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Original article

Synthesis of Poly (methyl methacrylate)-Titanium Dioxide Nanoparticles, Characterization, and Antibacterial Activity

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Abstract
In recent years, nanoparticle-enhanced polymers have garnered significant attention due to the increasing demand for clean, non-toxic chemicals, low-cost methods, environmentally friendly solvents, and renewable materials. In the present study, titanium dioxide nanoparticles (TiO_2NPs) were biosynthesized chemically and biologically using an extract from the stem of the *Balanites aegyptiaca* plant. The surface of the nanoparticles was enhanced by implanting PMMA onto the nanoparticle surface to improve the polymer's physical and mechanical properties. The titanium dioxide nanoparticles were characterized using FTIR, UV, XRD, and HR-TEM methods. X-ray diffraction revealed the presence of TiO_2NPs, confirmed by an occurrence peak at 25.28, which corresponds to anatase 101. High-resolution scanning electron microscopy (HR-TEM) results showed that the synthesized titanium dioxide nanoparticles were spherical, with individual nanoparticle sizes, as well as some aggregates, ranging from 1 to 9 nm. The antibacterial activities of biosynthesized nanoparticles (titanium dioxide nanoparticles) were investigated using the disc method. The polymer enhanced with titanium dioxide nanoparticles exhibited significant antibacterial activity.

Keywords. Antibacterial Activities, Balanites Aegyptiaca (L) Delile, Tio_2, PMMA, TTIP.

Introduction

In recent years, there has been a surge of interest in nanomaterials research. Nanotechnology is a powerful technique that manipulates materials at the nanometer scale. Titanium dioxide (TiO_2), non-toxic ceramic nanoparticles, are renowned for their physicochemical properties, including antibacterial efficacy, and for their applications in electronics, medicine, and environmental remediation [1]. Available in anatase, rutile, and brookite crystalline phases, TiO_2 nanoparticles enhance the functionality of polymer-based materials. Although polymers are widely used, their limitations in biological, electrical, and antimicrobial applications can be addressed by incorporating nanoparticles like TiO_2, thereby expanding their scope [2,3]. fields, including nanotechnology, biotechnology, materials science, physics, and chemistry. Beyond the diverse chemical and physical methods employed to synthesize nanoparticles. Nanomaterials, ranging in size from 1to100 nanometers, possess unique chemical, physical, and electrical properties. They have wide-ranging applications in medicine, polymer surface enhancement, microbiology, chemistry, engineering, low-cost catalysts, cytotoxicity studies, and other fields.

Previous studies have shown that using chemical reducing agents leads to the formation of large nanoparticles, consuming excess energy. Furthermore, chemical methods have been reported to be environmentally unfriendly and to have side effects. Chemically synthesized nanoparticles have also been known to be less stable and to increase agglomeration [4,5]. Therefore, researchers need to develop environmentally friendly methods that produce dispersible, stable nanoparticles of a suitable size while consuming less energy. Nanotechnology also plays a significant role in improving healthcare, safety, and environmentally friendly practices. Researchers have turned to green synthesis methods for preparing nanomaterials due to their ease of preparation, environmental friendliness, high efficiency, and lower cost [6,7]. Green synthesis is an exciting approach in materials science [7,8].

In recent decades, microorganisms and plant extracts have been used to prepare nanocomposites, and one such material used as a nanocomposite is titanium dioxide. Titanium dioxide (TiO_2) is stable in aqueous media and accepts both alkaline and acidic solutions. Titanium dioxide nanoparticles have been used in photocatalysts, food coloring, cosmetics, and pharmaceuticals [9]. Titanium dioxide nanoparticles can react with sodium hydroxide (OH) and oxygen gas (O_2), which are adsorbed onto the surface to produce oxygen and free hydroxyl radicals [10]. Due to the increased surface area of the nanoparticles, their interaction area with pathogenic bacteria is also increased, making them suitable as an antimicrobial agent. Their small size allows them to easily penetrate the bacterial surface, enabling them to damage the bacteria [11]. Titanium dioxide nanoparticles are of interest to many researchers due to their low cost. Previous studies have reported on the use of titanium dioxide in the life sciences [4].

It was reported that its antibacterial properties can be enhanced by adding it to the polymer matrix to increase its surface area. This study aimed to investigate the synthesis of titanium dioxide particles from the Balanites aegyptiaca plant and their dispersal on the surface of Polymethyl methacrylate after a demineralization process. The phytochemicals present in Balanites aegyptiaca stem extracts include alkaloids, coumarins, flavonoids, tannins, terpenoids, glycosides, compounds, steroids, essential oil, and saponins [12]. These phytochemicals are responsible for the reduction of titanium tetraisopropoxide to

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titanium dioxide nanoparticles, which then disperse on the polymer surface to improve its mechanical and physical properties. The structural, morphological, vibrational, and optical properties of the titanium dioxide nanoparticles were analyzed. In addition, the antibacterial activity against Gram-positive and Gram-negative strains was tested. The various processes for synthesizing titanium dioxide nanoparticles were also studied in detail. Polymethyl methacrylate (PMMA) was used in this research due to its wide and distinctive applications. It is considered a lightweight polymer with high rigidity and serves as an economic alternative to some polymers. It is also considered safe for medical applications after making certain improvements to its physical and mechanical properties by incorporating it onto the surface of nanoparticles.

Methods

Instrumentation

The functional groups of the plant were identified, and the presence of titanium particles was confirmed using an FTIR spectrometer (Thermo, Nicolet 380) equipped with Omnic software version 7.1. The spectral range of 400 to 4000 cm^{-1} was analyzed using KBr. X-ray diffraction (XRD) analysis showed that the green-synthesized titanium dioxide exhibited peaks corresponding to the anatase phase at 25.28°, 37.5°, 47.5°, and 55°, which is consistent with JCPDS 21-1272. Particle sizes were estimated using the Scherrer equation. Pure poly methyl methacrylate displayed an amorphous pattern. UV-Vis spectroscopy revealed strong absorption ranges for the titanium dioxide nanoparticles around 390-400 nm, corresponding to the anatase phase. HR-TEM images showed spherical nanoparticles ranging from 1 to 9 nm, well-dispersed within the PMMA matrix.

Materials and methods

PMMA (Mw 550,000 g mol−1) was purchased from Alfa Aesar, USA. Styrene was purchased from China, and 3-Aminopropyltriethoxysilane ($C_9H_{23}NO_3Si$) was purchased from High Purity Laboratory Chemicals, India. Titanium tetraisoperoxide (TTIP) was purchased From Cisco Research Laboratories, India. Ethanol and toluene were purchased from Molychem, India. Methanol and dimethyl sulfoxide were purchased from Finar Chemicals, India. Deionized water, NaOH, and HCl were purchased from Gujarat Natural Fertilizers Limited, India. All chemicals were used without further purification. Benzoyl peroxide $(C_6H_5.CO.O)_2$ was purchased from China. Tetrahydrofuran $(CH_2)_4O$.

Plant material

Balanites Aegyptiaca is a type of tree, classified as both a member of Zygophyllaceae and Balanitaceae. It is found in Africa, and parts of the Middle East, Balanites Aegyptiaca is a type of tree, classified as a member of both Zygophyllaceae and Balanitaceae, and has many common names for this plant, and it was used by the Pharaohs in many fields of an aesthesia, wound cleaning, treatment of constipation, and insulin is extracted from it as in)Figure 1)[13].

Figure 1. (The plant).

Preparation of plant extract

The plant was collected from Libya/Sebha/2023. The stems were washed thoroughly with rainwater and dried in the sun for a full day. They were then ground and sieved, and the water was used as an extract.

Synthesis of titanium dioxide nanoparticles by the sol-gel method

Minor modifications were made to the titanium dioxide nanoparticles obtained from the previously mentioned references. 4 mL of titanium tetraisoperoxide was dissolved in 35 mL of ethanol solution with

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continuous stirring for 30 minutes. Then, 5 mL of distilled water was added to form a dispersion medium. The product was shaken for 60 minutes, and the solution was transferred to an oven at 100 °C for 24 hours. The solution was then cooled to room temperature and mounted at 650 °C for 2 hours. The resulting titanium dioxide nanoparticles were collected. Further characterization follows [14].

Green synthesis of TiO_2 nanoparticles using Balanites Aegyptiaca

TiO_2 NPs were synthesized using a facile green synthesis. The method of Balanites aegyptiaca plant extract acts as a reducing/maximizing agent. The Balanites aegyptiaca plant extract was purchased from the local market in Sebha / Libya. The extract was prepared by adding 30 grams of the Balanites aegyptiaca plant extract, which was purchased from the local market in Sebha / Libya.to 100 ml of distilled water and boiling the mixture on the stove for 30 minutes. Then the aqueous solution was made. It was filtered and stored for further testing. Take 4 ml of titanium tetraisoperoxide (TTIP) in a 50 ml beaker and add 20 ml of the Balanites aegyptiaca plant extract drop by drop to the above TTIP solution. The solution was stirred for 1 h at room temperature. The color of the solution changed from pure white to reddish brown. The color change confirms the formation of titanium dioxide nanoparticles. And then the solution was filtered and dried at 100 °C for 24 h. The dried samples were then baked in a Muffle oven at 650 °C for 2 h [12, 13].

Synthesis and surface modification of porous fillers of PMMA-TiO_2, Nanocomposites

A mixture of 2.0 g of PMMA monomers, 0.03 g of nano-greens, and 0.1 g of benzoyl peroxide was subjected to ultrasonication for 1 hour in the presence of 3 mL of methanol at 50 μm. The mixture was then incubated at 60°C for 24 hours to promote in situ free radical polymerization. After polymerization was complete, the product was poured into excess methanol and stirred for 15 minutes. It was then washed several times with methanol and hot water before being filtered and dried in a container. The mixture was then placed in an oven at 80°C overnight (Figure 2).

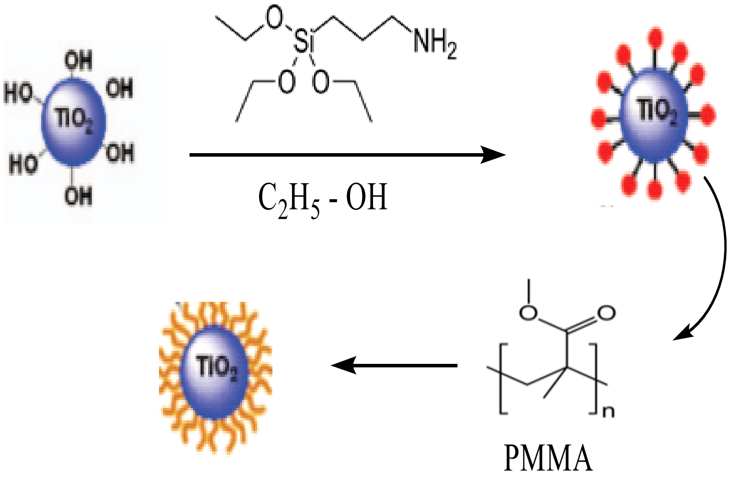

Figure .2 The synthetic route of PMMA-TiO_2 nanocomposites.

Fourier Transform Infrared Spectroscopy

The binding properties of titanium dioxide (TiO_2) nanoparticles synthesized from the extract of the heglig stem were investigated using Fourier transform infrared spectroscopy (FTIR). FTIR measurements were performed on a Proker Vertex 70 spectrometer. Powdered and dried titanium dioxide nanoparticles were granulated using potassium bromide (KBr) at a 2:4 ratio. Spectra were recorded in the wavenumber range of 400 cm^{-1} to 4000 cm^{-1} and analyzed by subtracting the purified potassium bromide spectrum.

UV–Vis absorbance spectroscopy

The UV–Vi's absorption spectra of the TiO_2NPs and TF-TiO_2NPs were chronicled at room temperature by Pharma spec UV-1700 (UV–Visible spectrophotometer. The scanning range of the samples was 200 - 800 nm in a resolution of 1 nm at a scan speed of 200 nm/min.

XRD

X-Ray Diffraction (XRD) is an analytical technique that is used to identify the phase formation and crystallinity of TiO_2NPs. XRD analysis was conducted via the PW 1148/89-based X-ray diffractometer employing nickel-filtered Cu α radiation (k = 1.54056 Å) at 298 K. The tool was armed with graphite monochromatic and then functioned at 30 mA and 40 kV. The diffractogram was attained in the 2θ range of 10–80°. The obtained raw data were processed in Origin 8 software and compared with the standard JCPDS database card no. 21-1272 (Anatase) and 21-1276 (Rutile)

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HR-TEM

The resulting PMMA-TiO_2 particles were characterized using high-resolution scanning electron microscopy (HR-TEM). A small amount of the sample was distributed in dry ethanol, sonicated for 10 minutes, and the resulting solution was then moistened onto a carbon-coated copper grid. The grid was left to dry under ambient conditions and then mounted for analysis using high-resolution scanning electron microscopy (HR-TEM).

Antibacterial activity

The antibacterial activity of TiO_2, PMMA, and PMMA-TiO_2 nanoparticles was evaluated using the standard disk diffusion method, Bacterial suspensions of Acinetobacter baumannii and Bacillus cereus were cultured on separate nutrient agar (NA) plates using an L-shaped applicator. 6 mm diameter Whatman filter paper discs (No. 1) were individually soaked in 20 µL of a 10 mg/ml PMMA-TiO_2 nanoparticle solution in tetrahydrofuran (THF). The discs were evaporated and then impregnated onto the plates. Samples were incubated for 24 hours at 37°C. Areas of inhibition were defined with a ruler, and the average value for each organism was recorded and expressed in millimetres [4].

Results and discussion

Characterization of PMMA-TiO₂ nanocomposites

The functional groups and chemical composition of the synthesized titanium dioxide (TiO2) nanoparticles were analyzed via Fourier-transform infrared (FT-IR) spectroscopy. As shown in (Figure (3, a broad absorption band centered at 3430 cm^{-1} corresponds to the O-H stretching vibration, likely originating from adsorbed moisture or hydroxyl groups. The band at 1535.18 cm^{-1} is attributed to the C-H bending vibration, while the sharp peaks at 1266 cm-1 and 1170 cm-1 signify the presence of alcohol functional groups and Ti-O-C stretching modes, respectively. In Figure 4, the spectrum reveals a peak at 1627 cm^{-1} associated with the stretching and bending vibrations of C=C bonds. A broad signal at 3477 cm^{-1} indicates the presence of overlapping N-H and C-H vibrations. Furthermore, the characteristic vibrational modes of the TiO2 crystalline lattice are observed at 624 cm^{-1}. The reappearance of the Ti-O-C signature at 1170 cm^{-1} further confirms the chemical environment of the nanoparticles [4, 15].

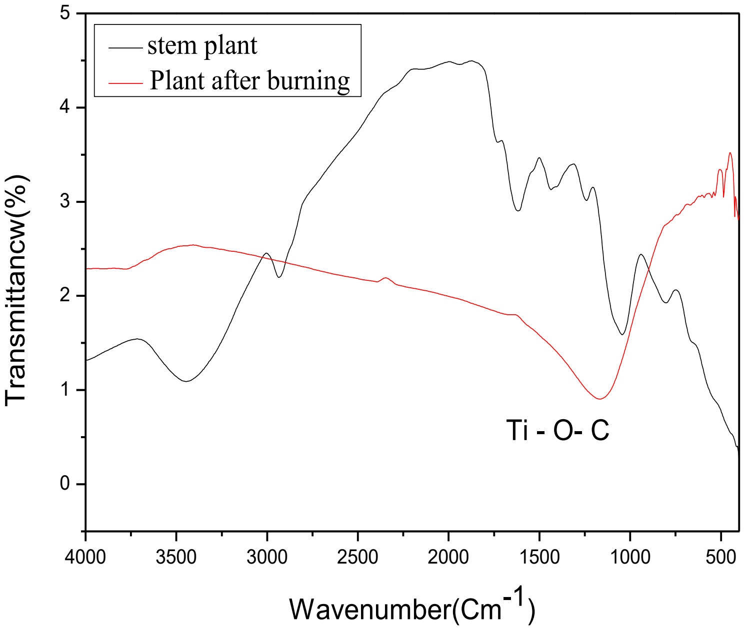

Figure 3. Shows FT–IR spectra of green synthesis, TiO₂ nanoparticles.

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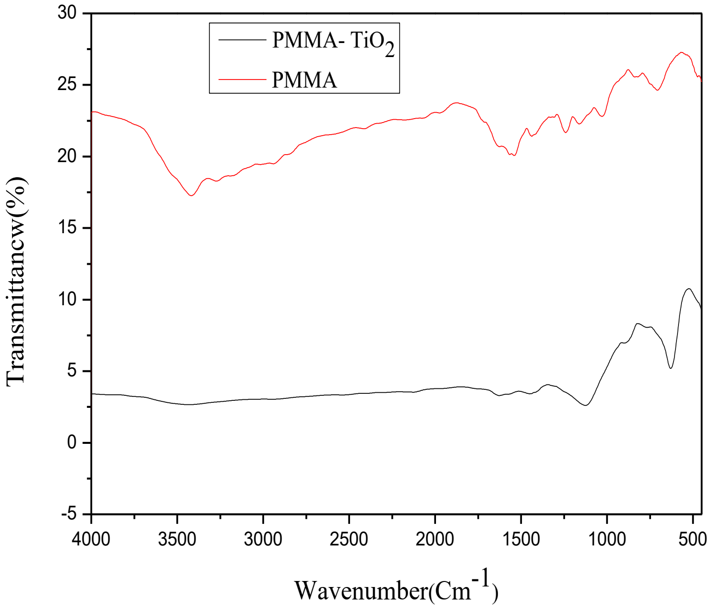

Figure 4. Shows FT–IR spectra of Polymer PMMA / PMMA-TiO_2 nanocomposite.

XRD

PAN analytical X-Ray Diffraction equipment model X'Pert PRO with Secondary Monochromatic, Cu-radiation (λ1.542Å) at 45 K.V., 35 M.A. and scanning speed 0.04 °/sec. were used. The diffraction peaks between $2\theta = 2^{o}$ and 66 θo, corresponding spacing (d, Å) and relative intensities (I/I^{o}) were obtained. The diffraction charts and relative intensities are obtained and compared with ICDD files.

The XRD pattern of green synthesized TiO_2NPs and PMMA-TiO_2 nanocomposites was displayed in (Figure 5, 6and 7). The sample validated a high crystallinity level with diffraction angles of 25.28, 37.5, 47.5, and 55, which confirms the formation of well-crystalline titanium with anatase phase, and it is well evident with the standard JCPDS database (# 21-1272). The diffraction angle perceived at 25.28 is associated with the (101) crystallographic plane of TiO_2 anatase only. The particle size estimation was performed by Scherer's equation. However, in Figure 8, nothing is shown because it contains pure PMMA [4,15].

$$d = \frac{0.9\lambda}{\beta\cos\theta}$$

where nanoparticles mean diameter is denoted by d, wavelength of X-ray radiation source denoted by λ, angular peak at the diffraction angle θ

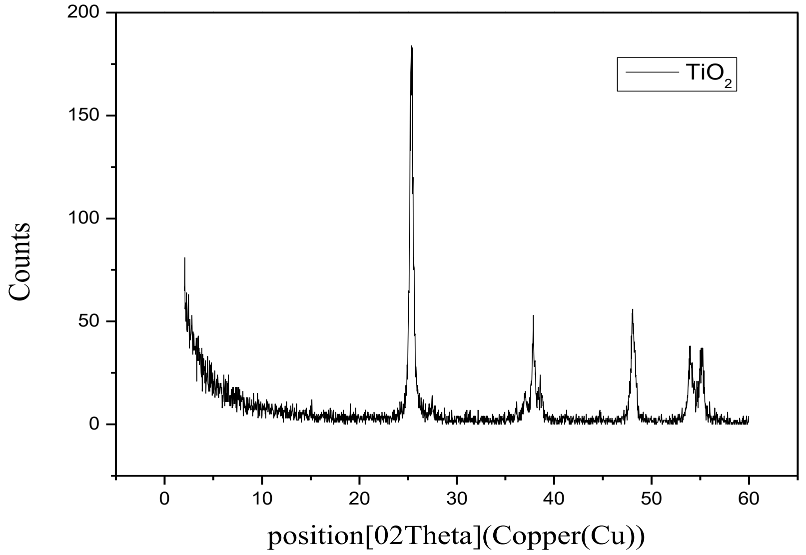

Figure 5. XRD pattern of green synthesized TiO_2NPs and from Balanites aegyptiaca extract.

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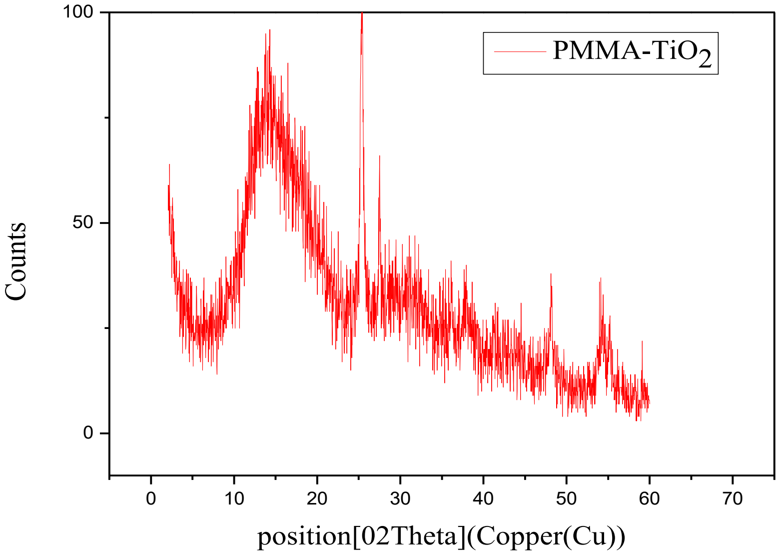

Figure .6 XRD pattern of synthesized PMMA-TiO₂ nanocomposites.

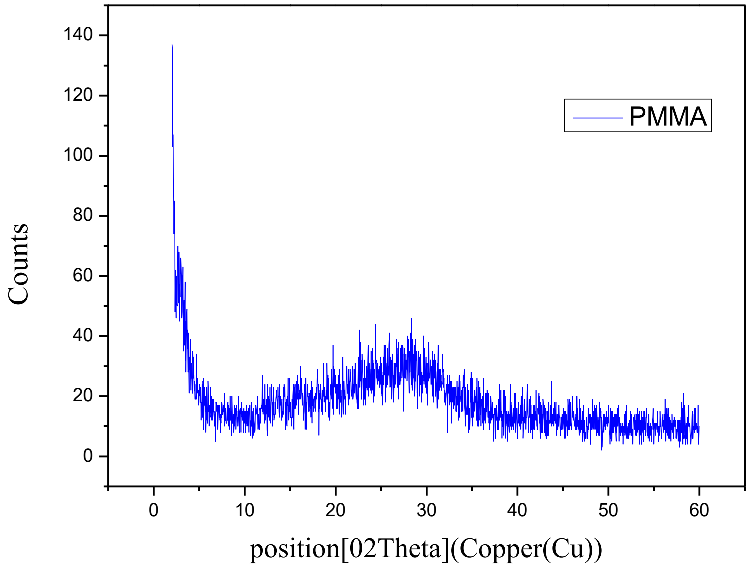

Figure 7. XRD pattern of pure PMMA.

Titanium dioxide nanoparticles synthesized using the stem extract of the *Balanites aegyptiaca* plant (TiO_2 nanoparticles) exhibited strong absorption)0.95) astronomical units) at a wavelength of 390 nm, while titanium dioxide nanoparticles exhibited strong absorption (0.94 astronomical units) at a wavelength of 400 nm (Figure 8). The results of the current work are consistent with previous reports, particularly those concerning the stem extract of the *Balanites aegyptiaca* plant, which indicate the presence of a titanium dioxide band at wavelengths of 320–400 nm [4,16]. High-resolution scanning electron microscopy (HR-TEM) images revealed spherical, multi-dispersed TiO2 nanoparticles, with the average size of the dispersed TiO2 NPs within the polymer matrix being 1 and 9 nm (Figure 9). The electron microscopy image clearly shows that the individual nanoparticles formed were nearly spherical [28]. This research finding offers hope for reducing the size and improving the efficiency of the plant synthesis process for TiO2 nanoparticles produced from anatase

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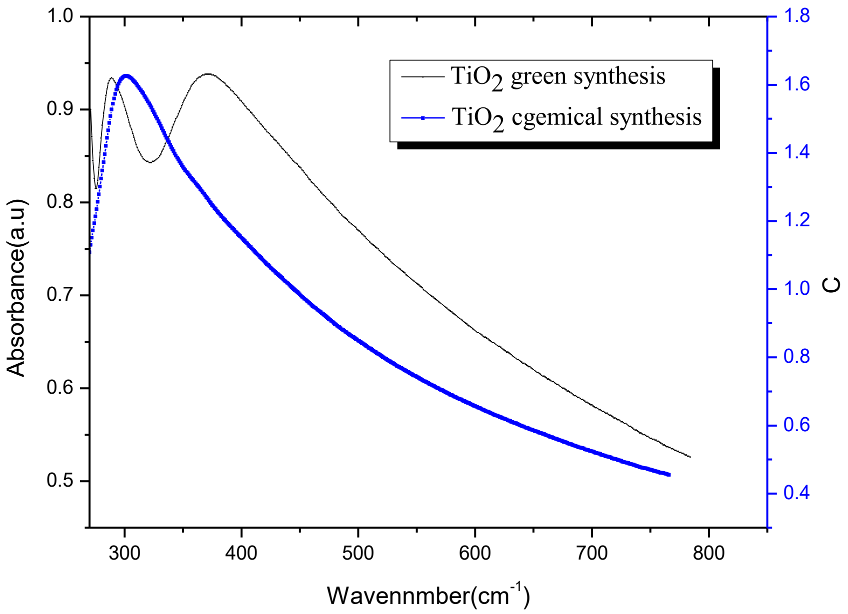

Figure 8. Ultraviolet–visible analyses at 200–800 nm range for chemically synthesized TiO_2 with (green synthesis-TiO_2 NPs)

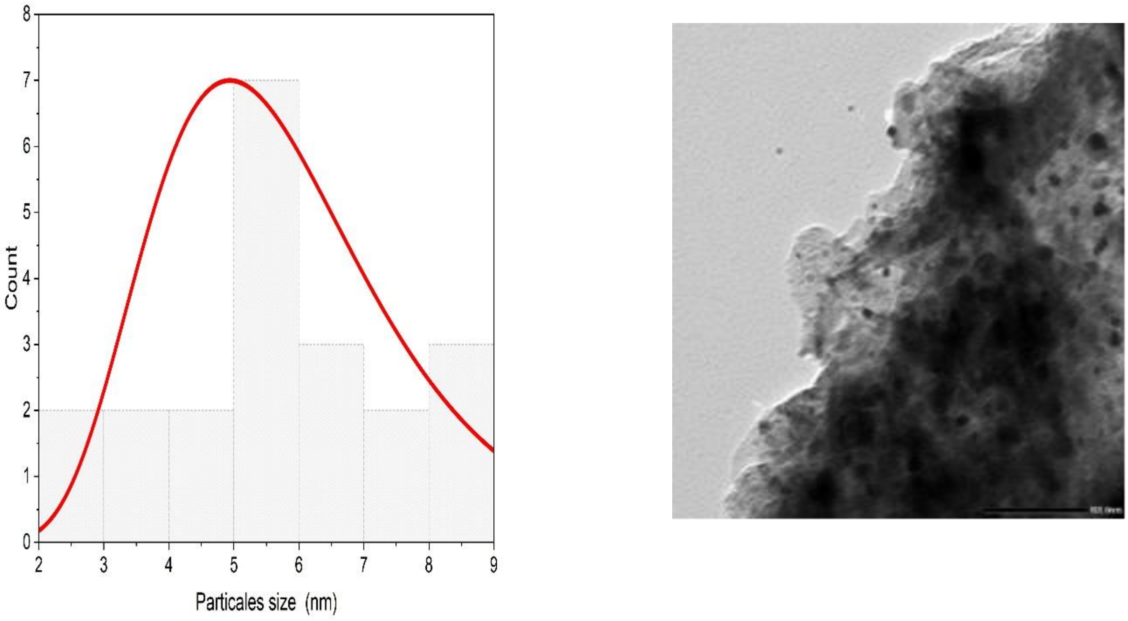

Figure 9. Titanium dioxide particles dispersed within the PMMA-TiO_2 polymer (HR TEM).

Antibacterial activity

The aim of this work was to evaluate the antimicrobial activity of titanium oxide nanoparticles dispersed in a polymer matrix and the dependence of this activity on the selected microbial species: *Bacillus cereus* and *Acinetobacter baumannii*. PMMA-TiO_2

nanoparticles exhibited antimicrobial activity compared to TiO_2 and pure PMMA nanoparticles against *Acinetobacter baumannii* (23 mm) and *Bacillus cereus* (12 mm) using PMMA-TiO_2 (Table 1) at a concentration of 700µm. They showed only a slight effect when using pure PMMA and TiO_2, as shown in (Table 1 and Figure 10 and11).

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Table .1 Mean zone of inhibition of synthesized PMMA, TiO_2, and PMMA-TiO_2 nanocomposites

C/A.bua	C/B.C	B/A.bau	B/B.C	A/A.bau	A/B.C	cone
-	3mm	10mm	-	14mm	-	c-trol
-	-	9mm	-	15mm	10mm	100µm
-	-	9mm	-	17mm	12mm	500µm
-	-	11mm	10mm	23mm	16mm	700µm


A B **C**

Figure 10. Study of the antibacterial activity of sample A (PMMA-TiO_2), B (PMMA) and C (TiO_2) at different concentrations for bacteria (B.C).

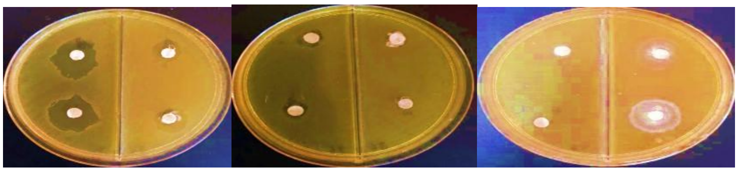
A **B** **C**

Figure 11. Study of the antibacterial activity of sample A (PMMA-TiO_2), B (PMMA), and C (TiO_2) at different concentrations for bacteria (A. baumannii).

Conclusion

With the significant advancements in polymers enhanced by the dispersion of nano-oxides on their surface, there has been considerable interest in improving polymer properties using titanium oxides prepared from plant extracts and applying them to bacteria. Nanotechnology is rapidly transitioning from laboratories to large-scale industrial production, and nanomaterials are being utilized in all biomedical applications. The current method promotes the pure biosynthesis of titanium nanoparticles using a stem extract of the stem *Balanites aegyptiaca* plant extract. The fully synthesized titanium dioxide nanoparticles were characterized using Fourier transform infrared (FTIR), ultraviolet-visible (UV-Vis), and XRD techniques. This state-of-the-art method offers faster and purer synthesis of titanium dioxide nanoparticles in a shorter and more reliable timeframe. The synthesized nanoparticles were found to exhibit excellent antibacterial activity against the tested microorganisms, paving the way for the development of a new variety of materials with antibacterial activity. The bio-reduction of metals and the enhancement of polymer properties will be a boon for developing a green, healthy, harmless, and environmentally sound method for producing metal nanoparticles. This could lead to economically viable and environmentally friendly applications for drug delivery, the treatment of various infectious diseases and cancers, commercial devices, sensors, and other electronic and medical applications.

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