

Hydrothermal Method of Synthesis of β -Ag₂MoO₄ Nanorods as a Peroxidase Mimetic for Glucose Sensing

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Abstract

In this study, β -Ag₂MoO₄ nanorods were synthesized using a hydrothermal method, aiming to explore their potential as a sensing material for L-glucose detection. The synthesis process was optimized to produce uniform nanorods with high crystallinity and surface area, which is essential for enhancing sensor performance. The synthesized β -Ag₂MoO₄ nanorods characterized by X-ray diffraction (XRD) and scanning electron microscopy (SEM), confirmed the successful formation of β -Ag₂MoO₄ nanorods with desired morphological features. The vibrations existing in β -Ag₂MoO₄ nanorods were characterized by FT-IR and Raman spectroscopic studies. The electrochemical properties of the nanorods were evaluated, demonstrating their sensitivity and selectivity towards L-glucose. The nanorods exhibited a rapid response time, low detection limit, and excellent reproducibility, making them promising candidates for glucose sensors. This work underscores the potential of hydrothermally synthesized β -Ag₂MoO₄ nanorods in the development of advanced glucose sensing platforms, offering a new approach for the efficient and reliable monitoring of glucose levels in medical diagnostics.

Keywords: β -Ag₂MoO₄ Nanorods, Biomimetics, Glucose Sensing, Hydrothermal.

Introduction

The synthesis of nanostructured materials has garnered significant attention in recent years due to their unique properties and wide range of applications, particularly in the fields of catalysis, electronics, and sensing technologies[1–3]. Among the various nanostructures, metal oxides have emerged as a prominent class of materials owing to their exceptional chemical stability, high surface area, and tunable electronic properties[4]. Silver molybdate (Ag₂MoO₄) is one such metal oxide that has demonstrated potential in various applications, including photocatalysis, antimicrobial activity, and sensor development [5, 6].

The hydrothermal method is a well-established technique for the synthesis of nanomaterials, offering several advantages over other methods, such as low-temperature synthesis, control over particle size and morphology, and the ability to produce highly crystalline materials. This method involves the use of a high-pressure, high-temperature aqueous environment to facilitate the nucleation and growth of nanomaterials [7, 8]. The versatility of the hydrothermal method allows for the fine-tuning of synthesis parameters, such as temperature, pressure, reaction time, and precursor concentration, enabling the production of nanostructures with

specific properties tailored for desired applications [9–11].

In the context of sensor technology, the detection of biomolecules, particularly glucose, is of critical importance due to its relevance in medical diagnostics and disease management. Glucose monitoring is essential for the management of diabetes, a chronic disease characterized by elevated blood glucose levels. Traditional glucose sensors are enzyme-based, relying on glucose oxidase to catalyze the oxidation of glucose. However, enzyme-based sensors offer the potential for improved stability, lower cost, and enhanced performance [12, 13].

β - Ag_2MoO_4 nanorods present a promising alternative for the development of non-enzymatic glucose sensors. The unique properties of β - Ag_2MoO_4 , such as its high surface area, excellent electrical conductivity, and catalytic activity, make it an ideal candidate for glucose sensing [14–16]. The nanorod morphology, in particular, offers a high aspect ratio and large surface-to-volume ratio, which are advantageous for sensor applications as they provide more active sites for glucose adsorption and oxidation. Moreover, the hydrothermal synthesis of β - Ag_2MoO_4 nanorods allows for precise control over the nanostructure, enabling the optimization of sensor performance [10].

In this study, we focus on the hydrothermal synthesis of β - Ag_2MoO_4 nanorods and their application in the sensing of L-glucose, an important stereoisomer of glucose. L-glucose, although not naturally occurring in significant amounts in the human body, is of interest in various research areas, including as a potential therapeutic agent and in studies of glucose metabolism. The ability to detect L-glucose with high sensitivity and selectivity is, therefore, valuable in both medical and research contexts.

The objectives of this research are to optimize the hydrothermal synthesis parameters to produce uniform, high-quality β - Ag_2MoO_4 nanorods and to characterize the

structural and morphological properties of the synthesized nanorods using various analytical techniques. Then the performance of β - Ag_2MoO_4 nanorods as a sensing material for L-glucose detection was examined to advance the development of enzymatic glucose sensors and contribute to the broader field of nanomaterial-based sensor technologies.

Experimental Section

Chemicals

Silver Nitrate and Sodium molybdate are obtained from Sigma-Aldrich. Sodium hydroxide, o-phenylene diamine, glucose oxidase, L-glucose, Ethanol are purchased from SRL Chemicals Pvt Ltd. Double distilled water was used throughout the experiments.

Synthesis of β - Ag_2MoO_4 Nanorods

The compound Ag_2MoO_4 was synthesized by combining a 0.1 M solution of AgNO_3 with a 0.1 M solution of sodium molybdate (Na_2MoO_4) using a thermo-stirrer. Additionally, the solution underwent ultrasonication for 40 minutes, after which 0.8 M NaOH was added gradually. Subsequently, the mixture of reactants was agitated for a duration of 10 minutes and then subjected to reflux for a period of 1 hr. The solution was centrifuged and the precipitate was rinsed many times with deionized water and ethanol. Afterward, the sample was subjected to dehydration in a hot air oven at a temperature of 90 °C for 60 mins. Then, calcined at a temperature of 500 °C for a duration of 1 hr to obtain Ag_2MoO_4 nanorods.

Sensing of L-glucose by β - Ag_2MoO_4

For the detection of L-glucose, firstly, 200 μL of 10 mM OPD was taken in a cuvette, then 100 μL of β - Ag_2MoO_4 NRs (1 mg/mL) was dispersed in 2.5 mL phosphate buffer (pH 7.0), followed by the addition of 20 μL of glucose solution with various concentrations in the presence of 200 μL of 10 mM H_2O_2 and Glucose oxidase.

Characterization Techniques

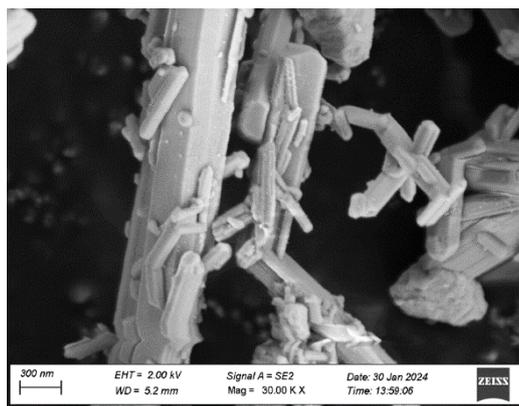


Figure. 1 FE-SEM Image of β - Ag_2MoO_4 shows the Formation of a Rod-Like Structure.

The UV-Vis absorption spectra of synthesized β - Ag_2MoO_4 NRs were recorded by using Hitachi UH5300 with deuterium and tungsten lamp in the range from 200- 800 nm. The IR spectrum of β - Ag_2MoO_4 NRs was measured by using Bruker IR spectrophotometer in ATR mode. The diffraction pattern of β - Ag_2MoO_4 NRs was recorded by using a powder X-ray

diffractometer (PANalytical Netherlands). The hydrodynamic size of β - Ag_2MoO_4 NRs NTs was examined with the dynamic light scattering technique by using the Horiba-SZ-100 DLS spectrometer. A pinch of sample was dispersed in an appropriate solvent before analysis.

Results and Discussion

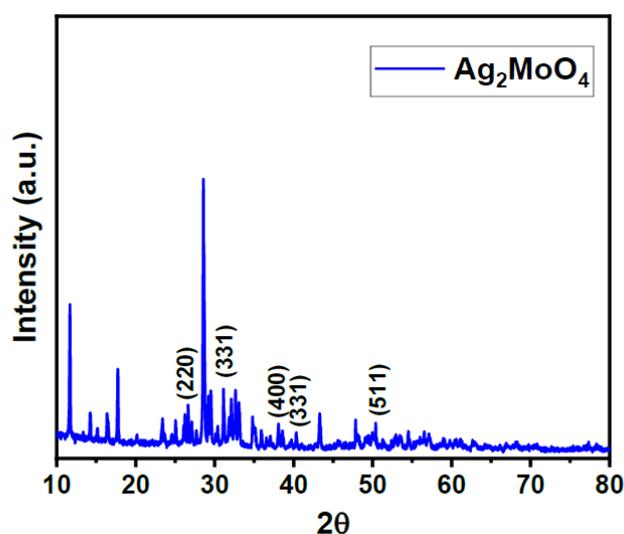


Figure 2. XRD Pattern of β - Ag_2MoO_4 Nanorods.

Characterization of β - Ag_2MoO_4 Nanorods

Fig. 1 shows the FE-SEM image of as-synthesized Ag_2MoO_4 . The morphology of Ag_2MoO_4 is confirmed to be rod-like with a diameter and length in nanometer ranges. The nanorods are uniformly distributed. The rod-like structure facilitates a higher surface area for enhanced photocatalytic activity of β -

Ag_2MoO_4 nanorods. The thermal treatment of β - Ag_2MoO_4 regulates the formation of rod-like structures.

The X-ray diffraction pattern of β - Ag_2MoO_4 nanorods is shown in **Fig. 2**. The peaks seen at 26.8° , 31.9° , 38.6° , 40.5° , and 50.4° corresponds to the (220), (331), (400), (331) and (511) planes respectively. The existing peaks resemble the characteristic peaks of β -

Ag₂MoO₄ nanomaterials reported in the literature. The 'n' number of peaks in the

diffraction pattern arise from the planes of various shapes of nanorods [17, 18].

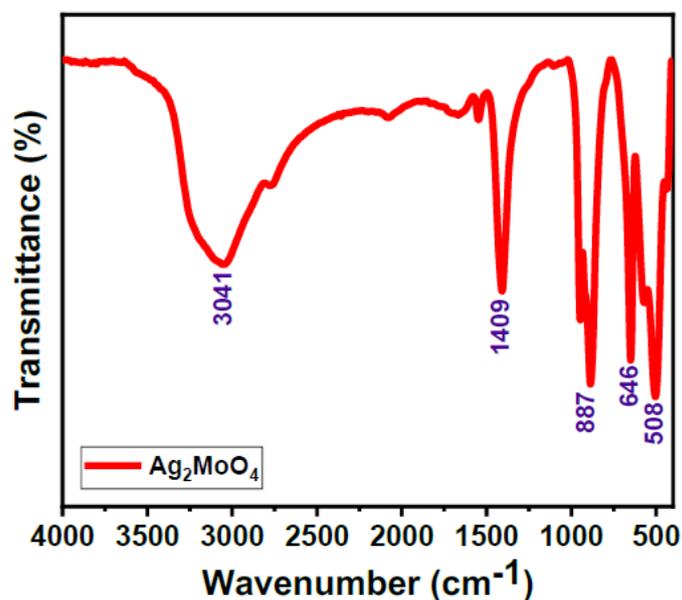


Figure 3. FT-IR Spectrum of β -Ag₂MoO₄ Nanorods.

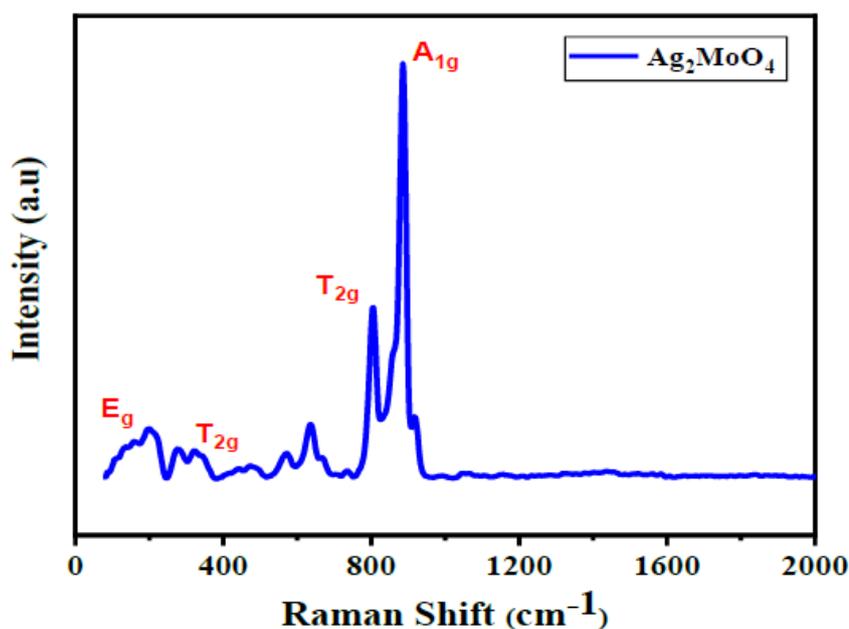


Figure 4. Raman Spectrum of β -Ag₂MoO₄ Nanorods.

The FT-IR spectrum of β -Ag₂MoO₄ nanorods is shown in Fig. 3. The peak at 3041 cm⁻¹ is attributed to the -OH stretching vibration. The peaks at 1409, 887, 646, and 508 cm⁻¹ arise from the stretching and bending vibrations of Mo-O bonding that existed in β -Ag₂MoO₄ nanorods. The results show the existence of bonding in Ag₂MoO₄ nanorods

suggesting the successful formation of nanorods [19].

The Raman spectrum of β -Ag₂MoO₄ nanorods is shown in Fig. 4. The peaks around 240, 364, 748, and 882 cm⁻¹ are attributed to the symmetric bending(E_g), anti-symmetric bending(T_{2g}), anti-symmetric stretching(T_{2g}), and symmetric stretching(A_{1g}) vibrations of

Mo-O bonding in Ag_2MoO_4 nanorods. The results show the vibrations existing in

Ag_2MoO_4 possess excellent optical characteristics of nanorods.

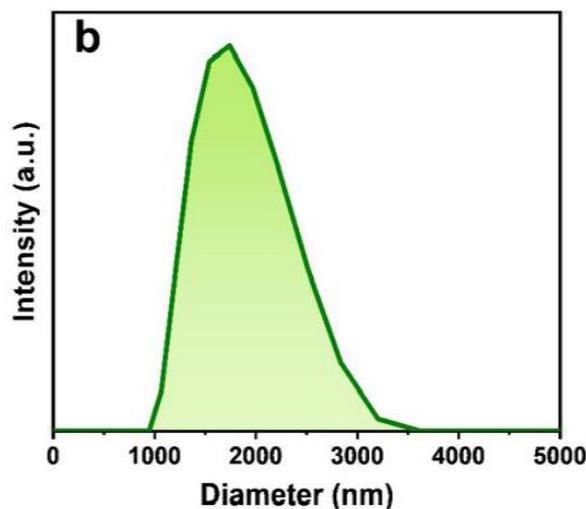


Figure 5. Dynamic Light Scattering Spectrum of Ag_2MoO_4 Nanorods.

Dynamic light scattering (DLS) is a tool used to measure the size of particles in the nanometer range using the Brownian motion of dispersed particles, which is dependent on the particle's core size, surface structures, concentration, and solvent medium. **Fig. 5** displays the results of the DLS measurement of synthesized Ag_2MoO_4 nanorods dispersed in ethanol. The image

demonstrates that the hydrodynamic diameter of particles in Ag_2MoO_4 nanorods has formed in the range of 1000 nm to 3500 nm, with the higher amount of particles falling in the hydrodynamic diameter of 2000 nm. The presence of ethanol during the synthesis may cause the NRs to aggregate.

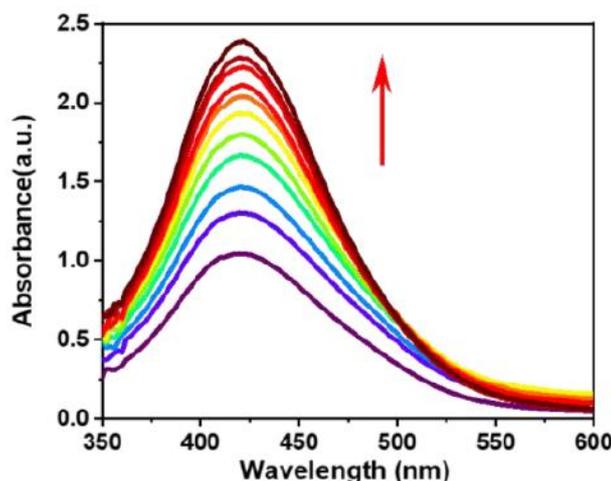


Figure 6. The Sensing Activity of $\beta\text{-Ag}_2\text{MoO}_4$ Nanorods Upon the Gradual Addition of Glucose in the Concentration Range Up to 10 μM .

Application of $\beta\text{-Ag}_2\text{MoO}_4$ in Sensing of L-Glucose

OPD, or o-phenylenediamine, not only results in a yellow solution but also produces a luminous product upon oxidation. Therefore, OPD was selected as the colorimetric substrate

for this investigation. The presence of H_2O_2 in the reaction of OPD leads to the formation of a yellow solution comprising mostly oxidized products, namely DAP. The DAP compound has a maximum absorption at a wavelength of 415 nm. Nevertheless, the process of reducing H_2O_2 is slow in the absence of a catalyst,

resulting in a low absorbance. Conversely, the inclusion of Ag₂MoO₄ nanotubes results in a significant increase in the absorbance. Ag₂MoO₄ nanotubes can enhance the rate of oxidation of OPD in the presence of H₂O₂. The

results demonstrate that this approach has a high level of selectivity for glucose while also demonstrating a great tolerance for other compounds [12, 20, 21].

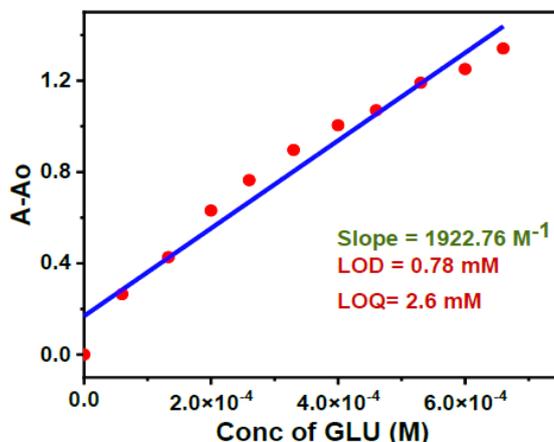


Figure 7. The Sensing of CoNCs with the Gradual Addition of Glutathione in the Concentration Range Upto 10 μ M.

The UV-vis responses of Ag₂MoO₄ NTs were measured under optimal conditions by introducing varying amounts of glucose (ranging from 0 to 1 mM) in the presence of OPD and H₂O₂. **Fig. 6** demonstrates that the absorbance increases gradually as the glucose concentrations increase, and a strong linear relationship is observed between the difference in absorbance and the glucose concentration. The detection limit is determined to be 0.78 mM, as depicted in **Fig. 7**. The sensitivity of measuring glucose concentration using colorimetric techniques was determined to be 1.9 mM⁻¹. Our approach has the capability to detect glucose across a wide linear range and with a low detection limit.

Conclusion

The hydrothermal process has been shown to be a successful technique for producing biomimetic β -Ag₂MoO₄ nanorods, which have

shown great promise for identifying L-glucose with high sensitivity. The synthesis technique is reasonably simple, ecologically sound, and yields nanorods with clearly defined structures and desired physical and chemical characteristics which were confirmed by FE-SEM, and XRD analysis. These nanorods provide exceptional sensing abilities as a result of their large surface area, stability, and improved electron transfer rates, rendering them ideal candidates for glucose sensing applications.

Competing Interests

The author declares no conflict of interest in the contents of the manuscript.

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