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Green Route: Synthesis and characterisation of Titanium Dioxide Nanoparticles Using Neem Extract

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ABSTRACT: TiO₂ nanoparticles have been prepared by green synthesis method using neem extract. The structural properties of the synthesised titanium dioxide nanoparticles have been studied using x-ray diffraction method. The grain size was observed to lie in the range of 65 - 75 nm. Surface morphology of the prepared TiO₂ nanoparticles has been carried out using SEM. The band gap energy has been found to be 3.43 eV from the absorption spectra of titanium dioxide nanoparticles. The sample is subjected to FTIR and EDAX studies.

KEYWORDS: Green Synthesis, TiO₂ nanoparticles, Neem extract

I. INTRODUCTION

Nanostructured materials have received considerable attention in recent years because of their proven and potential applications in various fields. TiO₂ has gained attention and interest due to its exceptional properties, such as high refractive index and ultraviolet absorption, excellent incident photoelectric conversion efficiency, good photocatalytic activity, photostability, chemical stability, long-time corrosion resistance and nontoxicity. Nanomaterial's are of strong interest for both fundamental and technological reasons. At the fundamental level, nano materials possess novel physical and chemical properties that differ from those of isolated atoms or molecules and bulk matter due to quantum confinement effects. As the particle size get reduced to a size below its critical size, it will show variations in a wide range of properties including optical, magnetic, thermal, mechanical, electronic and environmental. These novel properties are made use of in a wide variety of applications to improve its efficiency.

Neem has been regarded as a wonder medicinal tree capable of solving a large number disease. It has been an integral part of both traditional and modern systems of medicine. Neem in its raw form, in extracts and as finished products finds various applications. All parts of Neem are used for preparing many different medicines.

Green synthesis of nano materials are finding increased attention from the researches because of its simplicity, fastness, non-toxicity and economical approach. In the present work, we have prepared titanium dioxide nano particles by green route method using neem extract and the prepared nanoparticles were characterized using various techniques.

II. EXPERIMENTAL

Neem leaves were collected in and around the areas Coimbatore district, Tamil Nadu, India. All the chemicals and reagents were used as received without further purification. Aqueous leaf extract of neem was prepared using freshly collected leaves (25 g). They were surface cleaned with running tap water and followed by distilled water. The cleaned leaves were cut into small pieces and boiled with 250 mL of distilled water at 80⁰C for 2 hours. The extract was filtered using what man filter paper. The filtrate was stored for the synthesis of Nano particles.

The Neem leaves extract was added drop wise to 7.4 ml of titanium tetra isopropoxide under constant stirring to achieve a solution of pH 7. The mixture was subjected to stirring for 4 hours continuously. In this process Nano particles were formed and were separated using filter paper. The particles were washed with double distilled water repeatedly to remove the by-products if any. The Nano particles were dried at 100 °C for one hour and calcinated at 500 °C for 4hours. Flow chart depicting the green synthesis of Nano crystalline TiO₂ is shown in figure 2.1

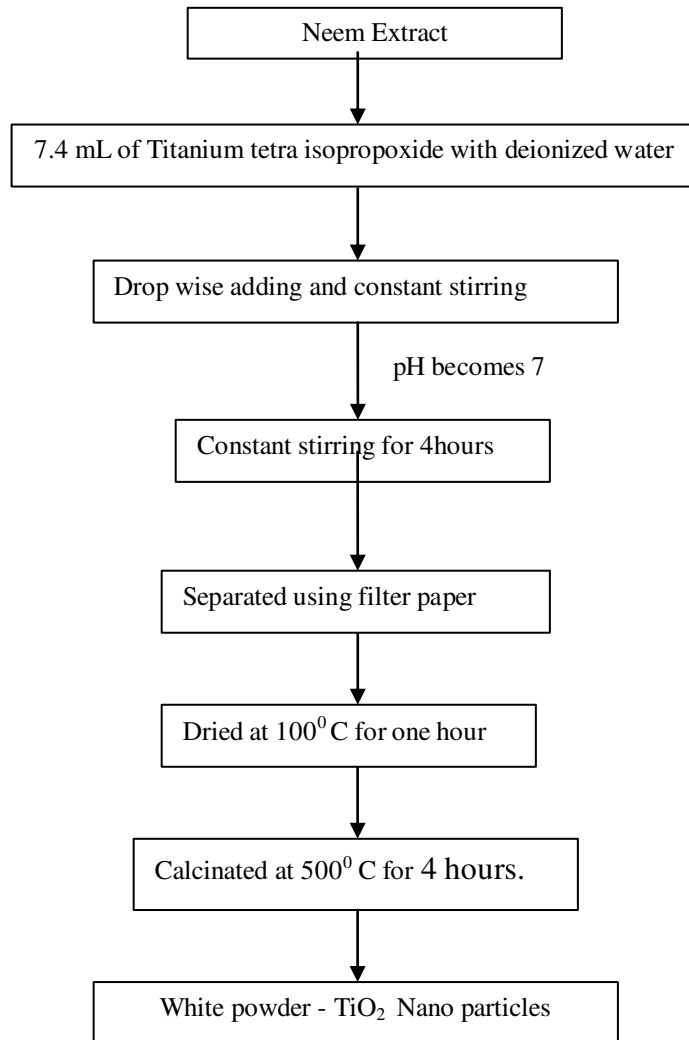


Fig 2.1 Flow chart for the synthesis of TiO₂ Nanoparticles

III. RESULT AND DISCUSSION

3.1 X-Ray Diffraction Analysis

XRD is a powerful non-destructive technique for characterizing crystalline materials. Hence, in order to study the phase formation and micro structure studies, the XRD analysis was carried out on the prepared TiO₂ nanoparticles. XRD pattern of the synthesized titanium dioxide nanoparticles showed the presence of anatase forms. The XRD sample showed a dominant peak at 25.4158, 48.1125 and 38.1166 which were found to be (101), (200) and (004) reflections, respectively and confirmed the nanocrystalline nature. The particles size estimation was done by the Debye-Scherrer's formula.

The Debye-Scherrer's equation is,

$$D = k\lambda / \beta \cos\theta$$

Where,

D = crystallite size



λ = wave length of the filament used in XRD machine (1.5418 Å)
 β = Full Width Half Maximum (FWHM)
 θ = angle of the corresponding peak
 k = shape factor(0.94)

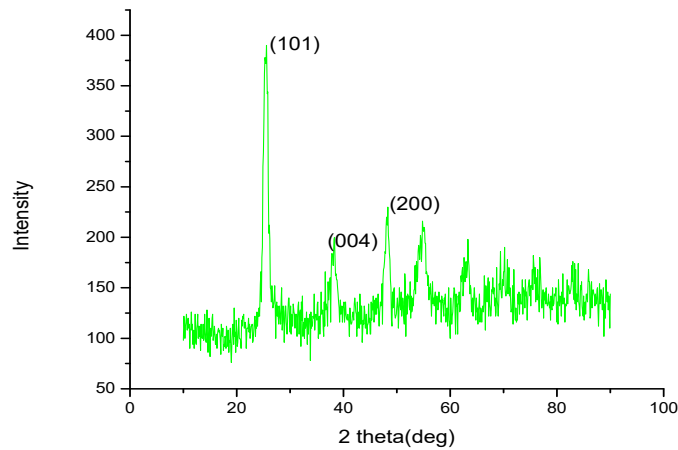


Fig 3.1 XRD pattern of TiO₂ nano particles calcinated at 500⁰C

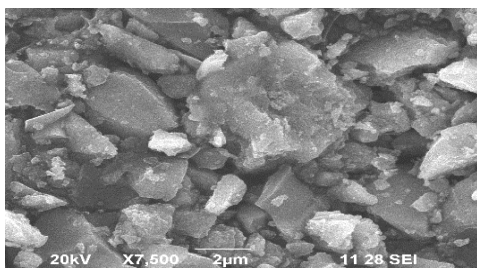
The peak details of the TiO₂ nanoparticles are listed below in the table 3.1.

2θ (deg)	FWHM (deg)	Crystalline size (nm)	d (Å)
25.4158	1.08380	75.1341	3.500
48.1125	1.05000	82.8488	1.888
38.1166	1.26670	66.3474	2.358

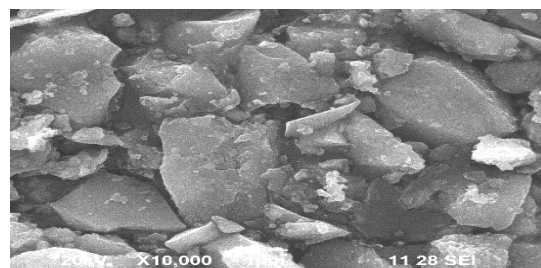
Table 3.1 - The peak details of the TiO₂ nanoparticles

3.2 Scanning Electron Microscope (SEM)

The surface morphology of titanium dioxide nanoparticles have been studied by SEM. Synthesized titanium dioxide nanoparticles was smooth and showed agglomeration of spherical cluster. The images showed the synthesized nanoparticles in various magnifications 7500x, 10000x, 30000x, 55000x which clearly gives physical morphology, particle size and aspect ratio (Figure 3.2 A, B, C and D).



(A)



(B)

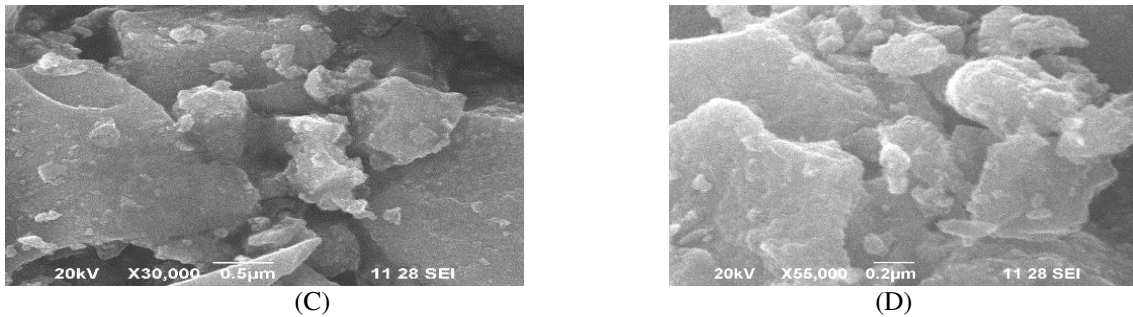


Fig 3.2 (A,B,C and D) SEM image of TiO₂ nanoparticle

3.3 ENERGY DISPERSIVE X –RAY ANALYSIS (EDAX)

The EDAX spectrum revealed the presence of titanium and oxygen in the prepared samples. It also indicated the presence of some impurities in the prepared sample which is due to neem extract involved in the preparation. The principal constituents of neem leaves include protein , carbohydrates , minerals, calcium, phosphorus etc., hence these elements were present in the prepared sample. EDX showing the chemical composition confirms the presence of the titanium metal.

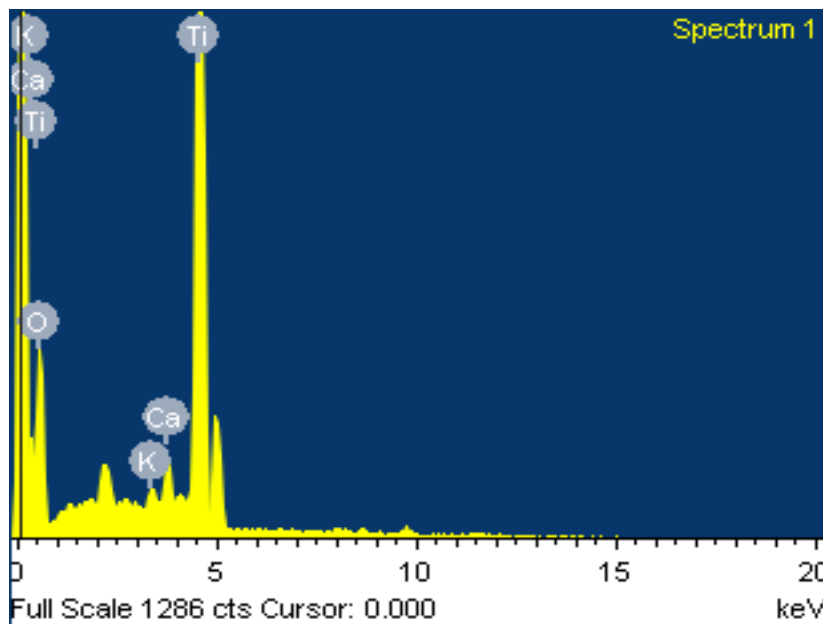


Fig 3.3 EDAX spectrum of TiO₂ nanoparticle

3.4 UV - Visible Spectroscopy

The energy band gap value was calculated based on the mathematical derivative of the optical absorption coefficient. The fundamental absorption method corresponds to band to band transitions by using energy relation. The energy band gap was found using the relation,

$$E = 1240/\lambda$$

The UV-VIS spectra of TiO₂ nanoparticles is shown in fig (3.4). The band gap energy calculated as 3.43 ev.

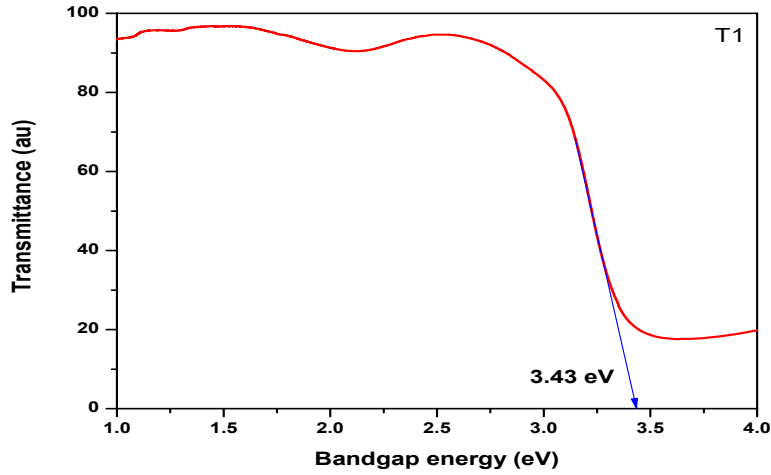


Fig 3.4 UV- VIS spectrum of TiO₂ nanoparticles

3.5 Fourier Transform Infra – Red Spectroscopy (FTIR)

FT-IR spectra identify chemical bonds in a molecule by producing an infrared absorption spectrum. FTIR spectrum of TiO₂ sample annealed at 500°C show peaks corresponding to stretching vibrations of the O-H.

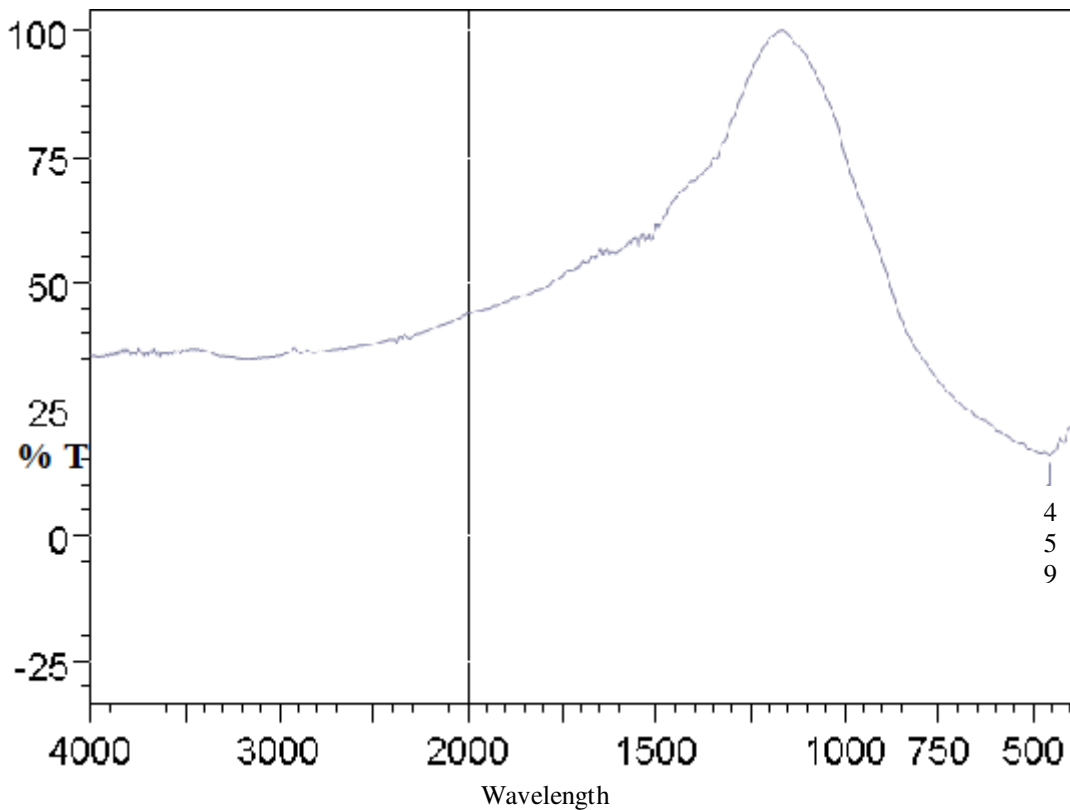


Fig 3.5 Fourier Transform Infra – Red Spectroscopy of TiO₂ nanoparticles



IV. CONCLUSION

The titanium dioxide nanoparticles were synthesized from Green synthesis by Neem. The XRD pattern shows a strong diffraction peaks at $2\theta = 25^\circ, 48^\circ, 38^\circ$. Along (101) (004) (200) directions in agreement with hexagonal structure. The SEM images clearly shows the agglomeration of TiO_2 nanopowders and formation of a spherical like structure. The FTIR spectrum showed that the synthesized product is pure TiO_2 nanoparticles with the monoclinic phase. The EDAX spectrum green synthesis of Ti and O present in the samples according to the energy dispersive spectrum (EDX) analysis of the annealed at 450°C of TiO_2 samples. The UV – visible spectroscopy shows a strong absorption and the band gap energy was found to be 3.43 eV.

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