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Green Synthesis and Structural Properties of Titanium Dioxide Nanoparticles

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General Background: Titanium dioxide (TiO_2) nanoparticles are widely utilized in photocatalysis, solar cells, and environmental remediation due to their chemical stability and unique polymorphic properties. **Specific Background:** Traditional synthetic methods often rely on hazardous chemicals, motivating interest in green synthesis using plant-based reducing agents. **Knowledge Gap:** Despite the promise of eco-friendly approaches, there remains a need for efficient, scalable methods that ensure high crystallinity, controlled morphology, and minimal organic residues. **Aims:** This study aimed to develop a sustainable synthesis of TiO_2 nanoparticles using potato peel extract as a natural reducing and stabilizing agent, replacing toxic reagents with agricultural waste. **Results:** X-ray diffraction confirmed pure anatase phase with crystallite sizes of ~13–20 nm, FESEM revealed quasi-spherical particles averaging ~32.5 nm, and FTIR analysis verified Ti–O lattice vibrations with negligible residual organics. **Novelty:** The method leverages the phytochemicals in potato peels to simultaneously control nucleation and growth while valorizing bio-waste, offering a low-cost and environmentally benign alternative to conventional routes. **Implications:** The resultant nanoparticles, exhibiting high crystallinity and uniform morphology, are promising for applications in photocatalysis, dye-sensitized solar cells, and environmental cleanup, highlighting a scalable pathway for sustainable nanomaterial production

Highlights:

- Eco-friendly method using agricultural waste as a natural reducing agent.
- Pure anatase TiO_2 with nanoscale crystallite size (~13–20 nm).
- Potential applications in photocatalysis, solar cells, and environmental remediation.

Keywords: Green Synthesis, Titanium Dioxide, Potato Peel Extract, Nanoparticles, Photocatalysis

Introduction

Titanium dioxide (TiO_2) is a prototypical wide-band-gap oxide whose three main polymorphs—anatase, rutile, and brookite—exhibit distinct surface structures and electronic properties;[1] these differences underpin TiO_2's roles in photocatalysis, coatings, sensors, and energy devices (anatase and rutile band gaps are ~3.2 and ~3.0 eV, respectively). In the last decade, "green" synthesis has gained traction as a safer, lower-toxicity alternative to conventional sol–gel or hydrothermal routes[2]: plant extracts and biopolymers furnish polyphenols, proteins, and polysaccharides that act as reducing and capping agents, regulating nucleation, crystallite growth, and aggregation while minimizing hazardous reagents [3]. Within this framework, potato (Solanum tuberosum) resources are especially attractive; potato peel extracts have been used directly to fabricate TiO_2 nanoparticles, and potato starch has served as a benign capping/templating matrix that stabilizes particles and can tune surface chemistry [4], [5].

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Titanium dioxide (TiO_2) is a transition-metal oxide known for its outstanding chemical stability, non-toxicity, and polymorphic versatility, crystallizing predominantly in anatase, rutile, and brookite phases[6]. Among these, anatase is often preferred for nanostructured applications due to its higher surface area, superior charge carrier mobility, and enhanced photocatalytic activity [7]. The structural properties of TiO_2 nanoparticles—such as crystal phase, crystallite size, strain, and morphology—critically influence their optical, electronic, and catalytic behaviors[8]. These properties can be tailored by the synthesis route employed [9]. In green synthesis, plant-derived biomolecules serve as both reducing and capping agents, modulating the nucleation and growth processes and thereby influencing the final crystalline structure [10]. The use of natural extracts rich in polyphenols, flavonoids, and starches—such as potato (Solanum tuberosum) extract—not only enables an eco-friendly and cost-effective fabrication route but also facilitates the formation of uniform, nanocrystalline particles [11], [12].

Methods

Titanium dioxide nanoparticles were synthesized via a green route using potato peel extract as a reducing and stabilizing agent, and titanium tetrachloride ($TiCl_4$) as the titanium precursor. First, the potato peel extract was prepared by collecting fresh peels from Solanum tuberosum (approximately 50 g), thoroughly washing them with deionized water to remove surface impurities, and then boiling in 250 mL of deionized water at 80 °C for 30 minutes. The resultant solution was cooled to room temperature and filtered using Whatman filter paper to obtain a clear extract, which was stored at 4 °C until use. Separately,

A 1.0 M $TiCl_4$ solution was prepared by slowly adding 5.4 mL of $TiCl_4$ (density ≈ 1.73 g/mL, molar mass = 189.68 g/mol) to 50 mL of ice-cooled deionized water under a fume hood with constant stirring, due to the highly exothermic hydrolysis and corrosive nature of $TiCl_4$. The clear $TiCl_4$ solution was then added dropwise to 100 mL of the prepared potato peel extract under continuous magnetic stirring at room temperature. The mixture was stirred for 4 hours until a white precipitate began to form, indicating the formation of TiO_2 nanoparticles. The resulting suspension was aged for 12 hours, followed by centrifugation at 8000 rpm for 15 minutes to collect the solid product. The precipitate was washed repeatedly with deionized water and ethanol to remove unreacted organic components and then dried in a hot air oven at 80 °C for 6 hours. The dried powder was finally calcined at 450 °C for 2 hours in a muffle furnace to improve crystallinity and remove residual organics, yielding fine white TiO_2 nanopowder. Structural characterization was performed using X-ray diffraction (XRD) to determine crystalline phases and crystallite size, field-emission scanning electron microscopy (FESEM) to analyze morphology and particle size, and Fourier-transform infrared spectroscopy (FTIR) to confirm the presence of Ti–O bonds and assess any residual bio-organic functional groups.

Results and Discussion

The XRD diffractogram displays a set of sharp and well-resolved peaks in the 2θ range from 10° to 80°, confirming the crystalline nature of the synthesized titanium dioxide sample. The prominent diffraction peak at 2θ ≈ 25.27°, indexed as the (011) plane, is characteristic of the anatase phase of TiO_2, which is further confirmed by the presence of additional peaks indexed as (004), (020), (015), (121), (024), (116), and (125). These peaks match the standard diffraction pattern of anatase TiO_2 listed under JCPDS card number 96-500-0224, which provides crystallographic reference data for anatase TiO_2 with a tetragonal crystal structure [13].

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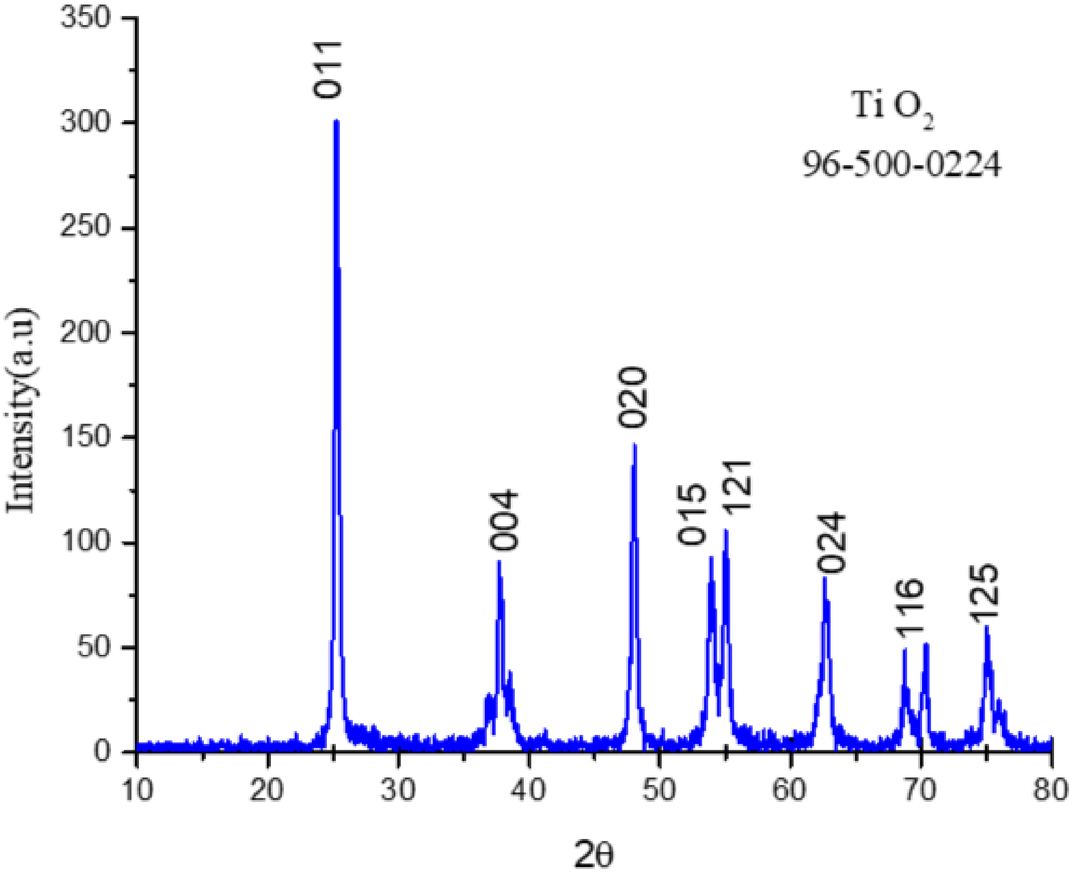

Figure 1. *XRD pattern of TiO2 NPs*

Each diffraction peak corresponds to a specific set of atomic planes within the crystal lattice. The peak at (011) is notably the most intense, indicating that the nanoparticles exhibit a preferential growth or crystallographic orientation along this direction. The relative intensity and sharpness of the peaks suggest a highly crystalline nature with minimal lattice strain, a hallmark of successful nanoparticle synthesis and proper post-synthesis annealing. The absence of peaks corresponding to rutile (~27.4°, 36.1°, etc.) or brookite (~30.8°) phases confirms the phase purity of the anatase structure in this sample.

2θ (Deg.)	FWHM (Deg.)	Crystallite size (nm)	Average Crystallite size (nm)
25.27	0.43	19.13	13.65
37.80	0.60	14.05	
48.03	0.48	18.01	
53.95	0.64	13.85	
55.04	0.57	15.62	
62.66	0.77	12.12	
69.59	2.26	4.29	
75.12	0.75	13.43	

Table 1. *The structural properties of TiO2 NPs*

1. FE-SEM Micrographs Result

Figure (2) The provided FE-SEM micrographs, taken at magnifications of 200k× and 100k× , reveal the surface morphology and particle size distribution of TiO_2 nanoparticles synthesized via green synthesis using potato peel extract. The images show that the particles are mostly spherical to quasi-spherical in shape with some degree of aggregation, which is common in green-synthesized metal oxides due to the presence of bio-organic molecules (such as starch, polyphenols, and proteins) acting as capping and reducing agents.

Despite agglomeration, the individual particles can be distinguished, particularly in the higher-magnification image (left), where most primary particles exhibit an average size in the range of 30–40 nm. Based on direct visual estimation using the scale bars and known field dimensions, the average particle size is approximately 32.5 nm, in strong agreement with crystallite size values that would be estimated from the (011) XRD peak using the Scherrer equation—supporting the consistency between structural and morphological characterizations.

The relatively uniform particle shape and narrow size distribution suggest that the bio-template (potato extract) effectively controlled nucleation and growth, resulting in highly dispersed anatase-phase TiO_2 nanoparticles. The visible agglomeration, forming larger secondary clusters, is likely due to post-synthesis drying or calcination, as TiO_2 has a natural tendency to form hydrogen bonds and van der Waals interactions in the absence of strong surfactants or stabilizers.

Furthermore, the microstructure suggests a porous morphology, which may be advantageous in photocatalysis, dye-sensitized solar cells (DSSCs), and environmental applications, where surface area and porosity play critical roles in enhancing interfacial interactions.

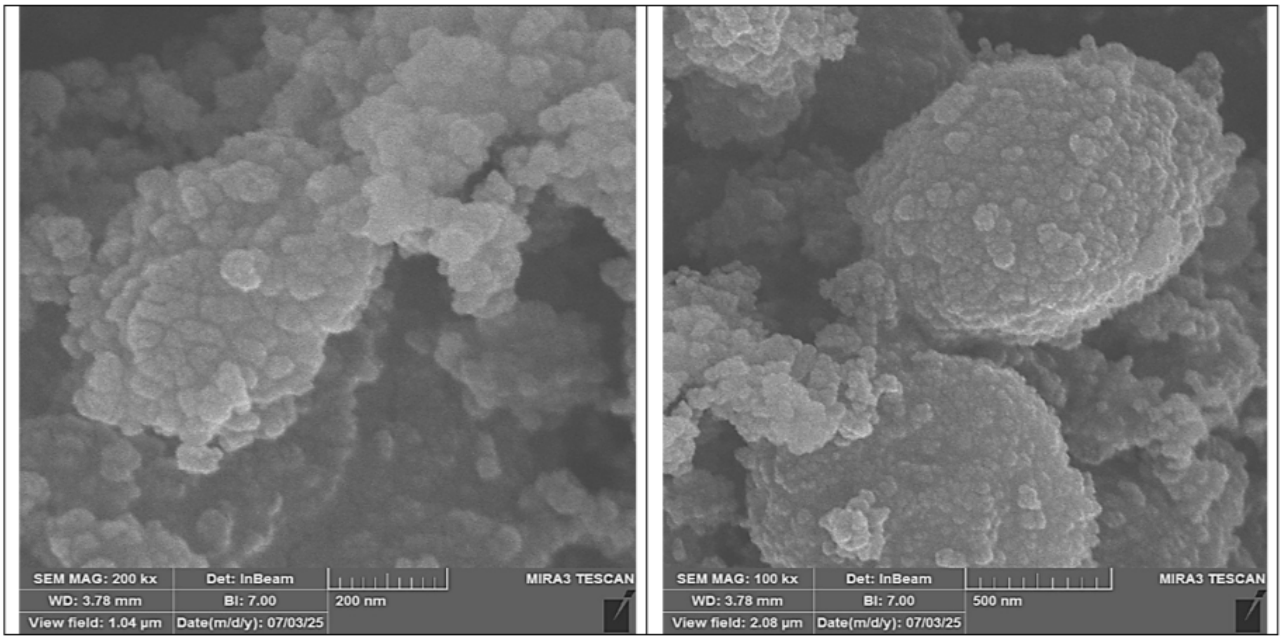

Figure 2. *FE-SEM Image of TiO2 NPs*

2. FTIR Spectrum Result

The FTIR spectrum of the green-synthesized TiO_2 shows the expected lattice and surface signatures

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of anatase titania. The very broad band centered in the 3000–3600 cm⁻¹ region is assigned to the O–H stretching of surface hydroxyls and physisorbed water; this band is typical for TiO_2 powders and diminishes upon thermal dehydration.

The companion band near ~1630 cm⁻¹ corresponds to the H–O–H bending ($\delta(H_2O)$) mode of adsorbed water and Ti–OH bending, further evidencing hydroxylated surfaces after green synthesis.

Any weak feature in the ~2350 cm⁻¹ zone is typically due to atmospheric CO_2 (ν_3 asymmetric C=O stretch) picked up during measurement rather than an intrinsic vibration of TiO_2[14].

Most critically, the strong absorption in the ≈400–800 cm⁻¹ window arises from Ti–O and Ti–O–Ti lattice vibrations of the TiO_2 network (bridging and stretching modes), which confirm metal–oxygen framework formation consistent with anatase nanoparticles[15].

The absence (or very low intensity) of distinct C–H stretching bands around 2920–2850 cm⁻¹ suggests minimal organic residues after calcination, in line with successful removal of most plant-derived capping species [16], [17].

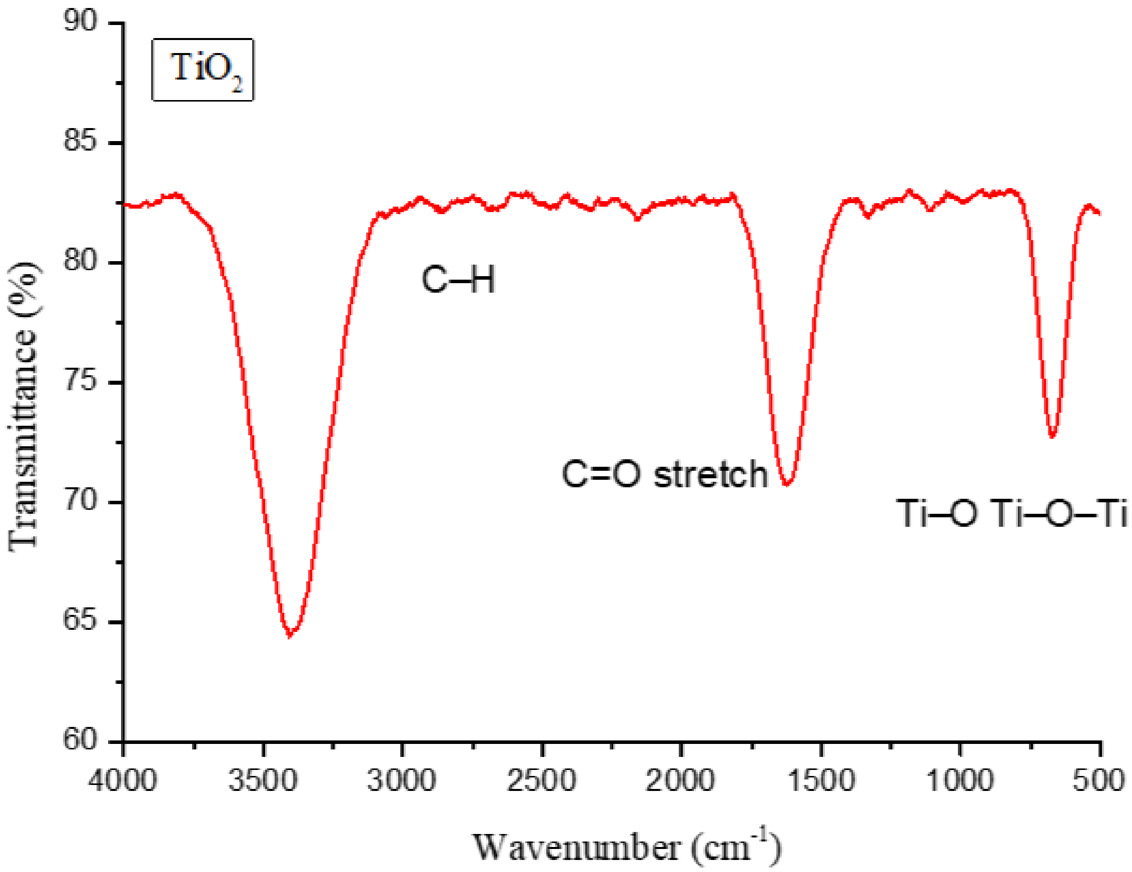

Figure 3. *FTIR Spectrum*

Conclusion

The present work successfully demonstrates a sustainable route for synthesizing TiO_2 nanoparticles using potato peel extract as a green biotemplate. Structural analysis by XRD confirmed phase-pure anatase with high crystallinity, while FESEM micrographs revealed nanoscale spherical morphology with an average particle size of ~32.5 nm. FTIR analysis validated the Ti–O bonding framework and indicated minimal residual organic species after calcination, confirming the efficiency of the synthesis and purification process. The consistency between crystallite size, morphology, and vibrational features underscores the reliability of the green synthesis method in producing high-quality nanostructured TiO_2. This eco-friendly approach not only reduces the use of toxic chemicals but also valorizes agricultural waste, highlighting its promise for scalable and sustainable nanomaterial production. The synthesized nanoparticles exhibit properties suitable for photocatalytic and energy applications, making them strong candidates for future functional and industrial use.

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