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Structural and Morphological Studies of TiO_2 Nanorods synthesized by sonochemical route

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ABSTRACT: Preparation technique of TiO_2 nanoparticles was carried out by Sonochemical method which uses ultrasound for chemical reaction. The ultrasound intensity plays two different roles. The dispersion and production of nanoparticles with broken chemical bonds. Titanium tetraisopropoxide, ethanol and sodium hydroxide are used as precursor materials. Sonochemical process at different amplitudes were carried out at room temperature. Further formed TiO_2 Nanoparticles were characterized through X-Ray diffraction (XRD), Particle Size Analyzer (PSA), Transmission Electron Microscopy (TEM).

Keywords: *Sonochemical method; Titanium dioxide; X-Ray diffraction; Particle size analyzer; TEM;*

1. INTRODUCTION

Titanium dioxide (TiO_2) is one of the naturally occurring oxide and it can be formed naturally by the oxidation of titanium. TiO_2 is available in different phases like rutile, anatase and brookite [1-2]. Rutile is more stable when compared with anatase and brookite. TiO_2 is used as pigment , food coloring etc. The photocatalytic properties of rutile and anatase can be used to purify water, air [3-4]. Each phase of TiO_2 has different properties. The photocatalytic properties of TiO_2 depends on the crystal structure, size of particles (which influence the surface area) etc. So it is important to control the crystal structure and size of particles while preparing TiO_2 [5-6]. The most common method to produce TiO_2 is sol-gel method but the produced precipitates are amorphous in nature [7-8]. So it requires further processes like heat treatment to get crystallization. But heat treatment leads to particle agglomeration and grain growth in the sample [9].

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Competing interests
The authors have declared that no competing interests exist.

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Sonochemical method is a non conventional method which uses ultrasound for chemical reaction. Ultrasonic cavitation is related with formation, growth, and implosive collapse of bubbles [10]. It produces extreme heat, pressure and cooling rate which provides the condition for chemical reaction. Suitable precursor and experimental conditions can produce large amounts (with high yield when compared to conventional methods) of pure anatase phase nanoparticles [11]. Additionally, with ultrasonic radiation assisted synthesis the time span involved is only 30-60 min. This method allows the preparation of large amounts of anatase phase in a very short time period and also successive reactions can be performed simultaneously [12]. Conventional methods for synthesis takes more time to produce nanoparticles with less yield. Sonochemical method is a new technique and gives high yield with less reaction time when compared to conventional methods. The present investigation is concentrated on study of suitable conditions for large scale synthesis of polymorphic materials nanostructures at low temperatures by the choice of a suitable precursor using sonochemistry. TiO_2 sample was obtained from synthesis of anatase phase nanoparticles. To get variable anatase-rutile ratio, the prepared sample was subject to thermal treatment. To understand the nature and relationship of the nanostructures synthesized as well as to characterize the prepared TiO_2 sample we used spectroscopy (XRD) and electron microscopy (TEM).

2. MATERIALS AND METHODS

Sonication is the act of applying sound energy to agitate particles in a sample, for various purposes. Sonication can be used for the dispersion of nanoparticles and the production of nanoparticles. Sonication is commonly used in nanotechnology for evenly dispersing nanoparticles in liquids. This energy even can change the chemical route of synthesized particles such as Tetra isopropoxide titanate, Ethanol, Sodium hydroxide, De-ionized water, Probe Sonicator Model: q- sonicator q-55. (Frequency 20khz, 0-100 amplitude). 1M of Sodium hydroxide pellets were dissolved in the 50 ml de-ionized water, 0.25M of Tetra isopropoxide Titanate solution dissolved in 50ml Ethanol. An aqueous solution of $C_{12}H_{28}O_4Ti$ solution was added drop wise to the NaOH solution for 30 mins. After adding drop wise the sonochemical reaction was continued for 60 mins at 50 amplitude. During the reaction the colour of the solution will change from colorless to white-lactic. The change in the color of the solution is due to the formation of TiO_2 nano particles. Overall reaction temp is found to be 50°C. Finally the particles collected were filtered and washed carefully with carbinol and the double distilled water. The prepared samples were dried at room temperature for 48 hours.

Fig.1: 1(a) NaOH solution, 1(b) drop wise addition of Tetra iso propoxide Titanate 1(c) Final TiO_2 product Synthesized by Sonochemical method.

3. RESULTS AND DISCUSSION

3.1 XRD Analysis

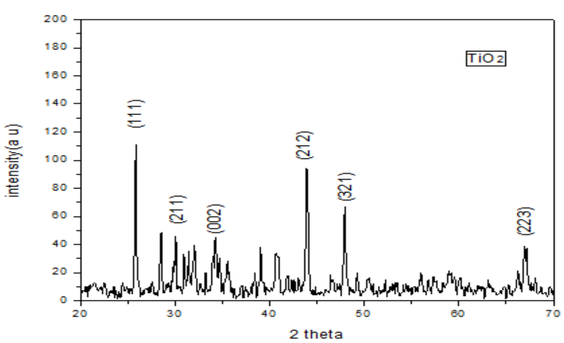

Fig. 2: XRD Pattern of TiO_2 nanoparticles

The XRD of TiO_2 nanoparticles obtained were as shown in Fig 2. The result showed that the structure is tetragonal structure Peaks obtained at 25°, 30°, 34°, 44° 48°, and 67° along with miller indices values (1 0 1), (1 1 2), (2 0 0), (1 0 5), (2 1 1) and (2 1 3) respectively. The lattice parameters were obtained a=b=0.3785 nm and c=0.9513 nm. The average crystallite size was measured by Debye-Schereer's equation as mentioned below.

$D = K.\lambda\ \beta.Cos\theta$

Where D is the average crystallite size of the particles, K- is Debye scherrer's constant (=0.94), λ- is the wavelength of the CuKα-radiation (=0.154 nm), β is the full width half maximum (FWHM) of the peak, θ is the Bragg's angle. The average crystallite size measured is about 38 nm.

3.2 Particle size analysis and Morphology

The average particle size was obtained by Particle Size Analyzer. The material was dispersed in distilled water using ultra-sonicator. Figure 3 represents the histograms of the dispersed nanoparticles. The average particle size was obtained as 50.2 nm. These results were supporting to XRD average crystallite size.

Calculation Results

Peak No.	S.P.Area Ratio	Mean	S. D.	Mode
1	1.00	50.2 nm	47.1 nm	22.9 nm
2	---	--- nm	--- nm	--- nm
3	---	--- nm	--- nm	--- nm
Total	1.00	50.2 nm	47.1 nm	22.9 nm

Histogram Operations

% Cumulative (2) : 10.0 (%) - 15.6 (nm)
% Cumulative (6) : 50.0 (%) - 32.7 (nm)
% Cumulative (10) : 90.0 (%) - 109.1 (nm)

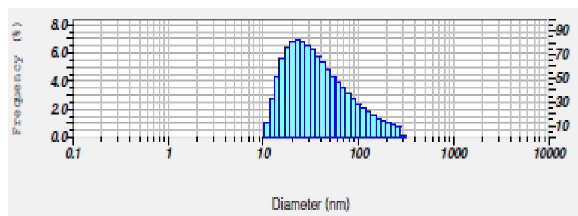

Fig. 3: Particle distribution of TiO_2 nanoparticles

The grain size, shape and surface properties like morphology were investigated by the Field Emission Scanning Electron Microscope shown in figure 4. This image was observed with in the magnification of 200nm.

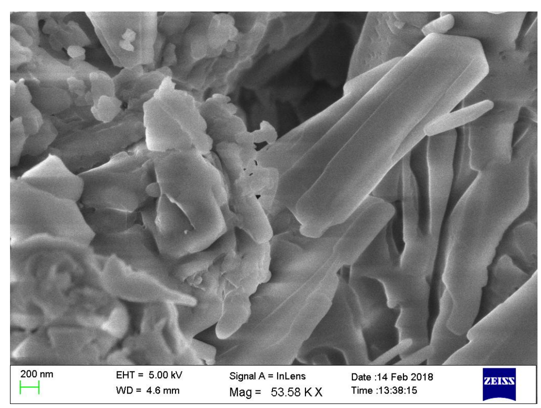

Fig. 4: FESEM image of TiO_2 nanoparticle at 700°C at 200nm

The TiO_2 nanoparticles were showing irregular particles structure. The size was ranging from 80 nm to 100 nm [13]. Figure 5 shows the TEM images of TiO_2 nanoparticle tubes were observed from TEM images at 700° degree temperature, which mean that the surface to volume ratio of the nanoparticle has increased. The average particle size from TEM is 98nm.

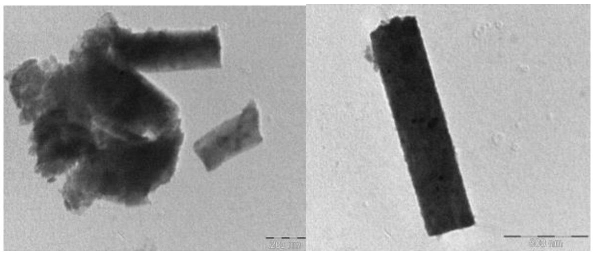

Fig.5: TEM image of TiO_2 nanoparticle at 700°C at 200nm and 500nm

3.3 Fourier Transform Infrared Spectroscopy (FTIR) analysis

FTIR (Make: SHIMIDZU, Model: IR-AFFINITY-1) spectrum was used to calculate the various functional groups present in Titanium dioxide nanoparticles. TiO_2 Nanoparticles FTIR spectra recorded from wavelength range 500 to 4000 cm^{-1} shown in figure 6.

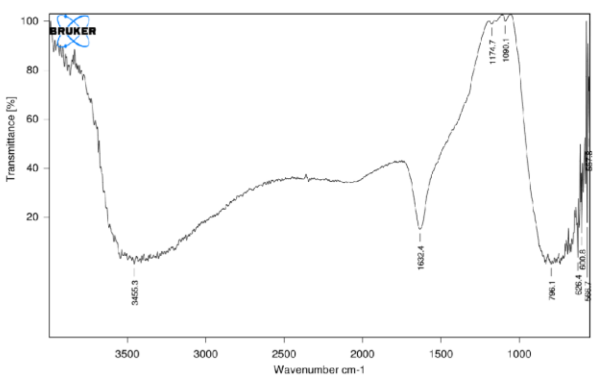

Fig.6: FTIR of of TiO_2 nanoparticle at 700°C

Ti-O bonding is confirmed by broad absorption frequency at 626.1 cm^{-1}. similarly O-Ti-O stretching is observed at 796 cm^{-1}, C-C stretching at 1090.1 and 1174.7 cm^{-1}, C=C stretching at 1632.4 cm^{-1} and 3455.3 cm^{-1} in the spectra are due to the bending vibration of the -OH group.

4. CONCLUSIONS

Nanoparticles in the size range of 50–100 nm were prepared by sonochemical synthesis. A two step reaction was used where initially the size of the precursors was controlled by sonic waves, followed by ageing of the material. This two step method not only helped to reduce the particle size of TiO_2, but also significantly reduced the crystallite size of the nanoparticles. It can be concluded that the TiO_2 are formed which were analysed by XRD , PSA and TEM analysis. FTIR showed a various functional groups in Titanium dioxide (TiO_2) nanoparticles and also determined by the transmission range. The SEM results showed that the formation of Titanium dioxide (TiO_2) nanoparticles in rod like structure.

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