

EFFECT OF CALCINATION TIME ON THE ADSORPTIVE CAPACITY OF MOLYBDENUM OXIDES FOR THE REMOVAL OF METHYLENE BLUE IN AQUEOUS SOLUTION

EFEITO DO TEMPO DE CALCINAÇÃO NA CAPACIDADE ADSORTIVA DE ÓXIDOS DE MOLIBDÊNIO PARA REMOÇÃO DE AZUL DE METILENO EM SOLUÇÃO AQUOSA

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ABSTRACT

The increasing industrialization process and the discharge of pollutants into water bodies have been concerns since the beginning of industrialization. To ensure safe access to water, new purification methodologies and stricter regulations are necessary. This study aims to develop materials for the removal of methylene blue (MB) dye in aqueous systems. Materials were synthesized through the oxidation of molybdenum disulfide (MoS_2) and subjected to different calcination times, seeking a balance between energy consumption and adsorption capacity. The elemental composition of the samples was analyzed by Scanning Electron Microscopy with Energy Dispersive X-ray Spectroscopy (SEM-EDS), X-ray Diffraction (XRD), and Fourier-Transform Infrared Spectroscopy (FTIR) revealed the transformation of MoS_2 to molybdenum trioxide (MoO_3) proportional to the thermal treatment time. The kinetic analysis of MB adsorption revealed that the sample calcined for 300 minutes exhibited the highest efficiency in dye removal from the solution, with an adsorption capacity of 41.6 mg.g⁻¹ after 300min. This capacity was significantly higher than that of the sample calcined for 30 minutes during the same period of time (18.6 mg.g⁻¹), representing a 124% increase. Compared to the precursor material (6.60 mg.g⁻¹), the improvement in adsorption capacity was even more expressive, with a 530% increase. Tests adsorption isotherms indicated that the maximum adsorption capacity of the sample calcined for 300 minutes was 379.0 mg.g⁻¹. Equilibrium tests indicated that the maximum adsorption capacity of the sample calcined for 300 minutes was 379.0 mg.g⁻¹. The greater dispersion in aqueous media of the calcined sample, compared to the others, is attributed to the presence of molybdenum oxide, favoring the adsorption of the dye onto the surface of the material formed.

Keywords: Adsorption; dyes; composites; MoS_2 ; MoO_3 .

RESUMO

O crescente processo de industrialização e o despejo de poluentes nos meios aquáticos têm sido preocupações desde o início da industrialização. Para garantir o acesso seguro à água, novas metodologias de purificação e regulamentações mais rígidas são necessárias. Este estudo visa desenvolver materiais para a remoção do corante azul de metileno (AM) em sistemas aquosos. Os materiais foram sintetizados através da oxidação

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do dissulfeto de molibdênio (MoS_2) e submetidos a diferentes tempos de calcinação, buscando um equilíbrio entre consumo energético e capacidade de adsorção. A composição elementar das amostras foi analisada por Microscopia Eletrônica de Varredura com Espectroscopia de Energia Dispersiva de Raios-X (MEV-EDS), Difractometria de Raios-X (DRX) e Espectroscopia no Infravermelho com Transformada de Fourier (FTIR) que revelou a transformação do MoS_2 para o trióxido de molibdênio (MoO_3) proporcional ao tempo de tratamento térmico. A análise cinética da adsorção de AM revelou que a amostra calcinada por 300 minutos apresentou a maior eficiência na remoção do corante da solução, com uma capacidade de adsorção de 41,6 mg.g⁻¹ após 300 min. Essa capacidade foi significativamente superior à da amostra calcinada por 30 minutos durante o mesmo período de tempo (18,6 mg.g⁻¹), representando um aumento de 124%. Em comparação ao material precursor (6,60 mg.g⁻¹), a melhoria na capacidade de adsorção foi ainda mais expressiva, com um aumento de 530%. Os testes de isotermas de adsorção indicaram que a capacidade máxima de adsorção da amostra calcinada por 300 minutos foi de 379,0 mg.g⁻¹. A maior dispersão em meio aquoso da amostra calcinada, comparada com as demais, é atribuída à presença de óxido de molibdênio, favorecendo a adsorção do corante sobre a superfície do material formado.

Palavras-chave: Adsorção; corantes; compósitos; MoS_2 ; MoO_3 .

INTRODUCTION

Although the exact origin of dye use is uncertain, it is widely accepted that humans have been using them since the dawn of civilization. Archaeological evidence, such as Egyptian mummies with colored adornments and cave paintings, attests to this fact. In Brazil, the exploitation of Brazilwood, a wood rich in red dye, was an important economic activity 500 years ago. The dye extracted from this wood was exported to Europe, where it was used in the textile industry to dye fabrics in shades of red, pink, and brown (DALLAGO; SMANIOTTO; OLIVEIRA, 2005; FERREIRA *et al.*, 2018).

Considered the most polluting of all industrial sectors, textile wastewater deserves special attention due to the complexity of its chemical components and the large volume discharged into waterways (OLIVEIRA; COELHO; DE MELO, 2018). Textile dyes require specific treatments and, due to their high chemical stability, are difficult to degrade (DALLAGO; SMANIOTTO; OLIVEIRA, 2005). Color intensity is also a highly relevant factor, as it influences the amount of light penetrating rivers and lakes, altering photosynthetic processes, disrupting water oxygenation, and increasing toxicity. Additionally, they can generate carcinogenic and mutagenic phenomena, negatively affecting the fauna and flora around watercourses (PEIXOTO; MARINHO; RODRIGUES, 2013).

There are no specific regulations regarding the disposal of these residues in the environment (PEIXOTO; MARINHO; RODRIGUES, 2013). CONAMA Resolution 357/05 stipulates that the discharged effluent cannot alter the classification of the receiving body and that the effluent color cannot exceed 75 mg Pt-Co/L (JORGE; TAVARES; SANTOS, 2015). Industries are required to install effluent treatment stations to treat the waste before it is discharged into the environment, as per the norm established in CONAMA Resolution 357/05.

Adsorption is a physicochemical phenomenon (adhesion) involving mass transfer, in which a substance (adsorbate) adheres to the surface of another material (adsorbent). This technique has emerged as a promising technology for the treatment of contaminated water. (PENHA, *et al.* 2021) Adsorbent materials, due to their high specific surface area and the presence of active sites, offer many advantages compared to traditional water treatment methods (PIMENTEL, *et al.* 2022). It is a relatively simple method and its efficiency depends on several factors such as the characteristics of the adsorbent, the properties of the adsorbate, and the operating conditions. The most common adsorbents are activated carbon, peat, silica, alumina, cellulose, chitin and chitosan, and clay (KIMURA *et al.*, 1999; OLIVEIRA *et al.* 2022). Despite being widely used in industry, they are expensive and not effective when the dye is cationic (DALLAGO; SMANIOTTO; OLIVEIRA, 2005).

Adsorbent materials provide significant contributions to the decontamination process of water. The most common ones are activated carbon, peat, silica, alumina, cellulose, chitin, and chitosan (KIMURA *et al.*, 1999). Although widely used in industry, they are expensive and ineffective when the dye is cationic (DALLAGO; SMANIOTTO; OLIVEIRA, 2005).

Molybdenum disulfide (MoS_2) has been studied due to its adsorption capacity derived from the metal's lamellar structure (SOUZA, 2022). It has a hexagonal structure and an affinity for heavy metals, interacting through Coulombic attractions exerted by the sulfur atom on the metals. However, it is insoluble in water, and its stability is lost when subjected to high temperatures and in the presence of oxygen, producing a water-soluble trioxide (MoO_3) (HAYNES, 2015). Intermediate states between the oxidized form and the disulfide form are determined by temperature, the presence of the oxidant, and the time in the calcination furnace.

Studies have already demonstrated the adsorption capacity of MoO_3 in applications with cationic dyes (PÉREZ-GONZÁLEZ *et al.*, 2021; RAKASS *et al.*, 2018; SOUZA, 2022; ZHANG; PARK, 2019). The efficiency results in the adsorption processes of dyes, such as methylene blue (MB), widely used in the textile industry for dyeing clothes, are promising. In this context, the present work aims at the experimental arrangement of the purification of water contaminated by the MB dye using MoS_2 and MoS_2 calcined for 30 and 300 minutes at 300 °C in an oxidizing atmosphere (Air) as adsorbent material. The difference in the calcination time of the samples aims to show if there is a significant difference in the adsorptive capacity of the material. If with a shorter calcination time the adsorption effectiveness is satisfactory, there is an energy gain compared to the longer calcination time. The performance of kinetic and adsorption tests is the first step towards understanding the efficiency of the capacity of these materials for water purification. Therefore, this work was divided into two parts, one for each test.

MATERIALS AND METHODS

MATERIALS

Methylene blue (99%) from the brand Neon and MoS₂ from the brand Jet-Lube, both commercially acquired, were used without further purification.

METHODS

Calcination of Molybdenum Sulfide

Molybdenum disulfide (MoS₂) was calcined in porcelain crucibles in a Jung muffle furnace, model 0261. To increase the contact area with the oxidizing atmosphere, small amounts of MoS₂ were spread on the inner walls of the crucibles. All crucibles were subjected to the same temperature of 300 °C for 30 and 300 minutes, respectively. After cooling in a desiccator, the samples were used in the experiments.

Preparation of Methylene Blue (MB) solution

Stock solutions of methylene blue were prepared at concentrations of 10, 40, 75, 100, 150, 175, 200, 275, 350, and 400 mg.L⁻¹. To do this, an exact amount of 0.5 g of methylene blue was weighed on an analytical balance and quantitatively transferred to a 1 L volumetric flask. The volume was completed with deionized water, homogenized by inversion for 2 minutes, and the solution was stored in an amber flask, protected from light, until use. Note: From the 500 mg.L⁻¹ stock solution, the remaining solutions were prepared by serial dilution, using calibrated volumetric pipettes and volumetric flasks.

Spectroscopy Ultraviolet/Visible (UV/Vis)

The samples were analyzed using a Thermo Scientific Genesys 10 UV-Vis spectrophotometer in the 600-700 nm range. The maximum absorption wavelength of methylene blue (MB) was determined to be 665 nm.

Scanning Electron Microscopy with Energy Dispersive X-ray Spectroscopy (SEM-EDS)

Powder samples were imaged using a Hitachi TM3000 SEM at an accelerating voltage of 5 kV without metal coating. EDS analyses were performed on the same equipment to determine the chemical composition of the analyzed material.

X-Ray diffraction

The powder sample X-ray diffraction pattern was obtained on a Shimadzu DRX-6100 X-ray diffractometer. Cu-K α radiation generated at 40 kV and 30 mA was used, with a scan speed of 2° per minute and a 2 θ angle range of 10° to 60°, using a disk sample **holder**.

Fourier Transform Infrared Spectroscopy (FTIR)

Characterization was performed on a PerkinElmer Spectrum 65 spectrophotometer using attenuated total reflectance (ATR) technique. Samples were analyzed before and after the methylene blue adsorption process at a concentration of 40 mg.L⁻¹. After adsorption, the materials were collected, dried at room temperature, and subjected to FTIR analysis to identify possible changes in their structures after interaction with the dye.

Adsorption Kinetics Studies

Kinetic studies aimed to determine the maximum adsorption capacity of the dye by the material under controlled conditions, with constant temperature but varying time. The adsorption capacity (q) is given in (mg.g⁻¹) of dye adsorbed per mass of adsorbent, according to Equation 1.

$$q = \frac{m_{dye}}{m_{adsorbent}} \quad (1)$$

A 10 mg.L⁻¹ dye solution was used in triplicate with 6 mg of each adsorbent material. A dye sample without adsorbent was measured for absorbance every 30 minutes to eliminate interference from possible dye degradation over time. The pH of the solution remained stable around 5.5, and sample preparation and measurement were carried out at 23°C. The samples were analyzed by UV/Vis spectroscopy, with a total of 10 measurements taken at 30-minute intervals, totaling 300 minutes of analysis. After each measurement, the sample was returned to the flask and allowed to rest.

Adsorption Isotherm Studies

To determine the maximum adsorption capacity of the dye by the material, dye solutions with different concentrations were prepared, ranging from 40 mg.L⁻¹ to a maximum of 500 mg.L⁻¹. The amount of adsorbent in each sample was kept constant at 5 mg. Samples were prepared in triplicate, and a control test was conducted with only the dye under agitation. A reference sample was prepared with demineralized water and the adsorbent materials. Each solution was contacted with a known mass of the adsorbent and subjected to constant agitation for 24 hours, under controlled room temperature and protected from light, to ensure homogeneous experimental conditions. After the

contact period, the samples were centrifuged to separate the phases. The supernatant, containing the unadsorbed adsorbate, was analyzed by UV-Vis spectrophotometry. In order to obtain a mathematical model that described the amount of adsorbate retained by the adsorbent, three widely used adsorption isotherms in the literature were fitted: Freundlich, Langmuir, and Sips. The equations of these isotherms (Equations 2, 3, and 4, respectively) were applied to the experimental data to determine the parameters that best represent the adsorption process.

Non-linear equation

$$q_e = K_f \sqrt[n]{C_e}$$

$$q_e = \frac{q_{max} b C_e}{1 + b C_e}$$

$$q_e = \frac{q_{max} (k_s C_e)^n}{1 + (k_s C_e)^n}$$

Linear equation

$$\ln q_e = \ln K_f + \frac{1}{n} \ln C_e \quad (2)$$

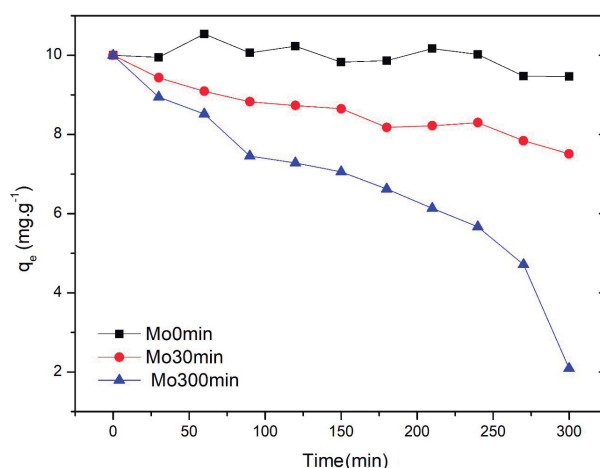
$$\frac{C_e}{q_e} = \frac{1}{b q_{max}} + \frac{C_e}{q_{max}} \quad (3)$$

$$\ln \frac{q_e}{q_{max} - q_e} = \frac{1}{n} \ln C_e + \ln k_s \quad (4)$$

Given the unknown nature of the adsorbent system, a multifaceted approach was adopted, employing the Freundlich, Langmuir, and Sips isotherms. The Freundlich isotherm, with its empirical nature and ability to describe heterogeneous systems, served as a starting point for modeling. The Langmuir isotherm, in turn, was used as a reference for homogeneous systems, providing a basis for comparison. The Sips isotherm, by combining aspects of both, allowed for a more comprehensive analysis, enabling the identification of the relative contribution of multilayer adsorption mechanisms and homogeneous sites.

RESULTS AND DISCUSSIONS

Figure 1 demonstrates the limited effectiveness of MoS₂ as an adsorbent for methylene blue. The low dye removal can be directly related to the poor dispersion of the material in water. The inability to form a stable suspension prevents intimate contact between the MoS₂ particles and the dye molecules, significantly compromising the adsorption process (HAYNES, 2015).

Figure 1 - Relationship between the amount of dye still present in solution as a function of time.

It was observed that both calcined compounds showed good effectiveness in removing AM dye from aqueous media, as evidenced by the reduction in the concentration of dye remaining in solution compared to the initial measurement (10 mg.L^{-1}). It is noteworthy that the sample calcined for 30 minutes showed a 25% reduction in the initial concentration, and the sample calcined for a longer time was more effective, with a reduction of almost 80% in the dye concentration in the sample. The adsorption capacity of the materials was determined using Equation 1, and the data are presented in Table 1.

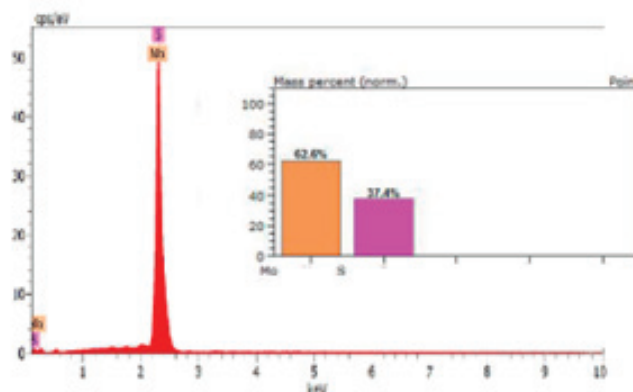
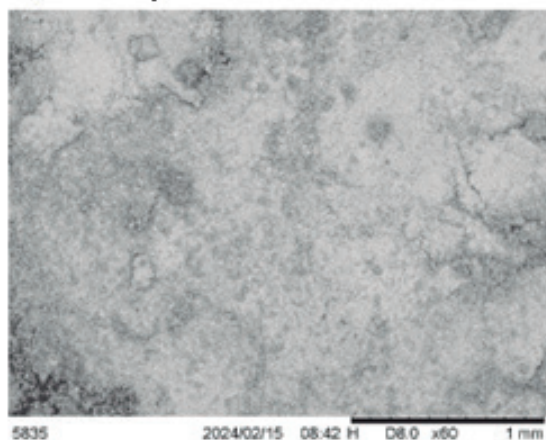
Table 1 - Adsorptive capacity of samples.

	Sample's mass ($\text{g} \pm 0.001$)	Initial mass of MB (mg)	Mass of adsorbed MB (mg)	Adsorptive capa- city (mg.g^{-1})
Mo0min	0.006	0.40	0.04 ± 0.004	6.60 ± 0.004
Mo30min	0.006	0.40	0.11 ± 0.009	18.3 ± 0.009
Mo300min	0.006	0.40	0.25 ± 0.013	41.6 ± 0.013

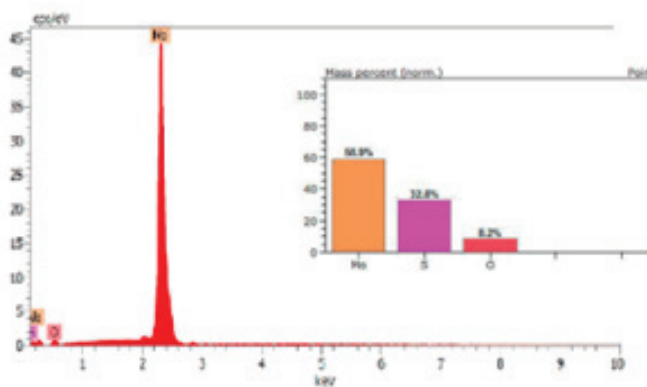
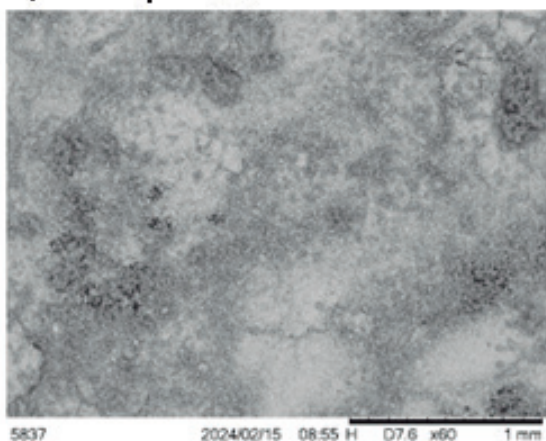
The high adsorption capacity shown by the sample calcined for 300 minutes (Mo300min) can be explained by the higher presence of the oxidized phase of molybdenum in its structure. This assertion is corroborated by the results of the SEM-EDS analysis, which revealed a significantly lower sulfur content (30.72%) in this sample compared to the one calcined for 30 minutes (Mo30min), with 47.7% sulfur. Figure 2 presents the detailed data of this analysis. The lower dispersibility of the non-oxidized phase of molybdenum in water, rich in sulfur, justifies its lower adsorption capacity. The formation of an efficient interface between the adsorbent and the adsorbate is fundamental to the adsorption process, and the high presence of sulfur in the Mo30min sample hinders this efficient contact. Thus, the partial transformation of MoS_2 into MoO_3 in the Mo300min sample significantly increases the adsorption capacity of the material, making it more effective in removing methylene blue dye from aqueous solution.

Figure 2 - Micrographs of samples with EDS a) Mo0min, b) Mo30min e c) Mo300min.

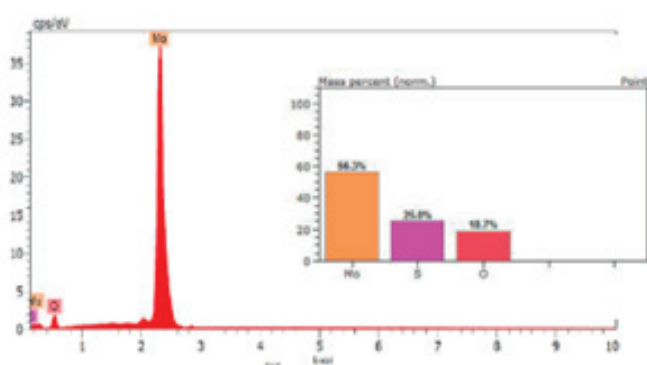
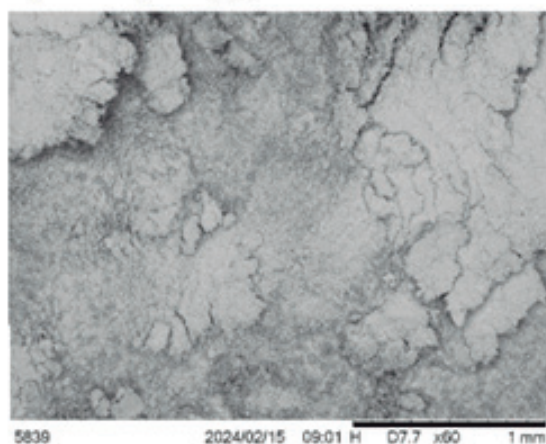
a) Sample Mo0min



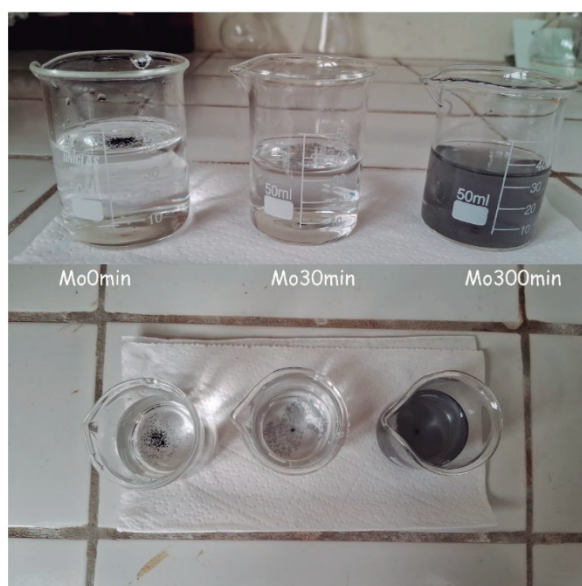
b) Sample Mo30min



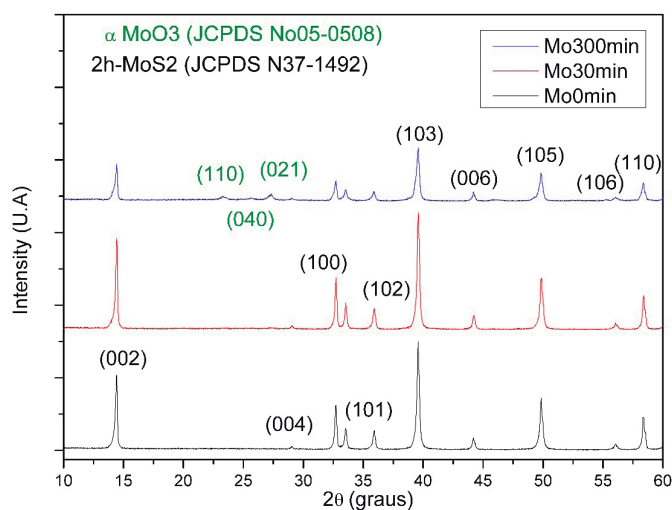
c) Sample Mo300min



The dispersion capacity of the materials under study is directly influenced by the oxygen content in their composition. Figure 3 presents a visual comparison of the three materials, evidencing that the dispersibility increases progressively from Mo0min to Mo30min and, finally, to Mo300min. This trend indicates that a higher oxygen concentration promotes better dispersion of the material in the liquid medium.

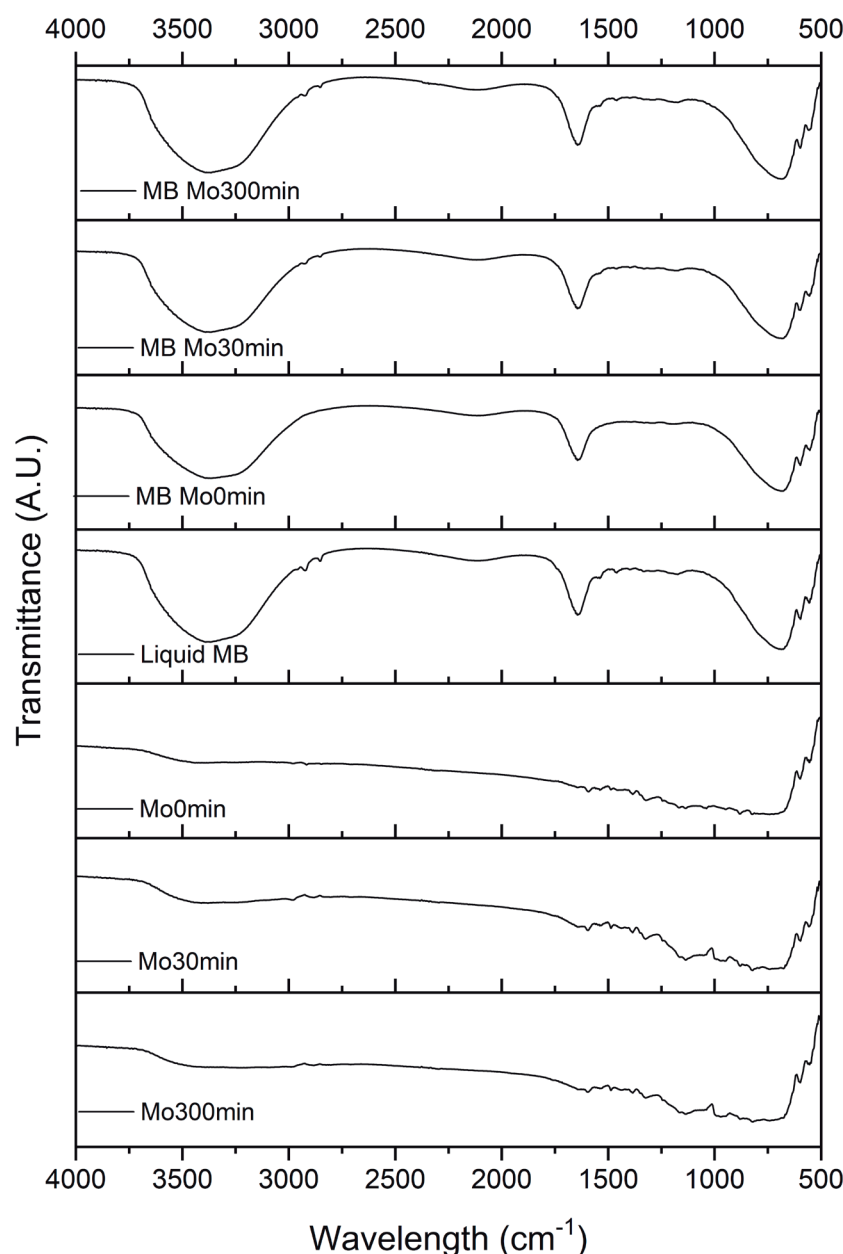
Figure 3 - Dispersibility of samples in aqueous medium.

The dispersion capacity of the studied materials is directly influenced by their oxygen content. Figure 3 visually compares the three materials, showing that dispersibility progressively increases from Mo0min to Mo30min and finally to Mo300min. This trend indicates that a higher oxygen concentration promotes better dispersion in the liquid medium. As the oxygen content increases, the material's structure is modified, enhancing its interaction with the aqueous medium. In the case of Mo300min, the higher presence of the oxidized molybdenum phase results in a more homogeneous and efficient dispersion, facilitating the formation of adequate interfaces between the adsorbent and the adsorbate. This is essential to optimize the adsorption process, confirming that prolonged heat treatment in an oxidizing atmosphere is a crucial factor for the effectiveness of MoS_2 in removing cationic dyes such as methylene blue.

Figure 4 - X-ray diffractograms of the samples.

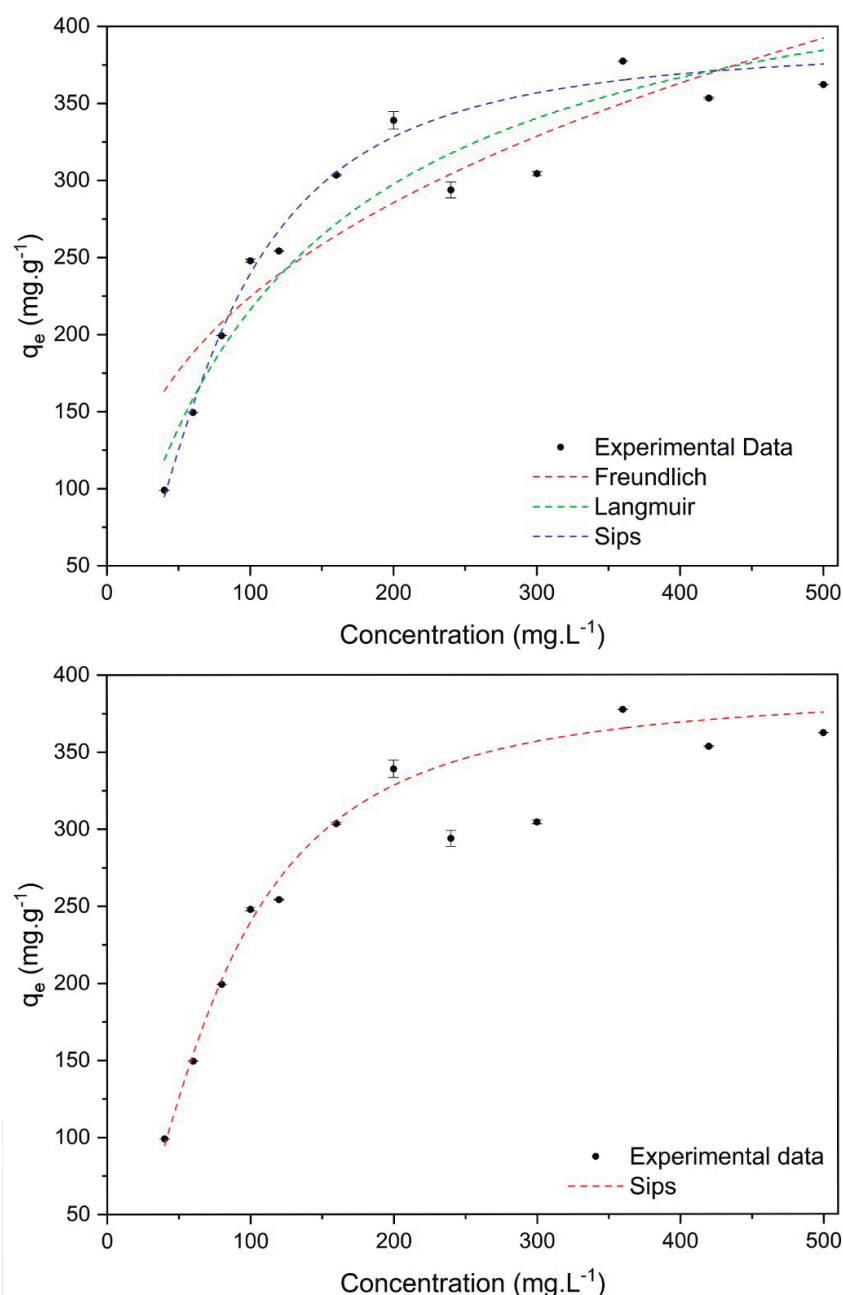
The XRD analysis, presented in Figure 4, reveals that the sample calcined for 30 minutes exhibits characteristic peaks of MoO_3 with low intensity, indicating a small amount of this phase. On the other hand, the sample calcined for 300 minutes shows intense and well-defined peaks, evidencing the predominant formation of the MoO_3 phase.

Figure 5 - FTIR analysis of samples both before and after adsorption experiments, including methylene blue.



The FTIR spectra of the samples after methylene blue adsorption showed the characteristic bands of the dye, evidenced in the region between 2800 and 3000 cm^{-1} , highlighting what suggests their presence on the surface of materials. The absence of new bands or significant shifts in the original bands suggests that the predominant adsorption mechanism was physisorption, characterized by relatively weak intermolecular forces.

Figure 6 - Nonlinear regression models based on Langmuir, Freundlich and Sips.



To obtain a more accurate non-linear fit of the experimental data, three adsorption isotherm models were tested: Freundlich, Langmuir, and Sips. The Freundlich model, which describes multilayer physical adsorption on heterogeneous surfaces, did not fit the data adequately. The Langmuir model, which represents monolayer chemical adsorption on homogeneous surfaces, showed a better but still non-ideal fit. The Sips model, combining characteristics of both Langmuir and Freundlich models and describing both homogeneous and heterogeneous systems, proved to be the most suitable for describing the experimental data, with an adjusted coefficient of determination (R^2 adjusted) of 0.986. This indicates monolayer adsorption in a system with intermediate characteristics between the Langmuir and Freundlich models (WANG; GUO, 2020). Equation 5 shows the Sips isotherm in terms of Q_m , K , and n :

$$Q_e = \frac{Q_m(K * C)^n}{1 + (K * C)^n} \rightarrow \frac{379(0.014 * C)^{1.52}}{1 + (0.014 * C)^{1.52}} \quad (2)$$

The low equilibrium adsorption constant (K) indicates a weak interaction between the adsorbate and the adsorbent. This weak interaction favors the desorption process, making the material promising for applications requiring adsorbate recovery. Additionally, the heterogeneity parameter (n) value greater than 1 indicates a predominantly homogeneous adsorbent surface, similar to the Langmuir model. This characteristic contributes to explaining the good fit of the Langmuir model to the experimental data. The maximum adsorption capacity (Q_m) of the investigated oxide, determined to be 379 mg.g⁻¹, positions it as a high-performance adsorbent compared to other commonly used materials, as shown in Table 2. These results indicate that the studied material has a high affinity for the adsorbate, making it promising for adsorption process applications.

Table 2 - Comparison of Q_m between different oxides.

Materials	Maximum adsorption capacity (mg.g ⁻¹)	Reference
Iron oxide	37.45	(RAZALI <i>et al.</i> , 2020)
Zinc oxide + Zeolite	41.32	(KABADAYI; ALTINTIG; BALLAI, 2024)
Activated carbon (AC)	222.20	(KOFFI, 2022)
Manganese oxide + AC	270.30	(KOFFI, 2022)
Material of the present study	379.00	Authors
Graphene oxide	714.00	(YANG, 2011)

CONCLUSIONS

In the present study, the effect of calcination time on the structure, composition and adsorption capacity of molybdenum sulfide was studied. Analysis showed that calcination results in the oxidation of the material, resulting in a solid containing both the sulfide and the metal oxide. The adsorption capacity was strongly affected by the calcination time used, with the sample obtained in 300 min being the one with the highest capacity. This fact was attributed to the presence of the oxide in the structure, which resulted in greater dispersion capacity in aqueous suspension, favoring interaction with the dye dissolved in the medium. The maximum adsorption capacity (Q_m) of the investigated oxide, determined to be 379 mg.g⁻¹, positions it as a high-performance adsorbent compared to other commonly used materials. These results indicate that the studied material has a high affinity for the adsorbate, making it promising for adsorption process applications.

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