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Influence of pH and Temperature on The Structure and Size of Tin Oxide Nanoparticles

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ABSTRACT

A systematic study on the preparation of tin oxide nanoparticles using the precipitation method has been conducted. The preparation of nanomaterials was by varying reaction parameters such as pH and temperature. The tin oxide nanoparticles were characterized by using AFM, SEM, XRD and UV-Vis. Particle size was obtained using XRD studies the value is 28.8 nm, 35.2 nm, 30.8 nm and 33.8 nm. It was found that the alteration of pH and temperature changes the particle size.

1. Introduction

Nanoparticles 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 to 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. The morphology of obtained materials is highly dependent on the chosen method, enabling to obtain nanoparticles, nanowires, nanorods, and other morphologies [1]. Tin oxide (SnO_2) is an n-type semiconductor with excellent optical and electrical properties, partly due to its wide band gap (Eg =3.6 ev). In sensor research, many semiconducting metal oxides are used of which tin oxide is the most widely studied and employed owing to its physicochemical properties [2].

Nano-sized tin oxide is regarded as a highly preferred multitasking metal oxide such as gas sensors and lithium rechargeable batteries .The transparent conducting oxide (TCOs) materials has been widely used for various optoelectronic devices, flat panel displays, liquid crystal displays, organic light emitting diodes, solar cells and etc. It has specific properties and advantages of high sensitivity, including conductivity, transparency in the visible region in addition to mechanical and chemical stabilities. However, thermal treatments lead to an increase of the average grain size, spreading of the grain size distribution, and changes in the phase composition with increasing annealing temperature [3]. Among various classes of Nanoparticles (Metals, Semiconductors and Insulators), semiconductor particles have attracted more interests because of their size-dependent optical & electrical properties [4]. This study attempts to provide some findings to this research area [5]. Chemical sensors have played very important roles in the detection of pollutant, toxic, and industrially important gas species such as NO_x, NH_3, COx, H_2 and ethanol. The study of dielectric properties and a.c. electrical conductivity throws light on the behaviour of charge carriers under an a.c. field, their mobility and the mechanism of conduction. Tin oxide nanoparticles are suitable for gas sensing applications due to high surface to volume ratio, compared to bulk tin oxide[6].The associated challenge with chemical precipitation methods is; when the product is calcined at high temperatures the crystallite size increases and the surface area decreases due to the particle growth [7]. Tin oxide has been used as solid state sensor mainly due to its sensitivity towards different gaseous species, photovoltaic energy conversion to make indium tin dioxide (ITO) transparent thin film coatings, etc., [8]. Tin oxide is an important oxide semiconducting material, which has been widely used in many applications such as catalysts agent, hazardous gas sensors, heat reflecting mirrors, varistors, transport conducting electrodes for solar cells and optoelectronic devices. Recent studies have shown that many fundamental physical or chemical properties of semiconductor materials strongly depend on the size and morphology of the materials [9]. Many processes have been developed to synthesis tin oxide nanostructures, e.g., spray pyrolysis, hydrothermal methods, chemical vapour deposition, thermal evaporation of oxide powders and sol-gel method. Annealing the SnO_2 nanoparticles prepared adding hydrochloric acid improved the crystallite size [10]. In the present work the fabrication and characterization of crystalline tin oxide nanoparticles powders by chemical precipitation method.

2. Experimental Methods

2.1 Chemical Precipitation Method

About 6 g (0.1 M) of stannous chloride dehydrate ($SnCl_2.2H_2O$) was dissolved in 300 mL of distilled water. After complete of dissolution, the ammonia solution was added to the above solution by drop wise under stirring. The pH is measured and it was adjusted to 11. The particles were then allowed to settle down at the bottom of the flask. The resulting gels were filtered and dried at 80 °C for 24 hours. The obtained product was heated to the temperature of 500 °C and 600 °C for 2 hours. The same procedure was repeated by changing the pH=9 also. The final obtained product was white tin oxide nano powder.

3. Results and Discussion

3.1 AFM Analysis

The atomic force microscope (AFM) was ideally suited for characterization of nanoparticles. It offers the capability of 3D visualization and both qualitative and quantitative information on many

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physical properties including size, morphology, surface texture and roughness. A wide range of particle sizes can be characterized in the same scan, from 1 nanometer to 8 micrometers. In addition, the AFM can characterize nanoparticles in multiple media ambient air, controlled environments and even in liquid dispersions. Resolution of AFM for SnO_2 sample was 300x300 pixels. The sample was analyzed in non-contact mode with nominal diameter of tip 10 nm.

The height of the nanoparticles roughness can be measured by atomic force microscope. Figs. 1(a, b) show tin oxide nanoparticles of maximum height 40 nm prepared in pH=8.0 at 500 °C and 100 nm prepared at 600 °C. Figs. 1(c, d) present the prepared tin oxide nanoparticles of maximum height 50 nm and 200 nm in pH=10.0 at 500 °C and 600 °C respectively.

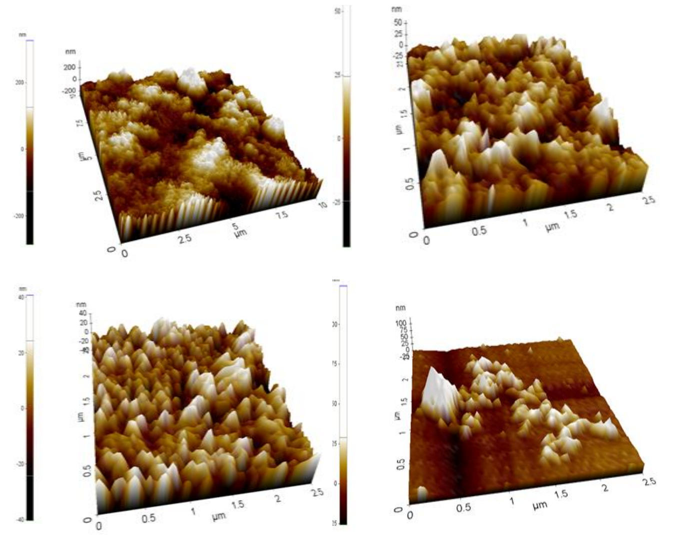

Fig. 1 AFM image of tinoxide nanoparticles prepared at pH=8 and pH=10 a) 500 °C, b) 600 °C and c) 500 °C d) 600 °C respectively

3.2 SEM Study

Scanning electron microscope (SEM) was used for the morphological study of tin oxide nanoparticles. Figs. 2(a-d) show SEM image of tin oxide nanoparticles. In Fig. 2a (pH=8, 500 °C) cauliflower like morphology structure can be seen. In Fig. 2b (pH=8, 600 °C) spherical like morphology structure was observed. In Fig. 2c (pH=10, 500 °C), highly porous foam like structure is observed and Fig. 2d (pH=10, 600 °C) few agglomeration has been observed. The particle sizes and the structures are varied due to the change in the reaction parameters such as pH and temperature.

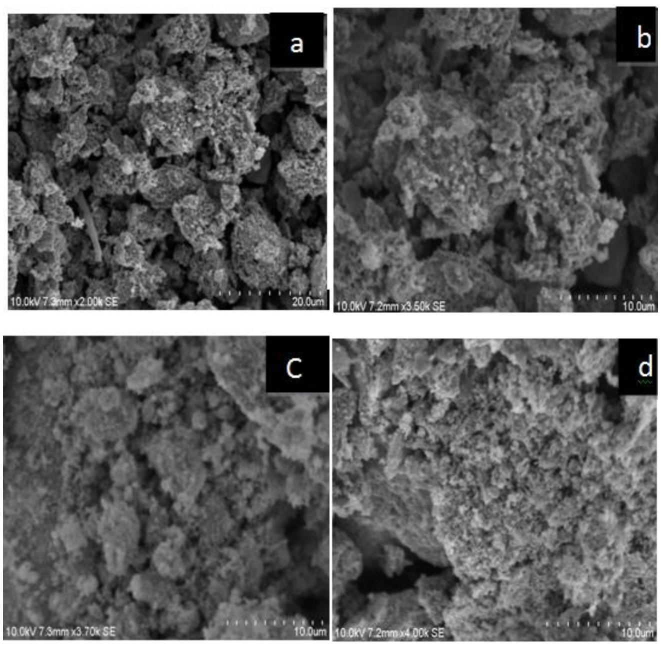

Fig. 2 SEM images of tin oxide nanoparticles prepared at pH=8 and pH=10 a) 500 °C, b) 600 °C and c) 500 °C d) 600 °C respectively

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3.3 XRD Analysis

In order to determine the size and structural properties of the synthesized tin oxide nano particles, the powdered XRD analysis was performed. Structural identification of SnO_2 nanoparticles were carried out with X-ray diffraction in the range of angle 2θ between 10° to 80°. Moreover, a sharper and higher relative intensity peaks at higher reaction temperature was attributed to highly crystallinity of SnO_2 NSs as the degree of crystallinity increase with reaction temperature [11]. Figs. 3(a-d) show XRD patterns for tin oxide nanoparticles, which was crystalline in nature. The diffraction peaks are markedly broadened, which indicates the crystalline. XRD spectrum of tin oxide nanoparticles sizes of samples are very small. According to the Debye-Scherer's equation [12], $D=0.9\lambda / \beta \cos \theta$, where λ is the wavelength, β is the width of half maximum of the diffraction peak and θ is Bragg diffraction angle. Fig. 3 shows the average crystalline size calculated using Scherrer formula are 28.77 nm, 35.19 nm, 30.82 nm and 33.8 nm respectively.

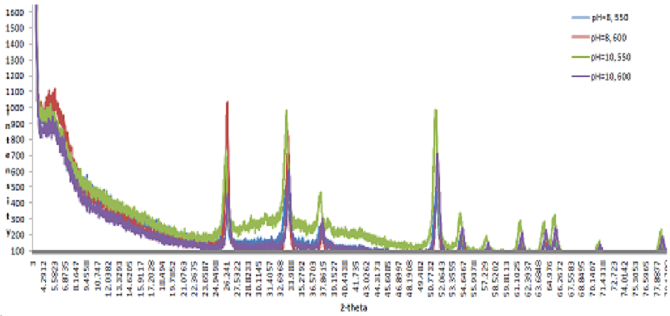

Fig. 3 XRD spectrum of tin oxide nanoparticles prepared at pH=8 & 10 at 500 °C, 600 °C respectively

3.4 UV Analysis

The size of the nanoparticles plays an important role in changing the entire properties of materials. Thus, size evolution of semiconducting nanoparticles becomes very essential to explore the properties of the materials. UV-visible absorption spectroscopy is widely being used technique to examine the optical properties of nanosized particles. Absorption bands confirm the formation of the tin oxide nanoparticles. Fig 4 shows that peaks were observed at 403 nm at 500 °C and 407nm at 600 °C in pH=8.0, 410 nm for 500 °C and 413 nm for 600 °C in pH 10.0.

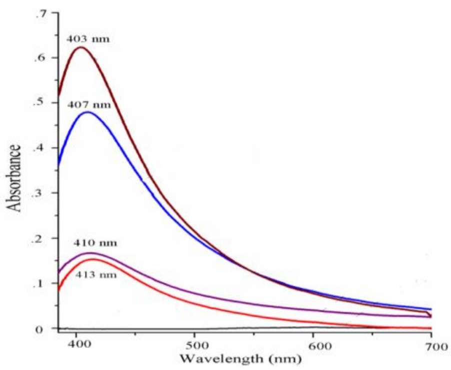

Fig. 4 UV spectrum of tin oxide nanoparticles prepared at pH=8 & 10 at 500 °C, 600 °C respectively

4. Conclusion

Tin oxide nanoparticles have been synthesized using chemical precipitation method. The resulting product was crystalline with agglomeration and hence more surface area. Surface morphology was studied using SEM and crystalline size calculated using XRD of tin oxide nanoparticles showed different structures and sizes due to variation in reaction parameters such as pH and temperature. The surface roughness of the tin oxide nanopartices were also measured using AFM. The maximum absorbance were observed at 403 nm at 500 °C and 407 nm at 600 °C in pH=8.0, 410 nm for 500 °C and 413 nm for 600 °C in pH 10.0 by UV-vis absorption spectrum. Also, the process used the economically lower cost chemical, tin (II) chloride as the precursor. The present study provides inexpensive and easy method to improve the quality of tin oxide nanoparticles. Thus, this synthesis method is fast, simple, convenient and feasible on industrial scale to synthesize SnO_2 nanomaterial. The possible applications of SnO_2 nanoparticles are in gas sensor, solar cells, optoelectronic devices, paint industry, and surface coating industry.

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