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Instant Synthesis of ZnO Nanoparticles by Microwave hydrothermal Method

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Abstract

ZnO nano particles has been prepared for fulfilling the necessity of large scale application in modern research world. The present article reports a new, simple and highly efficient microwave assisted hydrothermal chemicalized precipitation method from a mixture of solutions like zinc acetate and sodium hydroxide. The current synthesis method has more advantageous parameters like low cost, rapid reaction rate, large yield production of ZnO nano particles. The obtained ZnO nano particles are within averaged diameter of 30-35 mm and were characterized by scanning electron microscopy (SEM), X-ray diffraction (XRD), UV Spectroscopy, FTIR analysis from which the morphology and particle size of ZnO nano particles was revealed.

Keywords: ZnO nanoparticles, microwave radiation, hydrothermal

1. INTRODUCTION

The unique properties of ZnO nanostructures have attracted considerable attention for there potential technological applications [1]. The wide band gap of 3.3 eV and large excitonic binding energy of 60 meV as made ZnO nanostructures important both for scientific as well as industrial applications [2]. ZnO nanoparticles have pinnacle range of applications in ultraviolet (UV) lasers [3], power generators [4], solar cells [5], gas sensors[6], field emission devices [7], capacitors[8], photo printing [9],electro mechanical nano devices[10], sunscreen lotions[11]. In addition to ZnO nanoparticles [12-14] several forms like nanorods [15] nanofilms [16] can be synthesized. The ZnO Nanoparticles are having high surface area to volume ratio as well as good electrical, electro chemical and structural properties[17] in general there are many synthesis method for ZnO Nanoparticles [18-20], including vapor phase method[21] and

aqueous method[22] hydrothermal method[23-24]. Above all these methods microwave assisted hydro thermal method is found to is an efficient tool for the synthesis of ZnO Nanoparticles with low cost, low temperature and low complication technique.

2. EXPERIMENT

In a general ZnO Nanoparticles of spherical structures are synthesized by a 0.5Mol solution of zinc acetate dehydrate [Zn $(COOCH_3)2(H_2O_2)$] prepared by dissolving in 100ml of ethanol and stirred for 30minutes at 60^oC. Also 0.9M aqueous solution of sodium hydroxide (NaOH) was prepared in a similar way with stirring for 30 minutes. The 60^oC heated 0.9M NaOH aqueous solution was added to drop by drop (slowly for 1 hour) touching the walls of the conical flask containing zinc acetate dehydrate [Zn $(COOCH_3)2(H_2O_2)$] solution under high constant speed stirring by magnetic stirrer. The solution turns into jelly form and a milky white turbid solution was obtained after 2 hours. The solution was allowed to settle for 2 hours sealed. The turbid solution was then subjected to microwave radiation at a temperature of 90^oC heated for 20 minutes. The product becomes fluffy inside the oven, removed and dried at room temperature and grinded, the obtained is a powder consists a mixture of Nanoparticles of spherical structures. The total procedure is repeated for different time durations in microwave chamber.

3. RESULTS AND DISCUSSION:

3.1 X-Ray Diffraction Analysis:

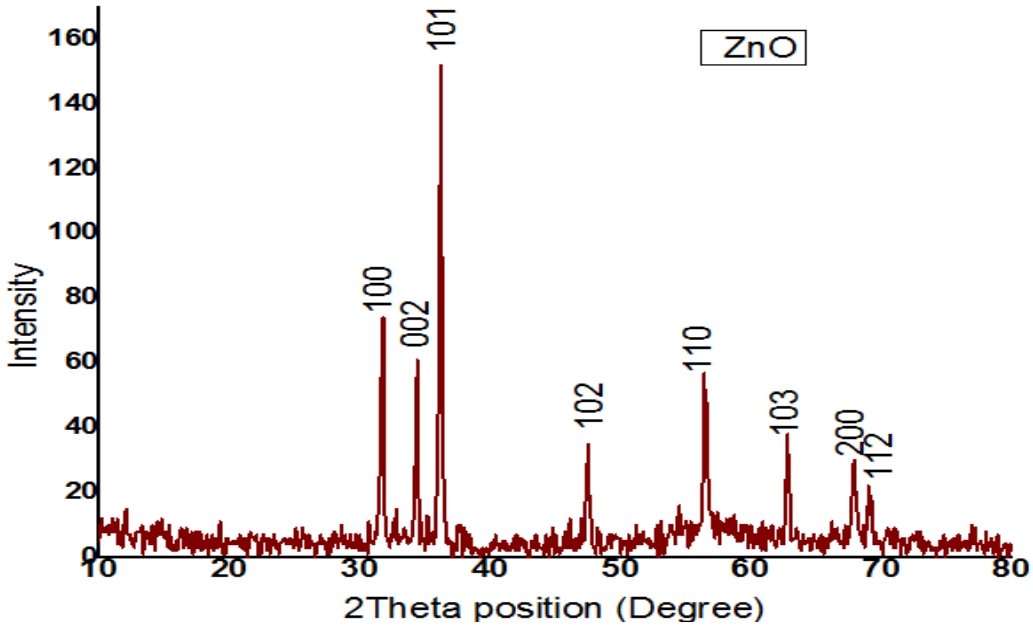

Fig. 1. XRD patterns of ZnO nanoparticle

XRD patterns of the synthesized samples were shown in Fig.1 XRD shows 2 theta values at 31.77^{o}C, 34.40^{o}C, 36.22^{o}C, 47.61^{o}C, 56.58^{o}C, 62.85^{o}C, 66.41^{o}C, 67.93^{o}C, 69.08^{o}C, 72.54^{o}C and 76.85^{o}C corresponds to (100), (002), (101), (102), (110), (103), (200), (112), (201), (004) and (202) planes confirmed the presence of ZnO NPs. All the peaks were duly assigned using JCPDS card no. 361451. Average crystalline size of ZnO NPs is found to be 30nm using Scherrer equation crystallite size of the sample is estimated from the full width at half maximum (FWHM) of the diffraction peak Diffraction pattern corresponding to impurities are found to be absent. This proves that pure ZnO NPs were as synthesized.

3.2 Fourier Transform Infra-red Spectroscopy Analysis:

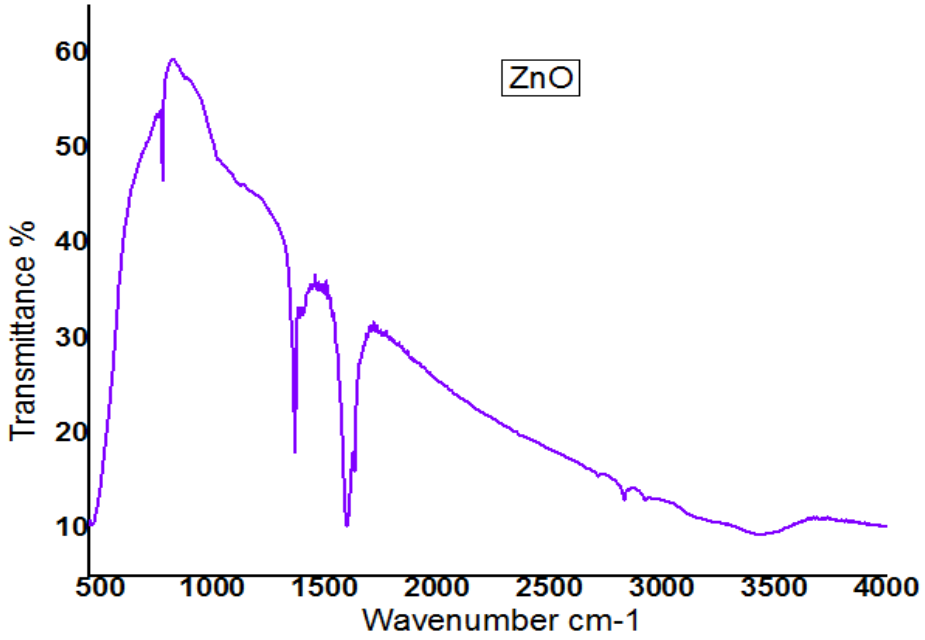

Fig.2. FTIR spectra of ZnO nanoparticle.

The identification of the atomic arrangement and the concentrations of the chemical bonds present in the samples have been carried using Fourier Transform Infra-red Spectroscopy (FTIR), in which percentage transmission and wave number are the output. Fig.2. shows FTIR spectra of ZnO nanoparticles which were acquired in the range of 450-4000 cm^{-1}. Infrared studies were carried out in order to ascertain the purity and nature of the metal nanoparticles. Metal oxides generally give absorption bands in fingerprint region i.e. below 1000 cm^{-1} arising from inter-atomic vibrations. The peak observed at 3000 and 1300 cm^{-1} are may be due to O-H stretching and deformation, respectively assigned to the water adsorption on the metal surface. The peaks at 1600 to 450 cm^{-1} are corresponding to Zn-O stretching and deformation vibration, respectively. The metal-oxygen frequencies observed for the respective metal oxides.

3.3 Particle Size Analysis :

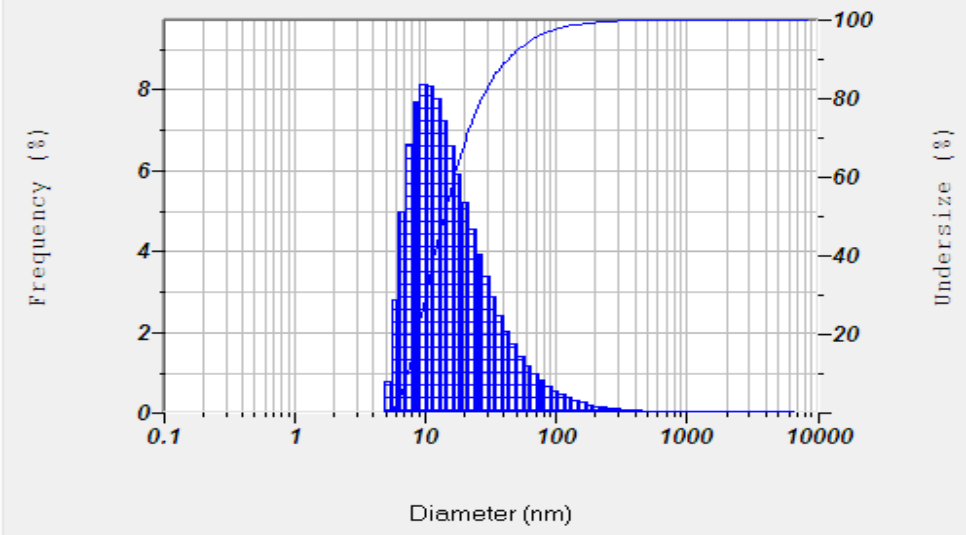

Fig.3. PSA spectra of ZnO nanoparticle

Particle Size Analysis (PSA) testing is essential to study the nano-particle size. The particle size distribution curve for ZnO nano-particles is shown in the Fig.3. The average particle size is calculated with the instrument HORBIA SZ100 and the scattering angle is 90˚. The average particle size is 29 nm. It corresponds with the crystallite size calculated from the XRD pattern.

3.4 Scanning Electron Microscope Analysis :

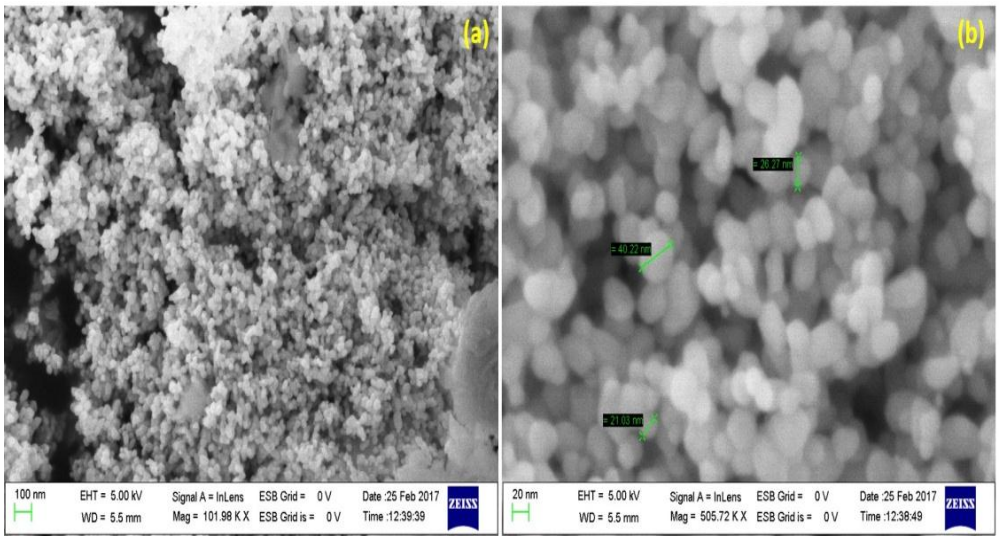

Fig 4(a) shows uniform growth of ZnO with spherical morphology diameter around 20-25 nm with magnification (100KX). **Fig.4 (b)** shows high magnification (500KX) for further confirmation of ZnO nanoparticle growth.

The surface morphology of ZnO NPs was thoroughly studied by FESEM micrographs. From Fig4 (a) (b), FESEM micrographs it was observed that ZnO Nanoparticles showed irregular surface morphology and was amorphous in nature, previously confirmed by XRD. Spherical structure was found which on higher magnification showed aggregation of smaller spherical particle and together forming chain like structure around 28nm.

3.5 UV-Vis Analysis:

The UV absorbance spectrum of ZnO nanoparticles is recorded in the wavelength range 300nm-800nm. Fig 5 shows optical absorbance spectrum of ZnO nanoparticles synthesized by microwave irradiation at 150^{o}C .The spectrum shows an absorption peak at 343nm which reveals the quantum size effect of ZnO Nanoparticles. The slight shift in the absorption may occur upon the change in particle size or particle shape.

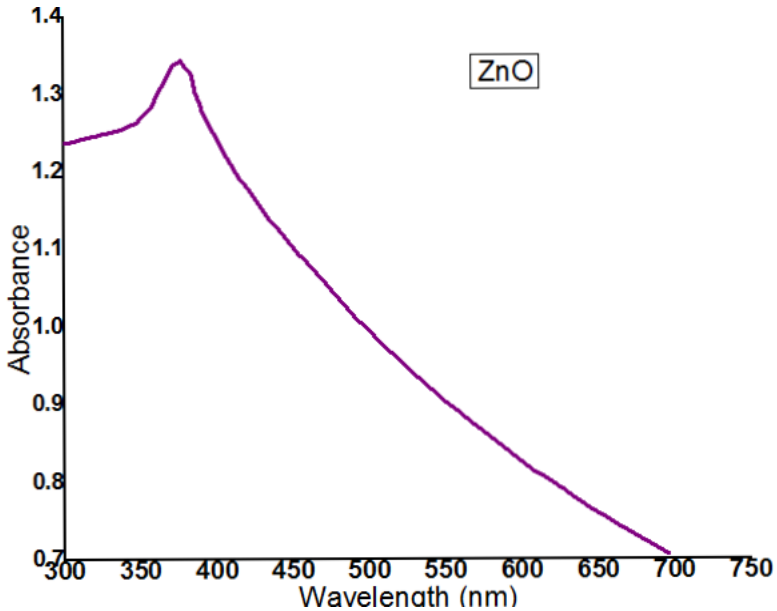

Fig.5. UV absorbance spectrum

4. CONCULSION:

The present work revealed a very simple approach for the synthesis of ZnO Nanoparticles by highly efficient microwave assisted hydrothermal method, which proved to be simple and reproducible for variety of nanostructures in large scale. The XRD, PSA analysis confirms the size of ZnO Nanoparticles, SEM images revealed the spherical granular ZnO Nanoparticles, Zn-O stretching and deformation vibration peaks were by disclosed by FTIR, and UV analysis technique confirmed the quantum size effect of ZnO Nanoparticles. The obtained ZnO nanoparticles are used in different research and industrial applications.

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