Shear bond strength evaluation of chemically-cured and light-cured orthodontic adhesives after enamel deproteinization with 5.25% sodium hypochlorite

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Abstract. This study aimed to assess the effect of enamel deproteinization with 5.25% sodium hypochlorite (NaOCl) before etching on the shear bond strength (SBS) of Unite (UN; 3M Unitek) and Xihu-BIOM adhesive (XB). Fifty-two maxillary first premolars were divided into four groups: (1) UN and (2) XB according to manufacturer's recommendation and (3) UN and (4) XB deproteinized with 5.25% NaOCl. Brackets were bonded, and a mechanical test was performed using a universal testing machine. The mean SBS value for groups A1, A2, B1, and B2 was 13.51 $\pm$ 2.552, 14.36 $\pm$ 2.902, 16.43 $\pm$ 2.615, and 13.05 $\pm$ 2.348 MPa, respectively. A statistically significant difference in SBSs was observed between chemically cured groups and between group B ($p < 0.05$). No statistically significant difference in SBSs was observed between light-cured adhesive groups and between group A ($p > 0.05$). NaOCl enamel deproteinization before acid etching has a significant effect on the SBS of Unite adhesive, but not on that of the Xihu-BIOM adhesive. Furthermore, a significant difference in the SBS of Unite and Xihu-BIOM adhesives within the enamel deproteinization group was observed in this study.

1. Introduction

Since the development of Buonocore's acid etch technique and Newman's orthodontic bracket bonding, many adhesive variants have been commercialized [1,2]. The most popular adhesive agent is chemically cured adhesive [2,3]. The main disadvantages of this material are the very short polymerization time and the fact that it cannot be manipulated [2]. Later, light-cured adhesive was introduced as an alternative. Such adhesives are cured under metal-based brackets by direct illumination on each side of the bracket and by trans illumination through the tooth structure [2,4,5]. Under visible light irradiation, the adhesives undergo rapid polymerization. Therefore, the working time can be adjusted and the bracket can be positioned more accurately [1,2].

Bracket failure is a common occurrence (0.5%–17.6%) during orthodontic treatment [5]. The shear bond strength (SBS) of a bonding agent should ideally be high enough to withstand intraoral forces throughout the treatment, and it should not lead to enamel damage during debonding [6]. Adhesion to enamel depends on the quality and quantity of the etched enamel surface. Previous study found that enamel deproteinization with sodium hypochlorite (NaOCl) before phosphoric acid etching doubled the etched surface area compared to that in conventional phosphoric acid etching [7]. Many studies have investigated enamel deproteinization with 5.25% NaOCl [7-11]. However, no study has compared the SBS of chemically cured and light-cured composite resin with or without sodium hypochlorite enamel deproteinization. In the present study, the SBS of chemically cured and light-cured orthodontic adhesives after enamel deproteinization with 5.25% NaOCl was evaluated.

2. Materials and Methods

Fifty-two human maxillary first premolars were collected, cleaned, and stored in saline solution at room temperature. The teeth were selected only if they had intact buccal enamel, had not been treated with chemical agents (such as H_2O_2, NaOCl, or alcohol), had no surface crack, and were caries-free. The teeth were cleaned in an ultrasonic cleaner for 5 min. Then, they were mounted on resin block frames and stored in saline solution.

These teeth were randomly divided into four groups (n = 13) and were treated as follows. Initially, prophylaxis was performed with a non-fluoride pumice paste and low-speed brush for 10 s, followed by rinsing for 15 s and drying for 10 s. In group A1, teeth were etched with 37% phosphoric acid for 15 s, rinsed, and dried. Primer was applied on the tooth surface and bracket mesh, and then, the tooth was bonded with Unite (Un; 3M Unitek). In group A2, teeth were etched with 37% phosphoric acid for 15 s, rinsed, and dried. Then, the teeth were subjected to primer application and fixation with Xihu-BIOM (XB) according to the manufacturer's recommendation. In group B1, teeth were deproteinized with 5.25% NaOCl, etched with 37% phosphoric acid, and bonded with UN. In group B2, teeth were deproteinized with 5.25% NaOCl, etched with 37% phosphoric acid, and bonded with XB. Before bonding, the width and length of each bracket were measured using Digimatic Caliper (Mitutoyo). The brackets were placed on the buccal surface at the center of the crown. In the XB group, each bracket was polymerized under LED light for 40 s (10 s on each side). After bonding, the specimens were stored in artificial saliva at 37 °C for 24 h. Each specimen was then subjected to a shear load test by using the Shimazu AG-5000 universal testing machine at a cross-head speed of 0.5 mm/min. Force was applied parallel to the tooth surface, and the blade was fixed on the top of the bracket and enamel junction. The shear load at point failure was recorded in units of kgF. The SBS was calculated as the quotient of the debonding force and the area of the bracket base in units of MPa. The surfaces of the enamel and bracket mesh were then examined using a Nikon SMZ800 stereomicroscope with 2x magnification to determine the location of adhesive failure. The SBS and Adhesive Remnant Index (ARI) values were analyzed statistically by the independent t-test by using SPSS V.20.

3. Results and Discussion

3.1 Results

Table 1 and Figure 1 show descriptive statistic data of the mean SBS of each group. The mean SBS of A1 and B1 was 13. 51 + 2.55 and 16.43 + 2.62 MPa, respectively. Furthermore, the mean SBS of A2 and B2 was 14.36 + 2.90 and 13.05 + 2.35 MPa, respectively.

Table 1. Group, mean, standard deviation, maximum, and minimum of shear bond strength values of evaluated groups

Group	n	Mean shear bond strength (MPa)	SD	Maximum	Minimum
Unite without NaOCl (A1)	13	13.5086	2.55212	12.052	14.965
Unite with NaOCl (B1)	13	16.4288	2.61501	14.972	17.885
Xihu-BIOM without NaOCl (A2)	13	14.3560	2.90157	12.900	15.812
Xihu-BIOM with NaOCl (B2)	13	13.0489	2.34776	11.592	14.505

The statistical significance in this study was calculated by using independent t-test analysis. According to independent t-test analysis of the SBS between groups, groups A1 and B1 showed a statistically significant difference with $p = 0.008$ (i.e., $p < 0.05$). Furthermore, groups A2 and B2 did not show a statistically significant difference, with p = 0.219 (i.e., p > 0.05). Table 2 shows the analysis data.

Table 2. Statistical Analysis of Difference between Unite and Xihu-BIOM Adhesive Groups With and Without Enamel Deproteinization

Adhesive	Mean without deproteinization (MPa)	Mean with deproteinization (MPa)	Mean difference (MPa)	p
Unite	13.5086	16.4288	-2.92018	0.008*
Xihu-BIOM	14.3560	13.0489	1.30716	0.219

Level of significance p < 0.05; * statistically significant

Independent t-test analysis was also performed to compare the means between the without and with deproteinization groups (Table 3). Groups A1 and A2 did not show a statistically significant difference, with p = 0.437 (i.e., p > 0.05). Furthermore, groups B1 and B2 showed a statistically significant difference, with p = 0.002 (i.e., p < 0.05).

Table 3. Statistical analysis of difference between with and without enamel deproteinization groups for unite and Xihu-BIOM adhesives

	Mean of Unite (MPa)	Mean of Xihu-BIOM (MPa)	Mean difference (MPa)	p
Without deproteinization	13.5086	14.3560	0.84738	0.437
With deproteinization	16.4288	13.0489	3.37995	0.002*

Level of significance p < 0.05; * Statistically significant

The result of the enamel surface examination using the stereomicroscope was classified using the ARI score proposed by Artun and Bergland (1984) [12]. Score of 0, 1, 2, and 3 indicate no adhesive, less than half of the adhesive, more than half of the adhesive, and all of the adhesive left on the tooth with distinct impression of bracket base, respectively. Table 4 shows the frequency distributions of the ARI score of the evaluated groups.

Table 4. Frequency distributions of ARI score of evaluated groups

Group	n	ARI score			
		0	1	2	3
Unite without NaOCl (A1)	13	0	9	3	1
Unite with NaOCl (B1)	13	0	6	5	2
Xihu-BIOM without NaOCl (A2)	13	0	6	6	1
Xihu-BIOM with NaOCl (B2)	13	0	5	5	3

3.2 Discussion

Since the introduction of the acid etch technique and bracket bonding system, many adhesive bonding agents have been developed. Nonetheless, bracket failure still occurs during orthodontic treatment. According to Espinosa *et al*. (2008) [7], the two key factors influencing adhesive failure were the quantity of the etched surface and the quality of the etching pattern. Phosphoric acid mainly acts on mineralized tissue, and it does not eliminate organic material from the enamel surface. The existence of this organic material reduces the quantity and quality of acid etching [13]. To overcome this limitation, enamel deproteinization must be performed before acid etching.

Sodium hypochlorite is a nonspecific proteolytic agent that effectively eliminates organic material at room temperature. Espinosa *et al*. [7] found that enamel deproteinization for 60 s with 5.25% NaOCl increases the quantity and quality of acid etching. In accordance with this finding, a study [11] found that enamel deproteinization with 5.25% NaOCl before etching with 37% phosphoric acid enhances the shear bond strength of RMGIC. This study evaluated the shear bond strength of brackets bonded with Unite and Xihu-BIOM orthodontic adhesives. This study aimed to determine whether applying NaOCl for 1 min before etching increases the SBS of the bracket. In the Unite group, the mean SBS without and with enamel deproteinization with 5.25% NaOCl before etching with 37% phosphoric acid was 13.51 ± 2.55 and 16.43 ± 2.62 MPa, respectively. Deproteinization increased Unite adhesive's SBS by 2.92 MPa, and the difference between the two groups was statistically significant ($p = 0.008$). In the Xihu-BIOM group, the mean SBS without and with deproteinization was 14.36 ± 2.90 and 13.05 ± 2.35 MPa, respectively. Deproteinization decreased Xihu-BIOM adhesive's SBS slightly, but the difference between the two groups was not statistically significant ($p = 0.219$). In all groups evaluated in this study, the mean SBS exceeded the minimum required orthodontic SBS value of 5.9–7.8 MPa as often cited in the literature for clinical success[14].

The significant increase in SBS in the Unite group and the insignificant decrease in the Xihu-BIOM group may be influenced by the difference in bonding method and adhesive viscosity. In the Unite group, primer was applied to the enamel surface and bracket mesh, whereas in the Xihu-BIOM group, primer was applied only to the enamel surface. The primer improved the adhesive's flow and penetration to the adhesive surface. Primer application increased the penetration of the adhesive into the enamel surface and bracket mesh. In clinical observations, Unite shows higher viscosity compared to Xihu-BIOM. High viscosity may complicate resin penetration into the etched enamel. Deproteinization increased the enamel's surface tension, thereby improving its wettability and making adhesive penetration easier [15]. This leads the significant increase in SBS in the Unite group. Xihu-BIOM's relatively lower viscosity leads to better adhesive penetration into the enamel. Deproteinization did not result in any significant difference in adhesive penetration. Another study also reported a similar finding with a Transbond XT group after deproteinization [11].

The ARI score showed that Unite adhesive without deproteinization had more bond failure within the adhesive-enamel interface compared to other groups. This finding supports the previous assumption that Unite's viscosity complicates adhesive penetration into the enamel. Inadequate adhesive penetration leads to inadequate adhesion of the adhesive to the enamel, resulting in less adhesive remnant on the enamel surface. The effect of deproteinization in the Unite group resulted in more bond failure within the adhesive-bracket interface, even though this increase was nonsignificant. This means that deproteinization increased the adhesion of the adhesive to the enamel surface. For Xihu-BIOM, the ARI score showed a balance in the bond failure locations between the enamel-adhesive interface and adhesive-bracket interface. This also supports the assumption of better adhesive penetration owing to the relatively lower viscosity of the Xihu-BIOM adhesive. In all groups with deproteinization, the ARI score indicated an insignificant increase in the adhesion of the adhesive to the enamel surface.

4. Conclusion

Enamel deproteinization with 5.25% NaOCl for 1 min before acid etching significantly enhances the SBS of brackets bonded with Unite. With regard to the ARI score, applying 5.25% NaOCl to the enamel surface results in an insignificant increase in the adhesion of the adhesive to the enamel surface.

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