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In vitro effects of several surface preparation methods on shear bond strength of orthodontic brackets to caries-like lesions of enamel

ABSTRACT

Aim The aim of this study was to evaluate the efficiency of several surface preparation methods for improving shear bond strength of brackets to demineralised enamel.

Materials and methods Study design: *in vitro* study. Eighty premolars were selected and divided into 5 groups. Group 1 served as the control, while the remaining 4 groups were immersed in a demineralising solution (pH 4.8) for 12 weeks. In groups 1 (control) and 2 (demineralised/control) conventional acid etching was used. In group 3, a solution of 5% sodium hypochlorite (NaOCl) was applied on enamel surface for one minute after acid etching. The brackets in group 4 were bonded with Transbond Plus self-etching primer, and group 5 underwent treatment with a 2% sodium fluoride (NaF) gel, which was applied on the enamel surface for 4 minutes before etching. Shear bond strength (SBS) and adhesive remnant index (ARI) were determined in all groups, and surface morphology was examined under scanning electron microscope (SEM).

Results The mean SBS of acid-etched demineralised enamel was significantly lower than that of acid-etched sound enamel ($p<0.05$). Treatment of caries-

like lesions with 5% NaOCl or self-etching primer failed to improve the bond strength. After NaF treatment and acid etching of demineralised enamel, both type 1 and type 2 etching patterns were observed and the resulting SBS was comparable to that of sound enamel ($p>0.05$).

Conclusion The application of 2% NaF on enamel caries before bracket bonding is an effective way for enhancing the bond strength.

Keywords Bonding; Caries-like lesion; Demineralised enamel; Demineralisation; Bond strength; Self-etching primer; Sodium fluoride; Sodium hypochlorite.

Introduction

The adhesion between enamel and adhesive resin is remarkably dependant on enamel surface properties. If there is any change in the physical and chemical structure of enamel, it may be difficult to achieve sufficient bond strength, because the etching pattern produced with phosphoric acid on normal enamel may not occur on the affected teeth [Mahoney, 2001]. Orthodontic patients occasionally show local or generalised demineralisation in one or more teeth before the start of the treatment. Furthermore, many patients experience the formation of white spot lesions during orthodontic treatment, especially those with poor oral hygiene [Hadler-Olsen et al., 2011; Ogaard et al., 1988; Tufekci et al., 2011; Gorelick et al., 1982]. In these cases clinicians may be obliged to bond brackets to demineralised enamel.

The altered enamel structure of teeth with caries-like lesions may reduce the bond strength of orthodontic brackets. This may result in high failure rates of brackets in clinical conditions, so the orthodontist should frequently rebond one or more accidentally detached brackets. Rebonding not only requires time and additional expenses, but also prolongs the duration of orthodontic treatment and may even increase the severity of white spot lesions, probably causing irreversible damage to the tooth structure.

According to our records, to date no study evaluated the bond strength of brackets bonded to caries-like enamel lesions. Previous authors reported that the bond strength of composite to teeth with molar incisor hypomineralisation (MIH) or hypocalcified amelogenesis imperfecta (AI) can be improved by application of self-etching primer [William et al., 2006a] or by enamel pretreatment with 5% sodium hypochlorite (NaOCl) [Mathu-Muju and Wright, 2006; Saroglu et al., 2006; Veneczel et al., 1994; William et al., 2006b]. Although the

decreased mineral content and the presence of enamel porosities are common in these conditions as in caries-like lesions, teeth with molar incisor hypomineralisation or hypocalcified amelogenesis imperfecta may also have other structural defects, so the result cannot be simply generalised to caries-like lesions. There is also some evidence that fluoride treatment before acid etching of enamel caries or hypomineralised enamel can restore the fluoride lost during lesion formation, while producing etching patterns that are suitable for placement of composite resins [Hicks and Silverstone, 1983; Hicks et al., 1984; Tandon and Mathew, 1997].

The purposes of the present study were to evaluate the effects of several enamel preparation methods, including 5% NaOCl, Transbond Plus self-etching primer, and 2% sodium fluoride (NaF), on shear bond strength (SBS) of brackets and type of bond failure in caries-like enamel lesions, and investigate the etching patterns produced by these methods under scanning electron microscope (SEM).

Materials and methods

Eighty premolar teeth extracted for orthodontic purpose were used in this study. The buccal surfaces of the teeth were intact and without any hypoplasia or enamel cracks. The teeth were stored in a 0.2 % (wt/vol) thymol solution for two weeks to inhibit bacterial growth and then kept in normal saline solution until the time of the study.

Each sample was randomly assigned to one of 5 groups of sixteen each. The first group was considered as the control group and the other groups were immersed in a cariogenic solution for 12 weeks to produce demineralised enamel. This solution consisted of 2.2 mM CaCl₂, 2.2 mM NaH₂PO₄, and 50 mM acetic acid with pH adjusted at 4.8 using potassium hydroxide (KOH) [Alencar et al., 2009]. Each tooth was individually immersed in a plastic container having 10 ml of cariogenic solution, and the solution was changed every week.

Before bracket bonding, the buccal surfaces of the teeth were cleaned with non-fluoridated pumice slurry and rubber prophylactic cups for 5 seconds, then rinsed with water and air-dried. Stainless steel standard edgewise second premolar brackets (0.018-inch slot; Dentaurum, Ispringen, Germany) were used in this study.

The bonding procedures were as follows.

- Group 1 (control): the sound enamel surface was etched with a 37% phosphoric acid gel for 30 seconds. The tooth was rinsed with a copious amount of water, and dried with an oil-free air spray. Then, a thin coat of Transbond XT primer (3M Unitek, Monrovia, California, USA) was applied on the enamel surface and the bracket was placed in the center of the crown with the use of Transbond XT adhesive (3M Unitek). The excess composite was removed from the

periphery of the base with a dental explorer and the bracket was light cured for 40 seconds from occlusal, gingival, mesial and distal directions (10 seconds each) using Bluephase C8 (Ivoclar Vivadent, Schaan, Liechtenstein) light-emitting diode (LED) at power density of 650 mW/cm².

- Group 2 (demineralised/control): the bonding procedure was the same as the control group (Group 1), but brackets were bonded on demineralised enamel.
- Group 3 (demineralised/NaOCl): after acid conditioning, a 5% sodium hypochlorite solution was applied on the enamel surface for 1 minute, then rinsed with water and air-dried. The subsequent steps were the same as the control group.
- Group 4 (demineralised/SEP): Transbond Plus self-etching primer (3M, Unitek) was activated according to the manufacturers' instructions. The primer was rubbed on the enamel surface for 10 seconds, and then thinned with a gentle air burst. Brackets were bonded with Transbond XT adhesive and light cured as the control group.
- Group 5 (demineralised/NaF): in this group, a 2% neutral sodium fluoride gel (Sultan Healthcare Inc., Englewood, New Jersey, USA) was applied on the enamel surface for 4 minutes. Subsequently, the teeth were rinsed with water for two consecutive periods of 5 minutes each to remove any readily-soluble reaction products [(Hicks et al., 1984)]. Then the teeth were etched with 37% phosphoric acid gel and bonded as the control group.

After enamel treatment and before placing the bonding agent, one specimen from each experimental group was prepared for scanning electron microscope (SEM) evaluation to determine the treatment effects on surface morphology of enamel caries.

The bonded teeth were kept in deionised water at 37°C for 24 hours and then mounted in self curing acrylic resin so that the buccal surface of the tooth was parallel to the direction of the debonding force. Shear bond strength test was performed on Zwick testing machine (model Z250, Zwick GmbH & Co, Ulm, Germany), using a cross head speed of 1 mm per minute until failure. The force required to fracture the bracket-tooth interface was recorded in Newtons and then converted to megapascals (N/mm²) by dividing the force value by the bracket base area (13.6 mm²).

After debonding, the teeth were examined at ×10 magnification to score the amount of remaining adhesive on the enamel surface using adhesive remnant index (ARI) of Artun and Bergland [1984]:

- 0: no adhesive remained on the tooth;
- 1: less than 50% of the adhesive remained on the tooth;
- 2: more than 50% of the adhesive remained on the tooth;
- 3: 100% of the adhesive remained on the tooth, with

GROUP	DEFINITION	NUMBER	MEAN (MPA)	SD	RANGE	DUNCANA
1	control	15	9.62	2.94	4.62_13.64	A
2	Demineralised/control	15	6.28	2.40	3.29_10.70	B
3	Demineralised/ NaOCl	15	6.20	2.09	2.54_8.90	B
4	Demineralised/SEP	15	6.03	1.75	2.04_8.11	B
5	Demineralised/NaF	15	8.92	2.97	5.47_16.43	A

aDuncan post-hoc comparison test; Different letters indicate significant differences between the groups.

TAB 1 Descriptive statistics and the results of the Duncan test comparing the shear bond strength (SBS) values of the study groups.

ARI	0		1		2		3	
	Number	%	Number	%	Number	%	Number	%
Group 1	5	37.5	4	25	4	25	2	12.5
Group 2	10	66.7	4	26.7	1	6.6	0	0
Group 3	10	66.7	4	26.7	1	6.6	0	0
Group 4	11	73.4	2	13.3	0	0	2	13.3
Group 5	11	80	2	6.7	1	6.7	1	6.6

TABLE 2 ARI scores in the study groups.

distinct impression of the bracket base.

Statistical analysis: The data were analysed by means of SPSS (Statistical Package for Social Sciences, Version 11.5, Chicago, Illinois, USA) software. One-way analysis of variance (ANOVA) was used to assess any significant difference in SBS values among the study groups, followed by Duncan multiple range test to evaluate the pairwise differences. The chi-square test was used to compare ARI scores among the study groups. Significance was determined at $p<0.05$.

Results

The descriptive statistics including mean, standard deviation (SD), and range regarding bond strength values of the study groups are presented in Table 1. ANOVA indicated a significant difference in SBS values between groups ($p=0.001$). Multiple comparisons by Duncan test (Table 1) revealed that groups 1 (control) and 5 (demineralised/NaF) had the highest SBS, with no statistical difference between them. The other three groups i.e. demineralised control (Group 2), demineralised/NaOCl (Group 3) and demineralised/SEP (Group 4) showed the lowest SBS, and did not statistically differ from each other.

The results for the ARI scores are presented in Table 2. The chi-square test indicated no significant difference in the distributions of ARI scores among the study groups ($p=0.225$).

Surface morphology

Enamel etching patterns observed on the representative

specimens were described according to the classification of Silverstone et al. [1975]:

- a. Type 1 pattern, the prism core material is preferentially removed;
- b. Type 2 pattern, preferential removal of prism peripheral regions;
- c. Type 3 pattern, general removal of tooth material occurs without relation to prism morphology.

The micrograph of hypomineralised enamel etched with 37% phosphoric acid agent represented areas with Type 3 etching pattern surrounded by sparse areas with featureless to slightly etched appearance (Fig. 1). After treatment with 5% NaOCl, a Type II etching pattern was observed with limited dissolution of prism peripheries

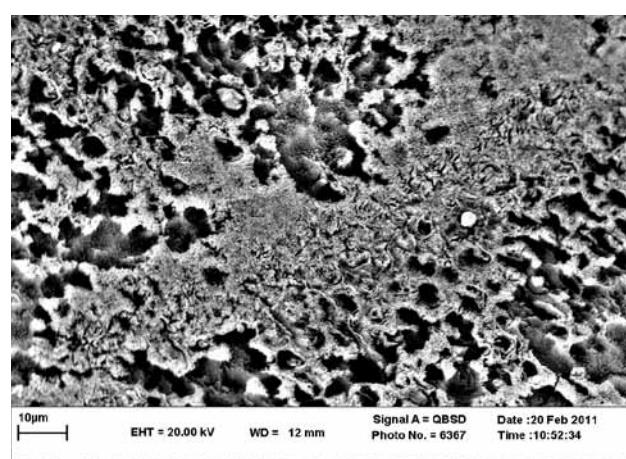


FIG. 1 SEM micrograph of hypomineralised enamel treated with 37% phosphoric acid (Magnification $\times 3000$).

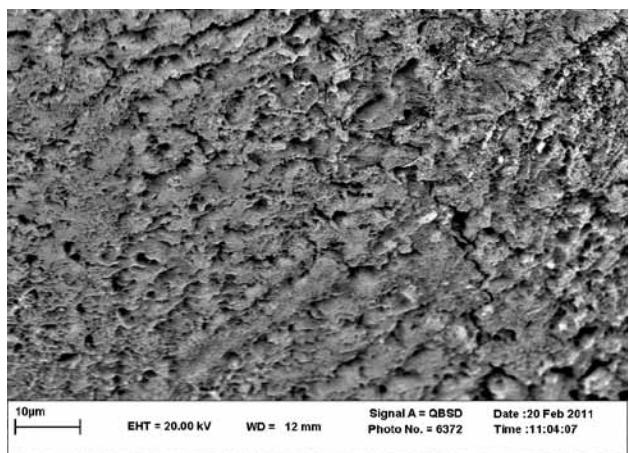


FIG 2 SEM micrograph of hypomineralised enamel after acid etching and NaOCl treatment (Magnification $\times 4000$).

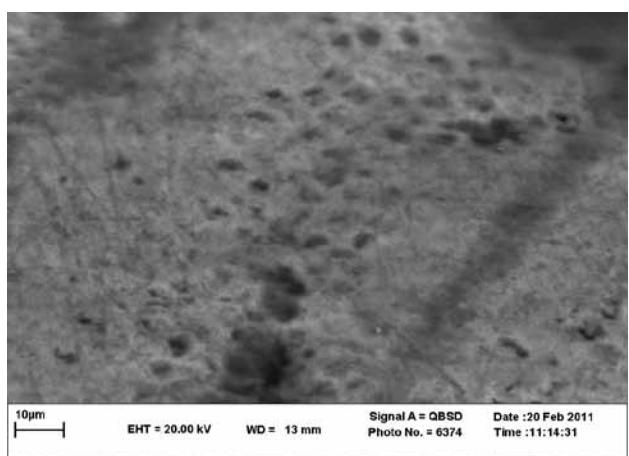


FIG 3 SEM micrograph of hypomineralised enamel after application of Transbond Plus self-etching primer (Magnification $\times 3000$).

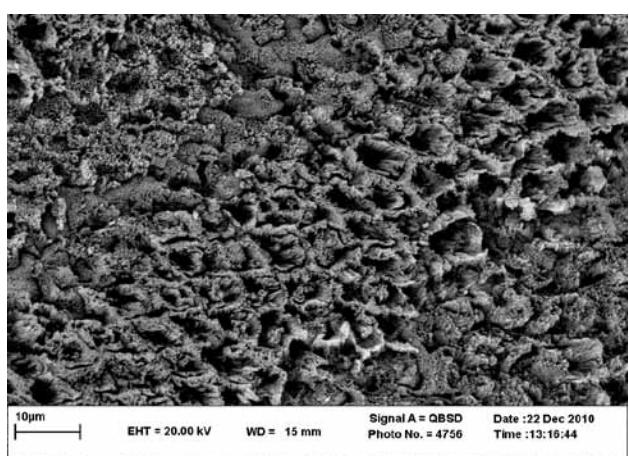


FIG 4 SEM micrograph of hypomineralised enamel after NaF treatment and acid etching (Magnification $\times 4000$).

producing a honeycomb appearance throughout the enamel surface (Fig. 2). Application of self-etching primer created an uneven etching effect, exhibiting islands of Type 1 etching pattern within large areas that remained unetched (Fig. 3). After NaF treatment and acid-etching of hypomineralised enamel, both Type 1 and Type 2 etching patterns were observed and the surface morphology seemed conducive to bonding (Fig. 4).

Discussion

The present study investigated the effects of several preparation methods of demineralised enamel on SBS values of orthodontic brackets. As expected, bond strength of brackets to acid-etched demineralised enamel was significantly lower than that for acid-etched sound enamel (6.28 MPa versus 9.62 MPa). The lower bond strength of brackets bonded to demineralised enamel may be related to its chemical and morphological structure that shows higher enamel porosity and lower mineral concentration than sound enamel [Hicks and Silverstone, 1985].

Pretreatment with a 5% NaOCl solution failed to improve the bond strength of brackets to demineralised enamel. Previous studies recommended pretreatment of teeth with molar incisor hypomineralization (MIH) [Mathu-Muju and Wright, 2006; William et al., 2006b] or hypocalcified amelogenesis imperfecta (AI) [Saroglu et al., 2006; Venezie et al., 1994] using 5% NaOCl in order to remove intrinsic enamel proteins and thus enhancing the bond strength. However, there is no study evaluating the effects of enamel deproteinisation on SBS values of brackets bonded to teeth with caries-like enamel lesions. It should be noted that Venezie et al. [1994] used NaOCl before acid conditioning of the enamel surface, but Saroglu et al. [2006] and Sonmez et al. [2009] used this technique after acid etching. William et al. [2006b] recommended initial etching of the hypomineralised defect with 37% phosphoric acid, applying 5% NaOCl and then re-etching the enamel surface before resin placement. It is possible that the latter technique provides higher bond strength to demineralised enamel, but further research is required to confirm this assumption.

Self-etching primers have gained a great popularity in today orthodontic practice, because they simplify treatment by reducing the steps required to prepare enamel for bonding, and thus reducing chair time. Transbond Plus, an orthodontic self-etching primer with a pH value of approximately 1, has shown promising results in several studies [Reis et al., 2008; Zanarini et al., 2011]. However, in the present study, the application of Transbond Plus self-etching primer did not improve the SBS of brackets to demineralised enamel. In a previous study, William et al. [2006a] explored the effects of self-etching adhesives on teeth

affected with molar incisor hypomineralisation and reported better bonding of a self-etch adhesive than a total-etch system to hypomineralised enamel, although the difference was not statistically significant. They ascribed this improved bonding to 2 factors:

1. elimination of rinsing in self-etching systems that prevents retention of excessive water in the porous lattice of hypocalcified enamel;
2. the chemical in addition to the mechanical bonding of self-etch primers to hypomineralised enamel.

Although Transbond Plus self-etching primer was not effective in enhancing the bond strength to caries-like lesions, it should be noted that the etching efficacy and the penetration depth of self-etching primers may be considerably different in contemporary self-etching systems [Pashley and Tay, 2001], and this may affect the bond strength, so further research with different self-etching primers is suggested on this subject.

In this study, NaF treatment of demineralised enamel increased the bond strength from 6.28 to 8.92 MPa, which was not statistically different from the control group. Fluoride application before, after or combined with acid etching has been used for years as a method of providing rapid uptake of fluoride into normal enamel, resulting in a higher concentration of fluoride in etched enamel than fluoride-treated sound enamel [(Kochavi et al., 1975; Belser et al., 1975)]. It has been reported that fluoride treatment followed by acid etching of caries-like lesions produced surface coating composed of calcium fluoride, providing an adequate source of mineral for remineralization, while producing etching patterns that resembled those described for etched sound enamel [Hicks and Silverstone, 1983; Hicks et al., 1984; Tandon and Mathew, 1997]. Although Hicks and Silverstone [1983], Hicks et al. [1984] and Tandon and Mathew [1997] did not measure the SBS, due to the suitability of etching patterns, they predicted that the bond strength of resins placed over fluoride-treated caries-like lesions would be comparable to that for normal enamel. This assumption was confirmed in the present study. The etching times of 1 or 2 minutes has been recommended in previous studies to be used on fluoride-treated caries-like lesions [Hicks and Silverstone, 1983; Hicks et al., 1984; Tandon and Mathew, 1997] but these etching times are higher than the etching time of 30 seconds usually employed in clinical practice [Sanchez-Quevedo et al., 2006]. For this reason and to have a proper comparison with other groups, the etching time of 30 seconds was preferred in this study.

Adhesive remnant index (ARI) is considered a method of determining bond failure interface. The results for the ARI scores indicated a higher frequency of bond failure at the enamel-adhesive interface in all demineralised groups (more than 66%) compared to that for normal enamel (37.5%). However, the difference in failure types among the study groups was not statistically

significant.

The SEM evaluation revealed remarkable differences in surface morphology between the groups, explaining to some extent the difference in SBS measurements. Conventional acid etching produced a type 3 etching pattern on demineralised enamel surrounded by islets with slightly etched appearance. The honeycomb topography after NaOCl treatment was associated with slight dissolution of prism peripheral regions, but it was not a characteristic Type 2 etching pattern as described by Silverstone et al. [1975]. The honeycomb appearance throughout the enamel surface was possibly due to the oxidant nature of NaOCl treatment. A mildly etched appearance after applying self-etching primer was consistent with observations of Pashley and Tay [2001] who reported faint relief of enamel surface following use of a self-etching adhesive with moderate aggressiveness. The surface morphology after acid-etching of NaF-treated demineralised enamel was considered acceptable for resin placement, because a mixture of characteristic Type 1 and Type 2 etching patterns could be detected on the surface.

If 7.8 MPa is accepted as the minimum bond strength required in orthodontic clinical practice [Reynolds, 1975], it can be drawn that fluoride treatment followed by acid etching provides sufficient bond strength of brackets to caries-like lesions of enamel, while enhancing remineralisation of the underlying lesion by creating a good reservoir of mineral for deposition. Considering the high prevalence and rapid development of dental caries in hypomineralised enamel, achieving sufficient bond strength while allowing enamel remineralisation to occur can be considered a great benefit for the orthodontist as well as for the child affected. The effect of fluoride application before acid etching on SBS values of teeth affected with molar incisor hypomineralisation or hypocalcified amelogenesis imperfecta needs further clarification in a future study. The change in fluoride concentration or pH of the prophylactic agent may affect the bond strength, so further research is suggested in this field.

Conclusion

- 1 The SBS of brackets bonded to acid-etched demineralised enamel was significantly lower than that for acid-etched sound enamel.
- 2 Enamel deproteinisation with a 5% sodium hypochlorite solution or the use of Transbond Plus self-etching primer failed to improve the adhesion of brackets to caries-like lesions of enamel.
- 3 The application of 2% sodium fluoride for 4 minutes before acid etching the demineralised enamel produced characteristic Type 1 and Type 2 etching patterns and the resulting bond strength was comparable to that of acid-etched sound enamel.

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