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## ► To cite this version:

S N Reddy Duvvuri, M. Mohamed Arafath, Rama Krishna Alla, . Rajasigamani, V. Bhaskar, et al.. Evaluation of the Shear Bond Strength of the Orthodontic Composites Modified with Various Concentrations of TiO<sub>2</sub> Nanoparticles: An In vitro Study. UTTAR PRADESH JOURNAL OF ZOOLOGY, 2023, 44 (1), pp.88-94. 10.56557/UPJOZ/2023/v44i13395 . hal-04029743

**HAL Id: hal-04029743**

**<https://hal.science/hal-04029743>**

Submitted on 15 Mar 2023

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# Evaluation of the Shear Bond Strength of the Orthodontic Composites Modified with Various Concentrations of TiO<sub>2</sub> Nanoparticles: An *In vitro* Study

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## Authors' contributions

This work was carried out in collaboration among all authors. All authors read and approved the final manuscript.

## Article Information

DOI: 10.56557/UPJOZ/2023/v44i13395

### Editor(s):

(1) Prof. Aurora Martínez Romero, Juarez University, Mexico.

### Reviewers:

(1) Gunasekar Manoharan, USA.

(2) Tarulatha Shyagali, MR Ambedkar Dental College and Hospital, India.

(3) Penke Vijaya Babu, India.

Original Research Article

Received: 17/11/2022

Accepted: 20/01/2023

Published: 30/01/2023

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## ABSTRACT

**Aim:** This study aimed to evaluate the effect of various concentrations of TiO<sub>2</sub> nanoparticles incorporation on the shear bond strength of the two different orthodontic composites.

**Materials and Methods:** A total of 80 freshly extracted premolars were divided into two groups of 40 teeth each. The extracted teeth in each of these two groups were further divided into four subgroups with ten teeth (n=10) in each, depending on the concentrations of TiO<sub>2</sub> NPs (0.0%, 1.0%, 5.0% and 10.0%) in the Transbond XT and Enlight composites. Then the teeth were mounted in cold-cure acrylic resin poured into PVC tubes. The teeth were vertically embedded in acrylic up to the cemento-enamel junction (CEJ), and a standard bonding procedure was carried out to bond the brackets using both composites. The shear bond strength was evaluated using a universal testing machine at a crosshead speed of 05mm/minute until the brackets de-bond from the tooth surface. The obtained data were subjected to statistical analysis using Oneway ANOVA and Posthoc tests.

**Results:** The control groups of both the composites showed more shear bond strength compared to the modified groups. One-way ANOVA showed significant differences (p=0.000) among the groups. Posthoc analysis showed significant differences between the control and modified groups and also between the modified groups in both the composites except between the control and 1.0 wt% and 5.0 and 10.0wt% groups of the Enlight composite.

**Conclusion:** A decrease in the shear bond strength was observed with an increase in the concentrations of TiO<sub>2</sub> nanoparticles in both the composites.

*Keywords: Shear bond strength; Transbond XT; Enlight; Titanium di oxide nanoparticles.*

## 1. INTRODUCTION

“Orthodontic bracket bonding to composite resin restorations is frequently carried out in orthodontic clinics in view of the growing demands for orthodontic treatment among adult patients” [1]. “Other than the enamel, obtaining a strong bond to dental substrate is challenging” [2]. “Numerous methods have been suggested to strengthen the bonding, including acid etching, micro-etching, surface roughening, and surface conditioning by chemical agents” [1]. “For securing orthodontic brackets to the surface of the teeth, the bonding technique with resin-based composite as an adhesive agent has been utilised predominantly in orthodontics” [3]. “Though these bonding techniques are simple and improve esthetics, however, they have some inherent disadvantages like plaque accumulation, development of white spot lesions and bond failure, which cause prolonging the treatment course, impose a high cost, consume more chair time, and less optimal esthetic result occurs after treatment, due to demineralization of enamel adjacent to the brackets, especially around the bracket margin, due to the surface exposed to composite” [3]. The development of biofilms, a factor in dental caries, has been inhibited using a variety of techniques. Two of the most prevalent instances of such attempts include the examination of the efficacy of adding various antimicrobial agents to adhesives, such as fluoride and chlorhexidine [4,5].

Numerous antibacterial Nanoparticles (NPs) have been developed since the development of nanotechnology [6]. Due to their reduced size, nanoparticles are thought to effectively exhibit their antibacterial characteristics by penetrating the bacterial cell wall [7,8]. The antimicrobial activity of numerous nanoparticles like nanosilver [9,10], Zinc Oxide (ZnO) [9,11,12], Chitosan NPs [13], etc., were investigated with the composites and reported that they demonstrated significant antimicrobial activity with the expense of decreased mechanical properties.

TiO<sub>2</sub> nanoparticles' antimicrobial capabilities and their applications in medicine, dentistry, and other scientific fields are well documented [3,14]. According to Haghi et al. [15], “who examined the antibacterial impact of TiO<sub>2</sub> NPs on the pathogenic strain of E. coli, the TiO<sub>2</sub> NPs generate microscopic pores in the bacterial cell walls, which increase permeability and cause cell death. However, the literature on the effect of these NPs incorporation on the shear bond strength (SBS) of Orthodontic composites was scarce”. Therefore, this study aimed to evaluate the effect of incorporating various concentrations of TiO<sub>2</sub> NPs on the SBS of two different orthodontic composites.

## 2. MATERIALS AND METHODS

Two different composite materials such as Trnsbond XT (3M Unitek, Monrovia, CA, USA) and Enlight (Ormco Corporation, USA), were

used in this *in vitro* study and modified with various concentrations of TiO<sub>2</sub> nanoparticles with an average particle size of 8-100 nm (Nanoresearch Lab, India)

## 2.1 Nanocomposites Preparation

Nanocomposites were fabricated using the method described by Sodagar et al. [3] For the fabrication of composite containing different concentrations of TiO<sub>2</sub> NPs, 400 mg of TiO<sub>2</sub> NP powder was mixed with 3600 mg of each of the composites such as Transbond XT and Enlight composite, to obtain 4000 mg of the composites containing TiO<sub>2</sub> NPs with 10% concentration. To fabricate a composite containing 5% TiO<sub>2</sub> NPs, 1200 mg of the composite containing 10% TiO<sub>2</sub> NPs was mixed with 1200 mg of plain composite. To fabricate a composite containing 1% TiO<sub>2</sub> NPs, 240 mg of 10% TiO<sub>2</sub> NPs was mixed with 2160 mg of plain composite. Thus, 2560 mg of the composite containing 10% TiO<sub>2</sub> NPs, 2400 mg of the composite containing 5% TiO<sub>2</sub> NPs and 2400 mg of the composite containing 1% TiO<sub>2</sub> NPs were thus obtained. The required amounts of composite and the NPs were blended by using a composite mixer (High Energy Ball Mill, Retsch – Emax) at a speed of 3500 revolutions per minute in a dark chamber for 5 minutes.

## 2.2 Specimen Preparation and Evaluation of SBS

Eighty premolars, freshly extracted for orthodontic treatment purposes, were divided into two groups of 40 teeth each. The extracted teeth in each of these two groups were randomly divided into four subgroups with ten teeth (n=10) in each, depending on the concentrations of TiO<sub>2</sub> NPs in the composites. The inclusion criteria for tooth selection were anatomically & morphologically well-defined extracted premolars with intact buccal enamel surfaces, and without any developmental defects, enamel caries, or fractured crowns. Then the samples were mounted in cold-cure acrylic resin poured into PVC tubes. The teeth were vertically embedded in acrylic up to the cemento-enamel junction (CEJ), and a standard bonding procedure was carried out.

Buccal surfaces were cleaned with a prophylaxis brush without powder, rinsed and dried. 35% phosphoric acid (Ultra-Etch, Ultradent, USA) was administered for 30 seconds, followed by rinsing

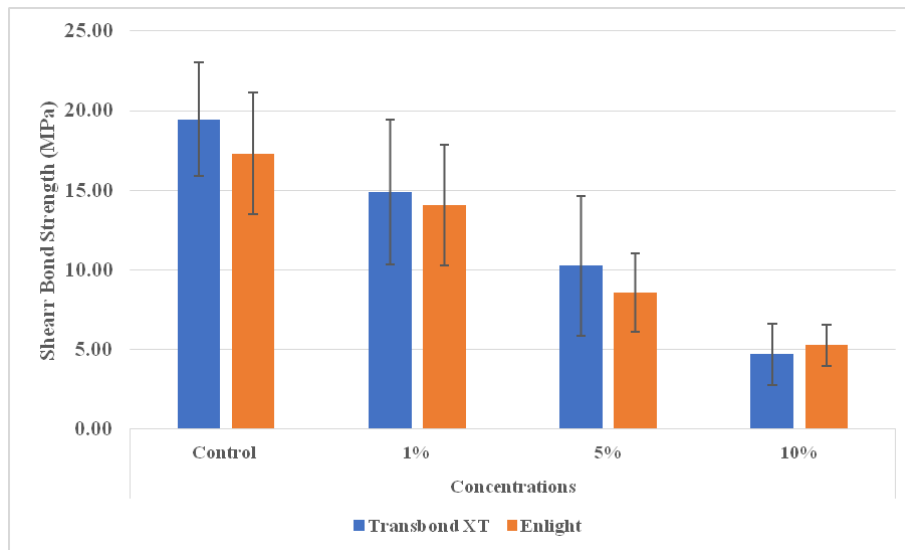
for 30 seconds and gentle air drying. After positioning the stainless-steel orthodontic brackets (Standard edgewise, 0.018-in slot, 12.62 mm<sup>2</sup> base area, Ormco Corporation, USA), a uniform, thin coating of adhesive (3M Unitek, USA) was applied to the buccal surfaces and light-cured (Woodpecker, UK) for 10 seconds. All specimens were stored in distilled water for seven days prior to the evaluation of the shear bond strength.

The shear bond strength (SBS) was measured using a universal testing machine (Model: AE-UTM-LC2, Advanced Equipments, India). The specimens were secured properly on the universal testing machine, and the bracket base was parallel to the direction of the applied force. Then, the shear load was applied at the bracket and the composite interface using a 0.6-mm metal blade in an incisogingival direction at a crosshead speed of 0.5 mm/min. The obtained debonding load (N) was divided by the bracket surface area (mm<sup>2</sup>) to calculate the SBS in megapascals.

The obtained data were subjected to statistical analysis using Statistical Package for Social Sciences, 21.0, NY, USA. The data were analyzed using one-way ANOVA, followed by Tukey's post-hoc test, used to find statistically significant differences between and within the groups. A p-value of less than 0.05 (p<0.05) was considered statistically significant, at a 95% confidence interval.

## 3. RESULTS

Table 1 shows the mean shear bond strength of Transbond XT and Enlight composites modified with various concentrations of TiO<sub>2</sub> NPs. The control groups of both the composite materials demonstrated more mean shear bond strength compared to the composites modified with various concentrations of TiO<sub>2</sub> NPs composite materials. Among the control groups, Transbond XT (19.45±3.56 MPa) showed slightly more SBS compared to the Enlight composite (17.31±3.81 MPa). Among the modified groups, composites incorporated with 1% of TiO<sub>2</sub> demonstrated more compared to the other modified groups. A decrease in SBS was observed as the concentration of TiO<sub>2</sub> NPs increased among both the modified composite groups (Fig. 1). One-way ANOVA showed significant differences (p=0.000) among both composite materials at various concentrations of TiO<sub>2</sub> NPs (Table 1).



**Fig. 1. Shear bond strength (MPa) of Transbond XT and Enlight composites modified with various concentrations of TiO<sub>2</sub> NPs**

**Table 1. Comparison of shear bond strength (Mean and Standard deviation) of Transbond XT and Enlight composites modified with various concentrations of TiO<sub>2</sub> NPs**

	Concentrations	Mean	Standard Deviation	F-value	p-value
<b>Transbond XT</b>	Control	19.45	3.562	28.462	0.000
	1%	14.90	4.545		
	5%	10.26	4.395		
	10%	4.68	1.909		
<b>Enlight</b>	Control	17.31	3.81	31.994	0.000
	1%	14.05	3.783		
	5%	8.55	2.47		
	10%	5.26	1.285		

**Table 2. Intergroup comparison of Shear bond strength (MPa) of Transbond XT and Enlight composites modified with various concentrations of TiO<sub>2</sub> NPs.**

	Concentrations		Mean Difference	Standard Error	Significance
<b>Transbond XT</b>	Control	1%	4.552*	1.678	0.048
		5%	9.196*	1.678	0.000
		10%	14.775*	1.678	0.000
	1%	5%	4.644*	1.678	0.042
		10%	10.223*	1.678	0.000
	5%	10%	5.579*	1.678	0.011
<b>Enlight</b>	Control	1%	3.257	1.352	0.094
		5%	8.759*	1.352	0.000
		10%	12.052*	1.352	0.000
	1%	5%	5.502*	1.352	0.001
		10%	8.795*	1.352	0.000
	5%	10%	3.293	1.352	0.088

\*statistically significant, Tukeys HSD

**Table 3. Comparison of Transbond XT and Enlight composites' shear bond strength at different concentrations of TiO<sub>2</sub> NPs**

	Group	Mean	Std. Deviation	t-value*	p-value
<b>Control</b>	Transbond XT	19.45	3.562	1.299	0.210
	Enlight	17.31	3.81		
<b>1%</b>	Transbond XT	14.90	4.545	0.453	0.656
	Enlight	14.05	3.783		
<b>5%</b>	Transbond XT	10.26	4.395	1.069	0.299
	Enlight	8.55	2.47		
<b>10%</b>	Transbond XT	4.68	1.909	0.798	0.435
	Enlight	5.26	1.285		

\*Independent sample t-test

On intergroup comparison, significant differences were observed between the unmodified and modified groups of Transbond XT composite material. Among the modified groups, significant differences were also displayed between the various concentrations of TiO<sub>2</sub> NPs incorporated in Transbond XT composites. The control group of Enlight composite material showed significant differences with the modified groups except with the 1wt% of TiO<sub>2</sub> NPs incorporated group. Among the modified Enlight groups, significant differences were observed except between 5wt% and 10wt% TiO<sub>2</sub> incorporated groups (Table 2). However, the independent sample t-test showed no significant differences between the composites at different concentrations of TiO<sub>2</sub> NPs (Table 3).

#### 4. DISCUSSION

Since their invention decades ago, composite resin adhesives have been the preferred option for bonding orthodontic brackets to the tooth; yet they may promote demineralization of adjacent enamel. According to De Soet and De Graaff, [16] "streptococcus mutans is the primary bacterium responsible for enamel demineralization". Several researchers attempted to improve the antimicrobial activity of orthodontic adhesives by incorporating various chemical agents [17,18]. Few studies [19,20], however, discovered that the bond strength of these orthodontic adhesives was reduced with the inclusion of various chemical agents.

Since the development of nanotechnology, NPs have found extensive usage in biological and pharmacological applications. According to Kim et al. [21], "NPs and their ions can generate free radicals, which trigger the production of reactive oxygen species (ROS), which can cause irreparable damage to the bacterial membrane, DNA, and mitochondria, ultimately leading to

bacterial death". "Several nanoparticles are known for their antimicrobial properties because of their small size and increased surface area" [22]. Numerous authors studied the effect of the incorporation of various nanoparticles on the biological and physico-mechanical properties of orthodontic adhesives. However, an increase in the NPs concentration demonstrated a decrease in the mechanical properties and the higher concentrations of NPs were reported to be cytotoxic [23,24]. The anatase form of TiO<sub>2</sub> NPS was proved to be an effective antimicrobial agent with superior mechanical strength and good biocompatibility [6].

The present *in vitro* study evaluated the effect of different concentrations of TiO<sub>2</sub> on the SBS of two Orthodontic composites, Transbond XT and Enlight. The present study reported that the addition of TiO<sub>2</sub> NPs decreased the SBS of both the orthodontic composites compared to the control groups. As the concentration of NPs was increased in the composites, a decrease in SBS was observed.

The particle size and shape of the NPs significantly affect the SBS of polymer-based materials to which they were added. Nanoparticles tend to agglomerate as their concentration is increased due to the interactions between them. The decrease in the SBS, in the current study, with an increase in the concentration of NPs can be assumed to be the agglomeration of the NPs resulting in their size. In addition, the agglomerated particles might have created voids, which act as stress concentrators and encourage crack propagation under stress [25,26]. The large-shaped particles were proved to be less cytotoxic compared to the smaller and finer particles [6]. The particle size of the NPs used in this study was in the range of 80-100 nm. However, the agglomeration might have resulted in increasing their size.

The high bond strength with the adhesive materials is primarily achieved by increasing the cohesion within the adhesive rather than the adhesion between the adhesive and the tooth structure [27]. The decrease in the bond strength with an increase in the NPs in the present study can also be attributed to the interference of the agglomerated NPs with the cross-linking of polymer chains resulting in the shearing of the polymer chains over each other under stresses reduced the bond strength with the natural tooth [28]. The depth of cure significantly influences the SBS of orthodontic composites. Several researchers reported that the agglomerated NPs could interfere with the curing process and reduce the depth of cure of resins and reduce the SBS [29].

According to Reynolds [30], "the clinically acceptable range for SBS is 6 to 8 MPa. The present study demonstrated that the mean SBS of the modified groups was greater than the clinically acceptable range except with the addition of 10 wt% TiO<sub>2</sub>. The results of the present study were in agreement with the study conducted by Mahendra TVD et al. [29] and Reddy et al. [9].

The present in vitro study investigated the effect of various concentrations of TiO<sub>2</sub> NPs on the SBS of two different Orthodontic adhesives. However, diverse oral conditions significantly influence the SBS of composites. Hence, further studies may be experimented with in vivo conditions and also decreasing the concentrations of TiO<sub>2</sub> NPs less than 1.0wt%.

## 5. CONCLUSION

Within the limitations of the present study, the following conclusions can be drawn.

- Incorporating TiO<sub>2</sub> NPs reduced the shear bond strength of the orthodontic composites compared to the unmodified composites.
- As the concentration of TiO<sub>2</sub> NPs increased a significant decrease in SBS was observed.
- Both Transbond XT and Enlight composite materials showed comparable SBS at different concentrations of TiO<sub>2</sub> NPs incorporated.

## COMPETING INTERESTS

Authors have declared that no competing interests exist.

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