

Evaluation of Physical and Mechanical Properties of a Novel Titanium Dioxide Nanoparticle Infiltrated Orthodontic Adhesive – An *In-Vitro* Study

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Abstract

This *in-vitro* study aims to assess and compare the physical and mechanical properties of a green synthesized (novel) Titanium dioxide nanoparticle infiltrated orthodontic adhesive with conventional orthodontic adhesive. A total of twenty disk-shaped specimens were fabricated by condensing the composite resin in a stainless-steel metal mold having a circular shape (10 x 2 mm) and polymerizing it using blue light (470nm). Scanning Electron Microscopy (SEM) and Fourier-transform infrared spectroscopy (FTIR) were used to characterize the TiO₂ NPs. The results of physical properties such as colour stability, and surface roughness showed no significant mean difference and the microhardness of two orthodontic adhesives showed a significantly greater hardness for conventional adhesives. Conventional orthodontic adhesive showed significantly increased compressive strength and greater tensile strength for novel TiO₂-NPs infiltrated orthodontic adhesive ($p > 0.05$). The results also showed improved mechanical properties for both groups.

Keywords: Green Synthesis, Mechanical Properties, Nanoparticles, Orthodontic Adhesives, Physical, Titanium Dioxide.

Introduction

A big challenge in orthodontic treatment for both patients and orthodontists with fixed orthodontic appliances is maintaining good oral hygiene, as fixed appliance components are plaque-retentive [1, 2]. Following this big challenge, occurs enamel demineralization during and after orthodontic treatment resulting in white spot lesions (WSLs). It has been reported that WSLs occur in 30% to 70% of the patients during orthodontic treatment [3-5] with a prevalence rate of 68.4% [6], affecting 18.5% of tooth surfaces [7]. These lesions are induced clinically within 4 weeks of orthodontic treatment occurring due to decreased oral pH and lactic acid produced by *Streptococcus mutans* [8-11]. Thus, the prevention of WSLs becomes inevitable to provide the integrity of dentition during orthodontic treatment [12].

This lesion can be managed by maintaining good oral hygiene, and use of topical fluorides including high-fluoridated toothpastes, fluoride, chlorhexidine mouthwashes, gels, and varnishes. Patient cooperation is of utmost importance in carrying out these preventive strategies, which is questionable. Short-term release of antimicrobial agents with reduced mechanical, and physical properties and possible tooth discolouration are some of the demerits of these preventive strategies [12, 13]. Thus, there is a need to produce agents with superior mechanical, chemical, physical, optical, and antimicrobial properties.

Recently, research has been directed at producing orthodontic adhesives with high antimicrobial properties to prevent WSLs. High charge density and large surface area of nanoparticles (NPs) which interact with bacterial cells exhibiting increased

antimicrobial efficacy are being introduced into the orthodontic adhesives without affecting the value of the bonded interface [14-18]. Nanoparticles such as gold [19], silver [20], copper [21], zinc oxide [22], silicon dioxide [23, 24], hydroxyapatite [25], and titanium dioxide (TiO₂) [26] have been added to orthodontic adhesive to prevent WSLs.

Compared to other NPs, Titanium dioxide nanoparticles are considered to have increased optical properties with a high reflective index, and chemical stability causing no colour change of resin composites around brackets. Also, TiO₂ has excellent mechanical properties such as increased microhardness, flexural strength, shear bond strength and modulus of elasticity [27-33]. Apart from augmented mechanical properties, TiO₂ NPs infiltrated into orthodontic adhesives display compelling antimicrobial properties without affecting the bond strength [34].

Nanoparticles are usually synthesized by chemical methods causing environmental pollution, increased energy consumption and imposing health problems. Green synthesis is the novelty which uses reducing agents derived from plants or microorganism extracts to reduce the metal ions, combating the above-mentioned problems [35]. Green synthesis costs less, decreases environmental pollution and improves human health and safety.

Though earlier studies assessed the mechanical properties of TiO₂ NPs, a literature search showed none of the studies assessed the physical and mechanical properties of green synthesized TiO₂ NPs infiltrated orthodontic adhesives. Thus, the present in-vitro study aims to assess and compare the physical and mechanical properties of a green synthesized (novel) Titanium dioxide nanoparticle infiltrated orthodontic adhesive with conventional orthodontic adhesive.

Materials and Methods

Sample Size Calculation

Based on the previous study [36], a sample size of 10 specimens per group was needed to exhibit a statistically significant difference in mechanical and physical properties between the groups upon alpha error of 5% and power of 80%.

Preparation of Titanium Dioxide Nanoparticle

Preparation of Leaf Extract

One gram of *Eucalyptus globulus* powder was mixed with 100 ml of distilled water to form a homogenous mix of the plant extract. The solution was then heated and boiled in a heating mantle. Using Whatman no:1 filter paper, the boiled plant extract was filtered, and the supernatant solution was collected in a beaker.

Preparation of TiO₂ NPs

About 50 ml of distilled water was mixed with 0.39g of titanium dioxide powder. To this, 50 ml of the prepared supernatant plant extract solution was added. The obtained solution was then placed on a magnetic stirrer/ orbital shaker overnight to observe the visual and chemical changes observed during the synthesis of TiO₂ nanoparticles. A colour change from white to whitish brown was observed. This was due to the reduction of the metal ion (Ti⁴⁺) indicating the synthesis of TiO₂ nanoparticles. Using hourly UV-vis spectrometric data, titanium dioxide nanoparticle production was evaluated and the same was used to confirm the presence of TiO₂ nanoparticles produced in the solution.

Upon confirmation, the solution was equally divided and poured into centrifuge tubes for centrifugation. The pellet obtained was collected in a separate tube and later poured into petri dishes. These Petri dishes were dried in a hot air oven, the dry substrate containing the TiO₂ nanoparticle was scraped, and the powder was collected and labelled.

Characterization of Synthesized TiO₂ Nps Using Scanning Electron Microscopy (Sem) and Fourier-Transform Infrared Spectroscopy (FTIR)

Scanning Electron Microscopy SEM

The JSM –IT800 Nano SEM (Joel, Nagoya University, Japan) was used to view the obtained nanoparticle and confirm its shape and size. SEM was used to examine external specifications of adsorbent morphology.

UV-Vis Spectroscopy

UV-Vis Spectroscopy (Malvern Panalytical Ltd; Enigma Business Park, Grovewood Road, Malvern WR14 1XZ, United Kingdom) was used to record the synthesized titanium dioxide nanoparticles' UV-Vis peak of absorption. The specimens' scanning ranged from 350 to 660 nm. A distilled water standard was used to interpret all UV-Vis absorption spectra.

FT-IR

An FTIR spectrophotometer (BRUKER; Massachusetts; United States) was used to study the functional groups on the biaxial surface. It is a good analytical tool to illustrate the bending properties of synthesized nanoparticles. The spectrum was recorded in the range of 400–4000 cm.

Preparation and Characterization of TiO₂ Infiltrated Orthodontic Adhesive

Preparation of Nano Infiltrated Adhesive

To achieve an orthodontic adhesive containing 1% nanoparticle, 0.04g of prepared titanium dioxide nanoparticle was mixed with 4 ml of dichloromethane (DCM) in a beaker. The beaker was covered with aluminium foil all around to ensure that no light passed through. To this mixture, 4g of orthodontic adhesive (Ormco Enlight light-cured adhesive, ORMCO) was added and continuously stirred manually. The beaker was then placed in an orbital shaker with an rpm (rotations per

minute) of 500 for 24 hours to ensure uniform distribution of nanoparticles within the matrix. The obtained material was then retro-filled into a black syringe to prevent light exposure.

Characterization of TiO₂ Nanoparticle Infiltrated Orthodontic Adhesive

The JSM –IT800 Nano SEM and EDX (Energy Dispersive X-ray) were used to view the obtained nanoparticle infiltrated orthodontic adhesive and confirm its shape and size. SEM was used to examine external specifications of adsorbent morphology.

Evaluation of Properties of the Novel TiO₂ Nanoparticle Infiltrated Orthodontic Adhesive

Grouping of Specimens

All tests evaluating the physical, chemical, and mechanical properties were conducted between two sample groups, Group 1 (G1): Conventional orthodontic adhesive (ENLIGHT ORMCO), Group 2 (G2): Titanium dioxide infiltrated orthodontic adhesive (TiO₂ nanoparticle + Enlight orthodontic adhesive).

Physical Properties

All physical and mechanical properties were measured before and after the ageing of the study material.

Ageing Procedure

All specimens of Groups 1 and 2 were subjected to thermo-cycling by being stored alternatively in water reservoirs at 5°C and 55°C, respectively, and remaining in each reservoir for 30 seconds.

Specimen Preparation

A total of twenty disk-shaped specimens were fabricated by condensing the composite resin in a stainless-steel metal mold having a circular shape (10 x 2 mm) and polymerizing it using blue light (470nm). Following polymerization, the specimens were removed from the mold and polished with a Sof-Lex polishing kit (3M ESPE). A digital calliper was

used to check the specimen's dimensions. The specimens with voids, defects, or incorrect dimensions were discarded. Specimens were then stored for 24 hours in distilled water in a dark container, at room temperature for complete polymerization. These prepared samples were used for evaluating the colour stability, surface roughness, and hardness before and after ageing.

Colour

Colour measurements were performed with a SPECTROPHOTOMETER CM-5 according to the CIE Lab (Commission Internationale de l'Eclairage, L*, a*, b*) coordinates. The target mask is selected based on the illumination area (specimen measuring port size) of 3 mm, and zero calibration is performed to compensate for the effects of stray light. The colour coordinates L, a and b of each specimen were measured relative to the standard illumination D65 and a 10-degree standard observer.

The CIE Lab coordinates were used to calculate the colour difference (ΔE) between the “before” and “after” periods of thermocycling. The colour difference (ΔE) between the two-time interval readings was calculated according to the following equation:

$$\sqrt{(\Delta L)^2 + (\Delta a)^2 + (\Delta b)^2} = \Delta E^2$$

Where ΔL , Δa , and Δb are the differences between the parameters before and after thermocycling. $\Delta E < 1$ were considered clinically imperceptible, between 1 to 3.3 as acceptable colour change, >3.3 were considered clinically relevant and highly noticeable.

Surface Roughness

The surface roughness parameters of the specimens were evaluated using SurfTest SJ-310 (Mitutoyo South Asia Pvt. Ltd, New Delhi). The surface profile was determined as average roughness (Ra), defined as the mean between peaks and valleys of the surface profile, Rq is the largest roughness considering all the cut-offs (Highest peak and highest valley), and Rz is the mean of 10 peaks and 10 valley heights in

each cut-off. Ra and Rz are average roughness parameters, and they are not enough to distinguish surfaces that differ in spacing or shape. Data for the surface roughness of all specimens before and after thermocycling were recorded separately for Ra, Rq and Rz.

Hardness

The Vickers hardness of the specimens was evaluated with a microhardness tester (Shimadzu HMV-G, Newage Testing Instruments, Southampton, PA). The indentations were made with a Vickers diamond indenter at a weight of HV 0.3 (2.942 N) with a dwell time of 20 seconds. The indentation was captured at a magnification of 40X. The values were noted for each of the specimens subjected to testing.

Mechanical Properties

Compressive and Diametral Tensile Strength

Mold Construction

A 3 mm diameter by 10 mm height cylindrical Teflon mold was fabricated according to International Standards Organization (ISO) No. 9917 (2000).

Specimen Preparation

The orthodontic Adhesive was condensed in the Teflon mold, which was then placed on a glass plate according to each group. Specimens were covered with celluloid strips and pressed with another glass plate. All the specimens were exposed to light cure for 40 seconds on both sides. The specimens were stored in distilled water for 24 hours before testing.

Compressive Strength Testing

Specimens were loaded on the Universal testing machine at a crosshead speed of 0.5 mm/min. The specimens were placed with their flat ends vertically laid between the two metal plates. The load was applied until the specimen was crushed; the peak force required to fracture each specimen was recorded in Newton from the stress-strain curve. The compressive

strength was calculated in megapascals (MPa) using the following equation

$$4P = \pi d$$

Where P is the load at the fracture point in Newtons and d is the diameter of the specimen.

Diametric Tensile Strength

Measuring the tensile strength of orthodontic adhesive is done using an indirect tensile test, in which a compressive load is placed on the diameter of a cylindrical specimen. The compressive strength induces a tensile stress in the plane of the application of the force. The tensile strength is directly proportional to the compressive load. In this test, the specimen cylinder was mounted on the universal testing machine and the load was applied to the specimen using a crosshead speed of 0.5 mm/min applying a compressive force to the specimen until fracture. The diametral tensile strength was calculated in MPa using the following equation

$$2P = \pi dt$$

Where P is the load at the fracture point in Newtons, d is the diameter of the specimen, and t is the thickness of the specimen.

Statistical Analysis

Data was collected and tabulated in Microsoft Excel and imported for statistical analysis to SPSS software (Statistical Package for the Social Sciences Inc. IBM, NY, USA) version 24.0. The normal distribution of the data was evaluated using Shapiro-Wilk's test. The data was normally distributed, hence parametric tests were used. Independent t-tests and paired t-tests were used to compare colour stability, surface roughness, hardness, compressive strength and diametral strength between and within the groups respectively. A significance level of $p < 0.05$ was considered.

Results

Green synthesis of TiO₂ NPs

A colour change from white to whitish brown was observed, as shown in Figure 1. This was due to the reduction of the metal ion (Ti⁴⁺) indicating the synthesis of TiO₂ nanoparticles. UV-vis spectrometry was used and confirmed the presence of TiO₂ nanoparticles in the solution.

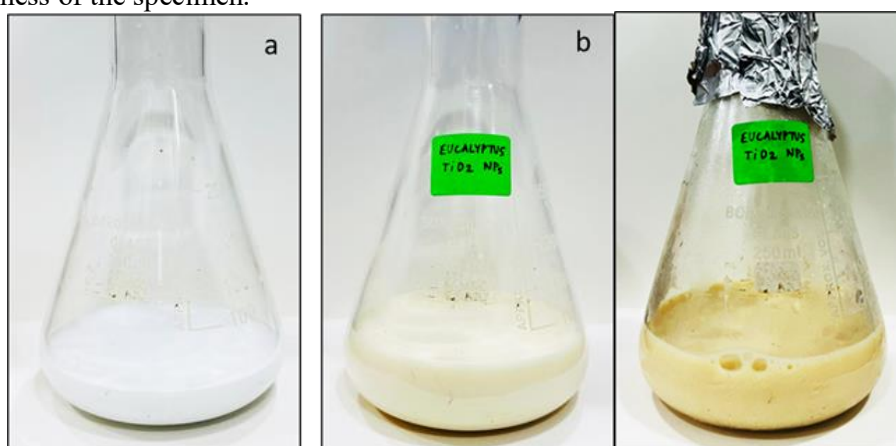


Figure 1. (a) TiO₂ Mixture with Distilled Water; (b) TiO₂ Solution Mixed with the Plant Extract

Characterization of TiO₂ NPs

The present study has described the synthesis of TiO₂ NPs by Eucalyptus globulus leaf extract mediated reduction of aqueous titanium ions. The formation of TiO₂ NPs in aqueous solutions was confirmed by using UV-

visible spectral analysis. Results showed the reduction of titanium ions, and the generation of TiO₂ NPs was completed after overnight incubation at room temperature. The formation of a whitish-brown colour indicated the reduction of titanium ions. The absorption

spectra of had absorbance peaks between 300-350 nm for the plant extract solution exposed to TiO₂ (Figure 2).

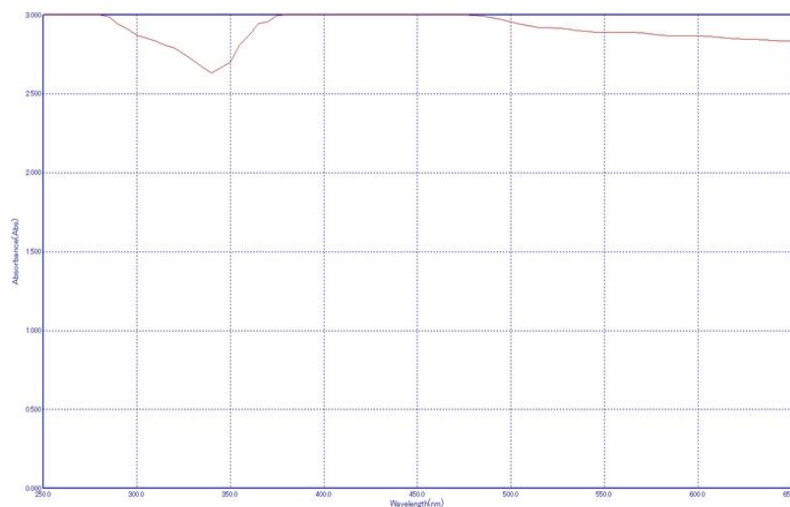


Figure 2. Uv-Vis Spectroscopy Graph

FT-IR

Estimation of bending properties of the TiO₂ NPs was done at room temperature between the wavelengths 400-4000 cm⁻¹. It can be observed

that at 584.24 cm⁻¹ TiO₂ bending mode and at 1101.46 cm⁻¹ stretching mode can be observed (Figure 3). This may be attributed to surface water sorption.

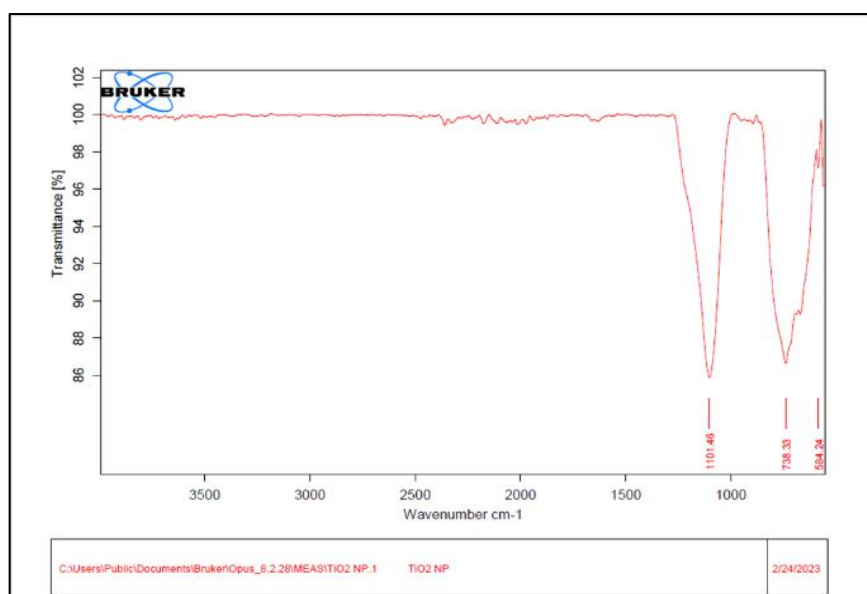


Figure 3. Ftir Graphic Representation

SEM and EDX

The scanning electron microscope was used to evaluate the size, shape, and surface properties, like morphology. SEM picture at 10x magnification of TiO₂ nanoparticles

synthesized using the leaf extract of Eucalyptus Globulus. The SEM image shows a uniform distribution of the TiO₂ nanoparticles, which are smooth and spherical (Figure 4). The average size of the nanoparticle ranged between 20 and 70 nm.

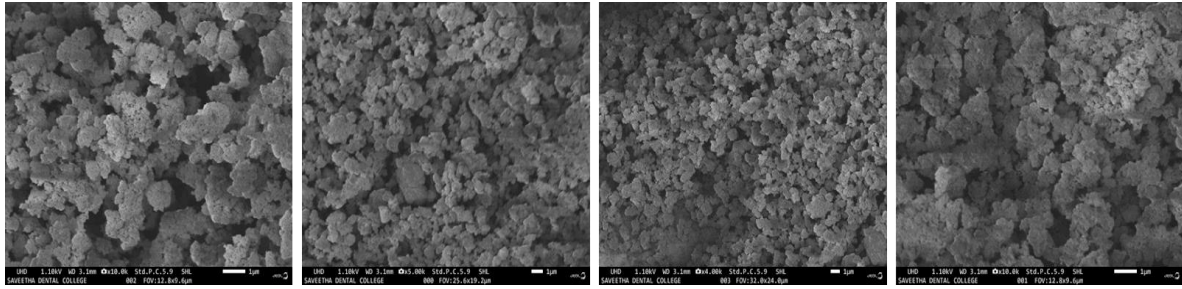


Figure 4. Sem Images of Tio2 Nanoparticle

EDX profile contained a strong TiO₂ signal along with other weak signals, which might be due to the biomolecules bound to the surface of the nanoparticles. Although strong signals were due to titanium in the nanoparticles, weaker signals for oxygen and chloride were due to the

biomolecules of *Eucalyptus globulus*. The signals might have been due to X-ray emission from carbohydrates/proteins/enzymes existing in the cell wall of the biomass. In the present study, TiO₂ NPs revealed a strong absorption spectrum at 4-5 keV (Figure 5).

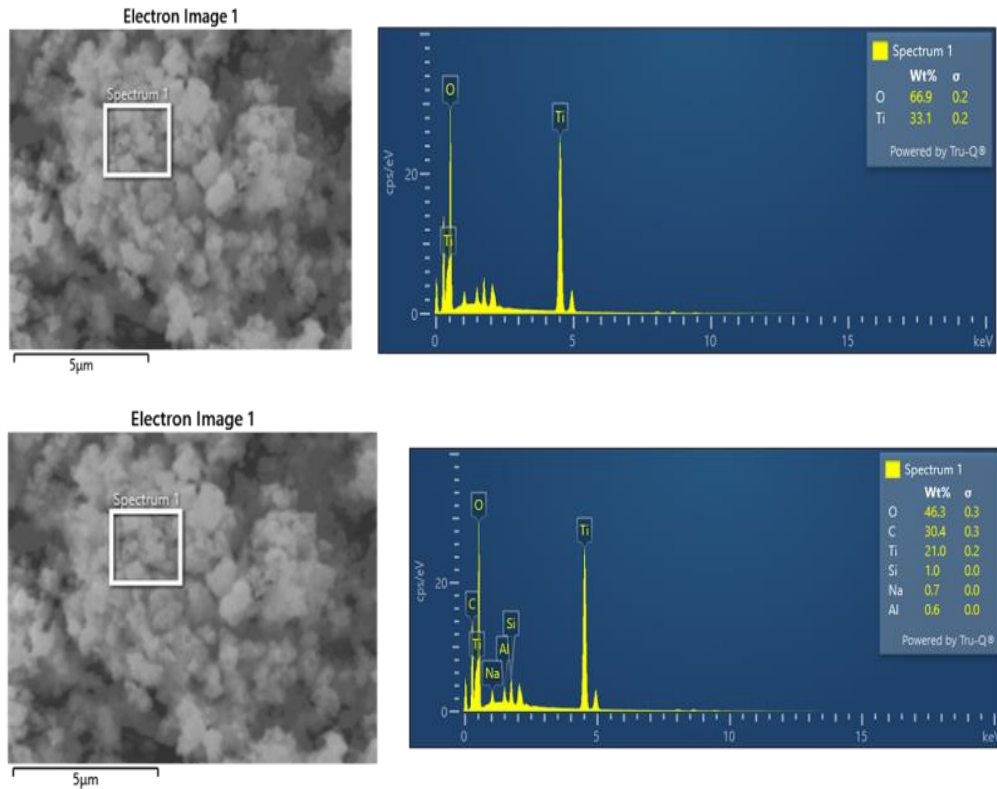


Figure 5. Edx Images of Tio2 Nanoparticle

Characterization of Novel TiO₂ Infiltrated Orthodontic Adhesive

SEM/EDX

The scanning electron microscope was used to evaluate the dispersion of TiO₂ NPs in the orthodontic adhesive matrix. Figure 6

illustrates the SEM picture at 10x magnification of TiO₂ nanoparticles infiltrating orthodontic adhesive. The SEM image shows a uniform distribution of the TiO₂ nanoparticles that are smooth and spherical. The average size of the nanoparticle ranges between 20 and 70 nm.

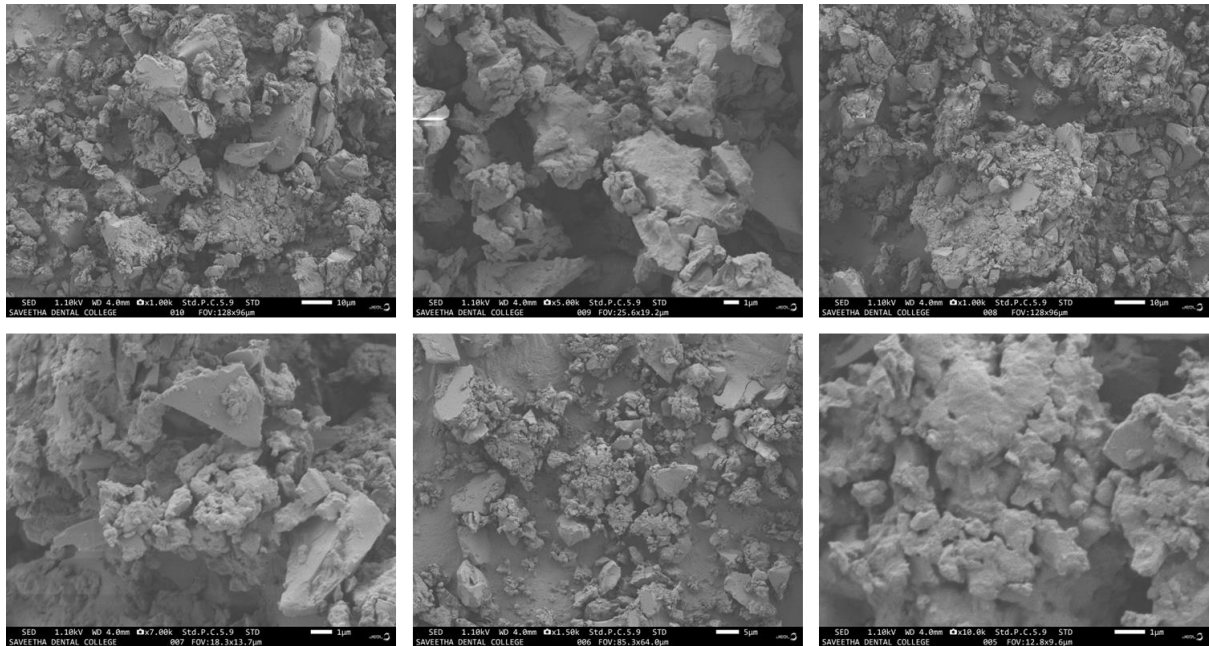


Figure 6. SEM Images of TiO₂ Nanoparticle Infiltrated Orthodontic Adhesive

The analysis of the TiO₂ nanoparticles infiltrated orthodontic adhesive by EDX showed the presence of titanium (Figure 7). The EDX profile contained a strong titanium dioxide signal along with other weak signals, which might be due to the biomolecules bound to the surface of the NPs. Although strong signals were due to titanium in the

nanoparticles, weaker signals for oxygen and chloride were due to other biomolecules. The signals might have been due to X-ray emission from carbohydrates/proteins/enzymes existing in the cell wall of the biomass. In the present study, TiO₂ nanoparticles infiltrated orthodontic adhesive and revealed a strong absorption spectrum at 4-5 keV.

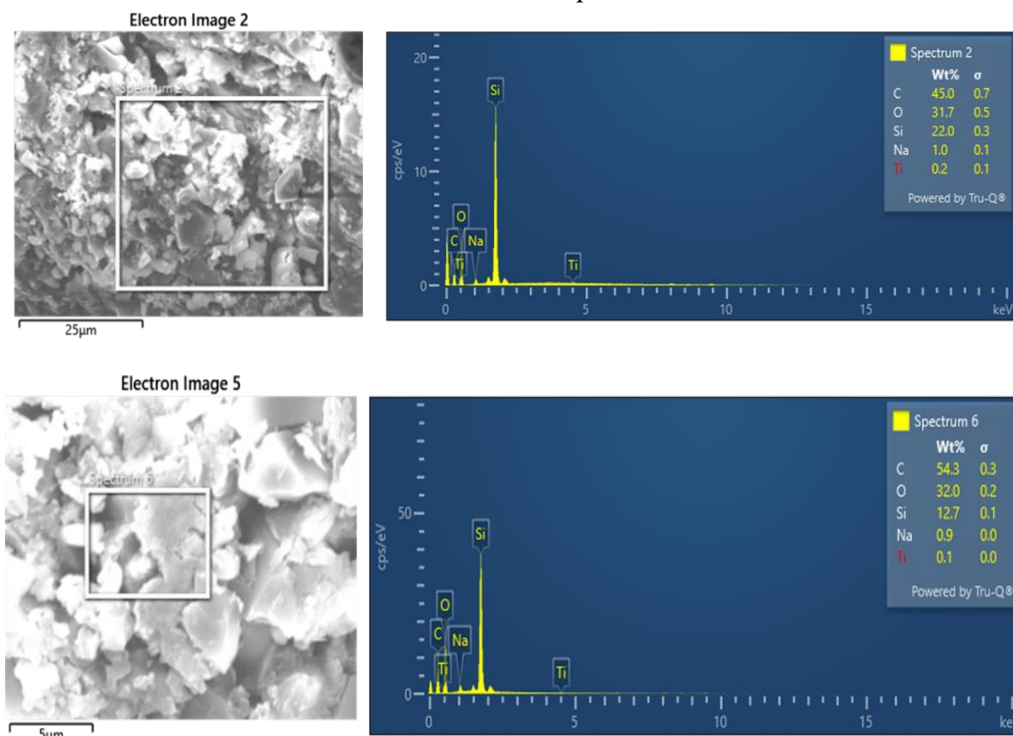


Figure 7. EDX Images of TiO₂ Nanoparticle Infiltrated Orthodontic Adhesive

Physical Properties

Colour Stability

The mean comparison of ΔE values is presented in Table 1. No statistically significant mean difference was observed between the two

groups ($P > 0.05$). Conventional orthodontic adhesive ($\Delta E = 1.71 \pm 0.80$) showed lower ΔE values when compared to TiO_2 infiltrated orthodontic adhesive (2.2 ± 1.1). ΔE values of both groups appear to be within the clinically acceptable range ($\Delta E < 3.3$).

Table 1. Comparison of Color Stability Values between the Study Groups

<i>Color Stability</i>	<i>Mean \pm SD</i>		<i>p value</i>
	<i>Group 1 (10)</i>	<i>Group 2 (10)</i>	
<i>ΔE value</i>	<i>1.71 \pm 0.80</i>	<i>2.2 \pm 1.1</i>	<i>0.248</i>

Independent t test

Surface Roughness

Comparison of mean R_a , R_q and R_z parameters showed no statistically significant

difference in surface roughness between ($p > 0.05$). No statistically significant difference was elucidated within the groups before and after thermocycling (Table 2).

Table 2. Mean Comparison of Surface Roughness among the Study Groups

<i>Surface roughness</i>	<i>Thermocycling</i>	<i>Mean \pm SD</i>		<i>p value</i>
		<i>Group 1</i>	<i>Group 2</i>	
<i>R_a (nm)</i>	<i>Before</i>	<i>0.23 \pm 0.07</i>	<i>0.47 \pm 0.21</i>	<i>0.004*</i>
	<i>After</i>	<i>0.25 \pm 0.06</i>	<i>0.41 \pm 0.11</i>	<i>0.001*</i>
<i>p value</i>		<i>0.428[#]</i>	<i>0.428[#]</i>	
<i>R_q (nm)</i>	<i>Before</i>	<i>0.32 \pm 0.10</i>	<i>0.63 \pm 0.28</i>	<i>0.005*</i>
	<i>After</i>	<i>0.36 \pm 0.09</i>	<i>0.54 \pm 0.15</i>	<i>0.004*</i>
<i>p-value</i>		<i>0.315[#]</i>	<i>0.498[#]</i>	
<i>R_z (nm)</i>	<i>Before</i>	<i>1.82 \pm 0.51</i>	<i>3.53 \pm 1.32</i>	<i>0.001*</i>
	<i>After</i>	<i>2.21 \pm 0.68</i>	<i>3.01 \pm 0.82</i>	<i>0.029*</i>
<i>p-value</i>		<i>0.111[#]</i>	<i>0.415[#]</i>	

* Independent t test; # Paired t test

Hardness

Table 3 shows the mean comparison of microhardness between two adhesives. There is a significant mean difference in microhardness between the adhesives ($p < 0.05$). However, no statistically significant difference was observed

within the adhesives (before and after thermocycling). Conventional orthodontic adhesive had a greater hardness value (HV) than TiO_2 -infiltrated orthodontic adhesive and the hardness value increased in both groups after thermocycling.

Table 3. Comparison of Mean Hardness between and Within the Groups

<i>Microhardness</i>	<i>Thermocycling</i>	<i>Mean ± SD</i>		<i>p value</i>
		<i>Group 1</i>	<i>Group 2</i>	
	<i>Before</i>	54.21 ± 6.70	44.64 ± 8.21	0.011*
	<i>After</i>	59.33 ± 15.4	47.19 ± 8.63	0.044*
<i>p value</i>		0.306#	0.294#	

* Independent t test; # Paired t test

Mechanical Properties

Compressive and Diametral Tensile Strength

Conventional and TiO₂ infiltrated Orthodontic adhesives showed no statistically significant difference in their compressive and

tensile strengths ($p > 0.05$). Conventional orthodontic adhesive had an increased compressive strength when compared to TiO₂-infiltrated orthodontic adhesive, while the result was vice versa for diametral tensile strength (Table 4).

Table 4. Mean Comparison of Compressive and Diametral Tensile Strength in MPA between the Study Groups

Mechanical properties	Mean ± SD		p value
	Group 1 (10)	Group 2 (10)	
Compressive strength (MPa)	202.39 ± 47.10	187.69 ± 38.73	0.456
Diametral tensile strength (MPa)	118.58 ± 22.71	125.14 ± 25.68	0.533

Independent t-test

Discussion

There is no literature explaining the efficacy of green synthesized TiO₂ NPs infiltrated orthodontic adhesives to prevent WSLs compared with conventional orthodontic adhesives. This in-vitro study investigated and compared the physical and mechanical properties of green synthesized TiO₂ NPs infiltrated orthodontic adhesives. The outcomes of physical properties such as colour stability and surface roughness of novel TiO₂ infiltrated orthodontic adhesives were significantly like that of conventional orthodontic adhesives. The hardness of conventional orthodontic adhesives was found to be significantly higher than that of

novel TiO₂-infiltrated orthodontic adhesives. Similarly, the mechanical properties of novel TiO₂-infiltrated orthodontic adhesives are significantly like conventional orthodontic adhesives.

As the present study is an in-vitro study, doubt may arise that thermal stresses may affect the physical and mechanical properties of orthodontic adhesives when used in the oral cavity. To overcome this problem, thermocycling, a laboratory procedure of exposing dental materials to temperature ranges which are like those occurring in the oral cavity is carried out for both conventional and novel TiO₂-infiltrated orthodontic adhesives [37, 38].

Scanning Electron Microscope

SEM images of *Eucalyptus globulus* leaf extracted TiO₂ NPs and infiltrated orthodontic adhesive showed uniform distribution which is smooth and spherical. The average nanoparticle size ranged between 20 – 70 nm which is inconsistent with the TiO₂ NPs prepared from jasmine flower and Aloe Vera whose average NPs size ranged between 32-48 nm [39, 40]. Decreased particle size is inversely proportional to the surface volume of the material. Thus, they easily bond to the tooth surface and penetrate bacterial surfaces and decompose them [41].

Energy Dispersive X Ray Analysis

EDX analysis of green synthesized TiO₂ NPs and TiO₂ infiltrated orthodontic adhesive showed strong titanium signals and weak oxygen and chloride signals with the absorption spectrum of TiO₂ NPs at 4-5 KeV. These strong and weak signals might be due to phytoconstituents from *Eucalyptus* leaf extract. This absorption spectrum is like the TiO₂ NPs synthesized from *Ocimum sanctum* leaf extract and *Sesbania grandiflora* leaf extract used to heal diabetic wounds [42, 43].

Fourier-Transform Infrared Spectroscopy

In the FTIR spectra showed a peak at 584.24 cm⁻¹ for bending mode and peak at 1101.46 cm⁻¹ for the stretching mode of TiO₂ NPs. This result is similar to the peak of TiO₂ NPs synthesized from *Ocimum sanctum* leaf extract was 590 cm⁻¹ which is used to heal diabetic wounds [42]. This peak at FTIR spectra of TiO₂ is due to the Ti – O -O bond [44].

Colour Stability

Green synthesized TiO₂ NPs infiltrated orthodontic adhesives from *Eucalyptus* leaf extract showed a color stability ΔE value of 2.2 ± 1.1 which is similar to the ΔE value of conventional orthodontic adhesives (1.71 ± 0.80). These ΔE values are up to the acceptable

range as reported in an in-vitro study of composite resins [45].

Surface Roughness

Results of Ra, Rq and Rz values of both the orthodontic adhesives before and after thermocycling showed no significant difference. In contrast to this result, an in-vitro study that evaluated the surface roughness of TiO₂ NPs to the heat cured denture-based resin reported increased mean surface roughness [46].

Hardness

The microhardness of two orthodontic adhesives showed a significant greater hardness for conventional orthodontic adhesives. Thermocycling has not affected the hardness of both orthodontic adhesives. A study on the hardness of TiO₂ nanotubes added to polycrystalline hydroxyapatite bio ceramic from bovine bones showed increased hardness [47]. Although chemically extracted TiO₂ nanoparticles increased the hardness, the constituents of *Eucalyptus* leaf extract would have reduced the hardness.

Compressive and Tensile Strength

Conventional orthodontic adhesives showed significantly increased compressive strength while novel TiO₂ infiltrated orthodontic adhesives had significant high tensile strength. An in-vitro study which evaluated 1% TiO₂ NPs infiltrated orthodontic adhesives showed high compressive and tensile strength [48]. As mentioned earlier, the constituents of *Eucalyptus* leaf extract would have reduced the compressive strength.

Strength and Limitations

This is the first study to evaluate the physical and mechanical properties of green synthesized TiO₂ nanoparticle-infiltrated orthodontic adhesives. The strength of the in-vitro study is that its design completely simulates in-vivo oral cavity temperatures by thermocycling. Though thermocycling is done to simulate the oral

cavity temperatures, factors such as patients' dietary habits and oral hygiene which may influence the study outcomes are not considered. Further, the influence of saliva on the properties of green synthesized TiO₂ nanoparticle infiltrated orthodontic adhesives has not been assessed. Thus, in-vivo studies to assess their clinical applicability and efficacy are needed. Also, identification of proper concentration of TiO₂ NPs is recommended.

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Conclusions

Mean color stability and surface roughness was similar in both orthodontic adhesives. High mean hardness was recorded in conventional orthodontic adhesives. The results of mechanical properties showed greater compressive strength for conventional orthodontic adhesives and greater tensile strength for novel TiO₂ NPs infiltrated orthodontic adhesives. Overall, the present in-vitro findings showed improved physical and mechanical properties of novel TiO₂ NPs infiltrated orthodontic adhesives.

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