International Research Journal of Engineering and Technology (IRJET) e-ISSN: 2395-0056
Volume: 02 Issue: 09 | Dec-2015 www.irjet.net p-ISSN: 2395-0072

Synthesis and Structural Investigations of Titanium Di-oxide (TiO_2) Nanoparticles by Microwave Assisted Method

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Abstract: *Nanoparticles of Titanium dioxide (TiO_2) have been synthesized via Microwave assisted method. The synthesized nanoparticles were characterized by powder X-ray diffraction (XRD), scanning electron microscope (SEM) and Fourier Transform Infrared (FT-IR). The XRD explains the interrelationship of particle size and specific surface area. SEM measurements were carried out on TiO_2 nanoparticles to uncover the morphological differences between the two systems at the nanometer and micrometer scales. The spectroscopic functional groups of TiO_2 nanoparticles were carried out by FT-IR.*

Keywords: Microwave, XRD, SEM, FT-IR

1. INTRODUCTION

Titanium dioxide (TiO_2) known as Titanium Oxide or titanium IV oxide or Titania, is naturally occurring oxide of titanium. TiO_2, has been studied extensively as photo catalyst to deal with environment pollution, water purification, wastewater treatment, hazardous waste control and air purification[1-5]. Titanium dioxide (TiO_2) is of great interest in technological application due to its morphology and crystalline phase[6]. TiO_2 has been widely studied regarding various applications utilizing the photo catalytic and transparent conductivity, which strongly depend on the crystalline structure, morphology and crystallite size [7]. TiO_2 nanoparticles have been prepared by different methods [8], chemical vapor deposition method [9], the sol-gel technique [10], sputtering[11],hydrolysis, Micro-emulsion method[12], Spray deposition method[13], aerosol-assisted chemical vapor deposition[14], Micro wave assisted method[15], thermal plasma[16], hydro thermal method[17], Solvo thermal method[18], and Flame combustion method[19]. TiO_2 has more application in various industries like aerospace, sports, medicine, paint, food and cosmetics. In this study, TiO_2 nanoparticles via Microwave assisted method using Acetic acid as a solvent. The prepared nanoparticles were characterized by Powder XRD, FTIR, and SEM.

2. EXPERIMENTAL

2,1 Materials

Titanium tetraisopropoxide (TTIP), acetic acid (Ae grade MERCK) and de-ionized water were used to prepare the nanoparticles of this study.

2.2 Synthesis

In a typical synthesis of TiO_2 nanoparticles, the amount of Titaniumtetraisopropoxide required to dissolve in 20ml of acetic acid was calculated to be 1M(sol-A) and the amount of acetic acid required to dissolve in 25ml(sol-B) . Solution of A and B was mixed in drop wise manner. The prepared solution was kept magnetic stirrer for 30 minutes at 60^0 C. Than the stirring solution was transferred to Teflon coated acutoclave and its kept in micro-oven at 90°C for 2 hrs. The sample was then filtered and it was dried at room temperature and obtain colorless powder.

2.3 Instrumentation

The as synthesized nanoparticles were characterized by powder XRD using an X-ray diffractometer (Model Bruker D-8). FTIR spectra of the as prepared nano TiO_2 were characterized by Jasco4100 spectrophotometer. SEM spectra of nano TiO_2 were recorded using Model VEGA3 TESCAN PMU Thanjavur, Tamilnadu, India.

3. RESULTS AND DISCUSSION

3.1 Structural characterization

XRD studies

The X-ray diffraction pattern of the synthesized Titania nanoparticles is shown in Fig.1 and the peak details are in Table.1. The experimental XRD pattern agrees with the JCPDS File no.21-1272 and the XRD pattern of TiO_2

nanoparticles other literature. The 2θ at peak confirms the TiO_2. Strong diffraction peaks at 25° and 47° indicating the TiO_2. The intensity of XRD peaks of the sample reflects that the formed nanoparticles are crystalline and broad diffraction peaks indicate very small size crystallite.

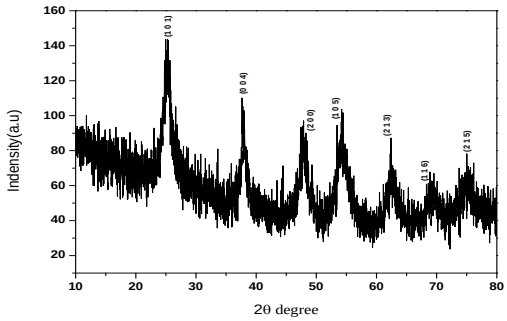

Fig.1. XRD pattern TiO2 nanoparticles

Table.1.XRD Data of TiO_2Nanopartices

2θ	FWHM (°)	βcosθ	λ (A°)	Si ze (nm)	d.spacing (A°)
25.29	0.803	0.0137	1.54	10.14	3.5181
37.87	0.535	0.0088	1.54	15.69	2.3732
47.82	0.803	0.0128	1.54	10.82	1.8998

Particle Size Calculation

From this study, considering the peak at degrees, average particle size has been estimated by using Debye-Scherer formula. Inter-planar spacing between atoms (d-spacing) is calculated using Bragg's law and enumerated in Table.1.

$$D = 0.9\lambda/\beta \cos\theta \quad (1)$$

$$2d \sin\theta = n\lambda \quad (2)$$

Where, λ is wavelength of X-Ray (0.1540nm), β is FWHM (full width at half maximum), θ is diffraction angle, d is d-spacing and D is particle diameter size and n is the order of diffraction.

Specific surface area (SSA)

The surface states will play an important role in the nanoparticles, due to their large surface to volume ratio with decrease in particle size. SSA is a material property. It is a derived scientific value that can be used to determine the type and properties of a material. It has a particular importance in case of adsorption, heterogeneous catalysis and reactions on surfaces. SSA is the Surface Area (SA) per mass. The average specific surface area of TiO_2 nanoparticles was calculated using equation (3).

$$SSA = 6000/D * \rho \quad (3)$$

Where D is the particle size in meter, SSA is the mean specific surface area of TiO_2 nanoparticles, ρ is the density of TiO_2 in kg/m^3. Table .2.shows calculation of specific surface area of the TiO_2 nanoparticles. The crystalline boundry sizes were increased to increasing in specific surface area of the TiO_2 nanoparticles. Figure.2. shows that specific surface area of TiO_2 nanoparticles.

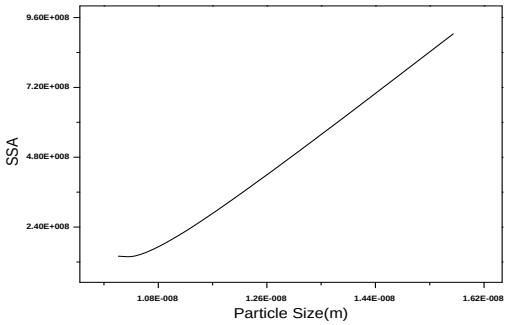

Fig.2. Specific Surface Area of TiO_2 nanoparticles

Table.2.specific surface area of TiO_2 nanoparticles

Cosθ	FWHM β (Radian)	β cosθ(deg)	Size(m)	Density (kg/m^3)	Surface area(m^2)	SSA
0.9758	0.014006	0.0137	1.01×10^{-8}	4.23×10^3	6.00×10^3	1.40×10^8
0.9459	0.009338	0.0088	1.57×10^{-8}	4.23×10^3	6.00×10^3	9.04×10^8
0.9142	0.014006	0.0128	1.08×10^{-8}	4.23×10^3	6.00×10^3	1.31×10^8
Average Specific Surface Area						1.20×10^8

3.2 FT-IR spectral analysis

The FT-TR spectra of the as-prepared TiO_2 nanoparticles is shown in fig.3. The strong speak of TiO_2 at 578cm^{-1} to 1618cm^{-1} was assigned all functional groups. The sample almost shows the same type of functional groups. The assignments for the peaks/bands of the FT-IR spectra of the sample have been given in accordance with the data reported in the literature.

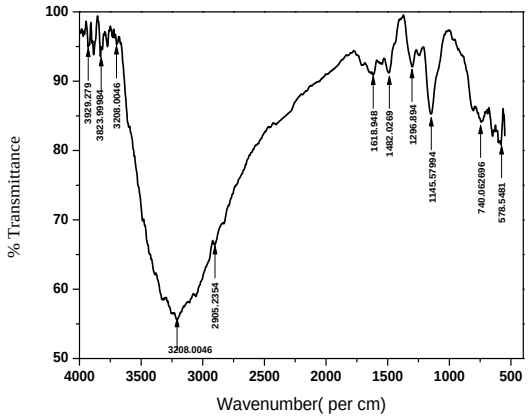

Fig.3. FT-IR spectrum of TiO_2 nanoparticles

3.3 Scanning Electron Microscope (SEM) analysis

The morphologies of the prepared samples were investigated through Scanning Electron Microscopic (SEM) images. SEM measurements were carried out on TiO_2 nanoparticles to uncover morphological differences between the two systems at the nanometer and micrometer scales. Fig 4a, and 4b, shows the SEM micrographs at different magnifications of TiO_2 nanoparticles.

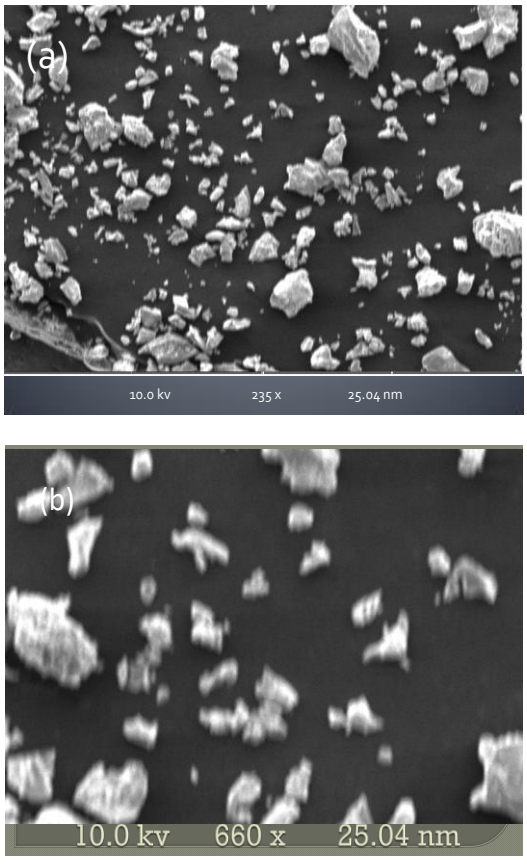

Fig.4(a)(b) SEM images of TiO_2 nanoparticles

4.CONCLUSION

TiO_2nano powders were successfully synthesized by microwave assisted method. The Miller Indices and the crystalline size of as-prepared TiO_2 nanoparticles were determined by powder XRD technique. Strong diffraction peaks at 25° and 47° indicating the TiO_2. The XRD studies were used to find specific surface area and d-spacing of TiO_2 nanoparticles. The morphological studies revealed that the particles were spherical in structure and slightly agglomerated. The phase purity and functional groups of the samples were analyzed by FT-IR studies. The strong speak of TiO_2 at 578cm^{-1} to 1618cm^{-1} was assigned all functional groups.

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