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To Study the Role of Temperature and Sodium Hydroxide Concentration in the Synthesis of Zinc Oxide Nanoparticles

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Abstract- In this work, zinc oxide (ZnO) nanostructures were synthesized by using zinc chloride and sodium hydroxide as the precursors at different temperatures. The ZnO nanostructures obtained were characterized by X-ray diffraction (XRD), Scanning Electronic Microscopy (SEM), Energy Dispersive X ray Spectroscopy (EDAX).The ZnO powders have hexagonal wurtzite structure and nanometric-sized crystallites. The size of the particle increases as the reaction temperature is increased. An attempt has been made to find out the effect of temperature on the morphology as well as particle size and to optimize the value of temperature for stable smaller sized particles.Once the temperature is optimised effect of base concentration is studied. The base concentration is varied and different samples are synthesized for various base concentrations keeping the temperature constant. The basic idea is to design materials with the proper morphology, size, shape, and texture for desired application. The size of the particle is found to be directly related to ionic strength. Increasing the base concentration increases the surface charge density and hence reduces the interfacial tension. This paper is an attempt to optimize the base concentration for desired particle size and shape to render it suitable for Biomedical application.

Index Terms- Nanotechnology, Base concentration, Scanning Electron Microscopy Zinc oxide nanoparticles

I. INTRODUCTION

Zinc Oxide nanostructures (ZnO) are interesting to study not only because of their unique physical properties but also a wide variety of morphologies. Some of the ZnO nanostructures exhibit nanowire [1], nanorod [2], nanocomb [3], nanoflowers and nanosheet [4] like structures. The multiplicity of morphologies is the speciality of ZnO nanosystems that forms the basis of its versatile applications [5]. Several chemical methods like sol-gel, micro emulsion, hydrothermal, self assembly, homogenous, microwave assisted hydrothermal and direct precipitation have been reported for synthesis of ZnO nanostructures[6]. Zinc oxide (ZnO) is an n-type semiconductor displays a hexagonal crystalline wurtzite type structure, with space group P63mc and lattice parameters of a = b = 0.3250 nm and c = 0.5207 nm. ZnO has long been recognized as having inhibitory effect on the microbes present in the Medical and Industrial process. They were found to be highly toxic against different multidrug resistant human pathogens. In our earlier work we have studied the antibacterial property of these ZnO nanoparticles against Escherichia coli and Staphylococcus aureus[7]. ZnO nanoparticles have good biocompatibility to human cell [8]. ZnO has long been recognized as having inhibitory effect on the microbes present in the Medical and Industrial process. They were found to be highly toxic against different multidrug resistant human pathogens. ZnO nanoparticles have good biocompatibility to human cell.

Currently ZnO is documented as safe material by FDA (Food and Drug Administration, USA)[9]. In this paper an attempt has been made to standardize the method for suitable temperature and base concentration so that smaller particle size can be obtained as smaller particle sized nanoparticles show enhanced antibacterial activity. Although antibacterial activity of ZnO nanoparticles is investigated, the mechanism of the antibacterial activity of ZnO nanoparticles is not well understood. There are several methods which have been proposed to explain the antibacterial activity of ZnO nanoparticles. The generation of hydrogen peroxide from the surface of ZnO nanoparticles is considered as an effective mean for the inhibition of bacterial growth. It is presumed that with decreasing particle size the number of ZnO powder particles per unit volume of the powder slurry increases resulting in increased surface area and increased generation of hydrogen peroxide. Another possible mechanism for ZnO antibacterial activity is the release of Zn^{+2} ions which can damage the cell membrane and interact with intracellular contents[10].

In our earlier work we have studied the antibacterial property of zinc oxide nanoparticles synthesized using zinc acetate precursor and its application in bandages has been studied [11]. This paper is an attempt to optimize the value of base concentration for suitable particle size and morphology.

II. EXPERIMENTAL

Zinc chloride is dissolved in 100 ml of distilled water. It is stirred continuously with magnetic stirrer and its temperature is raised to 80^0 C. Once the temperature of zinc chloride solution is reached to 80^0 C, add 5M NaOH solution drop by drop touching the walls of container. 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 solution was removed by washing with distilled water five times. After washing, the precipitate is allowed to dry in oven at 100^0 C for 2 hours [12]. Five samples sample 1,2,3,4 and 5 are synthesized using the above method by changing the temperature as 80^0C, 85^0C, 90^0C, 95^0C and 100^0C. For lowest

temperature particle size is lowest so the temperature is optimized and it is 80^0C. In the next part the temperature is kept constant at 80^0C while the concentration of sodium hydroxide is varied as 2M, 4M, 6M, 8M, 10M and again five samples, sample 6, 7, 8, 9 and 10 are synthesized.

III. RESULTS AND DISCUSSION

A. *X ray Diffraction Spectroscopy*

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 0.05^0s^{-1} in the 2θ range from 20^0 to 80^0. About 0.5 g of the dried particles was deposited as a randomly oriented powder onto a plexi glass sample container. The crystal structure and orientation of ZnO nanoparticles have been investigated by x-ray diffraction method using Panalytical Xpert Pro MPD using the software Panalytical Xpert High Score Plus.

The crystallite size of the prepared nanopowder can be calculated using Debye Scherer formula,

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

Where;

d_{avg} = Average crystal size,

λ= Wavelength of incident beam (1.5406Å),

β= FWHM in radians,

θ= Scattering angle in degree.

The lattice parameters a and c are determined using the formula

$1/d^2=(4a^2/3(h^2+hk+k^2))+ l^2/c^2$

As a=3.2508 A^0 and c=5.2108 A^0. The values obtained matches with the standard values (JCPDS Card No. 36-1451, a = 3.249 A °, c = 5.206 A °, space group:P63mc, No.186).

B. *Scanning Electron Microscopy*

The particle size using SEM data is approximately found to be 20nm, 30nm, 40nm, 50nm, 350nm for the samples 1,2,3,4,5 and 20nm,40nm,60nm,70nm and 350 nm for the samples 6,7,8,9 and 10.

Sample 1 consist of monodispersed and spherical shaped nanoparticles. As the temperature is increased further the shape of the particles is changed and size of the particles also increases. The particles take rod like morphology As the temperature is increased further the micron sized particles are formed having polygonal shape.

C . Effect of temperature on the particle size and morphology

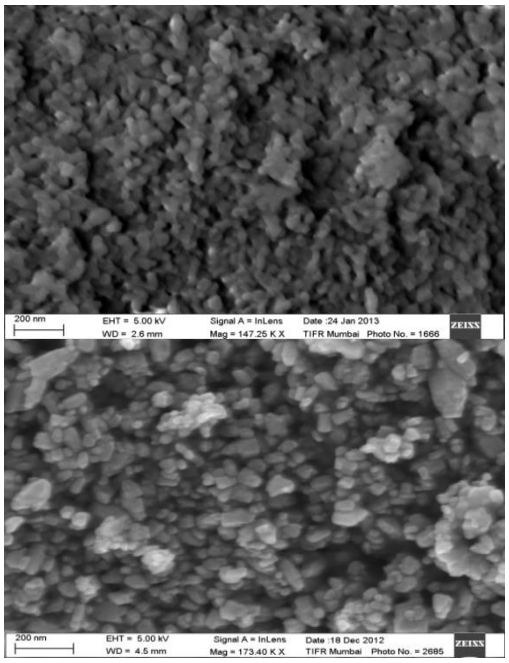
Reaction temperature is an important parameter which influences the structural morphology of the particles as well as the particle size. As the reaction temperature is increased there is increase in the particle size. In heating process when the particles are formed, they collide and either coalesce with one another to form a larger particle or coagulate. The process which occurs depends upon the temperature and available energy, that's why particle size increases with increasing temperature.

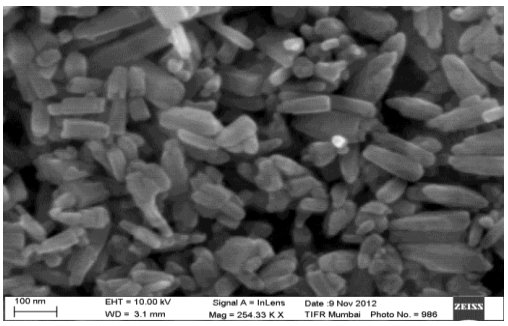
D.Effect of base concentration on the particle size and morphology

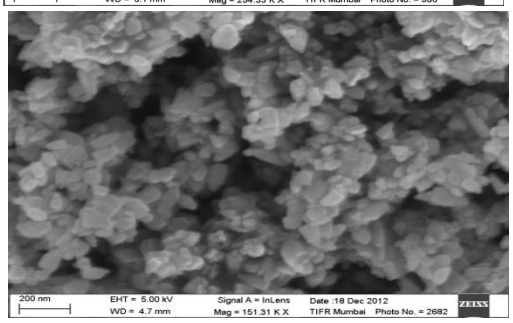
Herein as we increase the concentration from 2M to 10M the particle size increases. The reduction of Gibb's free energy is the driving force for both nucleation and growth.

$$\Delta G=(-KT/\Omega)\ln(C/C_0)$$

Where C is the concentration of the solute C_0 is the equilibrium concentration or solubility, Ω is the atomic volume. From above equation we can see that as the concentration increases Gibb's free energy increases. To lower this energy the particles continues to grow unless the minimum energy required for stability is achieved. The hydroxyl ion concentration plays an important role for the morphology and size. After nucleation the hydroxyl ions excess in solution is adsorbed on the polar faces of growing particle i.e. it takes nano rod shape. For even higher concentration hydrolysis/condensation is uncontrolled then no







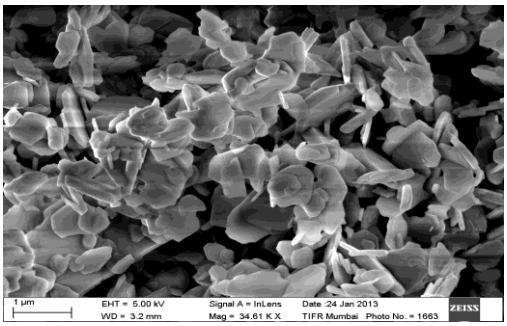


Fig 1: SEM Images of the synthesized ZnO nanoparticles at different temperatures 80^0C, 85^0C, 90^0C, 95^0C, 100^0C

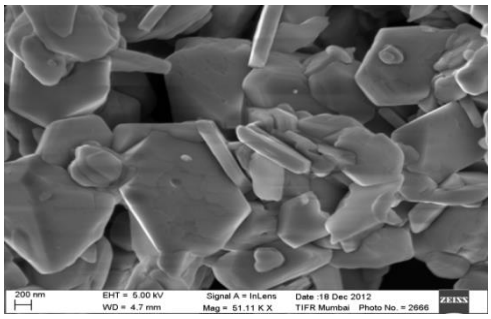
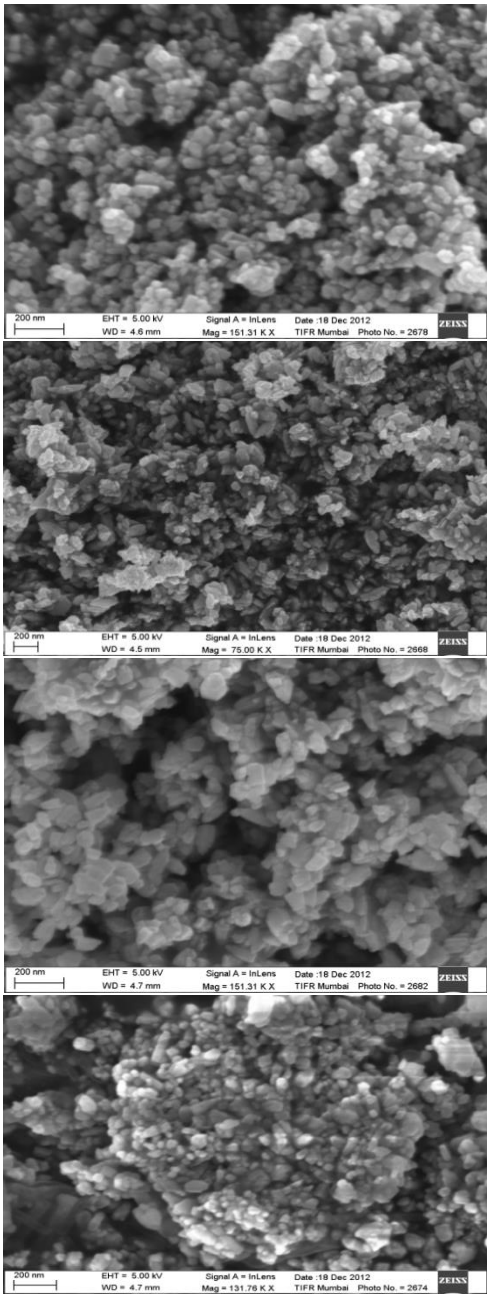


Fig. 2: SEM images of the synthesized ZnO nanoparticles at different molar concentrations of base 2M,4M,6M,8M,10M



preferential growth is observed along c axis and so the particles appear in quasi spherical shape. The critical size represents the limit how small nanoparticles can be synthesized. Critical size and critical energy is given by

$$r^*=-2\gamma/\Delta Gv$$

and

$$\Delta G^*=16\pi\gamma/3(\Delta Gv)^2$$

where γ is the surface energy per unit area and ΔGv change of Gibb's free energy or volume energy.

To reduce the critical size and free energy one need to increase the change of Gibb's free energy, ΔGv and reduce the surface energy of the new phase.

IV.CONCLUSION

In this paper wet chemical method for synthesis of zinc oxide nanoparticles is optimized for suitable temperature and base concentration. The temperature and base concentration is having profound effect on the size and shape of the nanoparticles. The particle size increases with increase in temperature and with increase in base concentration. The possible reason for both the observation is explained in terms of Gibb's free energy associated with the nanoparticles. The particles acquire the shape and size in such a way so as to minimize the Gibb's free energy.

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