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Comparison between adhesive remnant index of shear bond strength and tensile bond strength: An *in vitro* experimental study

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Abstract
Aims of the Study: The current study aims to evaluate the influence of adding zirconium oxide and Titanium dioxide nanoparticles alone or in combination at three different concentrations (0.02%, 0.04% and 0.06%) on shear and tensile bond strengths of Transbond™ XT orthodontic adhesive (3M), and to detect the site of bond failure after debonding.
Materials and Methods: One hundred freshly extracted human upper premolar teeth were used. The teeth were divided equally into two main groups, one group for the shear bond strength test and the other for the tensile bond strength test. Each group was further divided into four
Groups: The Control group, zirconium oxide nanoparticles group, Titanium dioxide nanoparticles group, and zirconium oxide nanoparticles mixed with Titanium dioxide nanoparticles group. These groups were further subdivided into three sub-groups according to the concentration of the additives. The labial surface of each tooth was cleaned with fluoride-free pumice and water, etched with 37% phosphoric acid, rinsed and dried. The Transbond™ XT orthodontic adhesive and/or the modified adhesive was placed on the bracket's mesh and bonded to the etched enamel. Universal testing
Machine (Tinius Olsen Ltd, England) was used to debond the brackets with a knife edge blade at cross head speed of 0.5 mm/min. for the shear bond strength, and 0.010 stainless steel ligature wire was used for debonding in tensile bond strength test. The bond strengths were in measured in Mega pascal, and the adhesive remnant was examined with a stereomicroscope using 10 X. Statistical analysis was done using SPSS Statistics, V21.
Results: The Shear bond strength of zirconium oxide nanoparticles mixed with Titanium dioxide nanoparticles group (0.06%) was statistically higher than the other groups. While in tensile bond strength of zirconium oxide nanoparticles mixed with Titanium dioxide nanoparticles group (0.06%) was significantly the highest.
Conclusion: Generally, the addition of ZrO2NPs and TiO_2NPs together at the studied concentrations improved the physical properties of orthodontic adhesive related to shear and tensile bond strength and reduced the bond failure rate.

Keywords: Bond strength, surface clean-up, resin removal, microetcher, debonding

Introduction

Debonding of orthodontic brackets occurs frequently when there is a problem with the orthodontic brackets bonding system, which delays treatment outcomes. These systems (and consequently, orthodontic brackets failure rates) can be affected by a number of tooth- or material-related variables; Clinical bonding failures can be attributed to other causes in 5-7% of cases [1].
Brackets for orthodontic treatment are often bonded using composite adhesive [2]. Inorganic fillers pre-treatment has been the primary focus of prior research into enhancing the characteristics of resin-based composites [3, 4]. It has been suggested that reinforcing fillers like nanofillers and fibers might be used in dental composite to boost the material's strength [5, 6].
Strengthening denture base resins by utilizing nanofillers has garnered a lot of interest recently due to the rapid advancement of Nano-phased materials and nanotechnology. This process results in a polymer nanocomposite which, compared to resins filled with micro-scale particles, possess enhanced physical and mechanical characteristics; furthermore, utilizing

several Nano fillers instead of just one allows for a higher performing composite than would be possible with adding just one nano-filler [7].

The application of nanotechnology has resulted in significant advancements in the area of orthodontics. Increasing the shear bonding strength of orthodontic materials, for example, just requires the addition of nanoparticles to the materials that are traditionally used [8].

The 3M orthodontic glue has been modified for this study by the addition of nanoparticles consisting of ZrO2 and titanium dioxide TiO2. The reason for utilizing these nanoparticles is that they possess intriguing photocatalytic, physical, and mechanical characteristics; Moreover, characteristics features of both (ZrO2:TiO2) nanostructured metal oxides were proven to be superior to adding just one. This was primarily attributed to titanium and zirconium's difference in size [9].

Nanoparticles composed of zirconium oxide are highly biocompatible and exhibit excellent aesthetic and mechanical characteristics [10].

TiO2 Nanoparticles are renowned for their chemical stability as well as photocatalytic activity in addition to their outstanding antimicrobial and mechanical capabilities. TiO2 NPs are hydrophilic because their surface contains hydroxyl groups, and they are nontoxic and cheap since titanium is the fourth most abundant metal in Earth's crust [11].

In this study, the tensile and shear bond strength (TBS and SBS, respectively) were examined to replicate the effects of various forces acting on the bonded areas, such as orthodontic forces and biting. The quantity of adhesive left after debonding on each tooth was also determined, and this information was used to calculate the Adhesive Remnant Index (ARI); ARI was used to identify the type of each failure of orthodontic adhesive.

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The amount of adhesive remained on buccal tooth surface by measuring the adhesive remnant index after orthodontic bracket de-bonding.

Materials and Methods

Failure Site and Adhesive Remnant Index

Failure site: At the time of bracket debonding after orthodontic treatment, preserving a sound, flawless enamel surface is a significant clinical concern [12]. Bond failure locations inside the bracket adhesive - enamel complex can occur within the bracket, at the bracket/adhesive contact, within the adhesive, and at the adhesive/enamel interface during debonding [12].

Bracket failure at the bracket/adhesive interface is safer and more advantageous than failure at the adhesive enamel interface because the enamel surface is left relatively intact; however, significant chair time is required to remove the residual adhesive, and the enamel surface is damaged during the cleaning process [13]. Although there is less leftover adhesive when brackets fail at the enamel/adhesive interface, enamel fracture and cracking can occur in this form of failure [14]. It is possible that the depth of etched enamel surface caused by phosphoric acid is a causative factor in the occurrence of enamel crack [12]; also, Chen *et al*. in 2008 described the sizes, locations, and incidences of enamel fracture coincided with the areas where tensile, shear, or torsion debonding force was applied and found no significant variance among these debond. After bracket debonding, there are two basic ideas: first, failure occurs at the bracket/adhesive interface (which is the most common site *in vitro* studies for both metal and ceramic brackets), leaving the adhesive resin primarily on the enamel surface [15]; second, failure occurs at the enamel/adhesive interface, meaning that there will be less adhesive on the enamel surface [1].

When brackets were given particular surface treatments such as etching [16] and bonding bases were covered with porous metal powder [17], the failure site did not represent differing bond strengths at different interfaces.

Separation at or near the bracket/resin junction has also been correlated by [18] to metal deformation. Metal deformation will lead to stress concentration and crack initiation, which progress a fracture plan.

Many factors have been identified in clinical studies as potential causes of bond failure:

1. Occlusal tension during function is a significant contributor [19].
2. A disruption in the connection during polymerization can result in resin cohesive failure [20].
3. Increased adhesive thickness leads to lower bond strength and more bond failure [21].
4. Excessive arch wire engagement force [22].
5. Inadequate enamel preparation leads to poor access and moisture contamination, particularly in the posterior teeth [23].

The Adhesive Remnant Indices

A. Scribante *et al*. (2020), used an ARI to determine how much adhesive was left on the tooth surface (Figure 1) [24]. As an example, the range is 0 to 3:

0 indicates that there is no adhesive remaining on the tooth.

1. Indicates that there is less than 50% adhesive left on the tooth.
2. More than half of the sticky is still on the tooth.
3. There is no more sticky on the tooth.

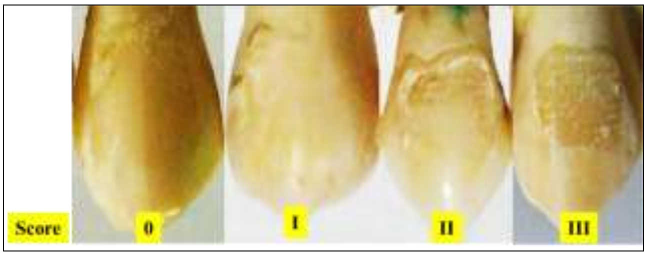

Fig 1: Scoring system for ARI.

B. By placing the teeth under a projection microscope and inspecting the enamel surface at magnification X 40, O'Brien *et al*. (1988) established a quantitative approach for determining the area of leftover adhesive as percentages of bracket base area [25]. Any adhering residues were carefully scrutinized and drawn out on high-quality tracing paper. The area under each tracing was determined and expressed as a percentage of the mean bracket base region after the tracings were digitized.

C. The modified adhesive remnant index (MARI) was developed by Bishara and Trulove (1990) [26]. Like Artun and Bergland's ARI, this is a qualitative index that takes into account how much resin material is stuck to the enamel surface when determining a final score [27]. The scores are as

follows:
Score 5 = Indicates that there was no composite left on the enamel.
Score 4 = Indicates that less than 10% of the composite was left on the enamel.
Score 3 = Indicates that more than 10% of the composite remained but less than 90%.
Score 2 = Indicates that more than 90% of the composite was retained.
Score 1: The whole composite, as well as the impression of the bracket base, remained on the tooth.
D. The following classification was proposed by Wang (1997) [28]:
Score 1 = The glue between the bracket base and the adhesive has failed.
Cohesive failure within the glue itself is a score of two.
Score 3 indicates that the glue has failed to adhere to the enamel. Enamel detachment is a score of four (Figure 2).

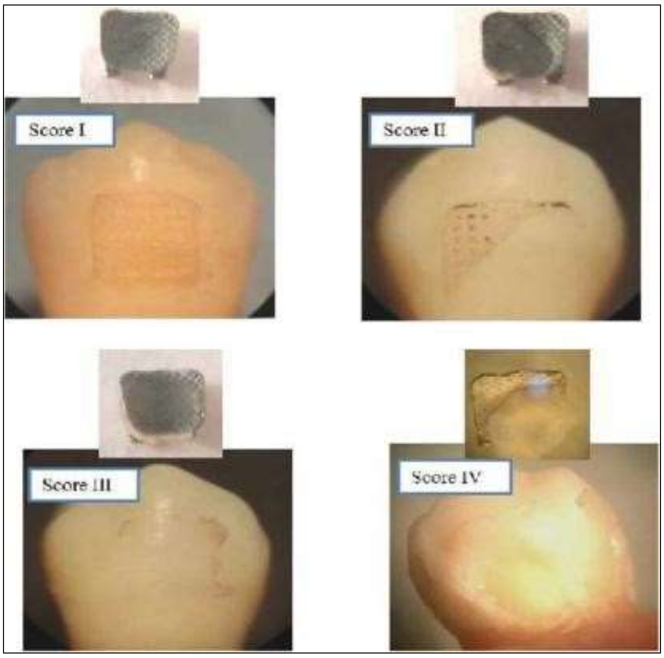

Fig 2: Adhesive remnant index scores.

E. David *et al*. (2002) developed a novel quantitative approach for determining how much residual adhesive remains on the enamel surface after debonding [29]. The following are the six score categories:
Score 1 equals 0% remaining.
Score 2 equals more than 0 percent but less than or equal to 25% remaining.
Score 3 equals greater than 25% but less than or equal to 50% remaining.
Score 4 equals greater than 50% but less than or equal to 75% remaining.
Score 5 equals more than 75% but less than 100% remaining.
Score 6 indicates that there is no adhesive left.
F. The 3D Modified Adhesive Remnant Index was developed by AL-Shamsi *et al*. (2007) [30]. After they were removed, polyvinyl siloxane (Lightweight) was used to make moulds of the teeth, which were then cast in a die stone. A 3 D laser scanner was used to scan the fabricated models, and a modified ARI was applied to analyze the resulting pictures for signs of bond failure.

Adhesive Remnant Index (ARI)

All samples were analyzed using a stereomicroscope with a magnification of X10 to determine how much adhesive remained on the tooth and bracket surfaces after debonding and to determine whether the bond had failed cohesively, adhesively, or in a mixed cohesive-adhesive manner. These assessments were made using scores developed according to the criteria described by Artun and Bergland in 1984 to minimize scoring errors, inter and intra examiner calibrations were carried out these scores are:
Score 0: indicate no adhesive remaining on the surface of the tooth.
Score 1: indicate that the quantity of the adhesive remaining on the surface of the tooth is less than half
Score 2: indicate that the quantity of the adhesive remaining on the surface of the tooth is more than half.
Score 3: indicate that All of the adhesive remained on the surface of the tooth, with the bracket's mesh leaving a recognizable imprint on the remaining adhesive.

Normality test of ARI of SBS

The raw data of all groups were normally distributed, as seen in table (1) which showed that the significant values were greater than (0.05) According to Kolmogorov-Smirnov tests.

Table 1. Normality test of ARI of TBS

Group	Kolmogorov-Smirnov		
	Statistic	df	Sig.
Control	0.329	4	0.406
ZrO_2NPs (0.02%)	0.341	4	0.401
ZrO2NPs (0.04%)	0.283	4	0.272
ZrO2NPs (0.06%)	0.298	4	0.224
TiO2NPs (0.02%)	0.298	4	0.224
TiO2NPs (0.04%)	0.298	4	0.224
TiO2NPs (0.06%)	0.298	4	0.224
ZrO2 TiO2NPs (0.02%)	0.303	4	0.086
ZrO2 TiO2NPs (0.04%)	0.298	4	0.224
ZrO2 TiO2NPs (0.06%)	0.283	4	0.272

ARI of SBS groups descriptive analysis

Table (2 and 3) demonstrate the descriptive data of the ARI for SBS. The data contain each group's sample numbers, standard deviation, mean, range, standard error, and all the study groups ARI minimum and maximum values. According to the descriptive data, the highest ARI mean scores belonged to the control group followed by ZrO_2NPs (0.02%), ZrO_2NPs (0.04%), ZrO_2TiO_2NPs (0.06%), ZrO_2NPs (0.06%), TiO_2NPs (0.02%), TiO_2NPs (0.04%) then ZrO_2TiO_2NPs (0.04%) groups. the lowest ARI mean scores belonged to the groups TiO_2NPs (0.06%) and ZrO_2TiO_2NPs (0.02%).

Table 2: ARI scores frequency distribution of the SBS across the study groups.

Groups	ARI Scores			
	0*	1*	2*	3*
Control	1	1	3	0
ZrO_2NPs (0.02%)	2	1	1	1
ZrO_2NPs (0.04%)	2	2	1	0
ZrO_2NPs (0.06%)	2	3	0	0
TiO_2NPs (0.02%)	2	3	0	0
TiO_2NPs (0.04%)	2	3	0	0
TiO_2NPs (0.06%)	3	2	0	0
ZrO2TiO2NPs (0.02%)	4	0	1	0
ZrO_2TiO_2NPs (0.04%)	2	3	0	0
ZrO_2TiO_2NPs (0.06%)	2	2	1	0

0*: sample numbers with an ARI score of 0,
1*: sample numbers with an ARI score of 1,
2*: sample numbers with an ARI score of 2,
3*: sample numbers with an ARI score of 3,
TiO_2NPs is titanium dioxide nanoparticles, and
ZrO_2NPs is Zirconium Oxide nanoparticles.

Table 3: The SBS study groups' ARI Descriptive statistics.

Groups	N	Min.	Max.	Mean	Std. Dev.
Control	5	0	3	1.25	1.258
ZrO2NPs (0.02%)	5	1	2	1.25	0.5
ZrO2NPs (0.04%)	5	0	2	1.25	0.957
ZrO2NPs (0.06%)	5	0	3	1.25	1.5
TiO2NPs (0.02%)	5	0	3	1.25	1.5
TiO2NPs (0.04%)	5	0	3	1.25	1.5
TiO2NPs (0.06%)	5	0	3	1.25	1.5
ZrO2 TiO2NPs (0.02%)	5	0	4	1.25	1.892
ZrO2 TiO2NPs (0.04%)	5	0	3	1.25	1.5
ZrO2 TiO2NPs (0.06%)	5	0	2	1.25	0.957

N is number, TiO2NPs is titanium dioxide nanoparticles, ZrO_2NPs is Zirconium Oxide nanoparticles.

Normality test of ARI of TBS

The raw data of all groups were normally distributed, as seen in table (4) which showed that the significant values were greater than (0.05) According to Kolmogorov-Smirnov tests.

Table 4: normality test of ARI of SBS

Tests of Normality	Kolmogorov-Smirnov		
Group	Statistic	Df	p value
Control	0.329	4	0.406
ZrO2NPs (0.02%)	0.329	4	0.406
ZrO2NPs (0.04%)	0.329	4	0.406
ZrO2NPs (0.06%)	0.283	4	0.272
TiO2NPs (0.02%)	0.341	4	0.401
TiO2NPs (0.04%)	0.341	4	0.401
TiO2NPs (0.06%)	0.329	4	0.406
ZrO2 TiO2NPs (0.02%)	0.329	4	0.406
ZrO2 TiO2NPs (0.04%)	0.298	4	0.224
ZrO2 TiO2NPs (0.06%)	0.329	4	0.406

Table (5 and 6) demonstrate The Descriptive data of the ARI for SBS. The data contain each group's sample numbers, standard deviation, mean, range, standard error, and all the study groups ARI minimum and maximum values. These data revealed that ZrO_2TiO_2NPs (0.04%) had the highest ARI mean scores followed by C group, ZrO_2NPs (0.04%), ZrO_2TiO_2NPs (0.02%), ZrO_2TiO_2NPs (0.06%), TiO_2NPs (0.06%). While TiO_2NPs (0.02%), ZrO_2NPs (0.02%), TiO_2NPs (0.04%) groups had the lowermost mean scores of ARI.

Table 5: ARI scores frequency distribution of the TBS across the study groups

Groups	ARI Scores			
	0*	1*	2*	3*
Control	0	1	1	3
ZrO_2NPs (0.02%)	0	1	1	3
ZrO_2NPs (0.04%)	0	1	1	3
ZrO_2NPs (0.06%)	1	0	2	2
TiO_2NPs (0.02%)	1	1	1	2
TiO_2NPs (0.04%)	1	1	1	2
TiO_2NPs (0.06s%)	1	0	1	3
ZrO_2TiO_2NPs (0.02%)	0	1	1	3
ZrO_2TiO_2NPs (0.04%)	0	0	2	3
ZrO_2TiO_2NPs (0.06%)	0	1	1	3

0*: sample numbers with an ARI score of 0,
1*: sample numbers with an ARI score of 1,
2*: sample numbers with an ARI score of 2,
3*: sample numbers with an ARI score of 3, TiO_2NPs is titanium dioxide nanoparticles, and ZrO_2NPs is Zirconium Oxide nanoparticles.

Table 6: The TBS study groups' ARI Descriptive statistics.

Groups	N	Min.	Max.	Mean	Std. Dev.
Control	5	0	3	1.25	1.258
ZrO2NPs (0.02%)	5	0	3	1.25	1.258
ZrO2NPs (0.04%)	5	0	3	1.25	1.258
ZrO2NPs (0.06%)	5	0	2	1.25	0.957
TiO2NPs (0.02%)	5	1	2	1.25	0.5
TiO2NPs (0.04%)	5	1	2	1.25	0.5
TiO2NPs (0.06%)	5	0	3	1.25	1.258
ZrO2 TiO2NPs (0.02%)	5	0	3	1.25	1.258
ZrO2 TiO2NPs (0.04%)	5	0	3	1.25	1.5
ZrO2 TiO2NPs (0.06%)	5	0	3	1.25	1.258

Additionally, the descriptive data revealed that the highest ARI mean scores belonged to the ZrO_2TiO_2NPs (0.04%) groups followed by ZrO_2NPs (0.04%), ZrO_2TiO_2NPs (0.02%), ZrO_2TiO_2NPs (0.06%), C group, TiO_2NPs (0.06%), ZrO_2NPs (0.06%), then While TiO_2NPs (0.02%), TiO_2NPs (0.04%), ZrO_2NPs (0.02%), and groups possessed the lowermost mean scores of ARI.

Discussion

The Adhesive Remnant Index (ARI) of SBS and TBS: The SBS groups had varying ARI scores (0-1). In a shear test, less than half of the adhesive persisted on the surface of the tooth following the bracket debonding, as shown in Table (4.7). Forty percent of the bond failure occurred cohesively inside the adhesive itself, and Forty-four percent of bond failure occurred at the enamel/adhesive interface. While the ARI scores for the TBS groups was predominately scores (3), as seen in Table (4.9). In which 54% of the samples were failed at the adhesive/bracket interface, means that more than half of the adhesive remained on the tooth surface after bracket debonding in tensile test.

This agreed with Kechagia *et al*. (2015), who reported that the adhesive/bracket interface was the most common site of failure in tensile test specimens with high ARI scores [31]. This is because the adhesive/bracket interface is more resistive to shearing/compression force than tensile/tearing load, and the stress or load distribution over the specimens was varied between the two types of testing ("different machine-sample alignment with distinct debonding techniques").

Nevertheless, obtaining an ARI score of 0–1 in SBS indicates successful polymerization in the area just below the bracket, which would be indirectly cured (since the curing light can't go through the bracket) by the light reflected from the enamel surface; this is corroborated by Mirzakouchaki *et al*. (2016) [32]. Despite the inclusion of the nanofillers in orthodontic adhesive, a mixed failure mode was seen in this investigation, with cohesive failures occurring inside the composite resin. This suggests that the degree of conversion of the monomer to polymer was sufficient, leading to a more homogenous polymerization and this is corroborated by Dimitriadi *et al*. (2021) [33].

While Ahmadi *et al*. (2020), reported that high ARI scores are linked to high SBS mean values, the present study contradicted this association by finding that highly significant SBS mean values were linked to low ARI scores (0-1) [34].

It may be preferable to have a low ARI in SBS (between 0 and 1) since it reduces the amount of adhesive residue left on the tooth surface after the brackets are debonded and the likelihood of iatrogenic harm to the teeth caused by the orthodontist during cleaning [35]. An additional benefit of low ARI scores is that rebonding on a previously bonded tooth surface following bracket bond failure takes much less time since there is less adhesive residual on the tooth surface. This agreed with Secilmis *et al*. (2013), who also discovered that the majority of specimens had ARI scores of (1-0) [36], but it was at odds with Yang *et al*. (2002), who discovered that having a high ARI of (3) "failure at the adhesive/bracket interface" was advantageous since it lowers the likelihood of enamel fracture during debonding forces [37].

Conclusion

The best SBS value was obtained by combining ZrO2NPs and TiO2NPs, particularly at a concentration of (0.06%). Whereas the control group's SBS values were low.

The best TBS value was obtained by combining ZrO2NPs and TiO2NPs, particularly at a concentration of (0.06%). Whereas the control group's SBS values were low.

Since SBS is strongly influenced by the geometry of the sample geometry and the topography of the surface, the findings pertaining to SBS are inconsistent among any given group's samples. This renders it challenging to predict the average failure loads within a given set of samples.

The resulting TBS readings are more foreseeable, and the failure load in the subsequent sample within the same group can be estimated with considerable certainty.

No changes in the 3M's Orthodontic Adhesive chemical structure were seen after introducing low concentrations of ZrO2NPs and TiO2NPs (0.02%, 0.04%, and 0.06%) to the adhesive.

Overall, the physical characteristics of orthodontic adhesive in relation to SBS and TBS were enhanced by the addition of ZrO2NPs and TiO2NPs at the tested concentrations, and the bond failure rate was decreased. This enhancement was less significant when only one of those NPs were added to the adhesive.

An essential aspect to consider is the particular concentrations of additional NPs. To maximize their beneficial effect on orthodontic adhesive's SBS and TBS, reducing the added NPs concentration is favourable

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