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Multifunctional performance of nanocrystalline tin oxide

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ABSTRACT

Crystalline tin oxide, with nanometre size range in diameter, was synthesized using a solution phase approach. The oxide material showed the photocatalytic performance for the reduction of a xanthene based dye in presence of ultra-violet irradiation, where the conduction band electron is responsible for the reduction mechanism. In addition to that the calculated power density and energy density values of the synthesized tin oxide nanoparticles, in combination with graphitic carbon, is comparable with the electrochemical supercapacitor like materials.

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1. Introduction

Nanotechnology has emerged as one of the most exciting fields in material sciences and the synthesis of nanocrystalline materials is becoming one of the most fascinating techniques that has been applied in most of the research areas in science and engineering. Nanosized semiconductor metal oxides are known to possess distinctive functionalities due to their high density surface sites and have emerged as an important class of materials with the great potential for diverse applications in electronics [1], optics [2] and catalysis [3]. Size of the material has a profound effect on a number of intrinsic properties in systems including the band gap [4] and at the same time the surface to volume ratio can also have the consequence on the electronic [5], magnetic [6] and optical [7] properties of the system. Among the various properties of nanomaterials, semiconductor nanocrystals with quantum confinement effect leads to spatial enclosure of the electronic charge carriers within the crystal dimension and thus the transport properties of the system are largely affected by the size and geometry of the materials [8,9].

Tin dioxide, SnO_2, is an n-type semiconductor with a wide-band gap of 3.6 eV [10], is considered as one of the smart materials, among the various semiconductor metal oxides, due to their good stability, nontoxicity and low cost which allow for its diverse application. The physicochemical properties of SnO_2 are closely linked to its size and shape. Report has been published for the synthesis of mesoporous SnO_2, with large surface area, using a hydrothermal synthesis route for the photo-degradation of organic dyes [11]. The sensing property of the large pore sized SnO_2-based thick films were investigated and reported for the detection of very low concentration of hydrogen sulphide [12]. A hydrothermal synthesis route was introduced for the synthesis of graphene-tin oxide composite and was applied as a catalyst for the electrochemical detection of dopamine [13,14]. A solution-based method has been employed for the synthesis of SnO_2-MnO_2 composites for the supercapacitor application [15]. SnO_2 and carbon cloth flexible composite system showed excellent performance as an anode material in lithium-ion battery application [16]. Micro structured SnO_2, has been reported [17] by a hydrothermal synthesis route, exhibit excellent photocatalytic performance with potential applications in waste water purification. Highly aligned nanorods of SnO_2 were synthesized by a hydrothermal method on a graphene matrix and has been reported [18] for a gas sensing application with improved performances. Excellent organic vapour sensing property with long-term stability has been obtained from the interconnected SnO_2 and Fe_2O_3 based nanotubes synthesized by combining the single nozzle electrospinning and thermal treatment methods [19].

In this work, crystalline SnO_2 particles, with the diameter ranging from 5 to 25 nm, have been prepared using a solution phase approach. The synthesized material was used as a photocatalyst for

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the reduction of a xanthene based dye. In addition to that, a detailed study indicates that the crystalline SnO_2 particles have the potential as an electrochemical supercapacitor when combined with graphitic carbon. The synthesized nanomaterial was also characterized using different optical, microscopic and surface characterization techniques.

2. Experimental

2.1. Materials

All the chemicals used for this study including stannous chloride, citric Acid, sodium hydroxide and xanthene based dye were purchased from Merck. The chemicals and solvents were used as received without further purification. All the solutions were prepared using mili-Q water.

2.2. Synthesis of nanostructured SnO_2

In a typical synthesis method, 40 mL of $SnCl_2$ (10^{-1} mol dm^{-3}) was taken in a three-neck flask equipped with a condenser and thermometer. A water solution of citric acid (10 mL, 10^{-2} mol. dm^{-3}) was added dropwise to the stannous chloride solution under continuous stirring condition under the room temperature. After the complete addition, the system was heated at 60 °C and water solution of sodium hydroxide (10^{-1} mol. dm^{-3}) was added dropwise to the above mixture solution until the complete precipitation was formed and later allowed to cool at the room temperature. The solution was centrifuged and washed with ethanol, acetone and water for several times. The solid material was collected and dried in oven. The dried material was further calcined at 500 °C for 4 h. Finally, the synthesized powder material was characterized using microscopic and spectroscopic techniques and also applied as a catalyst for a photochemical reaction. The synthesized material, in combination with graphitic carbon, was also studied for the electrochemical charge-discharge performance for the supercapacitor application.

2.3. Photocatalytic experiments

Initially, 20 mg of the synthesized material was suspended in 100 mL of xanthene dye (10^{-4} mol dm^{-3}) in a conical flask and placed inside the dark room, under the continuous starring condition for the period of 30 min, to allow an adsorption-desorption equilibrium. After that the conical flask was placed under the Philips UV-C (TUV T8) (germicidal) lamp with optical intensity value of ~40 mW/cm^2 adjacent to the solution. For a control experiment, the second set was exposed under daylight with optical intensity value of 3.5 mW/cm^2 adjacent to the solution. At a particular time interval, for the period of 3 h, 2.5 mL of the solution, from both the experiments, were collected and the intensity of absorption was monitored using a spectrophotometer.

2.4. Electrochemical experiments

The incorporation of graphitic carbon to the synthesized materials was done through a milling process for the period of 3 h by maintaining the ratio of 1:10, respectively. The electrochemical properties show the evidences for the successful incorporation and the composite formation.

2.5. Characterization

Transmission electron microscopy (TEM) studies were performed using a Philips CM200 TEM instrument equipped with a LaB6 source. The TEM samples were prepared by depositing small amount of synthesized material onto a 200 mesh size Cu grid. The X-ray diffraction (XRD) patterns were recorded on a Shimadzu XD-3A performed over a diffraction angle range of $2\theta = 20°-80°$. X-ray photoelectron spectra (XPS) were collected in a UHV chamber attached to a physical electronics 560 ESCA/SAM instrument. The UV–vis spectra were measured using a Shimadzu UV-1800 spectrophotometer using a quartz cuvette. Electrochemical studies were carried out with a Bio-logic SP-200 potentiostat connected to a data controller. A three-electrode system was used in the experiment with a glassy carbon electrode (GCE) as the working electrode. Ag/AgCl electrode (saturated KCl) and a Pt-electrode were used as the reference and counter electrodes, respectively.

3. Results and discussion

The X-ray diffraction study was done for the synthesized nanomaterial, which is indexed to the formation of rutile tetragonal phase of SnO_2 (Fig. 1A). All the crystalline peaks are well matched with the reference (JCPDS 00-041-1445) and no other impurity was observed [20]. Furthermore, the height and sharpness of the peaks suggest that the product is well crystallized. From the XRD pattern it is evident that the preferred orientation of the crystal is towards the direction of (110) plane. The detail formation mechanism of SnO_2 is mentioned in the supporting document. To identify the chemical state of the tin (Sn), X-ray photoelectron spectroscopy (XPS) measurements were performed. Fig. 1B shows the high resolution XPS spectrum of the SnO_2 nanocrystals, where two peaks, separated by 8.75 eV, with the binding energy values of 486.8 and 495.3 eV were observed due to the spin-orbit splitting of the Sn 3d level, namely Sn $3d_{5/2}$ and Sn $3d_{3/2}$ electrons, respectively, indicates the presence of Sn(IV) component [21]. The TEM image (Fig. 2) shows the wide size distribution, within the range between 5 and 25 nm, of the synthesized material. The image also indicates the collection of the SnO_2 particles with prominent grain boundaries for individual the particles. The study of the pore structure of the solids is connected with interpretation of adsorption-desorption isotherm. Fig. 3A indicate the present tin oxide sample can be categorized as the type-IV isotherms with the combination of H-1 and H-2 types of hysteresis loop, as per IUPAC recommendation, where the agglomeration of spheroidal and corpuscular types of

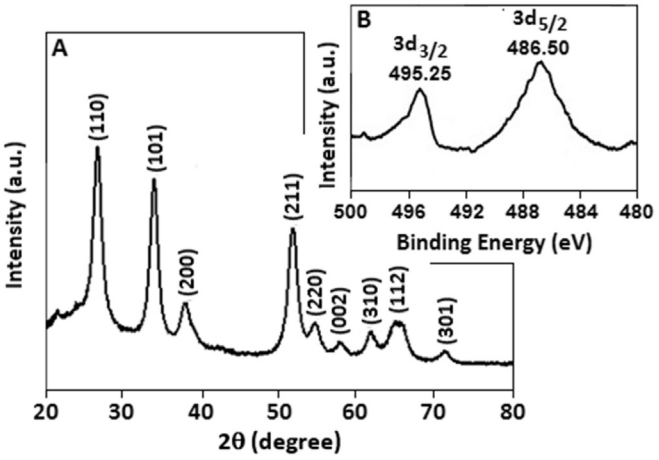

Fig. 1. (A) X-ray diffraction pattern of the tetragonal phase of SnO_2 nanocrystalline particles with the preferred orientation towards the direction of (110) plane. (B) H*igh resolution XPS spectrum of* the SnO_2 nanocrystals with the binding energy values of 486.8 and 495.3 eV represent the spin-orbit splitting of Sn 3d level and indicates the presence of Sn (IV) component.

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