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Preparation and Characterization of TiO_2 Nanoparticles by Green Method

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Abstract

In this study, we attempted to synthesize TiO_2 nanoparticles utilizing titanium tetrachloride as a precursor and an aqueous extract of Sansevieria plant leaves. Atomic force microscopy (AFM), X-ray diffraction (XRD) pattern, scanning electron microscopy (SEM), Fourier transform infrared (FTIR) spectroscopy, and Brunauer-Emmett-Teller (BET) analysis were used to identify the synthesized TiO_2 nanoparticles As per the surface topography, the average diameter of nanoparticles is considered as 67.73 nm. Debye-Scherrer equation was used to summarize the size of the crystals, the result shows that the average crystalline size for TiO_2 sample was 15.2 nm. Few spherical, non-agglomerated particles could be seen in the SEM image of TiO_2 nanoparticles. The appearance of the bending vibrations of Ti-O and Ti-O-Ti are shown at 688.59, 497.63, and 950.91 cm^{-1}, respectively, demonstrating the presence of TiO_2 nanoparticles. BET equation was used to estimate the porosity and surface area for the prepared sample.

Keywords: Green method, Sansevieria, titanium dioxide nanoparticles

INTRODUCTION

Nanotechnology is a fast-expanding discipline that is being used in science and technology to create novel materials at the nanoscale. Numerous uses of nanotechnology have been found in the sciences of pharmacology and biology [1]. Organic and inorganic type of nanoparticles are the scope of this study. The physical, chemical, biological, medicinal, optical, mechanical, and engineering sciences are very interested in metal and metal oxide nanoparticles [2]. For the synthesis of titanium dioxide nanoparticles, various methods are available, including solution combustion, sol-gel, hydrothermal, solvothemal, microwave assisted, co-precipitation, chemical vapor deposition, and green techniques [3]. Because of its ease, speed, eco-friendliness, and non-toxic nature, green synthesis of nanomaterials is gaining more attention [4]. Green synthesis is a simple process that utilizes a range of biological agents and yields no harmful by-products [5]. The success of green technologies is attributed to their lower chemical requirements. Thus, due to the simplest and cheapest procedures, plant extracts are much more interesting [6]. The use of plant extracts has many advantages, including being easily available, safe to handle and having a wide range of metabolites that can survive. Terpenoids, flavones, ketones, aldehydes and amides are the primary phytochemicals involved in the formation of nanoparticles. In this paper, we discuss green synthesis using Sansevieria leaves [7]. Sansevieria plant is one of the most well-known medicinal plants in the world, notably for dressing wounds. The dry Sansevieria aqueous extract has antibacterial effects on *Staphylococcus epidermidis* and contains mixtures such as flavonoids, phenols, proteins, carbohydrates, alkaloids, phenylethanoids, glycosides, quinine, oligosaccharides, saponins, steroids, triterpenoids, sesqui terpenoids, and tannins [8]. In this study, a novel green approach for producing TiO_2

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nanoparticles (NPs) was made using an extract from Sansevieria plants. The produced TiO_2-NPs were characterized by utilizing various techniques.

EXPERIMENTAL PART

Green Synthesis of TiO_2-NPs Using Sansevieria Plant (SP)

This process involved creating titanium dioxide nanoparticles from the extract SP leaves. To get rid of dust and dirt stuck from the plant, SP leaves were washed with distilled water several times, and then the plate was cut into small pieces. Twenty-five grams of leaves were added to 100 mL of boiling distilled water, the mixture was heated at 90°C for 2 hours. In order to separate the solid from the liquid phase, filter paper No.1 was used. In order to create titanium dioxide nanoparticles, 2.8 mL of titanium tetrachloride was added to 100 mL of distilled water and thoroughly shaken. The previously prepared SP leaf extract was gradually added to the titanium tetrachloride solution with constant stirring using (magnetic stirrer, Gallenkamp, England) for 4 hours continuously [3], then dried at 100°C for an entire day and the precipitate was continually calcined at 500°C for 4 hours. The precipitate was washed with distilled water to remove impurities and heated at 700°C for 1 hour to produce titanium oxide nanoparticles as a white precipitate. The precipitate was ground in a laboratory mill and stored in a clean and dry place for characterization. Figure 1 shows the procedure for creating TiO_2-NPs.

Figure 1. Steps of preparation of titanium dioxide nanoparticles (TiO_2-NPs) using Sansevieria plant (SP).

Characterization

Atomic force microscopy (AFM) measurements were taken using Nano AFM 2022, Nanosurf, Switzerland. X-ray diffraction (XRD) patterns for the prepared particles were obtained using XRD, PW1730, Philips, Holanda. Scannine electron microscopy (SEM) analysis of TiO_2-NPs was carried out using FESEM-EDS, MIRA III, TESCAN, Czech and Fourier transform infrared (FTIR) spectra were recorded using Shimadzu IR-Affinity-1 Japan. The Brunauer-Emmett-Teller (BET) specific surface area and pore size were determined by BET, BELSORP MINI II, BEL, Japan.

RESULTS AND DISCUSSION

Atomic Force Microscopy (AFM)

AFM is commonly applied to detect the nature of deposited materials and give information about surface roughness, shape, topography, thickness [2] and the average grain size [9]. The AFM analysis

for TiO_2 NPs sample is shown in Figure 2a and b. The images exhibit that the distribution of TiO_2 NPs was small with anaverage diameter of 67.73 nm. Through these findings, it was discovered that the sample created by green biological produce was positioned at the nanoscale and had particles with a narrow size distribution.

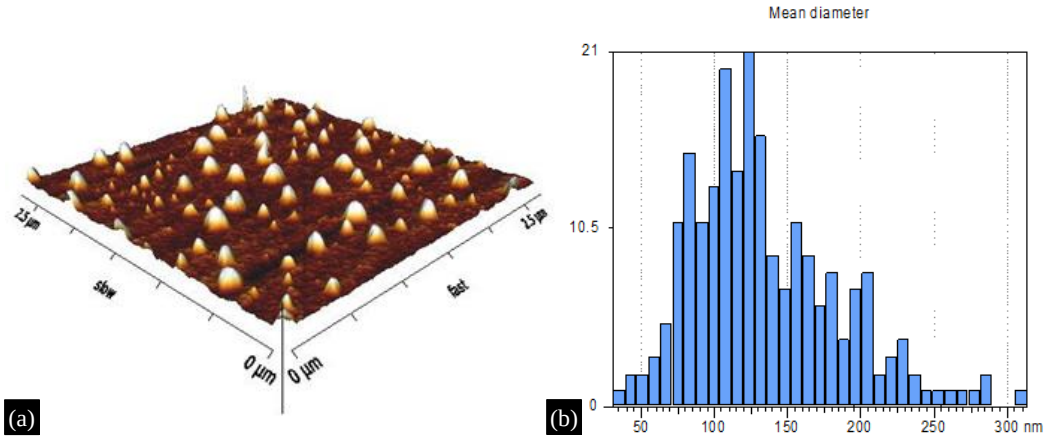

Figure 2. (a, b) View of atomic force microscopy (AFM) image and granularity cumulating distribution chart of TiO_2-NPs.

X-Ray Diffraction (XRD)

XRD pattern for TiO_2-NPs showed a polycrystalline structure of mixed anatase and rutile phases (Figure 3). The dominant anatase phase angles 2θ = 25.5795°, 37.1949°, 38.0553°, 38.7313°, 48.2265°, 55.2019°, 62.8841°, 69.0913°, 70.5356°, 75.1449° corresponding to crystalline planes (101), (103), (004), (112), (200), (211), (204), (116), (220), and (215), respectively according to the standard card No. 96-152-6932, while the diffraction peaks for the identified rutile phase appeared at 27.6076°, 36.2423°, 41.5277°, 54.3108° and 56.7998° corresponding to crystalline planes (110), (101), (111), (211), and (220), respectively according to the standard card No. 96-900-4142. The inter-atomic distance (d_{hkl}) was calculated from the diffraction angles according to Bragg law [10]:

$$n\lambda = 2 d_{hkl} \sin\theta \quad (1)$$

while the crystallite size (D) was determined by using Debye Scherrer equation [11]:

$$D = \frac{K\lambda}{\beta \cos\theta} \quad (2)$$

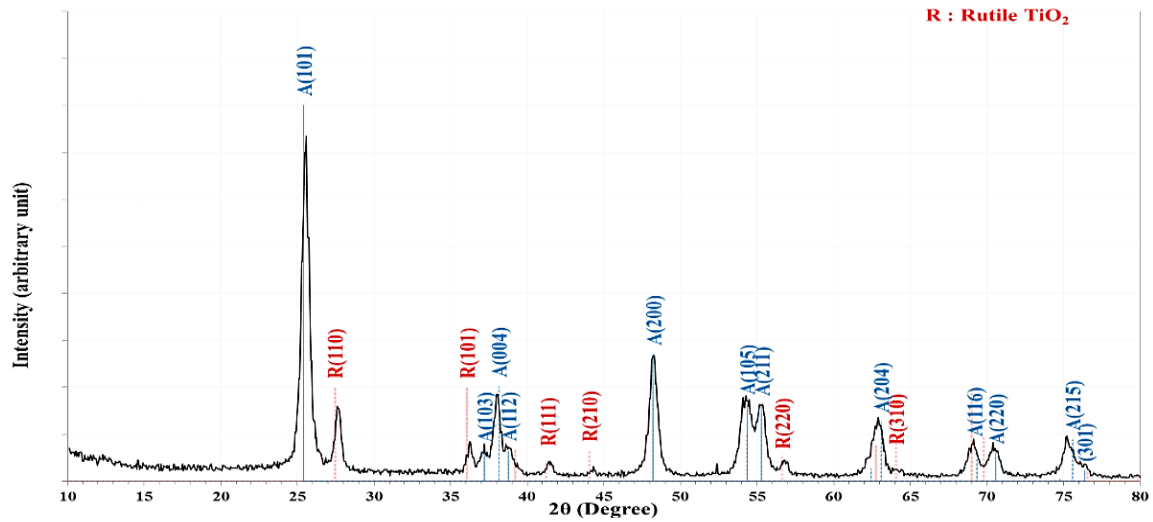

Figure 3. X-ray diffraction (XRD) pattern for titanium dioxide nanoparticles (TiO_2-NPs).

where K is the Scherrer constant, λ is the monochromatic wavelength of X-ray = 0.154061 nm, θ is the diffraction angle, and β is the full width at half maximum (FWHM) in radians. Table 1 shows the X-ray peaks parameters. In this table the values of d_{hkl} are close to the standard values. The average crystalline size was 15.2 nm.

Table 1. The X-ray diffraction results for titanium dioxide nanoparticles (TiO_2-NPs).

2θ (Deg.)	FWHM (Deg.)	d_{hkl} (Å)	D (nm)	hkl	Phase
25.5795	0.5532	3.4796	14.7	(101)	Anatase TiO_2
27.6076	0.4302	3.2284	19.0	(110)	Rutile TiO_2
36.2423	0.3687	2.4766	22.7	(101)	Rutile TiO_2
37.1949	0.5224	2.4154	16.1	(103)	Anatase TiO_2
38.0553	0.5531	2.3627	15.2	(004)	Anatase TiO_2
38.7313	0.5838	2.3230	14.4	(112)	Anatase TiO_2
41.5277	0.5838	2.1728	14.6	(111)	Rutile TiO_2
48.2265	0.6146	1.8855	14.2	(200)	Anatase TiO_2
54.3108	0.7375	1.6878	12.1	(211)	Rutile TiO_2
55.2019	0.6145	1.6626	14.6	211)	Anatase TiO_2
56.7998	0.4917	1.6196	18.4	(220)	Rutile TiO_2
62.8841	0.7682	1.4767	12.1	(204)	Anatase TiO_2
69.0913	0.7989	1.3584	12.1	(116)	Anatase TiO_2
70.5356	0.6453	1.3341	15.1	(220)	Anatase TiO_2
75.1449	0.8297	1.2633	12.1	(215)	Anatase TiO_2

FWHM, full width at half maximum.

Scanning Electron Microscopy (SEM)

SEM measurements are used to produce images of the materials under the scanning electron microscopy technique in order to examine the crystalline structure, porosity morphology, and surface features [12]. Figure 4 shows that the sample had high crystallinity. TiO_2 nanoparticles' SEM images showed that theparticles have a porous surface and a nano sheet-like structure [13]. Using the IMAGE J software, the TiO_2-NPs particle size was determined to be 42.039 nm. It was clear that TiO_2 nanoparticles had gathered and taken on an uneven shape. There were some spherical non-clumping particles present in the synthesized particles. Therefore, we might suppose that the phytochemicals of Sansevieria leaf extract coat the surface of TiO_2 nanoparticles, preventing their buildup [14].

Fourier Transform Infrared (FTIR) Spectroscopy

With the help of the fundamental FTIR spectroscopy technique, biological compounds such flavonoids, phenols, proteins, carbohydrates, alkaloids, phenylethanoids, glycosides, quinine, oligosaccharides, saponins, steroids, triterpenoids, sesqui terpenoids, and tannins can be visualized [8]. FTIR spectra are applied to determined various functional groups that are responsible for the formation of NPs [15]. FTIR spectra for green synthesized TiO_2-NPs are shown in Figure 5. A broad band was seen at 3448.72 cm^{-1}, which is caused by the hydroxyl O-H stretch, which represents the water as moisture. Peak at 2883.58 cm^{-1} confirmed the presence of secondary amines, and 2372.44 and 2318.44 cm^{-1} supported the presence of C-C. The peak at 1643.35 cm^{-1} was caused by the O-H bending vibration of adsorbed water molecules on the surface of TiO_2 which may play a significant role in photocatalytic activity. Strong peaks at 1562.34 and 1514.12 cm^{-1} indicate aliphatic nitro compounds with stretching of N-O [14]. The peak 1396.46 cm^{-1} was aliphatic amine and C-N-stretching [16]. The strong band at 688.59, 497.63 and 950.91 cm^{-1} demonstrate the emergence of Ti-O and Ti-O-Ti bending vibrations, respectively. The existence of TiO_2 in the produced TiO_2-NPs is confirmed by metal oxide linkages such Ti-O-Ti and Ti-O. Alkaloids, coumarins, flavonoids, tannins, and terpenoids are some examples of the biomolecules that strongly interact with TiO_2-NPs and produce the presence of the Ti-O-Ti link. In green synthesis, these phytochemicals convert the majority of titanium dioxide to stable TiO_2 [17].

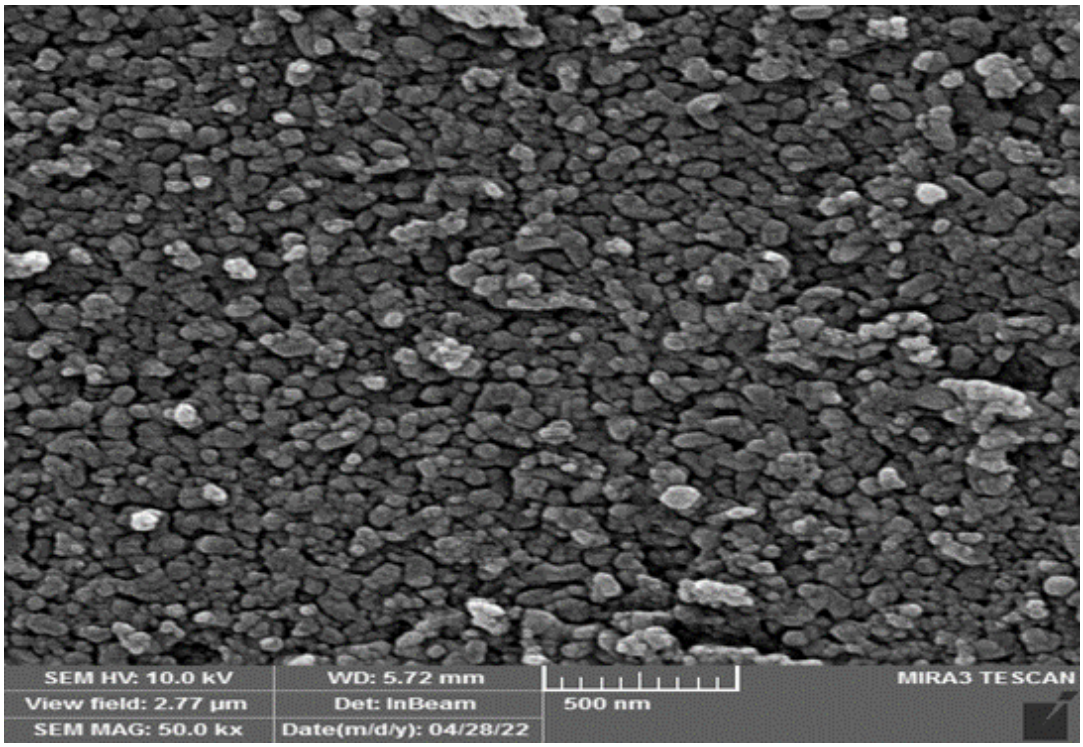

Figure 4. Scanning electron microscopy (SEM) image for titanium dioxide nanoparticles (TiO_2-NPs).

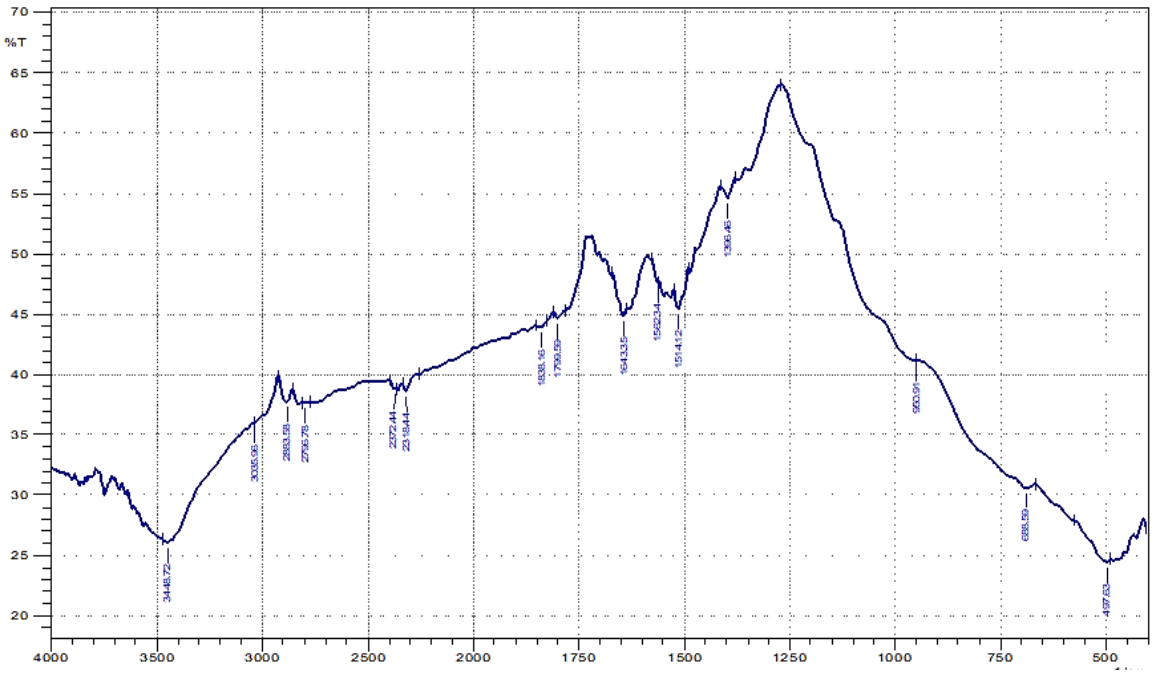

Figure 5. Fourier transform infrared (FTIR) spectrum of titanium dioxide nanoparticles (TiO_2-NPs).

BET Analysis

The surface area analysis and pore volume for synthesized TiO_2 nanoparticles was done by using BET equation [18]. Figure 6 depicts the TiO_2-NPs N_2 gas adsorption/desorption isotherm, this type of isotherm model is characteristic of a substance that involves mesoporosity [19, 20]. It was discovered that the specific surface area S_{BET} calculated using BET equation was 21.532 m^2/g. The mean pore diameter and total pore volume were determined to be equivalent to 27.262 nm and 0.1467 cm^3/g, respectively.

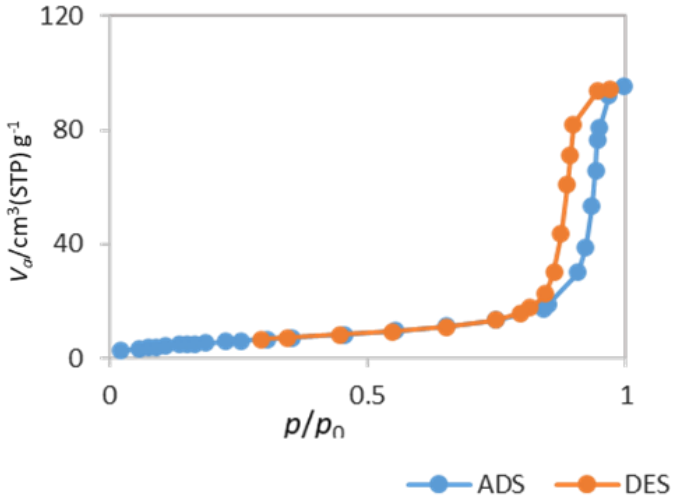

Figure 6. Adsorption/desorption isotherm curve for the produced titanium dioxide nanoparticles (TiO_2 NPs).

CONCLUSIONS

In comparison to other preparation techniques, the environmentally friendly method for producing TiO_2-NPs from Sansevieria plant extract could be a promising technology because it does not require any dangerous chemicals and is safe for the environment. Physical diagnostic techniques such as AFM, XRD, SEM, FTIR and BET, were used to characterize the prepared particles. An average diameter of 67.73 nm was found when the surface topography of TiO_2-NPs was examined. The Debye–Scherrer formula was used to calculate crystallite size, and the typical crystal size is obtained as 15.2 nm. The SEM image of TiO_2 nanoparticles showed few spherical, non-agglomerated particles. The emergence of the bending vibrations of Ti-O and TiO-Ti are shown in the strong bands at 688.59, 497.63, and 950.91 cm^{-1}, respectively, demonstrating the presence of TiO_2-NPs. The BET analysis shows that the specific surface area and total pore volume can be considered as 21.532 m^2/g and 0.1467 cm^3/g, respectively.

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