Shear and Tensile Bond Strengthsof Titanium Dioxide Nanoparticles Modified Orthodontic Adhesive

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ABSTRACT

Aims: to evaluate the shear bond strength and the tensile bond strength of Heliosit orthodontic adhesive system containing Titanium Dioxide nanoparticles.

Material and Methods: Forty freshly extracted human premolar teeth were used, (20) of them were embedded longitudinally into Poly Vinylchloride tubes with chemically-cured acrylic resin to the cemento-enamel junction for shear bond strength test, while the other (20)were cut at the cemento-enamel junction and the lingual aspect of the crown of the teeth were embedded into the cold cured acrylic. The labial surfaces were cleaned with pumice and water. The teeth were distributedrandomly into 4groups of 5 teeth each. The Control group, Titanium dioxide nanoparticles (0.02%) group, Titanium dioxide nanoparticles (0.04%) group and Titanium dioxide nanoparticles (0.08%) group.After acid etching with 37% phosphoric acid the modified adhesive and/or the Heliosit orthodontic adhesive placed on the bracket's mesh and bonded to etched enamel. Then test specimens were stored in distilled water at 37 ^{0}C for 24 hrs. Universal testing machine was used to debond the brackets with a knife edge blade at cross head speed 0.5mm/min. for the shear bond strength. A stainless steel ligature wire was used for debonding in tensile bond strengthtest.The bond strengths were in megapascaland statistical analysis was done using SPSS 19.

Results: The Shear bond strength of the Control group was statistically higher than the modified adhesives with Titanium dioxide nanoparticles. While in tensile bond strength, the modified adhesive group Titanium dioxide nanoparticles (0.08%) was statistically higher than the Control group.

Conclusion: addition of Titanium Dioxide nanoparticles to orthodontic adhesive improves the tensile bond strength while the shear bond strength remained higher than the minimally accepted range of the shear bond strength.

Key words: Titanium dioxide nanoparticles, shear bond strength, tensile bond strength, Metal brackets.

INTRODUCTION

Bond failure is a common unsatisfying problem occurs in a percentage of 0.5 – 17.6 in routine orthodontic treatment. Many reasons are responsible for failure in bonding procedure, such as stumpy retentiveness of certain bracket bases, reduced bracket base size, masticatory forces, and unusual patient's habits and sever mal teeth alignment which might interfere with final bracket seating. Therefore, it was important to enhance this particular part in order to eliminate its main drawbacks like delaying the treatment time, increases the cost, reduces the patient's cooperation and eventually lessens the confidence zone between the parents – patient and the orthodontist [1,2].

The main concern about bond strength testing is the lack of standardization, no specifications (International Organization for Standardization, American National Standard Institute/ American Dental Association) currently exist to standardize the test protocol for orthodontic bond strength assessments, and the large distribution of results often inhibit confident conclusions from being drawn [3].

For orthodontic treatment, clinical bonding could be considered successful if the SBS was between 5.9 – 7.8 MPa according to Reynolds. Fox *et al* [4]suggested a minimum required SBS to withstand both orthodontic and masticatory forces between 6 - 8 MPa. In general the maximum bond strength should be less than the tensile strength of enamel, which ranges between 11 and 25 MPa depending on the prismatic orientation [5, 6].

Nanodentistry, is the science and technology of nourishing near perfect oral health through the use of nanomaterials including tissue engineering and nanorobotics. Recently defined as the "science and technology of diagnosing, treating and preventing oral and dental diseases, relieving pain, preserving and improving dental health using nanostructured materials"[7,8].

Nanocomposites are a new set of materials that contain nanofillers. Due to the diminished dimension of the particles and a wide size distribution, the increased filler reduces polymerization shrinkage, enhancing the mechanical properties such as shear, tensile and compressive strength and increase the fracture resistance .The advantages of nanocomposite materials includeeasy handling characteristics , excellent optical properties, and superior refinementalso, decrease surface roughness of orthodontic adhesives, which is one of the greatest significant factors for bacterial adhesion[9].

Antimicrobial properties of TiO_2NPs have been widely recognized and therefore they have been applied in medicine, dentistry and other science. Many studies demonstrated the antimicrobial effect of TiO_2NPs [10].

Sodagar*et al.*[11], found that bacterial biofilm inhibition in composite containing TiO_2NPs is significantly greater than conventional composite resins, and such an effect increased as the percentage of NPs in the composites increased in such a way that the composite containing more than 5% NPs significantly reduced *S. mutans* and *S. sanguinis*. However, this had no effect on biofilm inhibition of L. acidophilus, which help the development of caries and are found in more advanced lesions.

TiO_2NPs have been used as an additive in dental materials to match the opaque properties of teeth, but using TiO_2NPs as additives to enhance the mechanical properties of dental resins, has not been successful due to the inconsistent agglomeration of TiO_2NPs [12,13].

Researchers have used TiO_2NPs as filler in epoxy, in order to overcome the disadvantages of commercial tougheners such as glass and rubber beads by improving the strength, stiffness and toughness of the epoxy without compromising its thermo-mechanical properties. TiO_2NPs addition to dental composites also augment the mechanical properties, including elastic modulus, micro hardness and flexural strength, and provided bond strength values that were equal or even higher than that of the nano particle free controls [14,15].

Felemban and Ebrahim[16], in their study concluded that the addition of TiO_2–ZrO_2NPs to resin-based adhesives increased the compressive, tensile, and shear bond strengths of the adhesive in vitro, furthermore, the use of multiple Nano fillers rather than a single additive develops a high performance composite that cannot be achieved by using a single filler.

The aim of this study was to evaluate the effect of addingTitanium dioxide nanoparticles on shear and tensile bond strengths of orthodontic adhesive.

MATERIALS AND METHODS

Sample Collection and Inclusion Criteria

The study samples are extracted human upper and lower premolar teeth, with normal size, shape and are recently extracted for orthodontic treatment purposes. They have been selected from a group of Iraqi patients in three different Iraqi cities (Mousl, Erbil and Duhok), who were attending the private clinics or other specialized dental centers. The inclusion criteria were sound teeth which have been examined for intact buccal tooth surface, with no previous bonding, no caries, cracks, fractures and no restorations, not subjected to any kind of orthodontic treatment or chemical agents like alcohol, formalin or hydrogen peroxide H_2O_2. A sample of 40 permanent premolars were stored in Thymol solution (0.1%) at room temperature for no longer than six months.

Preparation of the Modified Adhesive

Three different concentrations 0.02%, 0.04% and 0.08% were chosen of the modified orthodontic adhesive with TiO_2NPs weight to weight ratio with the aid of electrical sensitive balance (AZZOTA, USA). A sterile mixing glass slap has been used to weight the specific amount of Heliosit Orthodontic Adhesive (Light-curing, highly translucent single component bonding material for brackets in orthodontia. The monomer matrix consists of urethane dimethacrylate, Bis-GMA and decandiol dimethacrylate (85%). The filler consists of highly dispersed silicon dioxide (14%). Additional contents are catalysts and stabilizers (1%).Ivoclar Vivadent, Liechtenstein, Germany) and the TiO_2NPs amount to produce the desired concentra$_{tio}$n. Manual mixing was done with mixing spatula on the glass slap for approximately 60 secs in a semi dark environment till a uniform consistency was achieved [11], which is then transferred to a sterile disposable syringe, then covered with dark color tape to reduce the direct light exposure.

The Sample Preparation

A plastic Poly Vinylchloride (PVC) rings were used with 20 mm outside diameter, 18 mm inside diameter and 30 mm height. Regarding the SBS samples, the rings were filled with dental stone to approximately half of its height and after setting of the stone a sticky wax was used to fix the tooth apex on the stone surface with appropriate orientation of the long axis of the tooth so that the buccal portion of the tooth sample is parallel to a flat surface, which represent the direction of force application during shear bond strength test[17].Then filled the PVC rings with auto polymerizing cold cure acrylic resin to the level of the cemento-enamel junction (CEJ). Regarding the TBS samples before embedding in cold cure acrylic resin the tooth sample was sectioned at the CEJ by high speed hand piece with diamond fissure bur and copious water to reduce heat generation during cutting. After pouring of the auto polymerizing acrylic resin and before complete setting, the lingual portion of each sectioned tooth was embedded into the acrylic resin so that only the buccal portion is facing upward to receive the bracket later.

Bonding Procedure

The samples were polished with fluoride free pumice and a rubber prophylactic cups on a low speed hand piece for approximately 10 secs as seen in (Figure 1), rinsing with water spray and drying with oil free air for 30 secs. The buccal surface of each tooth was etched with a 37% phosphoric acid gel for 60 secs rinsed with water for 10 secs and subsequently dried by triple syringe till a chalky appearance was observed. The whole sample was positioned on the articulator by using a prefabricated base. The bracket (Stainless Steel Metallic Brackets, Standard Edgewise type, with 9 mm^2 bracket base surface area, Dentaurum, Germany) was hold by clamping tweezers and the resinous material (Heliosit orthodontic adhesive or the modified adhesives) was applied on the base of the bracket then transferred to the center of the buccal surface of the crown of the premolar tooth at a distance of 4 - 4.5 mm from the occlusal surface. Boons gauge (Dentaurum, Germany) was used to ensure correct bracket position (Figure 2). A load of 200 gm was directed at a right angle to the bracket slot to produce uniform thickness of the resinous material and to prevent air voids entrapment [18]. The excess resin was removed then curing started using LED light curing device with wave length of (420 – 480) and illumination of (1200 – 1500) mw/cm^2. Curing radiometer was used for calibrationfor each 5 samples.

Figure 1: Specimen polishing.

Figure 2: Bracket positioning

The curing light was applied for 20 secs for mesial side and 20 secs for the distal side, the tip of thecuring device is at a distance of 2mm from the mesial and distal edges of the bracket [19]. The specimens then were stored in container filled with distilled water and placed in an incubator at 37^0 Cfor 24 hrs prior to testing.

Measuring Shear Bond Strength

The SBS test was measured by using the Universal testing machine(SANS, China) with a crosshead speed of 0.5mm/min. A knife edge blade directed toward the tooth – bracket interface in an occluso-gingival direction (Figure 3). The necessary load to debond or initiate bracket failure was recorded in Newton unit and converted to Megapascal (MPa) unit by dividing the failure load in Newton unit to the surface area of the bonded bracket base.

Figure 3: The SBS testing in Universal testing machine with a knife edged blade

Measuring Tensile Bond Strength

The TBS test was measured by using the same Universal testing machine with a crosshead speed of 0.3mm/min, debonding was done using 0.010 S.S. ligature wire (Figure 4). The direction of force application was perpendicularly away from the buccal tooth surface, the force necessary to debond or initiate bracket failure was recorded in Newton unit and converted to Megapascal (MPa) unit with the same equation used for the SBS test.

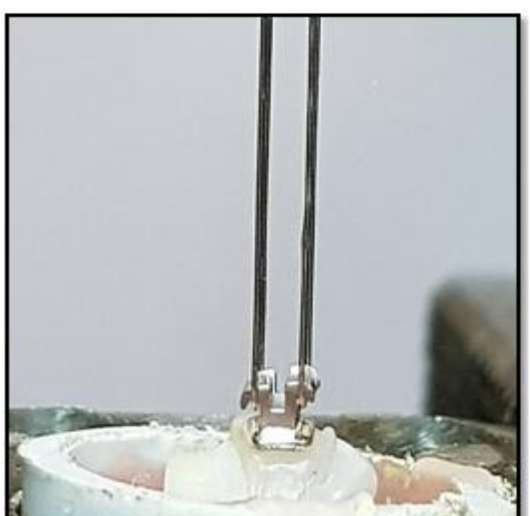


Figure 4: The TBS test via S.S. ligature wire of 0.010 gauge.

Transmission Electron Microscope (TEM)

TEM (Philips CM10, Germany) images were taken for the TiO_2NPs alone to check for the nanoparticle size and shape at different magnification power, the test was done in the Baghdad city at Al-Nahrain University.

Fourier Transform Infrared Spectrometry (FTIR)

The chemical characteristics of the modified Adhesive with TiO_2NPs were determined by the use of FTIR device (Alpha, Bruker Company, Germany), the test involves placing a small amount (drop like) of the modified adhesives of each concentration over a sterile mixing slap then initiated polymerization, after that the cured sample was placed over the FTIR table, (Figure 5), and pressed with the FTIR specialized lens holder.

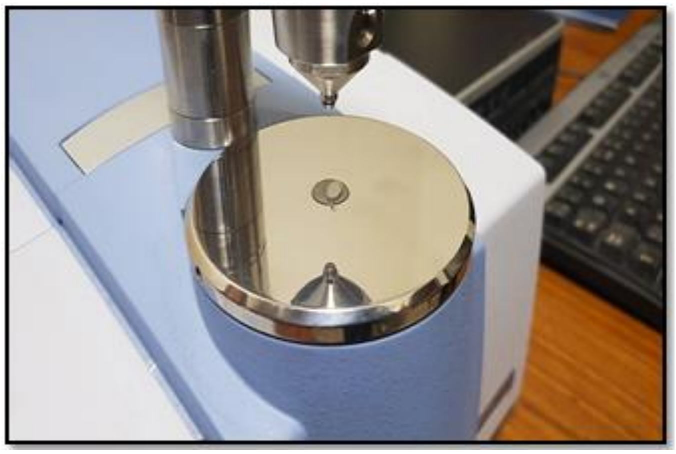
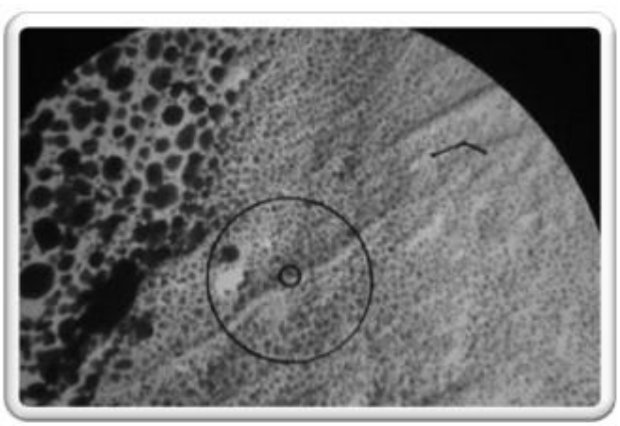


Figure 5: Sample placement on FTIR testing table and lens.

RESULTS

The TEM images for TiO_2NPs powder at a magnification power (x92000, x130000) revealed that the particles are spherical or semi oval in shape, and there were a mixture of nanoparticles with a different size ranging from (15 – 25 nm), with high tendency for agglomeration.



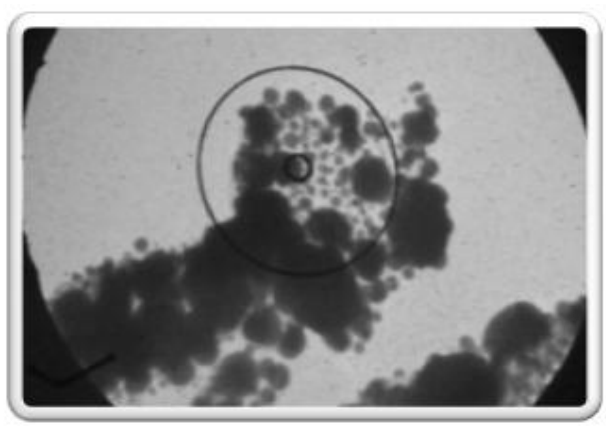


Figure 6: TEM image of TiO_2NPs at x92000 **Figure 7: TEM image of TiO_2NPs at x130000**

Fourier Transform Infrared Spectrometry (FTIR) Charts

The FTIR spectra of the Control showed some bands at 2931, 1711 and 1389 which are due to C-H stretching, C=O and C=C [20], when comparing them with the FTIR spectra of the modified adhesive with nanoparticles, (figures 8, 9 and 10), it was showed that these bands are at the same region of the Control spectra with no any shifting of the bands (no appearance of new bands nor disappearing bands), (Figures 8,9and 10).

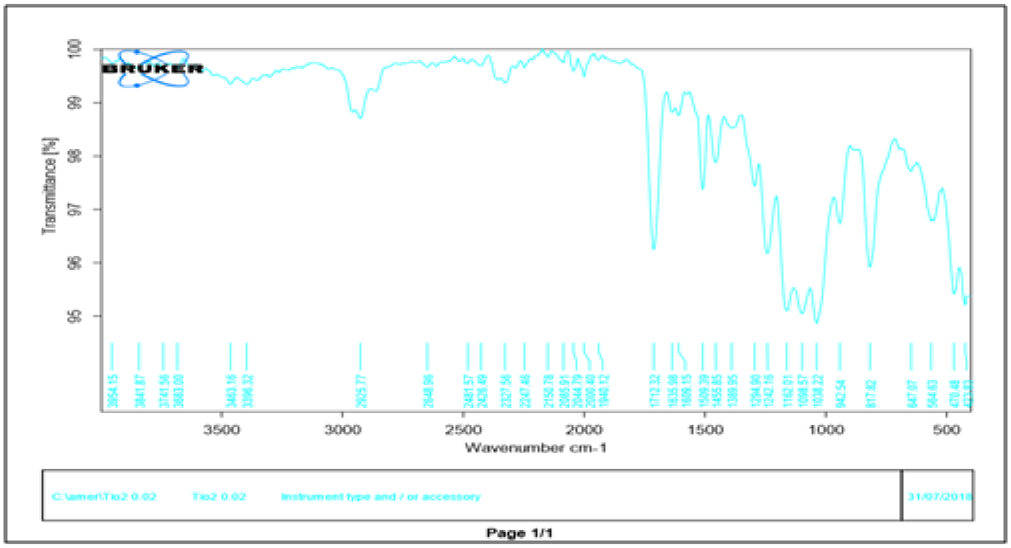


Figure 8: FTIR spectra of the modified adhesive with TiO_2 (0.02%).

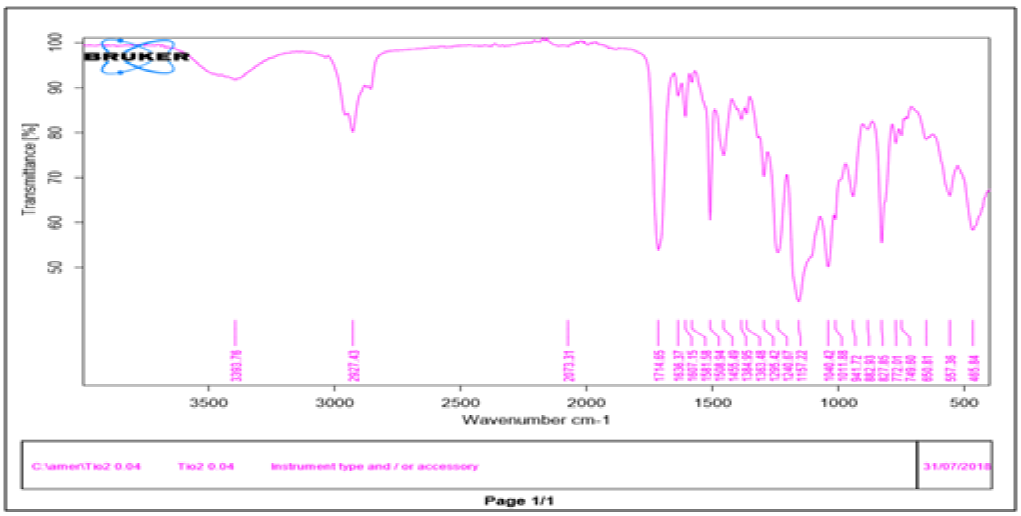


Figure 9: FTIR spectra of the modified adhesive with TiO_2 (0.04%).

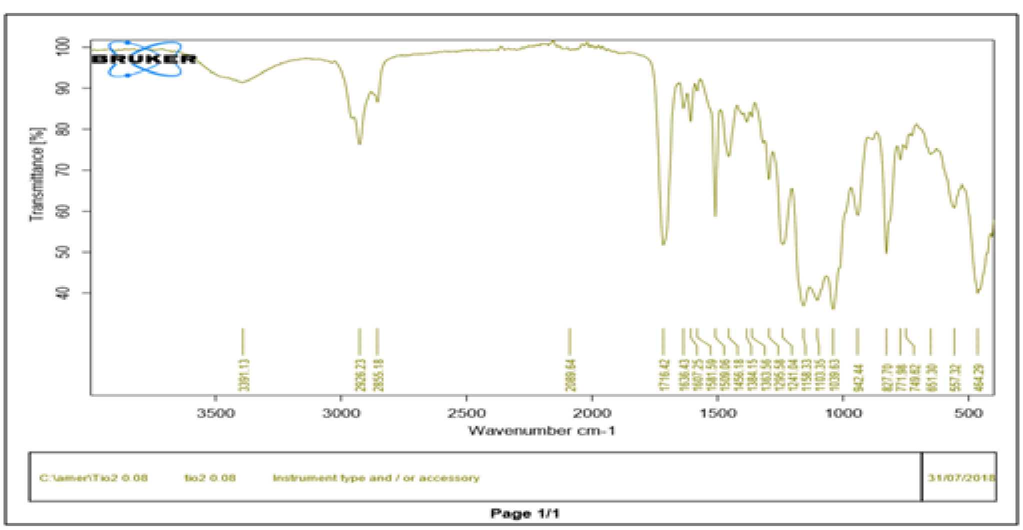


Figure 10: FTIR spectra of the modified adhesive with TiO_2 (0.08%).

Descriptive Analysis of Shear Bond Strength

The Descriptive data for SBS is demonstrated in (Figure 11). These data include the number of the samples in each group, range, mean, standard deviation, standard error, minimum and maximum values of the SBS for all the study groups. The descriptive analysis revealed that the **C** group showed the highest mean values of the SBS among the groups, followed by **TiO_2 (0.08%)**group**, TiO_2(0.02%)**group then **TiO_2 (0.04%)**group.

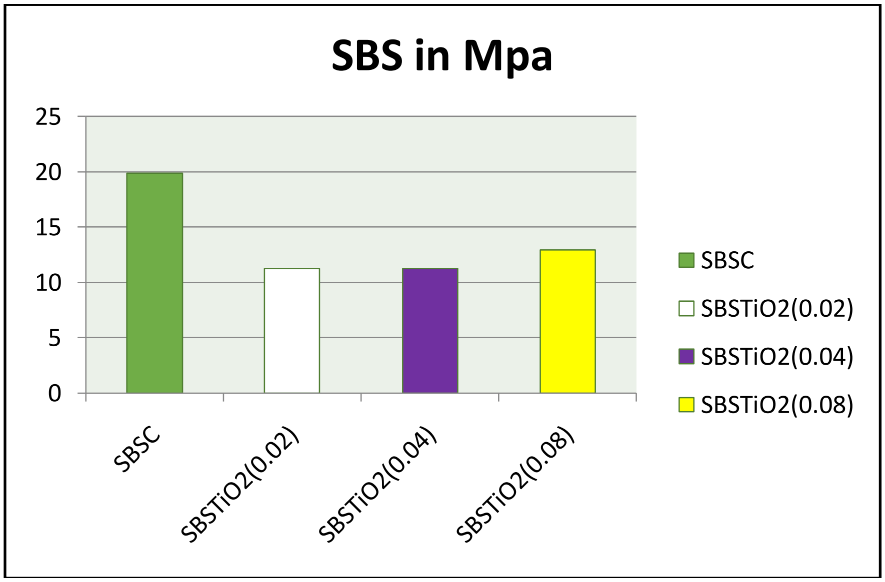


Figure 11: Bar diagram showing the mean values of the SBS among groups of study.

Analysis of Variance (ANOVA) of Shear Bond Strength

The result obtained from one way (ANOVA) statistical test is demonstrated in (Table 1) showed a significant difference at (***P*** **≤ 0.05**) among the mean values of the SBS for the different groups in the study.

Table 1: One way (ANOVA) for the mean values of SBS among the study groups.

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	250.906	3	83.635	21.947	0.000
Within Groups	60.973	16	3.811		
Total	311.879	19			

dfis degree of freedom **, F**is **F** test, **Sig**. is significant, Significant level is at (**P ≤ 0.05).**

A more specific Duncan's Multiple Range Test was conducted between the study groups as demonstrated in (Table 2), and revealed that **TiO_2 (0.04)** group had significantly the highest difference in SBS value, while the **C** group had significantly the lowest difference in SBS value

Table 2: Multiple Comparisons of the shear bond strength among the study groups using Duncan's Multiple Range Test.

Groups	N	Mean+ SE	Duncan Groups*
SBSC	5	19.84+1.39	D
SBSTiO_2 (0.02)	5	11.27+0.69	B
SBSTiO_2(0.04)	5	11.24+0.74	A
SBSTiO_2(0.08)	5	12.95+0.24	C

Nis number, **SE** is Standard error,***** Different litters mean significant difference (***P*** **≤ 0.05),SBS**is shear bond strength, Variable unit for **SBS** is in **MPa**,**C** is control group, **TiO_2** is titanium dioxide nanoparticles.

Descriptive Analysis of Tensile Bond Strength

The Descriptive data is demonstrated in (Figure 12). These data include the number of the samples in each group, range, mean, standard deviation, standard error, minimum and maximum values of the TBS for all the study groups. The analysis revealed that the**TiO_2 (0.08%)** group showed the highest mean values of the TBS between the groups, followed by **C**group**, TiO_2 (0.04%)** group then **TiO_2 (0.02%)** group.

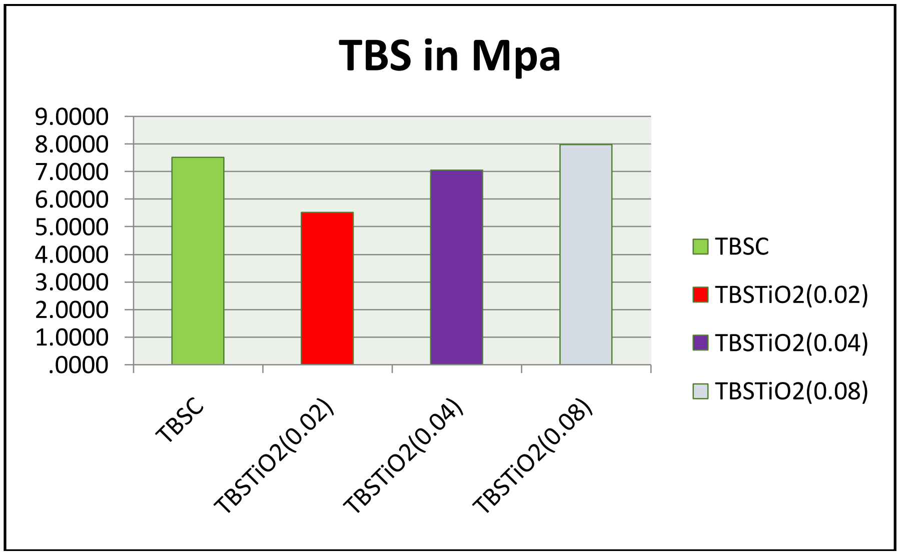


Figure 12: Bar diagram showing the mean values of the SBS among groups of study.

Analysis of Variance (ANOVA) of Shear Bond Strength

The result obtained from one way (ANOVA) statistical test is demonstrated in (Table 3) showed a significant difference at ($P \leq 0.05$) among the mean values of the SBS for the different groups in the study.

Table 3: ANOVA for the tensile bond strength for the study groups.

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	16.921	3	5.640	10.051	0.001
Within Groups	8.979	16	0.561		
Total	25.899	19			

df is degree of freedom ,**F**is **F** test, **Sig.** is significant, Significant level is at **($P \leq 0.05$).**

A more specific Duncan's Multiple Range Test was conducted between all the study groups as demonstrated in (Table 4), and revealed that **TiO_2 (0.02)** grouphad significantly the highest difference in TBS mean value, while the **C** group had the lowest difference in TBS mean value in comparison to all study groups.

Table 4: Multiple comparisons of the tensile bond strength among the study groups using Duncan's Multiple Range Test.

Groups	N	Mean+ SE	Duncan Groups*
TBSC	5	7.51+0.19	C
TBSTiO_2(0.02)	5	5.52+0.42	A
TBSTiO_2(0.04)	5	7.06+0.41	B
TBSTiO_2(0.08)	5	7.97+0.24	D

Nis number, **SE** is Standard error,* Different litters mean significant difference **($P \leq 0.05$), TBS** is tensile bond strength, Variable unit for **TBS** is in **MPa,C** is control group, TiO_2 is titanium dioxide nanoparticles.

DISCUSSION

Bond strength testing can be influenced by many factors, the tooth type, the presence of tracing elements like fluoride, smoothness and flatness of the tooth surface, presence of tooth convexity, tooth extraction time and storage, type of acid etching used, its concentration and time of etching, type of bonding agent used and its filler content, type of light curing device with its different intensities and time of curing, type of bracket material and its base configuration and base surface area, presence or absence of thermocycling, type of bond strength test performed (SBS, TBS, Microshear, microtensile, compression), cross head speed and the amount of the load applied. Therefore these kind of tests are so difficult to be standardized and draw a consistent results from them, which is in accordance to many authors and review studies [21, 22].

TiO_2NPs was chosen due to, low toxicity, white color, availability, low price, efficient photocatalytic action, chemical stability and its pleasing color which match the opaque characterization of teeth, also can provide better mechanical properties like micro hardness, modulus of elasticity, flexural strength and can increase the tensile and tear strength and percent elongation [23,24].

The reason for selecting such low concentrations is based on the main concept of nanoparticles,

It's important to add nanoparticles in low percentage, so that to maintain the adhesive flowability and low viscosity [17].

TiO_2NPs have high affinity to agglomerate with each other to form micro-sized particles especially when mixed with polymeric materials, such agglomerations act like a weak point where stress is concentrated, so that to reduce the agglomeration surface modification of TiO_2NPs is recommended by using organosilane material [25].

In the present study, the SBS mean values were higher than the TBS mean values, as it was shown in tables (1, 4). This is due to the difference in stress distribution between the two types of tests. This was in accordance to Leloup*et al.,* [26] and Arici*et al.,* [27] who found significantly higher shear bond mean values than tensile mean values.

The addition of TiO_2NPs improved the SBS mean value, although it wasn't the highest SBS between the groups but, it was above the recommended SBS mean values suggested by Reynolds[5] which was (5.9 – 7.8) MPa, and Fox *et al*., [4] which was (6 – 8) MPa. This was in agreement with Reddy *et al*., [28]and to Sodagar*et al.,*[11]. And in disagreement with Xia *et al*., [25] andSun *et al*., [29].

Studies regarding the TBS were limited, since the most common test for measuring the bond strength of orthodontic brackets is the shear test.However, there was a linear relationship between increasing the concentration and increasing the TBS. this may be due to the oxidative state of the TiO_2NPs which is (+2) which allow more attraction force between theTiO_2NPsespeciallyin tearing or elongation forces. Also some studies found that adding TiO_2NPs significantly increases the TBS as in Shirkavand and Moslehifard [30].

CONCLUSION

The addition of Titanium Dioxide nanoparticles to Heliosit Orthodontic adhesive improves the TBS while the SBS remained higher than the minimally accepted range.

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