Bond Strength of an Orthodontic Adhesive Containing an Antibiofilm Agent (Octafluoropentyl Methacrylate)

Abstract

Objective: The objective of this study was to determine whether an adhesive containing antibiofilm agent octafluoropentyl methacrylate (OFPA) has an acceptable bond strength as an orthodontic adhesive. **Methods:** Sixty human premolars were divided into four groups. In Groups 1 and 2, brackets were bonded to the teeth using experimental nanohybrid adhesive containing OFPA (ENH-OFPA) and Transbond XT (TXT) was used in Groups 3 and 4. In Groups 1 and 3, the samples were stored in water at 37°C for 24 h and in Groups 2 and 4 thermocycled between 5°C and 55°C. The entire samples were debonded utilizing a universal testing machine. Two-way ANOVA test employed to compare the shear bond strength (SBS) between two adhesives. Independent Student's *t*-test was utilized to compare the SBS at 24 h and followed thermocycling in each adhesive group. Adhesive remnant index (ARI) evaluated in different groups using Chi-square test. **Results:** The mean value for SBS in ENH and TXT groups were 8.13 MPa and 8.10 MPa, respectively. The inference of $P = 0.260$ was observed and concluded to be nonsignificant for adhesives in terms of SBS. Statistically, differences that observed in SBS at 24 h and following thermocycling in TXT ($P = 0.288$) and ENH-OFPA ($P = 0.145$) groups did not consider to be significant. Comparing ARI scores with bracket failure mode resulted in no significant difference ($P = 1$) between two adhesives in both 24 h and following thermocycling. **Conclusion:** Adding OFPA does not compromise SBS of the experimented orthodontic adhesive.

Keywords: *Composite resin, orthodontic bracket, thermocycling*

Elahe Vahid Dastjerdi, Golnaz Nahvi[1], Parisa Amdjadi[2,3], Farzad Aghdashi[4]

Department of Orthodontics, School of Dentistry, Shahid Beheshti University of Medical Sciences, [1]Student Research Committee, Department of Orthodontics,School of Dentistry, Shahid Beheshti University of Medical Sciences, [2]School of Dentistry, Shahid Beheshti University of Medical Sciences, [4]Department of Oral and Maxillofacial Surgery, Taleghani Hospital, Dental School, Shahid Beheshti University of Medical Sciences, Tehran, Iran, [3]Marquette University School of Dentistry, Milwaukee, WI, USA

Introduction

Decalcification of teeth containing fixed orthodontic appliances is a common iatrogenic effect of orthodontic treatment.[1] Accumulation of acidogenic microorganisms on salivary glycoproteins will increase the risk of developing secondary caries and white spot lesions (WSLs) along restorations.[2] Therefore, if a bonding material could impede bacterial accumulation, it might prevent demineralization near orthodontic appliances.

This caused much interest in different adhesives that could overcome such problems. Different materials including chlorhexidine,[3] fluorine-containing agents,[4] Proseal,[5] amorphous calcium phosphate,[6] iodide quaternary ammonium methacryloxy silicate,[7] nanoparticles included in the adhesive (i.e., nanofillers, silver, TiO_2, SiO_2, hydroxyapatite, fluorapatite, fluorohydroxyapatite) and bracket surfaces coated with a thin film of nitrogen-doped TiO_2,[8] triazine, and niobium pentoxide phosphate[9] have been added to orthodontic adhesives to decrease decalcification around bonded brackets. Incorporation of these materials suffers from some limitations such as compromising mechanical properties and limitation of active component release,[3,4] discoloration and esthetic problems,[10] and reduction of bond strength,[11] so neither of these materials are accepted as the best.

In a study by Dr. Ajmal *et al*. in 2017, the antibiofilm effects of octafluoropentyl methacrylate (OFPA) monomer as an anti-biofouling biocompatible material has been investigated. The authors have proposed that OFPA-coated materials express a protein repellent, the antifouling feature which could be effectively applied in materials which have direct contact with salivary glycoproteins such as orthodontic materials.[12] No previous study has investigated this material in the field of dentistry and orthodontics. Therefore,

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Address for correspondence:
Dr. Golnaz Nahvi,
Department of Orthodontics, School of Dentistry, Shahid Beheshti University of Medical Sciences, Tehran, Iran.
E-mail: golnaznahvi@gmail.com

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this study aimed to fill the gap by incorporating OFPA monomer into orthodontic adhesives and assessing its effect on the bracket bond strength. The null hypotheses to be tested were that the experimental nanohybrid composite containing OFPA has a considerable difference in shear bond strength (SBS) compared with conventional composites.

Methods

Preparation of nanocomposite

Brackets

Bicuspid stainless steel miniature mesh twin brackets (Generous Roth, GAC, NY, USA) with a 0.022-inch slot and 0 of tip and torque were used in this research. The mean bracket base surface area was about 12.4 mm^2. Brackets were placed on the tooth with a constant force by an operator.

Specimen selection and mounting

Sixty fresh extracted human premolar of Iranian population were included in the study. The samples were randomly assigned into four experimental groups. According to the criteria determined for this study, a specimen with sound buccal enamel, no pretreatment with chemical materials (e.g. H_2O_2), no signs of surface cracks, and free of caries were chosen.

The specimen was mounted using self-cured acrylic resin (Acropars 200, Marlik, Tehran, Iran) on a custom-made mounting jig (2 cm × 2 cm × 3 cm). The mounting jig was utilized for alignment of buccal surfaces of the teeth; therefore, they were parallel with the mold bottom. This kept the buccal tooth surface parallel to the applied force during the shear test.

Every specimen has an orientation with respect to the testing device as a guide. Thus, its labial aspect was perpendicular to the pressure, while conducting the SBS test. After mounting and right before brackets bonding, the teeth were cleaned and polished using coarse, oil-free pumice, and rubber prophylactic cups for 10 s. Then, the specimen was rinsed with water and evaporated for 15 s.

Groups tested

There were four experimental groups and each group contained 15 specimens.

- Group 1 – Etchant (Transbond XT [TXT] etching gel, containing 35% phosphoric acid from 3M/Unitek) was rubbed to the cleaned area of the tooth for 15 s, rinsed for 10 s, and evaporated using an air–water syringe for 20 s. For Group 1, each bracket was bonded using a direct bond technique. A tiny layer of TXT light-cured primer (3M Unitek) was rubbed on the surface of the specimen and cured for 10 s using a light curing unit (Astralis 7, Ivoclar, Vivadent, Schaan, Lichtenstein). TXT bonding agent was used to the base of the brackets, and the bracket was placed onto the facial tooth surface in the crown center over the long axis of the tooth using a metal index. Then, a 300-g-force was utilized (Correx force gauge, Bern, Switzerland) for 10 s.

The force gauge is applied for assuring a constant adhesive thickness between the bracket and the teeth surface. The hand instrument used to remove excessive adhesive, and the bracket was cured for 10 s from the distal and 10 s from the mesial. Then, the teeth contained in the deionized water for 1 day at 37°C. The teeth remained in distilled water at all times except when brackets were being bonded and artificial saliva debonded. The specimen was maintained in fresh distilled water at 37°C for 24 before debonding. Thymol crystals 0.2% (wt/vol) were added to inhibit bacterial growth. A storage period of 24 h was chosen to remain consistent with previous studies using TXT as a control.[13,14]

- Group 2 – In this group, the same bonding procedure similar to the first group was performed with the exception that the teeth were thermocycled after being stored in fresh distilled water at 37°C and thymol crystals 0.2% (wt/vol) for 24 h and before debonding
- Group 3 – The same bonding process similar to Group 1 employed for Group 3 with the exception that experimental nanohybrid adhesive containing OFPA was used for bracket bonding
- Group 4 – The same bonding process similar to Group 3 used for this group with the exception that the teeth were subjected to thermocycled after being stored in fresh distilled water at 37°C and thymol crystals 0.2% (wt/vol) for 24 h and before debonding.

Thermocycling

Following the recommendations of the International Organization for Standardization, the teeth preparation carried out at 23°C ± 2°C and stored in water at 37°C ± 2°C before testing at the temperature of the room.

The teeth were kept in water for 1 day to distinguish between materials that could or could not bear a wet environment.[15] After 24 h, the mounted teeth were thermocycled between 5°C and 55°C for 500 cycles. The exposure time to every water bath was 20 s. The transition period between the two baths was 5–10 s. Debonding was performed at room temperature.[15]

Shear Bond Strength Test

A universal testing machine (Zwick/Roell Z020, Zwick GmbH, Ulm, Germany) was utilized to determine the bond strength with 1-kN load cell with a crosshead speed of 1 mm/min. The brackets were loaded by an occlusal–gingival force applied by a blade-shaped steel rod inserted into the crosshead of the universal testing machine. This exerted a shear force at the interface of the bracket–enamel. The contact was made as close to the bracket–enamel interface as possible. The force at bracket failure was reported in Newtons by an electric connection to the Zwick machine. The SBS values calculations were performed in megapascal by force divided by the area of the bracket base (MPa = N/mm^2).

The highest required load for debonding or start bracket fracture was reported in Newton and then converted into megapascal as a ratio of Newtons to the bracket surface area.

Adhesive remnant index classification

Following the debonding, the specimen examination was performed by a stereomicroscope with × 16 magnification. The adhesive remnant index (ARI) score assessment was performed with regard to the remaining resin bonding on the teeth surface.[16]

0 – No bonding agent remaining on the enamel surface;

1 – Less than 50% of the bonding agent remaining on the enamel surface;

2 – More than 50% of the bonding agent remaining on the enamel surface;

3 – The whole bonding agent remaining on the tooth surface, with a distinguished sign of the bracket base.

Statistical analysis

SPSS software version 21.0 (SPSS Inc., Chicago, IL, USA) was utilized for statistical analysis. Data distribution was assessed using visual (histograms and probability plots) and statistical methods (the Kolmogorov–Smirnov test and Shapiro–Wilk test). Two-way ANOVA test was used for comparing SBS between two adhesive groups and independent *t*-tests for comparing 24-h and 24 h + thermocycling bond strengths of each bonding agent. Data were normally distributed (P = 0/085) [Figure 1]. ARI was compared between experimental groups using Chi-square test. SBSs correlation to the adhesive remnant scores was performed using Pearson's product–moment correlation coefficient. A level of 0.05 was chosen. Each specimen was scored for bonding agent remnant following debonding by two observers. There was no intraobserver or interobserver variations in adhesive remnant scores.

Results

Shear bond strength

SBS descriptive values in two experimental groups are demonstrated in Table 1.

Comparison of SBS in Group experimental nanohybrid adhesive containing OFPA (ENH-OFPA) at 24 h and 24 h + thermocycling showed no statistical differences (P = 0.145).

Comparison of SBS in Group TXT at 24 h and 24 h + thermocycling showed no statistical differences (P = 0.288).

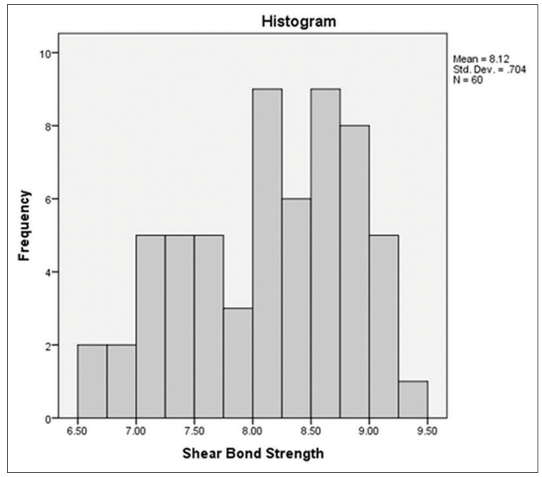

Figure 1: Histogram demonstrating normal data distribution

Table 1: Statistical descriptives of shear bond strengths of adhesives in megapascals

Dependent variable: SBS			
Group	**Time**	**Mean (MPa)±SD**	***n***
NH	Without thermocycling (24 h)	8.1313±0.64268	15
	With thermocycling	8.1447±0.71412	15
	Total	8.1380±0.66756	30
TXT	Without thermocycling (24 h)	8.3073±0.72590	15
	With thermocycling	7.9047±0.74371	15
	Total	8.1060±0.75055	30
Total	Without thermocycling (24 h)	8.2193±0.67955	30
	With thermocycling	8.0247±0.72671	30
	Total	8.1220±0.70441	60

SBS: Shear bond strength; SD: Standard deviation; TXT: Transbond XT; NH: Nanohybrid

Comparison of SBS between two adhesives showed no statistical difference (P = 0.260) [Figure 2].

Adhesive remnant index

Each specimen was given a score for bonding agent remnant after debonding by two observers. No intraobserver or interobserver errors were detected in adhesive remnant scores. None of the specimens tested showed detrimental effects on tooth surfaces following debonding in the testing machine.

The amount of residual adhesive on the enamel surface as evaluated by the ARIs scores is shown in Table 2. Comparison of ARI scores between two experimental group using Chi-Square showed no statistical difference (P = 1) [Figure 3].

There were negative correlations between bond strengths and adhesive remnant scores for the adhesives (r = −0.130).

Correlations for debondings demonstrated association between bond strength and adhesive remnant score: ENH-OFPA (r = 0.573), TXT (r = 0.690).

Table 2: The adhesive remnant index scores on enamel surfaces in different experimental groups

Time × ARI cross-tabulation			
Groups	**ARI**		**Total**
	1	**2**	
Time			
Without thermocycling (24 h)	22	8	30
With thermocycling	20	10	30
Total	42	18	60

ARI: Adhesive remnant index

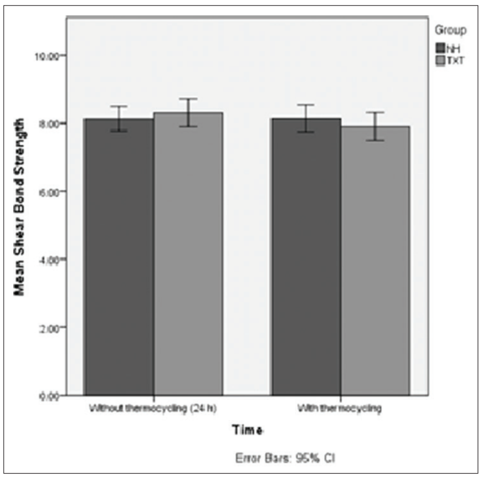

Figure 2: Error bar of 95% confidence intervals of the differences for the mean shear bond strength

Method error

Ten randomly selected samples were reexamined by the same examiner after 1 week, and the kappa test was applied to test intraexaminer reliability. Kappa values were more than 92% for the ARI.

As a measure of interexaminer reliability, the Cohen's Kappa coefficient was calculated. Cohen's Kappa coefficient was found to be 0.85, which is considered indicative of satisfactory interexaminer reliability.

In the few cases where disagreement in measurements observed, the two examiners reached a joint definition after reevaluation of the samples and sufficient discussion.

Discussion

Direct bracket bonding to the etched enamel surface has several disadvantages including enamel demineralization and WSL formation adjacent to the bracket.[17]

A considerable amount of research has been conducted to decrease bacterial-induced WSL during orthodontic treatment.[5-8,18] The use of nanoparticles in resin composites has been taken into consideration by researchers for bonding of orthodontic brackets.[13,19] Resin-based materials may express reduced mechanical properties including bonding strength when different antibacterial agents are added.[13]

The results of the current study showed that the OFPA-containing composite has a suitable antibacterial effect without compromising SBS.

An antifouling feature of the OFPA was investigated in a previous study by authors,[12] but literature lacks information regarding mechanical properties of an orthodontic adhesive containing OFPA. The novelty of the current study is that according to the literature, this is the first time that OFPA is included in dentistry materials, especially orthodontic adhesives. Hence, the current study was designed to investigate the SBS of an adhesive containing OFPA. Shear bond tests are known as *in vitro* testing procedure for measurement of adhesive force. To allow better comparison of the results recorded, they are converted by many authors from N/mm^2 into MPa.[20]

The mean debonding SBS recorded in ENH-OFPA group was 8.54 Mpa after 24 h and 8.51 following thermocycling. It is highly recommended that orthodontic adhesives should possess an appropriate bonding strength, ranging between 5.9 and 7.8 MPa, to allow adequate adhesion and ease of debonding.[21] ENH-OFPA possessed mean SBSs above the amount recommended by previous studies. This observation shows that adding OFPA does not compromise the SBS of the experimental adhesive. The bonding strength of ENH-OFPA was comparable with TXT as a control group. The TXT primer is regarded as one of the standard adhesive systems in orthodontics; therefore, it has been the subject of many studies examining its adhesive strength.[22-24]

In a previous systematic review on orthodontic bond strength, there are plentiful testing parameters that can impact *in vitro* adhesiveness values such as storage medium of teeth, cleansing of enamel, type of bracket, etchant type, etching time, adhesive type, photopolymerization device, specimen storage period, crosshead speed, force exertion location on bracket, and blade design of the jig of the universal testing machine, the shearing speed of the test machine, duration of light curing, and the type of used material.[25] With the exception of the adhesive type, all of the aforementioned parameters were compiled by the authors of the current study.

In contrast to the requirements for composite filling materials in conservative dentistry, where the fillings are intended to maintain for a long time, an adhesive used in orthodontics needs to be easily removable at the end of the treatment with no harm to teeth.

In orthodontics, less adhesive remained on the tooth after debonding requires less work and time spent by the orthodontist in removing it. A lower ARI score is favorable in this situation.[26] All four groups presented with a majority of specimens in the 0–1 range, thus a majority

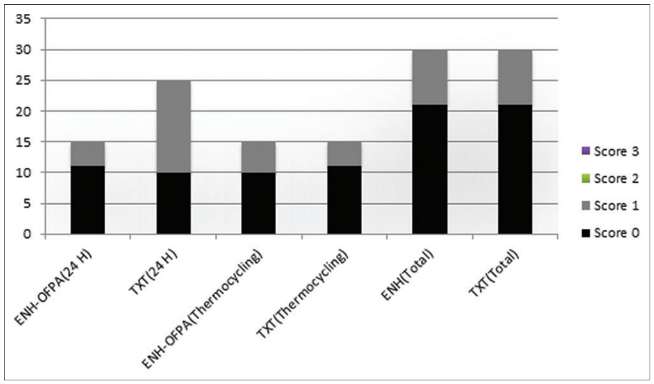

Figure 3: Bar chart comparing adhesive remnant index in four experimental groups in two adhesives (Experimental Nanohybrid adhesive containing octafluoropentyl methacrylate and Transbond XT)

of the adhesive was removed with the bracket during the process of debonding. The results of the ARI show a homogeneous distribution for both ENH-OFPM and TXT groups at 24 h and after thermocycling.

Brackets bonded in the clinic are exposed to a variety of forces at various temperatures.[27] Therefore, the research team decided to evaluate the SBS after reaching its maximum bond strength (24 h) and in different temperature variations (thermocycling).

In general, considering all limitation of *in vitro* studies, one of the shortcomings of this study is the fact that the results of *in vitro* experiments are never precisely comparable with those of clinical situations. The technique-sensitive materials and the complexity of the interactions involved are subject to error. The standardization can never reach 100% success since the bond strength in an *in vivo* situation is a feedback of all features of an adhesive. Examples are microleakage, debonding features, flow characteristics, amount of penetration, curing depth, and liquid absorption.[28] However, the results of *in vitro* experiments can provide valuable information for *in vivo* circumstances, specifically for clinical practice and everyday clinical practice.

Conclusion

With the limitations of the current study, we concluded that adding OFPA to orthodontic adhesive does not compromise the SBS. Data on the long-term performance of orthodontic adhesives using OFPA are lacking and necessitates further investigation and so do possible safety issues (toxicity).

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Nil.

Conflicts of interest

There are no conflicts of interest.

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