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Synthesis and Charaterization of SnO_2 Nanopowder

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Abstract— Nanocrystalline tin oxide (SnO_2) powders with the particle size distribution in the range of 50-100nm were synthesizes by hydrothermal method. The tin oxide nanopowder in higher temperature at 700˚C and time 2hrs. The X-ray diffraction pattern was used to analyze the formations of phase and crystal structure. The morphology and the microstructure of the nanosized tin oxide powders were studied using scanning electron microscope. The XRD measurement was used to investigate the structural changes observed in the formation of tin oxide nanopowders. The photoluminescence measurement showed that the material exhibited maximum emissions range.

Keywords*:* SnO_2, PL, UV-Vis.

I. INTRODUCTION

The wide variety of electronic and chemical properties of metal oxides makes them exciting materials. Oxides span a wide range of electrical properties from wide band gap insulators to metallic and super conducting. Tin oxide is also known as Stannic Oxide. Tin oxide (SnO_2) nanoparticles are available in the form of high surface area diamagnetic oxide nanostructures. Tin belongs to Block P, Period 5 and oxygen belongs to the Block P, Period 2 in the periodic table. It crystallizes with the rutile structure, where in the tin atoms are six coordinate and the oxygen atoms three coordinate. SnO_2 is usually regarded as an oxygen-deficient N-type semiconductor. It is an important material due to its properties such as.igh transparency, physical and chemical interaction, strong thermal stability, low operating temperature[1], high melting point 1630°C, boiling point 1800-1900°C.

The electrical conductivity and luminescence properties of SnO_2 are mainly decided by the oxygen vacancy present in SnO_2 lattice. Electrical and optical properties of SnO_2 nano particles alter due to their high surface to volume ratio. It is an N-type semiconductor has a high band gap energy 3.6-3.8eV [2].

Tin oxide (SnO_2) particles are relatively easy to disperse and compared to other particles such as aluminum oxide (Al_2O_3) or titanium dioxide (TiO_2), SnO_2 particles have almost no surface water & insoluble. SnO_2 have High-transparency semiconductors used in potential applications in flat panel displays. Particularly, bulk SnO2 has wide band-gap of 3.6eV and thin films can have high conductivity depending on oxygen vacancy. In addition to that tin oxide has optical and electrical property have been used in electrochemical and catalytic application.

Some of the application of tin oxide nanoparticles Magnetic properties of tinoxide nanoparticles is used in data storage and magnetic resonance imaging. As catalyst, energy saving coating and anti-static coatings. Tin oxide used as an electrode and anti-reflection coating in solar cells, making of liquid crystal displays.

Tin oxide has long been used as an opacifier and as a white colorant in ceramic glazes. Its use has been particularly common in glazes for earthenware, sanitary ware and wall tiles; see the articles tin-glazing and Tin-glazed pottery. Tin oxide remains in suspension in vitreous matrix of the fired glazes and with its high refractive index being sufficiently different from the matrix, light is scattered, and hence increases the opacity of the glaze. The degree of dissolution increases with the firing temperature, and hence the extent of opacity diminishes. Although dependent on the other constituents the solubility of tin oxide in glaze melts is generally low. Its solubility is increased by Na_2O, K_2O and B_2O_3, and reduced by CaO, BaO, ZnO, Al_2O_3, and to a limited extent PbO.

SnO_2 has been used as pigment in the manufacture of glasses, enamels and ceramic glazes. Pure SnO_2 gives a milky white colour; other colours are achieved when mixed with other metallic oxides e.g. V_2O_5 yellow; Cr_2O_3 pink; and Sb_2O_5 grey blue

Tin oxide can be prepared on a large scale at low cost by simple solution-based methods, such as sol-gel[3], co precipitation[4], microwave technique [5], solvothermal [6], hydrothermal [7], sonochemical [8] and mehcanochemical [9]. Hydrothermal techniques is a promising alternative synthetic method because of the low process temperature and very easy to control the particle size. The hydrothermal process have several advantage over other growth processes such as use of simple equipment, catalyst-free growth, low cost, large area uniform production, environmental friendliness and less hazardous. The low reaction temperatures make this method an attractive one for microelectronics and plastic electronics. This method has also been successfully employed to prepare nanoscale tin oxide and other materials. With this method, we can prepare SnO_2 nanoparticles in large amount with low cost precursors.

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II. Experimental Procedure

The tin oxide was prepared using commercially available chemical tin chloride as starting materials. 2g of $SnCl_2.2H_2O$ and was dissolved in 60ml distilled water .To this solution 4g of ammonium oxalate powder was added with slow stirring. Stirring was continued till this dissolution of ammonium oxalate takes place. The ammonia solution was added drop wise to raise the pH of the solution to about 10. A precipitate form and then settle precipitate was collected by filtration, washed with distilled water several times. The precipitate is then transferred on a watch glass kept in an air oven at 105˚C for a period of 1hour. The dried powder heated at 700˚C at 3hrs. To obtained tin oxide nanoparticles.

The synthesized sample was characterized by X-ray Powder diffraction (XRD) with a wavelength λ =1.5418 Å at 2θvalues between 20° and 80°. The surface morphology was studied using a Scanning Electron Microscope ((PHILIPS make, model-XL30). The surface roughness and morphology was studies using Atomic Force Microscope (Scanning Probe Microscope, PHILIPS make, model-XL30) in non-contact mode. The FTIR Spectrum was recorded in room temperature at 500-3500cm^{-1}(Bruker make, Alpha-E model, Germany). The photoluminescence was measured from 350 -400nm using Spectrofluorophotometer (Model No.: R7-5301 PC, Shimadzu, Japan)

III. Result and Discussion

A. Structure and Morphology

The X-ray diffraction pattern recorded for the milled Tin oxide nanopowder sintered at 700˚C is shown in Fig. 1. The XRD peaks were indexed using the JCPDS files [JCPDS card No.:71-0652] [10]. The tin oxide nanopowders crystallizes into orthorhombic lattice with lattice constants, a = 5.427 A ° , b = 32.736 A ° and c = 5.396 A ° . The lattice constants of tin oxide reported in the literature are a = 5.448 A °, b = 32.81 A ° and c = 5.41 A ° [11]. The good agreement of the obtained lattice constants with the standard values indicates that a single phase of SnO_2 with orthorhombic structure was obtained due to calcinations at 700˚C. The peaks centered at 2θ of 26.6827, 33.9520, 37.8965, 51.7696, 54.9162, 57.9145, 62.0194, 64.7448, 66.0384 and 71.3987 which are strongest, can correspond to orthorhombic structure (h k l) planes of (110), (101), (200), (211), (220), (002), (310), (301), (202), (100) of SnO_2.

The SEM images recorded for the tin oxide nanopowders prepared calcinations at 700˚C are shown in Fig.2 shows agglomeration of powder. Due to indistinct images, it is very difficult to determine the individual particle size. Therefore, the particle size was calculated using Scherrer's formula and it was found to be 21 nm. The morphology of the calcined SnO_2 powder is plate-like grain structure. The AFM image recorded for the calcined tin oxide nanopowders calcined at 700˚C is shown in Fig.3. The AFM image shows that there is a grain growth in the calcined tin oxide nanopowder. The average grain size obtained from the AFM image of the calcined tin oxide nanopowders varies from 30 nm to 40 nm.

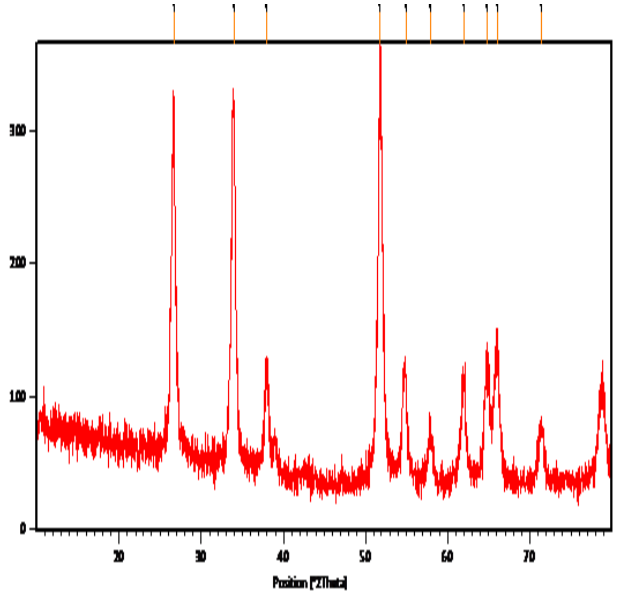

Fig1. XRD Pattern of SnO_2 nanopowder

B. FTIR Spectra

The FTIR spectra recorded at room temperature in the range 500-3500cm^{-1} the nanopowders calcined at 700˚C are shown in Fig 4. The sharp peak at centered at 603 cm^{-1} is observed. The broad band between 800 and 500 cm^{-1} was due to the vibrations of Sn-O [18, 19]. Remaining peaks at 1000 and 3000cm-1 are due to absorption of water.

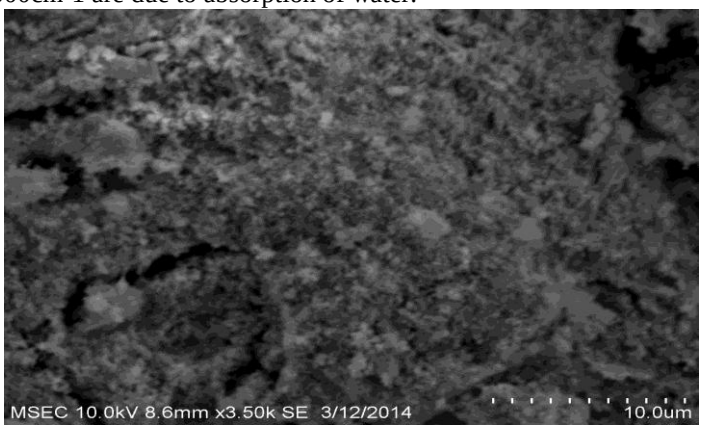

Fig2. SEM image of SnO_2

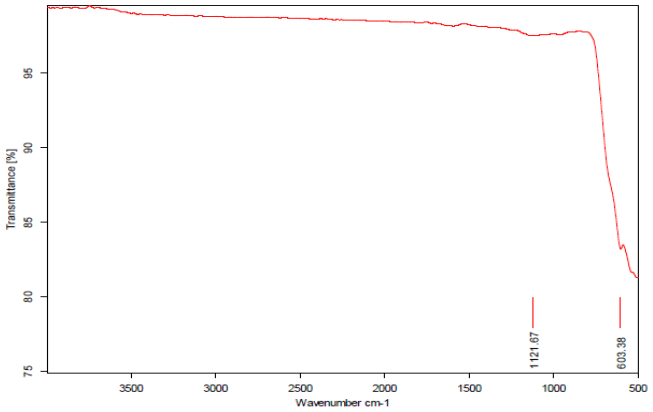

Fig3. FTIR Spectra of SnO_2

C. Photoluminescence

The photoluminescence spectrum recorded at room temperature for tin oxide nanopowders calcined at 700˚C is shown in Fig. 7. The excitation energy of the photoluminescence spectrum is found to be 2.95 eV (364 nm).

The maximum emission of photoluminescence occurs at 364 nm in a visible emission band around bluegreen colour. In the case of nanocrystals, the origin of photoluminescence is due to the dominant role of the surface states, since there are a large number of unsaturated atoms existing in the surface regions of nanometer crystallites and forming localized levels within the forbidden gap of the materials. The quantum size effects and the oxygen vacancies could play a dominant role in the luminescence processes.

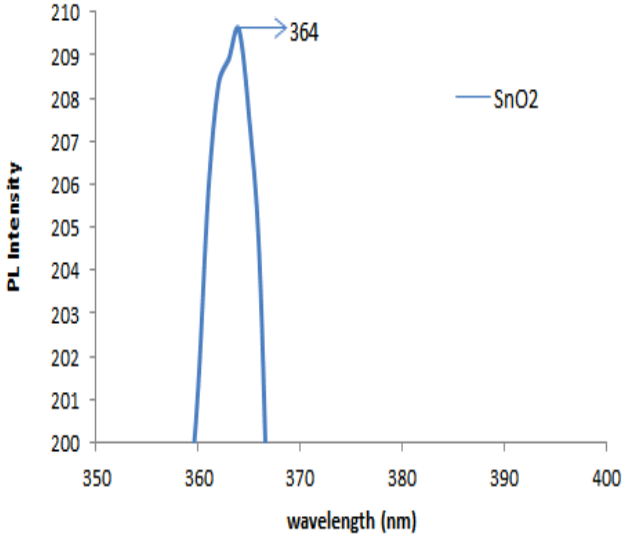

Fig4. Photoluminescence of SnO_2

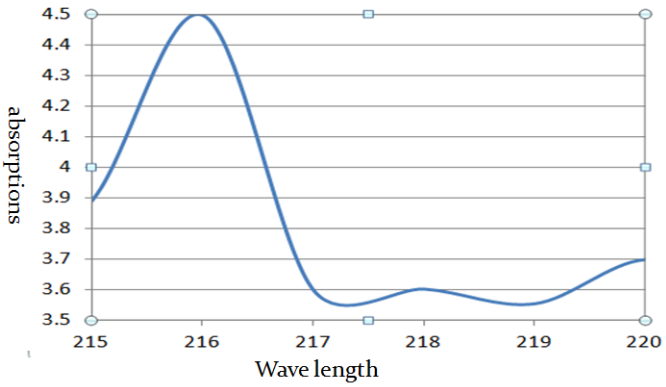

Fig5. UV-Vis absorption Spectrum of SnO_2

D. UV-Vis Spectrum

The UV-Vis spectrum depends on the size of the nanoparticles and the absorption maximum wavelength decreases with the particle size.[11] The UV-Vis absorption and band gap spectra of the synthesized nanoparticles as shown in Fig4. The absorption peaks around 216, 219nm is represented SnO_2.

The band gap values are calculated using wave energy formula

$$Eg = \frac{hc}{\lambda} eV$$

Where h is planks constant, c is velocity of light, λ wavelength of the sample.[12]. The calcuted band gap energy value 3.3eV.

IV. Conclusion

In summary tin oxide nanoparticles synthesised by hydrothermal method from $SnCl_2.2H_2O$ at 700 °C. All the peaks of XRD belong to tetragonal lattice of SnO2. The particle size around is 21nm. The spherical grain morphology of SnO_2 nanoparticles is observed using Scanning Electron Microscopy.In FTIR the broad band at 603 cm^{-1} was due to the vibrations of Sn-O and 1127 cm^{-1} was absorption of water.

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