



Synthesis and Characterization of SnO₂ Nano Particles for Carbon Absorbing Applications

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ABSTRACT

The SnO₂ nanoparticles was synthesized by Microwave assisted technique. From the powder XRD the particle size was calculated using Scherrer formula, it from 57 nm. The absorption spectrum was recorded from 1100 nm to 190 nm, and the optical band gap was calculated using Touc plot. The band gap value 3.16 eV. The functional groups were confirmed by FTIR spectrum. The FE-SEM analysis reveals the morphology of SnO₂ nano particles. The synthesized SnO₂ nano particles were used as a catalyst to reduce carbon from automobiles fume. The detailed study on carbon emission was reported.

Key words: Nano materials, SnO₂, SEM, Carbon Emission.

INTRODUCTION

In recent years the researcher's interests have been involved in on low dimensional inorganic semiconducting metal oxide nanomaterials owing to their unique optical, chemical, and electrical properties. As these properties were deeply influenced by the size and morphology of the nanostructures, the preparation of nano sized crystallites with different morphologies provided an opportunity to explore the possible changes in their physical and chemical properties with size and shape^{1,2,3}. Now days the researchers were involved to prepare the one-dimensional nonmaterials to

increasing their numerous applications⁴. The tin oxide (SnO₂) is the one of the interesting candidates due to its potential applications like gas sensors^{5,6}, solarcells⁷, lithium batteries⁸, and in transparent conductive electrodes⁹. In recent days, nanowire and nanotube based materials have been well demonstrated as building blocks for nano circuits, nano systems and nano-optoelectronics. For the same reasons, researchers focuses their interest on one-dimensional nano structured SnO₂ materials¹⁰⁻¹³.

In quantum size effect, when the particle size of a semiconductor is compared to the Bohr

radius¹⁴, the ratio of surface atoms to those with the interior enhances the surface properties of the materials. Due to these effects, band gap of the semiconductor nano particles has been modified, which makes a lot of changes in their electronic properties when compared to the bulk. Thus, there is an increase in the band gap i.e. the visible spectra show a blue shift, which is assigned to quantum size effect¹⁵. This stimulated a great interest in both basic and applied research in semiconductor oxide nano materials. Many methods have been applied by researchers to synthesize the SnO₂ nano materials¹⁶ like sol-gel route, thermal decomposition¹⁷, co-precipitation¹⁸, microwave-assisted solution¹⁹, gas phase condensation²⁰ and laser ablation²¹ etc.

In this paper, we have reported the SnO₂ nanoparticles as catalyst to reduce carbon from automobile engine. The synthesized SnO₂ material was characterized using various techniques like XRD, UV, FTIR, FE-SEM and EDX.

MATERIALS AND METHODS

The 0.1M solution of Tin (II) chloride was prepared using deionized water as solvent. The pH solution was maintained by liquid ammonia. The resulting precipitate was washed using deionized water, until no chlorine ions are detected (silver nitrate test). Further to remove NH₄⁺ ions the precipitate was washed using ethanol. Then it was irradiated with house hold microwave oven for 10 min. The radiation frequency was 2.45 GHz and its power up to 1KW. The final product gives SnO₂ nanoparticles.

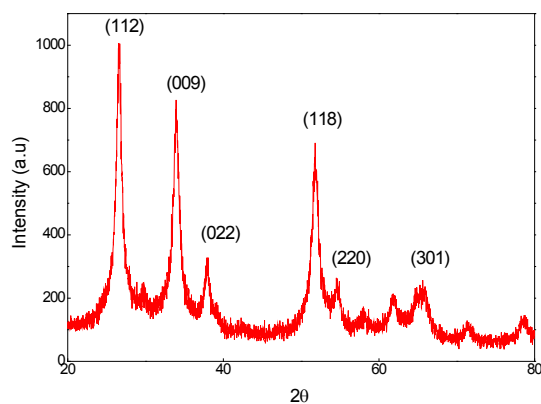


Fig. 1: XRD pattern SnO₂ nano particles

Characterization

XRD analysis

The fig. 1 shows powder XRD pattern of SnO₂ nanoparticles shown in fig.1. The powder sample was scanned in steps of 0.02 ° for time interval of 10 s over a 2θ range of 20-80 in Xpert high score. The observed pattern has prominent peaks at (112), (009), (022), (118), (220) and (301), are well coincide with JCPDS file no 41-1445 and confirms the formation of SnO₂ nano particles. The SnO₂ shows tetragonal structure. The sharpness of peaks shows that SnO₂ nanoparticles are highly crystalline. The particle size of the as-prepared powder was calculated using Debye Scherrer formula,^{2,7,32}

$$D = k\lambda / \beta \cos \theta$$

where D is the particle size, λ is the X-ray wavelength, β is the full width at half maximum of the diffraction peak, and θ is the Bragg diffraction angle of corresponding peaks. The average particle size was found to be 56 nm.

UV-vis absorption spectrum

The fig.2 shows that UV-visible absorption spectrum of SnO₂ nano particles. The UV spectrum carried from 190 -1100 nm. UV-visible spectroscopy provides useful information about the optical band gap of the semiconductors. For the semiconductor nanoparticles, the quantum confinement effect is expected and the absorption edge will be shifted to a higher energy when the particle size decreases^{22,23}. In the absorption spectrum shows the cut of wavelength of SnO₂ nano particles is 210 nm.

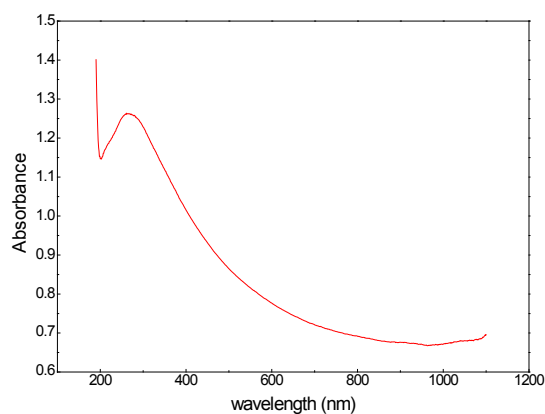


Fig. 2: UV absorption spectrum of SnO₂ nano particles

Calculation of optical band gap

The fig.2 shows the optical bandgap of various pH samples were determined from the absorption spectra. The relation between the optical absorption coefficient $h\nu$ and the photon energy was given by Mott and Davis²⁴ as

$$\alpha(h\nu) = B(h\nu - E_g)^n / h\nu \quad \dots(1)$$

where B is a constant dependent on the transition probability, h is Planck's constant, ν is the frequency of the radiation, and E_g is the optical energy gap. The type of transition responsible for the absorption depends on the value of n an index that can take any of the values 1/2, 3/2, 2 or 3 for direct-allowed, direct-forbidden, indirect-allowed, and indirect forbidden transitions, respectively. In the present case, $n=1/2$, which means an allowed direct transition. The optical absorption coefficient was calculated from the absorbance a using the following equation²⁵

$$\alpha(\nu) = 2.303 A/d \quad \dots(2)$$

Where d is the thickness of the sample in centimetre and A is calculated using the formula

$$A = \ln(I_0/I_t)$$

Where I_0 and I_t are the intensities of incident and transmitted light, respectively.

For the determination of the direct optical band gap E_g was plotted as function of photon

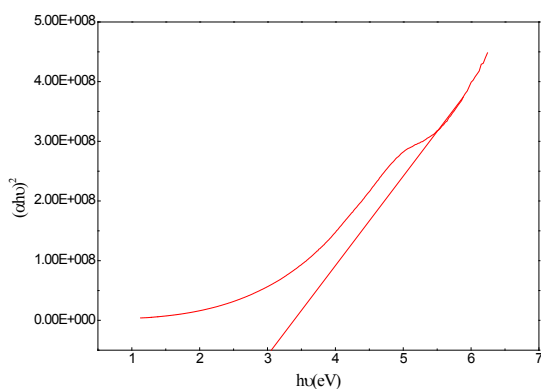


Fig. 3: Optical band gap of SnO₂ Nano particles

energy $h\nu$. When the dimensions of nanoparticles approach the exciton Bohr radius, a blue shift in energy is observed due to the quantum confinement. The band gap of SnO₂ was found to be 3.16 eV. The effective mass model [26] is commonly used to study the size dependence of optical properties of quantum systems. The resultant values of E_g for freshly prepared SnO₂ nanoparticle is found to be about 3.16 eV with splitting of energy^{2,27}.

FT-IR spectrum

The FT-IR spectrum was recorded for SnO₂ nano particles after microwave treatment (fig. 4). The FTIR spectrum is recorded using Avatar 330 FT-IR thermo nicolet spectrometer in the wavelength range 400–4000 cm⁻¹ by KBr pellet technique. The absorption band at 539 cm⁻¹ was ascribed to the terminal oxygen vibration ($\nu_{\text{Sn-OH}}$)²⁹ for the dried precipitate. It was obvious that the hydroxyl group changed into oxide group during microwave radiation. After heating the product at higher sintering temperatures (1KW) a new broad band centred at 620 cm⁻¹ appeared which was characteristic of oxide-bridge functional group ($\nu_{\text{O-SnO}}$)³⁰. It was obvious that at this temperature the SnO₂ had changed into SnO. The peaks at 1631 cm⁻¹ were assigned to NH deformation of ammonia and NH stretching vibration respectively. The absorption band at 3323 cm⁻¹ was mainly due to ν_{OH} stretching vibration of surface hydroxyl group or adsorbed water. For low pH value OH groups were still present at the surface and at the interior of the SnO particles. The remaining low signals attributed to some OH groups at the surface

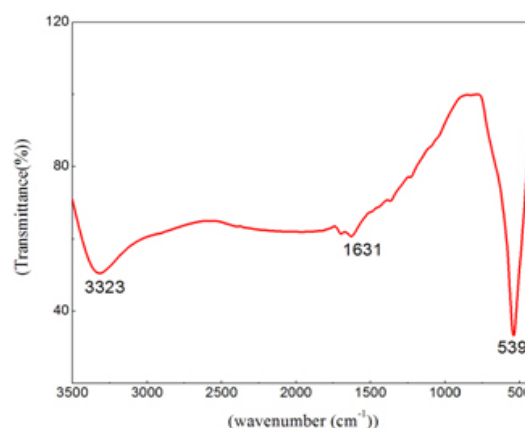


Fig. 4: FT-IR spectrum of SnO₂ Nano particles

were probably due to the re-absorption of water from the ambient atmosphere³¹.

Scanning Electron Microscopy (SEM)

The morphology and chemical composition of the microwave synthesized tin oxide nanostructures were analyzed by SEM (fig.5). The micrograph of SnO₂ sample shows the typical morphology of the SnO₂ powders synthesized,

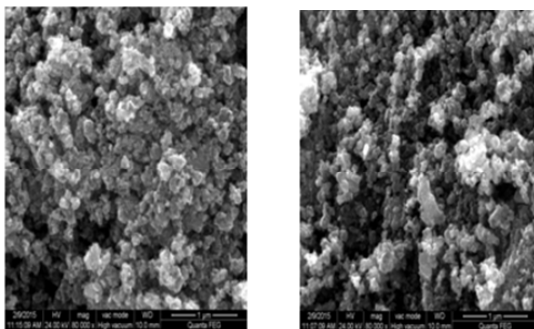


Fig. 5: SEM image of SnO₂ Nano particles

characterized by the presence of particles with an average size of about 1 μm or less. In this study SnO₂ nanoparticles found to be spherical.

EDX spectrum

The fig 6 shows the EDX spectrum of SnO₂ nano particles. The spectrum is confirmed the presence of elemental (stannum) tin oxide and oxygen. There was no other element not observed in EDX spectra. It shows the synthesized SnO₂ is in high pure form.

Measurements of CO₂ level

The synthesized SnO₂ nanoparticles were used as catalyst to reduce the carbon content of automobile fume. For this measurement we have chosen the pulsar bike (2002 model). The emission test was carried out by conventional emission test instrument, it records the emission for 0-60 seconds and it is shown in fig. 7a. Then the synthesized catalyst was spread on the wool rolled and put in the cylindrical tube. The tube fixed in the outer core

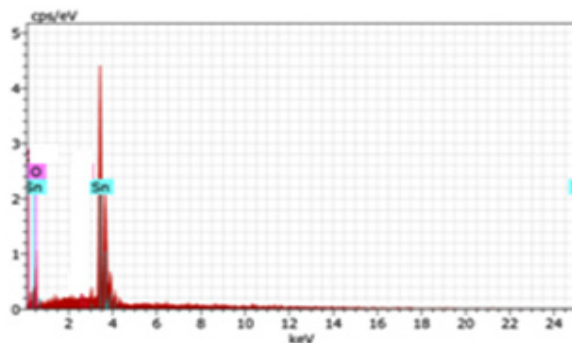


Fig. 6: EDX image of SnO₂ Nano particles

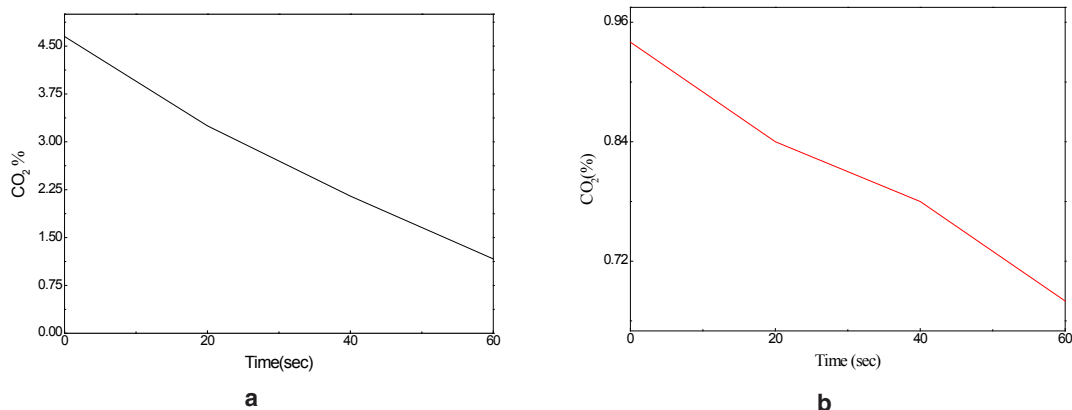


Fig. 7: (a) without catalyst (b) with catalyst

of the silencer. The bike get started the smoke was measured the CO₂ gas during the interval of the 0- 60 seconds and the results were shown in fig. 7 b. The amount of emitted CO₂ is found reduced while using SnO₂ catalyst. In this way the SnO₂ catalyst used as a converter used redox reactor to reduce the pollutants of the two wheelers.

CONCLUSION

We have successfully synthesized SnO₂ nano particles by microwave assisted method. The powder X-ray diffraction shows the good crystalline nature and particle size calculated using Debye Scherrer

formula. The calculated particle size is 57 nm. The band gap of SnO₂ nano particles is measured from Tauc relation. Morphology was investigated carried from SEM. The EDX spectrum gives atomic percentage level of SnO₂ nano particles. The SnO₂ is identified as suitable material to reduce the Carbon from automobiles fume.

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