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Department of Pedodontic orthodontics and Preventive Dentistry, College of Dentistry, University of Mosul, Mosul, Iraq.

Abstract.

Objectives: the study aims to assess the shear bond strength of modified- 3M orthodontic adhesive Transbond™ XT with different concentrations of Titanium Dioxide and zirconium oxide nanoparticles.

Materials and Methods: In a retrospective cross-sectional study a hundred sound-extracted human upper premolars were collected. 3M orthodontic adhesive Transbond™ XT was modified by (0.02%, 0.04%, and 0.06% Zirconium Oxide), and (0.02%, 0.04%, and 0.06% Titanium Dioxide) nanoparticles, and (0.02%, 0.04%, and 0.06% Zirconium Oxide and Titanium Dioxide). Dentaurum discovery pearl ceramic bracket were bonded to buccal enamel surfaces of the samples of control and nine modified adhesive groups. At 24 h after bonding, shear bond strength was measured. Adhesive remnant index was scored under (10X) magnification power of stereomicroscope after de-bonding. The chemical characteristics of orthodontic adhesive material were explored before and after mixing with Titanium Dioxide and zirconium oxide nanoparticles by using FTIR.

Results: (Zirconium Oxide nanoparticles) group and combination (Zirconium Oxide and Titanium Dioxide nanoparticles) had a higher shear bond strength mean value than the control group, 0.06% combination (Zirconium Oxide and Titanium Dioxide nanoparticles) group had the highest mean value. About adhesive remnant index, no significant differences were found among the studying groups.

Conclusions: the addition of 0.06% combination (Zirconium Oxide and Titanium Dioxide nanoparticles) had the best performance and improved shear bond strength of 3M orthodontic adhesive Transbond™ XT.

Key words: Zirconium oxide, titanium dioxide, nanoparticles, shear bond strength.

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 of 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 nano fillers 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]. This research's primary objective was to learn how nanoparticles incorporation (ZrO2 and/or TiO2) into orthodontic adhesive would affect its physical characteristics.

Materials and Methods.

Preparation of the Modified Adhesives: An electrical sensitive balance was used to aid in the process of preparing the modified adhesive containing ZrO2NPs. It was made in a weight-to-weight ratio of 0.02%, 0.04%, and 0.06%. The exact amount of ZrO2NPs and 3M orthodontic adhesive required to acquire the correct ratio were combined on a sterile glass slab. To ensure appropriate mixing and limit NP exposure, the glass slab was moved to a glass box. To ensure thorough soaking of the nanoparticles inside the resinous substance and consistent color dispersion, the mixture was manually mixed using a metal spatula for around 60 seconds in a semi-dark setting [9]. After being treated with ZrO2NPs, the orthodontic adhesive was transported to a sterile, disposable syringe which was then wrapped with a dark piece of tape to prevent it from being exposed to direct light.

1. Adhesive Modified TiO2NPs: Using a new glass slab, the same above steps were repeated but with TiO2NPs in place of ZrO2NPs.

2. Adhesive Modified with ZrO2NPs + TiO2NPs: using a new glass slab, the same process was repeated. Adding 0.01% of both ZrO2NPs and TiO2NPs to the adhesive yielded a final 0.02% concentration after mixing. In the same way, 0.04% & 0.06% w/w concentrations were achieved by mixing 0.02% and 0.03% of each additive respectively with the 3M orthodontic adhesive.

The tooth samples were rinsed with DW and scrubbed using a soft toothbrush to get rid of any remaining bits of soft tissue that had adhered to them during the extraction process. The teeth were then kept in a thymol solution of 0.1% at 37° degrees Celsius for storage. [10,11]. Each tooth was taken out of the thymol solution using a tweezer, submerged in distilled water for 10–15 seconds, and then dried on a clean, dry towel before being mounted. PVC (PolyVinyl Chloride) rings were utilized with 30 mm height, inside diameter of 18mm, and 20 mm outside diameter. In order to construct the samples designated for SBS testing, dental stone was loaded into the...
rings to about halfway up their height. Once the stone was set, sticky wax was employed to secure the stone surface and tooth apex together with the tooth’s long axis in its proper orientation, with the buccal section of in parallelism with a flat surface to simulate the path of force during the SBS testing [12]. Finally, cold-cure acrylic resin (auto-polymerizing) was utilized to pack the PVC rings up to the cemento-enamel junction (CEJ) level.

**Bonding Procedure:** The teeth samples were prepared by mounting them, then polishing them with rubber prophylactic cups and fluoride-free pumice for about 10 seconds using a slow-speed hand piece; finally, they were rinsed with a water spray to eliminate the remaining debris or pumice and then oil-free air was used to dry them for 30 seconds.

Etching for 15 seconds with 37% phosphoric acid gel, 10 seconds of rinsing with water, and then drying with a triple syringe until a chalky look is seen was the protocol followed on the buccal surface of each tooth. Utilizing a prefabricated base, the entire specimen was placed on the articulator. Clamping tweezers were used to keep the bracket steady while the resin material (3M orthodontic adhesive or the modified adhesives) was applied to the base and spread evenly using a dental explorer. The bracket was then positioned in the center (between 4 and 4.5 mm away from the occlusal surface) of the buccal surface of the sample. To ensure correct bracket position, Boons gauge was used. To ensure that the adhesive between the tooth surface and bracket base was of consistent thickness throughout all specimens and to avoid the trapping of air voids, a 200gm load was applied to the articulator arm and its direction was perpendicular in relation to the bracket slot [13]. With a sharp dental explorer, the extra resin was scraped off the bracket’s outside edges. LED light curing equipment with a (440 nm - 490 nm) wavelength and a (650 - 1500) mw/cm2 illumination was then used to initiate the curing process. A curing radiometer, with calibrations performed every 5 samples, was employed to maintain a constant light intensity throughout the polymerization of every specimen. At 2 mm away from the distal and mesial margins of the base of the bracket, the curing light was applied for 20 seconds on each side [14]. After 30 minutes of resting on the bench, the samples were sealed in a container with distilled water and incubated at 37˚ C for 24 hours before being tested.

**Measuring shear Bond Strength:** With a 0.5mm/min crosshead speed, the Tinius Universal testing machine was used to quantify the SBS. To guarantee that specimens are seated safely and securely and that their bracket bases are parallel to the direction of the shear stress, a prefabricated holder was constructed. A specially designed knife edge was aimed in an occluso-gingival orientation, targeting the tooth-bracket interface. The Failure loads unit of measurement was Newtons which was converted to megapascals (MPa) by dividing the failure load or force by the area of the bonded bracket base. Digitalized vernier was used to measure the bracket base surface area, and the result was 11.16 mm², which allowed the SBS to be computed and represented in megapascals (MPa) by utilizing the equation.

\[
\text{SBS in (MPa) unit} = \frac{\text{Force in Newton's unit}}{\text{Surface area of bracket base in mm}^2}
\]

**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.

**Fourier Transform Infrared Spectrometry (FTIR):** Using an FTIR instrument, the chemical properties of 3M orthodontic adhesive were examined both pre and post being mixed with NPs. Each concentration of 3M orthodontic adhesive and/or the modified adhesives were tested by depositing a tiny amount (drop-like) of adhesive on a sterile mixing slab and curing it for 40 seconds with the same LED curing apparatus. Then, using the FTIR table and a specific lens holder, the cured (drop-shaped) sample was pressed. The test was done in Ministry of Science and technology labs.

Transmission Electron Microscope (TEM): At Al-Nahrain University in Baghdad, TEM images of ZrO2NPs and TiO2NPs were obtained separately at several magnifications to determine the nanoparticle shape and size.

**Results.**

**Shear Bond Strength Results:**

**Descriptive analysis of tensile bond strength:** Table (1) and Figure (1) demonstrate the descriptive analysis data for shear bond strength. The data contain each group’s sample numbers, standard deviation, mean, range, standard error, and all the study groups SBS minimum and maximum values. These results show that the highest mean SBS among all the groups was reached in the (0.06%) ZrO2NPs group, while the lowest average SBS values were in the control group. On statistical levels, The rest of the groups were distributed between the maximum and minimum average values.

**SBS Analysis of Variance (ANOVA):**

Tables (2 and 3) show one way (ANOVA) statistical test results. The results show a significant differences at (P≤0.05) among the study groups SBS mean values.

**ARI of SBS groups descriptive analysis:**

Tables (4 and 5) and Figure (2) demonstrate The Descriptive data of the ARI for SBS. The data contains 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 ZrO2NPs (0.02%),
### Table 1. The study groups’ SBS Descriptive statistics.

<table>
<thead>
<tr>
<th>Groups</th>
<th>N</th>
<th>Min.</th>
<th>Max.</th>
<th>Mean</th>
<th>SD</th>
</tr>
</thead>
<tbody>
<tr>
<td>Control</td>
<td>5</td>
<td>11.19</td>
<td>12.84</td>
<td>11.948</td>
<td>0.626</td>
</tr>
<tr>
<td>2% Concentration of ZrO2</td>
<td>5</td>
<td>13.23</td>
<td>15.60</td>
<td>14.476</td>
<td>0.958</td>
</tr>
<tr>
<td>4% Concentration of ZrO2</td>
<td>5</td>
<td>16.60</td>
<td>17.30</td>
<td>16.900</td>
<td>0.273</td>
</tr>
<tr>
<td>6% Concentration of ZrO2</td>
<td>5</td>
<td>18.18</td>
<td>19.40</td>
<td>18.836</td>
<td>0.556</td>
</tr>
<tr>
<td>2% Concentration of TiO2</td>
<td>5</td>
<td>11.56</td>
<td>12.87</td>
<td>12.248</td>
<td>0.580</td>
</tr>
<tr>
<td>4% Concentration of TiO2</td>
<td>5</td>
<td>11.89</td>
<td>12.84</td>
<td>12.296</td>
<td>0.467</td>
</tr>
<tr>
<td>6% Concentration of TiO2</td>
<td>5</td>
<td>12.17</td>
<td>13.98</td>
<td>13.148</td>
<td>0.786</td>
</tr>
<tr>
<td>2% Concentration of ZrO2 and TiO2</td>
<td>5</td>
<td>14.23</td>
<td>15.60</td>
<td>14.876</td>
<td>0.554</td>
</tr>
<tr>
<td>4% Concentration of ZrO2 and TiO2</td>
<td>5</td>
<td>17.00</td>
<td>18.90</td>
<td>18.100</td>
<td>0.880</td>
</tr>
<tr>
<td>6% Concentration of ZrO2 and TiO2</td>
<td>5</td>
<td>19.20</td>
<td>19.90</td>
<td>19.516</td>
<td>0.307</td>
</tr>
</tbody>
</table>

N is number, SBS Variable unit is MPa, TiO2NPs is titanium dioxide nanoparticles groups, and ZrO2NPs is Zirconium Oxide nanoparticles, SD=standard deviation.

### Table 2. Descriptive analysis of the SBS mean values among the study groups using one way (ANOVA) test.

<table>
<thead>
<tr>
<th>Source of Variation</th>
<th>Sum of Squares</th>
<th>Df</th>
<th>Mean Square</th>
<th>F</th>
<th>Sig.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Between Groups</td>
<td>378.497</td>
<td>9</td>
<td>42.055</td>
<td>103.941</td>
<td>0.000*</td>
</tr>
<tr>
<td>Within Groups</td>
<td>16.184</td>
<td>40</td>
<td>0.405</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Total</td>
<td>394.681</td>
<td>49</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

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

### Table 3. Multiple Comparisons of SBS among the study groups using Duncan’s Multiple Range Test.

<table>
<thead>
<tr>
<th>Groups</th>
<th>N</th>
<th>Mean</th>
<th>Duncan</th>
</tr>
</thead>
<tbody>
<tr>
<td>Control</td>
<td>5</td>
<td>11.948</td>
<td>A</td>
</tr>
<tr>
<td>0.02% Concentration of TiO2</td>
<td>5</td>
<td>12.248</td>
<td>A</td>
</tr>
<tr>
<td>4% Concentration of TiO20.0</td>
<td>5</td>
<td>12.296</td>
<td>A</td>
</tr>
<tr>
<td>6% Concentration of TiO20.0</td>
<td>5</td>
<td>13.148</td>
<td>B</td>
</tr>
<tr>
<td>2% Concentration of ZrO20.0</td>
<td>5</td>
<td>14.476</td>
<td>C</td>
</tr>
<tr>
<td>2% Concentration of ZrO2 and TiO20.0</td>
<td>5</td>
<td>14.876</td>
<td>C</td>
</tr>
<tr>
<td>4% Concentration of ZrO20.0</td>
<td>5</td>
<td>16.9</td>
<td>D</td>
</tr>
<tr>
<td>4% Concentration of ZrO2 and TiO20.0</td>
<td>5</td>
<td>18.1</td>
<td>E</td>
</tr>
<tr>
<td>6% Concentration of ZrO20.0</td>
<td>5</td>
<td>18.836</td>
<td>EF</td>
</tr>
<tr>
<td>6% Concentration of ZrO2 and TiO20.0</td>
<td>5</td>
<td>19.516</td>
<td>F</td>
</tr>
</tbody>
</table>

N is number, ZrO2-NPs is TiO2-NPs is titanium dioxide nanoparticles, and Zirconium Oxide nanoparticles.

### Table 4. ARI scores frequency distribution of the SBS across the study groups.

<table>
<thead>
<tr>
<th>Groups</th>
<th>0*</th>
<th>1*</th>
<th>2*</th>
<th>3*</th>
</tr>
</thead>
<tbody>
<tr>
<td>C</td>
<td>1</td>
<td>1</td>
<td>3</td>
<td>0</td>
</tr>
<tr>
<td>ZrO2NPs (0.02%)</td>
<td>2</td>
<td>1</td>
<td>1</td>
<td>1</td>
</tr>
<tr>
<td>ZrO2NPs (0.04%)</td>
<td>2</td>
<td>2</td>
<td>1</td>
<td>0</td>
</tr>
<tr>
<td>ZrO2NPs (0.06%)</td>
<td>2</td>
<td>3</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>TiO2NPs (0.02%)</td>
<td>2</td>
<td>3</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>TiO2NPs (0.04%)</td>
<td>2</td>
<td>3</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>TiO2NPs (0.06%)</td>
<td>3</td>
<td>2</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>ZrO2-TiO2NPs (0.02%)</td>
<td>4</td>
<td>0</td>
<td>1</td>
<td>0</td>
</tr>
<tr>
<td>ZrO2-TiO2NPs (0.04%)</td>
<td>2</td>
<td>3</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>ZrO2-TiO2NPs (0.06%)</td>
<td>2</td>
<td>2</td>
<td>1</td>
<td>0</td>
</tr>
</tbody>
</table>

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, TiO2NPs is titanium dioxide nanoparticles, and ZrO2NPs is Zirconium Oxide nanoparticles.
Table 5. The SBS study groups’ ARI Descriptive statistics.

<table>
<thead>
<tr>
<th>Groups</th>
<th>N</th>
<th>Min</th>
<th>Max.</th>
<th>Mean</th>
<th>Std. Dev.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Control</td>
<td>5</td>
<td>0</td>
<td>3</td>
<td>1.25</td>
<td>1.258</td>
</tr>
<tr>
<td>ZrO2NPs (0.02 %)</td>
<td>5</td>
<td>1</td>
<td>2</td>
<td>1.25</td>
<td>0.5</td>
</tr>
<tr>
<td>ZrO2NPs (0.04 %)</td>
<td>5</td>
<td>0</td>
<td>2</td>
<td>1.25</td>
<td>0.957</td>
</tr>
<tr>
<td>ZrO2NPs (0.06 %)</td>
<td>5</td>
<td>0</td>
<td>3</td>
<td>1.25</td>
<td>1.5</td>
</tr>
<tr>
<td>TiO2NPs (0.02 %)</td>
<td>5</td>
<td>0</td>
<td>3</td>
<td>1.25</td>
<td>1.5</td>
</tr>
<tr>
<td>TiO2NPs (0.04 %)</td>
<td>5</td>
<td>0</td>
<td>3</td>
<td>1.25</td>
<td>1.5</td>
</tr>
<tr>
<td>TiO2NPs (0.06 %)</td>
<td>5</td>
<td>0</td>
<td>3</td>
<td>1.25</td>
<td>1.5</td>
</tr>
<tr>
<td>ZrO2 TiO2NPs (0.02 %)</td>
<td>5</td>
<td>0</td>
<td>4</td>
<td>1.25</td>
<td>1.892</td>
</tr>
<tr>
<td>ZrO2 TiO2NPs (0.04 %)</td>
<td>5</td>
<td>0</td>
<td>3</td>
<td>1.25</td>
<td>1.5</td>
</tr>
<tr>
<td>ZrO2 TiO2NPs (0.06 %)</td>
<td>5</td>
<td>0</td>
<td>2</td>
<td>1.25</td>
<td>0.957</td>
</tr>
</tbody>
</table>

Figure 1. Study groups Mean SBS values. SBS Variable unit is MPa. TiO2NPs is titanium dioxide nanoparticles, ZrO2NPs is Zirconium oxide nanoparticles.

Figure 2. ARI scores frequency distribution across the SBS study groups, TiO2NPs is titanium dioxide nanoparticles, and ZrO2NPs is Zirconium Oxide nanoparticles.
ZrO$_2$NPs (0.04%), ZrO$_2$TiO$_2$NPs (0.06%), ZrO$_2$NPs (0.06%), TiO$_2$NPs (0.02%), TiO$_2$NPs (0.04%) then ZrO$_2$TiO$_2$NPs (0.04%) groups. The lowest ARI mean scores belonged to the groups TiO$_2$NPs (0.06%) and ZrO$_2$TiO$_2$NPs (0.02%).

**Fourier Transform Infrared Spectrometry (FTIR) Charts:**

C=C, C=O, and C-H stretching can be seen in the control groups FTIR spectra at 1390, 1710, and 2929, respectively; when compared to the modified adhesive FTIR spectra, it is clear that the aforementioned bands remain in the same area as in the Control spectra, without shifting (no new bands appearing or bands disappearing). ZrO$_2$ and TiO$_2$ do not show up in FTIR charts because their metallic bands have absorption spectra in the sub-400 wavenumber cm$^{-1}$ area, which is outside the instrument measurement capabilities (Figure 3).

**Transmission Electron Microscope (TEM) Results**

TEM image of Zirconium Oxide Nanoparticles (ZrO$_2$NPs)

To determine the probable shape and size of particles, ZrO$_2$NPs powder TEM images were captured at varying magnifications (x180000, x130000, x92000). Although the ZrO$_2$NPs are neither modified nor coated with any modifying materials, transmission electron microscopy photos revealed less tendency for agglomeration. They also revealed a round or oval particle shape, and an around (20nm) particle size (Figure 4).

Titanium dioxide Nanoparticles (TiO$_2$NPs) TEM Images

The powdered TiO$_2$NPs have been imaged using transmission electron microscopy (TEM) at three different magnifications (x92000, x130000, and x180000) to determine their potential size and form. The transmission electron microscopy (TEM) images revealed semi-oval or round particles shape and a range of sizes of nanoparticles present, from (10-30nm). As the TiO2NPs employed in this study weren't coated with any altering agents or modified, they also shown a strong propensity for agglomeration into bigger particles (Figure 5).

**Discussion.**

Extracted human maxillary premolars were used as this study's sample. As orthodontic treatment frequently include their extraction. Due to potential negative effects of aging, such as a shift in the fluoride concentration of the enamel's outermost layer, the age group of (15-25) years old was selected [15]. The samples came from three separate governorates (Baghdad, babil) which contributed to the sample variance, which in turn affected the results. To prevent bacterial growth, we placed...
the samples in a 0.1% thymol solution, which also has the advantage of causing minimal alterations to the surface of the enamel during storage.

In accordance with other studies Transbond™ XT was chosen as the control adhesive [16] because it was viewed as the orthodontic gold standard adhesive because a) Immediate Bond Strength b) Extended working time c) Excellent handling e) Quick bracket cure. Transbond™ XT primer was composition from TEGDMA, Bis GMA 4-(dimethylamino)-Benzenethanol, DL-camphoroquinone hydroquinone Transbond™ XT paste BisGMA, Bisphenol A (2 Hydroxyethyl Ether), Dimethacrylate, Silane treated quartz, Silane treated silica, Diphenyliodonium hexafluorophosphate, with a bond strength value of (12) MPa for metallic brackets and (10) MPa for ceramic brackets, and it had been considered as the Control substance for this study, on which the ZrO2NPs and TiO2NPs were added in three different concentrations.

Generally, it is not necessary to obtain very extreme bond strength to refer to well clinical performance [17]. Therefore, is more important for clinical orthodontic practice to find adequate bond strength rate. That allow safe debonding of fixed appliance parts than to develop the highest Potential rate [18]. In vitro bond strength SBS and TBS tests were chosen for this study, these tests were carried out 24 hours after bonding. Approximately (5.9 - 7.8) and (6-8) MPa is the minimum SBS required to accommodate masticatory and orthodontic stresses, as stated by Reynolds (1975) and Fox et al. (1994) respectively. According to Fajen et al. (1990), (2.86) MPa is the bare minimum value for TBS needed to resist debonding stresses by orthodontic brackets.

Since the advent of nanotechnology, researchers have proven that nano-scale materials offer superior physical and mechanical qualities over their micron-scale counterparts [19]. The mechanical, antimicrobial, optical, chemical, and physical characteristics of orthodontic adhesives have been enhanced with the use of various nanofillers. The size, method of mixing these fillers with the adhesives, form, distribution or dispersion, and concentration are crucial factors in achieving these improvements [20].

Because of its accessibility as well as its many desirable qualities, ZrO2NPs was selected to serve as the filler in this study. Because of its high biocompatibility and white appearance, zirconium oxide (ZrO2) was chosen for this study. In this research, nanofiller particles were utilized because of their enhanced compatibility with organic polymer, improved dispersion, and elimination of aggregation [21].

TiO2NPs were selected for this work because of their many desirable characteristics, such as their chemical stability, efficient photocatalytic activity, cheap price, low toxicity, wide availability, white color, and attractive tint which coincide to the opaque nature of teeth. TiO2 has compelling antibacterial action and improved mechanical characteristics, such as bond strength value, flexural strength, modulus of elasticity, and micro hardness equivalent to or greater compared to controls lacking TiO2NPs and can enhance percent elongation and tear and tensile strengths [22,23].

This is the first study to our knowledge to examine the impact of adding TiO2NPs alone, ZrO2NPs alone, and a mixture of both to orthodontic adhesive, and also the first to evaluate the impact on TBS and SBS at three separate concentrations for each (0.02%, 0.04%, and 0.06%). Previous evaluations of nanofillers suggested that combining NPs of varying particle size makes for more advantageous characteristics than by adding just one...
type of NP, therefore in this experiment we combined ZrO2NPs and TiO2NPs to create a third group to test in the orthodontic adhesive [10].

Prior studies of ZrO2NPs and TiO2NPs proposed using concentrations of 1%, 1.2%, and 1.4% which were originally chosen for this study; however, during the pilot study, it was found that even at the lowest concentration of 1%, the resulting altered adhesive did not polymerize after 30, 60 and 120 seconds of curing. The same was true for the next lower concentrations: 0.4%, 0.5%, 0.2%, and 0.1%. Hence, the concentrations that were less than 0.1% were the primary emphasis. Concentrations of 0.02%, 0.04%, and 0.06% were chosen because they correspond to the three fundamental ideas of NPs:

All nanoparticles, and TiO2nanoparticles in particular, have a strong propensity to agglomerate with one another into micro-sized particles, particularly when combined with polymeric materials; these agglomerations behave as a weak spot wherein stress is focused, leading to compromising the translucency of the modified adhesive and bonding failure. Silanated nanofillers permit better dispersion into the resin matrix and thus, organosilane material is advised for surface modification of TiO2NPs to decrease agglomeration [24].

Zirconium nanoparticles are lighter and less susceptible to embrittlement by hydrogen. Available in the form of nanodots, Nano fluids nanocrystals with the white surface area. Provide great resistance to corrosion by acids, alkalis, salt water and other agents [25].

The photo activity of TiO2NPs have an important role as co-initiator for the photo activator present in the adhesive, and with increasing the ratio of the added TiO2NPs beyond 1% an interference with the translucency of the whole material will occur, resulting in low degree of conversion (DC) of the modified adhesives [26].

When adding nanofillers to any type of orthodontic adhesives, it is important to add them in low percentage, so that to preserve the adhesive flowability and maintain the low viscosity, these two properties are the main characteristics of the orthodontic adhesives [27].

SBS were greater than the mean values for the TBS in this study. This is because the two forms of testing cause different distributions of stress. Non-uniform stress distribution is typical in shear and tensile testing because of variability in the “loading configuration” used to debond the material, the resin’s modulus of elasticity, specimens’ geometry, specimen-machine alignment. In the tensile test, debonding was accomplished with an SS ligature wire, and in the shear test was accomplished using a chisel knife cross head, and in the tensile test, debonding was accomplished with an SS ligature wire. When taken together, these variables can have a significant impact on the total stress level, the region of stress concentration, and the resulting bond strength value. This result agreed with previous studies by Leloup et al. (2001), Arici et al. (2004), and Sunilkumar et al. (2013), all of which used a different resinous substance and substrate but also reported much greater shear bond mean values than tensile mean values.

In this study's SBS findings, the ZrO2TiO2 (0.06%) group was shown to have the most notable difference compared to the C group. Orthodontic adhesive's SBS values are improved significantly when ZrO2NPs are used, either alone or in combination with TiO2NPs. The ZrO2TiO2 (0.06%) had the greatest SBS mean value, followed by ZrO2(0.06%), ZrO2(0.04%), ZrO2(0.02%), TiO2(0.06%), and lastly TiO2(0.02%), which had the lowest SBS mean value.

While the inclusion of TiO2NPs did increase the SBS, the values obtained were still higher than the minimum recommended SBS values given by Reynolds (1975), which were between 5.9 and 7.8 MPa, and Fox et al. (1994), which were between 6 and 8 MPa. This was in agreement with the findings of Poost et al. (2013), who observed that including TiO2NPs into orthodontic adhesive enhanced its antibacterial capabilities without lowering the SBS mean values. The addition of very small amounts of TiO2NPs to orthodontic adhesives, as reported by Reddy et al. (2016), may reduce the SBS. In addition, as reported by Sodagar et al. (2017), the mean SBS values are kept within the acceptable range of SBS indicated by Reynolds when 1% and 5% TiO2NPs are added. While Xia et al. (2008) observed a significant enhancement in the mechanical characteristics of orthodontic adhesive after adding 1% TiO2NPs following surface modification with organosilane, the current findings contradict that finding. Also disagree with Sun et al. (2011)'s findings, according to which adding 0.1% weight percentage of TiO2NPs to conventional dentin adhesive causes a mean SBS increase of around 30%. TiO2 NPs increased the composite's shear bond strength and mechanical characteristics [28].

Regarding the ZrO2NPs addition and combination addition its effect on the SBS, Zirconium oxide (ZrO2) was selected due to its high level of biocompatibility as well as its white hue, which makes it less likely to experience deterioration in terms of its aesthetic. In this research, nanofiller particles were chosen because they provide greater dispersion, prevent aggregation, and are more compatible with organic polymer [29].

It has been reported that a combination of titanium dioxide (TiO2) and zirconium dioxide (ZrO2) nanoparticles is superior to either additive alone, mostly due to the size disparities between the two materials [30].

This study's findings corroborated those of Felemban and Ebrahim, who found that adding ZrO2-TiO2 nanoparticles to orthodontic adhesive increased its compressive, tensile, and shear bond strengths. These improvements can be related to the smaller particle sizes of the ZrO2 and TiO2, as well as the surface modification of the nanofillers with TMSPM coupling agents, which improves interfacial adhesion of the fillers to the polymer matrix, improves particle dispersion in the matrix, and prevents agglomeration [10].

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. 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 This was in agreement with [31], who reported that the adhesive/bracket interface was the most common site of failure in tensile test specimens with high ARI scores. 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 [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 [33].

While [34] 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).

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 was in agreement with [36] who also discovered that the majority of specimens had ARI scores of (1-0), but it was at odds with [37] 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.

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.

REFERENCES