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Synthesis and characterization of molybdenum trioxide: study of thermal effects on structural and morphological control.

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1. Introduction

Currently, molybdenum-based materials have aroused great scientific and technological interest due to their physicochemical properties and wide range of applications in areas such as catalysis, optics, sensors, and energy storage. Among these materials, molybdenum trioxide (MoO_3) stands out for presenting tunable electronic and optical properties, high chemical stability, and structural versatility, characteristics that enable its use in electrochromic devices, electrochemical energy storage systems, gas sensors, photocatalytic processes, and catalytic applications [1, 2, 3].

Among the different polymorphs of MoO_3, the orthorhombic phase (α-MoO_3) is the most thermodynamically stable under ambient conditions and features a lamellar crystalline structure composed of shared (MoO_6) octahedra, favoring the formation of planar structures. This structural configuration promotes properties of interest for applications in electronic devices, energy conversion and storage systems, as well as catalytic and photocatalytic processes. Despite the extensive studies reported in the literature, challenges related to the control of structure and morphology during the synthesis of the material still exist [4, 5].

The production of α-MoO_3 is strongly influenced by processing conditions, especially temperature and synthesis atmosphere, parameters that affect nucleation and crystal growth processes. Various synthesis routes have been employed to produce this material, including solid-state methods, chemical precipitation, hydrothermal, sol-gel, electrodeposition, and thermal evaporation, frequently followed by thermal treatments to promote the formation of the orthorhombic phase with structural and morphological rigor [6, 7].

In this context, the present work aims to synthesize α-MoO_3 using the coprecipitation method and to investigate its structural and local structural characteristics. To achieve this, structural characterization techniques and Time-Differential Perturbed Angular Correlation (TDPAC) spectroscopy were employed aiming to obtain information about the hyperfine environment of the probe-nuclei and contribute to the understanding of the mechanisms involved in the formation of the crystalline structure of the material. Additionally, scanning electron microscopy analyses were performed to evaluate the morphology of the obtained samples

2. Methodology

The α-MoO_3 samples were synthesized based on the procedure described by Jain et al., using the coprecipitation method. Initially, ammonium heptamolybdate was dissolved in 100 mL of Milli-Q water under heating and constant magnetic stirring. When the solution reached 60°C , this temperature was maintained for 30 min. Then, the temperature was raised to promote solvent evaporation and obtain the solid precursor. The resulting material was transferred to a porcelain crucible and subjected to thermal treatment, varying the calcination time for each sample. Subsequently, the samples were characterized by X-ray diffraction (XRD), Scanning Electron Microscopy (SEM), and Time-Differential Perturbed Angular Correlation (TDPAC) spectroscopy. XRD analyses were used to identify the crystalline phases present and verify the formation of the α-MoO_3 phase, while SEM measurements allowed the evaluation of the morphology of the obtained particles. The TDPAC technique was used to investigate the local hyperfine environment and possible structural changes associated with the material formation process. Figure 1 presents the synthesis process.

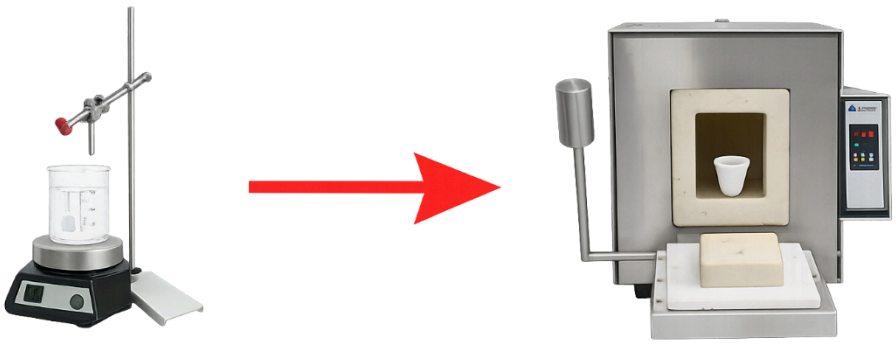

Figure 1: Diagram of the synthesis process.

3. Results and Discussion

The obtained diffraction patterns were refined in refinement cycles following the Rietveld method. The resulting diffractograms are compatible with the crystalline structure of α-MoO_3, ICD-35076. When analyzing the three obtained patterns, it is possible to observe that the 4-hour sample presents better-defined peaks and a higher definition in the baseline, which suggests greater crystallinity compared to the other samples (2h and 3h), as presented in Figure 2.

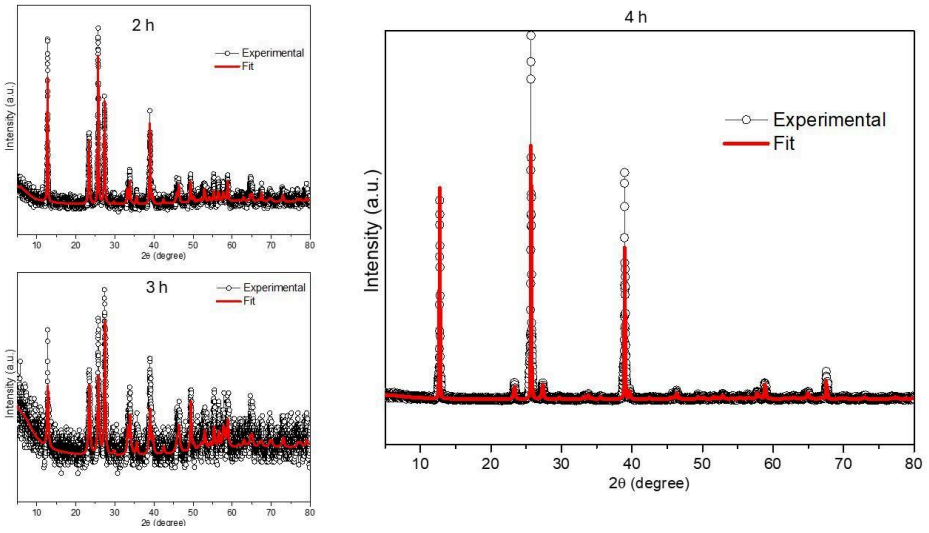

Figure 2: Diffraction patterns obtained at different thermal treatment times.

The SEM results indicate that the 2-hour and 3-hour samples do not predominantly present a defined morphology, showing regions of solid agglomerates. As for the morphology of the sample thermally treated for 4 hours, a predominant ribbon-like morphology is verified, possibly within the micrometric regime, as shown in Figure 3.

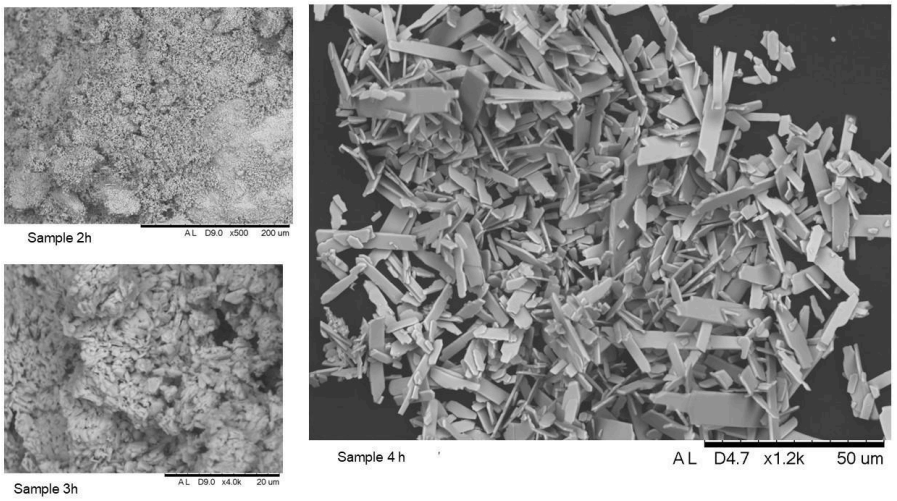

Figure 3: Scanning Electron Microscopy results for the 2h, 3h and 4h samples.

In view of the XRD and SEM results, the sample selected for the TDPAC measurement was the α-MoO_3 with 4 hours of thermal treatment. In this context, drops of ^{111}InCl were added during the synthesis, which was the only change to the previously reported protocol. After the 4-hour thermal treatment, the sample was sealed in a quartz tube and subjected to TDPAC measurement at room temperature. In the obtained spectrum, the probe is located in a non-symmetric local environment, characteristic of widely perturbed environments frequently associated with surface regions of the sample. The obtained spectrum is presented in Figure 4.

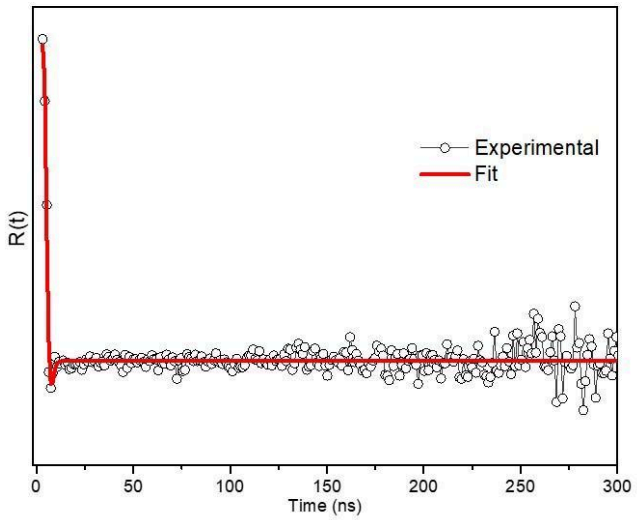

Figure 4: TDPAC spectrum obtained at room temperature.

4.Conclusions

In view of the presented results, it is possible to verify the attainment of the α-MoO_3 structure through the synthesis method reported by Jain et al. 2021. Furthermore, the influence of the thermal treatment on the formation of the α-MoO_3 structure is verified, as well as on its morphological control, where both results showing greater structural and morphological control were achieved after the 4-hour thermal treatment. The analysis of hyperfine interactions evidences that the crystalline environment where the probe is located is not symmetric, thus it is possibly located in surface regions of the sample. In general, the work highlights structural and morphological control parameters for the synthesis of α-MoO_3.

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