Research Article

Journal of Environmental Nanotechnology

Comparative Study of Chemically and Green Synthesized Titanium Dioxide Nanoparticles using *Leucas aspera* Leaf Extract

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Received: 14.08.2023 Accepted: 27.08.2023 Published: 30-09-2023

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ABSTRACT

In this research work, the chemical synthesis and green synthesis of Titanium dioxide nanoparticles using *Leucas aspera* leaf extract were successfully carried out with titanium isopropoxide as the precursor. Titanium dioxide nanoparticles are widely used in various fields such as biomedical applications (antibacterial, antifungal, anticancer, etc.). For the Titanium dioxide nanoparticles thus synthesized using a low-cost and eco-friendly approach, the morphology, crystalline size, functional group and bandgap were confirmed by Scanning Electron Microscope (SEM), X-ray Diffraction, Fourier Transform Infrared Spectroscopy (FTIR) and UV Spectroscopy techniques and antibacterial applications were carried out by Disc diffusion method.

Keywords: *Leucas aspera*; Green synthesis; Antibacterial; SEM; FTIR; TiO_2.

1. INTRODUCTION

Nowadays, cancer is a common and heterogeneous disease throughout the world. As the population increases (Kemp and Kwon, 2021) the probability of people getting affected by cancer also increases. Therefore, many studies aimed to treat cancer and overcome it as soon as possible. Surgery, chemotherapy and radiotherapy are the three rapidly used treatments with some side effects. Many studies are taken in this field to eliminate the side effects. Medical nanotechnology is one of the hopes towards this disease. Typical nanomaterials are from 1-100 nm in size (Nancy *et al.* 2022). They exhibit properties like high surface-to-volume ratio, unique fluorescence properties, permeability and outstanding compatibility, which are essential to overcome cancer (Ruirui *et al*. 2022). There are two approaches in nanotechnology. They are Top-down and Bottom-up approaches. Top-down approaches mainly involve evaporation–condensation processing methods, through which Titanium nanoparticles without atomic-level control are constructed from large entities. Meanwhile, Bottom-Up approaches mainly incorporate the electrochemical processing of metals (Bamal *et al.* 2021).

The main aim of nanotechnology studies in the cancer treatment area is discovering and designing nanoparticles for delivery and release of drugs to target cells (tumors) by enhancing the effect of the drug and also decreasing the side effects that are caused from surgeries or therapies. The extensively used nanoparticles for cancer treatment are zinc oxide (ZnO) (Shams *et al.* 2022) and titanium dioxide (TiO_2) (Caroline *et al.* 2023) nanoparticles. TiO_2 is a white, odorless and non-combustible powder which is abundantly available in nature. It has three different forms; these are anatase (tetragonal), rutile (tetragonal) and brookite (orthorhombic) which is more chemically active (Selin and Cigir, 2019).

2. MATERIALS AND METHODS

2.1 METHOD

Titanium dioxide (TiO_2) is a very good photocatalytic material with nontoxicity and environment-friendly characteristics. The other characteristics of this material are hydrophobicity, non-wettability, huge bandgap and chemical stability. *Leucas aspera* is a medicinal plant of Lamiaceae family which is distributed throughout India from the Himalaya down to Ceylon and commonly known as 'Thumbai'. The whole plant is used traditionally as an antipyretic and insecticide. The leaves are commonly used in chronic rheumatism, psoriasis and other chronic skin eruptions (Anjusha *et al*. 2017; Ramesh *et al*. 2021). Low-cost and eco-friendly methods are adopted for the preparation of TiO_2 nanoparticles (Karthika *et al.* 2021).

2.2 PREPARATION OF LEAF EXTRACTS

Experimental leaves of *Leucas aspera* were collected from the rural area around Erode district, Tamil Nadu, India. The leaves were washed thoroughly in running water for a few minutes to remove soil and dust particles. Again, the leaves were washed with deionized water. About 20 g of finely cut *Leucas aspera* leaves were taken and added with 100 ml of distilled water in a beaker and kept in a microwave oven for 30 minutes. The solution appeared pale green in colour and was filtered using Whatman No. 1 filter paper to get a clear solution. The collected solution was stored for further use.

Fig. 1: Physical appearance of TiO_2

2.3 SYNTHESIS OF PURE TiO_2 NANOPARTICLES

5 ml of titanium tetra isopropoxide was taken into a beaker and 100 ml of distilled water was added into it; the mixture was stirred for 30 minutes using magnetic stirrer. 1.0 g of sodium hydroxide pellets was added with 20 ml of distilled water and mixed well. This NaOH solution is added drop by drop to the stirred solution until it reaches the pH value 12. After maintaining pH, it was allowed to stir for 30 minutes by the magnetic stirrer. Then the stirred solution was kept for 30 minutes in a microwave oven at 70 W. Then the solution was dried in powder form, the above powder was ground finely by using mortar-pestle to obtained TiO_2 nanoparticles.

2.4 GREEN SYNTHESIS OF TiO_2 USING *Leucas aspera*

20 ml of freshly prepared pale green color Leucas aspera leaf extract was taken in a beaker and 5 ml of titanium tetra isopropoxide was added into a beaker with 100 ml of distilled water. The solution was stirred for 30 minutes by using a magnetic stirrer. To maintain pH 12, NaOH solution was added dropwise to the above mixture. The solution was again stirred for 30 minutes; the solution slowly changed from pale green to yellowish red colour. Then the solution was kept in a microwave oven at 70 W for 30 minutes. The dried sample was grained in a mortar with a pestle to finally get the green synthesized TiO_2 powder.

Fig. 2: *Leucas aspera*

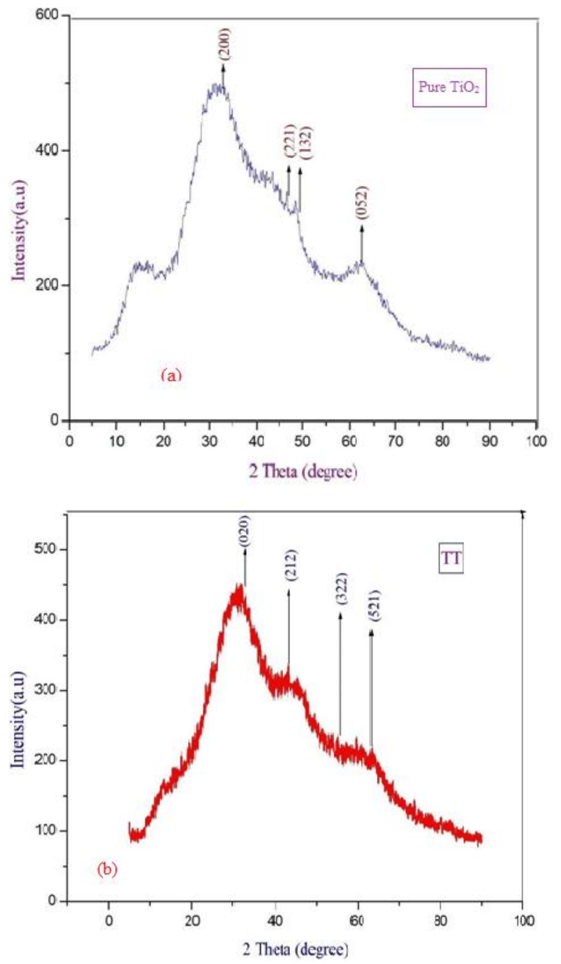

Fig. 3: XRD images of (a) Pure TiO_2 and (b) Green synthesized TiO_2 nanoparticles

3. RESULTS AND DISCUSSION

3.1 POWDER X-RAY DIFFRACTION ANALYSIS

XRD technique was used to confirm the structure and nature of the synthesized TiO_2 nanoparticles. The obtained XRD pattern for both pure and green synthesized TiO_2 are shown in Fig. 3. The prepared samples confirm the spherical structure and well matched with JCPDS file No: 761934. The broad diffraction peaks of the prepared TiO_2 are detected at values of 2ϴ=32.781, 43.58, 49.448, 62.5058, 32.32, 43.14, 57.77 and 63.86. The indexed hkl values are (2 0 0), (2 2 1), (1 3 2), (0 5 2), (0 2 0), (2 1 2), (3 2 2) and (5 2 1). The average crystalline sizes (D) of pure TiO_2 and green-synthesized TiO_2 were 3.683, 2.9697, 2.078, 2.7678, 2.0954, 1,594 and 1. 456 nm. Thus, the average crystalline size of pure TiO_2 is higher than that of the green synthesized TiO_2.

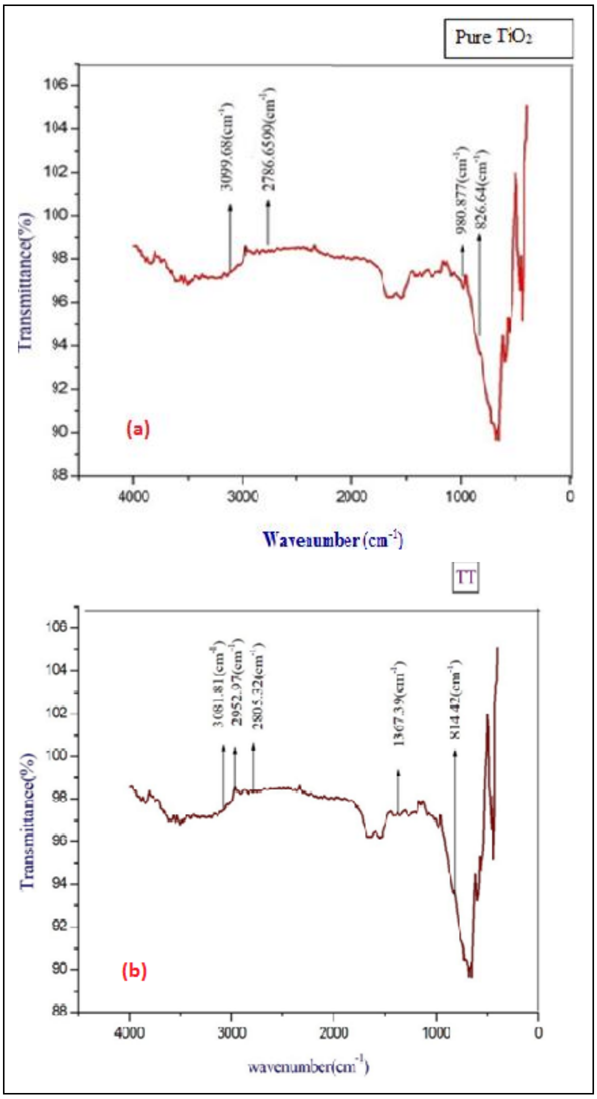

Fig. 4: FTIR images of (a) Pure TiO_2 and (b) Green synthesized TiO_2 nanoparticles

3.2 FTIR ANALYSIS

FTIR analysis was carried out to find the functional groups. The recorded spectrum is shown in Fig. 4. The board absorbed peak at 3099.68 cm^{-1} and 2989.65 cm^{-1} correspond to C-H stretching. C-H out-of-plane bending was confirmed at 814.42, 826.64 and 980.877 cm^{-1}. CH2 symmetric stretching was present at 2786.65 and 2805 cm^{-1}. The absorption peaks at 2952.97 and 1367.39 cm^{-1} were assigned to the antisymmetric C-H stretching and CH3 stretching. IR vibration observed at FTIR analysis confirms the successful formation of functional groups in both samples.

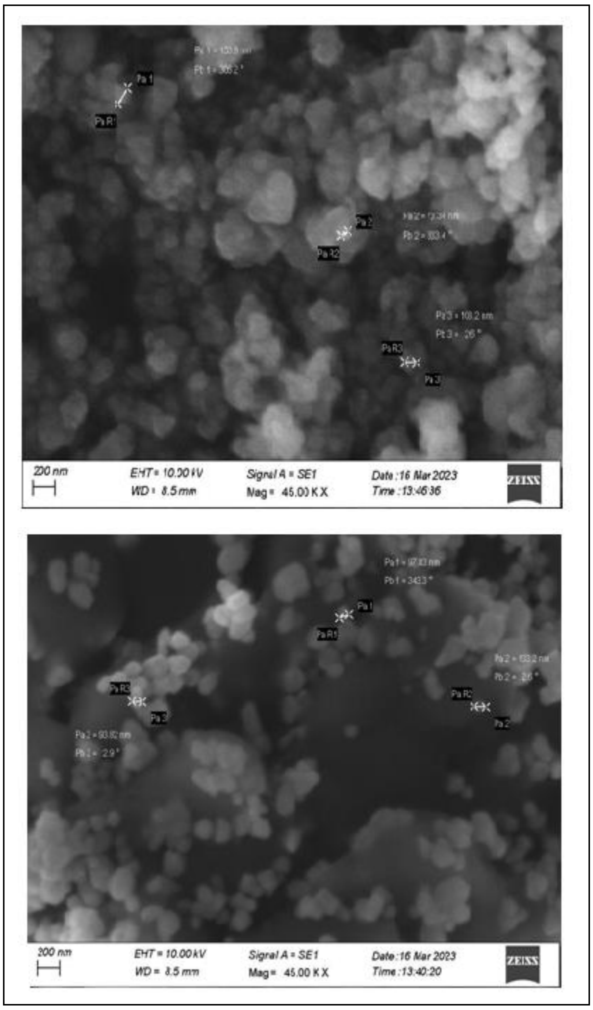

Fig. 5: SEM images of (a) Pure TiO_2 and (b) Green synthesized TiO_2 nanoparticles

3.3 MORPHOLOGICAL STUDIES

The surface morphology of pure prepared TiO_2 was characterized by SEM analysis. The synthesized pure particles are spherical in shape with good dispersion ranging from 93.82 to 103.2 nm in grain size. Fig. 5 shows the SEM image of green synthesized TiO_2 nanoparticles. The grain size of prepared green synthesized TiO_2 nanoparticles ranged from 73.34 to 150 nm of spherical shape; more agglomeration nanoparticles also appeared.

4. CONCLUSION

In this research work, both pure and green synthesized TiO_2 nanoparticles were successfully synthesized by Microwave irradiation method. The synthesized samples were characterized by XRD, FTIR and SEM analysis. The average crystalline size of pure TiO_2 is higher than green synthesized TiO_2 nanoparticles, due to the capping of *Leucas aspera* leaf extract.

The FTIR analysis has shown the different functional groups present in the two given samples. SEM has revealed the morphological structure of the prepared samples, depicting the spherical shape for both samples. Thus, the green synthesized TiO_2 nanoparticles are applied in biomedical applications such as treatment for cancer and snake bites.

FUNDING

This research received no specific grant from any funding agency in the public, commercial, or not-for-profit sectors.

CONFLICTS OF INTEREST

The authors declare that there is no conflict of interest.

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