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Synthesis of nano crystalline ZnO by Microwave Assisted Combustion method: An eco friendly and solvent free route

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ABSTRACT

ZnO nano crystals were synthesized rapidly using solvent free, economic and eco friendly method by microwave-assisted combustion method (MWAC). The economic and eco-friendly synthesis of ZnO nano particles using minimum amount of ethylene glycol as organic dispersant without use of any solvent was reported here for the first time. The study suggested that application of microwave heating to produce homogeneous and porous ZnO particles was achieved in a few seconds. The structure characterization and morphology of ZnO particles were investigated by XRD, FTIR, UV-Visible and SEM methods. The XRD and FTIR confirmed the formation of hexagonal wurtzite structure and SEM results confirmed the porous surface characteristic. The ZnO particles have band gap energy 3.4 eV.

1. INTRODUCTION

Nanostructured materials are known to exhibit interesting physical and chemical properties. These properties are significantly different form those of conventional bulk materials, due to their small size and pozolinic surface area. Especially inorganic materials like metal oxides have attracted mush attention due to their stability under hursh conditions and regarder as a safer materials for human and animals. The use of silver and zinc oxide nanoparticles has been established as a viable solution to stop infectious dieseases due to their antimicrobial properties. Various methods used for the synthesis of ZnO nano particles of contollled size and shape includes; pulse laser deposition [1] Microwave thermal evaporation [2] Hydrothermal [3] Vapor phase transparent process [4] Thermal evaporation [5] Sol–gelsynthesis [6] Combustion synthesis [7]. Microwave assisted synthesis of nanocrystalline materials has attracted much attention in recent years because of its several advantages such as unique synthetic pathways, rapid heating rates, short processing durations, uniformity and low power requirements [8]. Microwave irradiation as a heating method is different from conventional heating process. In this method heat will be generated internally within the material, instead of originating from external sources [9]. In the present work ZnO nano crystals synthesizd by microwave assisted combustion method (MWAC) using ethylene glycol use as dispersant. It is best, time saving and fast process for synthesis nano particles.

2. METHOD & MATERIALS

2.1 Chemicals and methods

All the chemicals and reagents used in the present study were highly pure and were of analytical grade. Zinc nitrate hexa hydrate Zn $(NO_3)_2$. $6H_2O$ (99% purity by wt.) was obtained from Merck (Germany) and ethylene glycol was obtained from SD fine chemicals, India. Microwave oven (Modal No. IFB 20SC2, 2000 W Power Consumption microwave 800 W, output range 230V-50 Hz.) was used for heating purpose. About 5.5 gm. of the Zn $(NO_3)_2.6H_2O$ was mixed with 2-3 drops of Ethylene glycol. The amount of Ethylene glycol used was optimized after several trials. The paste boils in a few seconds and undergoes dehydration followed by decomposition with the evolution of gases (O_2, NO_2) with yellow colors fumes. After 30 seconds it begins burning and releases lots of heat with vaporization of all the solution instantly to form foamy porous white solid powder.The expected microwave assisted combustion reaction to form ZnO nanoparticles is:

$Zn(NO_3)_2 \longrightarrow ZnO_2 + 2NO_2$ (at lower temperature)

$2ZnO_2 \longrightarrow 2ZnO + O_2$

The advantages in the above reaction is twofold, (i) since no solvent is used thus possibility of formation of any solid byproducts is very negligible.(ii) it requires few sec. to complete therefore very economic and eco friendly in terms of fuel consumption and production of very low toxic gases respectively.

3. RESULTS & DISCUSSION

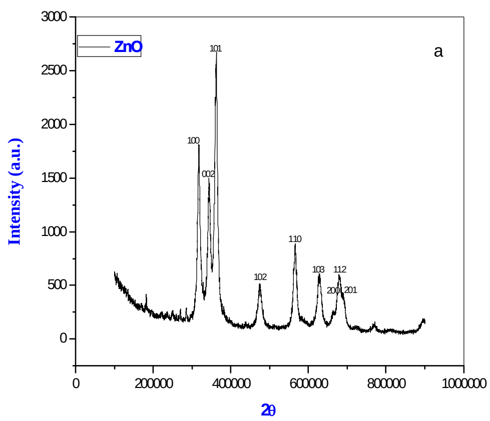
3.1 X-ray Diffraction pattern

The structural characterization confirm by X-ray diffraction (XRD) method using CuKα radiation (1.5418A^o). ZnO nano particles obtained typical XRD pattern (Fig. 1a, 1b). It is indicated in the reflecting planes (1 0 0), (0 0

2), (1 0 1), (1 0 2), (1 1 0), (1 0 3), (2 0 0), (1 1 2) and (2 0 1), which can be perfectly indexed to the hexagonal wurtzite structure (JCPDS card .No. 89-1397). XRD pattern shows that the sample formed is in single phase with the hexagonal symmetry. The absence of extra peak claims the purity of the substance and also the complete conversion of Zinc nitrate in to ZnO. The crystalline size of the sample obtained from Scherrer's formula.

D = 0.9λ / β cosθ (1)

Where D-Crystallite size λ- Wavelength (1.5418 Armstrong), β-Full maxima at half width of the ZnO nano particles. θ- Diffraction angle The average crystalline size found to be around 13 nm. This is confirmed that ZnO nano crystalline is hexagonal wurtzite structure.



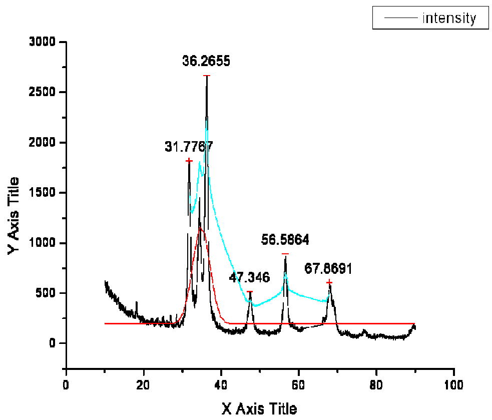
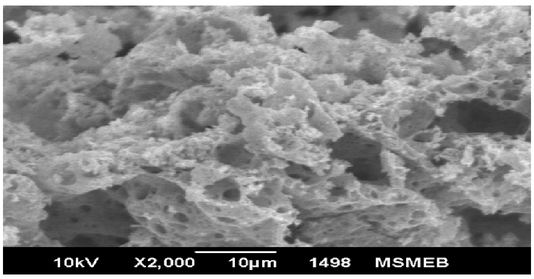


Fig. 1b. XRD OF ZnO X = 2θ and Y = intensity (a. u.)

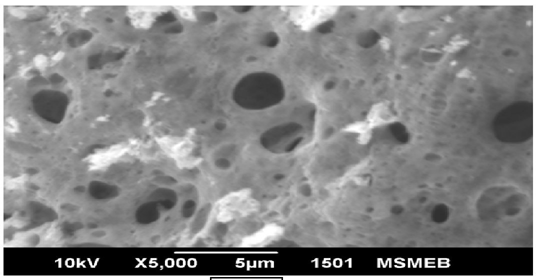
Fig. 1 XRD pattern of ZnO nano particles

3.2 Scanning electron microscopy

The surface morphology of the ZnO nanoparticles was examined using Scanning electron microscope. The SEM micrographs of ZnO nanoparticles at different magnification are shown in Fig 2 a and 2 b. The result reveal that the product is foamy and porous in nature having pore size 2 µm. This foamy structure is formed due to escaping gases like NO_2, O_2 etc. as mentioned in mechanism.



2a

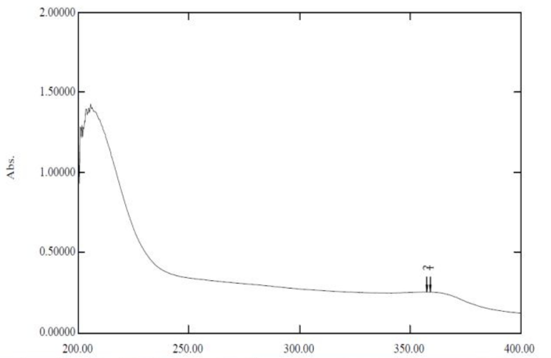


2b

Fig. 2 SEM micrograph of ZnO nanoparticles

3.3 Fourier transforms infrared spectroscopy

The FTIR graph of ZnO nanoparticles using ethylene glycol as organic dispersant are shown in Fig 3. Spectrum of the prepared nano crystalline ZnO observed main peaks below at ~500 cm^{-1} corresponding to the ν (Zn-O) modes confirm the formation of ZnO. A weak peak observed at near ~1323.11cm^{-1} is due to NO_3-bonding, which is due to the nitric acid group being adsorbed on the ZnO powder surfaces and peak at



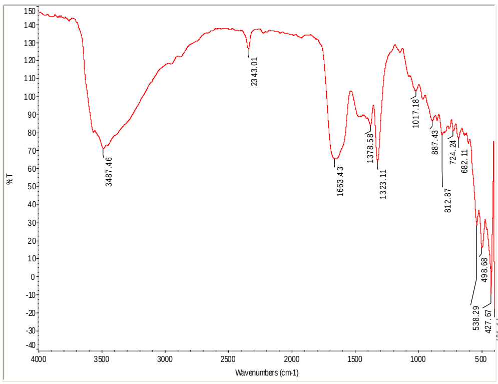


Fig.3 FTIR graph OF ZnO nano particle

Fig.4 UV–Visible of ZnO nano particle

~3487.46 cm^{-1} is the characteristic of O-H stretching and bending vibrations of adsorbed water molecules and another two bands at ~1550 and 1355 cm^{-1} due to O-H deformation and bonding between Zn and O (~400–650 cm^{-1}) is clearly represented [10].

3.4 UV-Visible spectroscopy

Size evolution of semiconducting nano particles become very essential to explore the properties of the materials. UV-Visible absorption spectroscopy is widely being used technique to examine the Optical properties of nano sized particles . The prepared Zinc oxide white crystalline powder was not soluble in water and almost in all organic solvents. ZnO nano particles UV–Visible spectra recorded by dispersed in methanol solution and sonicated for 5 to 10 min. The absorption band obtained at 357.60 nm. The band gap of ZnO nano particles can be determined by the following equation. The band gap of ZnO nano particles determined by formula.

Energy band gap = 1248/ $\lambda_{max.}$

The band gap of the ZnO particles is 3.4 eV. ***M. Saravana et al***. reported the band gap of ZnO is 3.9 eV.[11].

4. CONCLUSIONS

ZnO Nano crystal has been successfully synthesis by microwave assisted combustion method using ethylene glycol as organic dispersant within few seconds. X-ray diffraction confirm the formation of a hexagonal wurtzite phase. FTIR shows main peaks below at ~500 cm^{-1} corresponding to the ν (Zn-O) modes which revealed the formation of ZnO. SEM confirms the porous agglomeration free nanosize particles. UV- Visible spectra confirm absorbance at 357.60 nm and band gap energy of ZnO nano particles 3.4 eV is obtained. This process is an economical method for the preparation of nanocrystalline ZnO with respect to energy, time and simplicity.

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