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Annealing Effect on Structural and Morphological Study of ZnO Nanoparticles By A Microwave Irradiation Method

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ABSTRACT

Zinc Oxide (ZnO) nanostructures have been successfully prepared by microwave irradiation method. The crystallite size and morphology of ZnO have been investigated by X-Ray diffraction (XRD), fourier transform infrared spectroscopy (FT-IR), scanning electron microscopy (SEM), energy dispersive spectrum (EDS), and transmission electron microscopy (TEM) techniques. The XRD pattern of average particle sizes of ZnO is estimated to be around 14 nm. Furthermore, ZnO nanoparticles have the crystallite size in the range ~11-50 nm, as confirmed by TEM. Results obtained indicate that the microwave-assisted method is a promising low temperature, cheap, and fast method for the production of ZnO nanostructures.

Keywords: ZnO nanoparticles; Microwave irradiation; Structural; TEM:

INTRODUCTION

In recent years, noble metal oxide nano particles have been the subject of focused research due to their unique electronic optical, mechanical, magnetic and chemical properties. Zinc oxide is the topic of interest in these days due to its presence much unique and important morphology likes nanorods, nanoflowers, nanowires, nano dendrites and nanoparticles. Zinc oxide is a white solid inorganic powder. It is non flammable, stable and insoluble in water, II-VI semiconductor with wide band gap energy that is 3.3ev and high excitation energy that is 60ev [1]. This semiconductor has several favorable properties including good transparency, high electron mobility, strong room temperature, low toxicity, luminescence and photo chemical stability and higher breakdown field strength.

General, Zinc oxide crystallizes in two main form, hexagonal wurtzite and cubic zinc blende but the (B4 type) wurtzite structure is obtained only at optimum pressure and temperature [2, 3]. ZnO crystallizes in the typical wurtzite hexagonal structure where oxygen and zinc atoms are spatially arranged in a way that O atoms are arranged in a closed hexagonal structure, while the Zn atoms occupy the centre of the distorted tetrahedron structure [4]. The variety of structures of Nano metric zinc oxide means that ZnO can be classified among new materials with potential applications in many fields of nano technology. Zinc oxide can occur in one - (ID), two - (2D), and three-dimensional (3D) structures. One dimensional structure make up the largest group, including nano rod -needles, -helixes, - springs and -rings, - ribbons, - tubes - belts - wires and -combs. Zinc oxide can be obtained in 2D structures, such as Nano plate/Nano sheet and Nano pellets. Examples of 3D structures of zinc oxide include flower, dandelion, snowflakes, coniferous urchin-like, ZnO provides one of the greatest assortments of varied particle structures among all known materials [5, 6]. The large specific surface area high pore volume, nano structured properties, low cost and low toxicity of nano ZnO [7] make it a promising candidate, particularly in catalysts [8], photo catalysis, electrostatic dissipative coating, transparent UV protection films, and chemical sensors [9-12], gas sensor, solar cells. Moreover, ZnO nanoparticles have a tremendous potential in biological applications like biological sensing, biological labeling, gene delivery, drug delivery and nano-medicine [13-16].

One of the main environmental applications of nanotechnology is in the water sector. heterogeneous photocatalysts, one of the advanced oxidation process(AOPS),is a cost-effective treatment methods for the removal of toxic pollutants from industrial waste water sowing to its ability to convert these into safer and products such as CO_2, H_2O and mineral acids [17-18]. Several conventional methods have been used for synthesis of zinc oxide nanoparticles like chemical vapour synthesis [19], laser ablation [20]), solvothermal [21], thermal decomposition [22], and sol-gel method [23]. Here we present a

simple Co-precipitation method to synthesize uniform, spherically shaped and pure ZnO nanoparticles using zinc Sulphate as a metal precursor and ammonium hydroxide as a precipitating agent. In the present study was report the synthesis of ZnO nanoparticles using Co precipitation method and the characterization of ZnO nanoparticles using x-ray diffraction (XRD), transmission electron microscopy (TEM), selected area electron diffraction (SAED) , scanning electron microscopy (SEM), fourier transform infrared spectroscopy (FTIR) energy dispersive spectrum (EDS) are discussed [24 – 29].

EXPERIMENTAL PROCEDURE

MATERIALS

Zinc acetate (Merk) and ammonia solution (Merk, 98%) were used for the synthesis of ZnO nanoparticles. Zinc acetate were of analytical grade and used as received without further purification was used for the synthesis. Double distilled water was used for all the experiments.

SYNTHESIS

The initial solution was prepared dissolving 0.1 mol of zinc acetate in 20 ml of ethanol, which was later mixed with 175 ml of deionized water. The pH of the solution was maintained at 8 by adding a liquid ammonia solution dropwise. The resulting product was filtered and washed with double distilled water and ethanol until it became free from impurities. The precipitate was irradiated for 5 minutes in a household microwave oven (radiation frequency - 2.45 GH_Z, Power up to 1 $_KW$) with convection mode, giving a white product. Additionally, the resulting powders were thermally treated at 300°C in a muffle furnace for 4 hours.

CHARACTERIZATION

The resulting powders were analyzed by X-ray diffraction (XRD) using a Bruker AXS D8 Advance instrument diffractometer with monochromatic CuKα1 wavelength of 1.5406 Å. The samples morphology was observed by scanning electron microscopy (SEM), using a JEOL 5600LV microscope at an accelerating voltage of 10 kV. The microstructure was studied by transmission electron microscopy (TEM) and selected-area electron diffraction (SAED) in a Tecnai G20-stwin operated at 200 kV. The Fourier transform infrared spectra (FT-IR) of the samples were recorded by using a Nicolet 5DX FTIR spectrometer.

RESULTS AND DISCUSSION

X-Ray Diffraction analysis

The synthesized ZnO is particles were subjected to valuable studies to understand their structure, morphology size and other properties. The results obtained from those studies are discussed in this chapter. The powder X-ray diffraction pattern was studied to determine the structure of the as synthesized nanomaterials Zinc oxide. Fig. 1 depicts the Powder XRD pattern to determine the phase composition of the sample annealed temperature at 300°C. If the sample shows the diffraction peaks of crystalline ZnO_2 in its characteristic Hexagonal structure (JCPDS, No.36-1451).The average crystalline size of the crystallites was evaluated using the scherrer's formula,

$$D = \frac{K\lambda}{\beta \cos\theta}$$

Where D is the crystalline size (nm), K is a grain shape dependent constant (0.9), λ is the wavelength (1.5406 nm) of the incident beam, θ is a Bragg reflection angle and β is the full width at half maximum (FWHM) of the main diffraction peak. As estimated from the width at half-maximum of the main diffraction peak according to the Scherrer equation, are about 14 nm for the annealed temperature at 300°C. The calculated unit cell parameters are a= 5.145 Å, b= 5.205 Å, and c= 5.312 Å.

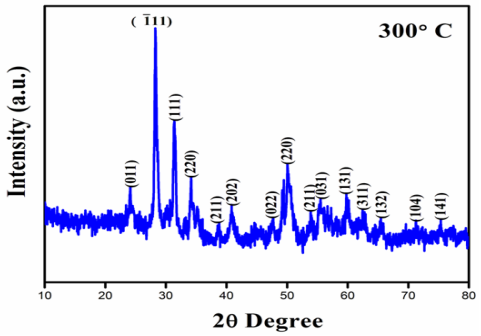

Fig.1 X-ray diffraction patterns of ZnO were annealed at 300°C.

Fourier Transform infra-red Spectroscopy (FTIR)

FT-IR spectrum was annealed temperature at 300°C. The IR spectroscopy was used to learn the surface interactions of the adsorbed water in dynamic symmetry with the gas phase on the ZnO surface. Frequency shifts and absorbance values were carefully observed to interpret the surface structure of the ZnO phase. It is well-known that H_2O and CO_2 molecules are simply chemisorbed onto the ZnO surface as soon as exposed to the ambiance.

WAVE NUMBER	ASSIGNMENT
501 cm^{-1}- 732 cm^{-1}	ZnO stretching frequency of Zn-O bond
3443 cm^{-1}	water re-absorption during the storage of the sample
1442 cm^{-1} **1627 cm^{-1}**	C=O stretching vibrational modes C=C stretching vibrational modes
2924 cm^{-1} **2854 cm^{-1}**	symmetric C-H bonds asymmetric C-H bonds

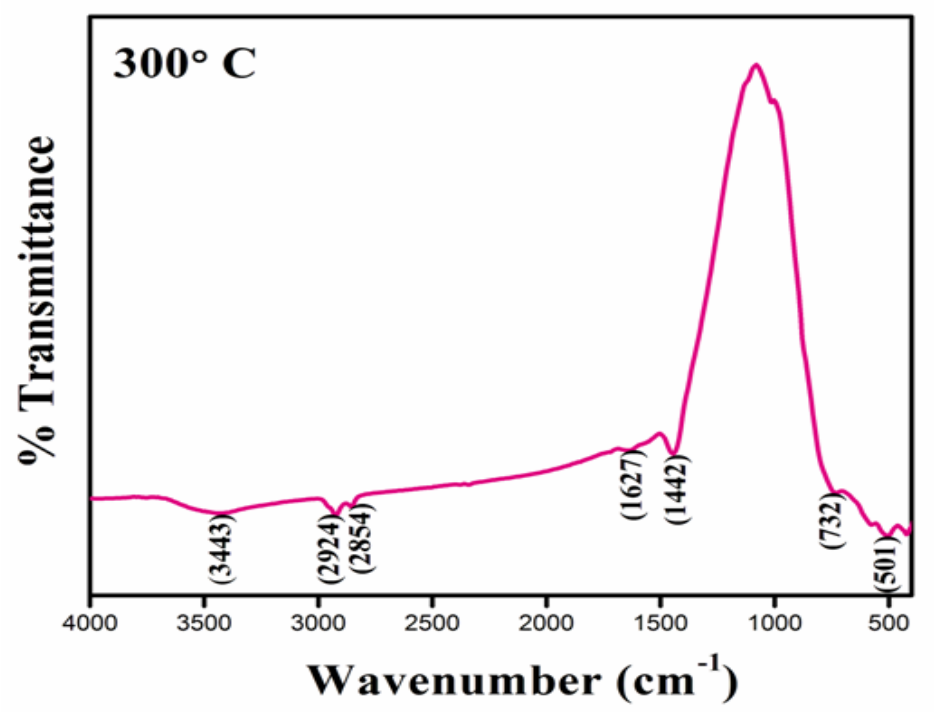

Fig.2 Fourier transforms infra-red spectroscopy of ZnO samples annealed at 300°C.

SCANNING ELECTRON MICROSCOPY (SEM)

The morphology of the synthesized nanocrystalline was analyzed by scanning electron microscope (SEM). The presence of the agglomerated with crystalline nature with composed by the agglomeration and non agglomeration of the smaller crystallites.

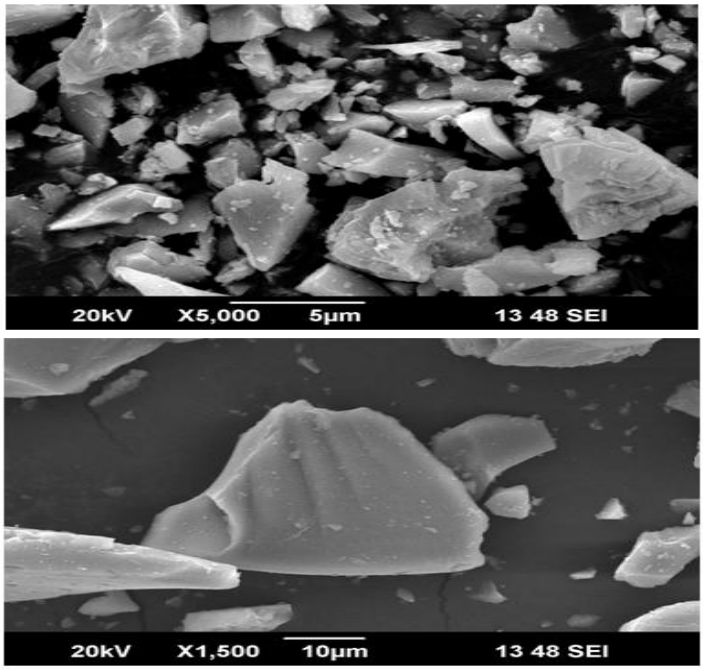

Fig.3. Scanning electron microscope of ZnO annealed temperature at 300°C.

Consequently the temperature distribution is homogeneous and is transferred to the materials interior, making an explosive reaction followed by vigorous evolution of the gases to form ZnO with good polycrystalline nature.

Energy Dispersive Spectrum (EDS)

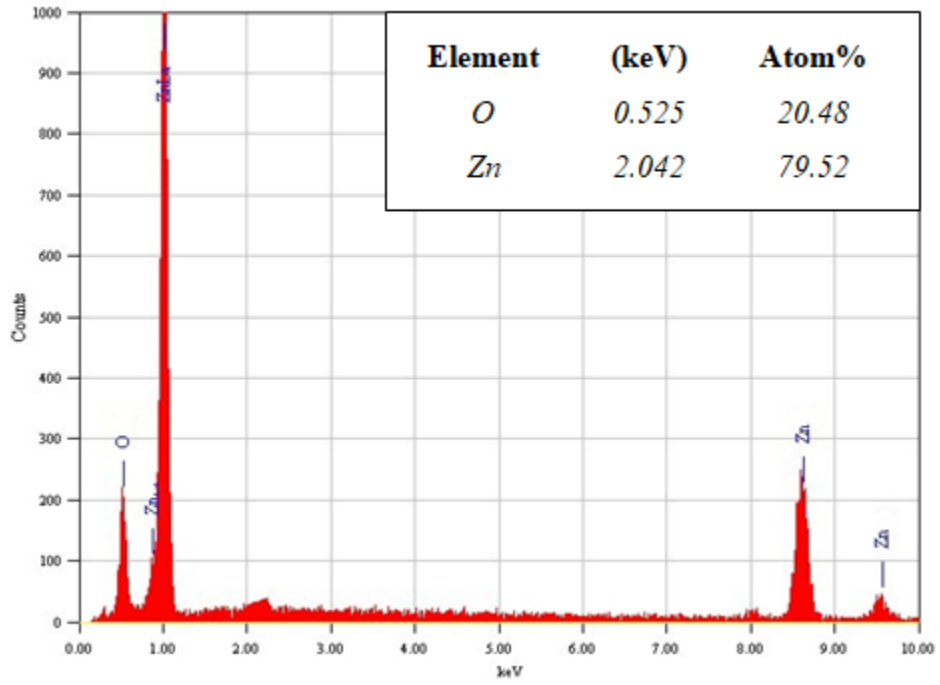

Fig.4. Energy dispersive X-ray analysis of ZnO annealed temperature at 300°C.

The composition of the obtained ZnO was analyzed by means of energy dispersive X-ray analysis (EDX) as shown in Fig. 4. The EDX result showed the presence of Zn and O by the appearance of Zn and O peaks.

Transmission electron microscopy (TEM)

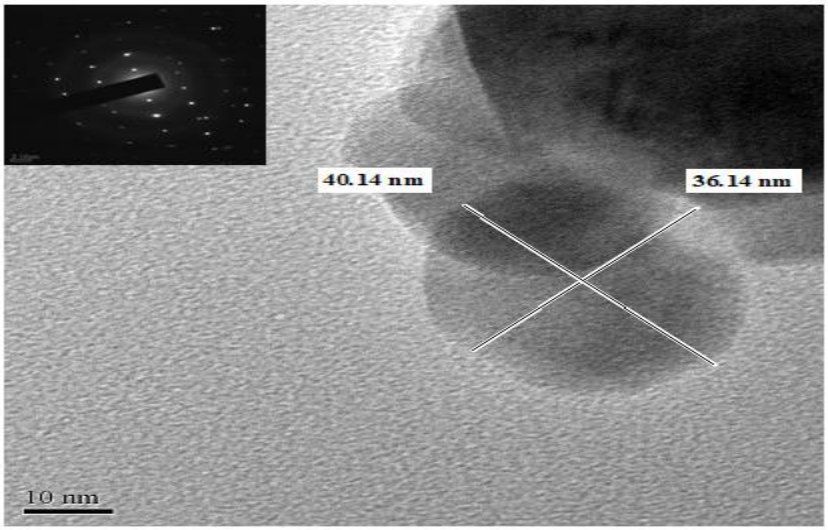

Fig.5. Transmission electron microscope of ZnO annealed temperature at 300°C.

The Particle size and nanostructure of ZnO nanoparticles have been examined through transmission electron microscope (TEM) are shown in Fig 5. The particle sizes of the zinc oxide samples are consistent with the results of XRD analysis. The pattern implies that the prepared ZnO nanoparticles are some particles spherical shape in the range of about 11-50 nm. The SAED pattern of ZnO nanostructures further confirms the crystalline nature of the sample. Fig 5 shows an electron diffraction pattern representing well-defined quasicontinuous diffraction rings. It is visible that the (011), (021), (211) and (131) planes were clearly distinguished as observed in XRD patterns. The SAED pattern of the high resolution TEM image conformed the nanoparticles corresponds to Hexagonal structure.

CONCLUSION

The ZnO_2 nanoparticles synthesized by Co – Precipitation method have been investigated. The sample have been investigates to the annealed at 300°C temperature. The Powder x-ray diffraction pattern of the nanocrystalline ZnO_2 possessed hexagonal structure with average crystallite size in the range of 14 nm for annealed temperature at 300°C. The FT-IR spectra of ZnO_2 are well-known that H_2O and CO_2 molecules are simply chemisorbed onto the ZnO_2 surface as soon as exposed to the ambiance. SEM results are presence of the agglomerated with crystalline nature with composed by the agglomeration and non agglomeration of the smaller crystallites. The EDS result showed the presence of Zn and O by the appearance of Zn and O peaks. The TEM pattern implies that the prepared ZnO_2 nanoparticles are some particles spherical shape in the range of about 11-50 nm. Thus the simple synthesis method of the zinc oxide nanoparticles was gas sensor applications.

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