ROLE OF SALT PRECURSOR IN THE SYNTHESIS OF ZINC OXIDE NANOPARTICLES

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Abstract

In this paper, Zinc oxide nanoparticles having wurtzite crystalline structure are synthesized. The temperature, base concentration and the salt precursor used for the synthesis affects the morphology and particle size. The synthesized nanoparticles are characterized by X-ray diffraction, Scanning electron microscopy and Diffused reflectance UV-visible spectroscopy. As the temperature is increased from 80^0 C to 100^0C, keeping the concentration of the base viz sodium hydroxide at 5M, the particle size increases from 30nm to 500nm. With change in base concentration from 2M to 10M, at constant reaction temperature of 80^0C, the particle size increases from 30 nm to 500 nm. Herein the precursor used is zinc chloride. The effect of salt precursor is studied for three different salt precursors, viz. zinc chloride, zinc nitrate and zinc acetate.

This paper is an attempt to give the information about salt precursor to be used, optimum values of temperature and base concentration for synthesis of the ZnO nanoparticles with enhanced antibacterial property for suitable biomedical application.

Keywords*: Zinc Oxide nanoparticles, metal oxide nanoparticles, nanotechnology.*

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1. INTRODUCTION

The nanoparticles possess unique physicochemical, optical and biological properties which can be manipulated suitably for desired applications [1]. Moreover the biological processes also occur at the nanoscale and due to their amenability to nanoparticles functionalization, the nanoparticles are finding important applications in the field of medicine [2]. Metal and metal oxide nanoparticles when embedded and coated on to surfaces can find immense applications in water treatment, synthetic textiles, biomedical and surgical devices, food processing and packaging [3]. Chemical synthesis is the one of the important technique for the synthesis of nanoparticles. It can be performed using range of precursors, temperature, time, concentration of reactants etc [4]. Physicochemical properties play a vital role in metal oxide antimicrobial activity, including particle aggregation, crystal phase and surface modification [5].

The use of metal and metal-oxide nanoparticles for antimicrobial and antifungal applications is being widely studied. The use of silver nanoparticles for their antimicrobial activity is being put to use in commercial applications. The metal oxides like ZnO and MgO are also being explored for their microbial inhibition in order to have cost effective systems. Our earlier study on the antimicrobial activity of ZnO nanoparticles showed its efficacy versus several pathogens [6]. In the present paper antimicrobial activity of the zinc oxide nanoparticles and its dependence on the morphology of nanoparticles synthesized using different precursors is studied.

2. EXPERIMENTAL

Zinc oxide nanoparticles are synthesized using different salt precursors.

2.1 Synthesis of Zinc Oxide Nanoflowers Using Zinc Nitrate

The production unit of ZnO nanostructures consists basically of a jacketed three-neck glass flask and of a magnetic stirrer with temperature control. In the three-neck glass flask, NaOH was dissolved in deionized water to a concentration of 1.0 M and the resulting solution was heated, under constant stirring, to the temperature of 70°C. After achieving this temperature, a solution of 0.5 M $Zn(NO_3)_2.6H_2O$ was added slowly (dripped for 60 minutes) into the three-neck glass flask containing the NaOH aqueous solution under continual stirring. In this procedure the reaction temperature was constantly maintained in 70°C. The suspension formed with the dropping of 0.5 M $Zn(NO_3)_2.6H_2O$ solution to the alkaline aqueous solution was kept stirred for two hours in the temperature of 70°C. The material formed was filtered and washed several times with deionized water. The washed sample was dried at 65°C in oven for several hours [7].

2.2 Synthesis of Zinc Oxide Rice like Nanostructures Using Zinc Chloride

5.5 gms of zinc chloride was dissolved in 100 ml of distilled water in a beaker. This solution was kept under constant magnetic stirring till zinc chloride totally dissolved in distilled water. The temperature of the beaker was raised to 90^0C by electric hot plate heating. Meanwhile 2 gms of NaOH was dissolved in 100 ml of distilled water in a separate vessel. From the prepared sodium hydroxide solution 16 ml of sodium hydroxide is added to the beaker with constant stirring touching the walls of the beaker. The aqueous solution turned into a milky white colloid without any precipitation. The reaction was allowed to proceed for two hours after complete addition of sodium hydroxide. After the complete reaction, solution was allowed to settle and the supernatant was removed by washing with distilled water five times. After washing, the powder is allowed to dry in oven at 100^0 C for 30 minutes [8]. The resulting powder showed rice-like structures.

2.3 Synthesis of Zinc Oxide Nanoparticles Using Zinc Acetate

Sample 3 is synthesized using zinc acetate as the precursor. Zinc acetate is kept in muffle furnace for 12 hours at 400^0C following the technique by S K Mishra et al [9]. Spherical zinc oxide nanoparticles are formed in this case.

2.4 Antimicrobial Activity

The cultures used are standard strains of *S.aureus, E. coli.* The antibacterial activity was determined by the disc diffusion method wherein 10mm paper discs impregnated with ZnO nanoparticles were placed on Mueller Hinton agar plates spread with the culture and testing the formation of inhibition zone compared to the controls [6].

3. RESULTS AND DISCUSSION

X-ray diffraction (XRD) was carried out on a XPERT-PRO X-ray diffractometer with Cu Kα radiation (λ =1.54060 nm) (applied voltage 45 kV, current 40 mA) at a scanning rate of $0.05^0 s^{-1}$in the 2θ range from 20^0 to 80^0. About 0.5 g of the dried particles were deposited randomly onto a plexi-glass sample container. The crystal structure and orientation of ZnO nanoparticles have been investigated by X ray diffraction method using the Panalytical Xpert High Score Plus Software.

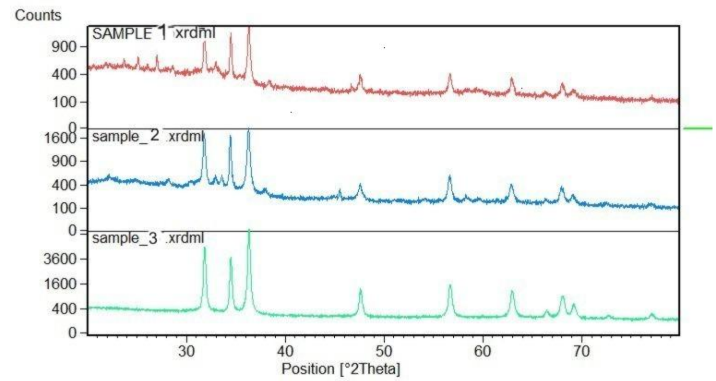

Fig 1: XRD plot of ZnO sample 1, 2 and 3 (different precursor)

All the peaks for the three graphs match with the standard plot. XRD spectrum of ZnO nanocrystals using the three precursors, labeled as sample 1,2 and3, using a Cu-Kα radiation source is shown in Fig. 1.

The average size of the ZnO particle is calculated using Debye scherer formula,

$$d_{avg} = 0.9\lambda / \beta \cos\theta.$$

Where
d_{avg} = Average crystal size,λ= Wavelength of incident beam (1.5406Å),
β= FWHM in radians, θ= Scattering angle in degree

The average crystallite size of the samples 1, 2 and 3 is found to be 36.06nm, 38.90nm and 39.91nm respectively.

Energy Dispersive X ray spectroscopic data gives the corresponding peaks of Zn and O in all the samples. In sample 1 some copper impurity is obtained while in sample 2 some chlorine impurity is found. But the sample 3 made from Zinc acetate precursor shows peaks corresponding to Zn and O only. This implies that sample 3 is extremely pure.

The fig 2 shows the SEM images for the samples 1,2 and 3. The particle size calculated using SEM data and XRD data is approximately equal.

The antibacterial activity tests are carried out using a given nanoparticle formulation versus a set of organisms and varying the concentration of the nanoparticles to determine the minimum inhibitory concentration. The results are to be performed in triplicate versus control. Preliminary investigations revealed that inhibitory activity was observed in case of ZnO nanoparticles obtained from Zinc acetate precursors while in case of the nanoparticles obtained from Zinc chloride precursors, the minimum inhibitory concentration was found to be very high. The tests are to be carried out selecting one set of nanoparticles of approximately same size and varying morphology against a given micro-organism.

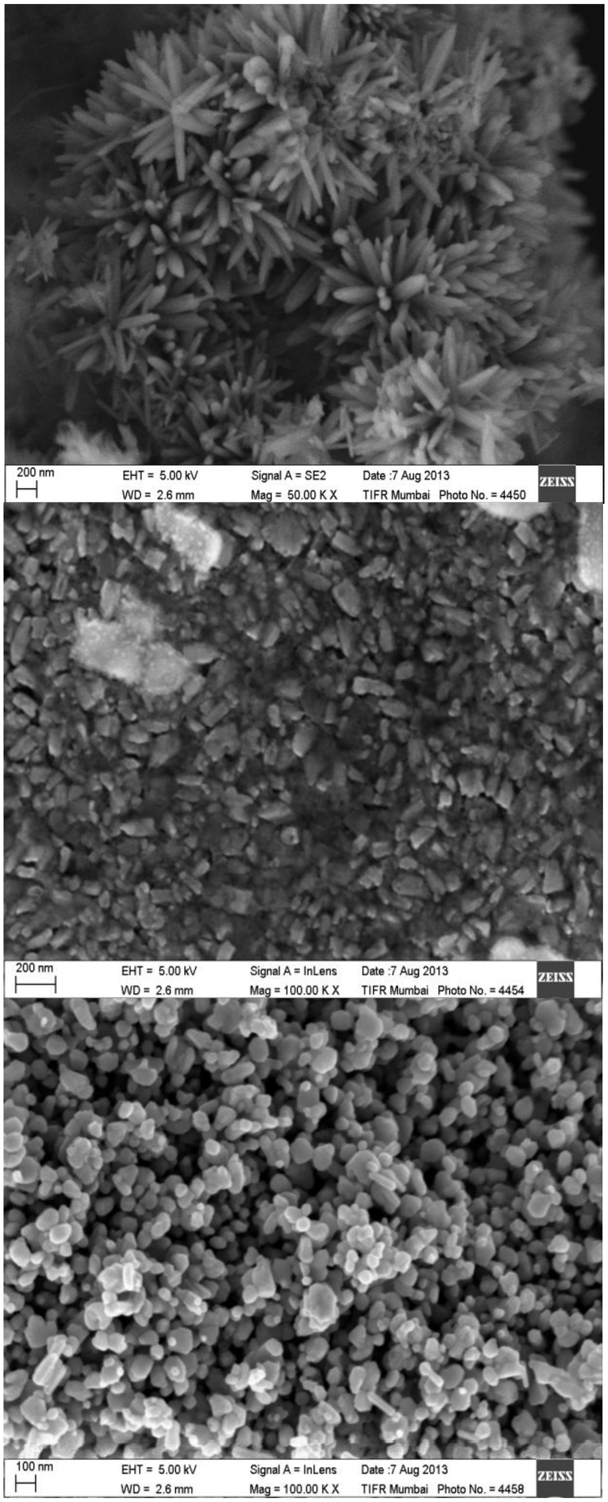

Fig 2: SEM images of the samples 1,2 and 3

4. CONCLUSIONS

Zinc oxide nanoparticles are synthesized using different precursors and are characaterized by X ray diffraction, scanning electron microscopy and Energy dispersive X ray spectroscopy. Different reaction temperatures and concentration values are tried so as to synthesize the nanoparticles of different shapes and having same crystallite size approximately 38nm. The use of different precursor leads to change in the particle shape. The change in antibacterial efficacy of the three samples is expected to throw light on the relation of morphology and the reaction parameters on their bioactivity.

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REFERENCES

[1]. Feynman R., There's plenty of room at the bottom. Science 1991; 254:1300-1301.
[2]. Parak W.J, Gerion D, Pellegrino T, Zanchet D, Micheel C, Williams CS, Boudreau R, Le Gros MA, Larabell CA, Alivisatos AP: Biological applications of colloidal nanocrystals. Nanotechnology. 2003;14:15-27
[3]. Gutierrez FM, Olive PL, Banuelos A, Orrantia E, Nino N, Sanchez EM, Ruiz F, Bach H, Gay YA., Synthesis, characterization, and evaluation of antimicrobial and cytotoxic effect of silver and titanium nanoparticles. Nanomedicine. 2010;6:681-688.
[4]. K. Renugadevi*, P. Raji and M. Bavanilatha, In-vitro evaluation of antibacterial activity and cytotoxicity effect of chemically synthesized ZnO nanoparticles, IJPSR (2012), Vol. 3, Issue 08
[5]. A. Mukherjee, Mohammed Sadiq I., Prathna T.C., N. Chandrasekaran, Antimicrobial activity of aluminium oxide nanoparticles for potential clinical applications, Science against microbial pathogens: communicating current research and technological advances, 2011, Vol. 1, pp.245-25
[6]. A.Surti, S. Radha and S.S. Garje, Study of the Antibacterial activity of ZnO nanoparticles, AIP Conf. Proc. 1512, 450 (2013)
[7]. http://www.aidic.it/icheap9/webpapers/286Gusatti.pdf
[8]Parthsarathi V.; Thilagawathi G.; Synthesis and characterization zinc oxide nanoparticle and its application on fabrics for microbe resistant defence clothing, Int. J. Pharm. Sci. 2011, 3,392-398.
[9]. Sheo K Mishra, Rajneesh K Srivastava, S G Prakash, ZnO nanoparticles: Structural, optical and photoconductivity characteristics, 2012, Journal of Alloys and Compounds, 539:1-6