unequal stress distribution resulting in cohesive bond failures in enamel can be avoided with the μ-TBS test.[15] The bond strengths of non-fluorosed enamel reported in this study are relatively lower than the previous studies, which used the same bonding system.[13,14] Operator variability[18] may have influenced this difference. In addition, the fact that these teeth were also collected from same areas as fluorosed teeth with high fluoride content in water may have contributed to relatively low enamel bond strengths. However, previous studies have indicated that enamel bond strengths in the range of 17-20 MPa as sufficient to ensure retention of composite resin restorations.[19] Moreover, there were no significant differences in the bond strengths with extended priming (90 s) compared to etched groups in non-fluorosed ground enamel. Therefore, non-fluorosed ground enamel does not need prior conditioning with H_3PO_4. However, fluorosed enamel need prior enamel conditioning as fluoroapatite is more resistant to acid dissolution than hydroxyapatite of non-fluorosed teeth. As the self-etching primer has a pH of two[20] compared to 0.6 of H_3PO_4 even extended priming is not sufficient to obtain adequate etching of the acid resistant fluorosed enamel. Therefore, according to the results of this study bonding systems that utilize phosphoric acid etching may be more suitable for bonding fluorosed enamel.

CONCLUSIONS

Phosphoric acid etching prior to application of the self-etching primer bonding system resulted in higher enamel bond strengths in ground fluorosed teeth. Accordingly, teeth with mild fluorosis can be bonded using a similar etching time as non-fluorosed teeth, while moderate fluorosis require longer etching time to obtain adequate bond strengths. Moreover, there are differences in the conditioning patterns obtained with the self-etching primer and H_3PO_4. It is therefore concluded that severity of fluorosis, etching time and pattern influence the bond strengths of the self-etching primer bonding system to fluorosed enamel.

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