Microwave-assisted precipitation synthesis of nanostructured SnO_2 particles: Studies on Structural, Morphological and Antimicrobial activities

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Abstract

Ultrafine tetragonal rutile phase tin oxide (SnO_2) nanoparticles (called as TONP's) are synthesized by microwave-assisted precipitation route using various solvents like water-methanol, water-ethanol, water-propanol and water. The structural, size and morphological features are studied using powder X-ray diffraction (XRD) and transmission electron microscope (TEM) analysis. The crystalline nature, crystallite size (~5-15 nm) and phase purity of the TONP's are observed from the powder XRD analysis. The elongated spherical shaped particles with size range of 5 nm are observed from the TEM analysis. The antimicrobial activities of the synthesized TONP's are examined using the selected gram negative bacteria, gram positive bacteria and fungi.

Keywords: Metal oxide, Nanoparticles, Microwave-Precipitation process, Structural properties, Antimicrobial activities

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Introduction

In recent years, semiconductor metal oxide nanoparticles like tin oxide (SnO_2) have received much attention in the industrial and technological point of view. Tin oxide nanoparticles (TONP's) are widely useful in catalysis, gas sensing devices, Li-ion batteries, dye-sensitized solar cells, optoelectronic devices and antimicrobial activities [1-4]. The size-dependent features of TONP's are important in modifying structural, morphological, and optical aspects. Many preparation routes are used to modify the properties of TONP's, paving the path for a various applications. Kumar et al. [5] examined the effect of solvent on nanostructured SnO_2 particles. Liu et al. [6] investigated the impact of various solvent ratios on the morphological and electrochemical properties of SnO_2 nanocrystals. Virender et al. [7] reported the crystallographic, morphological and optical features of nanostructured SnO_2 particles under the impact of different solvents effect. Chen et al. [8] studied single crystal SnO_2 nanorods via hydrothermal route. Haixia Xie et al. [9] used a solvothermal technique to create tin oxide nanoparticles by and then tested them in solar cells applications. Krishnakumar et al. [10, 11] used a microwave-assisted method with an operating frequency of 2.45 GHz to study tin oxide nanoparticles. Ashok K. Singh et al. [12] used microwave process to investigate the photocatalytic properties of nanostructured SnO_2 particles.

The microwave-assisted precipitation technique is used in this work for the mixture solvents assisted synthesis of tin oxide nanoparticles. The influence of various solvents on structural, size and morphological are examined using powder XRD and TEM analysis. The synthesized tin oxide sample is also used to examine the antimicrobial activity (Gram Positive, Gram Negative and Fungi) of selected pathogens such as *Bacillus subtilis*, *Staphylococcus aureus*, *Pseudomonas aeruginosa*, *Salmonella enteric* and *Candida albicans.*

2. Experimental procedure

2.25 g of tin chloride dihydrate ($SnCl_2.2H_2O$) was dissolved in 100ml distilled water at room temperature with magnetic stirring. The aforesaid mixed solution was then treated with 0.8 g caustic soda pellets, resulting in a milky white solution. The prepared mixture was then maintained in microwave oven for 7 minutes at 800 watts. The combinations were then washed with water as a solvent and heat-treated for 24 hours at 150 °C. The pale yellow SnO_2 nanopowders were generated after the intermediate products were calcined at 450 °C for 2 hrs. The schematic diagram of synthesis of TONP's is illustrated in Fig.1.

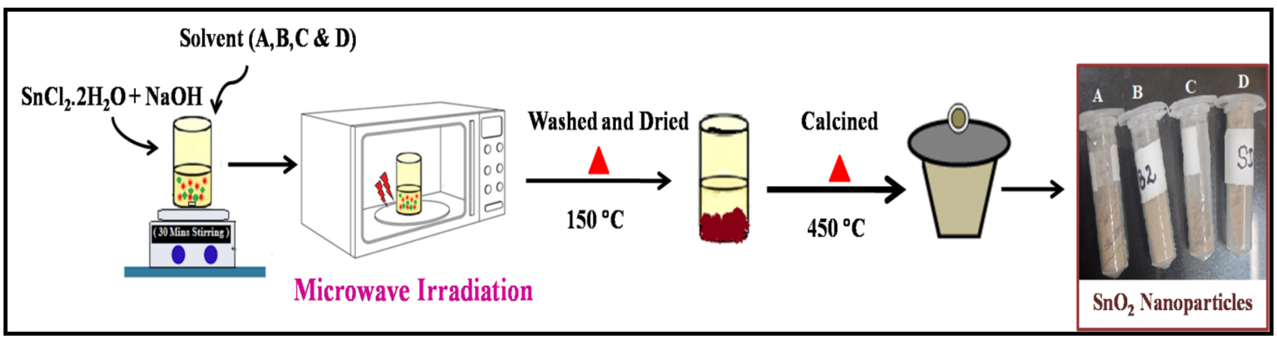

Fig. 1 Scheme of microwave-assisted synthesis of nanostructured SnO_2 nanoparticles.

TONP's samples are submitted to structural and morphological characterization tests such as powder XRD and TEM analysis. Powder XRD analysis of the prepared TONP's samples is performed with a Model-SmartLab SE X -Ray; make-Rigaku, Japan. HRTEM JEOL Japan, JEM-2100 Plus is used to examine the TEM images of the synthesized TONP's samples. TONP's compound was tested for in vitro antimicrobial activity against five human pathogens: Bacillus subtilis (Gram positive), Staphylococcus aureus (Gram positive), Pseudomonas aeruginosa (Gram

Negative), Salmonella enterica (Gram Negative), and Candida albicans (Fungal) using a well diffusion method, as detailed in the Supporting Information.

3. Results and discussion

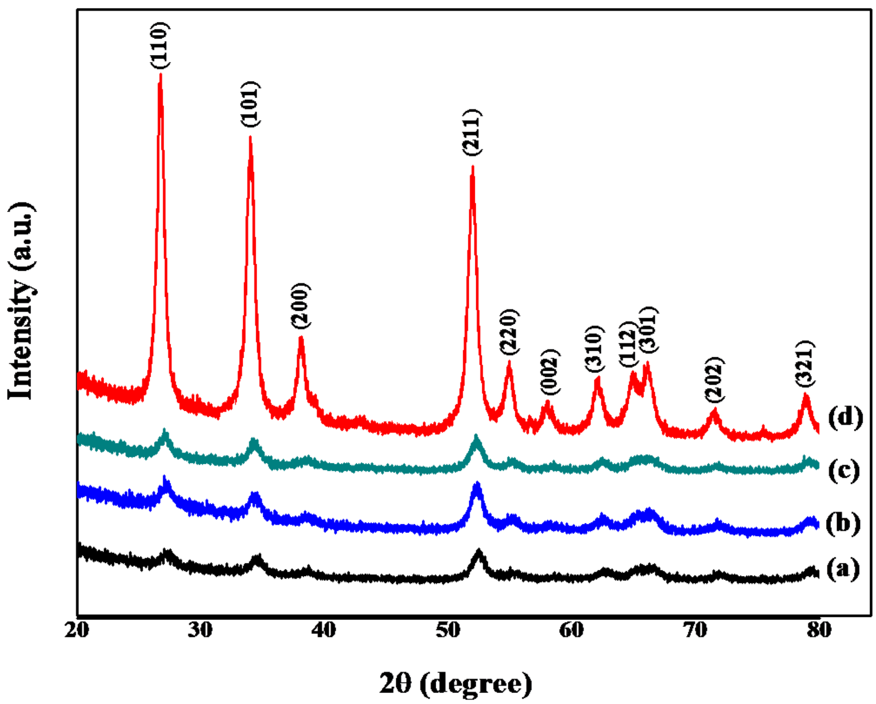

Fig. 2 Powder XRD patterns of (a) water-methanol (c) water-ethanol (c) water-propanol and (d) water-assisted TONP’s samples.

The powder XRD pattern of various solvents-assisted TONP’s samples is displayed in Fig.2. It demonstrated that TONP’s had a well-crystallized tetragonal rutile phase. The XRD pattern revealed no additional phases or impurity peaks. The micro-wave aided precipitation method was discovered to play an important function in the pure phase of TONP’s samples. Because of their higher crystallinity and larger crystallite size, water assisted TONP’s samples (Fig. 2d) exhibited more strong peaks than other mixture solvents-mediated samples. Peaks are found in the diffraction planes (110), (101), (200), (211), (220), (002), (310), (112), (301), (202), and (321), indicating the creation of a crystalline structure of SnO_2 (JCPDS 00-072-1147). The average crystallite size of the water-methanol, water-ethanol, water-propanol, and water-assisted TONP's is computed using the Debye-Scherrer's equation to be 5.2, 6.6, 7.8, and 15.7 nm, respectively.

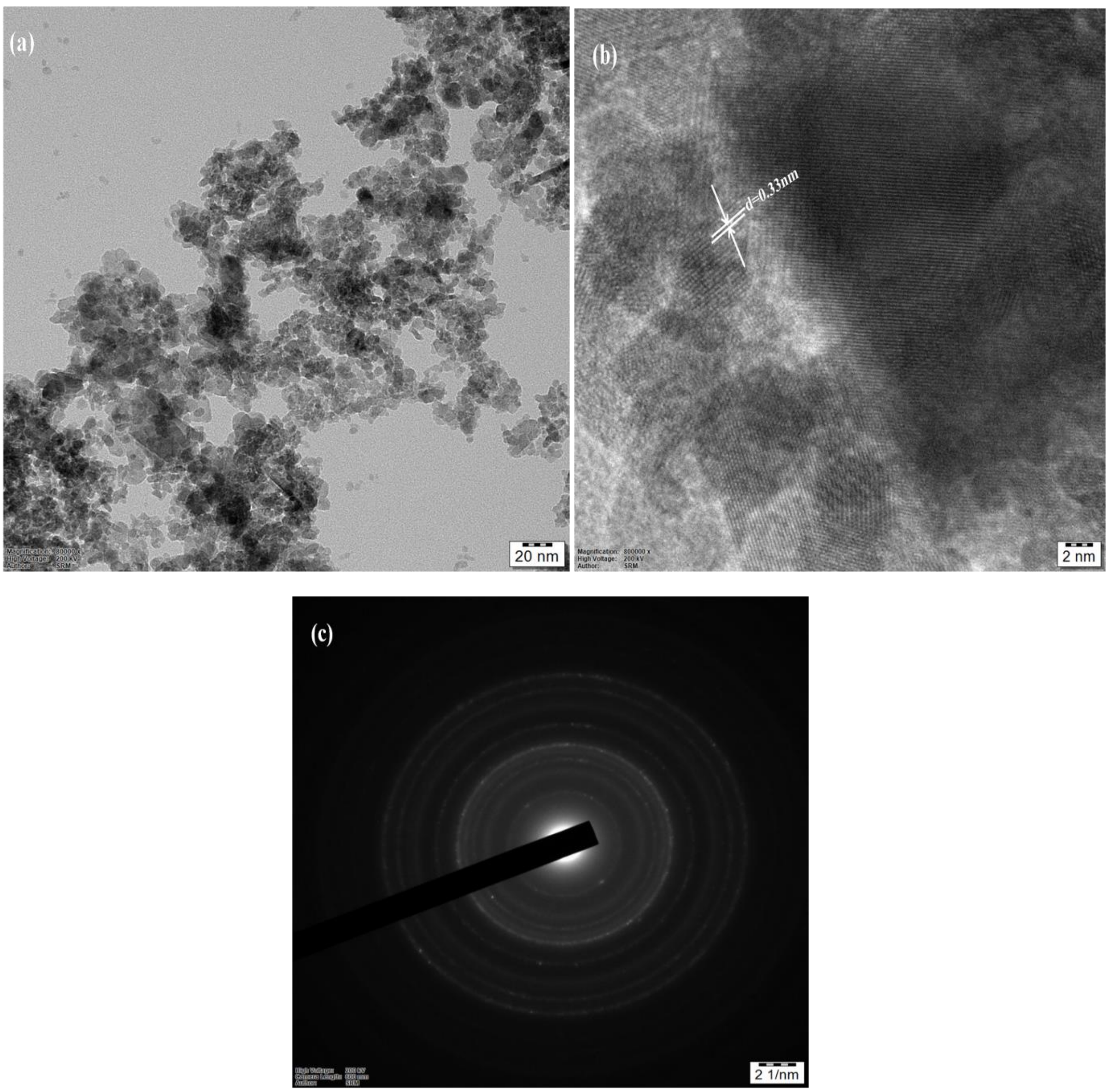

Fig.3 (a) TEM image (b) HR-TEM image and (c) SAED pattern of the water-methanol assisted TONP's samples.

Using Debye-Scherrer's equation, the powder XRD pattern revealed that among the solvent-assisted TONP's samples, the water-methanol assisted TONP's sample had the smallest crystallite size. As a result, it is subjected to TEM analysis in order to examine the morphological features of the TONP's sample. Figure 3(a) shows a TEM image of a water-methanol solvent mediated TONP's sample. It demonstrated that the elongated spherical-like smaller particles in the ~5 nm range are well-supported by XRD patterns. The clear lattice fringes in Fig. 3(b) indicate the crystalline characteristic of the TONP's. The distance between the lattice fringes was calculated as 0.33 nm, which is supported with the lattice spacing of the (110) plane in the TONP's sample. The polycrystalline nature of the synthesized TONP's sample was demonstrated by the obvious rings of the SAED pattern (Fig. 3c). The broadening of the diffraction rings indicates that the particles are small and have a high degree of crystallinity.

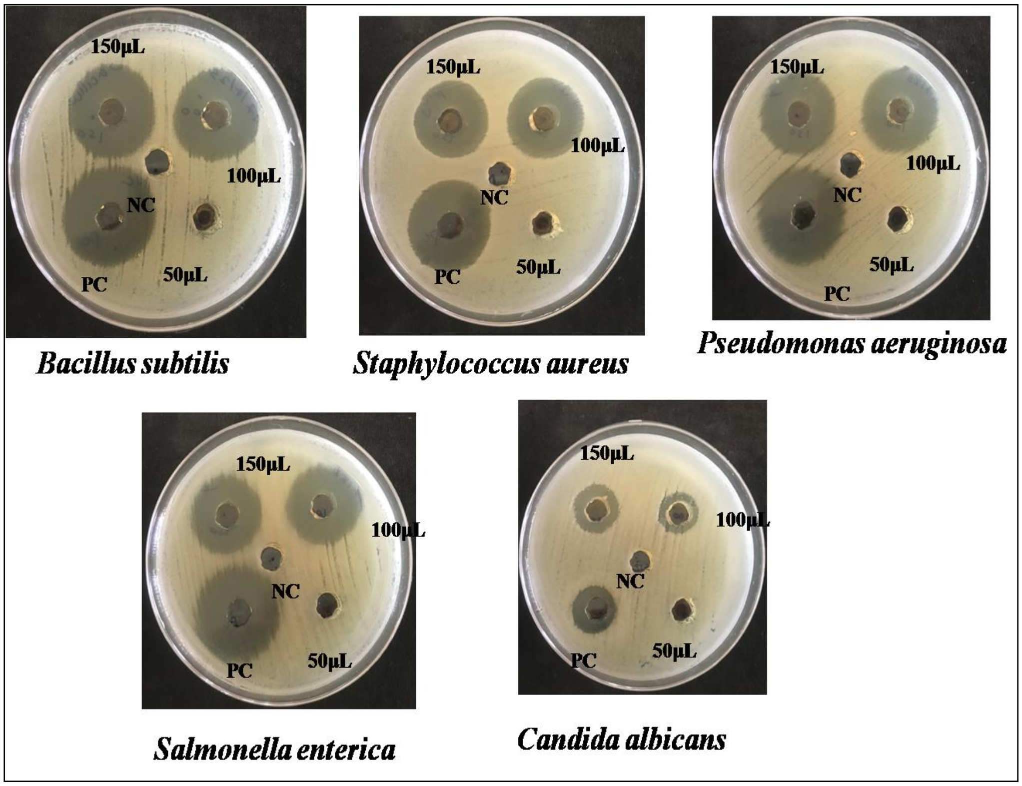

Fig. 4 Antimicrobial activities of the water-methanol assisted TONP's samples.

Fig. 4 displays the antimicrobial activities of TONP's samples. The prepared TONP's samples performed well in the well-diffusion assay. It demonstrated maximum activity against all five pathogens (Gram Positive, Gram Negative and Fungi) up to 29 mm. The concentration of 150 µl in the concentration of 150 g yielded the highest concentration. It performed better activity against gram positive and gram negative similar to the positive control i.e. streptomycin used for the study. Ketaconzole is used as a positive control for Fungi. Compound had the lowest activity against Candida albicans at a concentration of 150 µl. 10% DMSO was used as a negative control and had no effect on the human pathogens. According to the findings, the prepared TONP's samples had better antimicrobial activities. Table 1 summarizes the antimicrobial activities of TONP's samples against selected pathogens and their results.

Table 1: Antimicrobial results of TONP's samples.

S. NO	HUMAN PATHOGENS	SnO_2 (1mg/ml)			
		50 µl	100 µl	150 µl	PC
1	*Bacillus subtilis*	-	25mm	27mm	29mm
2	*Staphylococcus aureus*	-	29mm	29mm	29mm
3	*Pseudomonas aeruginosa*	-	23mm	25mm	30mm
4	*Salmonella enterica*	-	24mm	25mm	31mm
5	*Candida albicans*	-	13mm	15mm	17mm

*No activity was found in 10 % DMSO as negative control

4. Conclusions

In this work, the solvents mediated TONP's samples were successfully synthesized using a microwave-assisted precipitation method in this study. Powder XRD and TEM analyses were used to assess structural, size, and morphological aspects. Powder XRD investigation validated the tetragonal rutile phase crystal structure. The TEM investigation revealed elongated spherical-shaped TONP's with an average size range ~ 5 nm. The polycrystalline behaviour of the synthesized TONP's sample was evident in the SAED pattern. Antimicrobial investigations on the TONP's sample yielded positive results for selected pathogens such as Bacillus subtilis, Staphylococcus aureus, Pseudomonas aeruginosa, Salmonella enterica, and Candida albicans. Hence, TONP's could be a good antibacterial agent to develop against multidrug-resistant bacterium strains.

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