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Impact of fluoride-releasing orthodontic adhesives on the shear bond strength of orthodontic brackets to eroded enamel following different surface treatment protocols

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Abstract

PURPOSE: To assess the impact of enamel surface treatment protocols and the types of adhesive materials on the shear bond strength (SBS) of brackets to eroded enamel substrate.

MATERIALS AND METHODS: Eighty extracted premolars were randomly assigned to four main groups in which group C (no treatment) was the control group. The remaining groups were exposed to an erosion challenge through short-term acidic exposure to HCl solution (0.01 M, pH 2.3) for 30 s, with an agitation speed of 50 rpm at an environmental temperature of 25°C. The eroded enamel surface within each group was treated as follows: group N received no treatment; in group P, the eroded enamel was treated with 35% phosphoric acid (Ultradent Products, South Jordan, UT, USA) for 15 s, followed by a rinse for 10 s; and in group F, the eroded enamel was treated with fluoride gel (Bifluorid 12; Voco-GmbH, Cuxhaven, Germany) for 4 min. The brackets were bonded with either a resin composite adhesive (Transbond XT; light-cure adhesive, 3M Unitek, CA, USA) or resin-modified glass ionomer cement (Fuji Ortho LC-GC Corporation, Japan). The specimens were tested for SBS, and the bond failure was assessed according to the adhesive remnant index (ARI). Analysis of variance (ANOVA) and Tukey's post-hoc tests ($P < 0.05$) were used to compare the SBS of the groups. The ARI values between the groups were recorded.

RESULTS: Statistically significant differences were found among the tested variables ($P < 0.05$). Group P showed the highest mean SBS values regardless of the type of adhesive used, and the difference was statistically significant ($P < 0.05$). The application of the fluoride gel showed no statistically significant improvement in SBS values. The failure mode distribution among the test groups indicated that failures at the adhesive-bracket interface were predominant in group C compared with the other study groups.

CONCLUSIONS: Fluoride pretreatment, which was used to remineralize the eroded enamel surfaces before bonding, resulted in a decrease in the SBS of the orthodontic brackets *in vitro* compared with the other treated groups. The use of fluoride-releasing adhesive also enhances bonding to the eroded enamel surfaces.

Keywords:

Dental erosion, dentistry, orthodontic adhesives, orthodontics, shear bond strength

Introduction

Dental erosion is defined as the loss of tooth surface induced by acid without

bacterial involvement. In the initial stage of development, erosion is considered a superficial demineralization.^[1] This corresponds to the softening of the enamel surface, resulting in the loss of strength,

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resistance, and structural components of the tooth. Erosive tooth wear develops in more advanced stages, either by prolonged demineralization of the tooth surface or mechanical stress.^[2-4]

Erosive dental wear has become a more prevalent and growing clinical problem.

Excessive intake of acidic food and drinks has led to an increase in the incidence of erosive tooth wear. Gastroesophageal reflux and eating disorders often result in erosive lesions.^[5,6] The key to avoiding the progression of erosive lesions is to lower the direct interaction of exogenous or endogenous acids with the tooth surface.^[7] However, this is not always achievable, and strategies have been suggested to restore enamel loss at the initial stages of dental erosion. The prevention of enamel erosion and remineralization of enamel through orthodontic treatment is a crucial issue.^[2-4,8,9]

In early erosive lesions, remineralization occurs as a repair process. The process of remineralization entails inducing hydroxyapatite crystals to form through the precipitation of calcium and phosphate ions aided by fluoride.^[10] Fluoride ions influence this process through two mechanisms: hydroxyl ion substitution into hydroxyapatite and formation of calcium fluoride.^[11]

For the prevention of erosion in tooth surfaces with fixed orthodontic appliances, various approaches have been implemented, one of which is the topical application of fluoride. Fluoride can be administered in various forms (toothpaste, mouth rinse, gels, varnishes, and fluoride-releasing bonding materials) for patients undergoing orthodontic therapy. At low concentrations, fluoride has been proven to promote remineralization across orthodontic brackets.^[12-14] Fluoride-containing adhesives have the potential to release and absorb fluoride to protect teeth from erosion. The majority of the fluoride is released during the setting reaction, with only a small amount of long-term fluoride release remaining.^[14] Glass ionomer cements (GICs) and composite resins have been combined to provide increased fluoride release while maintaining bond strength. Resin-modified glass ionomers (RMGIs) are GIC-composite composed of two components that have a true acid-base reaction on mixing. The physical properties of RMGIs are better. Between the cement and the tooth surface, they have a diffusion-based adhesion that releases fluoride continuously.^[14]

Resin infiltration is a different approach that has been developed to protect the enamel from dental erosion. The use of enamel-penetrating, low-viscosity light-curing resins have been suggested as a method for penetrating and protecting against incipient erosive lesions.^[15] The

purpose of this approach is to occlude pores of untreated enamel lesions by creating a diffusion barrier between the enamel and the acidic attack.^[16]

Bonding orthodontic materials to a weakened tooth structure has been a crucial problem in orthodontics. Reduced bracket bond strength during orthodontic therapy with fixed appliances may increase the incidence of bracket detachment. Frequent failure of the brackets leads to a prolonged treatment period and thus patient dissatisfaction. The use of durable adhesive materials is therefore needed.^[17,18]

Orthodontic brackets are subjected to a combination of shear, tensile, and torsion forces inside the mouth.^[19] The bond strength of orthodontic adhesives varies depending on several factors, such as the type of adhesive used, the bracket design, enamel morphology, and orthodontist technique. The ideal adhesive for orthodontic bracket bonding must have sufficient bond strength to prevent detachment during regular oral function and to distribute the needed orthodontic forces to the tooth.^[2,19] Moreover, when orthodontic therapy is complete, the adhesives should be removed easily without damaging the tooth surface.^[20]

Several studies have reported on the impact of different surface treatment methods on the shear bond strength (SBS) of orthodontic adhesives to eroded enamel.^[21,22] To the best of the author's knowledge, no study has evaluated the impact of lesions resulting from gastric hyperacidity on these bond strengths. The goal of the present study is, therefore, to determine and compare the effects of different enamel surface pretreatment methods combined with fluoride-releasing adhesives on the SBS of orthodontic brackets bonded to eroded enamel. The null hypotheses were as follows:

- (1) Different enamel surface pretreatments have no impact on the SBS.
- (2) Different fluoride-releasing orthodontic adhesives have no effect on the SBS.

Materials and Methods

Specimen preparation

Eighty recently extracted sound human maxillary premolar teeth were collected following the patients' verbal consent to use their teeth in the study. Approval to utilize human teeth was acquired from the Dentistry Research Ethics Committee of Taibah University Dental College and Hospital (TUCDREC/06092020/NALTHAGAFI). Adherent soft tissue was removed with a disposable scalpel, and then the teeth were stored in distilled water until use. All teeth were cleansed using a mixture of water and fluoride-free pumice in a

slow-speed rubber prophylaxis cup for 2 or 3 s, washed thoroughly with water, and air-dried. The teeth were divided randomly into four groups of 20 specimens each.

Initial evaluation of enamel hardness

The superficial layer of the buccal surface of the teeth was polished and flattened in order to assess the hardness.^[4] Superficial hardness was evaluated using a Vickers indenter linked to a microhardness tester (FutureTech, Tokyo, Japan). Six indentations per test were made at an interval of 300 μm in the middle section of each specimen. The indentation load was 100 g with 15 s dwell time. Any specimen with a mean hardness of 10% above or below the general mean was excluded (mean surface hardness of 334.56 ± 30.71 VHN).^[20]

Evaluation of enamel hardness after erosive lesion formation

All teeth were exposed to an erosion challenge, except for the control group (group C). The erosive challenge was based on that proposed by Saker *et al.*^[23] An erosive lesion was produced by subjecting the enamel specimens to a short-term acidic exposure with hydrochloric acid (HCl) solution (0.01 M, pH 2.3) for 30 s, with an agitation speed of 50 rpm at an environmental temperature of 25°C.^[23,24] Surface hardness was assessed again to verify the formation of the lesion (mean surface hardness of 180 ± 22.3 VHN). Following the erosive lesion exposure, each specimen was washed in distilled water for approximately 30 s and immersed in 20 mL of artificial saliva for 30 min.^[20]

Experimental groups

Eighty specimens with erosive lesions were randomly divided equally into four treatment groups according to the etching protocol ($n = 20$ each) as follows:

Group C: Control group, intact enamel etched with 35% phosphoric acid-etching gel (Ultradent Products, South Jordan, UT, USA) for 30 s, followed by a rinse for 10 s and air-dried gently.

Group N: Eroded enamel without any etching.

Group P: Eroded enamel treated with 35% phosphoric acid-etching gel (Ultradent Products, South Jordan, UT, USA) for 15 s, followed by a rinse for 10 s and air-dried gently.

Group F: Eroded enamel treated with fluoride gel (Bifluorid 12; Voco-GmbH, Cuxhaven, Germany). The fluoride gel was applied for 4 min before bonding, with the aid of a cotton tip, and was later removed by squirting deionized water to rinse thoroughly.

Bonding procedures

The bonding procedure was performed by one of the two orthodontic adhesive systems in accordance with the manufacturer's instructions, which are as follows:

A liquid primer Transbond XT (primer, 3M Unitek, CA, USA) was applied to the etched enamel surfaces with a disposable applicator, which was then blown using a gentle air burst to dry the primer into a thin film. The applied primer was polymerized via a light-curing unit (Elipar S10, 3 M ESPE Co., Seefeld, Germany) for 10 s. After the primer was applied to all experimental groups, each treatment group was then subdivided into the following subgroups ($n = 10$) based on the type of orthodontic adhesive used:

- Subgroup 1: Resin-modified glass ionomer cement (Fuji Ortho LC-GC Corporation, Japan)
- Subgroup 2: Resin composite adhesive, Transbond XT (light-cure adhesive, 3M Unitek, CA, USA).

The adhesive was applied to the mesh back of the metal bracket (universal bracket, upper bicuspid; Ormco Corporation, CA, USA) and was firmly pressed against the buccal enamel surface of each tooth. Any excess adhesive material was carefully removed from the periphery of the bracket base using an explorer. Then, the adhesive was light-cured with a light-curing unit (Elipar S10, 3 M ESPE Co., Seefeld, Germany) applied on each side of the bracket for 20 s.

After bonding, the specimens were stored individually in 2 mL of non-fluoride artificial saliva (20 mmol/L NaHCO_3 ; 3 mmol/L NaH_2PO_4 ; and 1 mmol/L CaCl_2 , neutral pH) inside a plastic container at 37°C for 24 h.^[2]

Shear bond strength testing

The specimens were mounted in acrylic cylinders of 3 mm internal diameter and 4 mm height, with the root embedded in an acrylic resin and the crown exposed. The teeth were mounted vertically, and the long axis of each tooth was vertically aligned to the base of the cylinder using a dental surveyor. The brackets were debonded by applying a load in the occluso-gingival direction to deliver a shear force parallel to the buccal enamel surface and at the bracket-tooth interface. The SBS was measured using a universal testing machine (EZ-test-1 KN Shimadzu, Kyoto, Japan). A shear force was applied to the specimens via the flattened end of a steel rod with a 30° beveled termination. The maximum load at failure required to debond each bracket was registered in Newton (N) at a crosshead speed of 1 mm per min and 1 KN cell load.^[2] The SBS (MPa) was calculated by dividing the load at failure (N) by the bracket base surface area (mm^2): $\text{MPa} = \text{N}/\text{mm}^2$. With the use of a digital caliper (Absolute Digimatic, Kawasaki, Japan), the

average surface area of the brackets' base was calculated as 7.06 mm².

ARI

The debonded enamel surfaces were examined under a stereomicroscope (Meiji Techno, Japan, Saitama, Japan) at 7X magnification to determine the amount of residual adhesive on the enamel surface. According to the original description of Artun and Bergland, the adhesive remnant index (ARI) scores were recorded as follows: 0 = no adhesive is remaining on the enamel surface, 1 = less than half of the adhesive is remaining on the enamel surface, 2 = more than half of the adhesive is remaining on the enamel surface, and 3 = the entire adhesive remains on the enamel surface.^[16]

Statistical analysis

The data were analyzed using the Statistical Package for the Social Sciences (SPSS) software, version 20.0 (SPSS, Inc., Chicago, IL, USA). The normality test of Shapiro-Wilk's and Levene's variance homogeneity test were applied to the data. The SBS data were found to be normally distributed, and there was homogeneity of variance among the groups. Thus, the statistical evaluation of SBS values among all groups was performed using parametric tests. Descriptive statistics, including mean values and standard deviation of the SBS, and minimum and maximum values, were calculated for all groups.

Comparisons of means of SBS values were made with a two-way analysis of variance (ANOVA). Post-hoc multiple comparisons of the SBS values among different groups were made with the Tukey Honestly Significance Difference test. The failure scores were analyzed with the Chi-square and Kruskal-Wallis tests to disclose the differences between the tested groups. The level of statistical significance was defined at ($P < 0.05$).

Results

Descriptive statistics, including the mean, standard deviation, and minimum and maximum values of the SBS for all experimental groups, are presented in Table 1. Figure 1 shows the differences in SBS values

among the groups. The results of the statistical analysis revealed statistically significant differences between the SBS values among the groups. Thus, the null hypothesis of this study was rejected. Group P showed the highest mean SBS values regardless of the type of adhesive used (group P: 25.20 ± 3.58 MPa, for Fuji Ortho LC), (group P: 20.99 ± 2.45 MPa, for Transbond XT), and the difference was statistically significant ($P < 0.05$). The fluoride-pretreated group, group F, showed the lowest mean SBS values among test groups (group F: 15.50 ± 3.53 MPa, for Fuji Ortho LC) (group F: 14.49 ± 2.67 MPa, for Transbond XT). The control group, group C showed comparable SBS values with group F for both types of adhesives used (group C: 15.50 ± 2.79 MPa, for Fuji Ortho LC) (group C: 14.40 ± 2.01 MPa, for Transbond XT). In addition, lower mean SBS values were recorded for group N (group N: 18.38 ± 2.68 MPa, for Fuji Ortho LC) (group N: 16.40 ± 2.11 MPa, for Transbond XT).

A two-way ANOVA revealed that both the etching protocol, as in group P and the type of adhesive used affected the SBS values, whereas the interaction between the test variables was insignificant [Table 2].

The distribution of ARI scores among the different experimental groups is presented in Table 3. The failure mode distribution among the test groups indicated that failures at the adhesive-bracket interface were predominant in group C compared with the other study groups.

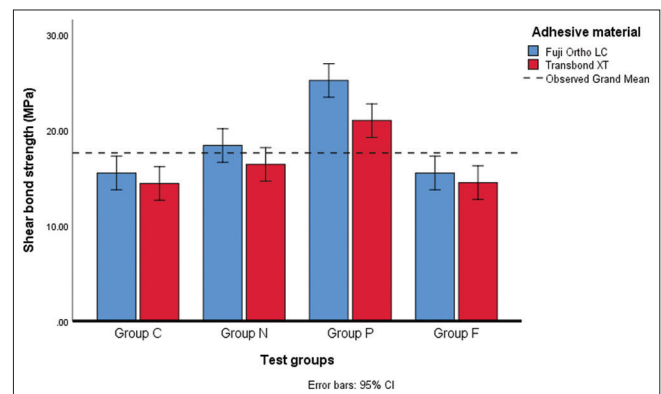


Figure 1: Differences in shear bond strength values among the groups

Table 1: Descriptive statistics of shear bond strength in MPa for all experimental groups

Group	n	Adhesive	Shear Bond Strength			
			Mean (MPa)	SD	Min	Max
Group C: Control group, intact enamel, etched with 35% phosphoric acid for 30 s	10	Fuji Ortho LC	15.500	2.79881	13.744	17.256
	10	Transbond XT	14.400	2.01108	12.644	16.156
Group N: Eroded enamel without any etching	10	Fuji Ortho LC	18.380	2.68734	16.624	20.136
	10	Transbond XT	16.400	2.11870	14.644	18.156
Group P: Eroded enamel treated with 35% phosphoric acid for 15 s, followed by a rinse for 10 s	10	Fuji Ortho LC	25.200	3.58391	23.444	26.956
	10	Transbond XT	20.990	2.45423	19.234	22.746
Group F: Eroded enamel treated with fluoride gel	10	Fuji Ortho LC	15.500	3.53553	13.744	17.256
	10	Transbond XT	14.490	2.67102	12.734	16.246

SD: Standard deviation

Table 2: Two-way analysis of variance test for all study groups

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	1,000.321 ^a	7	142.903	18.412	0.000
Intercept	24,801.924	1	24,801.924	3,195.587	0.000
Etching protocol	880.949	3	293.650	37.835	0.000
Type of adhesive	86.113	1	86.113	11.095	0.001
Etching protocol* Type of Adhesive	33.261	3	11.087	1.428	0.242
Error	558.814	72	7.761		
Total	26,361.060	80			
Corrected total	1,559.135	79			

^aR²=0.642 (adjusted R²=0.607)

Table 3: Distribution of the adhesive remnant index (ARI) scores for all groups tested (n=10)

Groups	Adhesive	ARI Scores %			
		Score 0	Score 1	Score 2	Score 3
Group C	Fuji Ortho LC	10	30	20	40
	Transbond	20	10	20	50
Group N	Fuji Ortho LC	10	40	40	10
	Transbond	20	30	30	20
Group P	Fuji Ortho LC	0	40	50	10
	Transbond	10	50	40	0
Group F	Fuji Ortho LC	10	50	10	30
	Transbond	20	30	20	40

Discussion

This study was performed to evaluate the effects of fluoride-releasing adhesives together with enamel surface pretreatment methods on the SBS of orthodontic brackets bonded to eroded enamel surfaces. To the best of our knowledge, no study has evaluated the impact of eroded enamel surface pretreatment combined with the bonding effect of fluoride-releasing orthodontic adhesives. Based on the results, the research hypotheses of this study were rejected.

The protocol of Saker *et al.*^[23] described the use of artificially eroded surfaces in the present study. As shown in the earlier studies, when the erosive challenge was produced, the SBS values of the orthodontic brackets decreased compared with the control group.^[12,13] Uysal *et al.*^[2] reported that the reduced SBS of the brackets was due to the atypical enamel surfaces and the lack of resin tag formation, which are responsible for the micromechanical interlocking achieved at the enamel. Reynolds^[19] affirmed that a minimum SBS of 5.9–7.8 MPa is adequate for the orthodontic brackets to withstand masticatory and orthodontic forces without detachment. The differences between the SBS values for sound and eroded enamel were significant in this study. The highest mean values of SBS were shown by experimental group P. These findings support those of Lenzi *et al.*,^[25] who reported an increase in the bond strength of eroded enamel when an etch-and-rinse adhesive system was used. Furthermore, all SBS values recorded were above the minimum requirement (5.9–7.8 MPa) suggested for a durable adhesive system.

The effects of fluoride on the prevention of tooth decay and remineralization of decalcified enamel have been elaborately described.^[1,5,9,11,22] In the present investigation, fluoride-treated groups showed the lowest SBS values, which could be attributed to the ability of fluoride to react with the enamel surface to form calcium fluoride and fluorapatite, making the surface more resistant to demineralization. This finding was supported by previous reports, which showed that topical application of fluoride can interfere with the etching effect of phosphoric acid on enamel surfaces, resulting in the reduced bond strength of dental resins.^[26] However, controversial results on the effect of topical fluoride application on bracket bond strength have been reported.^[27]

Other remineralizing agents, such as casein phosphopeptide-amorphous calcium phosphate (CPP-ACP) have been linked to a decrease in SBS of orthodontic appliances. CPP-ACP application reduced SBS of orthodontic brackets and resulted in enamel damage upon debonding.^[28] Furthermore, SBS of orthodontic appliances was reduced after the use of biomimetic nano-hydroxyapatite as a remineralizing agent.^[29]

The ARI score evaluation system has proven to be valuable in the studies of orthodontic adhesive systems. The failure pattern of debonded orthodontic brackets was inspected visually. The ARI was used to determine whether the failure was adhesive or cohesive. Adhesive failure occurs at the enamel–adhesive interface; it is scored as 0 or 1. Adhesive failure at the adhesive–bracket interface is scored as 3, whereas cohesive failure within the adhesive material itself is scored as 2 or 3. Bond failure is preferable at the adhesive–bracket interface or within the adhesive rather than at the enamel–adhesive interface to reduce the risk of enamel damage.^[29,30]

The results of ARI score comparisons in the present study indicated that all experimental groups, except group P, showed ARI scores ranging from 1 to 3, demonstrating that all or more than half of the adhesive was sustained on the enamel surface. The ARI score of group P ranged from 1 to 2. However, the enamel fractures were not registered.

The higher ARI scores could also be attributed to the fact that 35% phosphoric acid was used for enamel conditioning. When acid-etching techniques were used, fewer bonding failures occurred at the enamel–adhesive interface compared with the adhesive–bracket interface, which was true for group C.^[7,12]

In vitro bond strength testing of orthodontic adhesives will provide guidance to orthodontists in their clinical practice. It will help them understand the indications and clinical performance of the tested material.^[31] In the present investigation, testing protocols were regulated and followed properly to simulate the clinical setting. However, future clinical trials are needed because of limitations in the current research design (*in vitro* study, artificial erosive lesions). Evidence-based recommendations on the durability of orthodontic bonding materials, particularly in terms of prolonged erosive challenges, will be beneficial. Knowing the effects of other adhesive systems on erosion inhibition, such as self-etch adhesives, is also important. Together with erosion, other variables could alter the surface morphology of enamel and dental materials, such as toothbrushing^[32] and wear.^[33] Therefore, it would be interesting in the future to test remineralizing materials also in combination with these factors.

Professionals should inform their patients about the possibility of acidic substances causing irreversible damage to dental hard tissue in order to prevent dental wear. Patients with a high risk of dental erosion should limit their intake of potentially acidic drinks and foods, as well as their time in contact with their teeth. Fluoride home-care products should also be used as an additional preventive measure.^[6,9,25]

This study has some limitations, such as the SBS values were calculated *in vitro*, and more clinical research is needed to confirm the findings *in vivo*. Furthermore, since this current study only looked at one fluoride product, more research is needed in the future to compare the results to those of the other remineralizing agents.

Conclusions

The following conclusions can be drawn based on the results of this study:

1. Fluoride pretreatment, which was used to remineralize the eroded enamel surfaces before bonding, resulted in a decrease in the SBS of the orthodontic brackets compared with the other treated groups.
2. The use of fluoride-releasing adhesive enhances bonding to the eroded enamel surfaces.

Disclosure

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Conflicts of interest

There are no conflicts of interest.

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