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Phase formation and structural studies of tungsten oxide wo$_3$–al nanoparticles for future photocatalytic applications

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Abstract
Creation and analysis of Aluminum-doped WO_3 nano-sized materials, made using microwave heating (2.45 GHz, 180 W for 10 minutes). The Aluminum-doped $WO_3.H_2O$ samples all showed a crystalline structure, with the orthorhombic shape being the most common. However, when the samples were heated to anneal, both the pure and doped samples exhibited a monoclinic structure. These findings suggest that the dopant plays a crucial role in stabilizing the oxygen content and adjusting the crystal structure of the final products.

Keywords: Tungsten oxide, Microwave irradiation, photocatalytic activity, Aluminium doped metal oxide, semiconducting metal oxides

Introduction

In recent years, nanomaterials have garnered significant attention in research due to their distinctive properties when compared to bulk materials, as well as their current and potential applications across a diverse range of fields, including information storage, electronics, sensors, structural components, and catalysis, among others [1]. In recent years, nano crystalline materials based on tungsten oxide have garnered significant interest due to their exceptional electrical, optical, mechanical, and gas sensing characteristics. Beyond their ability to withstand high temperatures, tungsten is also of commercial significance for applications that depend on high density and elastic modulus. The incorporation of certain metallic ions enhances the functional properties of tungsten oxide, particularly in terms of mechanical, gas sensing, and optical performance [2]. Tungsten oxide nanoparticles are synthesized using a range of techniques suitable for industrial applications. These methods include the microwave hydrothermal approach [3]. Surfactant-mediated technique [4]. Sol-gel process [5]. Acidification method [6]. Precipitation method [7]. electrodeposition [8]. Laser ablation method [9] and ball milling [10]. Additionally, these nanoparticles exhibit antifungal activity [11]. Environmentally friendly nanoparticles that exhibit durability over time. [12].$W_{18}O_{49}$ Nano crystals exhibit a wide absorption spectrum that encompasses both visible light and near-infrared wavelengths. [13]. Examine the photocatalytic efficacy of the SA/PVP/WO_3 nanocomposite [14]. Co-Precipitation Technique ($Fe_{3O4)}$ [15]. The successful commercialization of MONPs greatly depends on the ability to achieve well-controlled properties during the synthesis process [16]. In reaction to this request, we devised a straightforward electrospinning method for the synthesis of a porous tungsten oxide nano framework integrated with graphene fibers [17]. The characteristics of oxygen vacancies in WO3 nanofibers [19]. The presence of aluminum and copper enhances the photocatalytic activity [20]. The processes of dehydrogenation and oxidation [21]. The variety in structure and the transitions between crystal phases of WO3-x. [22]. Significant opportunities for chemical transformation exist [23]. The process of water splitting activity [24]. The enhanced surface area of the WO_3-nCdS nanocomposite contributes to its photoelectrochemical performance [25]. The photocatalytic process effectively eliminates pollutants from water [26]. The crystalline structure's ammonia concentration is influenced by photo-thermal interfacial evaporation [29]. Coating method (PEQ) [30]. Possible optoelectronic applications of nanoparticles on aluminum through plasma electrolytic oxidation [31]. The synthesis of trioxide (WO_3) was achieved through a hydrothermal method [33].

Advancements in the area of heterogeneous visible-light photocatalysis have been noted, particularly in the plasma processing technique utilizing aluminum nitrate [35]. These nanoparticles can be produced using conventional techniques, including both chemical and physical methods [36]. The method of deposition through chemical bath techniques [37]. The compounds that facilitate magnetic recoverability are based on Aluminum Ferrite in this study [38]. This indicated that the use of an efficient nano Al_2O_3 adsorbent resulted in an increase of nearly two times [39]. The optical analysis revealed that the introduction of dopants results in a red shift of the band gap [40]. The methods outlined previously are both cost-effective and require a more time-intensive process, which will be taken into consideration moving for [41]. In order to address the challenges associated with the synthesis of semiconductor oxide-based nanomaterials, there is a necessity for the development of methods that are both cost-effective and efficient in terms of time and space utilization. A key objective of the current invention is to introduce an innovative approach for synthesizing semiconducting metal oxide-based nanomaterials that minimizes costs and reduces synthesis time. The inventors have undertaken comprehensive research to tackle the aforementioned issues related to the synthesis of tungsten oxide-based nanomaterials. In light of the existing circumstances, they have successfully identified a straightforward method for producing oxide-based nanomaterials. The applications of nanoparticles are rapidly expanding across various domains, including electrochemical sensors, biosensors, medicine, targeted drug delivery, healthcare, agriculture, and wastewater treatment. Due to their higher surface area, nanomaterials, particularly nanocatalysts, exhibit remarkable surface activity. For instance, the reaction rate of nano-aluminum can be so elevated that it is employed as a solid fuel in rocket propulsion, in contrast to bulk aluminum, which is commonly used in cookware. Nano-aluminum's heightened reactivity enables it to generate the necessary thrust for launching payloads into space. Likewise, the effectiveness of catalysts in either accelerating or inhibiting reaction rates is contingent upon their surface activity, which can be effectively utilized to influence the rate-controlling steps of reactions. In the microwave irradiation method, the synthesis time was approximately 10 minutes, and the reaction process was notably straightforward.

Materials and Methods

This article discusses how nanomaterials were created and analyzed to understand their structure, optical properties, and microscopic characteristics for potential use in photo catalytic applications. The study introduces a new material, both in its pure form and doped with "Al" at different levels, as a potential option for photo catalysis. The researchers used a simple microwave method to create nano-sized tungsten oxide (WO_3) powders with varying levels of aluminum doping. They dissolved 4.98 grams of tungstic acid (H_2WO_4) in 20 ml of sodium hydroxide (NaOH) with a one molar ratio to achieve this. The yellow solution was stirred for 20 minutes to make sure it was uniform. This yellow hydrated sodium tungstate solution was created through a process called proton exchange. Additionally, a 2% solution of Aluminum Sulfate was made using deionized water and mixed with the sodium tungstate solution, then stirred for 30 minutes. The two solutions were mixed slowly together and stirred again for 30 minutes. The pH of the solution was neutral, but was adjusted to one by adding a few drops of Hydrochloric acid to help with precipitation and provide a suitable environment for the final products to take on a specific shape. To help with the microwave irradiation process, 5 ml of double distilled water was added. The final solution was then placed in a household microwave oven with a frequency of 2.45 GHz and a power of 900 W. Microwaves were used to control the shape and size of products. The difference in microwave extinction coefficient between the solvent and reactant, along with the values of dielectric constant, played a key role. Considering the significance of using microwave irradiation (MWI) instead of traditional heating methods, the conditions in the microwave oven were adjusted to 240 W for 10 minutes at room temperature to synthesize nano-sized particles. A yellow-colored precipitate was produced after the microwave irradiation process. The resulting powders were then heated in a muffle furnace at 550°C for 6 hours in normal conditions to eliminate any by-products and enhance the crystallinity.

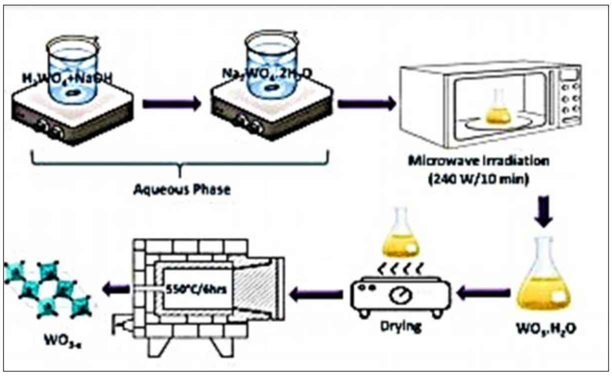


Fig 1: Schematic representation of the preparation by microwave irradiation method

Results and Discussion

Powder XRD analysis

The present result analysed Fig. 1.3.1 (a) and (b) shows the respective diffraction pattern for Pristinedemonstrate the diffraction pattern for both the original and metal-doped tungsten anhydride samples after annealing. The results indicate the formation of the orthorhombic phase of tungsten anhydride with a high level of crystallinity, matching JCPDS 43-1035.A noticeable change in the diffraction pattern of the doped sample suggests the presence of dopant ions within the lattice of WO_3, likely due to the larger atomic size of the dopantions. Additionally, the dopants contribute to adjusting the crystalline nature of the final products. The average crystallite size of the samples was determined using Scherrer's equation based on the XRD peaks in the diffraction pattern. The corresponding average crystallite size of the prepared samples was calculated from the diffraction pattern for corresponding XRD peaks according to using Scherrer's equation.

$$D = K\lambda / \beta \cos \Theta$$

Where

D - Average crystallite size in nanometer
λ - Wavelength of the X-ray radiation (10^{-10} m)
K - Constant
β - Full width at half maximum intensity
Θ - Half diffraction angle

All samples was calculated and they were given a tetragonal system,

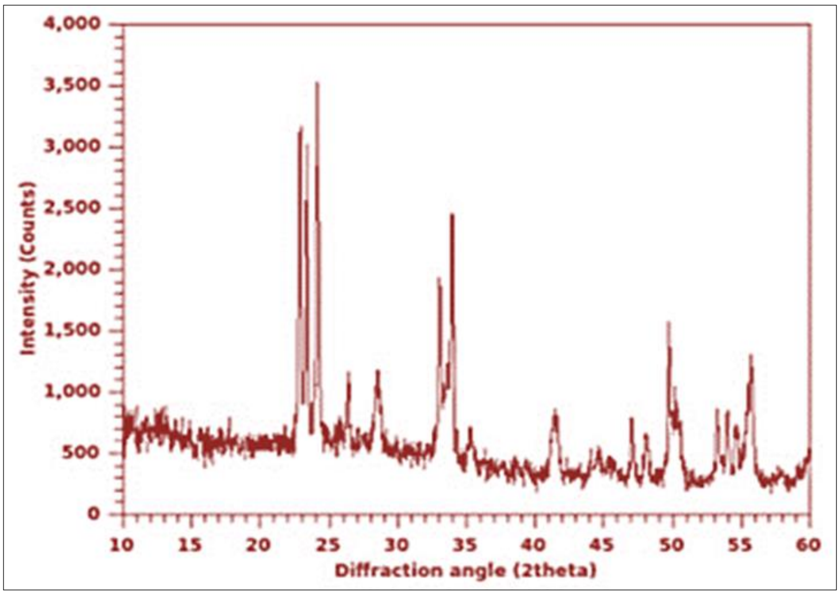
$1/d^2 = (h^2 + k^2/a^2) + (l^2/c^2)$

Eg – Band gap

n - Direct the type of transition

Where
a, c -The Lattice parameters
d –The Interplaner angle

The samples calcined at 600^{0}C in an ambient atmosphere with orthorhombic and monoclinic phases which is highly favorable for photo catalytic activity.



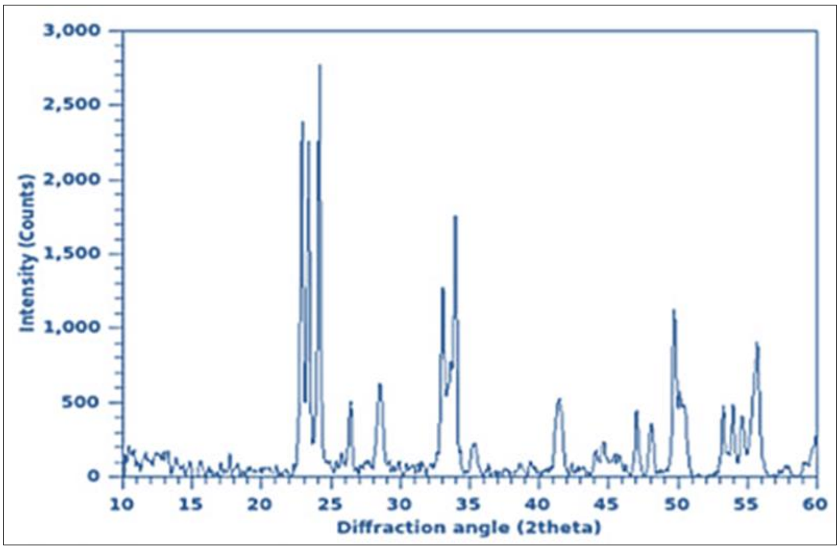


Fig 2: a) wo_3 without doping b) wo_3 with doping

Conclusions

The synthesis and characterization of pure, doped a hydrated tungsten oxide (WO_3) nano dimensional particles for photocatalytic applications with novel approach for the first time. The work successfully declares the synthesis and characterization of Aluminium doped tungsten anhydride nanomaterials prepared by using household microwave irradiation techniques for photocatalytic applications. The corresponding powder diffraction using X-ray as a source confirmed the phase formation of the end products with orthorhombic and monoclinic phases respectively for anhydride and hydride tungsten oxide nanoparticles in the case of both pure and doped.The microscopic analysis exhibited the contribution of dopant while changing its morphology during the synthesis process.

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