Impact of Titanium Oxide Nanoparticles on Microleakage and Bond Strength of Orthodontic Brackets: An In Vitro Analysis

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Abstract: The present study compares the microleakage and shear bond strength (SBS) of orthodontic brackets bonded with conventional composite resin and composite resin containing Titanium oxide (TiO2) nanoparticles after thermocycling. Eighty human extracted premolars bonded with 0.022 slot MBT brackets were divided into Group I (TiO2 nanoparticles composite) and Group II (conventional Transbond XT composite). After bonding, samples were thermocycled between 5°C and 55°C and evaluated for microleakage (stereomicroscope, 40X) and SBS (Universal testing machine). Adhesive Remnant Index (ARI) was scored under 10X magnification, and data was analyzed using a t-test and Chi-square test. The results showed that Group II (conventional composite) demonstrated higher shear bond strength (19.01 MPa) than Group I (15.05 MPa, $p<0.001$). Group II also showed lower microleakage (0.42 mm) than Group I (0.83 mm, $p=0.01$). Incisal microleakage was lower ($p=0.16$), with a higher ARI score ($p=0.03$) in the Group II samples. The current study revealed that the conventional composite resin (Transbond XT) has a higher shear bond strength and decreased microleakage compared to Titanium oxide nanoparticle incorporated composite within a clinically significant range. Conventional composite resin bonding also exhibited higher Adhesive remnant index scores, indicating a reduced risk of enamel damage during debonding.
Keywords: Orthodontic brackets; Composite resin; Titanium oxide; Nanoparticles; Debonding.

INTRODUCTION

In orthodontics, fixed mechanotherapy involves bracket bonding on enamel surfaces. Ensuring adhesive longevity prevents patient discomfort, delays, or enamel damage (Bakhadher *et al.*, 2015). Potential effects of mechanotherapy include enamel demineralization causing white spot lesions (WSLs) and early dental caries around bonded brackets (Leloup *et al.*, 2001). WSLs impact 2-96% of patients within 4 weeks, often influenced by compromised oral hygiene and harmful biofilm, potentially leading to treatment disruptions (Buonocore, 1955). Microleakage, the infiltration of bacteria, fluids, and ions between prepared cavities and restorative materials, contributes to WSL development. Managing microleakage is complex, leading to enamel decalcification, visible as white spot lesions (Bishara *et al.*, 1999; Vicente *et al.*, 2009).

After bracket bonding, the bond strength of the composite resin should be capable of resisting the forces applied during the

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orthodontic treatment, mastication, and stresses exerted by the archwires (Sharma *et al.*, 2014; Thekiya *et al.*, 2018). In orthodontics, acid-etch bonding is the most often used surface preparation method and may serve as a medium for plaque accumulation, leading to an increase in enamel demineralization and eventually causing bond failure (Bishara *et al.*, 1998).

Nanoparticles as fillers in nanocomposites leverage their high surface-to-volume ratio to enhance mechanical and aesthetic properties, with higher concentrations displaying increased microleakage resistance (Jandt *et al.*, 2020). In fixed mechanotherapy, enamel demineralization affects 50-70% of patients due to bacterial aggregation at the adhesive-enamel junction (Borzabadi-Farahani *et al.*, 2014; Park *et al.*, 2009). Nano adhesives, with particle sizes ≤ 100 nm, are extensively used in orthodontic bonding for superior dentin and enamel bond strength and marginal seal. Nanoparticles integrated into orthodontic materials exhibit biocidal properties, deterring oral biofilm growth and demineralization around brackets (Hedayati *et al.*, 2018). Salman OL *et al.* (2021) demonstrated that exposure to oral fluids induces elastic deformation, biodegradation, and physical changes in the tooth and adhesive, leading to micro-leakage and reduced bond strength.

Nanoparticles in biomaterials offer antibacterial potential in orthodontics, achievable through their integration with composites, Glass ionomers, or coatings. Notably, nanoparticles like chitosan, zinc oxide, silver, titanium, and copper exhibit antibacterial properties (McInnes *et al.*, 1992; Asiry *et al.*, 2018). Researchers highlighted the nanoparticle-reinforced composites for their exceptional antibacterial properties while maintaining shear bond strength. Titanium dioxide (TiO2) nanoparticles have gained interest for their photocatalytic activity, with TiO2-containing resins demonstrating potent antibacterial effects to counter enamel demineralization and recurrent caries (Reddy *et al.*, 2016; Felemban *et al.*, 2017). There is not much-published research on the impact of TiO2 nanoparticles on microleakage and SBS following thermocycling. Hence, this study investigates the microleakage and shear bond strength in orthodontic brackets bonded with conventional composite resin (Transbond XT) and titanium oxide nanoparticles-incorporated composite resin after thermocycling.

MATERIALS AND METHODS

The present research was conducted at the Department of Micro and Nano-mechanical Testers, Indian Institute of Science (IISc), Bangalore, after obtaining an Institutional Ethical Clearance Certificate (IEC: TODC/221/ECAL/2020-21) from The Oxford Dental College, Bangalore.

SAMPLE SIZE CALCULATION

The GPower software (v. 3.1.9.4) was used to determine the sample size. Considering the effect size to be measured (d) at 80% for the Two-tailed hypothesis, the power of the study at 80%, and the alpha error at 10%, the total sample size needed was 40 for each group. This study consisted of two main groups: Group I, an experimental group (n=40), and Group II, the control group (n=40).

Group I (Experimental group): This group consisted of 40 premolars, and they were further subdivided into Subgroup-A (20 premolars) to assess for shear bond strength and Subgroup-B (20 premolars) for microleakage testing which were bonded with 5% Titanium oxide nanoparticles incorporated in Transbond XT composite resin.

Group II (Control group): The control group consisted of 40 premolars, and they were further subdivided into Subgroup-A (20 premolars) to assess for shear bond strength and Subgroup-B (20 premolars) for microleakage testing, which were bonded by using conventional composite resin (Transbond XT, 3M Unitek Dental Products, CA, USA).

The PEA brackets (Ortho Organizers, MBT 0.022 slots, Carlsbad, USA) were used in both groups I and II.

MATERIALS

In this study, the conventional composite resin used was Transbond XT, a light-cured orthodontic adhesive containing Bisphenol A-glycidyl methacrylate (Bis-GMA), manufactured by 3M Unitek Dental Products, CA, USA. Transbond XT is well-regarded for its effectiveness and widespread use in orthodontic applications. The titanium oxide nanoparticles employed were obtained in powder form from Kronos Titan GmbH, Leverkusen, Germany, with particle sizes less than 100 nm. To prepare the composite resin with TiO2 nanoparticles, 400 mg of the nanoparticles were manually mixed with 3600 mg of Transbond XT, resulting in a composite with a 5% TiO2 nanoparticle concentration and a total weight of 4000 mg.

PHYSICOCHEMICAL CHARACTERISTICS OF THE MATERIALS

Conventional composite resins used in this study are primarily composed of a resin matrix with monomers like Bisphenol A-glycidyl methacrylate (Bis-GMA) or Urethane Dimethacrylate, combined with glass or ceramic fillers and silane coupling agents to enhance bonding. These composites exhibit various properties, including compressive strengths between 200 and 300 MPa, shear bond strengths from 15 to 25 MPa, and flexural strengths of 80 to 150 MPa. Light-cured composites typically have a setting time of 20-30 seconds and viscosities between 200 and 500 Poise, with hardness values ranging from 40 to 70 HV. They are known for their good color stability and moderate wear resistance and are available in various viscosities to meet different orthodontic needs. Titanium dioxide (TiO_2) nanoparticles, typically ranging from 1 to 100 nanometers and often in anatase form, can enhance the mechanical properties of composites due to their high hardness and strength. They also exhibit antimicrobial properties by generating reactive oxygen species under UV light.

Preparation method of Titanium oxide nanoparticles incorporated into composite resin

To prepare the composite containing 5% titanium oxide nanoparticles, 400mg of titanium oxide nanoparticle powder was manually mixed in a glass beaker with approximately 3600mg of Transbond XT composite to obtain 4000mg of the composite (Fig. 1).

Figure. 1. Manual incorporation of Titanium oxide nanoparticles into Transbond XT composite.

Group I sample bonding: The teeth were bonded with TiO_2 nanoparticles composite resin.

Group II samples bonding: The conventional composite resin (Transbond XT, 3M Unitek Dental Products, CA, USA) was used for bonding.

THERMOCYCLING AND MICROLEAKAGE TEST

Forty specimens were subjected to thermocycling from each group at Praj Metallurgical Laboratory, Pune, India. The thermocycling test consisted of 1000 cycles in distilled water between 5°C and 55°C. After thermocycling, the teeth were sectioned longitudinally in an incisor-cervical direction with a diamond saw (Fig. 2), and each section was examined on both sides. The percentage of microleakage for each surface area was ascertained by using a Stereomicroscope (Stereomicroscope 40x, Lynx, EVO Dynascope®) at IISc, Bangalore, with 40X magnification on the enamel-adhesive interface.

Figure 2. Cut section of tooth showing the bracket, adhesive, and tooth surface interface.

Shear bond strength test and Adhesive remnant index (ARI)

The Universal testing apparatus (Universal Testing Machine, Mecmesin OmniTest-25, Britain) was utilized to determine the shear bond strength at IISc, Bangalore (Fig. 3). A 0.6mm metal blade was applied in an incisor-gingival direction at a crosshead speed of 0.5mm/min to apply a shear force to the composite interface. The value found was then divided by the bracket surface area to get the SBS. After debonding, the teeth and the brackets were examined under a stereomicroscope of 10X magnification (Lynx, EVO Dynascope®) at IISc, Bangalore to calculate the ARI on a scale between Score 0 and 3.

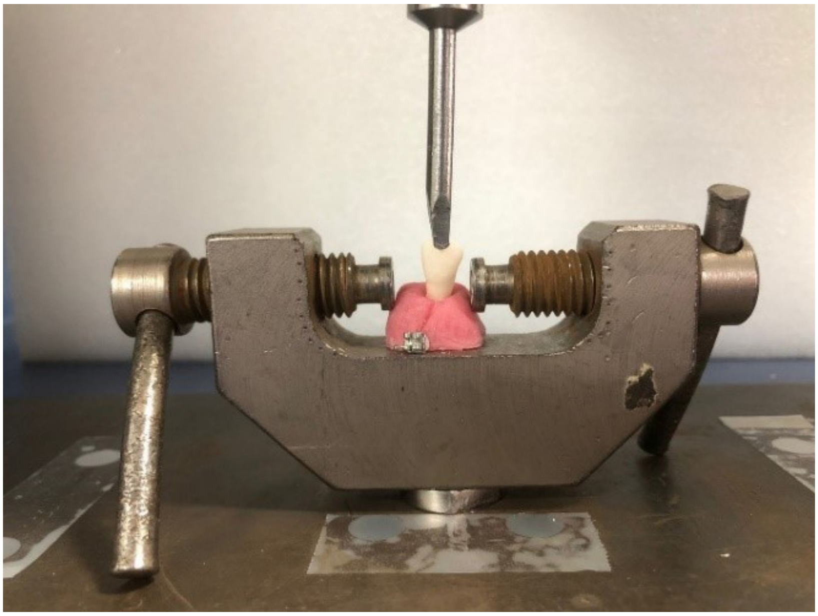

Figure 3. Evaluation of shear bond strength using a Universal Testing Machine.

Statistical Analysis

The Statistical Package for Social Sciences (SPSS, Version 22.0, SPSS Inc., IBM Corporation, New York, USA) was used to analyze the statistical data. An independent Student t-test was used to compare mean shear bond strength and microleakage. The chi-square test was used to compare the mean scores of microleakage testing and the ARI between the two groups. The level of significance (p-value) was set at $P<0.05$.

RESULTS

The shear bond strength (Table 1) varied significantly between Group I (15.05 ± 3.00Mpa) and Group II (19.01 ± 1.84Mpa), with Group II demonstrating higher bond strength ($p<0.001$). Microleakage distance (Table 2) was also significantly lower in Group II (0.42 ± 0.43mm) compared to Group I (0.83 ± 0.47mm with p=0.01). While microleakage scores (Table 3) showed no significant difference between the groups (p=0.16), Group II exhibited higher proportions of no microleakage (35%) and score 1 compared to Group I (10% and 55%, respectively). Distribution of microleakage (Table 4) in the incisal region was lower in Group II (15.4%) compared to Group I (38.9%) and higher in the gingival region (84.6% vs. 61.1%), though not statistically significant (p=0.16). Adhesive remnant index scores (Table 5) revealed a significant predominance of Score 1 in Group II compared to Score 2 in Group I (p=0.03).

Groups	Number of samples	Mean	Standard Deviation	Mean Difference	p-value
Group I	20	15.05	3.00	3.96	0.001*
Group II	20	19.01	1.84		

Table 1. Comparison of mean Shear bond strength (in Mpa) between 2 groups using an independent Student t-test.

Groups	Number of samples	Mean	Standard Deviation	Mean Difference	p-value
Group I	20	0.83	0.47	–0.41	0.01*
Group II	20	0.42	0.43		

Table 2. Comparison of mean Microleakage distance (in mm) between 2 groups using an independent Student t-test.

Variable	Category	GROUP I		GROUP II		p-value
		Number of samples	%	Number of samples	%	
Micro Leakage Region	Incisal	7	38.9%	2	15.4%	0.16
	Gingival	11	61.1%	11	84.6%	

Table 3. Comparison of Microleakage Scores between 2 groups using the Chi-Square Test.

Variable	Category	GROUP I		GROUP II		p-value
		Number of samples	%	Number of samples	%	
Micro Leakage Scores	Score 0	2	10.0%	7	35.0%	0.16
	Score 1	16	80.0%	11	55.0%	
	Score 2	2	10.0%	2	10.0%	

Table 4. Comparison of Microleakage region (Incisal/ gingival) between 2 groups using the Chi-Square Test.

Variable	Category	GROUP I		GROUP II		p-value
		Number of samples	%	Number of samples	%	
Adhesive Remnant Index	Score 0	7	35.0%	9	45.0%	0.03*
	Score 1	3	15.0%	8	40.0%	
	Score 2	6	30.0%	0	0.0%	
	Score 3	4	20.0%	3	15.0%	

Table 5. Comparison of Adhesive Remnant Index (ARI) Scores between 2 groups using the Chi-Square Test.

DISCUSSION

In the initial phases of orthodontic therapy, bracket attachment to stainless steel bands was uncomfortable, causing gingival trauma and decalcification. Extensive space creation and closure around each tooth prompted innovative bonding approaches, including new adhesives, base designs, bracket materials, curing methods, primers, fluoride-releasing agents, and sealants (Khosravanifard *et al.,* 2011). Successful orthodontic bonding requires mechanical and chemical surface conditioning, optimal material selection, and careful bracket handling (Ferracane *et al.*, 1992; Proffit *et al.*, 1993).

Orthodontic appliances disrupt teeth's natural cleaning, leading to plaque accumulation and decay. Antimicrobial agents like Silver and Titanium dioxide nanoparticles prevent enamel demineralization (Park *et al.*, 2009; Shaik *et al.*, 2018). Studies show silver nanoparticles reduce bacterial adhesion compared to traditional composites, preventing decay. However, periodontal diseases and white spot lesions remain challenges in orthodontic treatment outcomes.

Reynolds emphasized the crucial bond strength requirement between brackets and composite materials to withstand masticatory forces. He suggested a range of 5.9-7.8 MPa as adequate, while others noted higher values, highlighting shear bond strength variability across oral regions (Sodagar *et al.*, 2013; Hedayati *et al.*, 2018). The direct bonding technique revolutionized orthodontic interventions, stressing the importance of maintaining clinically acceptable bond strength with minimal enamel damage.

Orthodontic bonding materials undergo aging in the oral environment, leading to microcrack formation and eventual debonding (Park *et al.*, 2009). Microleakage, a primary contributor to enamel decalcification, worsens due to intraoral temperature fluctuations (Salman *et al.*, 2021). Factors like polymerization shrinkage, water sorption, and differences in thermal expansion coefficients affect microleakage (McInnes *et al.,* 1992). Bacterial activity, chewing-related wear, and acidic food erosion also contribute to adhesive biodegradation (Vicente *et al.*, 2009).

Nanotechnology has enhanced orthodontic materials by improving antimicrobial properties and mechanical strength (Arhun *et al.,* 2006). Nanoparticles, such as Titanium dioxide (<100 nm), act as nano adhesives, antimicrobial agents, remineralization agents, coating brackets, wires, and anchorage devices. Nano adhesives offer increased dentin and enamel bond strength, stress absorption, shelf life, and fluoride release without separate etching. Incorporating zirconium oxide and titanium oxide nanoparticles improves mechanical properties in orthodontic adhesives (Sodagar *et al.*, 2013).

Literature confirms the antimicrobial properties of nanoparticle-containing composites for preventing enamel demineralization around brackets (Ahn *et al.*, 2009). Titanium dioxide nanoparticles combined with orthodontic materials exhibit antimicrobial effects against bacteria (Reynolds *et al.*, 2010).

The temperature variations significantly impact adhesive stress and microleakage. Thermocycling studies have revealed reduced bond strength and increased microleakage due to thermal stress. Polymerization shrinkage with light-cured adhesives contributes to microleakage. Achieving an effective tooth-adhesive interface involves addressing polymerization shrinkage, post-curing water sorption, and differences in thermal expansion coefficients. Oral fluid exposure disrupts the adhesive interface, leading to deformation, biodegradation, and microleakage. In vitro thermocycling simulates oral temperature changes and aging, affecting adhesive properties (McInnes PM *et al.*, 1992).

Various studies suggest the potential of nanoparticles to overcome microleakage and enhance mechanical properties (Hegde *et al.*, 2010; Hedayati *et al.*, 2018). Studies have shown that 5%

TiO2 nanoparticles maintain clinically acceptable shear bond strength without compromising other mechanical properties (Sodagar *et al.*, 2013; Shaik *et al.*, 2018). Orthodontic composites with 1% TiO2 nanoparticles significantly reduce microbial overgrowth without compromising shear bond strength. TiO2 nanoparticles, combined with silica and silver nanofillers, aid in preventing enamel demineralization while maintaining physical properties (Felemban *et al.*, 2017). The concentration of TiO2 nanoparticles is crucial, with 5% showing favorable results compared to 1% and 10%. The present study incorporated 5% titanium oxide nanoparticles to assess their effects on shear bond strength and micro leakage during aging (Murray *et al.*, 2003; Asiry *et al.*, 2018).

Comparisons of shear bond strength between the groups Transbond XT with 5% titanium oxide nanoparticles (Fig. 4) and Transbond XT (Fig. 5) revealed significantly higher mean shear bond strength in conventional bonding, which was statistically significant ($p<0.001$). However, the two groups had no statistically significant difference in mean microleakage ($p=0.16$). Microleakage was lesser in conventional composite bonding (0.42 ± 0.43) compared to titanium oxide nanoparticles in composite resin (0.83 ± 0.47). Differences in microleakage distribution between incisal and gingival surfaces were statistically insignificant ($p=0.16$). The Adhesive Remnant Index score was significantly higher in conventional composite compared to Score 2 in titanium oxide nanoparticles in composite resin ($p=0.03$).

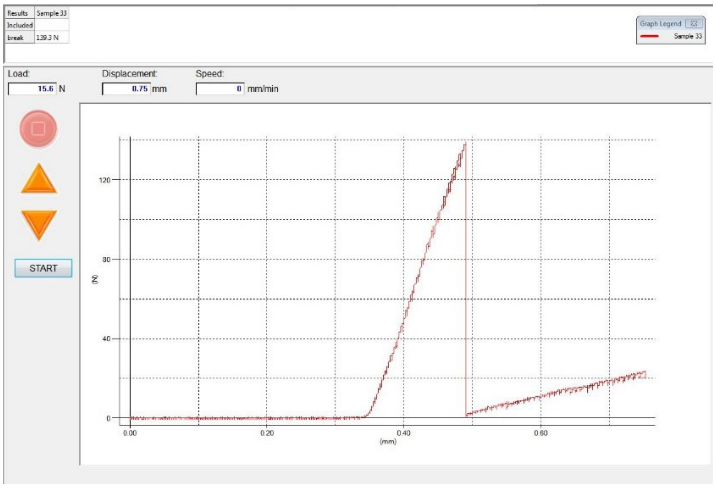

Figure 4. Group I samples showing shear bond strength breakage point at 139Newton

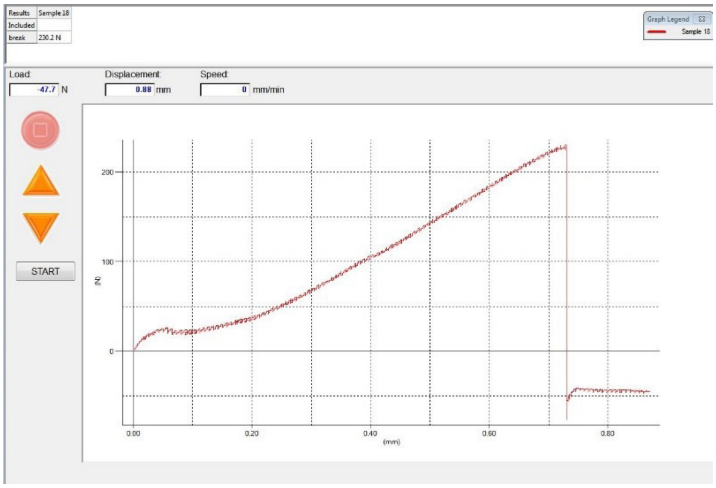

Figure 5. Group II samples showing shear bond strength breaking point at 230Newton

Achieving adequate shear bond strength is crucial, with low ARI scores indicating a lowered risk of enamel damage. The current research demonstrated that the conventional composite (Transbond XT) offers favorable debonding characteristics, microleakage, and a high ARI score compared to titanium oxide nanoparticles incorporated in composite resin. The conventional adhesive composite resin displayed a high ARI score on the bracket surface, indicating that debonding occurs at the tooth resin interface. This minimizes the risk of enamel damage from debonding forces.

LIMITATIONS AND FUTURE SCOPE

This study has a few limitations, including its in-vitro nature and smaller sample size. Therefore, the results should be cautiously compared with clinical research findings and validated with a larger sample size. Analyses such as biosorption, time-dependent toxicity, material-dependent toxicity, and dose-dependent toxicity are critical factors for understanding these materials' long-term safety and biocompatibility. However, this study did not analyze these aspects due to its in vitro nature. These factors are inherently related to biological interactions and long-term exposure, which are beyond the scope of in vitro experiments. Future clinical research involving actual patients and biological fluids, such as saliva, will be necessary to evaluate these safety parameters comprehensively. Additionally, it is suggested that manufacturers design and introduce nanocomposites with appropriate viscosity to enhance their effectiveness as a preferred orthodontic adhesive.

CONCLUSIONS

Orthodontic brackets bonded with conventional composite resin (Transbond XT) exhibited a superior shear bond strength compared to Titanium oxide nanoparticle incorporated composite within a clinically significant range. The titanium oxide nanoparticle composite exhibited greater microleakage, particularly in the gingival third. Furthermore, the conventional composite resin had a higher ARI score on the bracket surface, indicating that debonding occurs at the tooth-resin interface, thereby minimizing the risk of enamel damage from debonding forces.

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Conflict of interest

None. ♦

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