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Synthesis and Investigation of Antimicrobial Activity of Tin Oxide Nanoparticles by Microwave Assisted Solution Method

S. Rajeswari¹, J. Uma Maheswari¹, D. Muthuraj^{1,*}, E. Kumar², V. Veeraputhiran³¹PG and Research Department of Physics, The M.D.T. Hindu College, Tirunelveli – 627 010, Tamilnadu, India.²Department of Physics, School of Science, Tamil Nadu Open University, Chennai – 600 015, Tamilnadu, India.³R & D Department, Alba Industries Pvt. Ltd., Thoothukudi – 628 103, Tamilnadu, India.

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ABSTRACT

Tin oxide (SnO₂) nanoparticles have been synthesized via microwave-assisted solution method. The structural analysis was carried out using X-ray diffraction. It showed that the SnO₂ nanoparticle exhibited primitive tetragonal structure. The SnO₂ product was investigated by FT-IR, UV, SEM and antimicrobial activity studies of SnO₂ have confirmed that they have biomedical applications.

1. Introduction

Nanotechnology is the most essential and electrifying forefront field in physics, chemistry, engineering and biology. In semiconductor nanoparticles, the particle size influences the optical, catalytic, electric and magnetic properties [1]. Tin oxide (SnO₂) is an n type semiconductor with direct band gap, transparent and sensitive to gases [2-4]. SnO₂ nanoparticles used in gas sensors, solar cells, transistors, etc. Many processes have been developed to the synthesis of SnO₂ nanostructures includes spray pyrolysis, hydrothermal methods, chemical vapor deposition, thermal evaporation of oxide powders and sol-gel method [5-10]. In the present work the synthesis and characterization of antimicrobial activity of SnO₂ nanoparticles powder by microwave-assisted solution method was studied.

2. Experimental Methods

2.1 Materials

All the chemicals were used as analytical grade without any further purification. Stannous chloride dihydrate and ammonia were used to prepare the nanoparticles of this work. Double distilled deionized water used in this investigation. Disc diffusion method has employed with *Escherichia coli* and *Staphylococcus aureus*.

2.2 Synthesis

Initially, in a typical synthesis of synthesis of SnO₂ nanoparticles, 3 g of stannous chloride dihydrate [SnCl₂·2H₂O] was completely dissolved in 50 mL deionized water. Then 50 mL ammonia solution was added drop by drop in the solution with constant stirring at 70 °C and its pH value of the mixture was maintained at 12. The stirring was continued for 1 hour. Then the prepared mixture solution was kept in the microwave oven (900 W, 2450 MHz Onida, India) at a temperature of 70 °C for about 20 minutes. Finally, the as prepared sample was centrifuged and washed several times in double distilled water to remove the by-product, and dried in room temperature for 24 hours results in the formation of SnO₂ nanoparticles.

The synthesized nanoparticles were observed to be Pale yellow in colour and very fine powder of SnO₂.

2.3 Instrumentation

Powder X-ray diffraction pattern of the nanoparticles was obtained using a Rigaku Ultima IV powder X-ray diffractometer. The sample was scanned over the required range for 2θ values. The FTIR spectrum of the sample was recorded within the wavenumbers from 4000 cm⁻¹ to 400 cm⁻¹ by using Perkin Elmer Frontier FTIR. Optical properties were examined by UV-Vis spectrophotometer (UV-1800 PC Shimadzu). HRTEM measurements were done by HITACHI H-800, operating at 200 kV. The antimicrobial activity was determined by disc diffusion method.

3. Results and Discussion

3.1 X-Ray Diffraction Method

The XRD pattern of pure SnO₂ synthesized in this work is presented in Fig. 1. It is observed that the XRD reflection peaks of pure SnO₂ sample are found perfect match with the diffraction pattern of SnO₂ published in the JCPDS File No. 41-1445. All the reflections of powder XRD patterns of this work are indexed using the TREOR and INDEXING software packages. The exhibited peaks for nanocrystalline SnO₂ are observed and indexed to (1 1 0), (1 0 1), (2 0 0), (2 1 1), (3 1 0) and (3 0 1) having primitive tetragonal structure of SnO₂. The position and the relative intensities of the tetragonal SnO₂ are in good agreement with JCPDS no. 41-1445. It reveals that the material deposited is SnO₂ of polycrystalline in nature. No diffraction peaks of other impurities are detected, suggesting the SnO₂ product with a high purity. The obtained values in this work are in good agreement with the values reported by other research group [11]. The unit cell parameters of the sample are found using the software 'UNITCELL' and the observed values are a = b = 4.7382 Å and c = 3.205 Å. Due to the nanocrystalline nature of the sample, the peaks are broadened and diffused considerably. Hence, the peak position and resolution are determined using Gaussian peak fitting method. The particle size is calculated using Scherrer equation [11]:

$$d = \frac{0.9\lambda}{\beta \cos \theta}$$

where β is the full width at half maximum in radians, λ is the wavelength of X-rays used and θ is the Bragg's angle [12]. For the various reflection

*Corresponding Author: muthurajmdt@gmail.com (D. Muthuraj)

peaks of the XRD pattern, the particle size is estimated and the average size of nanoparticles of the sample is found to be around 16 - 20 nm.

Fig. 1 XRD pattern SnO₂ nanoparticles

3.2 FT-IR Method

The infrared spectroscopy is effectively used to identify the functional groups of the synthesized compounds. The FT-IR spectrum of as-prepared SnO₂ nanoparticles is shown in the Fig. 2.

Fig. 2 FT-IR spectrum of SnO₂ nanoparticles

The band at 3477 cm⁻¹ is appeared due to OH stretching vibrations of free and hydrogen-bonded hydroxyl groups [13]. The narrow peak at 616 cm⁻¹ confirms the formation of crystalline O-Sn-O mode of the tin oxide [16]. It confirms the presence of SnO₂ as crystalline phase. This is in a good agreement with the results of the XRD analysis. The peak at 1631 cm⁻¹ may be due to the adsorbed water and ammonia [14]. The intense and broad bands at 3433 cm⁻¹ and 1638 cm⁻¹ can be attributed to the O-H vibration in adsorbed water on the sample surface [15]. The broad bands between 450 and 790 cm⁻¹ are attributed to the framework vibrations of the Sn-O bond in SnO₂ [16]. It can be seen that, the absorption spectrum is dominated with very strong and broad absorption peak spread over the spectral region (3600–3000 cm⁻¹). This effect is thought to be due to adsorbed water vapor since these features are consistent with underlying (H-bonded) OH stretching bands. However, one can see weak shoulders riding over this broad peak. The aromatic C-H stretching vibration is easily identifiable above 3000 cm⁻¹ as a weak peak at 3141 cm⁻¹. The absorption band appeared at 3201 cm⁻¹ and a shoulder at approximately 3439 cm⁻¹ are assigned to hydrogen bonded symmetric and asymmetric N-H stretching vibrations.

3.3 UV-Vis Analysis

UV-Visible absorption spectral study may be assisted in understanding electronic structure of the optical band gap of the material. The room temperature ultraviolet-visible spectra of SnO₂ nanoparticles samples were recorded using UV-1800 series spectrophotometer in the absorption mode. The UV-absorption edge provides a reliable estimate of the band gap of any system. The optical absorption spectra of the SnO₂ nanoparticles are presented in Fig. 3. The absorption band edge is observed at 295 nm, which clearly indicates a blue shift from the bulk SnO₂ (340 nm) [17].

Fig. 3 UV spectra of SnO₂ nanoparticles

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3.4 SEM and HRTEM Analysis

The morphology and size of nanoparticles are analysed using a scanning electron microscope (SEM). The SEM images of as-prepared tin oxide are shown in Fig. 4. Also it is observed that the most of the particles of the samples are spherical shape of tin oxide with some agglomeration of finer particulates to form bigger clusters. The particles are fine and granular in nature and seemed to be nanosized, typically in the range of < 30 nm.

Fig. 4 SEM image of SnO₂ nanoparticles

The HRTEM images of as-prepared SnO₂ and are presented in Fig. 5. Also, it is observed that the shapes of most of the particles are nearly spherical and can be arranged in different ways. To obtain a particle size distribution from transmission electron micrographs we manually measured the particle sizes for 10 particles to ensure a reliable representation of the actual size distribution. The crystallite size is about 5-20 nm as estimated from the TEM micrographs. The experimental and calculated XRD patterns provide a volume-weighted average grain size of 16 nm, which is in good agreement with the particle size observable on the TEM image. The interplanar spacing of the SnO₂ nanoparticles is about 0.33 nm, which is identical with the (1 1 0) facet distance of bulk SnO₂ phase. From SEM and HRTEM images, the morphology of nanoparticles is observed to be spherical shaped particles with slight agglomeration.

Fig. 5 HRTEM image of SnO₂ nanoparticles

3.4 Antimicrobial Activity

The antimicrobial activity of SnO₂ was investigated against gram-negative bacteria (*Escherichia coli*) and gram-positive bacteria (*Staphylococcus aureus*), respectively. The prepared SnO₂ nanoparticles showed a good inhibitory action against *Escherichia coli* (inhibition zone size of 22 mm) at the 100 μg/mL compared to other bacteria due to the negatively charged, *Staphylococcus aureus* (inhibition zone size of 18 mm) cell wall readily attracting positively charged SnO₂, and in that way inhibiting microbial actions. In addition, the SnO₂ nanoparticle was directly proportional to their inhibitory actions beside the tested microorganism. Finally, confirmed prepared SnO₂ nanoparticles are more stable and have antimicrobial activity behavior.

4. Conclusion

SnO₂ nanoparticles powder was synthesized at 70 °C by microwave-assisted solution method. The XRD pattern exhibited peaks for nanocrystalline SnO₂ are observed and indexed to (1 1 0), (1 0 1), (2 0 0), (2 1 1), (3 1 0) and (3 0 1) having primitive tetragonal structure of SnO₂. From SEM image, it is observed that the most of the agglomerated particles of the samples are spherical shape of tin oxide. FTIR broad bands between 450 and 790 cm⁻¹ are attributed to the framework vibrations of the Sn-O bond in SnO₂. At room temperature ultraviolet-visible spectrum of SnO₂

nanoparticles were recorded using UV-1800 series spectrophotometer in the absorption mode and absorption band edge is observed at 295 nm with blue shift. A good antimicrobial activity of SnO₂ was found against gram-negative bacterium *Escherichia coli* and gram-positive bacterium *Staphylococcus aureus*, respectively, and thus the synthesized SnO₂ nanoparticles have confirmed that they have possible biomedical applications.

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