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Synthesis of Multifunctional TiO_2 Nanoparticles from Punica granatum Peel: A Sustainable Route to Advanced Nanomaterials

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Abstract: The growing demand for sustainable nanomaterials has encouraged the development of green synthesis approaches that make effective use of natural resources and agricultural waste. In this study, titanium dioxide (TiO_2) nanoparticles were synthesized using Punica granatum peel extract as a plant-derived medium, providing a sustainable route for converting fruit-processing waste into functional nanomaterials. The peel extract was prepared in distilled water and combined with titanium tetraisopropoxide (TTIP) to facilitate nanoparticle formation. The resulting material was separated, dried, and calcined at 450 °C to obtain TiO_2 nanoparticles. The synthesized material was characterized using Fourier-transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), scanning electron microscopy (SEM), and transmission electron microscopy (TEM). The FTIR spectrum indicated the presence of surface hydroxyl groups and absorption features associated with Ti-O and Ti-O-Ti vibrations. The XRD pattern exhibited a prominent diffraction peak at approximately 25.3°, together with additional reflections consistent with crystalline anatase TiO_2. SEM and TEM observations revealed irregularly distributed, predominantly rounded to quasi-spherical particle-like features, with noticeable aggregation. These findings support the successful preparation of crystalline TiO_2 nanoparticles through a plant-mediated synthesis route. The study highlights the potential of pomegranate peel as a value-added resource for sustainable nanomaterial production and provides a foundation for further investigations into the applications of the synthesized nanoparticles.

Keywords: Green synthesis; titanium dioxide nanoparticles; Punica granatum peel; anatase TiO_2; structural characterization; waste valorization.

1. Introduction

Nanotechnology has become an important area of materials science because it offers opportunities to develop materials with distinctive structural, optical, chemical, and surface properties. Among the different nanomaterials, metal oxide nanoparticles have received considerable attention

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owing to their applications in environmental remediation, catalysis, energy conversion, and biomedical research. However, the properties and performance of these materials depend not only on their chemical composition but also on their particle size, morphology, crystalline structure, and surface characteristics. Consequently, developing reliable and sustainable synthesis strategies is an important step towards producing nanomaterials with desirable functional properties [1,2]. Titanium dioxide (TiO_2) is one of the most extensively investigated semiconductor metal oxides because of its chemical stability, relatively low cost, useful optical properties, and photocatalytic potential. It occurs mainly in three crystalline forms: anatase, rutile, and brookite, each possessing distinct structural characteristics. TiO_2 has been widely studied for photocatalytic oxidation, self-cleaning surfaces, water purification, and antimicrobial applications [3,4]. When exposed to light with sufficient energy, TiO_2 generates electron–hole pairs that can participate in surface redox reactions. These charge carriers may produce reactive oxygen species, including hydroxyl radicals and superoxide radicals, which contribute to the transformation of organic contaminants under suitable experimental conditions [3]. Nevertheless, conventional TiO_2 exhibits limitations, particularly its relatively wide band gap and the rapid recombination of photogenerated charge carriers. These factors can restrict its efficiency under visible-light irradiation and motivate continued research into controlling its structure, surface chemistry, and composition [4,5]. Conventional approaches for synthesizing TiO_2 nanoparticles include sol–gel processing, hydrothermal treatment, precipitation, and related chemical methods. Although these techniques provide control over crystallinity and particle morphology, some procedures require substantial energy input, additional chemical reagents, or carefully controlled reaction conditions. The increasing emphasis on sustainable materials production has therefore encouraged researchers to explore synthesis routes that reduce hazardous chemical use and make better use of renewable resources. Green chemistry provides a useful framework for this transition by encouraging waste prevention, safer reagents, improved resource efficiency, and reduced environmental impact [6]. Plant-mediated synthesis represents one promising approach because naturally occurring biomolecules can interact with metal precursors and influence the formation and stabilization of nanostructured materials [1,2,7]. Plant extracts contain a complex mixture of phytochemicals, including polyphenols, flavonoids, tannins, organic acids, sugars, and other functional compounds. Depending on the plant source and reaction conditions, these constituents may participate in precursor complexation, hydrolysis, nucleation, particle growth, and surface modification. Their influence can extend beyond the initial formation process by affecting particle aggregation, surface functional groups, and the physicochemical characteristics of the final material [1,2,7]. Previous studies have demonstrated the feasibility of producing TiO_2 nanoparticles through plant-assisted routes, while also showing that the extract composition and concentration can influence the resulting crystalline phase, particle size, and structural properties [8,9]. However, green synthesis should not automatically be considered nontoxic or environmentally superior in every respect; the precursor, extraction solvent, energy requirements, calcination conditions, and final material toxicity must also be considered. Among the available plant resources, Punica granatum L. (pomegranate) peel is particularly interesting because it is commonly discarded during fruit processing despite containing a substantial concentration of valuable phytochemicals. Pomegranate peel has been reported to account for approximately 30–40% of the fruit and to contain phenolic acids, flavonoids, and hydrolysable tannins, including punicalagin, punicalin, ellagic acid, and gallic acid [10,11]. These constituents have attracted considerable interest in food chemistry, natural-product research, and waste

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valorization. Their abundance makes pomegranate peel a potentially useful biological resource for developing plant-mediated synthesis methods. Using this material as a synthesis aid could provide an additional use for an agricultural by-product while supporting the principles of resource recovery and circular economy [10–12]. The chemical composition of pomegranate peel provides a reasonable basis for investigating its role in TiO_2 nanoparticle formation. Phenolic hydroxyl groups and other oxygen-containing functional groups may interact with titanium-containing species during precursor hydrolysis and condensation. Such interactions could influence nucleation, growth, and the distribution of organic species on the developing particle surface. Subsequent thermal treatment may remove a substantial proportion of the organic constituents and promote the formation of crystalline TiO_2. Nevertheless, the exact role of individual phytochemicals should be established through experimental evidence rather than assumed from the known composition of the extract. Similarly, the final crystal phase and particle morphology must be confirmed using appropriate characterization techniques [7,8,10]. Previous research has already explored the use of plant extracts and fruit-processing residues in TiO_2 synthesis. For example, Isnaeni and co-workers investigated the green synthesis of different TiO_2 nanoparticle phases using mango-peel extract and demonstrated that the extract-to-precursor ratio could influence the phase composition and particle characteristics [8]. A separate study reported the preparation of a TiO_2-based material using pomegranate peel extract and investigated its antimicrobial potential for water disinfection [13]. More recent research using Kinnow peel has also demonstrated the continued interest in fruit-waste-mediated TiO_2 synthesis and the evaluation of its physicochemical and antioxidant properties [14]. Together, these studies establish that plant and fruit-derived materials can serve as useful components in the development of TiO_2-based nanomaterials. Despite this progress, the use of a particular fruit peel does not, by itself, establish the novelty of a synthesis method. The scientific value of a new study should instead arise from a clearly defined research question, reproducible synthesis conditions, detailed structural characterization, and convincing evidence of functional performance. In the case of P. granatum peel, it is important to determine how the extract concentration, precursor chemistry, solution pH, reaction temperature, and calcination conditions affect the properties of the resulting TiO_2 nanoparticles. A systematic relationship between these synthesis parameters and the final material characteristics would help distinguish the contribution of the plant extract from the effects of conventional processing variables [1,2,7]. TiO_2 nanoparticles also offer opportunities for multifunctional applications, particularly in environmental and antimicrobial research. In photocatalytic treatment, light-generated charge carriers can initiate oxidative reactions that transform organic pollutants, although the extent of degradation depends on the light source, pollutant concentration, catalyst loading, pH, and other reaction conditions [3,4,15]. In antimicrobial studies, TiO_2-based materials may affect microbial viability through surface interactions and, under appropriate illumination conditions, reactive oxygen species generation. However, antimicrobial performance should be evaluated experimentally, with suitable controls, and should not be inferred solely from nanoparticle formation or the known biological properties of pomegranate peel [13,15]. Establishing more than one demonstrated function would provide a stronger basis for describing the synthesized material as multifunctional.

Based on these considerations, the present study focuses on the synthesis of TiO_2 nanoparticles using Punica granatum peel extract as a plant-derived synthesis medium. The study aims to investigate the structural, morphological, and optical characteristics of the prepared material and to assess its potential for multifunctional applications through appropriate experimental evaluations. X-ray

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diffraction, FTIR spectroscopy, electron microscopy, elemental analysis, and UV–visible diffuse reflectance spectroscopy can provide complementary information about the crystalline phase, surface chemistry, particle morphology, elemental composition, and optical response. Depending on the experimental findings, photocatalytic and antimicrobial studies may further establish the practical utility of the synthesized nanoparticles. By integrating fruit-waste valorization with plant-mediated nanomaterial synthesis, this work seeks to contribute to the development of more sustainable TiO_2 production strategies and to clarify the relationship between synthesis conditions, material properties, and functional performance.

2. Experimental

2.1. Materials

Titanium tetraisopropoxide (TTIP), isopropanol, and ethanol (Qualigen) from Avra Chemicals were utilised exactly as supplied. Punica granatumwas purchased from local market. Experimental solutions were made using double-distilled water.

2.2. Catalyst synthesis

2.2.1. Isolation of Peel Extract

Peels of Punica granatum were washed systematically with distilled water and oven-dried. The extract was made ready by boiling dry peel Punica granatum in distilled water with a concentration of 70.0 g/L at 90 °C for 60 min and filtered [16].

2.2.2. TiO_2 preparation

Titanium tetraisopropoxide (TTIP) was dissolved in 60 mL of isopropanol, and the mixture was agitated for an hour at room temperature. Next, 20 mL of Punica granatum extract was progressively added to the mixture. The solution turned after 4 hours of stirring, indicating the formation of TiO_2-NPs [17]. Centrifugation at 5000 rpm for 15 minutes allowed the precipitate to be separated from the solution. The resultant precipitate was dried for six hours at 80 °C in a hot air oven. White TiO_2 NPs were produced by calcining the obtained samples for five hours at 450°C. After that, these TiO_2 NPs were stored in a vacuum-sealed bag until further examinations could be performed.

2.5. Characterization Techniques

The infrared (IR) spectra were recorded using the tidy method on an AVATAR-330 FT-IR spectrometer (4000-650 cm^{-1}). Using powder X-ray powder diffraction (XRD), the crystalline phase of the produced nanomaterials was determined over a 2θ range of 20 to 80º using Cu Kα radiation (λ=1.5418 Å). The surface morphology was analyzed using SEM (ZEISS EVO18) and HRTEM (Jeol/JEM 2100).

3. Results and Discussion

3.1. Characteristics of TiO_2

3.1.1. FT-IR analysis

The Fourier-transform infrared (FTIR) spectrum (Fig.1.) of the TiO_2 nanoparticles synthesized using Punica granatum peel extract provides information about the functional groups associated with the synthesized material. As shown in the spectrum, a broad absorption feature is observed in the higher-wavenumber region around 3200–3500 cm^{-1}, which can be attributed to O-H stretching vibrations associated with surface hydroxyl groups and adsorbed water molecules. These hydroxyl groups may contribute to surface reactivity and influence the photocatalytic behaviour of TiO_2 nanoparticles. The absorption features in the region around 1500-1700 cm^{-1} may be associated with bending vibrations of adsorbed water or stretching vibrations of residual organic functional groups

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originating from the pomegranate peel extract. Furthermore, the absorption bands in the fingerprint region below approximately 1000 $cm^{-1}$are generally associated with Ti- O and Ti-O-Ti lattice vibrations, supporting the formation of titanium oxide. The presence of these features is consistent with the surface chemistry expected for plant-mediated TiO_2 synthesis [17]. The observed FTIR spectrum suggests that surface hydroxyl groups and possible plant-derived organic residues may be associated with the synthesized nanoparticles. The phytochemicals present in Punica granatum peel extract may interact with titanium-containing species during synthesis and influence particle formation. Overall, the FTIR analysis provides supporting evidence for the formation of TiO_2 nanoparticles and offers insight into their surface functional groups, which may be relevant to their potential environmental and photocatalytic applications.

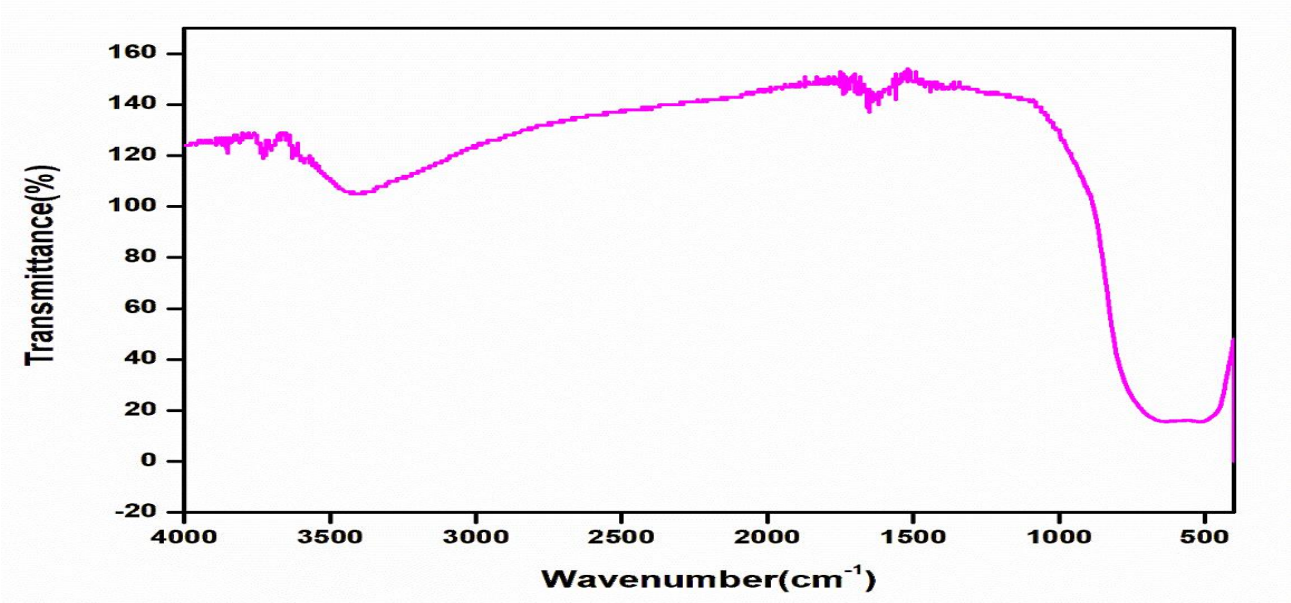


Figure. 1. FT-IR Spectrum of TiO_2

3.1.2. XRD analysis

The X-ray diffraction (XRD) pattern (**Fig.2.**) of the synthesized TiO_2 nanoparticles exhibits several distinct diffraction peaks across the 2θ range of approximately 10°- 80°, indicating the formation of a crystalline material. The most prominent diffraction peak appears at around 25.3°, accompanied by additional peaks at approximately 37.8°, 48.0°, 54.0°, 55.1°, 62.7°, 68.8°, 70.3°, and 75.0°. These reflections are broadly consistent with the characteristic diffraction pattern of anatase-phase TiO_2. In particular, the intense reflection near 25.3° is commonly assigned to the (101) crystal plane of anatase TiO_2, while the reflections around 37.8°, 48.0°, and 54° correspond to other characteristic anatase lattice planes [17]. The presence of these characteristic reflections suggests that the green synthesis successfully produced crystalline TiO_2. The sharp and intense peak at approximately 25.3° indicates a well-defined crystalline arrangement, whereas the comparatively weaker peaks at higher diffraction angles reflect additional crystallographic planes within the anatase structure. The observed diffraction pattern is consistent with the standard anatase TiO_2 phase, although

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confirmation of phase purity requires comparison with a reference diffraction database and assessment of any additional reflections. The use of Punica granatum peel extract provides a sustainable synthesis approach, as its phytochemical constituents may influence precursor hydrolysis and particle formation. Overall, the diffraction results support the formation of crystalline TiO_2 nanoparticles with a pattern consistent with the anatase phase.

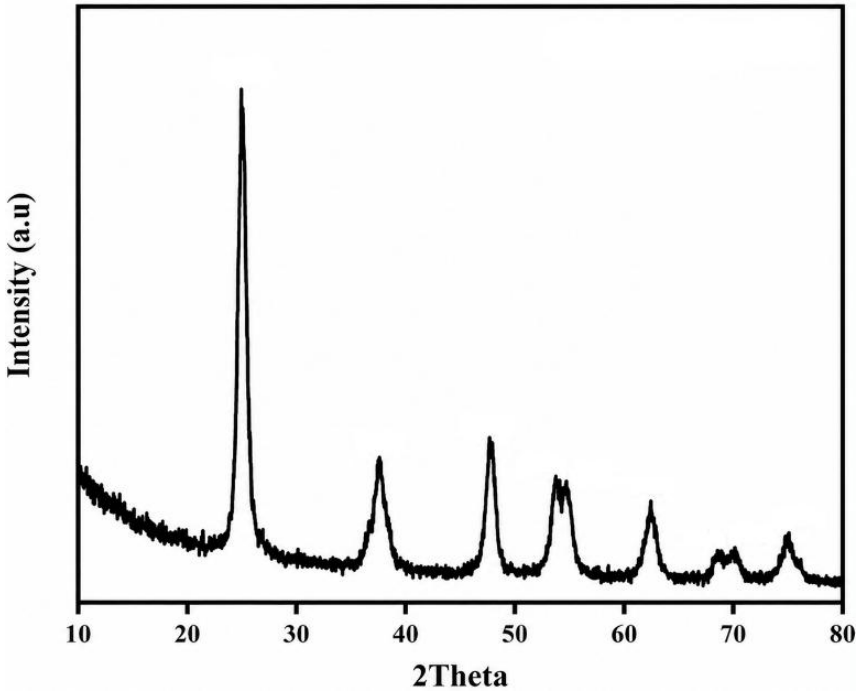


Figure. 2. XRD pattern of synthesized TiO_2

3.1.3 SEM analysis of TiO_2

The scanning electron microscopy (SEM) images (**Fig.3.**) of TiO_2 nanoparticles synthesized using Punica granatum peel extract reveal a densely packed, irregular surface morphology with noticeable particle agglomeration, as shown in the low-magnification image. The particles are distributed throughout the observed surface, forming clusters rather than remaining completely separated. This aggregation may be associated with the high surface energy of nanosized particles and interactions between neighbouring particles during synthesis and drying. The higher-magnification image provides a clearer view of the individual particle-like features, which exhibit predominantly rounded to irregular morphologies with variations in apparent size and shape. These morphological characteristics suggest that the plant-mediated synthesis produced a heterogeneous particulate structure. Similar aggregation and morphology variations are commonly reported for TiO_2 nanomaterials prepared using plant extracts, in which naturally occurring biomolecules can influence nucleation and particle growth.

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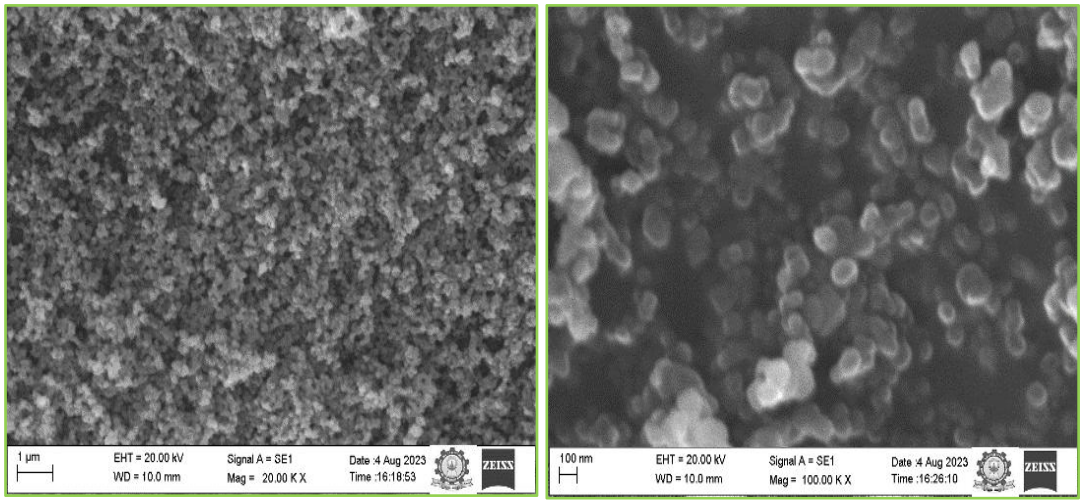
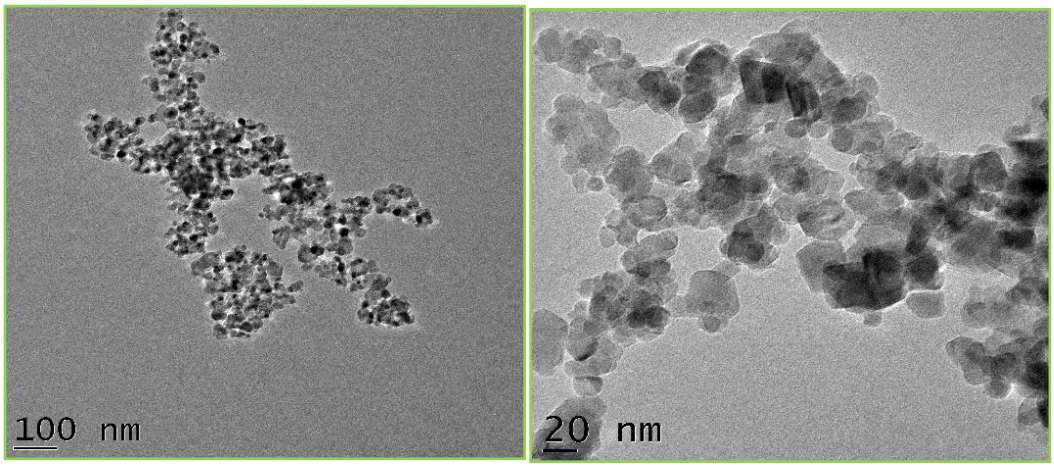


Figure 3. SEM images of TiO_2

3.1.4 TEM analysis of green-TiO_2

The transmission electron microscopy (TEM) images (**Fig.4.**) provide further insight into the particle morphology, nanoscale structure, and aggregation behaviour of the TiO_2 nanoparticles synthesized using Punica granatum peel extract. The lower-magnification image (100 nm scale bar) reveals that the particles are arranged in interconnected clusters, forming an irregular, branched assembly. At higher magnification (20 nm scale bar), the individual particle-like features appear predominantly rounded to quasi-spherical, with some irregular shapes and considerable particle-to-particle contact. The overlapping contrast regions indicate that the nanoparticles are closely packed, which may result from agglomeration during synthesis, drying, or sample preparation. The observed aggregation is common in TiO_2 nanomaterials because nanosized particles possess high surface energy and tend to associate with neighbouring particles. The use of Punica granatum peel extract may influence particle nucleation and growth through interactions between titanium-containing species and naturally occurring phytochemicals.



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Figure 4. TEM images TiO_2

4. Conclusion:

In this study, titanium dioxide (TiO_2) nanoparticles were successfully synthesized using Punica granatum peel extract through a simple and sustainable plant-mediated approach. The utilization of pomegranate peel, an abundantly available fruit-processing waste, offers a promising alternative for producing value-added nanomaterials while reducing dependence on additional synthetic chemicals. The synthesized TiO_2 nanoparticles were systematically characterized using FTIR, XRD, SEM, and TEM techniques to investigate their structural and morphological properties. FTIR analysis revealed characteristic absorption features associated with surface hydroxyl groups and Ti-O-Ti vibrations, supporting the formation of titanium oxide. The XRD pattern exhibited a prominent diffraction peak at approximately 25.3°, along with other characteristic reflections, confirming the formation of crystalline TiO_2 with a diffraction pattern consistent with the anatase phase. SEM observations revealed a densely packed surface with irregularly distributed particles and noticeable agglomeration, while TEM images further demonstrated the formation of predominantly spherical to quasi-spherical nanoparticles arranged in interconnected clusters. These findings highlight the potential of Punica granatum peel extract as a natural synthesis medium for preparing crystalline TiO_2 nanomaterials. Overall, this work demonstrates a promising waste-to-value approach that integrates agricultural waste utilization with sustainable nanomaterial development. The synthesized TiO_2 nanoparticles may serve as potential candidates for environmental remediation and other functional applications

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