**GREEN SYNTHESIS OF TITANIUM DIOXIDE NANOPARTICLES USING *MELIA AZEDARACH* LEAF EXTRACT AND EVALUATION**

**Abstract**

The present work reported the green synthesis characterization of (TiO2) Titanium dioxide nanoparticles the synthesis of TiO2 has been produced by the sol gel process with aqueous *Melia Azedarach* leaf extract. Synthesized nanoparticles were characterized with the aid of the usage of and verify the physical pattern by means of SEM (Scanning Electron Microscopy), TEM (Transmission Electron Microscopy), XRD (X-Ray diffraction analysis), UV(Vi’s spectroscopy), FTIR (Fourier-rework infrared spectroscopy) and EDAX (Energy-dispersive X-ray analysis). X-Ray diffraction analysis (XRD) research to be taken to expose the crystalline nature of Titanium dioxide nanoparticles. Transmission Electron Microscopy and (SEM) pix to expose the shape and size. SEM (Scanning Electron Microscopy) analysis shows the small spherical nanoparticles with a size ranged from 25nm to 87 nm. TEM (Transmission Electron Microscopy) analysis shows the size in the range of 15–45 nm. XRD (X-Ray diffraction analysis) analysis shows that were observed at 25.36, 26.54, 37.05, 37.78, 38.54, 48.12, 54.02, and 55.04° can be assigned to the crystal 101, 110, 04, 103, 2 lines of the anatase and rutile structures of the synthesized titanium dioxide nanoparticles. UV(Vi’s spectroscopy) UV–Vis spectra of the green synthesized TiO2 NPs were recorded using a dual-beam spectrophotometer (Shimadzu UV-2450 PC) in the range of 200–900 nm. EDAX (Energy-dispersive X-ray analysis) Chemical composition and crystalline nature of the green synthesized TiO2 were obtained by the EDAX analysis. Generally, it is known that TiO2 display typical optical absorption peaks 4.3-5.3Ke V due to surface plasmon resonance.

**Keywords:** Titanium dioxide nanoparticles (TiO2)-(NPS), Green synthesis, *Melia Azedarach* and Multiple applications of Physics.

**Introduction**

Many academics have been working on creating long-lasting and ecologically friendly nanoparticles during the past few years. Emerging industries are greatly impacted by nanoparticles (NPs) and nanotechnologies, which are becoming more and more restricted in their applications across a variety of fields including mechanical systems and biological sciences. Due to titanium dioxide (TiO2) NPs' exceptional durability, strong electrical conductivity, nontoxicity, capacity to absorb UV radiation, and antibacterial activity, studies have grown interested in the size and form of these particles. These qualities also make TiO2 NPs a suitable material for nanostructures. Nanoparticles are created by physical, chemical, and biological processes; despite their small size, they have a huge surface area (Prammitha Rajaram *et al*.,2023).

Metal nanoparticles such as Ag, Au, MgO, CuO, Al, TiO2, etc., which are effective against various drug-resistant bacterial, viral and fungal strains have received attention in recent years (Rai and Bai, 2011). TiO2 nanoparticles are more remarkable nanomaterials due to their photocatalytic properties, chemical stability and non-toxicity. It has huge applications in the cosmetic industry, solar cells, electrochemical devices, pollution control and antibacterial coatings (Amarnath *et al*., 2013). In recent years, the synthesis of nanoparticles using a green approach has increased. There are reports on the synthesis of TiO2 nanoparticles using various plant extracts (Nasrollahzadeh and Sajadi, 2015, 2016; Rostami-Vartooni *et al*., 2016; Nasrollahzadeh *et al*., 2016; Maham *et al*., 2017).

The bioactivity of such an herbal plant is mainly due to the occurrence of a high level of limonoid compounds in addition to other phenolic acids and flavonoid glycosides, especially rutin (Dougnon, 2022). On the other hand, *M. azedarach* is native to Africa, Asia and northern Australia and is commonly used as an antiparasitic and antifungal agent with significant free radical scavenging activity (Maciel *et al*., 2006, Farag *et al*., 2011). The leaves of *M. azedarach* L. contain significant amounts of nimbolinin-type limonoids that have various therapeutic effects, including antimicrobial, antioxidant, and anticancer potential (Kanwal *et al*., 2011).

There are many methods for the synthesis of TiO2 NPs, such as batch and semi-batch two-step mixing method or microemulsion method. But the sol-gel method is the simplest and most effective bottom-up approach. The word sol-gel is made up of two words, sol and gel. The word sol means a colloidal solution formed in a continuous liquid, while the word gel means a solid molecule dispersed in a liquid. The chemical solution is treated as a catalyst. There are many titanium precursors, including TiCl3, Ti (OCH(CH3)2)4 (TTIP), TiCl4, and Ti (OBu) (Ramesh, 2013). TiO2 NPs help result in cytotoxicity to the tumor cells, that allows you to assist reduce the cancerous activities among all patients of all age agencies (Zhangjian Chen *et al*.,2020).

**2.Experimental**

**2.1. Materials**

Healthy leaves of *Melia Azedarach* were collected from Coimbatore district. Tamil Nadu. India. For antimicrobial activity, bacterial pathogens viz. *E. coli*, *Klebsiella pneumoniae*, *Bacillus subtilis*, *Staphylococcus aureus* and *Salmonella typhi* were obtained from PSG-Postgraduate Institute of Medical Education and Research (PSG-PGIMER), Coimbatore district. Tamil Nadu. India.

**2.2. Preparation of *Melia Azedarach*** **leaf broth**

Briefly, 20 g of finely chopped fresh leaves of *Melia Azedarach* were thoroughly washed with distilled water and then boiled in 100 mL of sterile doubly distilled water for 30 min in a water bath (Fig.1.). The resulting extract was then filtered to remove particles and stored at 4°C for further use.

  

**a**

**b**

**Fig:1. *Melia azedarach* leaves (a) and *Melia azedarach* powder (b)**

**2.3. Green synthesis of TiO2 nanoparticles**

Green synthesis of TiO2 nanoparticles was performed as previously described by Krishnasamy *et al*., (2015) with modifications. Briefly, 10 mL of *Melia Azedarach* leaf broth was added to 90 mL of 5 mM aqueous titanium dioxide for nanoparticle synthesis. The titanium dioxide nanoparticles were purified by repeated centrifugation at 7826 x g for 20 min using doubly distilled water and the pellet was dispersed in deionized water. Finally, heat treatment was performed at 60 °C for 1 h and stored for further use. A stock solution was prepared by suspending the nanoparticles in methanol to give a final conc. 10 mg/ml. The stock solution was sonicated for 15 min and each assay was performed within 1–2 h after sonication. The suspension was kept at 4°C.

**2.4. Sol-Gel Process**

The sol-gel process is carried out at low temperatures (usually less than 100 °C) and in the liquid state. The final product is of course a solid, and these solids are formed as a result of a polymerization process that involves the formation of M-OH-M or M-O-M (where M represents a metal atom) between the metal atoms in the raw materials. The synthesis of aerogels using the sol-gel process consists of two steps, which are as follows (Wang *et al*., 2018, Zhang *et al*., 2016, Chen *et al*., 2021 and Ji *et al*., 2020): (I). The first stage involves the formation of individual colloidal solid particles with dimensions of nanometers. (II). The second stage involves the association of colloidal particles in a solvent to form a gel.

**2.5. Sol-gel route**

All reagents used were of analytical grade and no further purification was performed before use. The sol-gel synthesized TiO2 was obtained from titanium isopropoxide (TTIP) was dissolved in absolute ethanol and distilled water was added to the solution in a molar ratio of Ti:H20=1:4. Nitric acid was used to adjust the pH and to limit the hydrolysis process of the solution. The solution was stirred vigorously for 30 min to form the salts. After aging for 24 hours, the sols were transformed into gels. In order to obtain nanoparticles, the gels were dried at 120 °C for 2 h to maximally evaporate water and organic material. After that, the dry gel was sintered at 450 °C for 2 h, and then the desired nanocrystalline TiO2 was obtained.

**2.6. Characterization of green synthesized TiO2-NPs**

**2.6.1. UV–visible spectroscopy**

UV-vis spectroscopy is based on the adsorption mechanism of spectroscopy. The principle of UV-visible spectroscopy relies on the interception of ultraviolet or visible light by chemical compounds that produce distinct spectra. This mechanism operates under the UV-visible spectrum at a wavelength of 200–800 nm. Different metal nanoparticles operate at different wavelengths ranging from 2 to 100 nm. A wavelength of 250–400 nm is commonly used to characterize nanoparticles. A graph is made between absorbance and wavelength to see the UV-vis spectrum of the synthesized nanoparticles.

**2.6.2. X-ray diffraction analysis (XRD)**

X-ray diffraction analysis of TiO2 nanoparticles was performed using a Smart Lab 9 kW rotating anode X-ray diffractometer (Rigaku Corporation) at diffraction angles (29) from 200 to 800 (scan range) at a scan rate of 20/min.

**2.6.3. Scanning Electron Microscopy (SEM)**

The morphology and size and synthesized TiO2 nanoparticles were analyzed by field emission scanning electron microscope (NOVA NANOSEM 450, FEI, USA).

**2.6.4. Energy-dispersive X-ray analysis (EDAX)**

Energy-dispersive X-ray analysis was used to obtain the composition and the elemental analyses of the nanoparticles. The EDAX detector attached to the SEM instrument.

**2.6.5. Fourier-rework infrared spectroscopy (FTIR)**

The FTIR spectroscopy study of the synthesized nanoparticles was carried out using a Cary, 630 FTIR (Agilent Technologies, India) in the spectral range from 4000 to 400 cm-1.

**2.6.6. Transmission electron microscopy (TEM)**

Transmission electron microscopy (FP 5022/22-Tecnai G2 20 S-TWIN, FEI, USA) was performed to characterize the size and shape of TiO2 nanoparticles.

**2.6.7. Statistical analysis**

All experiments were performed in triplicate, and mean and standard error were determined using GraphPad Prism 5.0.

**3. Results and discussion**

**3.1. Green synthesized TiO2-NPs and its UV–vis characterization**

The addition of *M. azedarach* aqueous extract of the TiO2-NPssolution creates a change in the color of the reaction mixture from yellow to brown (Fig.2). Such change in color occurred because of the active molecules present in the *M. azedarach* aqueous extract of the TiO2-NPs. Moreover, it has been established that TiO2-NPsshow strong surface plasmon resonance (SPR) properties, a consequence of the collective oscillations of free electrons on the metallic particle surface. These oscillations are defined by the particle size which therefore determines the specific wavelength range of the absorption in the visible spectrum. Indeed, The UV-visible spectrum confirmed the formation of TiO2-NPsby showing a typical silver surface plasmon resonance at a wavelength of about 400nm (Fig.2). The presence of a single surface plasmon resonance peak in the absorption spectrum of the formed TiO2-NPsgives evidence to their spherical shape. Similarly, many other reports confirmed that the resonance peak of TiO2-NPscolloidal solutions appear in this region. UV–Vis spectra of the green synthesized TiO2 NPs were recorded using a dual-beam spectrophotometer (Shimadzu UV-2450 PC) in the range of 200–900 nm. Shows the absorption spectrum of the aqueous extract of *Melia Azedarach* leaves and the synthesized TiO2 NPs. The absorption maximum at 317.6 nm supports the formation of TiO2 NPs (Zangeneh,2019). UV-Vis spectroscopy is used to study the absorption spectra of TiO2 NPs that are produced using *Trema Orientalis* leaf extract. An estimated absorption band was found close to 351 nm. Using the following Kubelka-Munk equation, we were able to determine the optical bandgap of produced TiO2 NPs by using the Tauc relation. The absorbance, A, d, and photon energy are represented in the Tauc equations (Equation 1) as α, eV, and d, respectively, representing the thickness of the sample and the UV light traveling through it (Norouzzadeh *et al*.,2020).

 

**Fig:2. UV- Visible spectrum and Chromatic variation of TiONPs after adding extract with 2mM of titanium solution (i) Extract, (ii) titanium and (iii) TiO2NPs**

**3.2. SEM and Energy-dispersive X-ray analysis (EDAX)**

The SEM surface morphology and size of the TiO2 nanoparticles were investigated by scanning electron microscopy (SEM), which revealed that the TiO2 nanoparticles were spherical and ranged in size from 0.5µm to 1µm (Fig. 3 a, b) (Muthukrishnan *et al*.,2019). Chemical composition and crystalline nature of the green synthesized TiO2 were obtained by the EDAX analysis. Generally, it is known that TiO2 display typical optical absorption peaks 4.3-5.3Ke V due to surface plasmon resonance (Fig.3. c) (Muthukrishnan *et al*.,2019). Using the SEM image, the surface morphology of the produced TiO2 was examined an uneven particle structure was detected in the produced TiO2 nanoparticles. The formation of aggregate nanoparticles after the nanoparticles were uniformly distributed throughout the surface showed that the powder particles are very slightly agglomerated (Purkait *et al*.,2020).

  

**a**

**b**

 

**c**

**Fig.3. (a) The SEM images of green synthesized TiO2-NPs at X30,000 (b) at X10,000, and (c) EDX images for TiO2-NPs**

**Table:1. EDAX elemental analysis of green synthesized TiO2**

|  |  |  |  |
| --- | --- | --- | --- |
| **S.No** | **Element** |  **Mass (%)** | **Atom (%)** |
| 1. | Ti | 59.25 | 31.96 |
| 2. | O | 36.08 | 57.99 |
| 3. | C | 4.67 | 10.05 |
|  | Total | 100.00 |  |

**3.3. XRD**

The XRD patterns of green synthesized TiO2 nanoparticles were observed at Scan Axis Gonio Start Position [°2Th.]10.0231 End Position [°2Th.]80.9231 Step Size [°2Th.] 0.0500 Scan Step Time [s10.7950 Scan Type Continuous PSD Mode Scanning PSD Length [°2Th.] 2.12Offset [°2Th.] 0.0000 Divergence Slit Type Fixed Divergence Slit Size[°] 0.4785 Specimen Length[mm]10.00 Measurement Temperature [°C] 25.00Anode Material CuK-Alpha1 [Å]1.54060K-Alpha2 [Å]1.54443K-Beta[Å]1.39225K-A2 / K-A1 Ratio0.50000Generator Settings 30 mA, 40 kV Diffractometer Type 0000000011024644 Diffractometer Number-0Gonio meter Radius [mm]240.00 Dist. Focus-Diverg. Slit [mm] 91.00(Fig. 4). These results were confirmed using Joint Committee on Energy Diffraction Standards (JCPDS) The average crystalline size of the synthesized nanoparticles was 41.9 nm calculated using the Scherrer equation (D = Kλ/βcosθ). λ is the X-ray wavelength (CuKα = 0.15406 nm), K is a constant taken as 0.89, β is the line width at half maximum (FWHM) of the peak, and θ is the diffraction angle. The average crystalline size of the synthesized nanoparticles is 15–45 nm calculated according to the Scherrer formula based on the XRD pattern (Patterson, 1939). XRD was used to examine the crystalline nature of the produced TiO2 NPs. The XRD plot revealed major peaks that corresponded to the (101), (112), (211), and (301) planes of TiO2 with 2θ values of 25.360, 36.200, 37.200, 54.240, 56.610, and 69.530. The anatase phase is represented by the lattice parameters found for TiO2 NPs (JCPDS file no. 84-1285) (Hudlikar *et al*.,2012).

 

**Fig.4. XRD patterns of green synthesized TiO2-NPs using *M. azedarach* leaf extract**

**Table:2. XRD patterns of green synthesized TiO2-NPs using *M. azedarach* leaf extract**

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| Pos. [°2Th.] | Height [cts] | FWHM Left [°2Th.] | d-spacing [Å] | Rel. Int. [%] |
| 14.2051 | 88.83 | 0.1476 | 6.23505 | 85.43 |
| 19.9824 | 24.71 | 0.2952 | 4.44352 | 23.77 |
| 32.0700 | 103.99 | 0.1968 | 2.79098 | 100.00 |
| 42.4555 | 20.67 | 0.2952 | 2.12921 | 19.88 |
| 47.8083 | 22.13 | 0.2952 | 1.90257 | 21.28 |

**3.4. FTIR**

The FTIR spectrum of the biosynthesized titanium dioxide nanoparticles showed peaks at 3443.96, 2025.46 cm-1, 1633.37 cm-1, 1383.94 cm-1,1270.75 cm-1,1102.04 cm-1and 665.79cm-1 (Fig. 5). The peak at 3581.96 corresponds to O–H stretching of alcohols and phenolic compounds, the peak at 1166.92 is attributed to C=C groups of aromatic rings. The peak at 1102.04 indicates the C=O vibration of the carboxylic acid and the peak at 665.79 corresponds to the Ti–O–Ti stretching vibration of the titanium dioxide nanoparticles. The presence of the C-O stretch of the ester group, which is classified as hydrolyzable tannic acid present in M. azedarach leaf extract and is the main biomolecule responsible for the formation and stabilization ofTiO2-NPs. Similar observations were also reported by Vasconcelos *et al*., (2011) and Yilmaz *et al*., (2011).

 

**Fig. 5. FT-IR spectrum of green synthesized TiO2-NPs using *Melia azedarach* leaf extract**

**3.5. TEM**

TEM studies showed that the nanoparticles have a spherical shape with a smooth surface and a size in the range of 10–20 nm, which is in close agreement with the size estimated by the Scherrer formula based on the XRD pattern (Fig. 6) (Dougnon,2022).

  

**a**

**b**

**Fig. 6. The TEM images of green synthesized TiO2-NPs using *M. azedarach* leaf extract at a)10nm and b)20nm scale**

**4. Conclusion**

The green synthesis of titanium dioxide nanoparticles was achieved due to the presence of terpenoids, flavonoids and proteins in *Melia azedarach* as these bioactive compounds were responsible for the synthesis of these nanoparticles. Synthesis using a green approach is a simple, cheap and environmentally friendly process that reduces the use of toxic chemicals. The interest in green synthesis is due to its advantages of being harmless to the environment. Different plant parts are used for synthesis purposes and their characterization using different techniques helps to generate TiO2 NPs of different shapes and sizes. To summarize, green technology through biosynthesis, as discussed in the paper, provides excellent insights that can encourage foster researchers and novices to continue and expand their exploration of nature's potential, as well as the development of efficient and sustainable methods for the synthesis of nanoparticles with desirable properties, which can be used in various fields.

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