

Structural and Morphological Analysis of Aluminum (Al) Doped Tungsten Oxide (WO₃) Nanoparticles

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Abstract: Al doped WO₃ nanoparticles have been successfully synthesized by the solvo thermal cum chemical method. Al doped WO₃ nanoparticles is prepared at different temperature like room temperature, 100°C and 400°C respectively. The nanoparticles have been characterized by a number of techniques as x-ray diffraction (XRD), scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDS). The XRD result suggested that the nano particles have a polycrystalline spherical structure with well developed crystalline. The SEM results noted that the agglomeration increases with increasing temperature

1. Introduction:

Current research is moving towards the use of small-band-gap photo catalysts that can promote photo catalytic reactions using ultraviolet and also visible light which is about 45% of the terrestrial solar energy [1-3]. Tungsten oxide (WO₃) is a semiconductor oxide material with a band-gap of 2.6–3.0 eV [4], and it is becoming the focus of research attention due to its unique electronic properties. In particular, various WO₃ nanostructures (nanoparticles, nanoplatelets, nanorods, and nanowires) are of special interest as promising candidates for photo catalyst [4], electrochromic devices [5–7], and gas sensors [8, 9] because of their high surface area and novel properties [10].

Many physical properties of WO₃, such as piezoelectricity, electrical conductivity and defect structures, are greatly influenced by the presence of impurities. Several dopants such as Fe, Cr, Al, Cu, Co, Mn, Mg, S, P, N etc. can lead to an increase in the surface area of the WO₃ based nano powders. Doping with other elements can greatly improve the performance of Metal oxide [11]. Among these doped metal oxide materials, aluminum (Al) doped WO₃ (AWO₃) nanoparticles have received considerable attention [12-16]. And also have the advantage of abundant and cheap raw materials. However, the application range of Al doped WO₃ nanoparticles have been limited by the limitation of particles characteristics.

The present investigations are aimed at the characterization of Al doped WO₃ nano particles synthesized by solvo thermal cum chemical method. The influence of Al content on the structural and morphological properties is discussed in this paper.

2. Experimental

Tungsten chloride (Sigma Aldrich 99.99%) and Cyclohexanol were used as a precursor. 50 mg of Tungsten chloride was slowly dissolved in 25 ml of Cyclohexanol to obtain a uniform solution with the help of

magnetic stirrer. Then the solution was centrifuged and washed with distilled water until to reach neutral pH of the solution. After evaporation we got WO₃ nanoparticles. To prepare Aluminum (Al) doped WO₃ nanoparticles, we used the paste preparation method. In paste preparation method, the paste was produced by mixing of 2.0 g of Al anatase powders and 5 g of WO₃ nanoparticles with a mixture consisting of 10.0 g of α -terpineol, 1 g of cellulose, and 25 ml of ethanol, which was solicited for 48 hrs at 1200 Wcm⁻². By using the prepared paste thin films were prepared by coating the paste on a FTO conducting glass plate (Hartford FTO, ~30 Ω cm⁻², 80% transmittance in visible region) using the doctor blade technique. The Al doped WO₃ is prepared at different temperature like room temperature, 100°C and 400°C respectively.

X-ray diffraction method using CuK α radiation has been used to study the structure of the synthesized nanoparticles. This study was carried out by employing a Bruker Axs D8 Advance X-ray diffractometer with CuK α ($\lambda=1.5406$) radiation using a tube voltage and current of 40kv and 30mA respectively. The sample was scanned from 10°-80° in 2 θ with step size of 0.5° 2 θ and scan speed of 0.5°2 θ per second. Surface morphology of the synthesized nanoparticles was studied using scanning electron microscopy (SEM; Philips XL40), and the atomic compositions of the nanoparticles was measured by energy dispersive X-ray analyses (EDXA; Inca, oxford instruments) operated at 120 kV.

3. Results and Discussion.

3.1 X-ray diffraction analysis

X-ray diffraction pattern has been used to investigate the phase of the synthesized Al doped WO₃ nanoparticles. The X-ray diffraction pattern of Al doped WO₃ nanoparticles prepared at 100°C is shown in Figure 1.

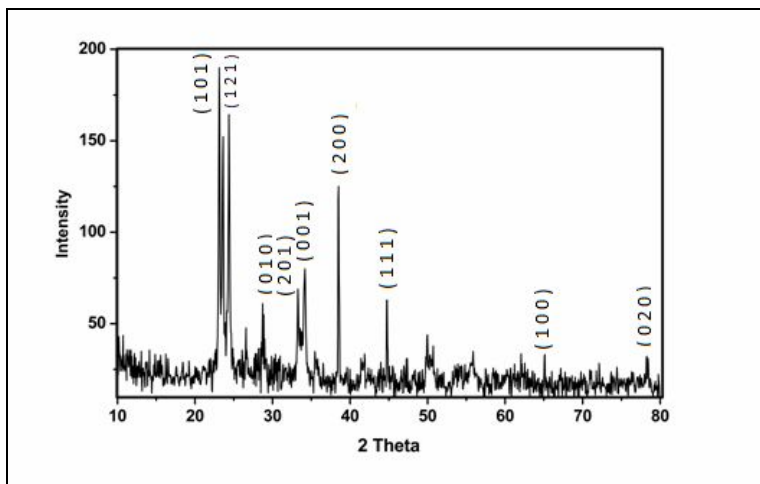


Figure 1 X-ray diffraction pattern of Al doped WO₃ at 100°C

Figure 1 show the XRD patterns of Al doped WO₃ nanoparticles prepared at 100°C in the 2 θ range 10–80 degree. The patterns were indexed using origin software and all peaks were well matched with hexagonal structure of WO₃ using the standard data (JCPDS No. 05-0392). Here, no other impurity peaks were observed in the XRD patterns which show the purity of the nanoparticles formation. This result suggested that the nano particle have a polycrystalline spherical structure with well developed crystallinity. The average crystallite sizes (D) was calculated using well known Scherer's equation

$$D = 0.9\lambda / (\beta \cos \theta)$$

Where λ , β and θ are the x-ray wavelength (0.154 nm), full width at half maxima (FWHM) of the diffraction peak and the Bragg's diffraction angle, respectively. Crystallite size of each sample was calculated using most intense peak (101) of Al doped WO₃ nanoparticles (not shown). When more number of Al³⁺ enters into the lattice sites in the place of W²⁺, the lattice distortion is indentified which results in larger strain and consequently it affects the normal growth of WO₃ crystals. This may be the cause for the average crystallite size of Al doped WO₃ to be reduced to 27 nm.

3.2 SEM and EDS analysis

The scanning electron microscope images of the Al doped WO₃ nanoparticles are shown in Figure 2.

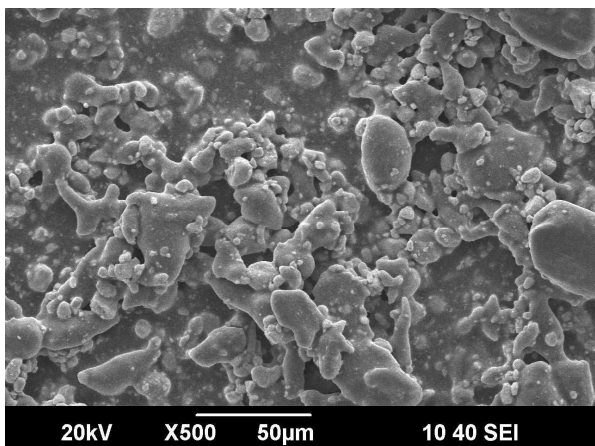


Figure 2 SEM images of Al doped WO₃ nanoparticles at 100°C

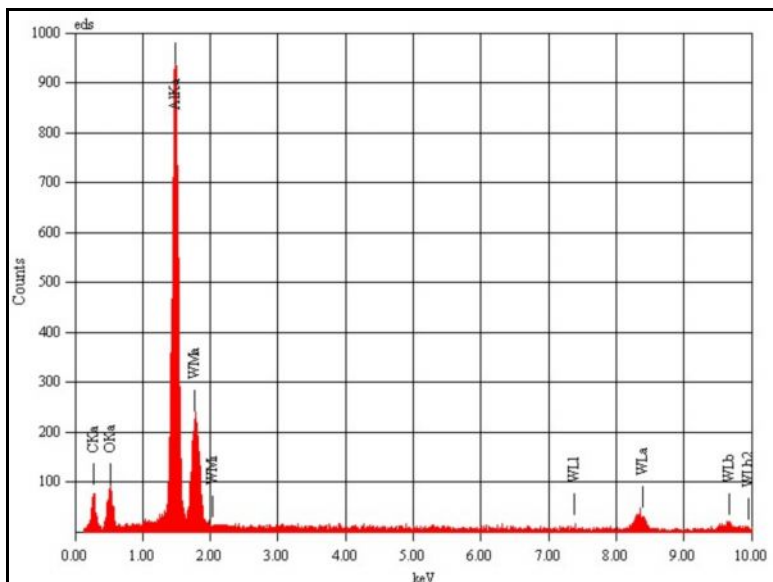


Figure 3 Energy dispersive x-ray analysis (EDS) pattern of the Al doped WO₃ nanoparticles at 100°C.

The morphological and structural studies for Al doped WO₃ nanoparticles at 100°C were investigated using scanning electron microscopy and displayed in Figure 2. These micrographs exhibited the formation of nanoparticles of Al doped WO₃. The all fine nano particles is agglomerated and spherical like in shape. It was noted that the agglomeration increases with increasing temperature. To check the chemical composition of the material, an energy dispersive X-ray (EDS) spectroscopy analysis was performed. Figure 3 shows the EDS spectra of Al doped WO₃ nanoparticles at 100°C, which confirms the synthesis of Al doped WO₃ nanoparticles at 100°C. The surface of the WO₃ nanoparticles also exhibited elements of C, O, W and Al. All the carbon had been removed during the sintering process. The tungsten element is found in large that are reliable with the concentration of XRD patterns and SEM micrographs.

4. Conclusion

Al doped WO₃ nanoparticles were synthesized by solvo thermal cum chemical method. The structural and morphological properties of Al doped WO₃ nanoparticles have been investigated. The XRD result suggested that the nano particle have a polycrystalline spherical structure with well developed crystalline. The SEM results noted that the agglomeration increases with increasing temperature. The EDS confirms the formation of all Al, W, and O.

5. References.

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