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ZnO nanopowders by Microwave Assisted Hydrothermal Method

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Abstract –ZnO nanopowders were synthesized via microwave assisted hydrothermal method at different precursor solution molarities. For this, the aqueous solutions of zinc aceatate dihydrate and sodium hydroxide were prepared at different molarity as precursor materials. The synthesized ZnO nanopowders were further calcined in air at 500ºC for 2 h. The effects of different precursor solution molarity on the crystalline structure of the ZnO nanopowders were investigated by X-ray diffraction study. Field emission scanning electron microscope was used to analyze the surface morphology of the ZnO nanopowders. As the NaOH solution molarity increases, the rod-like structure of ZnO begins to be seen. The optical band gap values of the ZnO nanopowders were also determined by reflectance measurements.

Keywords – *ZnO, nanorod, microwave assisted hydrothermal method, precursor molarity, FESEM*

I. Introduction

Zinc oxide (ZnO) is an important transparent semiconductor conductive oxide material. Due to its trancparency in the visible region, non-toxic structure and abundant nature, it is very popular. Thanks to these features, ZnO can be used in many technological applications such as solar cells [1], gas sensors [2], varistors [3] , heterojunction diode [4] [5], transistors [6]. ZnO nanostructures synthesized in different morphologies such as nanowire [7], nanorods [8], nanobelts [9], nanotubes [10] and nanoflowers [11] are available in the literature. Even the different morphology of ZnO is obtained in different deposition processes of the same method. ZnO nanostructures are produced by production methods such as homogeneous precipitation [12], solvothermal method [13] and sol-gel [14] and microwave assisted hydrothermal method (MWHT) [15]. Among these, microwave assisted hydrothermal method is a synthesis method that is completely different from the other method. This method has many advantages, such as not requiring complex equipment, being easy, allowing controlled nanostructure synthesis. ZnO nanostructures can be produced in different morphologies depending on the solution parameters (type and molarity of precursors and solvents, pH of solution, surfactant, solution aging) by using this method.

In this study, ZnO nanopowders have been deposited by MWHT at different solution molarity and type (zinc aceatate dehydrate (ZnAc) and sodium hydroxide (NaOH)). The effects of solution molarity and type on structural, morphological and optical properties of ZnO nanopowders have been investigated using XRD, SEM and diffuse reflectance measurements, respectively.

II. Materials and Method

ZnO nanopowders were synthesized by MWHT different molarity of ZnAc and sodium hydroxide NaOH. ZnO nanopowders were produced using zinc acetate dihydrate ($Zn(CH_3COO)_2.2H_2O$, 99.5%, Merck) as the zinc cation precursor and, sodium hydroxide (NaOH, 98%, Sigma Aldrich), as the hydroxide anion source. For this, the aqueous solutions of zinc aceatate dihydrate (ZnAc; 0.1, 0.15 and 0.2) and sodium hydroxide (NaOH; 0.1, 0.2, 0.3 and 0.4M) were prepared as precursor materials. Aqueous solutions were mixed in a magnetic stirrer at 30 ° C. After stirring the zinc acetate aqueous solution, the NaOH aqueous solution was slowly added until a milky solution formed. The solutions were stirred for 20 minutes at room temperature in a magnetic stirrer and then placed in a microwave oven (CEM Mars 6). These solutions irradiated at 300W for 10 min. After, the solution was cooled to room temperature. The powders formed after the microwave process were filtered and washed with deionized water and ethanol for several minutes. The final nanopowders were dried in an oven at 60ºC for 24 h and then calcined in air at 500ºC for 2 h.

XRD measurements were performed by X-ray powder diffractometer (BRUKER D8 Advance). Surface morphology was studied using ZEISS Ultraplus model field emission scanning electron microscopy (SEM). The optical reflectance measurements of were carried out by double beam SHIMADZU UV 2450 spectrometer with an integrating sphere.

III. Results

The XRD study was performed to determine the crystallographic properties of ZnO powders prepared in different molarities. Fig. 1a and Fig. 2a shows the XRD patterns of ZnO nanostructures synthesized at different molarity of ZnAc and NaOH, respectively. As shown in these Figures, the XRD pattern belongs to pure zinc oxide and does not contain any impurity. ZnO nanopowders are crystallized in hexagonal wurtzite structure and the five peaks on the jcpds card are seen here (JCPDS # 36-1451). These peaks are

(100), (002), (101), (102) and (110). The texture coefficient values (TC(*hkl*)) of all the powders were calculated (not given here) and they were structured in the direction of (002). The crystallization of the nanopowders increased with increasing solution of molarity. The increase in the molarity of NaOH has great effect on the crystallization of the nanopowders.

The SEM images of the ZnO nanopowders deposited in different ZnAc and NaOH solution molarity are given in Fig 1b and Fig. 2b, respectively. The molarity of the solution affected the morphology of the powders. As the molarity increases the flowering structures began to grow and the diameter of the rods in the flower structure increases.

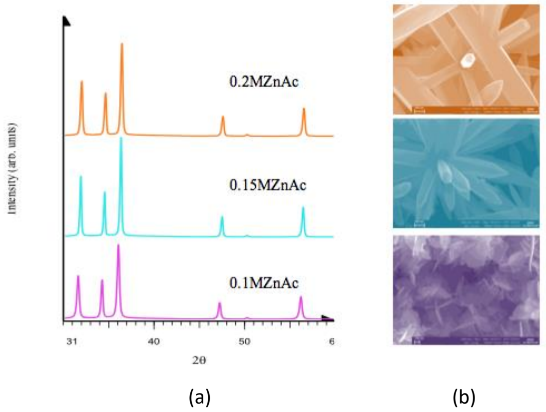

Fig. 1 XRD spectra and SEM images of the ZnO nanopowders synthesized at different molarity of ZnAc.

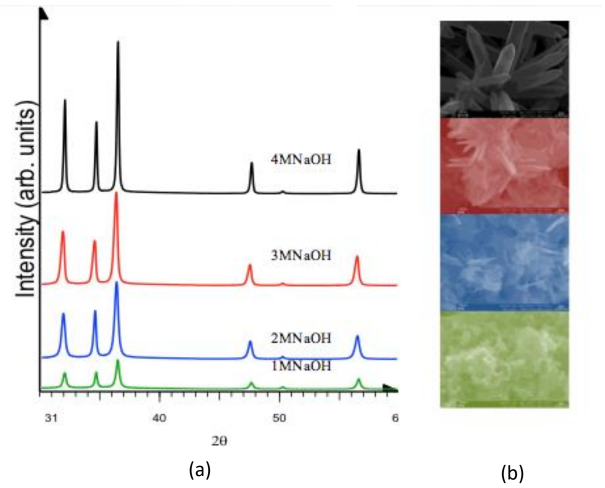

Fig. 2 XRD spectra and SEM images of the ZnO nanopowders synthesized at different molarity of NaOH.

Diffuse reflectance spectra (*R%*) was carried out to determine optical band gap (*Eg*) of the nanopowders. These spectra of the ZnO nanostructures synthesized at different molarity of ZnAc and NaOH were given in Fig. 3. As illustrated in this figure, the R_{ave} values of the nanopowders increase with increasing solution of molarity.

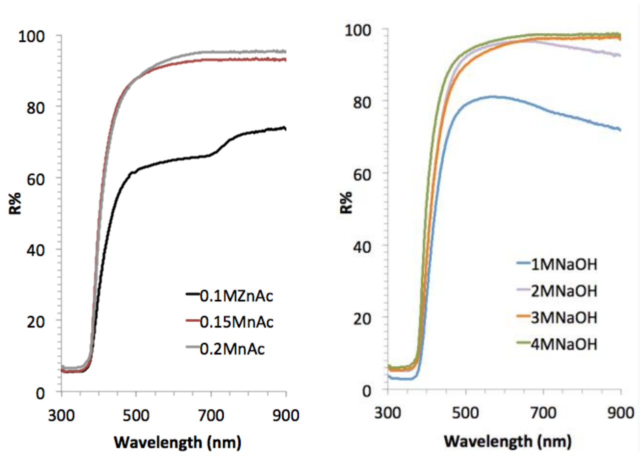

Fig. 3 R% spectra of the ZnO nanopowders synthesized at different molarity of ZnAc and NaOH.

The Kubelka Munk function (*F(R)*) was used to find the values of *Eg* [16]. The *Eg* values were determined by extrapolating the linear portion of $(F(R)h\nu)^2$ vs. hν curves (Fig 4) and given in Table 1.

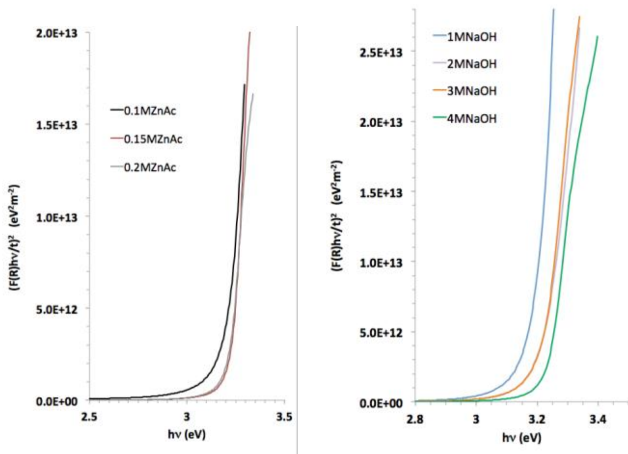

Fig. 4 $(F(R)h\nu)^2$ vs. hν graph of the ZnO nanopowders synthesized at different molarity of ZnAc and NaOH.

Table 1. The *Eg* values of the nanopowders

Nanopowder	*Eg* (eV)
0.1MZnAc	3.21
0.15MZnAc	3.23
0.2MZnAc	3.19
0.1MNaOH	3.17
0.2MNaOH	3.22
0.3MNaOH	3.22
0.4MNaOH	3.24

IV. CONCLUSION

Microwave assisted hydrothermal method was used to synthesize ZnO nanopowders. The powders were obtained with aqueous solutions prepared in different ZnAc and NaOH molarities. By using XRD measurements, the effects of ZnAc and NaOH solution on the crystal structure of ZnO nanopowder were investigated. It was found that the crystalline quality of ZnO nanopowder increased with increasing solution molarity. The surface morphology of ZnO nanopowder was analyzed by SEM. The solution molartiy has a significant effect on the surface morphology of ZnO nanopowder. The optical band gap values of ZnO nanopowder were determined by using Kubelka-Munk fraction.

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