Abstract

Objectives: The AZO nanoparticles are prepared by novel microwave assisted precipitation method with varying annealing temperature. Methods/Findings: The research work explores preparation and Structural, Morphological and Photoconductivity investigations of aluminum doped Zinc oxide (AZO) nanoparticles. Structural, Morphological and Optical Characteristics are examined by X-Ray Diffraction (XRD), Scanning Electron Microscope (SEM) and UV Visible Spectroscopy (UV-Vis) respectively. The XRD analysis reveals that the crystallite size was 42.3 nm, 45.2 nm and 50.7 nm for the annealing temperatures of 450 °C, 600 °C and 750 °C. The shape of the nanoparticles observed in hexagonal wurzite format. The maximum level of peaks is observed in (100), (002), (101), (102), (110), (103), (200), (112), (201), (004) and (202) planes for various annealing temperatures. The SEM image indicates that the morphology has rod shape structure. The optical absorbance spectra of AZO nanoparticles were about 384.00 nm. The FTIR spectra shows the presence of aluminum content in the synthesized nanoparticles. Applications: The prepared AZO nanoparticles can be applied to prepare the gas sensors.

Keywords: Nanorods, AZO, EDAX, SEM, FTIR, XRD

1. Introduction

ZnO is an N type semiconductor having direct energy band gap (3.37 eV) and high binding energy (60 meV)\(^1\). ZnO nanoparticles can be used in sensors, catalysis, water purification, antibacterial, nano electronics, solar energy, cosmetics, paints, synthetic textiles, food packaging, medical care, healthcare, tooth paste, detergent and sun screen. At room temperature the electrical resistivity of the Zinc oxide was in the order of 0.75 MΩ. The resistivity of the ZnO material can be reduced by doping with group – III materials. Zinc oxide materials can be easily doped with group – III material such as Ga, In, B and Al\(^1\). In this work, aluminum is doped with ZnO nanoparticles. AZO is a valuable alternative to ITO materials. It has increased optical transmittance and low electrical resistance\(^2\).

Synthesized AZO nanoparticles can be prepared by sol gel, spray pyrolysis, precipitation, hydrothermal method, chemical vapor deposition and microwave irradiation. Extensively the microwave irradiation method is used to synthesize the AZO nanoparticles. Microwave irradiation method draws all notice owing to its homogeneous volumetric heating, simple and economical method\(^3\). The prepared AZO nanoparticles having peculiar optical property and also it has predominant application in electric transducer, solar cell and gas sensors\(^3\).
2. Experimental Section

Figure 1 shows the synthesized producer of AZO nanoparticles for different annealing temperature. In this synthesized method the aqueous solution of Zinc acetate dihydrate. \((\text{Zn(CH}_3\text{COO)}_2\cdot2\text{H}_2\text{O})\) is an precursor and Aluminum nitrate nano hydrate \((\text{Al(NO}_3)_3\cdot9\text{H}_2\text{O})\) was a source of aluminum. 1.0 M of Zinc acetate dihydrate was dissolved in 100 ml of double distilled water by using hot plate magnetic stirrer. 1 at % of aluminum nitrate nano hydrate was mixed with this dissolved solution. Liquid ammonia also added in to it drop wise to adjust the Ph value to 8.0. The mixed solution was stirred vigorously for 40 min at 80 °C. The prepared solution was allowed to get cool at room temperature. At last white colored AZO precipitate formed at the bottom of the beaker. The same method was followed to prepare two more aluminum doped zinc oxide nanoparticles with same molar concentration.

All three white colored prepared precipitates were filtered separately using whatman filter paper and was washed for four times using double distilled water and ethanol. The washed precipitates were dried using microwave oven to evaporate water molecules. The microwave irradiation was performed in convection method for 10 min at 150 °C. Moreover, it was placed in muffle furnace for 4 hours at 450 °C, 600 °C and 700 °C to dry the synthesized nano particles again. The dried synthesized nanoparticles have been powered using mortar and pestle.

3. Results and Discussion

3.1 Structural Analysis

The structural analysis of 1 at % of synthesized AZO nanoparticles for different annealing temperature were examined in X-RD pattern (XPERT- PRO, 30mA, 40kV) with range from 20-80 degree. Figures 2(a), (b) and (c) shows the XRD pattern of synthesized AZO nanoparticles. We found that the maximum peaks at \((100), (002), (101), (102), (110), (103), (200), (112), (201), (004)\) and \((202)\) for 450 °C, 600 °C and 750 °C which were matched with [Card no : 36 – 1451] of AZO material. Among these intense peaks appeared at \((101)\) for all the annealing temperature.

It shows that the synthesized AZO nanoparticles were in hexagonal wurtzite structure also perfect orientation. The average size of the synthesized nanoparticles is calculated by Scherrer formula. The XRD analysis reveals that the crystallite size was 42.3 nm for 450 °C, 45.2 nm for 600 °C and 50.7 nm for 750 °C.

\[ D = \frac{K \lambda}{\beta \cos \theta} \]

where \(D\) is the crystalline size of AZO nano particle
\(\lambda\) represent the wavelength of x-ray source 0.1541 nm used in XRD
\(\beta\) is the full width at half maximum of the diffraction peaks
\(K\) is the Scherrer constant with a value from 0.9 to 1 and \(\theta\) is the Bragg angle

3.2 Morphological Analysis

The morphology and chemical composition of synthesized nanoparticles has been studied by using Scanning Electron Microscope. Figures 3 (a), (b) and (c) shows the morphological structure of synthesized AZO nanoparticles. From the SEM analysis morphology of
synthesized nanoparticles were rod shape. At 450 °C, the nanorods were more agglomerated. But at 600 °C and 750 °C, the synthesized nanorods were separated with each other. Clear nanorods appeared when the annealing temperature increased. Figure 4 shows the EDAX spectra of 1 at % AZO nanoparticles for 600 °C. It confirms that the synthesized nanoparticles contain Zn, O, Al and no other impurities.

![Figure 2](image2.png)

(a) X-RD Pattern of 1 at % of AZO nanoparticle for 450 °C, (b) X-RD Pattern of 1 at % of AZO nanoparticle for 600 °C, (c) X-RD Pattern of 1 at % of AZO nanoparticle or 750 °C.

![Figure 3](image3.png)

(a) SEM image of 1 at % of AZO for 450 °C, (b) SEM image of 1 at % of AZO for 600 °C, (c) SEM image of 1 at % of AZO for 750 °C.

![Figure 4](image4.png)

EDAX Spectra of 1 at % of AZO for 600°C.

### 3.3 UV-Vis Absorption Technique

Figure 5 shows the optical absorbance spectra of synthesized AZO nanorods. The optical absorbance spectra of 1 at % of Aluminum doped ZnO for annealing temperature 600 °C analyzed by UV-Vis spectrometer in the wave length range from 200 – 800 nm. The maximum peak found at 384.00 nm.
Impact of Annealing Temperature on Structural, Morphological, Photoconductivity of Aluminum Doped Zinc Oxide (AZO) Nanorods Prepared by Microwave Assisted Precipitation Method

3.4 FTIR Analysis

Fourier Transform Infra Red Spectra reveals the chemical property of the synthesized nanoparticleless. Figure 6 shows the FTIR spectra of 1 at % of AZO for 600 °C. The FTIR spectra were recorded from 500 to 4000 cm⁻¹. The absorbance peaks at 560 cm⁻¹ and 860 cm⁻¹ represents the presence of Al. The peak at 1500 cm⁻¹ corresponds to C=O groups. The peaks about 2400 cm⁻¹ corresponds to C=H groups. The maximum peaks between 3000 cm⁻¹ and 3500 cm⁻¹ corresponds to hydroxyls groups.

4. Conclusion

AZO nanoparticles can be prepared by microwave assisted precipitation method. The prepared nanoparticles were analyzed by XRD, SEM, UV-Vis, EDAX and FTIR method. The synthesized nanoparticles characterized by XRD analysis shows that the synthesized particles are wurite shape and size is 42.3 nm for 450 °C , 45.2 nm for 600 °C and 50.7 nm for 750 °C. The SEM analysis shows that morphology of AZO using microwave assisted precipitation method having nonorods. The UV-Vis study also shows that absorbance spectra were 384.00 nm. The FTIR spectra confirmed that the presence of Al, Zn and O in synthesized nanoparticles.

5. References

1. Khan W, Khan Z, Saad AA, Shervani S, Saleem A, Naqvi AH. Synthesis and characterization of Al doped ZnO nanoparticles. World Scientific. 2013; 22:630–6. https://doi.org/10.1142/S2010194513010775
2. Krishnakumar T, Jayaprakash R, Pinna N, Singh VN, Mehta BR, Phani AR. Microwave - assisted synthesis and characterization of flower shaped zinc oxide nanostructures. Materials Letters. 2009; 63:242–5. https://doi.org/10.1016/j.matlet.2008.10.008
3. Kiruthiga A, Krishnakumar T, Synthesis and characterization of microwave-assisted ZnO nanostructures. Chemistry Tech Research. 2015; 8(7):104–10.
4. Rai P, Song H-M, Kim Y-S, Song M-K, Oh P-R, Yoon J-M, Yu Y-T. Microwave assisted hydrothermal synthesis of single crystalline ZnO nanorods for gas sensor application. Materials Letters. 2012; 68:90–3. https://doi.org/10.1016/j.matlet.2011.10.029
5. Lupan O, Chow L, Shishiyanu S, Monaco E, Shishiyanu T, Sontea V, Roldan Cuenva B, Naitabdi A, Park S, Schulte A. Nanostructured zinc oxide films synthesized by successive chemical solution deposition for gas sensor applications. Materials Research Bulletin. 2009; 44:63–9. https://doi.org/10.1016/j.materresbull.2008.04.006
6. Hjiri M, El Mir L, Leonardi SG, Pistone A, Mavilia L, Neri G. Al-doped ZnO for highly sensitive CO gas sensors. Sensors and Actuators B: Chemical. 2014; 196:413–20. https://doi.org/10.1016/j.snb.2014.01.068