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Invitro evaluation of shear bond strength of orthodontic brackets bonded with modified orthodontic adhesive and / primer containing various concentrations of tio$_2$ nanoparticles

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Abstract

Objective: The present study aims to assess the effect of incorporating different percentages (wt %) of Titanium dioxide(TiO_2) nanoparticles into the bonding system's components to obtain a composite and primer with sufficient bond strength to enamel surface for use in Orthodontics.

Materials and Methods: Incorporation of 1%(w/w) Titanium dioxide nanoparticles and 5%(w/w) Titanium dioxide nanoparticles into the components of the orthodontic bonding system and subdivided into three groups, i.e., nanocomposite, nano primer and combination of both used for bonding the orthodontic stainless steel brackets.

Results: The mean shear bond strength of the 1%(w/w) and 5%(w/w)Titanium dioxide nanocomposite(20.243.46; 14.893.59) showed highest followed by nanoprimer(13.014.10; 10.162.28) and combination of both(9.592.97; 7.471.40) the nanocomposite and nanoprimer used for bonding. Amongst the two concentrations 1%(w/w) Titanium dioxide nanoparticles showed higher shear bond strength(SBS) than 5% (w/w) Titanium dioxide nanoparticles.

Conclusion: The addition of 1% (w/w)Titanium dioxide and 5%(w/w) Titanium dioxide nanoparticles into the orthodontic bonding system components seems to affect the mechanical properties of experimental groups shows higher than clinically acceptable SBS value.

Keywords: Titanium dioxide, Nanoparticles, Shear Strength, Adhesive Remanent Index.

Introduction

The technique of bonding orthodontic accessories, with the introduction of enamel acid etching by Buonocore and its association with organic material, bisphenol A glycidyl methacrylate(Bis GMA) developed by Bowen has made a tremendous evolution in developing various bonding resins to enamel surfaces, Newmann introduced the technique of bonding orthodontic brackets to the tooth.[1, 2]

Decades since their introduction into the field of orthodontics, composite resin adhesives undoubtedly remain the first choice of bonding material.[3] However, it also associates risks and complications of fixed appliance therapy leads to plaque accumulation, a rapid shift in the bacterial flora of plaque occurs elevated levels of acidogenic bacteria are present in the plaque, most notably Streptococcus mutans and Lactobacillus acidophilus.[4] High levels of these bacteria can decrease the PH of plaque in orthodontic patients to a greater extent than in non-orthodontic patients. The acidic byproducts of these bacteria in plaque are responsible for the subsequent enamel decalcification and white spot lesions(WSLs).[5-9] Several methods have been used to inhibit bacterial growth and reduce the incidence of decalcification and formation of WSLs.[10] The use of antimicrobial agents such as nanoparticles has attracted much attention in medicine and dentistry, nanoparticles with commercially available composite for the development of antimicrobial nanocomposite for prevention of formation of WSLs.

Nanoparticles are believed to efficiently penetrate the cell wall of bacteria due to their smaller size, effectively exerting their antibacterial properties.[11] Streptococcus mutans are sensitive to nanoparticles of TiO_2, which allows achieving acute clinical effects.[12] Ghada Salem et al.[13] studied the effect of adding nanoparticles into the primer and the composite. The results showed that incorporating nanoparticles into orthodontic primer decreases the SBS, while composite containing nanoparticles show acceptable SBS for bonding the orthodontic brackets. AK Reddy et al.[14] investigated the influence of various nanoparticles on shear bond strength. They concluded that incorporating nanoparticles, even in minimal amounts, may decrease shear bond strength and may lead to bracket or adhesive failure.

The development of clinically acceptable orthodontic bonding systems with additional antimicrobial effects could be undertaken if their mechanical properties have also been considered. Therefore, incorporating various concentrations of TiO_2 nanoparticles(TiO_2 NPs) into the orthodontic bonding system components, I.e., orthodontic primer, orthodontic composite resin alone, thereby increasing the overall concentration of TiO_2 NPs but maintaining the minimum concentration within the individual components of the bonding system.

The present study aims to assess the effect of incorporating different percentages (wt %) of TiO_2 nanoparticles into the bonding system components to obtain a nanocomposite and nanoprimer with sufficient bond strength to enamel surface for orthodontic bonding.

Materials and Methods

The materials and the equipment used for the study are tabulated as follows (Table 1)

Table 1: Materials and the equipment used for the study are

Materials	Equipment
● TiO_2 NPs (dry nanopowder, average primary particle size: 30 -50 nm;purity: >99.5%, Nano Research Lab, Jharkhand, India) ● 37% phosphoric acid (Eazetech, Anabond). ● Primer (Orthosolo, Ormco, CA). ● Conventional composite (Enlight, Ormco Crop, CA) ● 0.022 slot MBT premolar brackets (Ortho Organizers, Di-MiM, Mini -Twin)	● Digital weighing machine ● Composite mixer: High energy ball mill (RETSCH-EMAX, Andhra University, visakhapatnam, AP). ● Universal testing machine: INSTRON model-8801 (Gitam university, Visakhapatnam, AP). ● Stereomicroscope x20 magnification (zeiss, ProgRes, C3 (Andhra University, Visakhapatnam, AP).

Methodology

The study consists of various concentrations of TiO_2 nanoparticles blended with orthodontic adhesive and orthodontic primer for the preparation of nanocomposite and nanoprimer used for bonding the orthodontic brackets and were divided into groups and subgroups.

TiO_2 Nanocomposite preparation

Addition of TiO_2 Nanoparticles into the composite for the preparation of 1% (w/w) and 5%(w/w) TiO_2 nanocomposite. The desired quantity of TiO_2 Nanoparticles for the sample preparation was measured using a digital weighing machine. The first sample, 20 mg of TiO_2 Nanoparticles, was added to 2000 mg of Conventional composite (Enlight, Ormco Crop, CA). For the second sample, 100 mg of TiO_2 Nanoparticles was added to 2000 mg of Conventional composite (Enlight, Ormco Crop, CA) and blended by using a composite mixer (High energy ball mill, RETSCH-EMAX, Andhra University, Visakhapatnam, Andhra Pradesh) at a speed of 3500 revolutions per minute(RPM) in dark environment for 5min.[3]

TiO_2 nanoprimer preparation

Addition of TiO_2 Nanoparticles into the primer for the preparation of 1% (w/v) and 5%(w/v) TiO_2 nano primer. For the first sample, 1%(w/v) TiO_2 nanoparticles were added to every 1mm of the primer using a graduated pipette. For the second sample, 5%(w/v) TiO_2 nanoparticles were added to every 1mm of the primer using a graduated pipette, and thorough mixing was performed using a vortex shaker (V-MIX, Department of microbiology, GEMS, Srikakulam, Andhra Pradesh) at a speed of 2500 revolutions per minute(RPM) in dark environment for 5min. [13]

After the TiO_2 nanocomposite and nano primer preparation, the samples were a scanning electron microscope (SEM) examination(Andhra University, Visakhapatnam, Andhra Pradesh) was performed to check the uniform distribution of nanoparticles within the composite and the primer. (Fig 1)

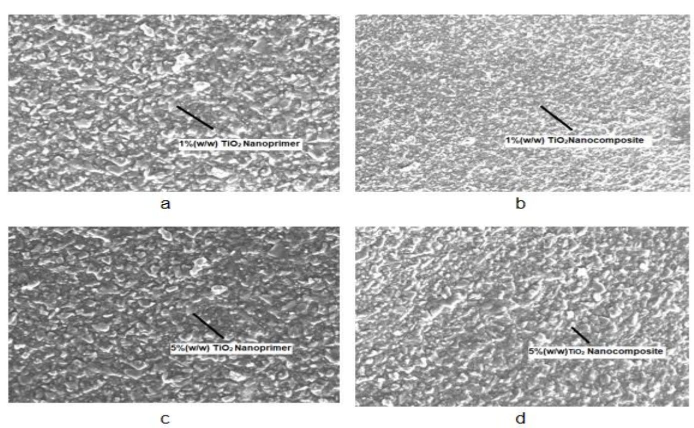


Fig 1: SEM images for uniform distribution of NPs in primer (a,c) and composite resin (b,d).

Preparation of samples for evaluation of shear bond strength

Sixty freshly extracted premolar teeth for orthodontic treatment purpose. The inclusion criteria for tooth selection were anatomically and morphologically well defined extracted premolars with intact buccal enamel surface. The teeth excluded from the study are with any developmental defects, Enamel caries, or Fractured crowns. Then the samples were mounted in cold-cure acrylic resin poured in polyvinyl chloride(PVC) tubes. The teeth were embedded in acrylic vertically up to the Cemento enamel junction.

Samples distribution

After preparing the 1%(by weight) and 5%(by weight) orthodontic bonding system containing TiO_2 nanoparticles, the experimental bonding systems were used to bond the brackets to the enamel surface teeth. The groups and subgroups of the study are tabulated as follows (Table 2, 3)

Table 2: Frequency distribution of samples in 1%(w/w) TiO_2 NPs

Group I: 1%(w/w) TiO_2 NPs		
Sub groups	TiO_2 NPs in bonding system used for bonding	n
Group Ia (1%TiO_2 nanocomposite)	composite	10
Group Ib(1% TiO_2 nanoprimer)	primer	10
Group Ic(combination of both Ia, Ib)	Both (composite + primer)	10

Table 3: Frequency distribution of samples in 5%(w/w) TiO2 NPs

Group II: 5%(w/w) TiO_2 NPs		
Sub groups	TiO_2 NPs in bonding system used for bonding	n
Group IIa (5% TiO_2 nanocomposite)	composite	10
Group IIb (5% Ag nanoprimer)	primer	10
Group IIc (combination of both IIa, IIb)	Both (composite + primer)	10

Bonding procedure

Bonding with nanocomposite: The buccal surface of the tooth was etched using 37% phosphoric acid (Eazetech, Anabond) for 30 secs, rinsed thoroughly with running water for 30 seconds, and gently dried with air spray then applied a thin coat of primer (Orthosolo, Ormco, CA) with applicator tip and light-cured for 10 secs followed by bonding of MBT 0.022" slot stainless steel premolar brackets (Ortho organizers), with TiO_2 nanocomposite.

Bonding with nano primer: after etching and rinsing the tooth surface then applied a thin coat of TiO_2 nano primer with applicator tip and light-cured for 10 secs followed by bonding of MBT 0.022" slot stainless steel premolar brackets (Ortho organizers), with conventional composite (Enlight, Ormco Crop, CA)

Bonding with both the nanocomposite and nano primer: after etching and rinsing the tooth surface, then applied a thin coat of TiO_2 nano primer with applicator tip and light-cured for 10 secs followed by bonding of MBT 0.022" slot stainless steel premolar brackets (Ortho organizers), with TiO_2 nanocomposite.

Evaluation of shear bond strength

Sixty samples from each of the 1%(by weight) and 5%(by weight) TiO_2 groups were subjected to a shear bond strength test using a Universal Testing Machine (Instron machine, model - 8801, Gitam Univesity, Visakhapatnam, Andhra Pradesh) performed testing at a crosshead speed of

1mm/minute. The force required to shear the bracket, causing the bond failure, was recorded in Mega Pascals(MPa).

Evaluation of adhesive remnant index(ARI)

The Adhesive Remnant Index (ARI) was used to assess the amount of composite resin retained on the tooth's enamel surfaces (Artun and Bergland, 1984).[15] The evaluation was done using a stereomicroscope (Zeiss, ProgRes, C3, Andhra University, Visakhapatnam, Andhra Pradesh) at ×20 magnification.

The ARI has a scale range between 0 and 3:

0 = no adhesive retained on the enamel.

1 = less than 50 percent of adhesive retained on the enamel.

2 = more than 50 percent, but less than 100 percent of adhesive retained on enamel.

3 = all adhesive retained on the enamel.

Statistical Analysis

One-way ANOVA was used to compare the mean shear bond strength among the groups and used Tukey's post hoc test for pairwise comparisons. ARI scores were analyzed using the Kruskal Wallis test. $P \leq 0.05$ was considered Significant. Using SPSS software version 20.

Results

Table 4: Comparision of mean shear bond strength(MPa) values of 1%(by weight) TiO_2 NPs in orthodontic bonding agents.

Group I: 1%(w/w) TiO_2 NPs

	N	Mean	Std. Deviation	Std. Error	95% Confidence Interval for Mean	
					Lower Bound	Upper Bound
Group Ia	10	20.2470	3.46309	1.09512	17.7697	22.7243
Group Ib	10	13.0140	4.10383	1.29775	10.0783	15.9497
Group Ic	10	9.5900	2.97809	.94175	7.4596	11.7204

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	592.039	2	296.020	23.554	.000
Within Groups	339.331	27	12.568		

One way ANOVA, $p < 0.001$ shows statistically significant

There is a statistically significant difference amongst the 1%(by weight) TiO_2 nanoparticles used in components of the orthodontic bonding system of which Group Ia (1%(w/w) TiO_2 nanocomposite) shows the highest shear bond strength followed by Group Ib (1%(w/v) TiO_2 nano primer) and Group Ic (a combination of both) used for bonding.

Table 5: Comparision of mean shear bond strength(MPa) of 5%(by weight) TiO_2 NPs in the orthodontic bonding system.

Group II: 5%(w/w) TiO_2 NPs

	N	Mean	Std. Deviation	Std. Error	95% Confidence Interval for Mean	
					Lower Bound	Upper Bound
Group IIa	10	14.8940	3.59372	1.13644	12.3232	17.4648
Group IIb	10	10.1640	2.28798	.72352	8.5273	11.8007
Group IIc	10	7.4750	1.40043	.44286	6.4732	8.4768

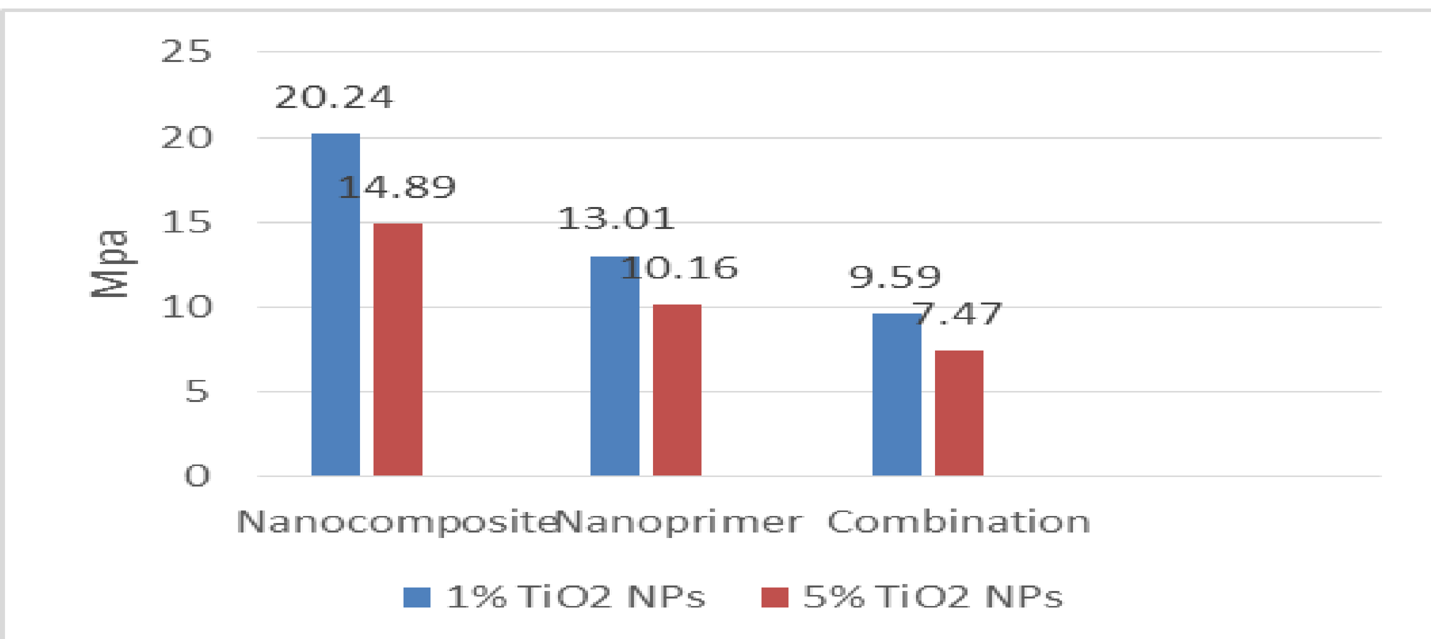
	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	282.151	2	141.075	21.045	.000
Within Groups	180.998	27	6.704		

One way ANOVA, $p < 0.001$ shows statistically significant difference

The mean shear bond strength values showed a statistically significant difference amongst 5%(by weight) TiO_2 NPs used in orthodontic composite resin and primer for bonding the brackets. In which Group IIa (5%(w/w)TiO_2 nanocomposite) showed the highest SBS followed by Group IIb (5%(w/v) TiO_2 nano primer) and Group IIc (combination of both nanocomposite and nano primer).

In the present study of the two concentrations of the TiO_2 NPs, the mean shear bond strength(Mpa) values of the 1%(by weight) TiO_2 NPs showed higher value than the 5%(by weight) TiO_2 NPs. (Graph 1)

Graph 1: The shear bond strength(Mpa) values of the tested groups



The Kruskal-Wallis test did not show any significant difference among groups in terms of the ARI (*p*= 0.81)

Table 6: ARI score of 1% (w/w)TiO_2 NPs

Group I: 1% (by weight)TiO_2 NPs ARI SCORES					
	0	1	2	3	P value
Group Ia	0	5(50%)	4(40%)	1(10%)	0.81
Group Ib	1(10%)	6(60%)	3(30%)	0	
Group Ic	3(30%)	4(40%)	3(30%)	0	

ARI score of 1%(by weight) TiO_2 nanocomposite, nano primer, and combination of both were not significantly different after debonding (*P* = 0.81)

Table 7: ARI score of 5% (by weight) TiO_2 NPs

Group II: 5%(w/w) TiO_2 NPs ARI SCORES					
	0	1	2	3	P value
Group IIa	1(10%)	6(60%)	3(30%)	0	0.81
Group IIb	1(10%)	7(70%)	2(20%)	0	
Group IIc	2(20%)	6(60%)	2(20%)	0	

ARI score of 5%(by weight) TiO_2 nanocomposite, nano primer, and combination of both were not significantly different after debonding (*P* = 0.81)

Discussion

Enamel demineralization and formation of white spot lesions during or at the end of the fixed orthodontic therapy is the most significant concern for the orthodontist as they give the unaesthetic appearance. Patient co-operation in preserving good oral hygiene is always challenging during orthodontic treatment; therefore, many clinicians prefer methods that do not rely on patient compliance.[16]

Previous studies[17, 18] have confirmed that incorporating nanoparticles into other orthodontic material has shown significant effects in terms of antimicrobial and mechanical properties. It helps convert inactive oxygen into an active one, which causes structural damage to the bacterial cell. It binds to disulfide or sulfhydryl groups present in the cell wall proteins and binds to the nucleus's DNA, causing cell death. So the present study aims to investigate such interventions on the mechanical properties of impregnated TiO_2 nanoparticles in orthodontic adhesives, orthodontic primer, and a combination of both used for bonding.

According to Reynolds et al. [19], the clinically acceptable SBS value to withstand the mechanical forces is 6-8Mpa. The results of the current study showed 1%(w/w) TiO_2 nanoparticles has highest SBS than increasing the concentration of nanoparticles to 5%(w/w) amongst the groups, TiO_2 nanocomposite showed the highest SBS followed by nano primer and combination of both used for bonding the orthodontic brackets and all the experimental groups had shown greater the clinically acceptable SBS value(6-8 Mpa).

The addition of 1%(w/w) TiO_2 nanoparticles in composite resin and primer showed a significant increase in both strengths than using both the 1% (w/w) nanocomposite and nano primer for bonding and the results of the present study was in accordance with the Ak Reddy et al.[14]

The addition of 5%(w/w) TiO_2 nanoparticles showed adequate SBS but less than the 1%(w/w) TiO_2 NPs group. According to Akhavan et al.[20] an increase in the concentration of nanoparticles decreases the SBS due to the agglomeration of nanoparticles that interfere with the curing process, and the results in accordance with the A Sodagar et al. [21]

Evaluation of the ARI scores following the orthodontic brackets' debonding is essential for verifying the amount of composite left on the enamel surfaces. The results showed that there was no significant difference between the experimental groups regarding the mode of failure. Most fractures happened at the bracket/composite interface with different amounts of material left on the enamel surface (ARI scores = 0, 1, and 2) and decreasing the possibility of enamel fracture.

Conclusion

The addition of nanoparticles into the orthodontic bonding system seems to affect the mechanical properties of experimental groups but shows higher than the clinically acceptable SBS value (6 - 8MPa).

1%(by weight) TiO_2 NPs showed higher SBS than 5%(by weight) TiO_2 NPs. Among the orthodontic bonding system components in which TiO_2 NPs are incorporated in the nanocomposite showed the highest SBS value than the nano primer and the combination groups.

Abberivations

SBS - Shear bond strength

TiO_2 - Titanium dioxide

NPs - Nanoparticles

SS - Stainless Steel

ANOVA - Analysis of variance

SPSS - Statistical Package for the Social Sciences

W/W - Weight by weight

W/V - weight by volume

RPM - Revolutions Per Minute

SEM - Scanning Electron Microscope

MPa - Mega Pascals

WSLs - White Spot Lesions

BISGMA - Bisphenol A glycidyl methacrylate

Mg - milli grams

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