TiO_2 NANOPARTICLES: SYNTHESIZED BY SIMPLE MICROWAVE – ASSISTED METHOD USING ROOMTEMPERATURE IONIC LIQUIDS

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ABSTRACT

TiO_2 is an important material because of its special properties like chemical stability, high photo catalytic activity, high electric permittivity and large birefringence. So it is used in various applications like optical, electrical, electronic and catalytic devices. Present work focused on to synthesis TiO_2 nano particles using simple microwave-assisted method using Room Temperature ionic Liquids (RTIL) as precursor materials. Microwave-assisted method is a simple, biodegradable and rapid heating technique. Titanium isopropoxide and NaOH used as precursor materials. The synthesized TiO_2 nano particles have been characterized by X-ray Diffractometer (XRD), Dynamic Light Scattering (DLS) and Scanning Electron Microscope (SEM), Transmission Electron Microscope (TEM) for average crystallite size, average particle size, morphology and structure respectively.
Keywords: TiO_2 Nanoparticles, RTILs, XRD, SEM, TEM

Introduction:

Titanium dioxide (TiO_2) is one of the semiconducting materials which as used in various applications due to its wide range of band gap, high chemical stability, controlling oxidation strength and non-toxicity nature (Saowaluk Boonyod, 2011 et al.). Semiconductor materials are having very interesting properties, so these are used in various applications like electronic, humidity sensors, gas sensors, paints, cosmetics, health care products and solar cells (C. Bandas (Ratiu), 2011 et al., Vilas G. Pol, 2007 et al., Seigo Ito, 2007 et al.). TiO_2 having different crystalline phases like anatase phase, rutile phase and brookite phase depends on its physical property (Meilong Hu, 2012 et al.). Titanium isopropoxide and NaOH were taken as precursor materials. TiO_2 nano particles were synthesized by Microwave-assisted method. It is a simple, fast and rapid heating method compared with other synthesis methods. This method is suitable for nano particle preparation because of its lower by-products and higher homogeneity (L. Cui, 2012 et al., Lucky M. Sikhwivhilu, 2012 et al.).

Methodology:

Titanium isopropoxide (TTIP) and NaOH were used as starting materials. TiO_2 nanoparticles were successfully prepared by Microwave-assisted method. TTIP (5 ml) solution mixed with 50 ml of distilled water under vigorous stirring, 0.01M NaOH was added to the above solution up to the solution gets white colour. These obtained mixture solution is introduced in the Microwave oven, under microwave radiations for 5 minutes. Then white colour precipitate was formed. These precipitate was centrifuged using with water and ethanol, finally we obtained TiO_2 nano particles.

Present paper deals with the TiO_2 nanoparticles which are before calcination and after calcination at 600^0C.

Characterization Techniques:

The obtained TiO_2nanoparticles were characterized by Bruker D8 advanced X-ray diffractometer using Cu$K\alpha$ radiation for crystal structure and average crystallite size.The average particle size obtained from HORIBA SZ-100 Particle size analyzer. HITACHI S 3400N Scanning Electron Microscope for observe Morphology and Size. JEM 100 CXII Transmission Electron Microscope used for structure and d-spacing.

Results & Discussions:

a) X-Ray Diffraction-

XRD – pattern of TiO_2 nanoparticles was shown in figure 1. In this pattern two types of materials were mentioned: one is before calcination and another one is after calcination at 600^0C.

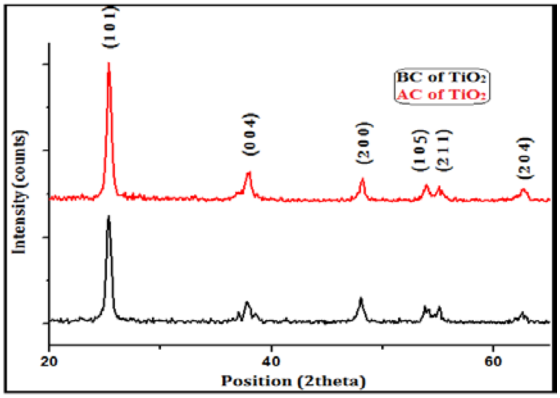

Figure 1 XRD pattern of TiO_2 nano particles for before and after calcinations

From the above XRD-pattern, before calcination temperature and after calcination temperatures peak positions were same as 25^0, 37^0, 48^0, 53^0, 55^0 and 62^0, the corresponding miller indices were (1 0 1), (0 0 4), (2 0 0), (1

0 5), (2 1 1) and (2 0 4) respectively. Sharp peaks were observed at after calcination. The TiO_2 nanoparticles crystal structure is hexagonal and the lattice parameters were a=b=0.3785 nm and c=0.9513 nm, this data is good agreement with JCPDS card number 21-1272.

The average crystallite size was measured by Debye-Scherer's equation $D = K\lambda/\beta \cos\theta$, Where D – is the average crystallite size of the particle, λ – is the wavelength of the radiation, β – is the full width half maximum (FWHM) of the peak, θ – is the Bragg's angle.

The obtained crystallite sizes were before calcination 18 nm and after calcination 21 nm (PradeepanPeriyat, 2010, et al.).

b) Dynamic Light Scattering-

As prepared TiO_2 nanoparticles were ultra-sonicated in the distilled water. Characterize these particles using particle size analyser. It works with the basic phenomenon of dynamic light scattering. Figure 2 Shows that particles distribution in the particle size analyser.

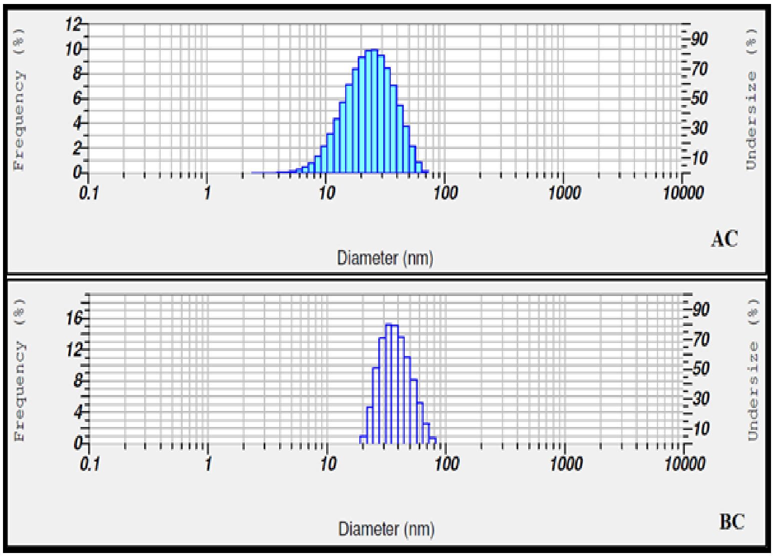

Figure 2 Particles distribution in Particle Size analyser for before and after calcinations

The average particle sizes were obtained 20 nm and 23 nm for before calcination and after calcination respectively.

c) Scanning Electron Microscopy-

SEM images of TiO_2 nanoparticles were shown in figure 3. The morphology of the nanoparticles was observed by SEM. From the images, before calcination of TiO_2 nanoparticles were shown agglomerated, whereas after calcination of TiO_2 nanoparticles were shown spherically granules structure.

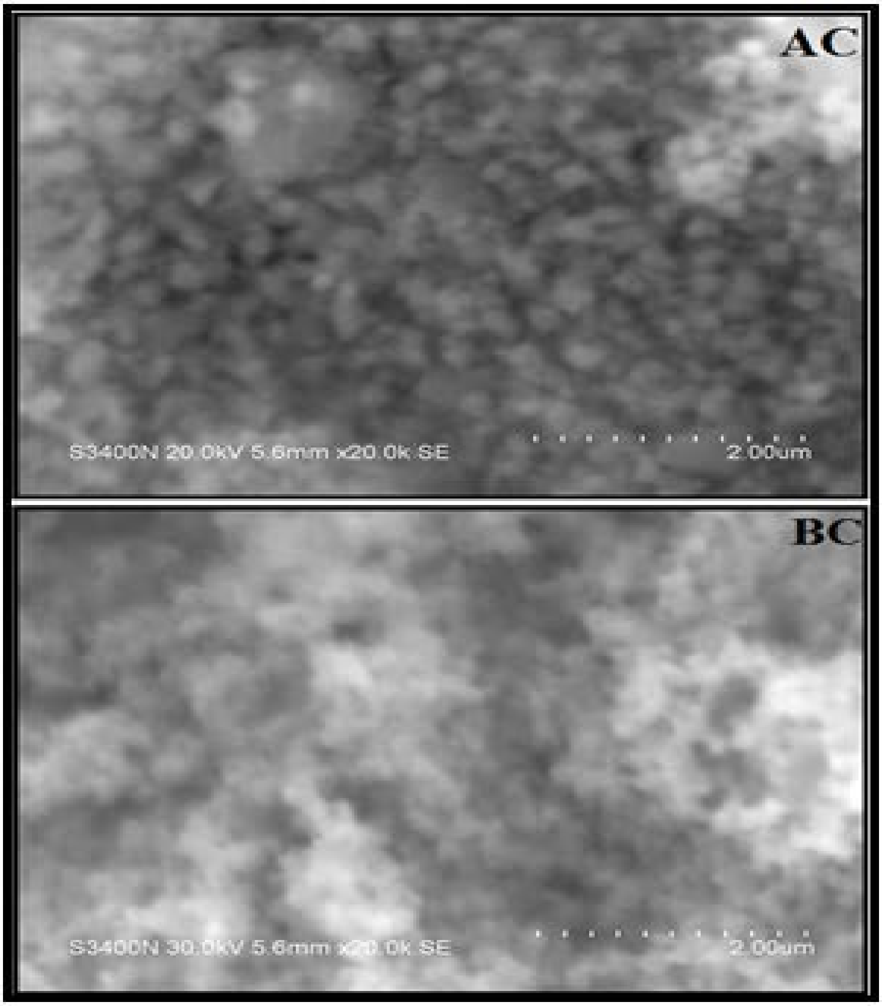

Figure 3 SEM images of TiO_2 nanoparticles for before and after calcinations

The size range of the particles was 60-80 nm and 100-120 nm for before calcination and after calcination respectively (Hanbin Lee, 2012 et al.).

d) Transmission Electron Microscope-

TEM images of TiO_2 nanoparticles were shown in figure 4. The structure and surface morphology was observed by TEM. The images show before calcination and after calcination of TiO_2 nanoparticles. It infers that, the structure is hexagonal structure and after calcination the agglomerated and spherical granules like structure was formed.

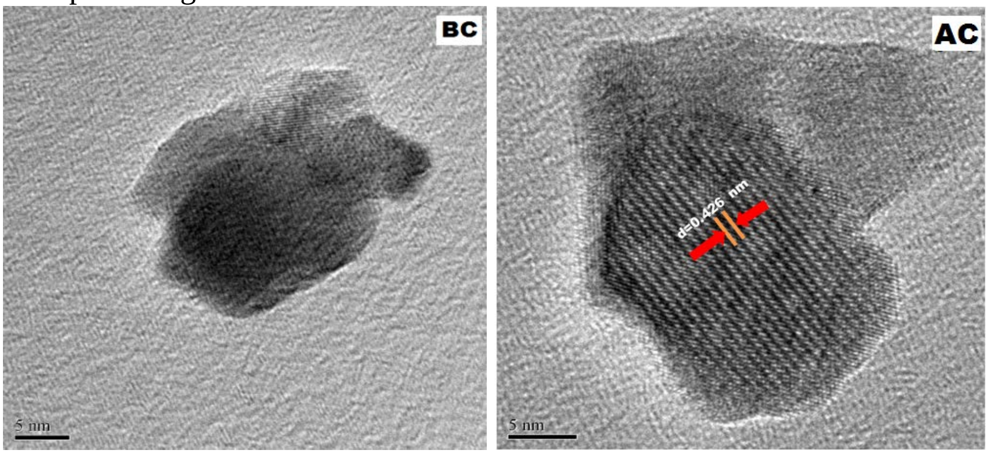

Figure 4 TEM images of TiO_2 nanoparticles for before and after calcinations

The d-spacing value was possible to measure the after calcination of TiO_2 nanoparticles only, because before calcination image was showing uneven distribution of planes. The d-spacing value of after calcination was 0.426 nm which is nearly equals to XRD d-spacing value.

Conclusions:

The TiO_2 nanoparticles had been synthesized by simple Microwave – assisted method using titaniumisopropoxide and NaOH as precursor materials. The XRD pattern shows that the structure of TiO_2 nanoparticles were hexagonal, the average particle sizes ware18 nm and 21 nm for before calcination and after calcination respectively. The sharpness of the peaks was increased at after calcination. The average particle size was obtained from particle size analyser was 20 nm and 23 nm for before calcination and after calcination. These sizes are nearly equals to XRD average crystallite sizes. After calcination SEM image shows that agglomerated spherical granules like structure, whereas before calcination it was agglomerated. The size ranges from SEM images were 60-80 nm and 100-120 nm for before and after calcination. From the TEM images the structure and d-spacing values supports the XRD data. The all above structural properties were concluded that the synthesized TiO_2 material was in the nano range.

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