

ALUMINUM DOPED ZINC OXIDE (AZO) NANOPARTICLE PREPARATION AND CHARACTERIZATION BY MICROWAVE SINTERING TECHNIQUE

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Abstract

This research work focus on the preparation and characterization of Aluminum doped Zinc oxide (AZO) nanoparticles by Microwave sintering technique. The nanostructure, morphology and optical properties of the synthesized AZO nanoparticles were characterized by X-ray Diffraction (XRD), Scanning Electron Microscope (SEM), Energy Dispersive X-ray (EDAX), Ultraviolet-Visible (UV-Vis) spectroscopy and Fourier Transform Infrared (FTIR) respectively. The XRD analysis exhibits that the AZO nanoparticles were in hexagonal wurtzite structure. The average crystallite size was estimated as 34.66 nm to 45.54 nm for 0.5 at.% - 1.5 at.% aluminum concentrations. The SEM micrographs showed the morphology of AZO nanoparticles. The presence of Al, Zn and O also were confirmed by EDAX analysis. The FTIR analysis exposes the composition of prepared AZO nanoparticles. The optical absorbance of 0.5 at.% - 1.5 at.% of aluminum doped nanoparticles was estimated between 354 nm and 357 nm. The prepared nanoparticles exhibit the transmittance between 88 % - 99.5 % in the visible region. The obtained energy band gap values of the prepared samples are in the range of 3.50 eV to 3.47 eV.

Keywords: AZO nanoparticles, XRD, SEM - EDAX, UV – Vis, FTIR.

1. Introduction

ZnO has n-type semiconductor property. It contributes more electrons for current conduction. ZnO has wide direct energy band gap about 3.37 eV also high binding energy (60 meV) at room temperature [1]. The large forbidden energy gap in ZnO requires more break down voltage. ZnO nanoparticles can be used in gas sensors, biosensors, catalysis, water purification, nanoelectronics, photo electronic devices, cosmetic industry, anti-corrosive coating, synthetic textiles, rubber industry, food packaging, anti-fungal ointment in health care, toothpaste, detergent and UV filter in sunscreen, Additive in the manufacture of concrete, ceramic industry. The electrical resistivity of the Zinc oxide was in the order of 0.75 MΩ at room temperature. ZnO materials having poor electrical conductivity. The conductivity of the ZnO material can be enriched by mixing the impurity of transition (group – III) materials. Zinc oxide materials can be easily doped with a group – III materials. Gallium, Indium, Boron, and Aluminum are Group - III materials. Ga, In, B, and Al are a transition as well as conductive materials. Present work, a trivalent impurity of aluminum is doped with ZnO nanoparticles with a different molar ratio. The AZO materials are suitable substitute for ITO in order to low cost, abundant in nature, low electrical resistivity and high- level transmission [2].

Several methods were available to synthesize the AZO nanoparticles such as sol-gel, spray pyrolysis, precipitation, hydrothermal method, soft chemical method, and chemical vapour deposition method. The microwave sintering technique was widely used to synthesize AZO nanorods. Microwave sintering technique draws all the attention due to its unique features like uniform volumetric heating, trouble-free, less process time and cost-effectiveness [3-6]. The AZO nanoparticles were found applications in an electric transducer, solar cell and gas sensors fabrication [7-9]. The present study has been intended to synthesize AZO nanorods by microwave sintering technique with different doping concentration. The synthesized AZO nanoparticles were characterized by XRD, SEM-EDAX, UV-Vis and FTIR studies.

2. Experimental Section

Microwave sintering technique was used for synthesizing AZO nanoparticles. The synthesizing process was shown in Figure 1. The Zinc acetate dihydrate ($\text{Zn}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$) was a host precursor and Aluminum nitrate nano hydrate ($\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$) were added as dopant precursor.

0.5 M of Zinc acetate dihydrate was dissolved in 50 ml of double distilled water by using a hot plate magnetic stirrer. The mixer was stirred for 20 minutes at room temperature to get the crystal clear solution. 0.5 at.% of Aluminum nitrate nano hydrate was mixed with this dissolved solution. Liquid ammonia also added in it drop wise to balance the pH value to 8.0. The mixed solution was stirred very strongly for 40 minutes at 80 °C. Finally, milky white coloured AZO precipitation was formed at the bottom of the beaker. The same work was done to prepare another two more aluminum doped zinc oxide nanoparticles with different molar concentration.

All the prepared precipitates were filtered separately using filter paper and washed with double distilled water. The washed precipitates were dehydrated using a microwave oven. The microwave was passed for 15 minutes at 150 °C on all samples. Moreover, the prepared samples were sintered in a microwave oven at 160 W for 30 minutes and the power ramped to 320 W for 30 minutes to evaporate the solvent.

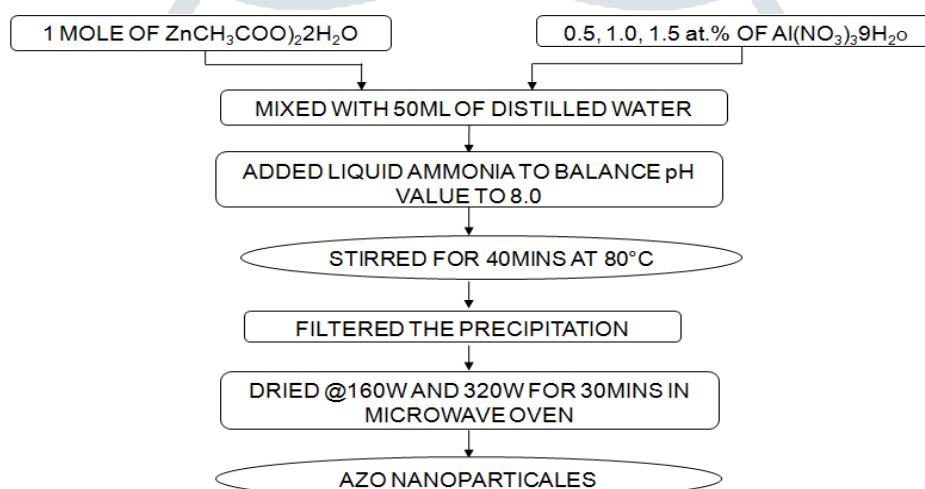


Figure 1. Flowchart shows the preparation of AZO nanoparticles

3. Results and Discussion

3.1 Structure analysis

The structure analysis of synthesized AZO nanoparticles was performed by non-destructive analytical method (XRD method) with ranges from 20°- 80°. Figures 2(a)-2(c) show the XRD patterns of synthesized AZO nanoparticles. The peaks appeared in the XRD patterns at (100), (002), (101), (102), (110), (103), (200), (112), (201), (004) and (202) for 0.5 at.%, 1.0 at.% and 1.5 at.% of alumina which was rightly matched with JCPDS data (Card no: 36-1451) and the synthesized AZO nanoparticles were in hexagonal wurtzite structure. The diffraction peak at (101) was found as the highest for all doping concentration of alumina which indicated the preferred growth orientation. There was no evidence to indicate impurities. The strength of peak intensity (count value) were decreased. It shows that the incorporation of Al dopent in ZnO. The average crystalline size of the synthesized nanoparticles was estimated using Scherrer's formula [11]. The crystalline size of the nanoparticles was 34.66 nm, 41.06 nm and 45.54 nm for 0.5 at.%, 1.0 at.% and 1.5 at.% of alumina respectively. The size of the crystalline was a function of dopent.

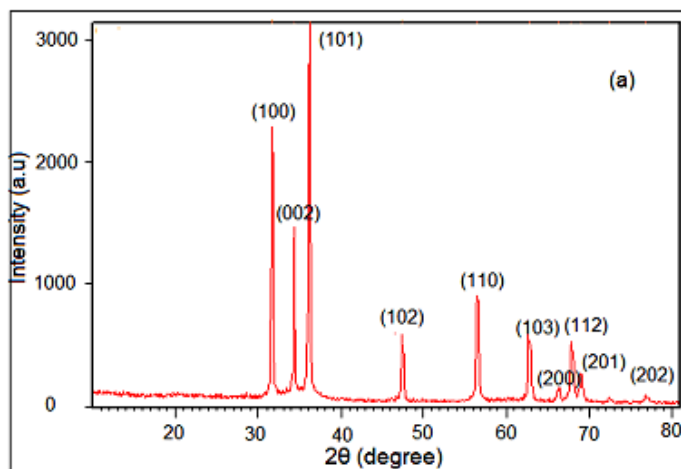


Figure 2(a). XRD Pattern of 0.5 at.% of AZO

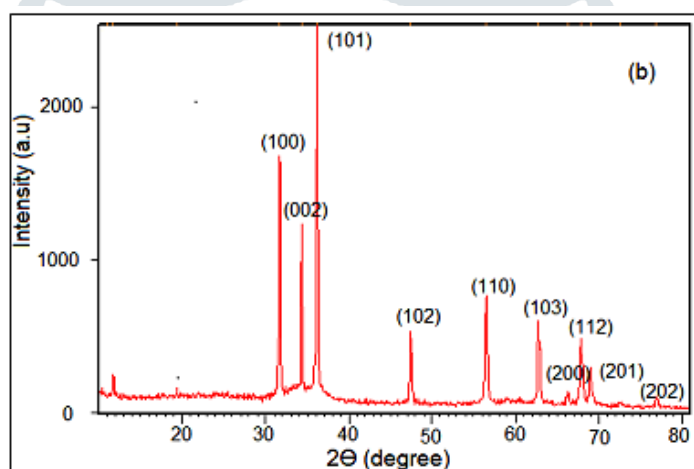


Figure 2(b). XRD Pattern of 1 at.% of AZO

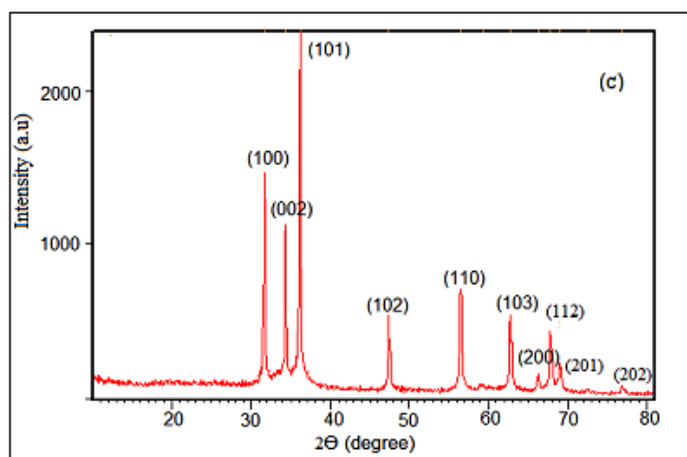


Figure 2(c). XRD Pattern of 1.5 at.% of AZO

3.2 Morphology analysis

The morphology and elemental composition of synthesized nanoparticles have been investigated through SEM analysis and EDAX spectra. Figures 3(a)-3(c) show the morphology of synthesized AZO nanoparticles. SEM micrographs depict the formation of excellent nanorods. The length and width of nanorods were not identical for all

doping concentration. Length and Width of nanorods were starts to diminished while increased the concentration of aluminum. At last excellent AZO nanorods were synthesized. Figures 4(a)-4(c) show the EDAX spectra of synthesized AZO nanoparticles. EDAX analysis identified the presence of elements such as Zn, O, and Al in synthesized nanoparticles. There were no other impurities found in synthesized AZO nanoparticles in EDAX analysis. There was more solubility of Al in ZnO for 1at.% of dopant than 0.5 at.% and 1.5 at.%.

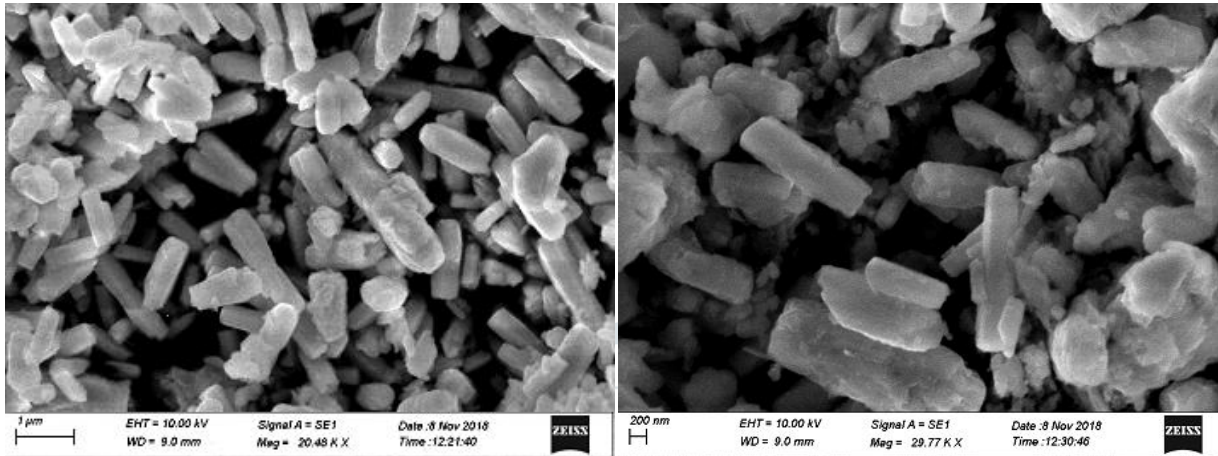


Figure 3(a). SEM image of 0.5 at.% of AZO

Figure 3(b). SEM image of 1 at.% of AZO

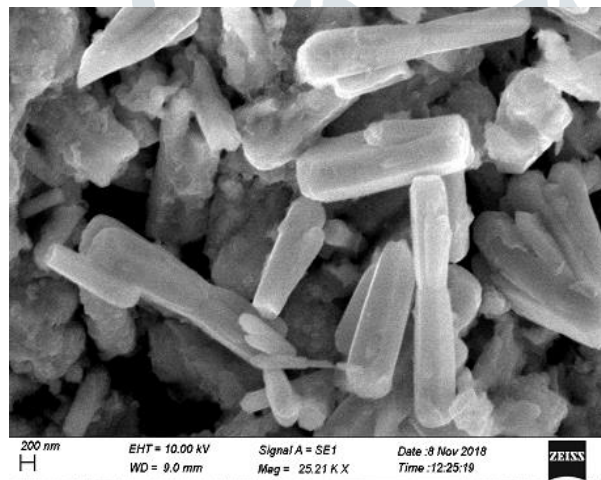


Figure 3(c). SEM image of 1.5 at.% of AZO

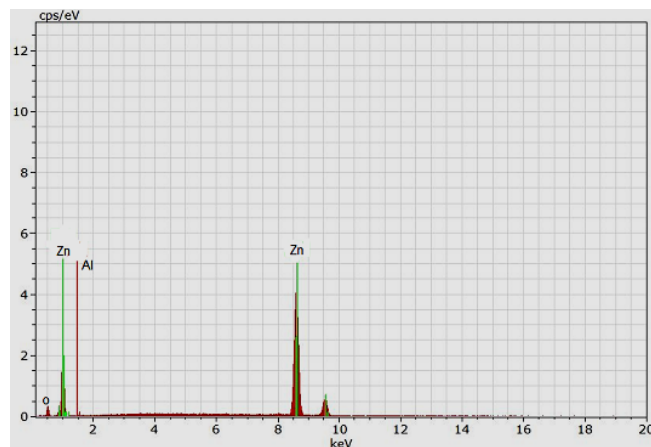


Figure 4(a). EDAX spectra of 0.5 at.% of AZO

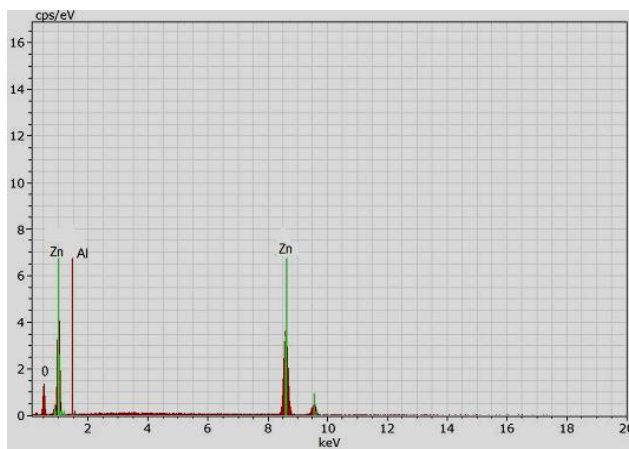


Figure 4(b). EDAX spectra of 1.0 at.% of AZO

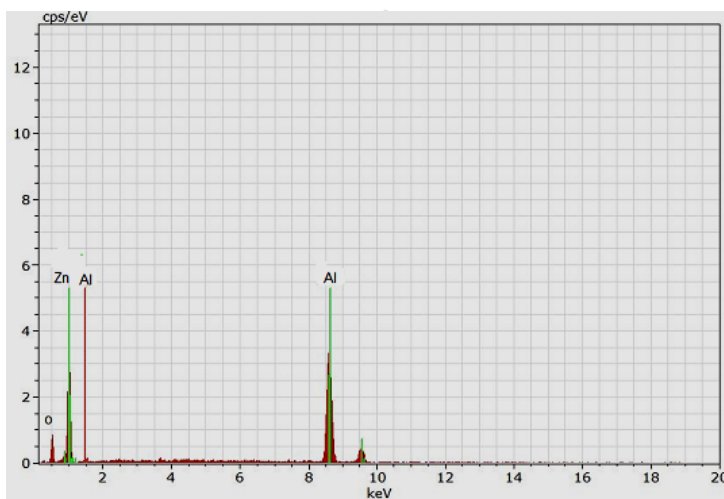


Figure 4(c). EDAX spectra of 1.5 at.% of AZO

3.3 UV-Vis Transmittance analysis

Figure 5 shows the optical transmittance spectra of synthesized AZO nanoparticles. Optical transmittance spectra of synthesized AZO nanoparticles were recorded in the wave length range from of 100 nm to 700 nm at room temperature. The optical transmittance spectra of 0.5 at.%, 1.0 at.% and 1.5 at.% of Aluminum-doped ZnO was 88 %, 99.5 % and 92 % respectively. 1.0 at.% of synthesized AZO nanoparticles had predominant transmittance spectra in the visible region.

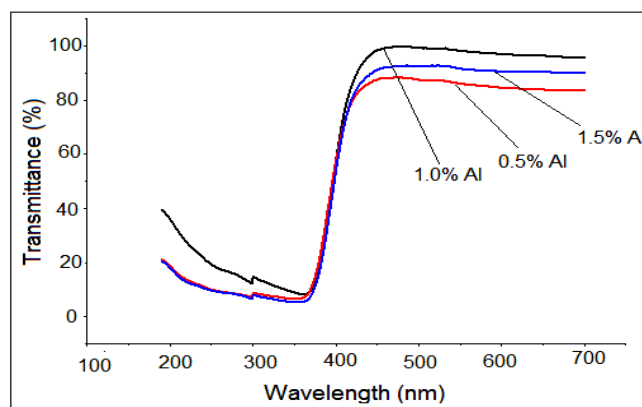


Figure 5. UV-Vis transmittance spectra of AZO

3.4 UV-Vis Absorbance analysis

The optical absorbance spectra of synthesized AZO nanoparticles were shown in Figure 6. The optical absorbance spectra of 0.5 at.%, 1.0 at.% and 1.5 at.% of Aluminum-doped ZnO was 354 nm, 362 nm, and 357 nm respectively. 1.0 at.% of alumina zinc oxide had highest optical absorbance spectra than other two. It clearly shows that there was blue shift.

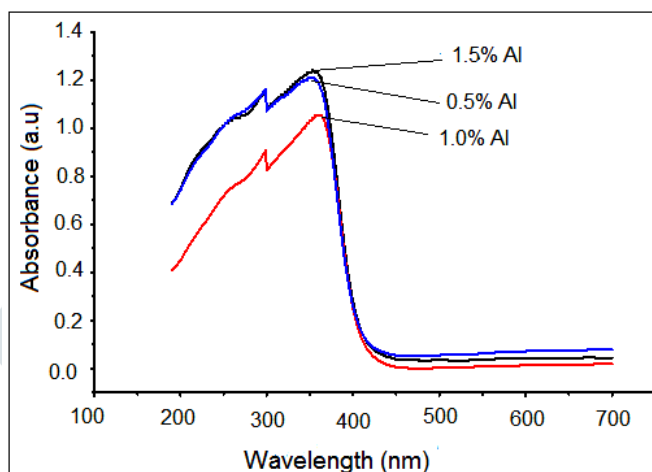


Figure 6. UV-Vis absorbance spectra of AZO

3.5 Energy gap analysis

The distance between valance and conduction band were called Energy gap. The energy gap of synthesized AZO nanoparticles was evaluated by using Energy equation of quantum mechanics. Energy gap values for synthesized AZO nanoparticles were 3.50 eV, 3.42 eV and 3.47 eV. The 1.0 at. % of AZO nanoparticles had a lower value than the other two. Therefore it requires less amount of potential difference to move the electrons as charge carries to conduction band from the valence band for current conduction. The value of optical absorption determines the energy gap. Energy gap decreases while increasing the optical absorption.

$$\text{Energy gap} = (\text{Planks constant} \times \text{Speed of light}) / \text{Wavelength} \quad (1.1)$$

$$E = (h \times c) / \lambda \quad (1.2)$$

Where

Energy (E) = Energy gap (eV)

Plank's constant = 6.626×10^{-34} joules

Velocity of light (c) = 3×10^8 m/s

Wavelength (λ) = Absorption peak value.

3.6 FTIR analysis

The FTIR analysis unveiled the chemical property of the synthesized AZO nanoparticles. Figure 6(a) shows the FTIR spectra of synthesized AZO nanoparticle. The FTIR spectra were recorded from 400 to 4000 cm^{-1} . The peak at 540 cm^{-1} corresponds to the presence of aluminum. The peak at 1500 cm^{-1} corresponds to C = O groups. The maximum peak at about 3400 cm^{-1} corresponds to hydroxyls groups.

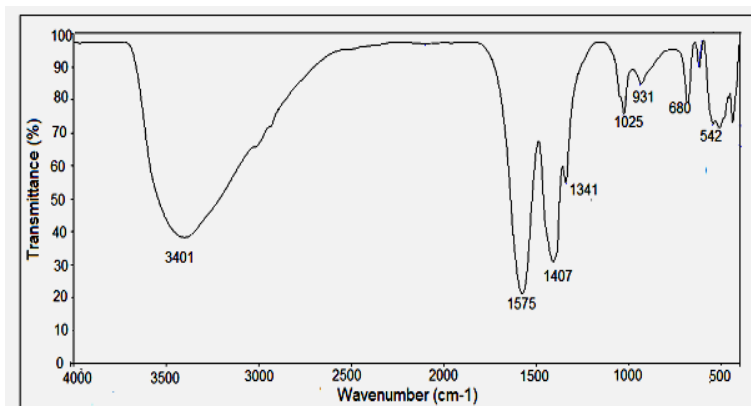


Figure 6. FTIR Spectra of 1 at. % of AZO

4. Conclusion

Excellent AZO nanoparticles were synthesized by microwave sintering technique. The synthesized nanoparticles were characterized by XRD, SEM, UV-Vis, EDAX and FTIR analysis. XRD analysis revealed synthesized nanoparticles were hexagonal wurtzite structure with an average crystallite size of 34.66 nm for 0.5 at.%, 41.06 nm for 1.0 at.% and 45.54 nm for 1.5 at.% of Alumina dopant. SEM micrographs indicated the topography of synthesized AZO nanoparticles were excellent nanorods. The optical transmittance spectra of 0.5 at.%, 1.0 at.% and 1.5 at.% of Aluminum-doped ZnO was 88 %, 99.5 %, and 92 % and absorbance spectra were 354 nm, 362 nm and 357 nm respectively. EDAX analysis proved the presence of Al, Zn, and O in synthesized AZO nanoparticles. The peak at 540 cm^{-1} in FTIR analysis confirmed the incorporation of Al in the synthesized nanoparticles. These results shown that the formation of good quality AZO nanoparticles.

References

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