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Green synthesis of TIO_2 nanoparticles using *Ocimum basilicum* extract and its characterization

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Abstract
Nanotechnology is an escalating field, which has an growing impact in each and every sector. There are many ways for synthesising nanoparticles but biosynthetic route of nanoparticle synthesis is emerging as a new and safer alternative to the conventional methods. *Ocimum basilicum* is an age old herb native to Southeast Asia and Central African countries. These plants are generally known to be used in different cuisines world-wide. The aroma emitted from the leaves and the twigs of the fresh and dried plants are well known to have insect repellent property. Estragole, citronellal, limonene and nerolidol are the natural volatile compounds present in the basil leaves which acts as repellents. This plant is also widely used as a scent through which it netted its title Queen (*basileus*) of aromatic herbs. The present study focused on the preparation of Titanium dioxide (TiO2) nanoparticles with basil leaf extract using Green synthesis method in two forms before and after calcination to be used as mosquito repellent and antibacterial finishes to textiles. The obtained nanoparticles were characterized by X-ray Diffractometer (XRD), Particle size Analyzer (PSA), Scanning Electron microscopy (SEM) and Fourier Transform Infrared Spectroscope (FTIR) for average crystalline size, average particle size, morphology and structure respectively.

Keywords: Green synthesis method, TiO2 nanoparticles, *Ocimum basilicum*, XRD, SEM, FTIR, PSA

Introduction

In recent years nanotechnology gained utmost importance in industrial, pharmaceutical, environmental and health care applications due to their unique physical and chemical characteristics [3]. TiO2 is poorly soluble, non-flammable, thermally stable and not classified as hazardous according to the United Nations (UN) Globally Harmonized System (GHS) of Classification and Labeling of Chemicals [4]. Titanium is a strong metal which is corrosion resistant and lustrous in appearance. Titanium based nanoparticles were proven to be chemically stable, high photocatalyst activity and strong oxidizing power. This is one of the metal which is nontoxic in nature. The main uses of titanium-based nanoparticles include - it helps in pollution degradation, Self-cleaning and sensors fabrication. In plants, titanium helps in stimulation and production of carbohydrates and improves the rate of photosynthesis. Conventional method of metal and metal oxide nanoparticle preparation involves various physical and chemical methods which includes non-sputtering, solvothermal, reduction, sol-gel technique and electrochemical technique. These methods are costly, lethal and potentially unsafe to environment. Metal and metal oxide nanoparticles were said to have higher surface area and high fraction of atoms. Because of these reasons green synthesis of nanoparticles, a one-step cost effective, eco-friendly, pollution free method of synthesis was given major importance in this experiment. This method consumes less time and energy and provides homogenous composition of particles with high yield compared to other methods. The present study on green synthesis of TiO_2 nanoparticles is based on *Ocimum basilicum* (Basil) Plant extract. Basil is also called with other names like sweet basil because of its sweet flavour with strong and pungent smell. Basil belongs to the family Lamiacea which is most popular and extensively grown herb especially in Asia. The extracts of *Ocimum basilicum* is frequently cited as being used in many medical treatments, antibacterial and many insect repellent activities. The rich pungent aroma of basil leaves is said to be because of linalool, methyl chavicol and 1,8-cineole [5], and methyl cinnamate [2, 5] as the dominant component which helps in repelling mosquitoes.

Methodology

Experimental Details

The preparation of Titanium based nanoparticles using green synthesis method was carried at Department of Nano Science and Technology, JNTU Hyderabad, Telangana. The fresh leaves of Basil were collected from PJTSAU campus Hyderabad. Fifty grams of fresh leaves were separated from the plant and was thoroughly washed 3-4 times under running water and 2 times with distilled water. These leaves were dipped in 125ml of distilled water and was boiled for 2 hours at 70^0C. The extract was filtered using what man filter paper. This precursor (extract) was used for the synthesis of TiO_2 nanoparticles.

10 ml of Titanium Isopropoxide (TTIP) was added drop wise to 250 ml of distilled water while the distilled water is on stirring continuously. This whole solution was allowed to stir for half an hour and precursor was added to this solution till the solution achieves a PH of 7. This whole mixture was subjected to stirring at 980 RPM for 4 hours and kept aside. After 12 hours, the solution was filtered using what man paper in order to remove by-products and the wet cake was collected dried at 70^0C for 12 hrs in Hot air Oven. The nanoparticles were collected in powder form as Before Calcinated Nano particles. Measured quantity of Nano powder was kept in Muffle furnace and calcinated at 400^0C for 2 hours in order to collect After Calcinated Nanoparticles.

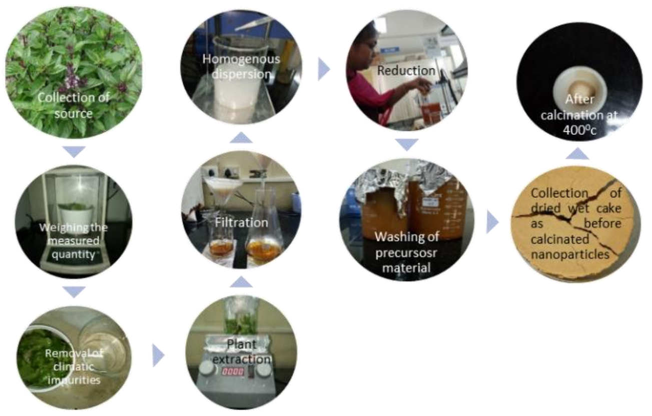

Fig 1: Preparation process of TiO_2 nanoparticles using Basil extracts through Green synthesis Method

Characterisation Techniques

The crystalline structure and average crystalline size was measured by Bruker D8 X-ray diffractometer. Shape and elementary analysis of the nanoparticles were estimated by S 3400 N Scanning Electron Microscope. The average particle size was obtained by HORIBA SZ-100 Particle Size analyser.

Results and Discussion

X-Ray Diffractometer

The XRD pattern of TiO_2 nanoparticles before and after calcination at 400^0c obtained from green synthesis were shown in the figure 2 and 3. The results of the XRD showed that the structure of nanoparticles were tetragonal (anatase) and the results were coincided with JCPDS Card no 21-1272. Peaks were observed at 25^0, 38^0, 48^0, 53^0, 55^0, 62^0 and 75^0 along with miller indices values (1 0 1), (0 0 4), (2 0 0), (1 0 5), (2 1 1), (2 0 4) and (2 1 5) respectively. The average crystalline size was calculated by Debye-Scherrer's formula [6].

$$D = \frac{K\lambda}{\beta.\ Cos\ \theta}$$

Where D represents the average grain size of the material. K- is Debye Scherrers Constant (= 0.94), λ is the wavelength of the radiation and β is the full width half maximum of the peak, θ is the Braggs angle. The sharp and strong peaks indicates the high crystalline structure of the nanoparticles. The size of the nanoparticles are inversely proportional to the width of the peak ie., as the width of the peak increases, size of the nanoparticles decreases, which represents the presence of material in nonorange.

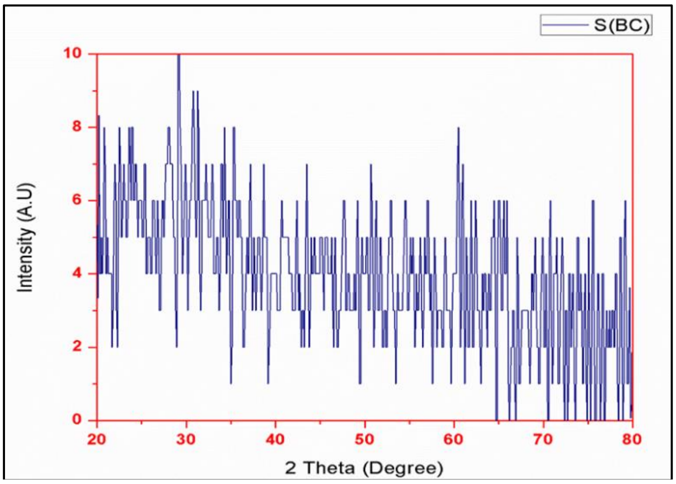

Fig 2: XRD Pattern of TiO_2 Before Calcinated Nanoparticles

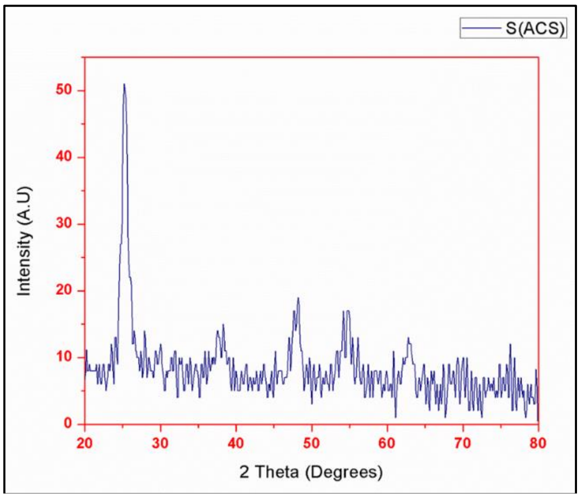

Fig 3: XRD Pattern of TiO_2 After Calcinated Nanoparticles at 400^0C

A sharp diffraction peak was observed in after calcinated TiO2 nanoparticles, whereas, the intensity of diffraction peak of green synthesized before calcinated TiO2 nanoparticles is less with slight broadening. The lattice parameters obtained were close and consistent with standard data for TiO2 (JCPDS 21-1272).The calculated crystallite was found to be 18 nm for before calcinated nanoparticles and 20nm for after calcinated TiO2 nanoparticles respectively. The XRD peaks of green synthesized before and after calcinated TiO2 nanoparticles obtained using extract of basil differ in the broadening and intensity. The diffraction peak of the green synthesised before calcinated TiO2 nanoparticles is broadened, whereas the peak of after calcinated TiO2 nanoparticles is comparatively sharp [7]. Thus, the broadening of XRD peak of green synthesised before calcinated TiO2 nanoparticles observed confirms the size reduction. The XRD peak of green synthesized after calcinated TiO2 nanoparticles is sharp, thus indicating that their size is still larger than the before calcinated TiO2 nanoparticles. The intensity of the diffraction peak of before calcinated TiO2 nanoparticles is less when compared to after calcinated TiO2 nanoparticles. Therefore, it can be suggested that the phytochemicals present in the *Ocimum basilicum* extract would have coated the surface of the TiO2 before calcinated nanoparticles, resulting in decreased intensity in XRD peak. This phytochemical coating may enhance the stability and the dispersibility of the nanoparticles, which in turn may enhance their bioavailability, making them suitable for biological applications [8].

Particle size Analyser

The particle size analyser works on the principle of dynamic light scattering and is used to know about average particle size of the nanoparticles. TiO_2 based basil nanoparticles were dispersed in distilled water and ultrasonicated for half an hour. The particle size distribution was studied using the histogram of the dispersed nanoparticles which was represented in Fig. 4 & 5

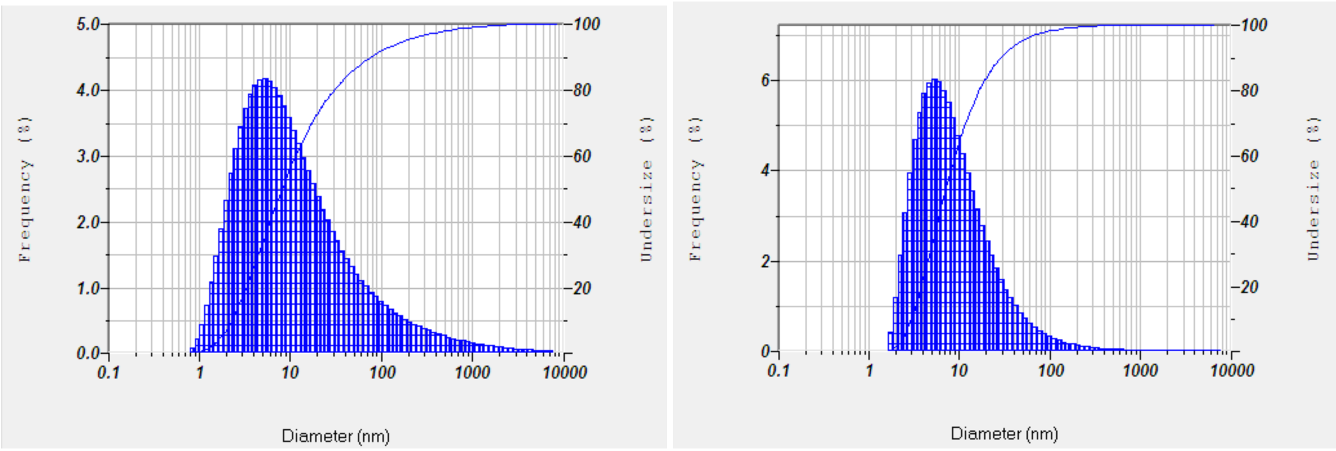

Fig 4 & 5: Particles Distribution of TiO_2 nanoparticles both Before calcination and After calcination at 400^0c

The mean value of the histogram was taken as average particle size. The average particle size was obtained as 20 nm for nanoparticles before calcination and 32 nm for after calcinated nanoparticles (400^0c). The results has proven that the nanoparticles increase with increase in temperature, as they absorb the heat energy and expanded their size. The results were supported to XRD average crystalline size [9].

Scanning Electron Microscope

The SEM images of TiO_2 nanoparticles both before and after calcination at 400^0c were shown in fig 6 & 7. Various properties like grain size, shape and surface properties like morphology can be investigated through Scanning Electronic Microscopy (SEM). The image was observed with in magnification of 20 µm. The TiO_2 nanoparticles were observed with irregular particle structure. The size was ranging from 100-120 nm for before calcinated nanoparticles and 120-140 nm for after calcinated nanoparticles. The nanoparticles were dispersed evenly on the surface with development of aggregate nanoparticles which revealed that powder particles are marginally agglomerated showing the view of spherical nanoparticles [10, 11].

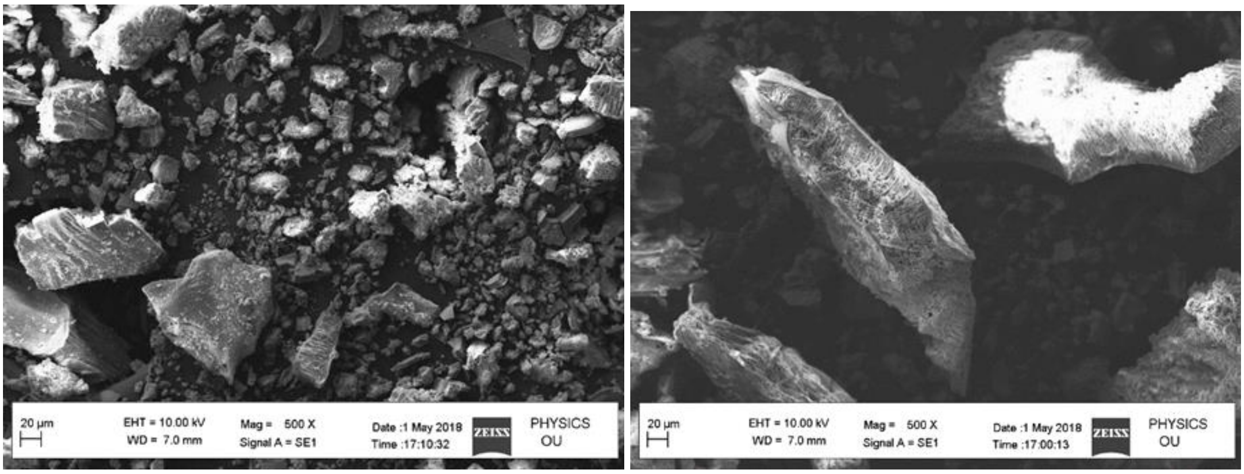

Fig 6 & 7: SEM images of TiO_2 Before and After calcinated Nanoparticles at 20µm magnification

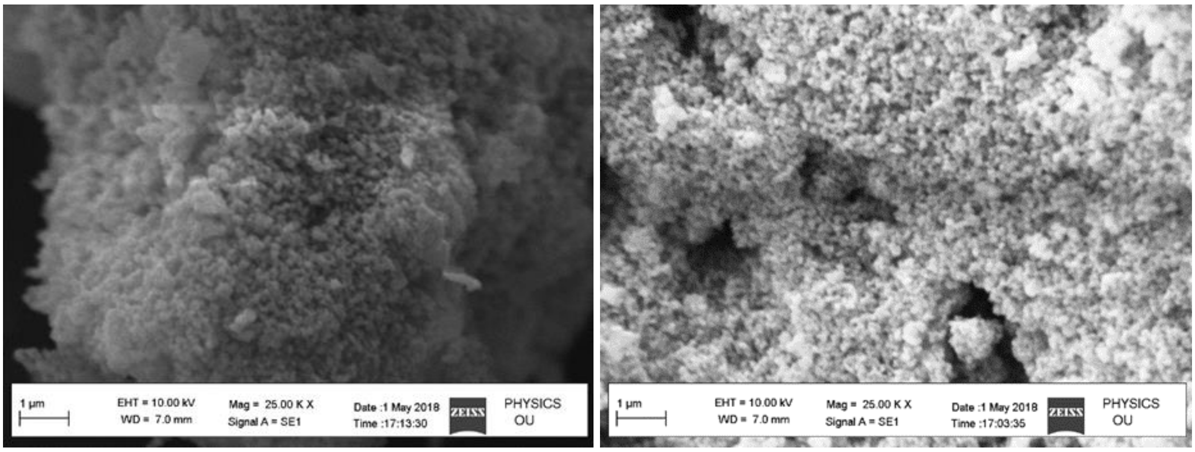

Fig 8 & 9: SEM images of TiO_2 Before and After calcinated Nanoparticles at 1µm magnification

Fourier Transform Infrared Spectroscope

The information on functional material was analysed by Fourier transform infrared spectroscopy, the FTIR spectrums of TiO2 nanoparticles before and after calcination were shown in figure 8. FTIR analysis was done from the wavenumber 500 cm-1 to 4000 cm-1 range. O-H stretching vibration bonds represented in the range of 4000 cm-1 3400 cm-1 are due to the water molecules. From 3400 cm-1 to around 1600 cm-1 endothermic peak observed due to very weak bonding vibration of water molecules.

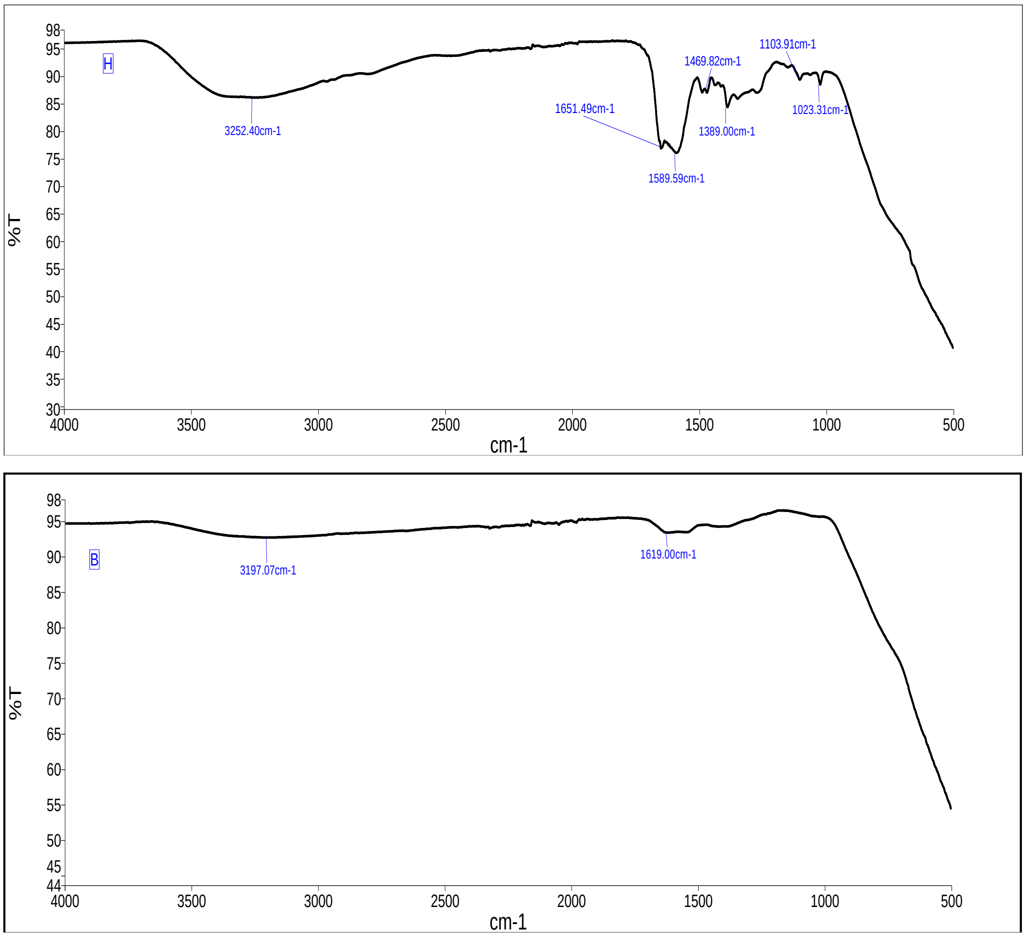

Fig 7 & 8: FTIR Spectrum of TiO_2 Nanoparticles before and after Calcinated Nano particles at 400^0c

The stretch around 3400 cm-1 to 3200 cm-1 for both the nanoparticles (before and after calcinated) represents Normal polymeric "OH" stretch indicating the presence of alcohol and hydroxy compound. The bonds showing around 1680 cm-1 to 1620 cm-1 range signifying Alkenyl C = C stretch representing Olefinic (alkene) group frequencies. Aromatic ring vibrations centered around 1600 and 1500 cm-1. Group frequencies with wave number 1150- 1000 cm-1 embodies Aliphatic fluoro compounds with C- F stretch. Stretch at 3570-3200 cm-1 exemplifies Hydroxy group, H-bonded OH stretch [12]. Stretch at 3400-3200 normal "polymeric" OH stretch. The Ti-O-Ti stretching vibration bonds formed within the range of 1000 cm-1 to 800 cm-1 [13]. All the above results confirmed that TiO_2 Nano particles formation explained by Chae *et al*

Conclusion

The use of nanoparticles was rapidly moving from laboratory to large-scale industrial production owing to the technological benefits. The present study focused on the green synthesis of TiO2 nanoparticles in two forms before and after calcinated. Synthesized TiO2 nanoparticles were characterized using XRD, FTIR, SEM and PSA. From XRD analysis the average crystalline size of the sample was calculated as 18nm for before calcinated nanoparticles and 20nm for after calcinated nanoparticles. The results of Particle size analysis revealed that the average particle size of the after calcinated nanoparticle was 20nm and before calcinated nanoparticle was 32nm. SEM analysis results revealed that, the nanoparticles were showing irregular particle structure and the size of the nanoparticles was reported to be 100-120nm for before calcinated nanoparticles and 120nm-140nm for after calcinated nanoparticles respectively. The results of FTIR disclosed the formation of TiO2 nanoparticles. Linalool, Naphthalene, Dodecane, Estragole, Tetradecane, are the biological compounds which were found to act as insect repellent compounds present in basil leaves revealed through GC-MS analysis as explained by Bagavathi *et al* [9]. It was also demonstrated that Alkaloids, Saponnins, Steroids, tannins, and terpenoids along with flavonoids and cardiac glycerides are the phytochemicals present in the leaves of basil plant that help in repelling insects [15]. TiO2 Nanoparticles prepared by green synthesis method using basil leaf extract can be used in finishing textiles for mosquito repellent, antibacterial and antifungal finishes as this green method helps in retaining the bioactive compounds available even in nanoforms.

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