



Shear Bond Strength of Self-Adhesive Composite Containing TiO₂ and SiO₂ Nanoparticles with an Additional Etching Step for Orthodontic Brackets Bonding to Enamel

Soheil Nikpour¹, Atefe Saffar Shahroudi², Aida Saffarpour³, Azam Akhavan⁴, Ahmad Sodagar⁵

¹ Faculty of Dentistry, International Campus of Tehran University of Medical Sciences, Tehran, Iran

² Dental Research Center, Dentistry Research Institute, and Department of Orthodontics, Dental School, Tehran University of Medical Sciences, Tehran, Iran

³ Radiation Application Research School, Tehran, Iran

⁴ Department of Operative Dentistry, International Campus of Dental School, Tehran University of Medical Sciences, Tehran, Iran

⁵ Department of Orthodontics, Faculty of Dentistry, Tehran University of Medical Sciences and member of Dental Research Center, Dentistry Research Institute, Tehran University of Medical Sciences, Tehran, Iran

Corresponding author: Ahmad Sodagar, Department of Orthodontics, Faculty of Dentistry, Tehran University of Medical Sciences, North Kargar Street, Tehran, Iran; E-mail: sodagara@tums.ac.ir; Tel.: (+98)9124070738

Received: 18 July 2020 ♦ **Accepted:** 19 Feb 2021 ♦ **Published:** 31 Dec 2021

Citation: Nikpour S, Shahroudi AS, Saffarpour A, Akhavan A, Sodagar A. Shear bond strength of self-adhesive composite containing TiO₂ and SiO₂ nanoparticles with an additional etching step for orthodontic brackets bonding to enamel. *Folia Med (Plovdiv)* 2021;63(6):865-74. doi: 10.3897/folmed.63.e56657.

Abstract

Introduction: Recently, nanoparticles such as nano-TiO₂ have been added to some dental materials for enhancing dental carries prevention due to their antibacterial activity.

Aim: This study aimed to assess the shear bond strength of a self-adhesive composite containing TiO₂ and SiO₂ nanoparticles for orthodontic bracket bonding.

Materials and methods: This in vitro, experimental study was done on 70 extracted human premolars divided into 7 groups. Six groups of Vertise Flow self-adhesive composite samples were prepared: without any nanoparticles, with 0.5% and 1% TiO₂ nanoparticles, 0.5% and 1% SiO₂ nanoparticles, and 1% mixture of TiO₂ and SiO₂ nanoparticles so that nano-hybrid composites were prepared. Metal brackets were bonded with these samples as well as Transbond XT as the control group. The shear bond strength of the brackets to enamel was measured using a universal testing machine. The adhesive remnant index (ARI) score was also determined by a stereomicroscope. Data were analyzed by one-way ANOVA, Tukey's test and Kruskal-Wallis test.

Results: The shear bond strength of the groups was significantly different ($p=0.000$). Pairwise comparisons revealed that the bond strength of Transbond XT group was significantly higher than others ($p<0.05$), followed by the 1% TiO₂ group with significant differences with Vertise Flow and 0.5% TiO₂ groups. The lowest value belonged to Vertise Flow with no nanoparticles. The ARI scores was different in the control group ($p=0.000$).

Conclusions: Adding TiO₂ and SiO₂ nanoparticles to Vertise Flow self-adhesive composite not only did not adversely affect its shear bond strength, but also slightly increased it. Overall, the self-adhesive nano-hybrid composite containing TiO₂ and/or SiO₂ nanoparticles, following an additional etching step would be acceptable for bracket bonding and can be used clinically to benefit from the antimicrobial activity of these nanoparticles.

Keywords

nanoparticles, shear bond strength, orthodontic brackets, self-adhesive composite, TiO₂-SiO₂

INTRODUCTION

Enamel demineralization around and under orthodontic brackets is an important, yet common, complication during the course of orthodontic treatment with fixed appliances.¹ Composite resins used for direct adhesive bonding have a polymer matrix and can provide a suitable environment for the growth and proliferation of aerobic and anaerobic microorganisms. Accumulation of these microorganisms in the marginal areas of composite resins and brackets can lead to early debonding, enamel demineralization, and periodontal disease.^{2,3} Moreover, oral hygiene maintenance is more difficult in such patients. Therefore, researchers and manufacturers have tried to benefit from the application of antibacterial agents with cariostatic properties to prevent caries around orthodontic brackets.

Application of nanotechnology in production of composite resins is among the important advances made in dental materials.⁴ Use of nano-scale materials (1 to 100 nm) enables benefitting from their unique physical, chemical, mechanical, and optical properties. Nanotechnology aims to control and manipulate the materials to prepare them for certain applications.⁵ This new era of science has been used in many fields in the recent years and was also employed in dentistry to produce materials with high mechanical properties and antimicrobial activity.⁶⁻⁸

Titanium dioxide (TiO₂) nanoparticles have optimal antibacterial activity and have been incorporated in the composition of several biomaterials. They have antimicrobial activity against *Escherichia coli*, *Giardia lamblia*, *Staphylococcus aureus*, *Pseudomonas aeruginosa*, *Candida albicans*, and *Lactobacillus acidophilus*; they are also capable of eliminating tumoral cells.^{9,10} White colour, high stability, affordable cost, and low toxicity are among the other favourable characteristics of TiO₂.⁸ Composite resins containing TiO₂ have been produced as well and their antibacterial properties have been previously confirmed.^{6,11} Wires coated with a layer of TiO₂ have also shown antibacterial properties against *Streptococcus mutans*.¹² These nanoparticles also incorporated antifungal effect in acrylic resin.¹³

Silicon dioxide (SiO₂) nanoparticles have a porous structure and high surface absorption, and are therefore highly suitable for incorporation in biomaterials to serve as a carrier for other agents. These carriers let the antimicrobial agents release slowly, which in turn enhances the safety and durability of the agent.^{8,14} Accordingly, the combination of TiO₂-SiO₂ can be more promising than TiO₂ alone.¹⁵ It was shown that polymethyl methacrylate polymers containing SiO₂ and TiO₂ nanoparticles have strong antibacterial activity against the cariogenic bacteria.¹⁵ Although the antibacterial property of nanoparticles is a great advantage, incorporation of these agents should not interfere with the mechanical properties of the respective material.

Early debonding of brackets is a problem commonly encountered in orthodontic treatment. Thus, there is always an attempt to produce materials with the fastest, easiest and most suitable bonding quality. Several studies have assessed

the efficacy, durability and strength of new bonding agents by the shear bond strength and tensile bond strength tests. Shear bond strength testing is more commonly used since it simulates the oral clinical setting in terms of loading conditions and load application in a better way.^{16,17}

The conventional orthodontic bracket bonding systems included several procedural steps including etching, rinsing, drying, and resin primer application before bracket bonding with resin cement.¹⁶ In an attempt to simplify the bonding procedure and decrease the chair-time, self-adhesive composites were recently introduced to the dental market, and these decrease the procedural steps and facilitate the process of bracket bonding.¹⁷

Recent studies did not report adequate shear bond strength for orthodontic brackets bonded with self-adhesive composites.¹⁷⁻¹⁹ However, phosphoric acid etching of tooth surface prior to composite application significantly increased the bond strength of composite to tooth structure to the clinically acceptable level and it was reported that etching of the enamel surface prior to the use of self-etch systems would significantly increase the shear bond strength.²⁰⁻²²

Although the incorporation of TiO₂ nanoparticles into conventional composite resins as orthodontic bracket adhesives did not show any adverse effect on their shear bond strength^{6,23}, no data is available on the effect of incorporation of TiO₂ and SiO₂ nanoparticles in the composition of self-adhesive composites.

AIM

This study aimed to assess the shear bond strength of self-adhesive composite containing TiO₂ and SiO₂ nanoparticles following an additional etching step for bonding of orthodontic metallic brackets to enamel.

MATERIALS AND METHODS

In this in vitro experimental study, sample size was calculated to be 10 in each of the 7 groups according to a previous study by Sodagar et al.¹⁵ using one-way ANOVA power analysis feature of PASSII software, assuming alpha = 0.05, beta = 0.2, standard deviation of the mean = 7.2 MPa and effect size of 0.47.

Preparation of nano-hybrid composites containing nano-TiO₂ and SiO₂

The nano-TiO₂ powder with 20 nm particle size and nano-SiO₂ powder with 20 nm particle size (Degussa, Germany) were used to prepare nano-hybrid composites containing TiO₂-SiO₂. In order to prepare nanoparticle-containing composite, in the first stage, the amount of composite required for each single tooth was estimated based on clinical experience. Next, by some overestimation

to compensate possible errors, the amount of composite required for each group (n=10) was estimated to be approximately 500 mg. In order to prepare experimental nano-hybrid composites which contained 1% TiO₂ nanoparticles, 5 mg of nano-TiO₂ powder was manually mixed with 495 mg of self-adhering flowable resin composites (Vertise Flow, Kerr, Orange, CA, USA) on a glass slab in a semi-dark room away from direct light until obtaining a homogeneous mixture.

To prepare 500 mg composite containing 0.5% nano-TiO₂ nanoparticles, 250 mg from the aforementioned prepared nano-hybrid composites containing 1% nano-TiO₂ was mixed with the intact Vertise Flow composite.

The same procedure was performed to prepare a nano-hybrid composite containing SiO₂ nanoparticles in two concentration of 0.5% and 1%. Subsequently, in order to produce a composite containing both TiO₂ and SiO₂ nanoparticles, 2.5 mg of nano-TiO₂ and 2.5 mg of nano-SiO₂ powder were mixed with 495 mg of the intact Vertise Flow composite to obtain 1% concentration of both nanoparticles. Finally, experimental nano-hybrid composites with 0.5% and 1% nano-TiO₂, 0.5% and 1% nano-SiO₂ and 1% mixture of nano-TiO₂-SiO₂ were prepared.

The applied percentage of nanoparticles was determined according to previous studies.^{7,23,24} Homogeneous distribution of the nanoparticles in the composite resin was ensured by inspection under a scanning electron microscope (Fig. 1).

Preparation of teeth

Seventy sound maxillary premolars without cracks that had been extracted for orthodontic purposes were selected for

use in this study. The teeth were stored in saline until the experiment. The calculus and soft tissue debris were removed and the teeth were immersed in 0.5% chloramine T solution at 4°C for 1 week for disinfection. Then, their buccal surface was cleaned with a prophylactic brush and was etched with 37% phosphoric acid for 30 s and rinsed with water spray for 60 s followed by of air-spray drying for 10 s. In the next step, the Vertise Flow composite was applied on the surface. Standard premolar brackets (Mini Master series, American Orthodontics, Sheboygan, NY, USA) were then placed on the tooth surface. Light curing of composite was performed using Demi (Kerr, USA) light-curing unit (Fig. 2A). The teeth were randomly divided into 7 groups (n=10) as follows:

Group 1: This group served as the control group. Transbond XT light-cure composite (3M Unitek, Monrovia, CA) was used with Transbond XT primer (3M Unitek, Monrovia, CA). The primer was first applied on the tooth surface and gently air-sprayed for 5 seconds. Next, the composite was applied at the middle of the buccal surface of the tooth beneath the bracket. Excess composite was removed by a scaler and the composite was light-cured from mesial, distal, occlusal and gingival directions for a total of 40 seconds.

Group 2: In this group, Vertise Flow self-adhesive composite without nanoparticles was applied between the buccal surface of the teeth and the brackets. Excess composite was removed by a scaler and the composite was light-cured as explained for group 1. The other five groups were also bonded with similar procedure with nano-hybrid composites and the seven study groups are shown in Table 1.

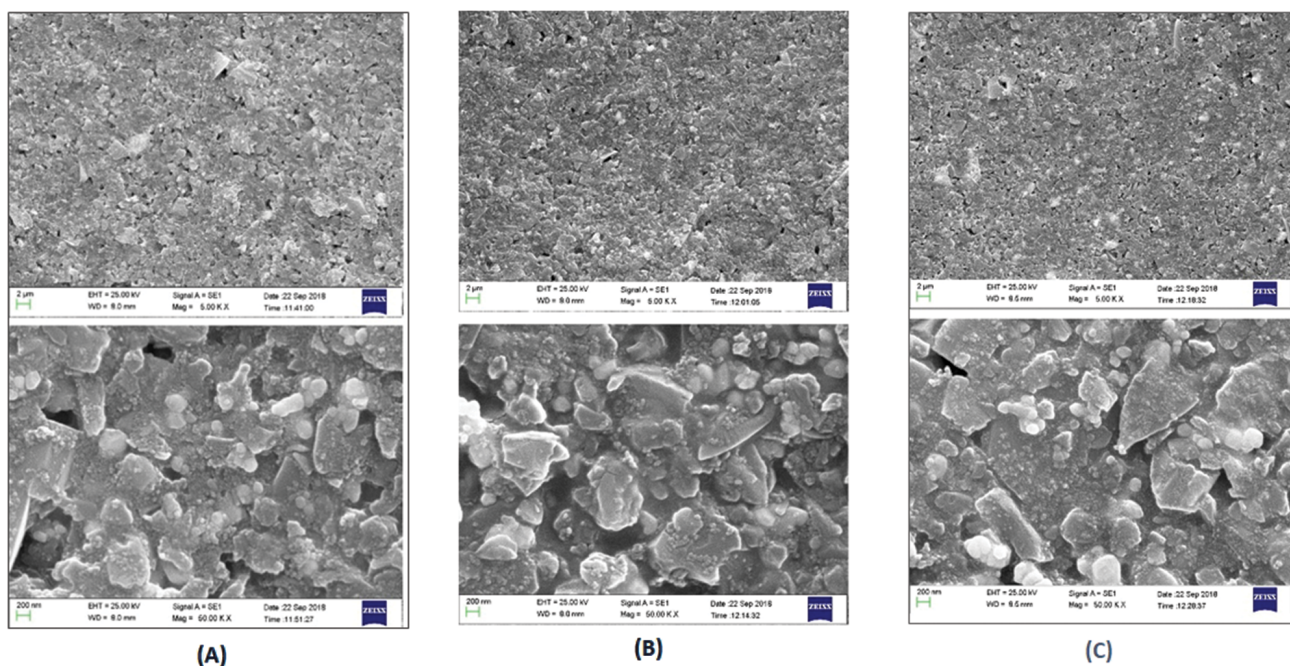


Figure 1. Scanning electron microscope micrograph of the distribution of nanoparticles in the Vertise Flow nano-hybrid composite with two different magnification: A) nano-TiO₂; B) nano-SiO₂; C) nano-TiO₂-SiO₂.

Table 1. Definition of study groups

Group	Applied Adhesive
1	Transbond XT (Control Group)
2	Vertise Flow
3	Vertise Flow containing 0.5% SiO ₂ nanoparticles
4	Vertise Flow containing 1% SiO ₂ nanoparticles
5	Vertise Flow containing 0.5% TiO ₂ nanoparticles
6	Vertise Flow containing 1% TiO ₂ nanoparticles
7	Vertise Flow containing 1% SiO ₂ and TiO ₂ nanoparticles

The teeth were mounted in auto-polymerizing acrylic resin in metal molds measuring 2.5 cm in diameter. Petroleum jelly was applied to the internal surface of the molds and the teeth were fixed to the wire using ligature wire. Each tooth was positioned at the center of each mold and then 0.016×0.22-inch stainless steel rectangular wires were fixed to the mold using sticky wax (**Fig. 2B**). This was done to prevent their movement during the application of acrylic resin. Auto-polymerizing acrylic resin was prepared in the form of a paste and applied into the molds such that the teeth were embedded in the acrylic to the level of their cemento-enamel junction. After polymerization of acrylic resin, the teeth were separated from the rectangular wire, and the acrylic blocks containing the teeth were removed from the metal molds (**Fig. 2C**).

The samples were subjected to thermocycling (Vafaei Industrial, Iran) for 1500 cycles between 5-55°C for 24 hours

with a dwell time of 15 seconds and a transfer time of 10 seconds. This was done for a better simulation of the oral physical conditions.

Measuring the bond strength

The shear bond strength of the teeth was measured using a universal testing machine (Roel 7060; Zwick Roell, Germany). The samples were placed in the machine in the way that the bracket's base was parallel to the direction of the load application. Load was applied by a metal blade with 0.6 mm thickness to the bracket base-enamel interface. It had a smooth surface pointing to the bracket and a steep surface pointing to the tooth. Load was applied incisogingivally with a speed of 0.5 mm/min until bracket debonding (**Fig. 2D**). The load at debonding was recorded in newtons (N) and divided by the bracket base surface area in square millimetres ($9.15 \pm 0.02 \text{ mm}^2$) to calculate the shear bond strength in megapascals (MPa).

Adhesive remnant index (ARI) score

After the debonding process, each debonded bracket and tooth surface was inspected under a stereomicroscope (SMZ800, Nikon, Japan) at ×10 magnification. Digital photographs were taken of both bracket bases and teeth surfaces at this magnification and the percentage of adhesive remnant surface area was calculated by computer. Two researchers determined the ARI score distribution according to the remained adhesive on the enamel for each specimen according to a modified score which was explained

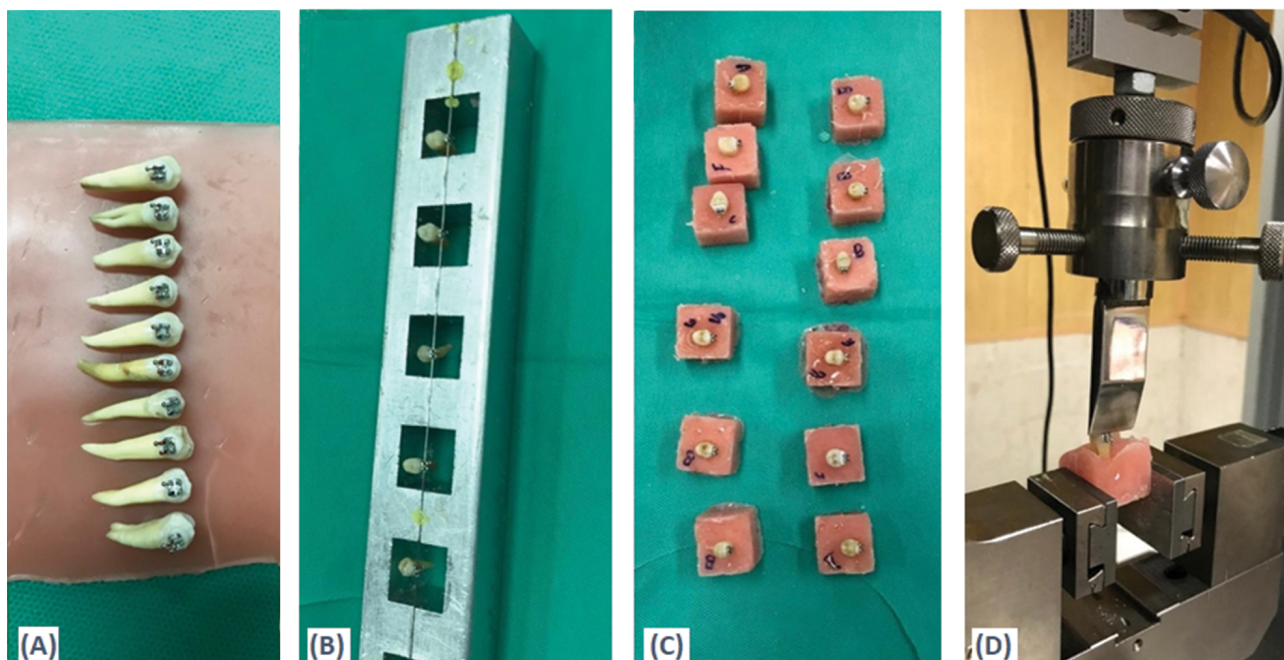


Figure 2. A) Orthodontic braces were bonded on human premolars with experimental composites. B) The bonded teeth were fixed by a stainless steel wire on a fabricated metallic mold. C) Acrylic blocks containing bonded teeth were prepared. D) Blocks were inserted in a universal testing machine.

in Ritter et al.²⁵ study. The Kappa coefficient of agreement between researchers in an ARI score assessment was 0.863 ($p < 0.0001$). The ARI score was determined in a 1-5-score scale as follows:

1: All the bonded tooth area (100%) is covered with the adhesive (**Fig. 3A**).

2: More than 90% of the bonded tooth area is covered with the adhesive.

3: Between 10% and 90% of the bonded tooth area is covered with the adhesive.

4: Less than 10% of the bonded tooth area is covered with the adhesive.

5: No adhesive remained on the tooth surface (**Fig. 3B**).

Statistical analysis

Data were analyzed using SPSS version 25 (SPSS Inc., IL, USA). The maximum, minimum, mean, and standard deviation of shear bond strength were reported for all 7 groups. The groups were compared in this regard using one-way ANOVA. Since the result was significant, pairwise compar-

isons were carried out using the Tukey's post-hoc HSD test (considering the homogeneity of variances). The ARI score was analyzed using the Kruskal-Wallis test.

RESULTS

Table 2 presents the mean shear bond strength of the bracket to the tooth structure in the 7 groups; as shown, the maximum shear bond strength was noted in Transbond XT control group while the minimum shear bond strength was recorded in Vertise Flow group. The Levene's test showed no significant difference in data distribution of different groups ($p > 0.05$). One-way ANOVA found a significant difference in the shear bond strength of the 7 groups ($p = 0.000$). Thus, pairwise comparisons were performed using the Tukey's test (**Table 3**). As shown in **Table 3**, the shear bond strength of bracket to tooth structure in Transbond XT control group was significantly higher than that in other groups followed by 1% TiO₂ group, which had significant differences with Vertise Flow and 0.5% TiO₂ groups.

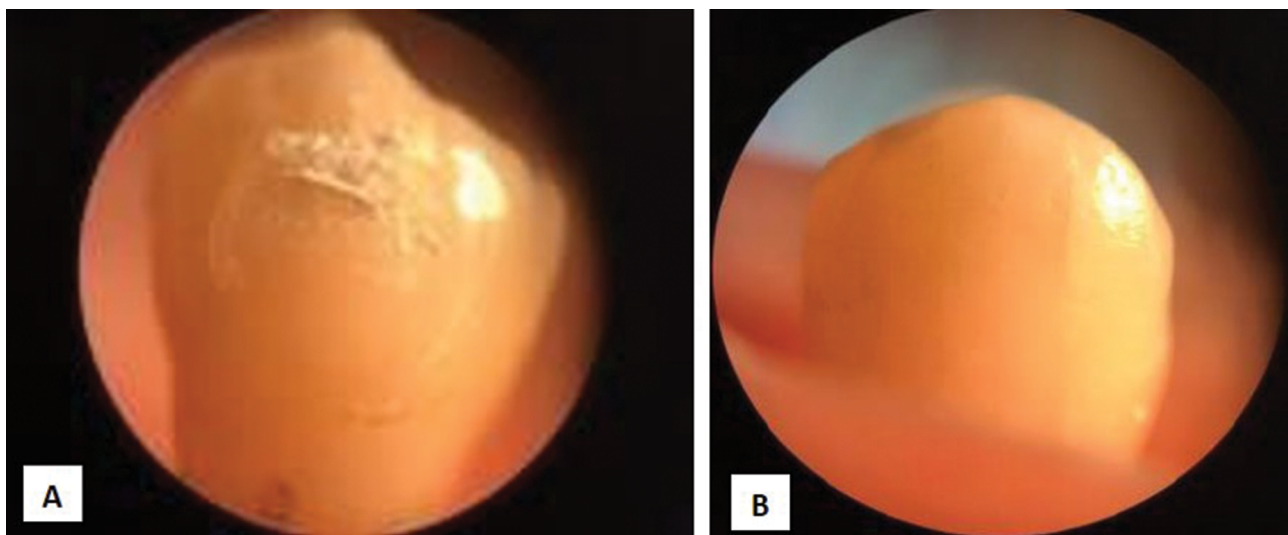


Figure 3. Adhesive remnant index: **A)** Maximum adhesive remaining on the tooth surface; **B)** no adhesive remaining on the tooth surface.

Table 2. Mean shear bond strength of bracket to tooth structure in the 7 groups in Mpa (n=10)

Group	Minimum	Maximum	Mean	Std. Deviation
1. Transbond XT	8.38	14.29	11.3230	1.69952
2. Vertise Flow	4.53	7.15	6.1710	0.81499
3. VF with SiO ₂ -0.5%	5.51	7.55	6.7300	0.60307
4. VF with SiO ₂ -1%	6.20	9.20	7.6150	1.03841
5. VF with TiO ₂ -0.5%	4.80	7.62	6.3540	0.93370
6. VF with TiO ₂ -1%	7.02	9.17	7.8410	0.78741
7. VF with SiO ₂ -TiO ₂ -1%	5.50	7.18	6.5250	0.57859

VF: Vertise Flow

Table 3. Pairwise comparisons of the groups regarding shear bond strength using the Tukey's test

Group (I)/group (J)	Mean difference (I-J)	Std. Error	Sig.*	95% Confidence interval		
				Lower Bound	Upper Bound	
Transbond	Vertise Flow	5.15200*	0.44146	0.000	3.8075	6.4965
	SiO ₂ -0.5%	4.59300*	0.44146	0.000	3.2485	5.9375
	SiO ₂ -1%	3.70800*	0.44146	0.000	2.3635	5.0525
	TiO ₂ -0.5%	4.96900*	0.44146	0.000	3.6245	6.3135
	TiO ₂ -1%	3.48200*	0.44146	0.000	2.1375	4.8265
	SiO ₂ ,TiO ₂ -1%	4.79800*	0.44146	0.000	3.4535	6.1425
Vertise Flow	SiO ₂ -0.5%	-0.55900	0.44146	0.865	-1.9035	0.7855
	SiO ₂ -1%	-1.44400*	0.44146	0.027	-2.7885	-0.0995
	TiO ₂ -0.5%	-0.18300	0.44146	1.000	-1.5275	1.1615
	TiO ₂ -1%	-1.67000*	0.44146	0.006	-3.0145	-0.3255
	SiO ₂ ,TiO ₂ -1%	-0.35400	0.44146	0.984	-1.6985	0.9905
VF with SiO ₂ -0.5%	SiO ₂ -1%	-0.88500	0.44146	0.422	-2.2295	0.4595
	TiO ₂ -0.5%	0.37600	0.44146	0.978	-0.9685	1.7205
	TiO ₂ -1%	-1.11100	0.44146	0.171	-2.4555	0.2335
	SiO ₂ ,TiO ₂ -1%	0.20500	0.44146	0.999	-1.1395	1.5495
VF with SiO ₂ -1%	TiO ₂ -0.5%	1.26100	0.44146	0.080	-0.0835	2.6055
	TiO ₂ -1%	-0.22600	0.44146	0.999	-1.5705	1.1185
	SiO ₂ ,TiO ₂ -1%	1.09000	0.44146	0.188	-0.2545	2.4345
VF with TiO ₂ -0.5%	TiO ₂ -1%	-1.48700*	0.44146	0.021	-2.8315	-0.1425
	SiO ₂ ,TiO ₂ -1%	-0.17100	0.44146	1.000	-1.5155	1.1735
VF with TiO ₂ -1%	SiO ₂ ,TiO ₂ -1%	1.31600	0.44146	0.059	-0.0285	2.6605

*: The mean difference is significant at the 0.05 level; VF: Vertise Flow

The 1% SiO₂ group ranked next which had a significant difference with Vertise Flow group. No other significant differences were noted. Fig. 4 shows the error bar of the mean shear bond strength of bracket to tooth structure with 95% confidence interval.

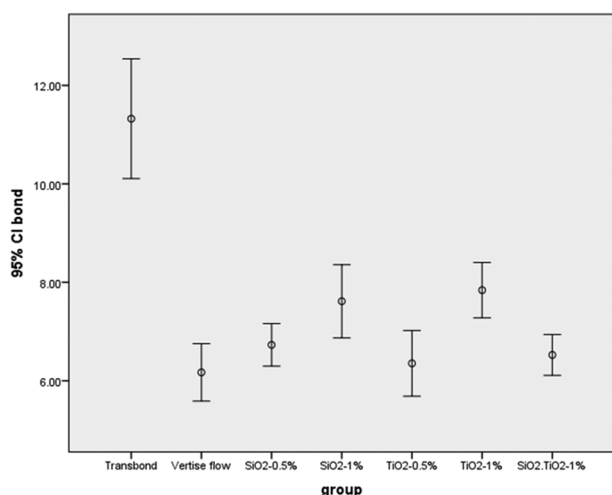


Figure 4. Error bar of the mean shear bond strength of bracket to tooth structure with 95% confidence interval.

Table 4 shows the ARI scores in the 7 groups. According to the Kruskal-Wallis test, only the control group had significant differences with other groups in terms of ARI scores ($p=0.000$).

Table 4. Adhesive remnant index (ARI) scores in the 7 study groups

Study groups	ARI score					Total
	1	2	3	4	5	
Transbond XT	1	7	2	0	0	10
Vertise Flow	0	0	0	5	5	10
VF with SiO ₂ -0.5%	0	0	1	8	1	10
VF with SiO ₂ -1%	0	0	4	6	0	10
VF with TiO ₂ -0.5%	0	0	0	7	3	10
VF with TiO ₂ -1%	0	0	3	7	0	10
VF with SiO ₂ , TiO ₂ -1%	0	0	0	8	2	10
Total	1	7	10	41	11	70

VF: Vertise Flow

DISCUSSION

This study aimed to assess the shear bond strength of a nano-hybrid composite made out of Vertise Flow self-adhesive composite containing TiO₂ and SiO₂ nanoparticles to tooth enamel following an additional etching step.

The concentration of nanoparticles was so that it caused minimal change in mechanical properties of the composite while maintaining optimal antimicrobial activity.^{6,7,23,24} An additional etching step prior to the application of composite on the enamel surface was also performed to improve the bond strength.²⁰⁻²² The results showed that the shear bond strength was significantly different among the 7 groups ($p=0.000$). Pairwise comparisons revealed that the bond strength in Transbond XT (control) group was significantly higher than that in the other groups ($p<0.05$) followed by 1% TiO₂ group with significant differences with Vertise Flow and 0.5% TiO₂ groups. SiO₂ 1% ranked next with a significant difference with Vertise Flow. Regarding the ARI scores, significant differences were detected only between the control group and other groups ($p=0.000$).

A literature search by the authors yielded only two studies on the application of Vertise Flow for bracket bonding. Gungor et al.¹⁸ reported that the bond strength provided by Vertise Flow was 5.90 ± 0.90 MPa, which was lower than the required threshold for bracket bonding. Goracci et al.¹⁷ reported that the bond strength of Vertise Flow was high prior to thermocycling but it significantly decreased afterwards and reached to 2.99 MPa, which is lower than the required bond strength for bracket bonding. However, they showed that etching of the enamel surface prior to the application of Vertise Flow yielded an acceptable bond strength before and after thermocycling. The value after thermocycling was 6.56 MPa; their results were in line with our findings. With regard to the optimal efficacy of enamel etching prior to the application of self-etch adhesive, a number of studies have shown that additional etching of enamel prior to the application of self-etch adhesive would improve the bond strength of adhesive to the enamel.²⁰⁻²² This finding was compatible with the current results and those of Goracci et al.¹⁷ since an additional etching step increased the bond strength of bracket to tooth structure.

Some previous studies have assessed the effect of incorporation of nanoparticles in composites for orthodontic bracket bonding on bond strength and other mechanical properties of composites. Evidence shows that increasing the concentration of nanoparticles in composite would enhance the antimicrobial properties, but may adversely affect the bond strength or other composite properties.^{24,26} Poosti et al.⁶ found no significant difference in bond strength of composites containing 1% TiO₂ and the conventional composite; the bond strength was within the clinically acceptable range and was therefore similar to that in our study. Akhavan et al.²⁴ showed that silver/hydroxyapatite nanoparticles had a significant effect on the shear bond strength; this effect was dose-dependent such that maxi-

mum shear bond strength was noted in the group with 1% concentration of nanoparticles, with no significant difference with the control group. Sodagar et al.⁷ indicated that incorporation of nano-chitosan increased the shear bond strength such that maximum mean bond strength was noted in the group containing 1% chitosan nanoparticles while minimum bond strength was recorded in the control group. In the present study, all groups containing nanoparticles had higher bond strength than the Vertise Flow group, which was in accordance with the findings of the aforementioned study.⁷ However, due to the differences in the type of nanoparticles, their results of the two studies cannot be accurately compared.

The current results indicated that 1% TiO₂ or 1% SiO₂ added to Vertise Flow may be able to slightly increase the shear bond strength of bracket to tooth structure. Another research demonstrated that addition of TiO₂ and SiO₂ nanoparticles to acrylic resin can have an adverse effect on flexural strength of the final product.⁸ Although the percentage of nanoparticles in their study was similar to ours, different results were obtained in the two studies, which may be due to the fact that they studied acrylic resin and measured the flexural bond strength while we studied composite resin and measured the shear bond strength. The type of applied adhesive resin could be also an important factor. It was observed that the addition of TiO₂ nanoparticles to Transbond XT composite adversely affected the shear bond strength and this effect was dose-dependent.²³ Their control group showed maximum bond strength with significant differences with other groups, which was in agreement with our results about Transbond XT. While in our study, Vertise Flow without any nanoparticle showed lower bond strength than those containing nanoparticles. On the other hand, nano-hybrid composite containing 1% TiO₂ had clinically acceptable shear bond strength, which was in line with our results.

In this study, a manual mixing method on a glass slab was applied which was successfully used in previous studies.⁶⁻⁸ The advantage of this method is that it is simple and thus more clinically applicable. According to the result of this study, adding nano particles to orthodontic composites by this method did not adversely affect its shear bond strength. Consequently, the clinicians who apply Vertise Flow composite can easily mix it with nanoparticles before its application to benefit from nanoparticles' antimicrobial activity.

Another important factor which should be considered for appropriate orthodontic adhesives is the amount of adhesive remaining on the tooth surface or bracket which can be evaluated by adhesive remnant index (ARI). Some previous studies found no significant difference between the groups in ARI scores.^{6,7,23,24} However, Gungor et al.¹⁸ reported that the distribution of ARI scores in Transbond XT group was significantly different from that in Vertise Flow group; this result was similar to our findings. But, Goracci et al.¹⁷ showed that the distribution of ARI scores after thermocycling was lower in Vertise Flow groups and had

significant differences with Transbond XT control group, which was different from our result.

According to Reynolds²⁷, the acceptable range of shear bond strength for bracket bonding is between 6 to 8 MPa. The shear bond strength in all groups in our study was within this range. Thus, it can be concluded that Vertise Flow with an additional etching step can result in a clinically acceptable bond strength in presence of minimum percentage of TiO₂ and SiO₂ nanoparticles (for their antibacterial property).

The homogeneous distribution of nanoparticles was investigated in 200 nm scale which was chosen according to previous publications.^{28,29} An acceptable homogeneity was observed in this study (Fig. 1). However, in the case that prepared nano-hybrid composites fail to show expected properties, it is also recommended to use higher magnification of SEM images to investigate the nanoparticles distribution following preparation of nano-hybrid composites.

This study had an in vitro design which could not perfectly simulate the oral clinical conditions because a combination of tensile, shear, and torsional forces are applied in the oral cavity. Also, stresses such as thermal alterations, humidity, acidity, and microbial plaque are present in the oral cavity, which are hard to simulate in vitro. Thus, generalization of results to the clinical setting must be done with caution. Future studies are required to incorporate nanoparticles in the composition of other composite resins to assess their effect on shear bond strength. Moreover, the effect of incorporation of other percentages of nanoparticles in Vertise Flow is worth being considered in further studies.

CONCLUSIONS

- Adding TiO₂ and SiO₂ nanoparticles to Vertise Flow self-adhesive composite not only did not adversely affect its shear bond strength, but also slightly increased it.
- The shear bond strength of Vertise Flow self-adhesive composite containing 0.5% and 1% TiO₂ and SiO₂ nanoparticles and combination of both after an additional etching step of tooth structure was within the clinically acceptable range.
- By adding TiO₂ and SiO₂ nanoparticles to self-adhesive composites we can benefit from the antimicrobial effect of these nanoparticles around orthodontic brackets without any adverse effect on the shear bond strength of the adhesive to the tooth enamel.

Funding

This research was a part of an MS thesis with the reference number of 257 conducted in and funded by International Campus of Dental School of Tehran University of Medical Sciences.

Ethics approval

Not applicable. This article does not contain any studies with human participants or animals. The study was approved by the ethics committee of Tehran University of Medical Sciences (IR.TUMS.DENTISTRY.REC.1396.2057).

Disclosure of interest

The authors declare that they have no competing interest.

REFERENCES

1. Derks A, Katsaros C, Frencken J, et al. Caries-inhibiting effect of preventive measures during orthodontic treatment with fixed appliances. *Caries Res* 2004; 38(5):413–20.
2. Matasa CG. Microbial attack of orthodontic adhesives. *Am J Orthod Dentofacial Orthop* 1995; 108(2):132–41.
3. Lim B-S, Lee S-J, Lee J-W, et al. Quantitative analysis of adhesion of cariogenic streptococci to orthodontic raw materials. *Am J Orthod Dentofacial Orthop* 2008; 133(6):882–8.
4. Uysal T, Yagci A, Uysal B, et al. Are nano-composites and nano-ionomers suitable for orthodontic bracket bonding? *Eur J Orthod* 2010; 32(1):78–82.
5. Yadav V. Nanotechnology, big things from a tiny world: a review. *AEEE* 2013; 3(6):771–8.
6. Poosti M, Ramazanzadeh B, Zebarjad M, et al. Shear bond strength and antibacterial effects of orthodontic composite containing TiO₂ nanoparticles. *Eur J Orthod* 2013; 35(5):676–9.
7. Sodagar A, Bahador A, Jalali YF, et al. Effect of chitosan nanoparticles incorporation on antibacterial properties and shear bond strength of dental composite used in orthodontics. *Iran J Ortho* 2016; <https://doi.org/1017795/ijo-7281>.
8. Sodagar A, Bahador A, Khalil S, et al. The effect of TiO₂ and SiO₂ nanoparticles on flexural strength of poly (methyl methacrylate) acrylic resins. *J Prosthodont Res* 2013; 57(1):15–9.
9. Choi JY, Kim KH, Choy KC, et al. Photocatalytic antibacterial effect of TiO₂ film formed on Ti and TiAg exposed to *Lactobacillus acidophilus*. *J Biomed Mater Res B: Applied Biomaterials: An Official Journal of The Society for Biomaterials, The Japanese Society for Biomaterials, and The Australian Society for Biomaterials and the Korean Society for Biomaterials* 2007; 80(2):353–9.
10. Feng X-X, Zhang L-L, Chen J-Y, et al. Preparation and characterization of novel nanocomposite films formed from silk fibroin and nano-TiO₂. *Int J Biol Macromol* 2007; 40(2):105–11.
11. Su W, Wei S, Hu S, et al. Preparation of TiO₂/Ag colloids with ultraviolet resistance and antibacterial property using short chain polyethylene glycol. *J Hazard Mater* 2009; 172(2-3):716–20.
12. Yao Y, Ohko Y, Sekiguchi Y, et al. Self-sterilization using silicone catheters coated with Ag and TiO₂ nanocomposite thin film. *J Biomed Mater Res B: Applied Biomaterials: An Official Journal of The Society for Biomaterials, The Japanese Society for Biomaterials, and The Australian Society for Biomaterials and the Korean Society for Biomaterials* 2008; 85(2):453–60.
13. Shibata T, Hamada N, Kimoto K, et al. Antifungal effect of acrylic res-

- in containing apatite-coated TiO₂ photocatalyst. *Dent Mater J* 2007; 26(3):437–44.
14. Jia H, Hou W, Wei L, et al. The structures and antibacterial properties of nano-SiO₂ supported silver/zinc-silver materials. *Dent Mater* 2008; 24(2):244–9.
 15. Sodagar A, Khalil S, Kassaei MZ, et al. Antimicrobial properties of poly (methyl methacrylate) acrylic resins incorporated with silicon dioxide and titanium dioxide nanoparticles on cariogenic bacteria. *J Orthod Sci* 2016; 5(1):7.
 16. Al-Saleh M, El-Mowafy O. Bond strength of orthodontic brackets with new self-adhesive resin cements. *Am J Orthod Dentofacial Orthop* 2010; 137(4):528–33.
 17. Goracci C, Margvelashvili M, Giovannetti A, et al. Shear bond strength of orthodontic brackets bonded with a new self-adhering flowable resin composite. *Clin Oral Investig* 2013; 17(2):609–17.
 18. Gungor AY, Alkis H, Turkkahraman H. Shear bond strengths of brackets bonded with a new self-adhering resin composite. *Int J Artif Organs* 2016; 39(8):431–4.
 19. İşman E, Karaarslan EŞ, Okşayan R, et al. Inadequate shear bond strengths of self-etch, self-adhesive systems for secure orthodontic bonding. *Dent Mater J* 2012; 31(6):947–53.
 20. Erickson RL, Barkmeier WW, Kimmes NS. Bond strength of self-etch adhesives to pre-etched enamel. *Dent Mater* 2009; 25(10):1187–94.
 21. Lühns A, Guhr S, Schilke R, et al. Shear bond strength of self-etch adhesives to enamel with additional phosphoric acid etching. *Operative Dentistry* 2008; 33(2):155–62.
 22. Van Landuyt K, Kanumilli P, De Munck J, et al. Bond strength of a mild self-etch adhesive with and without prior acid-etching. *J Dent* 2006; 34(1):77–85.
 23. Sodagar A, Akhouni MSA, Bahador A, et al. Effect of TiO₂ nanoparticles incorporation on antibacterial properties and shear bond strength of dental composite used in orthodontics. *Dental Press J Orthod* 2017; 22(5):67–74.
 24. Akhavan A, Sodagar A, Mojtahedzadeh F, et al. Investigating the effect of incorporating nanosilver / nanohydroxyapatite particles on the shear bond strength of orthodontic adhesives. *Acta Odontol Scand* 2013; 71(5):1038–42.
 25. Ritter DE, Bruggeman G, Locks A, et al. Bond strengths and adhesive remnant index of self-etching adhesives used to bond brackets to instrumented and uninstrumented enamel. *Am J Dent* 2006; 19(1):47–50.
 26. Hojati ST, Alaghemand H, Hamze F, et al. Antibacterial, physical and mechanical properties of flowable resin composites containing zinc oxide nanoparticles. *Dent Mater* 2013; 29(5):495–505.
 27. Reynolds IR. A review of direct orthodontic bonding. *Br J Orthod* 1975; 2(3):171–8.
 28. Sodagar A, Akhavan A, Hashemi E, et al. Evaluation of the antibacterial activity of a conventional orthodontic composite containing silver/hydroxyapatite nanoparticles. *Progress in Orthodontics* 2016; 17(1):1–7.
 29. Sodagar A, Bahador A, Pourhajibagher M, et al. Effect of addition of curcumin nanoparticles on antimicrobial property and shear bond strength of orthodontic composite to bovine enamel. *J Dent (Tehran, Iran)* 2016; 13(5):373.

Прочность сцепления при сдвиге самоклеящегося композита, содержащего наночастицы TiO_2 и SiO_2 , с дополнительным этапом травления для крепления ортодонтических брекетов к эмали

Сохеил Никпур¹, Атефе Саффар Шахруди², Аида Сафарпур³, Азам Акхаван⁴, Ахмед Содагар⁵

¹ Факультет стоматологии, Международный кампус при Тегеранском университете медицинских наук, Тегеран, Иран

² Стоматологический исследовательский центр, Исследовательский институт стоматологии и кафедра ортодонтии, Факультет стоматологии, Тегеранский университет медицинских наук, Тегеран, Иран

³ Центр исследования применения радиации, Тегеран, Иран

⁴ Кафедра хирургической стоматологии, Международный кампус факультета стоматологии при Тегеранском университете медицинских наук, Тегеран, Иран

⁵ Кафедра ортодонтии, Факультет стоматологии, Тегеранский университет медицинских наук и член Исследовательского института стоматологии, Исследовательский институт стоматологии, Тегеранский университет медицинских наук, Тегеран, Иран

Адрес для корреспонденции: Ахмед Содагар, Кафедра ортодонтии, Факультет стоматологии, Тегеранский университет медицинских наук, ул. „Северный Каргар“, Тегеран, Иран; E-mail: sodagara@tums.ac.ir; Тел.: (+98)9124070738

Дата получения: 18 июля 2020 ♦ **Дата приемки:** 19 февраля 2021 ♦ **Дата публикации:** 31 декабря 2021

Образец цитирования: Nikpour S, Shahroudi AS, Akhavan A, Saffarpour A, Sodagar A. Shear bond strength of self-adhesive composite containing TiO_2 and SiO_2 nanoparticles with an additional etching step for orthodontic brackets bonding to enamel. Folia Med (Plovdiv) 2021;63(6):865-74. doi: 10.3897/folmed.63.e56657.

Резюме

Введение: В последнее время наночастицы, такие как нано- TiO_2 , были добавлены в стоматологические материалы для улучшения профилактики кариеса благодаря их антибактериальной активности.

Цель: Данное исследование направлено на оценку макрошлифа (прочности сцепления на сдвиг) самоклеящегося композита, содержащего наночастицы TiO_2 и SiO_2 , для фиксации ортодонтических скоб.

Материалы и методы: Это экспериментальное исследование *in vitro* было проведено на 70 удалённых человеческих пре-молярах, разделённых на 7 групп. Были подготовлены шесть групп, на которые были нанесены образцы самоклеящихся композитов Vertise Flow: без наночастиц, с 0.5% и 1% наночастиц TiO_2 , 0.5% и 1% наночастиц SiO_2 и 1% смеси наночастиц TiO_2 и SiO_2 для получения наночастиц гибридные композиты. Были скреплены металлические брекеты с этими образцами, а также Transbond XT в качестве контрольной группы. Прочность на сдвиг брекетов к эмали измеряли на универсальной испытательной машине. Результат индекса остатков адгезива (ARI) также был определён с помощью стереомикроскопа. Данные были проанализированы с помощью однофакторного дисперсионного анализа, теста Тьюки и Краскела-Уоллиса.

Результаты: Была обнаружена значительная разница в прочности на сдвиг в группах ($p=0.000$). Парные сравнения показали, что сила прикрепления группы Transbond XT была значительно выше, чем у других ($p<0.05$), за ней следовала группа 1% TiO_2 со значительным различием с группами Vertise Flow и 0.5% TiO_2 . Наименьшее значение было обнаружено у Vertise Flow без наночастиц. Показатель ОРВИ отличался в контрольной группе ($p=0.000$).

Заключение: Добавление наночастиц TiO_2 и SiO_2 в самоклеящийся композит Vertise Flow не только не повлияло отрицательно на прочность на сдвиг, но и несколько повысило её. Как правило, самоклеящийся наногибридный композит, содержащий наночастицы TiO_2 и/или SiO_2 , после дополнительного травления может быть приемлем для крепления брекетов и может использоваться в клинике, чтобы воспользоваться антимикробной активностью этих наночастиц.

Ключевые слова

наночастицы, прочность сцепления на сдвиг, ортодонтические брекеты, самоклеящийся композит, TiO_2 - SiO_2
