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Solochemical synthesis and characterization of ZnO nanostructures with different morphologies and their antibacterial activity

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ABSTRACT

Zinc oxide nanostructures were synthesized by solochemical method using different precursors. ZnO nanostructures were characterized by X-ray diffraction, Scanning electron microscopy, Transmission electron microscopy and UV – vis diffuse reflectance/absorption spectroscopy. The X-ray diffraction patterns of different ZnO nanostructures suggest that the synthesized structures have hexagonal *wurtzite* structure of zinc oxide. Scanning electron microscopic and Transmission electron microscopic images revealed the different shapes and sizes of the zinc oxide nanostructures. The UV – vis absorption spectra of the zinc oxide nanostructures show blue-shift in wavelength corresponding to bulk. Band gap energies of zinc oxide nanostructures are determined by UV – vis diffuse reflectance spectra. The zinc oxide nanostructures were evaluated for their antibacterial activity against Gram-positive and Gram-negative bacteria. Gram-negative bacteria seemed to be more resistant to ZnO nanostructures than Gram-positive bacteria.

1. Introduction

Nanoscale semiconducting materials represent a new class of important materials that is increasingly being developed for use in research and health-related applications. Among many nanoscale semiconducting materials, zinc oxide (ZnO) is a typical *n*-type inorganic semiconductor that displays a hexagonal crystalline *wurtzite*-type structure, with space group $P6_3mc$ and lattice parameters of $a = b = 0.3250$ nm and $c = 0.5207$ nm [1]. The importance of ZnO is due to its high conductance, good mechanical and thermal stability, wide and direct band gap of 3.37 eV and a large exciton binding energy of 60 MeV [2–4]. In addition, it has good radiation resistance and is harmless to the environment [5,6]. It is widely applied in the field of optoelectronics, pharmaceutics, cosmetics, food science and agriculture [7–14]. The antibacterial activity of zinc oxide has been widely explored. It has been documented that concentration, size and healing temperature can affect the antibacterial activity [15]. Zinc oxide as an inorganic antibacterial reagent is more stable than the organic reagents [16]. ZnO nanoparticles have been shown to be useful antibacterial and antifungal agents when used as a surface coating on materials and textiles [17]. Zinc is an essential element and ZnO nanoparticles are considered to be non-toxic. Toxicity studies have shown that zinc ions do not cause any damage to the DNA of human cells [18]. From the literature survey, it is found that various approaches for the preparation of ZnO nanopowders have been developed, namely, microemulsion, thermal decomposition of organic precursor, spray pyrolysis, electrodeposition, ultrasonic, microwave-assisted technique, chemical vapour deposition, hydrothermal, sol – gel, solochemical and precipitation method. Among the techniques employed, those belonging to the chemical routes are suitable for the preparation of ZnO nanostructures in industrial scale since are relatively cheap and provide a high uniformity of the final product [19–21].

Zinc oxide nanopowder production by solochemical (SC) processing method involves preparation of a solution containing zinc complex and subsequent decomposition of the complex into the zinc oxide nanopowder. Another name for this method is two-stage solochemical (TSSC) method. TSSC method can also be used for production of other oxides such as Mn_2O_3 and NiO. Pouring of a limpid chemical (containing zinc complex) onto a second chemical leads to the formation of a nanoscale powder. Due to the simplicity, versatility and low cost of this route, the solochemical method is a process extremely viable for industrial production of zinc oxide [22].

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In this study, ZnO nanostructures were synthesized in short reaction time and in relatively low temperature by the solochemical method using different precursors zinc nitrate tetrahydrate and zinc acetate dihydrate without any posterior treatments. The structural and phase formation of the samples were identified by X-ray diffraction (XRD) technique. The morphology and size of the ZnO nanostructures were evaluated by scanning electron microscopy (SEM) and transmission electron microscopy (TEM). The optical properties were confirmed by UV – vis diffuse reflectance/absorption spectroscopy. The antibacterial activity of ZnO nanostructures towards *Staphylococcus aureus, Escherichia coli* and *Enterobacter aerogenes* was tested by an agar disc diffusion assay.

2. Experimental

2.1. Materials

The source materials, such as, zinc nitrate tetrahydrate [$(Zn(NO_3)_2{\cdot}4H_2O$], zinc acetate dihydrate [$Zn(CH_3COO)_2{\cdot}2H_2O$], sodium hydroxide (NaOH), potassium hydroxide (KOH) and ammonia solution (25% NH_4OH) were of analytical grade and used as purchased. Solutions were prepared by dissolving appropriate amount of the compounds in double distilled water.

2.2. Synthesis of ZnO nanostructures

2.2.1. Synthesis of cauliflower-like ZnO nanostructures (Sample code: A)

In this method 0.45 M aqueous solution of zinc nitrate tetrahydrate and 1.0 M aqueous solution of sodium hydroxide (NaOH) were prepared in distilled water. Then the beaker containing NaOH solution was heated at the temperature of about 60 °C. The zinc nitrate tetrahydrate solution was added dropwise for 45 minutes to the above heated solution under high speed stirring. The beaker was sealed at this condition for 2 h. The precipitate obtained was cleaned with deionized water and ethanol and then dried in air atmosphere at 60^0C.

2.2.2. Synthesis of ZnO nanoflowers (Sample code: B)

In this method 6 mL of NH_4OH was first dissolved in 94 mL of distilled water in a 600 mL conical flask. Simultaneously, 0.2 M zinc acetate dihydrate was dissolved in 100 mL of distilled water and then added dropwise into NH_4OH solution. White precipitate was formed immediately and it was heated at 70 °C for 1 h. The precipitate was filtered after being cooled and then rinsed with distilled water and absolute ethanol. After that it was dried in air atmosphere at 60 °C.

2.2.3. Synthesis of hexagonal ZnO nanostructures (Sample code: C)

In this method 1.0 M aqueous solution of NaOH was added dropwise into 0.3 M aqueous solution of zinc acetate dihydrate under continuous stirring. The solution was then heated for 30 min at 80 °C. The resulting white precipitate was washed with methanol several times and dried at room temperature.

2.2.4. Synthesis of spherical ZnO nanostructures (Sample code: D)

In this method 0.5 M aqueous solution of zinc nitrate tetrahydrate and 1.0 M aqueous solution of potassium hydroxide (KOH) were prepared in distilled water. The reaction solution was heated at 50 °C under continuous stirring for 1 h. The precipitate formed was collected and washed with ethanol and dried at room temperature.

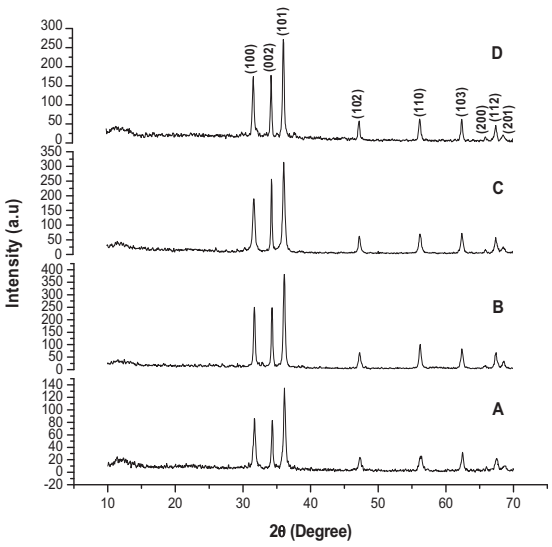

Fig. 1. XRD patterns of ZnO nanostructures.

2.3. Characterization of ZnO nanostructures

The structural and phase formation of the samples were identified by Reich Seifert XRD 3000 diffractometer using Cu-Kα (λ = 1.5406 Å). The morphology and size of the ZnO nanoparticles were evaluated by scanning electron microscopy (SEM, FEI-Quanta 250) and transmission electronic microscopy (TEM, FEI-Technai Sprit). UV – vis measurements were made by Lamda 650 UV – vis diffuse reflectance spectrometer (PerkinElmer).

2.4. Test of antibacterial activity

Antibacterial tests were performed by an agar disc diffusion assay using a Gram positive bacteria *S. aureus* and Gram-negative bacteria *E. coli* and *E. aerogenes* as indicator strains. The three species of bacteria grown individually on nutrient agar plates at 37 °C for 16 – 18 h were resuspended in 0.85% sterile saline equivalent to a turbidity of standard McFarland No. 0.5 with approximate 1.5×10^8 colony forming units/mL (CFU/mL). The suspension of testing bacteria was spread on the Mueller – Hinton agar plate and left at room temperature for 5 minutes so as to allow the moisture from the inocula to absorb into the medium. Sterile disc of 6 mm dia were prepared by pipetting various concentration of (250, 500, 750 and 1000 μg/mL) ZnO samples A, B, C and D to each disc and placed on the plate. The agar plates were left at room temperature for 15 minutes and then incubated at 37 °C for 16 h. The clear halo zone surrounding each disc on cultivated agar plate was measured and the zone of inhibition was expressed in mm. The antibacterial activity of the ZnO samples was compared to that of the standard antibiotic Streptomycin, which is a positive control.

3. Results and discussions

3.1. Structural characterization of ZnO samples

The XRD patterns of the synthesized ZnO samples are shown in Fig. 1. The XRD patterns of the samples reveal that all peaks correspond to the characteristic peaks of the hexagonal *wurtzite* structure of ZnO with space group $P6_3mc$ and lattice parameters of $a = b = 0.3250$ nm and $c = 0.5207$ nm according to the JCPDS database 36-1451. Based on the XRD results the average crystallite size of ZnO samples is determined by Debye – Scherrer's formula [23] ($D = k\lambda/\beta\cos\theta$, where D is the average crystallite size, k is a