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Synthesis and characterization of nano sized ZnO using conventional and microwave heating methods

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Abstract: Nano Zinc Oxide (ZnO) nano particles were synthesized through a simple and cost effective chemical precipitation method. The synthesized samples were characterized by XRD and SEM. The influence of the double heating on the particle size was investigated. It was observed that the particle size of samples increased upon microwave heating. The product formation was confirmed from FT-IR spectra.
Key words: Zinc Oxide, Nano particles, SEM and XRD.

Introduction

Today in nanoscience and nanotechnology the synthesis of nanostructures through environmentally friendly methods attracts every ones sight towards it. Materials scientists achieved to design, obtain, control, manipulate and modify structures at the nanometer range by many synthetic routes. Zinc oxide, an amphoteric oxide has been found to have numerous applications such as photo catalysis, solar cells, varistors, chemical sensors etc. [1-4] due to its exceptional physical and chemical properties. ZnO is also a n -type semiconductor material of II-VI compound of the periodic table whose ionicity resides at the borderline between covalent and ionic semiconductor with a direct band gap of 3.2eV at 294K making it potentially useful for opto-electronic devices that emit at shorter wavelengths [5].

Nano structured ZnO materials have drawn much attention due to their distinguished performance in various fields. Synthesis of ZnO has been received the attention of almost all the materialists because of its versatile properties, such as the direct wide band gap (3.37 eV), large excitation binding energy (60meV at room temperature), good piezoelectric characteristics, chemical stability and non-toxicity suggest a good candidate of many practical applications, notably in the area of ultraviolet/blue emission devices [6]. Microwave heating shortens the reaction time compared to other usual heating methods [7]. In recent years a number of ZnO nanostructures have been fabricated using different synthesis methods for many potential applications.

In our project, we reported for the synthesis and characterization of nano zinc oxide using wet chemical -one pot method from Zinc Acetate with aqueous Potassium Hydroxide without adding any capping agent. We also compared the effect of heating methods viz., heating the samples in a hot air oven and double heating that means both in hot air oven and MW heating. This method is advantageous one because it is simple and cost effective.

Materials

Zinc acetate (Purity - 98%, Spectrum) and potassium hydroxide (Purity - 85%, Spectrum) were used for synthesis of Zinc Oxide without further purification.

Methods

One hundred milliliters of 0.1 M Zinc Acetate were added drop wise from the burette to beaker containing one hundred milliliters of 0.2 M Potassium Hydroxide solution kept in at room temperature on a magnetic stirrer under constant stirring using Teflon coated magnet. When the viscosity of the mixture increases, the revolution of the Teflon coated magnet adjusted that vortex of stirring was seen. The mixture was then stirred further for 20 minutes using magnetic stirrer. The reactions taking place in our synthesis are as follows:

$H_2 \rightarrow H^+ + OH$
$KOH \rightarrow K^+ + OH^-$
$Zn(CH_3COO)_2 \rightarrow Zn^{2+} + 2CH_3COO^-$
$Zn^{2+} + 2OH^- \rightarrow Zn(OH)_2$

The precipitated zinc hydroxide were filtered and washed several times with distilled water. They were then heated in a hot air oven at 150º C for 20 minutes and a part of these samples were heated in a microwave oven at 180W for 30 minutes and the as-synthesized nano Zinc oxide were named as AA1 and AA 2 respectively.

Charaterisation

The formation of Zinc oxide was investigated and confirmed by FT-IR spectra (Model-SHIMADZU spectrometer). The structural characteristics were determined by means of powder X-ray diffraction patterns using X'PERT- Goniometer ultima3 theta-theta gonio, Multipurpose diffractometer operating at 40kV/300mA using nickel filtered CuKα radiation in the 2 θ range of 10º – 80º at 2θ /min. The surface morphology, agglomeration level and crystalline state were studied by cold field emission scanning electron microscopy.

Results and Discussion:

The FT-IR spectra of the nano ZnO powders show main absorption at ~ 3400 $cm^{-1}$and ~1600 cm^{-1} which correspond to O-H stretching and bending mode respectively, The absorption band at around 2300cm^{-1} is due to absorbed CO_2 molecule. The standard absorption band at ~ 464 cm^{-1} in samples AC I and AC II corresponds to Zn-O stretching mode [8].

XRD patterns in Fig.1.a and 1b, show narrow peaks that indicate the crystallinity of the samples. All the diffraction peaks can be indexed as hexagonal wurtzite structure (JCPDS Card no: 36-1451). The crystalline sizes were determined using Scherer's Equation and are listed in Table 1. [9]

$$\langle D \rangle = 0.9\lambda / \beta \cos \theta$$

This table revealed that particle sizes of the ZnO that were obtained by double heating were higher than the samples obtained from simple heating in hot air oven. This may be due to attributed to strong agglomeration of the particles.

The SEM micrographs were as shown in Fig. 2.a and 2b. The SEM photographs revealed that the samples obtained by conventional heating exhibited a spongy morphology. The samples obtained from microwave heating showed a similar morphology but it clearly indicated the the ZnO started to crystallize out as spherical balls with uneven distribution of particle size on MW irradiation [10].

Table 1: Average particle sizes of the synthesized Nano ZnO

Heating method	Samples	Average Particle Size nm
Conventional	AAI	12
Microwave	AAII	17

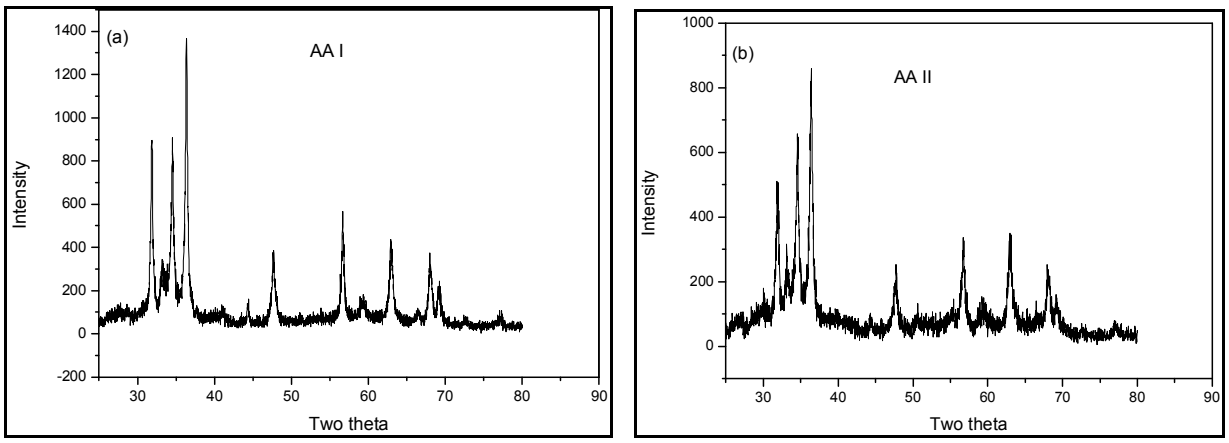

Fig1: XRD pattern of nano ZnO obtained from $Zn(CH_3COO)_2$ by Conventional MW irradiation

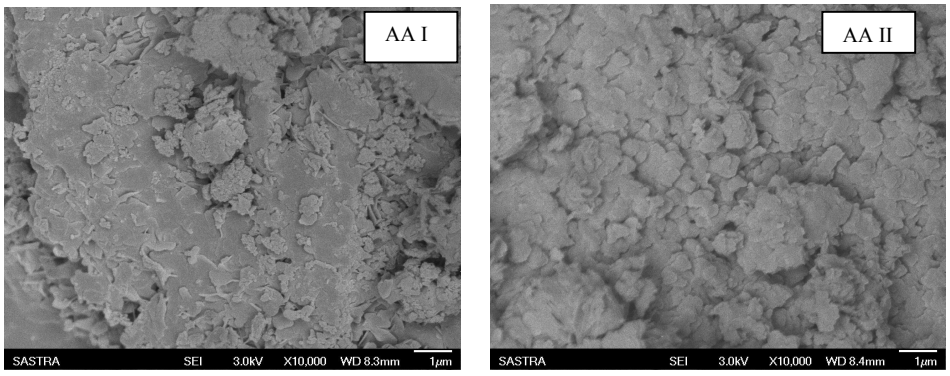

Fig2. SEM photographs of nano ZnO obtained from $Zn(CH_3COO)_2$ a) Hot air oven b) MW irradiation

Conclusion:

Nano crystalline ZnO powders were successfully synthesized by a simple and cost effective method utilizing Zinc acetate, Zinc Chloride, Zinc nitrate and KOH. The XRD patterns and FT-IR spectra suggested the formation of Wurtzite nano crystals in the synthesized powders after calcinating the precursors at 150° C in Hot air oven and irradiating with MW at 180 W. The nano powder consists of mixture of particles with sizes of 50-100 nm and 12-18 nm as revealed by SEM and XRD. The current simple synthesis method using desk top chemicals to prepare nano crystalline powders can be extended to synthesize other metal oxide powders.

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