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Research Article

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Effect of Boat Lilly Leaf Extract on the Properties of Titanium Dioxide Nanoparticles

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Abstract

Nature inspires researcher to carry out new research in the science and technology, especially in the field of nanotechnology. In those natural systems Boat Lilly plant is one having very important medical properties and hence have used here. Now-a-days green synthesis metal oxide nanoparticles gained importance in the field of opto-electronics as well as in photo-electronics industry. Keeping this in view, in our present work we made an attempt for the synthesis of Titanium dioxide (TiO_2) nanoparticles from leaf extract of Boat Lilly plant using green synthesis method. The obtained TiO_2 nanoparticles have been characterized by X-ray Diffractometer (XRD), UV-Visible Spectroscopy (UV-Vis), Scanning Electron Microscopy (SEM), Energy Dispersive X-ray Spectrometer (EDAX) and the dielectric measurements.

Keywords

Boat lilly; Green synthesis; TiO_2 nanoparticles; X-ray diffractometer; Scanning electron microscopy; Dielectric measurement

Introduction

A significant region of examination in nanotechnology manages the blend of nanoparticles of various compound arrangements, measurement and controlled monodispersity. Nanotechnology is rising as a quickly developing field with its application in science and innovation to fabricate new materials at the nanoscale level [1]. Titanium dioxide (TiO_2) is an inert, non-toxic and inexpensive material, whose high refractive index and high capability to absorb UV light make it an interesting white pigment and environmentally friendly catalyst [2]. The nanosized TiO_2 particles are widely used to provide whiteness and opacity to products such as sunscreen lotions, paints, plastics, papers, inks, food colorants and toothpastes [3]. There are various methods available for synthesis of Titanium dioxide nanoparticle, green synthesis is one of them. The green synthesis process is eco-friendly technique due to use of extracts of plant (leave, flower, seed and peels), bacteria, fungi and enzymes for synthesis of nanoparticles instead of large quantity of chemicals [4]. Green synthesis provides more advantages over physical methods and chemical methods because it is very cost effective, easy process and scalable for large scale production. This method do not require high temperatures, high pressure, costly equipment and hazardous chemicals so it is eco-friendly and pollutant free [5].

The plant extracts used in the green synthesis method may act as reducing agent or stabilizing agent, so the characteristic of nanoparticle depends on the source of plant extract [6]. In the recent past many researchers investigated the importance of green synthesis NP over chemically synthesized NP in various aspects [7-10].

The boat lilly leaves are used for cough, colds, hemoptysis, whooping cough, nose bleed, bacillary dysentery, blood in the stool and it is also used for lymphatic tuberculosis. With reference to the present literature available, we found that, no work was reported with boat lilly extract till date. So we made a first attempt to prepare TiO_2 nanoparticles using boat lilly extract via green synthesis method and the results are reported herein.

Materials and Methods

Collection and preparation of leaf extract

Fresh and young greenish leaves were collected from boat lilly plant grown in the red clay soil in the area of Nagercoil, Kanyakumari District, Tamil Nadu (Figure 1). The surface of the leaves were washed thoroughly using distilled water to remove dust particles for about 3 times. Each of these leaves were completely wiped with white color Khadhi cloth and dried at room temperature for 2 hrs. Then the weight of the leaves were measured as needed for testing. The weighed leaves were cut into tiny pieces, grinded and then were mixed with 50 ml of distilled water. The obtained solution was filtered separately in a clean beaker using Whatmann filter paper.

Synthesis of titanium dioxide nanoparticles

In the present study, titanium dioxide has been prepared using Solvothermal method. 18 ml of titanium tetra chloride was completely dissolved in 50 ml of Ethylene glycol and 18 grams of Urea was mixed with 50 ml of Ethylene glycol. The precursors were mixed together and heated in microwave oven for nearly half an hour to obtain pure titanium dioxide nanoparticles. The nanoparticles were separated

Figure 1: Leaves of boat lilly.

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using Whatmann filter paper and the resultant sample was washed using distilled water repeatedly to remove the by-products, the sample was dried for few days and again it was washed using acetone to remove the organic impurities (if present any). To the above precursors extract of boat lilly leaves were added to get boat lilly mixed TiO_2 samples. This procedure was repeated for different concentrations of boat lilly leaf extracts (3 ml and 5 ml respectively).

The prepared samples of pure TiO_2 and boat Lilly leaf extract added TiO_2 samples were characterized by X-ray Diffractometer (XRD), UV-Visible Spectroscopy (UV-Vis), Scanning Electron Microscopy (SEM), Energy Dispersive X-ray Spectrometer (EDAX) and the dielectric measurements.

Characterization

To identify the structural properties of green-synthesized TiO_2 nanoparticles, X-ray diffraction studies were carried out using Pro Penalty CAL with CuKα (λ=1.5461 Å) radiation. Shimadzu UV-2400 PC spectrophotometer was utilized to study the different regions such as ultraviolet and visible regions between 200 nm and 800 nm. Ultraviolet/visible analysis was used to study the effect of TiO_2 NPs on the optical properties and used to calculate the energy gap (Eg) of prepared nanocomposites. Morphological properties were studied using SEM. The elemental composition of the prepared nanoparticle samples were analysed by EDAX studies. The electrical properties of TiO_2 nanoparticles were studied using HIOKI 3532-50 LCR HITESTER, at room temperature, between the frequency ranges of 50 Hz and 5 MHz. The parameters such as ac conductivity, dielectric constant and dielectric loss were also measured.

Results and Discussion

Powder X-Ray Diffraction (PXRD) analysis

The prepared samples were subjected to X-ray diffraction measurements. XRD analysis showed six distinct diffraction peaks at 34°, 49°, 54°, 55°, 56°, 62°, 65°, 70°, 73°, 75°, and 79° along with the miller indices values (0 2 0), (1 2 2), (1 3 1), (5 1 1), (4 1 2), (3 3 1), (6 1 1), (3 3 2), (6 2 1), (0 0 4), (5 3 1) and (0 4 2) respectively (For pure). TiO_2 mixed with 3 ml of boat lilly leaf extract has 2θ peaks absorbed at an angles 27°, 36°, 44°, 54°, and 76° along with the miller indices values (1 1 0), (1 0 1), (2 1 0), (2 1 1) and (2 0 2) respectively, similarly TiO_2 mixed with 5 ml of boat lilly leaf extract has 2θ peaks absorbed at an angles 38°, 39°, 60°, 63°, 74 and 77° along with the miller indices values (1 2 1), (4 0 0), (6 0 0), (5 2 1), (1 0 4) and (4 2 3) respectively of the Orthorhombic titanium dioxide((JCPDS No.76-1934). The average grain size formed in the green synthesis was determined using Scherrer's formula, d=0.9l/bcos.

X-ray diffraction patterns are shown in the Figures 2-4 below. The average crystallite size for pure and boat lilly leaf extract added samples were found as 49 nm, 45 nm and 46 nm respectively using above formula.

The lattice parameter was calculated from the formula is,

$$\frac{1}{d^2}=\frac{h^2}{a^2}+\frac{k^2}{b^2}+\frac{l^2}{c^2}$$

Using the above relation, the lattice parameter value of pure TiO_2 is a=9.22661, b=5.42418, c=5.1428 and mixed with 3 ml of boat lilly leaf extract is a=9.21254, b=5.45363, c=5.14004 and mixed with 5ml of boat lilly leaf extract is a=9.19341, b=5.49716, c=5.15123. It matched with the JCPDS value a=9.174, b=5.449, c=5.138.

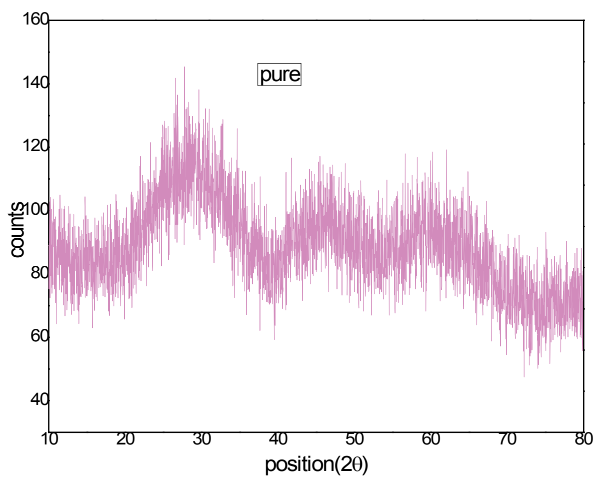

Figure 2: XRD pattern of pure TiO_2.

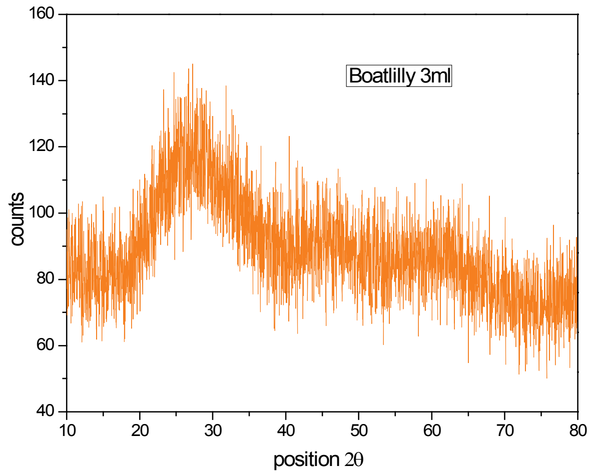

Figure 3: XRD pattern of TiO_2 mixed with 3 ml of boat lilly extract.

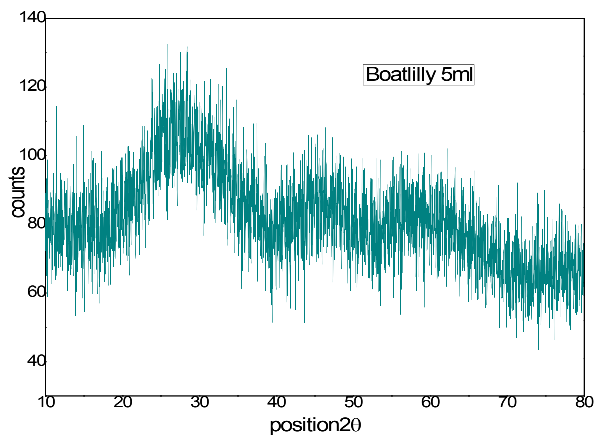

Figure 4: XRD pattern of TiO_2 mixed with 5 ml of boat lilly extract.

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UV visible studies

The Optical properties of the prepared nanoparticles were studied using UV-Visible spectrometer. Figures 5-7 show the energy band gap of the sample in nano-range. The spectra exhibited between 200 nm and 800 nm ranges were studied. The optical measurements of the pure TiO_2 and samples mixed with boat lilly leaf extract can be used to calculate the optical band gap energy (Eg) by using Tauc plot. Energy Band gap of the material was calculated by the formula,

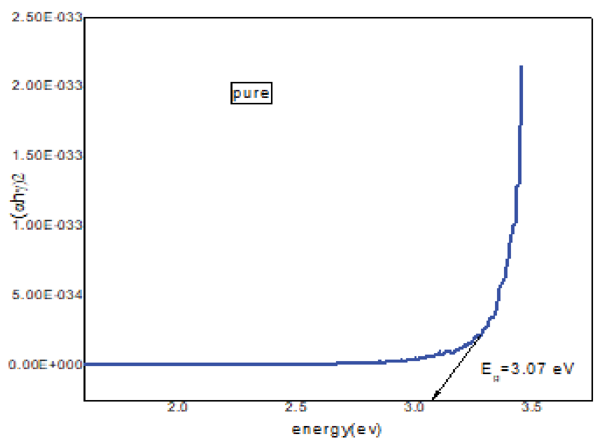

Figure 5: Tauc plot for pure TiO_2.

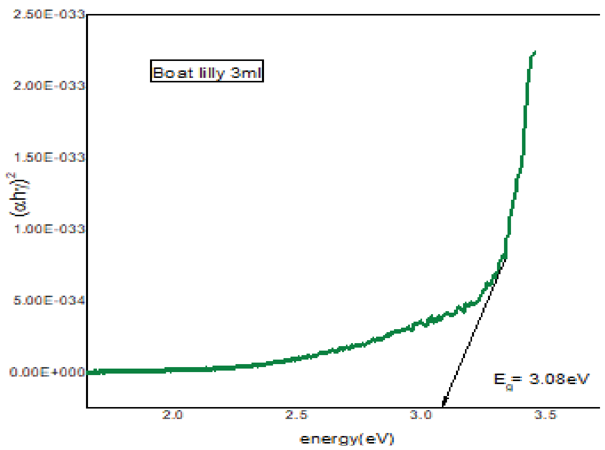

Figure 6: Tauc plot TiO_2 mixed with 3 ml of boat lilly leaf extract.

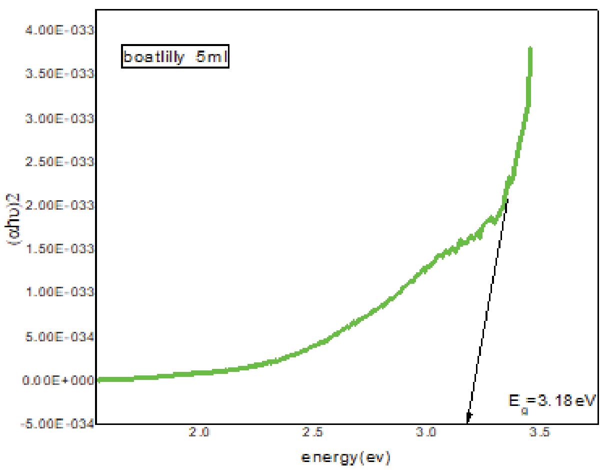

Figure 7: Tauc plot TiO_2 mixed with 5 ml of boat lilly leaf extract.

$$E=h.c/\lambda$$

Where h is Planck's constant, c is the velocity of light and l is the wavelength of maximum absorbance. The relation between the incident photon energy (hν) and the absorption coefficient (α) was got from the formula

$$(\alpha h\nu)n=A\ (h\nu\text{-}Eg)$$

where A is the constant related to the material, Eg is the band gap energy of the material in eV, h is the planck's constant (h = 6.626 × 10^{-34} J s), n is an exponent that can take a value of n=2 for direct allowed transition, n=½ for an indirect allowed transition and n=2/3 for the direct forbidden transition.

In order to find the possible transitions, the Tauc plots were drawn (αhν)n versus photon energy (hν). The corresponding band gap energy was got by extrapolating the linear portion of the curve to the photon energy (hν) axis (absorption equal to zero). The optical band gap observed for the samples under study are in the range 3eV-3.5eV. Energy band gap calculated from the above formula for pure and boat lilly mixed TiO_2 are 3.07 eV, 3.08 eV and 3.18 eV respectively.

SEM studies

The grain size, shape and surface properties like morphology were investigated by the scanning electronic microscopy shown in Figures 8-10. This image was observed within the magnification of 10 µm. From the figures, the nanoparticles are appearing to be aggregated and the surface of the aggregates is rough. Scanning Electron Micrograph (SEM) was equipped with Energy Dispersive Spectroscopy (EDX). The TiO_2 nanoparticles were showing irregular particles structure.

Elemental analysis (EDAX)

EDAX is an analytical technique used for the elemental analysis or chemical characterization of a sample. EDAX spectrum was recorded for the prepared samples. Figures 11-13 show the presence of Ti and

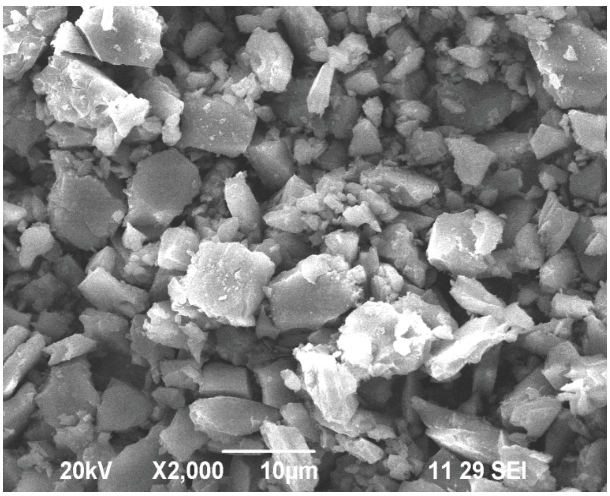

Figure 8: SEM micrograph of pure TiO_2.

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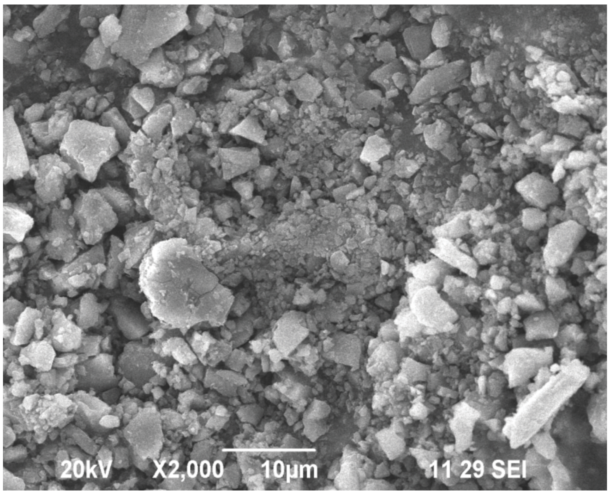

Figure 9: SEM micrograph of TiO_2 mixed with 3 ml of boat lilly extract.

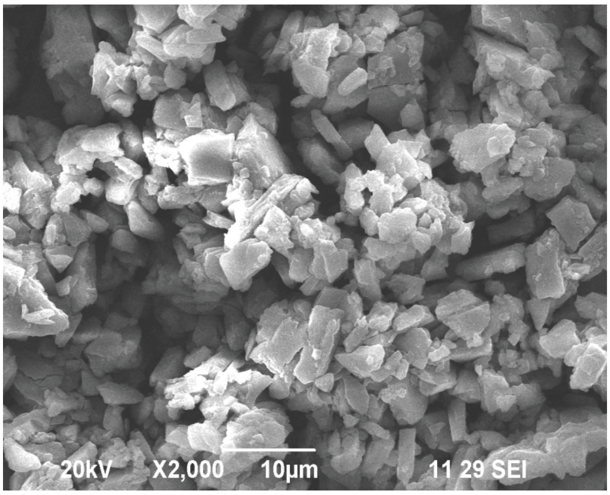

Figure 10: SEM micrograph of TiO_2 mixed with 5 ml of boat lilly extract.

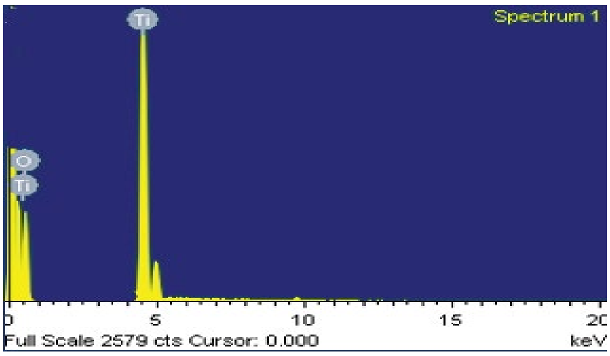

Figure 11: EDAX spectrum of pure TiO_2.

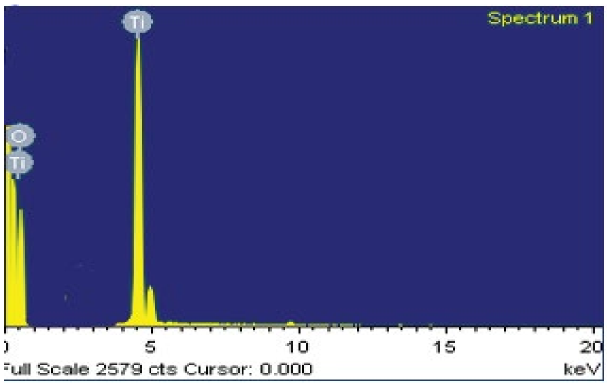

Figure 12: EDAX spectrum of TiO_2 mixed with 3 ml of boat lilly extract

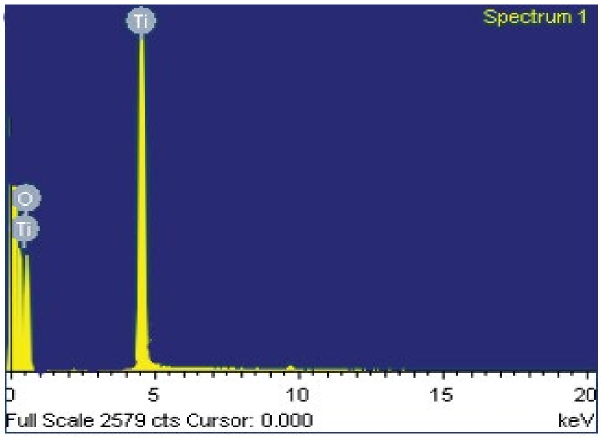

Figure 13: EDAX spectrum of TiO_2 mixed with 5 ml of boat lilly extract

O in the prepared samples at the exact energy region. It confirms that they are free from any other contaminants.

Electrical studies

The change in the dielectric constant (ε_r) and the dielectric loss (tan δ) with the function between 50 Hz and 5 MHz at various temperatures for TiO_2 nanoparticles are recorded in the figures below. The measurement of dielectric constant and dielectric loss as a function of frequency and temperature is of interest both from the theoretical point of view and from the applied aspects. Figures 14-16 show the variation of dielectric constant (ε_r) with various temperature and frequency. It is observed that for all the proposed samples the ε_r values increases with increase in temperature and decreases with increase in frequency. The tan δ values also increases with increase in temperature and it decreases with increase in frequency like dielectric constant. The increase of dielectric constant and dielectric loss is due to dipolar polarization [9-11].

Figures 17-19 show the variation of AC electrical conductivity (σac) with various temperature and frequency. The AC conductivity results clearly indicates that for all the proposed samples the σac values increase with increase in temperature and frequency. It also indicates that σac remains constant at low frequencies and as frequency increases, the conductivity increases exponentially. Hence the conduction mechanism of the proposed samples is localized. When the frequency increases, the localized state also increases. Therefore, high conductivity value was observed for high frequency and it originates from hopping of charge carriers between two states which confirms small polar on hopping present in the proposed samples.

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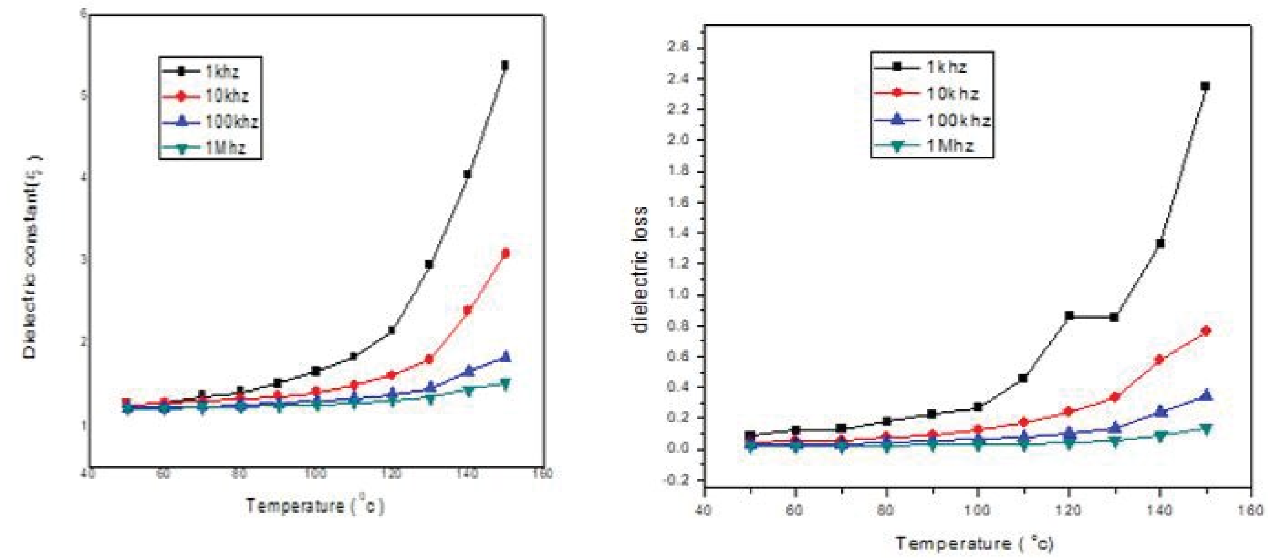

Figure 14: Variation for pure TiO_2.

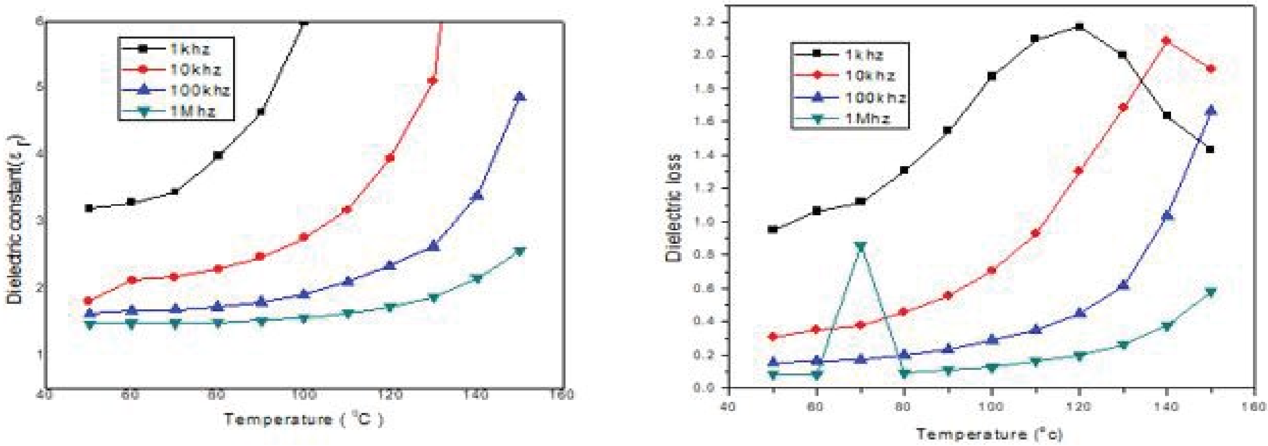

Figure 15: Variation for pure TiO_2 mixed with 3 ml of boat lilly extract.

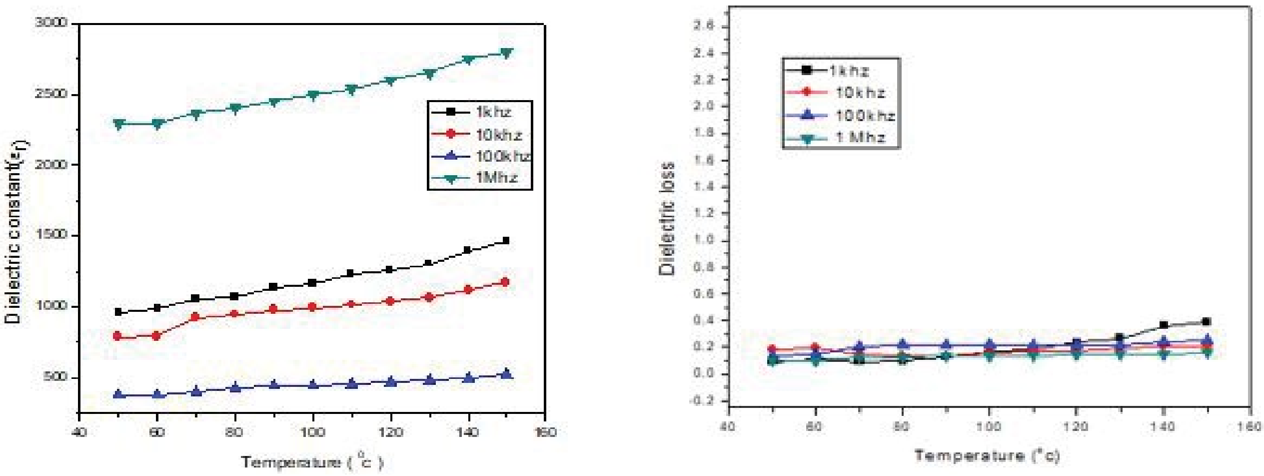

Figure 16: Variation for pure TiO_2 mixed with 5 ml of boat lilly extract.

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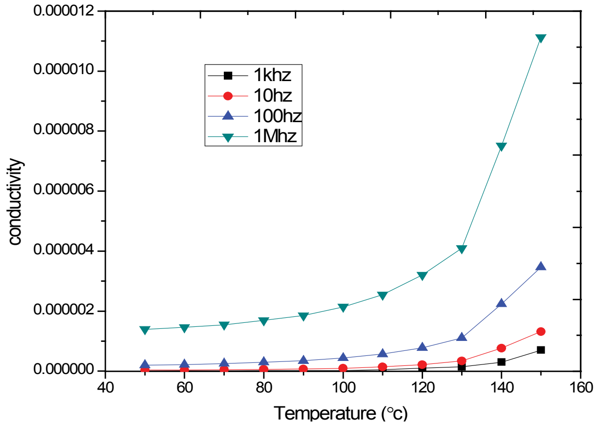

Figure 17: Variation of ac conductivity with temperature for different frequencies of pure TiO_2 nanoparticles.

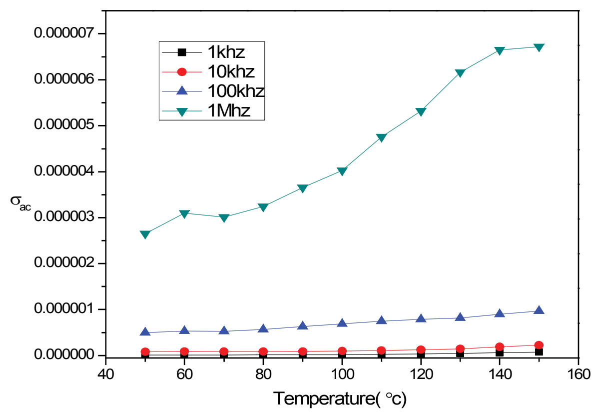

Figure 18: Plot of ac conductivity (In σac) versus 1000/T of 3 ml boat lilly leaf extract mixed TiO_2 nanoparticles.

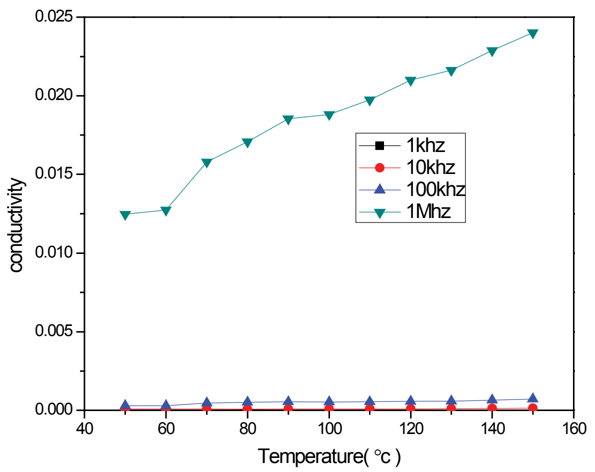

Figure 19: Plot of ac conductivity (In σac) versus 1000/T of 5 ml boat lilly leaf extract mixed TiO_2 nanoparticles.

Conclusion

The TiO_2 nanoparticles were prepared using Solvothermal method mediating green and characterized. From XRD analysis average crystallite size of the pure TiO_2 and TiO_2 mixed with 3 ml and 5 ml of boat lilly leaf extracts were obtained as 49 nm, 45 nm and 46 nm respectively. The XRD pattern depicted that the prepared nanoparticles were orthorhombic in structure. The optical properties were studied by UV-visible spectroscopy, it infers that the energy band gap of pure TiO_2 and TiO_2 mixed with 3 ml and 5 ml of boat lilly leaf extract has been obtained as 3.07 eV, 3.08 eV and 3.18 eV respectively, which is in good agreement with that reported in literature. The orthorhombic irregular particle structure was observed in SEM image. EDAX confirms that they are free from any other contaminants. The variation of dielectric constant and dielectric loss at greater frequencies range is salient for photonic and electro-optic application. The increase of dielectric constant and dielectric loss with temperature is due to the interfacial and dipolar polarization. The value of σac increases with increasing temperature and frequencies due to the migration of electron which may depend upon the polarization effects.

TiO_2 has certain applications in our day to day life such as water purification, photo catalytic properties, hence its used in antiseptic and antibacterial composition, UV resistant material etc. The present work proves that the boat lilly leaf extracted green synthesis is a new useful, non-toxic method using cheap precursors for the preparation of titanium dioxide nanoparticles.

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