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Preparation and characterization of SnO_2 nanoparticles by hydrothermal method

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Abstract: In present investigation we report the synthesis of Tin oxide nanoparticles via hydrothermal method using cetyltrimethyl ammonium bromide (CTAB). The cationic surfactant CTAB is a key in determining the morphology of the products. The crystallite size and morphologies of the SnO_2 were characterized by XRD and Field Emission Scanning Electron Microscopy (FESEM). XRD pattern shows that the obtained powder is SnO_2 with tetragonal crystalline structure. The results of FTIR are also discussed.
Keywords: CTAB; Hydrothermal; SnO_2.

Introduction and Experimental:

Nanomaterials have attracted great interest due to their intriguing properties, which are different from those of their corresponding bulk state. Enormous efforts are being taken towards the development of nanometer sized materials in studies related on one hand to their fundamental mechanism such as the size effect and the quantum effect and on the other hand towards application of these materials. Nanometer sized material and semiconductor particles have a large potential for industrial applications. Metal oxide semiconductors are low cost and effective gas sensing material. Among the various metal oxide semiconductor, SnO_2have been attracting much attention since they are highly conducting, transparent and sensitive to gases. SnO_2 is highly interesting as it is an n-type semiconductor with direct band gap of 3.6eV between the full oxygen 2 p Valence band and the tin states at the bottom of the conduction band and offers many technological applications such as catalysts for oxidation of organics, solid state gas sensors and optical electronic devices. Many processes have been developed to the synthesis of SnO_2 nanostructures e.g. hydrothermal method, microwave assisted, chemical precipitation method, sol-gel [3-6] and Spray pyrolysis and conventional co-precipitation method. A number of researchers have reported the use of surfactants in the synthesis of nanometal oxides. An optimized amount of surfactants can achieve high specific surface area and narrow pore size. Surfactants are used in the preparation of crystalline metal oxide in the nano scale to control crystal growth and to provide solubility[1,7].

In this paper we report the simple hydrothermal synthesis of SnO_2nanoparticles using $SnCl_45H_2O$ as the inorganic precursor, CTAB (cationic surfactant) as an organic template and Urea as a reagent to control the pH and to obtain a pure and dense hydrous tin oxide.

The chemical reagents used were hydrated Tin chloride ($SnCl_4$ $5H_2O$) fromSigma, Cetyltrimethyl ammonium bromide ($C_{16}H_{33}$) N ($(CH_3)_3Br$), Urea $(NH_2)_2CO$,ethanol(C_2H_5OH).All chemicals used were as received without further purification. Initially 2mmol of hydrated Tin chloride and 2mmol of CTAB was dissolved in 50 ml of distilled water in a beaker under continuous stirring. 2mmol of urea was added drop by drop to adjust pH at 8.The solution was stirred to homogeneity, and then the mixture was transferred to a 100ml Teflon- lined autoclave. The solution was heated in an oven at 100^{o}C for 24 hours. The resulting product was washed with distilled water and ethanol several times via centrifugation to remove any impurities and then dried at 80^{0}C overnight. The dried solids were calcinated at 600^{o}C for 2 hours. The calcined powders were examined by X-Ray diffraction, FTIR and FESEM techniques.

Results and Discussion:

The XRD pattern of SnO2 nanoparticles recorded in the region of 2ϴof 0 – 80^o are displayed in Fig.1 the XRD results show that all diffraction peaks at 26.6^0, 33.9^0 and 51.9^0, indicate the formation of SnO_2 nanoparticles. These three peaks correspond to the (110),(101) and (211) reflection planes of a tetragonal lattice of SnO_2 as identified using the standard JCPDS file no.41 -1445. No obvious reflection peaks from impurities such as unreacted Sn or SnO were detected, thus indicating the high purity of the product with a tetragonal rutile structure. The average crystallite grain size calculated using Scherrer formula of $SnO_2$is 19 nm.

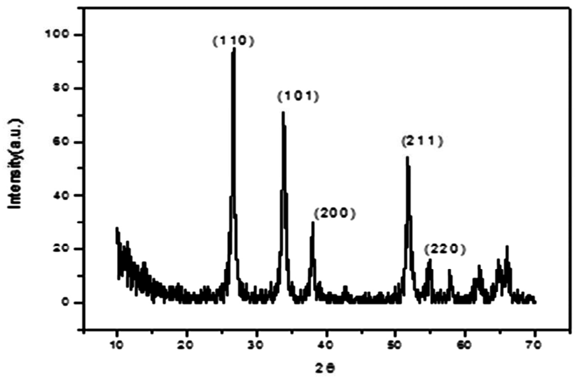

Fig. 1.XRD pattern of SnO_2 nanoparticle

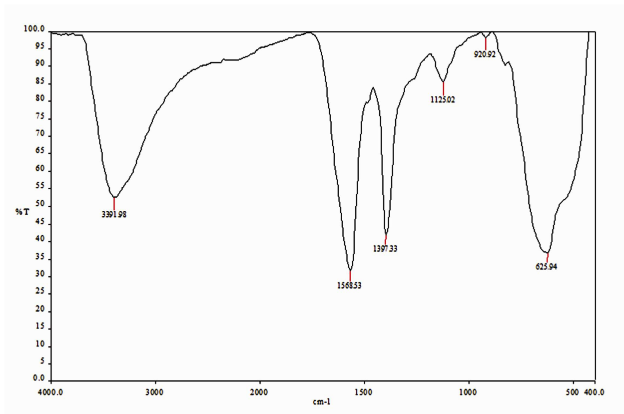

Fig. 2. FTIR Spectra of SnO_2 nanoparticle

The FTIR transmission spectra of SnO_2 synthesized using CTAB is shown in Fig.2.The peaks at 3391cm^{-1}and 1568 cm^{-1} were due to stretching vibrations of water molecules or hydroxyl groups absorbed at the surface of the Tin oxide.The bandsat 625cm^{-1} and 930 cm^{-1} refers to Sn – O stretching modes of Sn-O-Sn[1,5]. The band around 1400cm^{-1} was assigned to NH deformation of ammonia and the NH stretching vibration from decomposition of urea.

The morphology of the calcined material was obtained by Field Emission Spectroscopy (FESEM).It can be seen from Fig.3. That the samples are composed of spherical particles which are smaller in size. These small particles are aggregated into larger particles leaving some pores in between which make them ideal for various applications. In order to check whether the template molecules are completely removed during the calcinations process, the calcined sample was analyzed with FESEM-EDX (Fig.4.).The sample exhibits the peaks relating to only Sn and O.

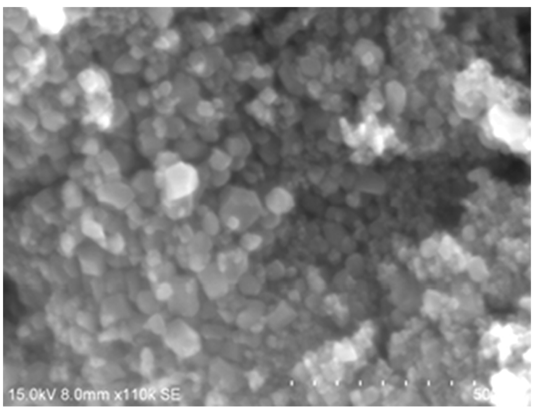

Fig.3. FESEM image of SnO_2 nanoparticle

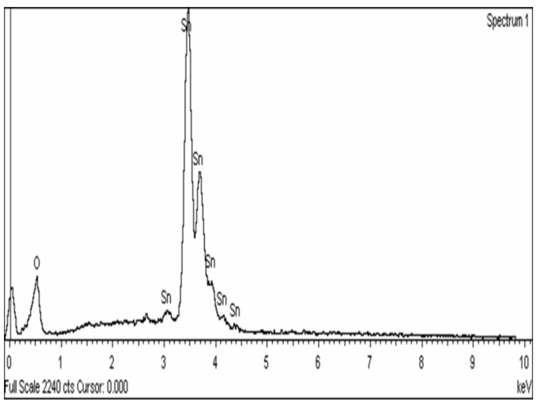

Fig.4. EDX spectrum of SnO_2 nanoparticle

Thus the SnO_2 nanoparticle have been successfully synthesized by a simple hydrothermal method using CTAB, a cationic surfactant. XRD spectra indicated the tetragonal structure of the particle and the surface morphology was investigated by FESEM.

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