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Structural and Morphological Properties of Tin Oxide (SnO_2) Nanoparticles by Microwave Irradiation Method

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ABSTRACT

In the present work SnO_2 nanoparticles have been successfully prepared by a microwave irradiation method. The crystallite size and morphology of SnO_2 have been investigated by X-ray diffraction (XRD), scanning electron microscopy (SEM), energy dispersive spectrum (EDS), and transmission electron microscopy (TEM) techniques. The XRD pattern of average particle sizes of SnO_2 is estimated to be around 14 nm. Furthermore, SnO_2 nanoparticles have the crystallite size in the range ~11-50 nm, as confirmed by TEM. Results obtained indicate that the microwave-assisted method is a promising low temperature, cheap, and fast method for the production of SnO_2 nanostructures.

KEYWORDS: Structural; XRD; SEM; TEM; Microwave irradiation method

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INTRODUCTION

If a particle size of a semiconductor becomes comparable to the Bohr radius of the exiciton, the ratio of the surface atoms to those in the interior increases remarkably, leading to the materials[1.] Two tin and four oxygen atoms per unit cell the ideally stoichiometric SnO_2 is an insulator, however, the real SnO_2 structure contains oxygen vacancies, which make this material an oxygen deficient tin (IV) oxide is n-type semiconductor with band gap 3.6 to 3.8 eV which has rutile- type Cassiterite structure. As one of the most important classes of materials metal oxide semiconductor are presenting themselves in various are of science and technology due to[2-3] using a nanocrystalline metal oxide as an anode materials has many advantages because of the larger surface are and high sensitivity[4-5]. Tin oxide enjoys a place of pride because of its very high specific capacity (>600 mAh/g). SnO_2 is transparent conducting oxides (TCOS). SnO_2 has important physical properties such as a high optical transparency in the visible-light high electrical conductivity, low electrical resistance, good chemical and thermal stability in various environmental conditions. It is fully explored due to its potential applications in catalysis[6].There are two obvious approaches to improve the gas sensing sensitivity and selectivity of SnO_2 one strategy is doping with some rare earth ions or noble metals ions such like Y[7], La[8], Ce[9], Pr[10], Nd[11] and Sm[12], another approach is to increase the specific surface area because SnO_2 is a surface-resistance-control semiconductor material for this reason, significant efforts including Nano structured tin oxides have been synthesized by a Varity of techniques such as sol-gel[13], Ultrasonic[15], spray Pyrolysis[16], Thermal evaporation[17], co-precipitation[18], hydrothermal[19], solve thermal[20], SnO_2 different morphologies have been synthesized such as nanoparticles[21], Nanowires[22], Nanotubes[23], Nanoroads[24], Nanobelts[25] , Nanofibers[26], Grass-like nanostructures[27], Core-shell nanostructures[28].The studies of the micro-wave heating are even complicated by fact that the rate enhancement of chemical reactions core depends on many complex factors[27] and the effects of several factors The usage of energy electronic devices has been increased in recent years due to the rapid growth of smart Phones, Smart watches, Camera, Laptop and personal digital assistant (PDAS) electrochemical capacitor has stood for energy storage system.

The electrode materials such as Carbon materials, Transition metal oxides and conducting polymers play an important role in the performance of super capacitors, Ru[28], MnO_2 [29], Co_3O_4 [30], NiO [31], and SnO_2 [32], are the promising electrode materials due to the high specific capacitance. Among various metal oxide nano particles, SnO_2 is inexpensive and Non-toxic in nature and Gas sensors[33], Dye-based solar cells[34], Light emitting diodes[35], Transistors[36] etc. Such as volume[37], solvent[38], size of the reaction vessel and power level in micro-wave assisted organic synthesis. SnO_2 nanoparticles with sizes less than 30 nm were synthesized by a micro-wave assisted solution process in which amorphous oxy-hydroxy precipitate sn^{2+} was crystallized by micro-wave heating. Sn^{2+} oxidation otherwise prevailed in the conventional thermal heating process. The micro-wave is highly efficient and energy saving in the process, including short reaction time, tiny energy consumption and product yield. The microwave assisted method offers to larger reaction volumes , allows faster reaction time and removes the need of high demands post synthesis of annealing , huge requirement of time and large energy consumption[39]. Microwave assisted synthesis effects rapid processing speed, homogeneous heating and simple control of processing condition and thus attracted much attention in last four years. Synthesis SnO_2 nanocrystals via microwave assisted process and demonstrated that anatase nanocrystals are highly crystalline, low in Ti^{3+} effect and free of aggregation. The Chemical synthesis in a liquid phase through microwave irradiation mainly involves dipolar polarization and ionic conduction heating mechanisms. Nature of properties of the SnO_2 crystals depends on different kind of defects and impurities that are present in structure of material. These defects could affect its structural, electronic, optical and/or magnetic properties [40].

In this paper reports on tin oxide synthesized by micro-wave irradiation method. The effect of conventional heating treatment. In this paper we focused a microwave irradiation with 2.45 GHZ frequency to obtain SnO and SnO_2 after calcinations, nanocrystalline powder in rapid volumetric heating which increased reaction rates and shortened the reaction time.

EXPERIMENTAL

Material

All chemical regents were of analytical grade and used without purification. The tin Chloride was purchased from (Merck product 98% AR grade) and ethanol solution (Merck product) were used to synthesis SnO_2 nanoparticles sample was prepared by using double distilled water.

Synthesis

A pure tin (II) ($SnCl_2.2H_2O$) was used as precursor, and ethanol was used as solvent. Solution of $Sncl_2$ in ethanol was prepared by a 0.1 M of solution under continuous stirred at room temperature until the transparent and colorless sol was obtained. The pH was maintained at 7 to 8 by using ammonia solution and continuously stirred. That sol further washed with ethanol to remove $NH4^{+}$ions. The resulting precipitate was irradiated with house hold micro-wave oven for 5 minutes. The radiation frequency was kept between 2.45 GHZ to 1KW between to 1KW with convection mode. The finally, a white product was annealed at various temperature.

CHARACTERIZATION TECHNIQUES

The microstructure of the sample was analyzed by X-ray diffraction (XRD) using a Bruker AXS D8 Advance instrument and the monochromatic CuK1 wavelength of 1.5406 Å. The average crystalline size of the crystallites were evaluated using Scherrer’s formula, where is the mean crystalline size, is a grain shape dependent constant (0.9), is the wavelength of the incident beam, is a Bragg reflection angle, and is the full width at half maximum (FWHM) of the main diffraction peak. The sample morphology was observed by scanning electron microscopy (SEM), using a JEOL 5600LV microscope at an accelerating voltage of 10 kV. High resolution transmission electron microscopy (HRTEM) and selected-area electron diffraction (SAED) was recorded on a Tecnai G20-stwin using an accelerating voltage of 200 kV.

RESULTS AND DISCUSSION

X- Ray Diffraction (XRD)

The XRD pattern of tin oxide nanoparticles after microwave treatment and different annealing (or) sintering temperature (at 300°C - 600°C) showed the formation of SnO_2 nanocrystals due to the microwave radiation which changed the hydroxyl group into the oxide group. It was oxidant that the different matched perfectly with the SnO_2 tetragonal structure. When the sample was sintered at 300°C - 600°C, the particles were oxidized at SnO_2. The sintered samples at 300° C-600° C showed typical SnO_2 tetragonal phase which could be attributed to following miller indices (110), (101), (220), and (211).

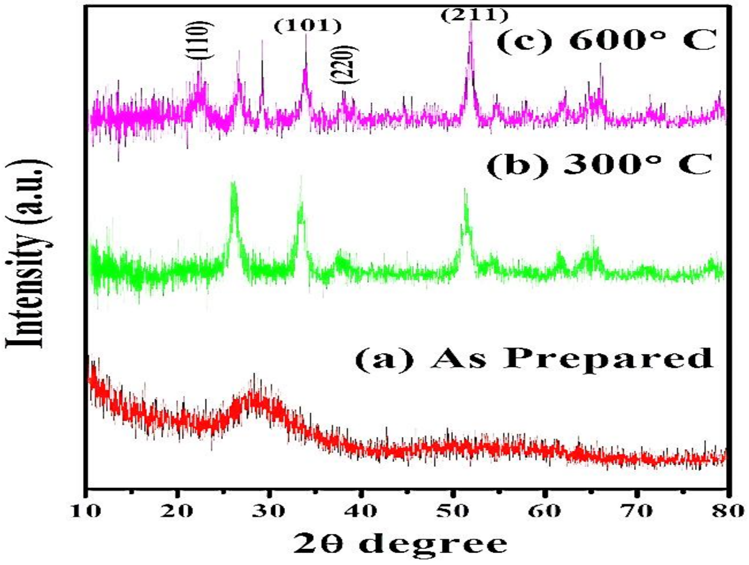

Figure No. 1: “XRD patterns of SnO_2 nanoparticles.”

The obtained XRD patterns of tin oxide (SnO_2) nanoparticles were in good agreement with the standard JCPDS card no 88-0287.

No significant variations were observed while increasing sintering temperature from 300°C-600°C, but the lattice parameter tended to slightly decrease. Such behavior might be attributed to the complete dehydroxylation of the material suggesting the presence of hydroxyl group in the tin oxide crystallites sintered at 300°C-600°C.

Scanning Electron Microscopy (SEM)

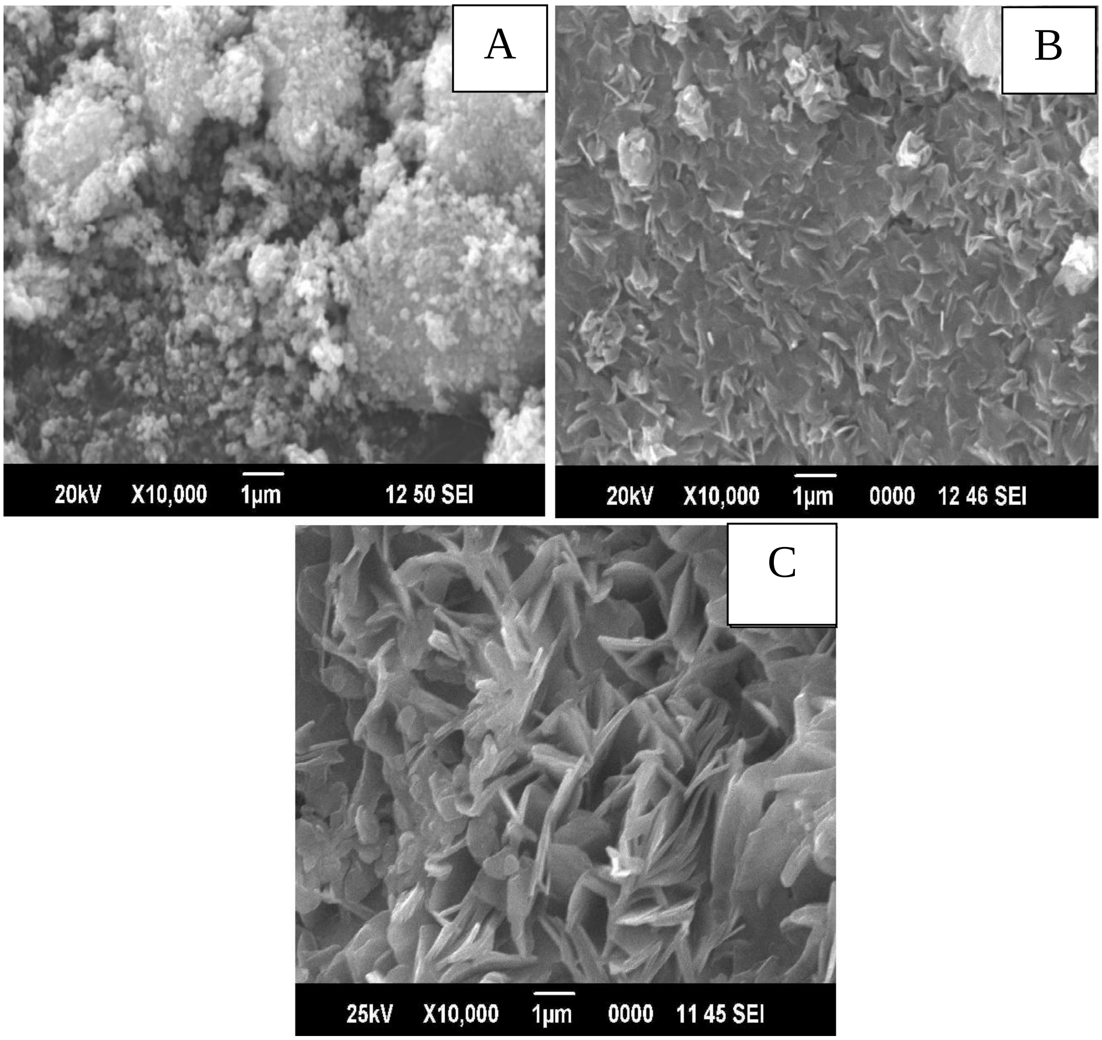

Figure No. 2: "SEM images of SnO_2 nanoparticles."

The Morphology of the synthesized nanocomposite was analyzed by scanning electron microscope. A typical micrograph of Samples (a), (b) and (c) are shown in fig.3. SEM images also show that the synthesized samples are agglomerated and smaller crystallites joint together.

Therefore the temperature distribution is uniform and is transformed the materials inside, making a volatile effect followed by vigorous growth of the gases to form SnO_2 with good polycrystalline nature. It was observed that for each annealing temperature, the synthesized nanoparticles uniform surface without any significant defects and cracks. Surface of the particles consisted spherical particles fused each other.

Energy Dispersive Spectrum (EDS)

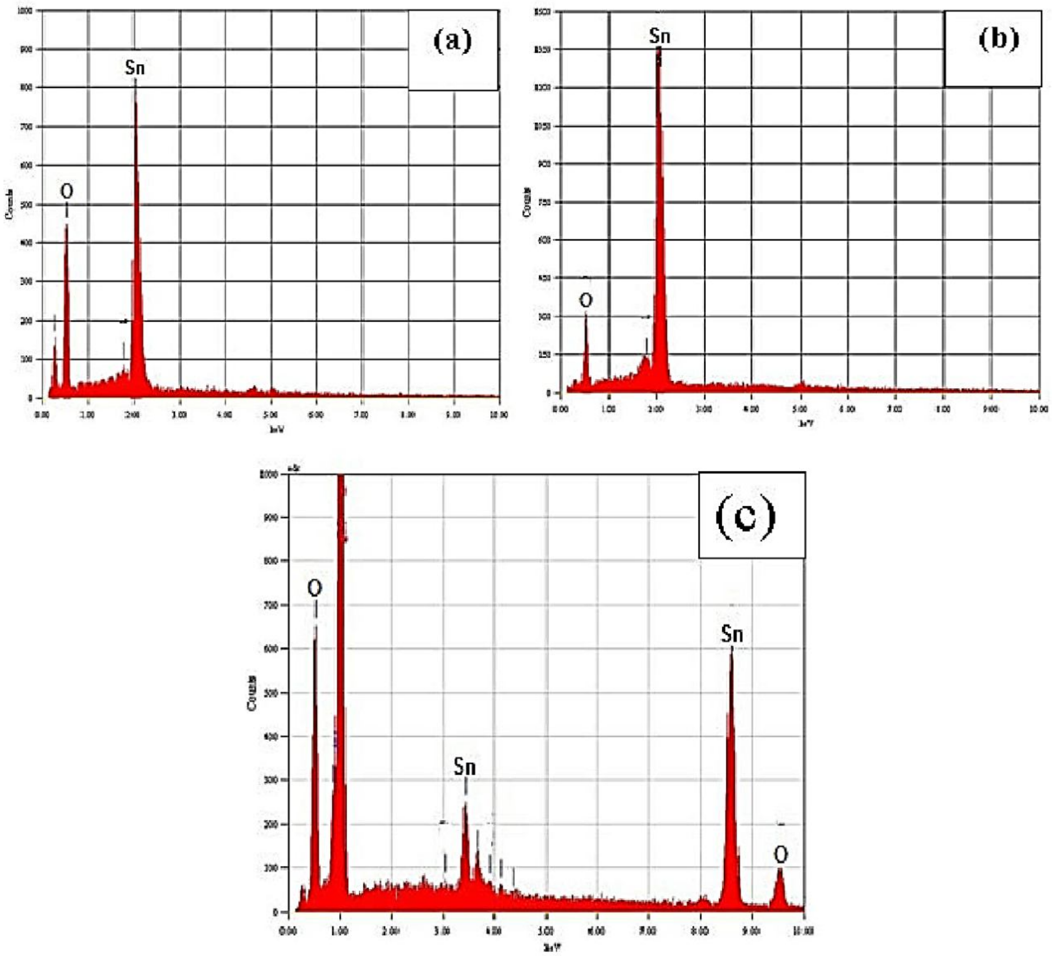

Figure No. 3: "EDS spectrum of SnO_2 nanoparticles."

The spectrum of the synthesized nanocomposite was analyzed by energy dispersive spectrum (EDS). A typical spectrum of sample (a), Sample (b) and sample (c) in shown fig. This spectrum is performed to investigate the element composition of SnO_2 nanostructures. EDS analysis confirms that the presence of SnO_2 nanostructures. The emission peak such as O and Sn observed in EDS Spectrum shows the presence of tin and oxygen element and confirms the Stoichiometry of nanoparticles.

Transmission Electron Microscopy (TEM)

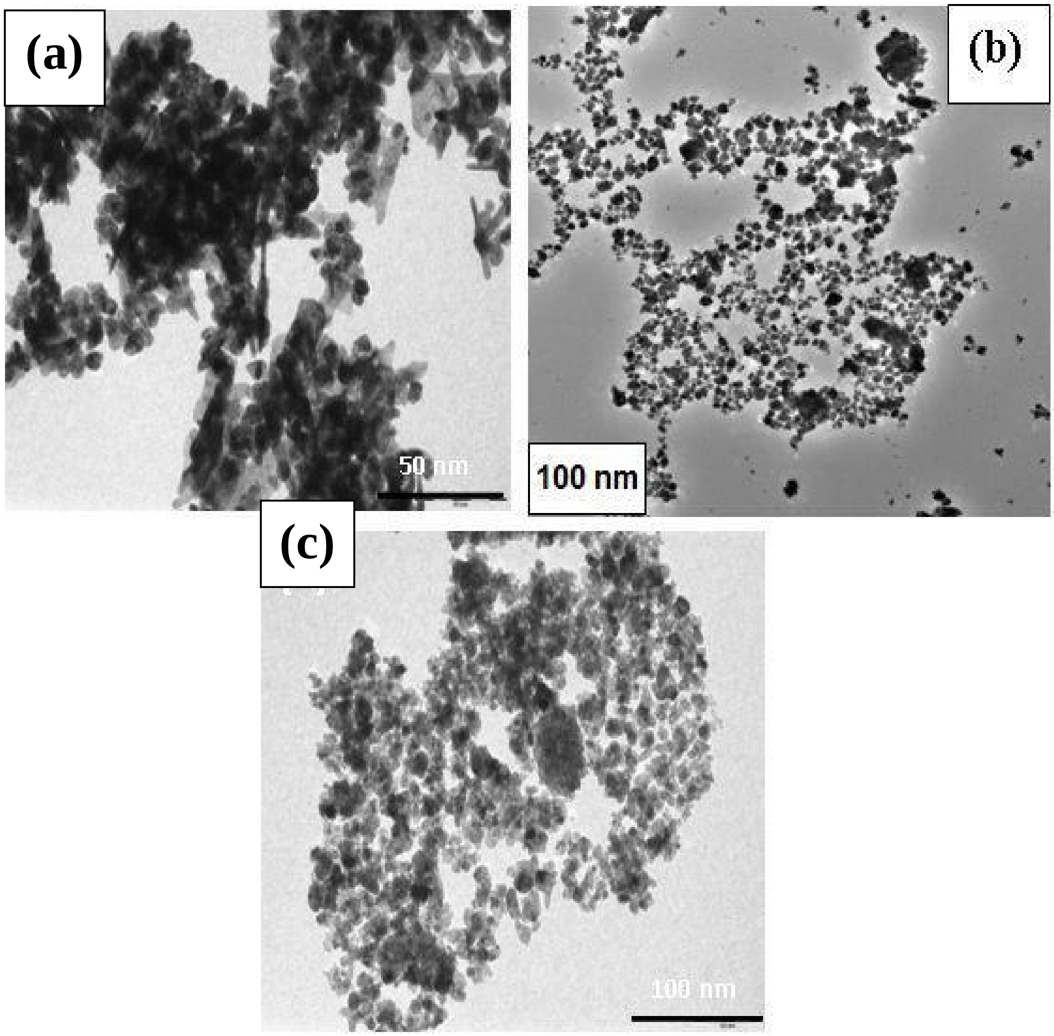

Figure No. 4: “TEM images of SnO_2 nanoparticles.”

The Morphologies of the tin oxide nanoparticles were analyzed using TEM micrographs (fig 3.4). The SnO_2 particles showed needle and spherical shaped morphology with different magnification such as 50 and 100 nm. The estimated particle size well matched with XRD measurements. Thus the larger particles are composed of many smaller particles. After annealing the sample, it showed that the mainly spherical shaped particles with sizes in range from 50 nm-100 nm have formed (fig 3.b, c). It appears that the spherical shape breaks in to the smaller nanoparticles annealing at 600° C for 12h in an air atmosphere.

CONCLUSION

Pure SnO_2 nanopowders with tetragonal phase were successfully synthesized by microwave irradiation technique and no other impurity was observed it's confirmed by XRD. SEM morphology of nanoparticles showed good agglomeration with small crystallite joint together and EDS analysis confirms that the formation of SnO_2 nanostructures. TEM image reveals clearly the formation of tetragonal face nanorods when the SnO_2 sample annealed at 600 °C. The obtained SnO_2 product is potential material for optoelectronic device applications.

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