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Investigation of Methyl Orange Adsorption onto Powdered Activated Carbon: Equilibrium, Kinetic, and Thermodynamic Analysis

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1. Introduction

Due to the rapid industrialization and the increasing exploitation of natural resources, environmental pollution due to the release of various harmful chemicals to the environment has become a serious issue. Many of these chemicals, which contain organic substances such as phenols, dyes, and other related compounds, are toxic and rarely degrade easily in the environment. Industrialization processes and associated emissions of hazardous substances such as acids, metal ions, and oils waste disposal have led to the increasing demand for effective and economic treatment technologies for the removal of these pollutants [Ajay Kumar et al., 2022; Ahmaruzzaman et al., 2024]. Adsorption has become a prominent technique for the removal and recovery of environmental pollutants such as heavy metal ions, reactive dyes, color pigments, and organic substances due to their effectiveness in the recovery of solutions containing these

Abstract

Adsorption is a widely used method for the removal of dyes from wastewater due to its simplicity, effectiveness, and economic viability. Methyl Orange is a commonly used azo dye in textile industries, and its presence in water bodies poses environmental and health hazards. Activated carbon is a popular adsorbent for dye removal due to its high surface area and porous structure. This study investigates the adsorption behavior of methyl orange onto powdered activated carbon.

We carried out experiments on batch adsorption by varying the initial dye concentration and tracking the adsorption over time. To ascertain the kinetic parameters of adsorption, the experimental data were examined using kinetic models such as pseudo-first-order and pseudo-second-order kinetics. In order to understand the spontaneity of the adsorption process, we also performed a thermodynamic analysis to assess the adsorption equilibrium by computing thermodynamic parameters like enthalpy and entropy changes.

The adsorption equilibrium of apparent methyl orange and powdered activated carbon was obtained after 60 minutes. Adsorption follows pseudo-second-order kinetics ($R^2=0.999$). It turned out that the Freundlich model is the most compatible with these kinetics ($R^2=0.988$).

According to the analysis of thermodynamic parameters, the adsorption process occurs with favorable and spontaneous reactions ($\Delta G_o < 0$), and the adsorbent-adsorbate reaction is endothermic ($\Delta H^\circ > 0$).

Keywords: Methyl Orange, Adsorption kinetics, Equilibrium of adsorption, Thermodynamic.

pollutants, simplicity in operation, potentially low cost, and easy regeneration of the adsorbent, and can be used for the treatment of low concentration of pollutants [R. Miglani et al., 2022].

These promising factors have driven the researchers' interest to investigate the efficiency and feasibility of various materials as adsorbents such as activated carbon, agricultural by-product materials, minerals, zeolites, and polymers [D. Sahu et al., 2024, Ahmaruzzaman et al., 2024]. Among these absorbent materials, activated carbon stands out as the most promising adsorption material for the removal of organic contaminants such as color dyes due to its large surface area, high porosity, and various functional groups covering its surface.

Water sources contaminated by synthetic dyes pose a serious threat to global health and the environment. Among these dyes, methyl orange stands out due to its extensive use in a variety of industries, including textiles, paper, and plastics, which frequently results in its disposal in industrial effluents [Ajay Kumar et al., 2022; A. Prince et al., 2024]. Methyl orange-containing wastewater leaks into aquatic environments have the potential to cause major environmental harm, including the death of aquatic life and contaminating water sources.

In response to these challenges, researchers and environmental engineers have explored various treatment methods to mitigate the harmful effects of dye contamination. Among these techniques, we can summarize these chemical [P. Kumar et al., 2012, R. Ganaie et al., 2023], physical [H. Bessaha et al., 2017, H. Liu et al., 2023, Q. Hu et al., 2007, A. Aluigi et al., 2014], physico-chemicals [Singh et al., 2017, H. A. Erkurt, 2010, Md. Pervez et al., 2024], and biological processes [A. Christina et al., 2022, M. Amran et al., 2011] in Table 1:

Table 1: Dye treatment methods

Chemical Processes	Physical Processes	Physico-Chemical Processes	Biological Processes
Oxidation - AOPs - Fenton's Reagent Reduction Neutralization - pH Adjustment	Adsorption - Activated Carbon - Natural Adsorbents Membrane Filtration - Ultrafiltration - Reverse Osmosis Sedimentation and Filtration - Coagulation and Flocculation	Electrochemical Methods - Electrocoagulation - Electrooxidation Ion Exchange Photocatalysis	Biodegradation - Microbial - Enzymatic Phytoremediation Constructed Wetlands

Because it is simple to use, efficient, and affordable, activated carbon adsorption has become a highly promising technique. Activated carbon, with its high surface area and porosity [N. Yalçın and V. Sevinç, 2000, D. Vasconcelos et al., 2021], offers an ideal medium for the adsorption of organic contaminants, including methyl orange dye molecules, from aqueous solutions.

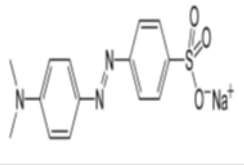
While numerous studies have investigated the adsorption of methyl orange dye onto activated carbon, there remains a need for a comprehensive understanding of the adsorption kinetics, particularly concerning commercial activated carbon products. Powdered activated carbons are widely available and utilized in water treatment applications, yet their performance and kinetics concerning methyl orange adsorption may vary depending on factors such as pore structure, surface chemistry, and manufacturing processes [B. Li, 2012, Huang, L., 2011].

2. Materials and Methods

2.1.Characterization of the chemical products used

- Methyl orange is a synthetic organic dye that is frequently used and falls under the azo dye class. Its chemical formula is $C_{14}H_{14}N_3NaO_3S$, and because of its ability to change color, it is frequently used as an acid-base indicator. Methyl orange is soluble in water and usually has a red or orange powdery appearance. Table 2 indicates the main physico-chemical characteristics of the model dye study.

Table 2: Physico-Chemical characteristics of Methyl Orange

Characteristics	Dye
Trade name	Methyl orange
Brute formula	$C_{14}H_{14}N_3NaO_3S$
Chemical structure	
Molar mass (g/mole)	327,33
λ_{max} (nm)	464
Family	Anionic
pH	3.1 (red) to 4.4 (orange)
Density (g/cm ³)	1.28
Melting point (°C)	>300

- In this work, powdered activated carbon, which is available commercially, was used. The characteristics of him are as follows: conductivity of 752 $\mu S/cm$, actual density of 945.3 kg/m^3 , effective size of 0.94 mm, and porosity of 24%.
- Solutions prepared in distilled water are used in all experiments; the conductivity of distilled water is less than 10 $\mu S/cm$, and its pH is approximately 6.0.

2.2.Experimental protocol

Methyl orange (MO) dye was used in batch experiments for this study, with concentrations ranging from 1 to 120 mg/L. A concentrated solution containing 1 g/L was diluted with distilled water to create MO solutions. To 50 milliliters of the MO solution, a predetermined quantity of powdered activated carbon (PAC) was added. Using a magnetic stirrer, the mixture was agitated at room temperature ($25 \pm 2^\circ C$) at a speed of 160 rpm. A Spectro Scan 80DV-UV/Vis spectrophotometer with a spectral range of 190 to 1100 nm was used to analyze the samples, which were collected at predefined intervals, filtered. The UV range that was appropriate for the dye was used for the analysis.

The adsorption yield, or rate, is computed using equation (1) to ascertain the efficacy of the dye removal procedure.

$$Re (\%) = \left(1 - \frac{C_t}{C_0}\right) 100 \quad (1)$$

C_0 and C_t : The initial and final dye concentration in the liquid phase.

3. Results and discussion

3.1. Effects of various factors on dye adsorption

a. Contact time

Figure 1 shows how the contact time affects the dye adsorption yield.

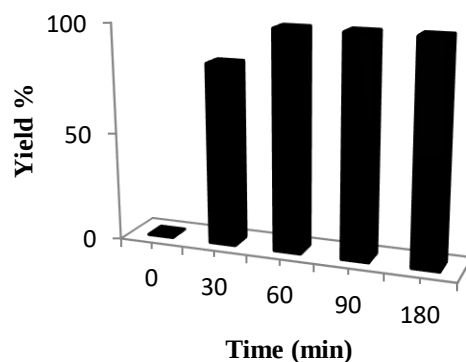


Figure 1: Adsorption kinetics of MO on PAC.

The results show that the dye adsorbs onto the PAC quickly and uniformly, with a rapid increase in adsorption percentage over time, as seen in the figure. This rapid increase indicates strong interactions between the dye molecules and the PAC surface in the solution [H.Tran, 2017, M. Al-Ghouti, 2003]. The effective adsorption process peaks after a 60-minute contact period, resulting in maximum decolorization. This is the ideal duration for maximum adsorption efficiency.

b. Quantity of PAC

By altering the amount of adsorbent in the water while maintaining a constant solution volume, the dye adsorption on PAC was investigated. For the tests, an hour was selected as the stirring period. The results are shown in figure 2.

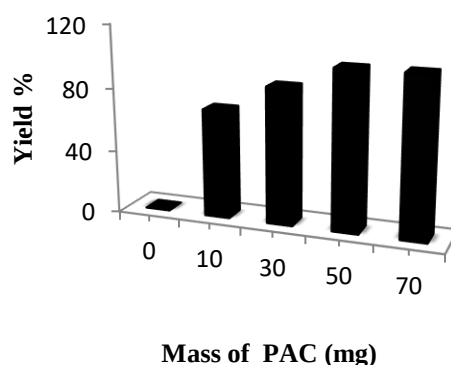


Figure 2: Dye removal efficiency as a function of PAC mass.

The results in the figure demonstrate that the adsorption efficiency of the dye increases with the mass of the adsorbent; PAC. This trend can be attributed to the increase in the number of available adsorption sites on the PAC surface, which enhances the capacity for dye fixation [George Z. et al., 2013]. However, it is observed that the adsorption efficiency reaches equilibrium at a mass of 50 mg

of adsorbent. This plateau indicates that beyond this point, additional adsorbent mass does not significantly enhance the adsorption efficiency, likely due to the saturation of adsorption sites [S. Wong et al., 2020] and the establishment of equilibrium between the dye in solution and the dye adsorbed on the PAC.

c. Dye concentration

The initial concentration of the dye significantly affects the adsorption of PAC. Figure 3 displays the results that were obtained.

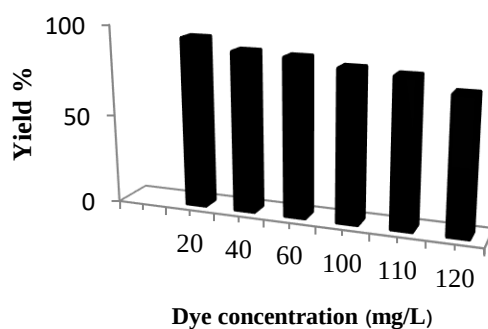


Figure 3: Effect of MO dye's initial concentration on adsorption

At lower concentrations, the methyl orange dye in the adsorption medium can interact with the active sites on the surface of the PAC adsorbent, as figure 5 illustrates, increasing the adsorption yields. Conversely, at high concentrations, adsorption efficiency decreased due to saturation of the adsorption sites.

d. Temperature

The effect of temperature is another crucial parameter to consider in adsorption study, as the process involves heat transfer between a liquid phase and a solid, which can be either exothermic or endothermic; we conducted experiments at successive temperatures of 25°C, 35°C, and 55°C. The results obtained are shown in figure 4:

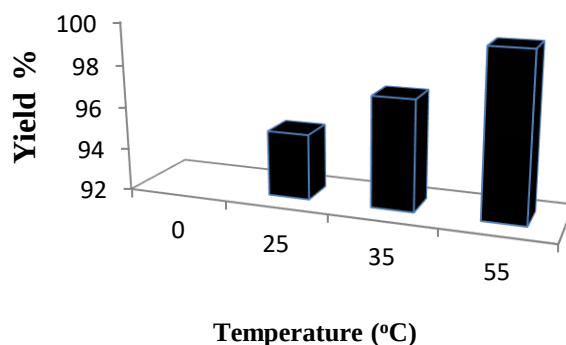


Figure 4: Effect of temperature on the adsorption of MO on PAC.

The findings showed that increasing the temperature to 55 °C improved adsorption. This implies that the higher temperature improved the dye molecules' kinetic energy and improved their diffusion towards the PAC surface [P. Senthil Kumar et al., 2014, A. M. Abbas et al., 2018]. The dye molecules were probably able to move more easily and get past energy barriers to reach the adsorption sites more successfully because of the higher thermal energy at 55°C. This implies that the adsorption process in this instance is endothermic, meaning that a rise in temperature enhances PAC's ability to absorb dye molecules.

3.2. Adsorption kinetics on PAC

Adsorption kinetics are essential in understanding how quickly a dye; Methyl Orange (MO), can be removed from an aqueous solution using PAC. The study of adsorption kinetics involves analyzing how the rate of adsorption changes over time and provides insights into the mechanisms controlling the adsorption process. The kinetics of adsorption were studied under the following conditions:

- Normal pH of the colored solution;
- Initial concentration of 100 mg/L of MO;
- Mass of activated carbon of 50 mg;

From the study, the adsorption equilibrium time on the PAC was determined to be 60 minutes.

3.2.1. Kinetic models

The equilibrium adsorption capacity (Q_e) is calculated according to equation (2) as follows:

$$Q_e = \frac{(C_o - C_e)V}{m} \quad (2)$$

In this section, first and second order kinetic laws were used.

The first-order kinetic model's equation takes the following form:

$$\text{Log}(Q_e - Q) = \text{Log } Q_e - \frac{K_1}{2.303} t \quad (3)$$

The second-order kinetic model's equation takes the following form:

$$\frac{1}{(Q_e - Q)} = \frac{1}{Q_e} + K_2 t \quad (4)$$

Figure 5 displays the curve representing the dye adsorption kinetics on the PAC according to this model.

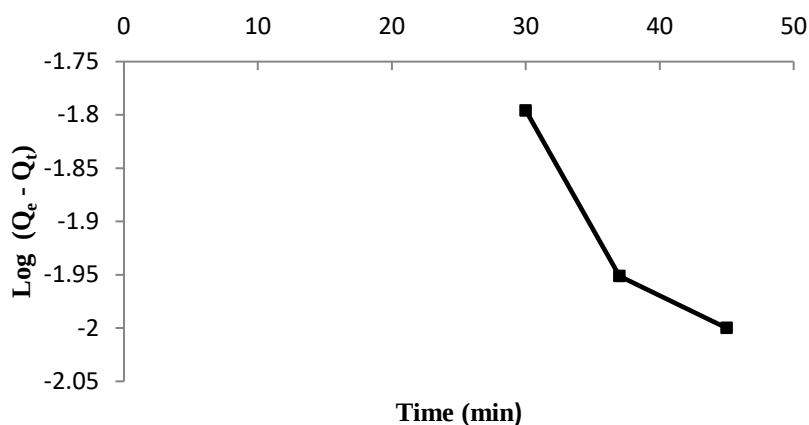


Figure 5: Graph of the linear form of the first-order kinetic model of MO utilized.

Figure 6 shows the results that were obtained with the pseudo-second-order model.

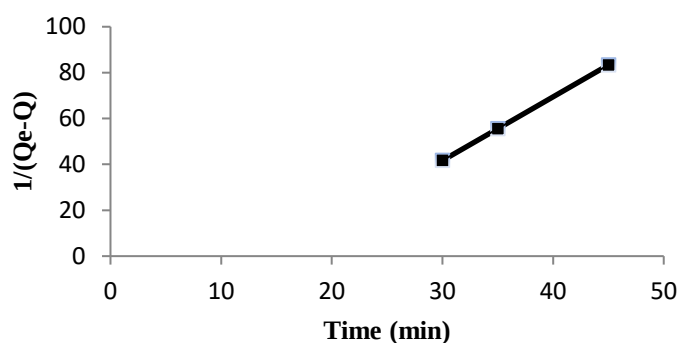


Figure 6: Graph of the linear form of the second order kinetic model of MO utilized.

The constants and values of the correlation coefficient R^2 according to the results obtained in figures 5 and 6 are grouped together in Table 3.

Table 3: Kinetic adsorption constants of methyl orange for two models

First order	K_1	0.085
	R^2	0.897
Second order	K_2	0.171
	R^2	0.999

This table helps in comparing the fitting of the adsorption data to different isotherm models, enabling the determination of the most appropriate model for describing the adsorption process of MO on PAC. Higher R^2 values indicate a better fit of the model to the experimental data, providing insights into the nature and capacity of the adsorption process.

From these results, we see that the pseudo-second-order kinetics model is the best for studying the kinetics of adsorption of dyes on PAC. The correlation coefficient (R^2) is very near to unity, which supports this conclusion.

3.2.2. Adsorption isotherms

Understanding and perfecting the adsorption process depend heavily on adsorption isotherms. Several theoretical models have been proposed to describe adsorption isotherms. This work will only focus on the most fundamental and widely used models; the Freundlich [Freundlich, 1926] and Langmuir models [Langmuir, 1918].

Linear equations for the Langmuir and Freundlich adsorption models ((5) and (6)) respectively:

$$\frac{C_e}{Q_e} = \frac{C_e}{Q_{\max}} + \frac{C_e}{Q_{\max} \times K_L} \quad (5)$$

$$\ln Q_e = \ln K_F + \left(\frac{1}{n}\right) \cdot \ln C_e \quad (6)$$

The graphical representation of Langmuir isotherm is as follows:

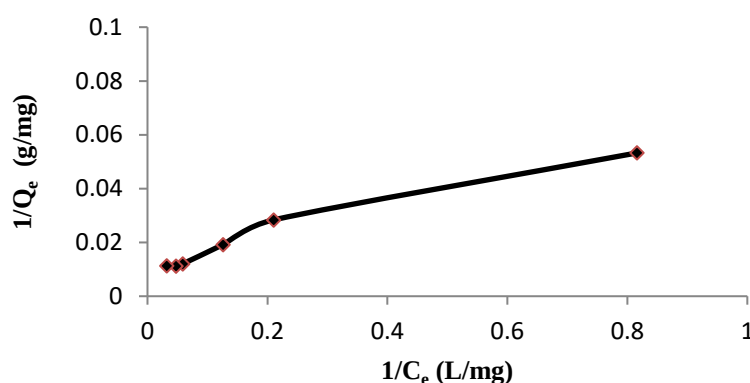


Figure 7: The linear Langmuir model for MO.

The graphical representation of Freundlich isotherm is as follows:

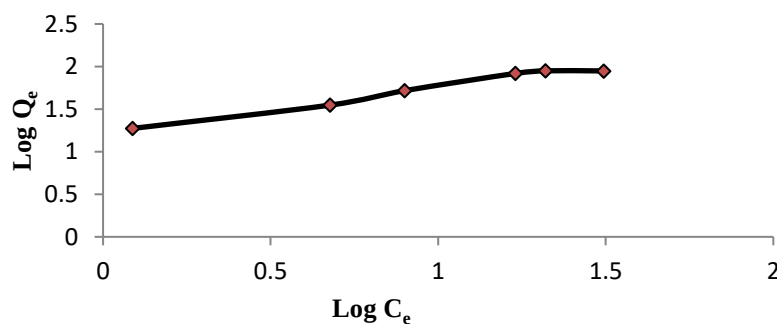


Figure 8: The linear Freundlich model for MO.

The results obtained enabled us to deduce the main parameters characterizing each model (Table 4). These parameters are essential for characterizing the adsorption process and understanding the nature of the interaction between the adsorbent and adsorbate. The Langmuir model is particularly useful for systems where monolayer adsorption dominates, while the Freundlich model is better suited for heterogeneous surfaces and multilayer adsorption.

Table 4: Main parameters characterizing the Langmuir and Freundlich Isotherm Models

Langmuir	$Q_{\max}$	87.720
	K_L	0.0950
	R^2	0.934
Freundlich	K_F	0.0940
	$1/n$	0.530
	R^2	0.988

The fixation of the dye on the PAC appears to follow the Freundlich model, based on the values obtained from the R^2 . This implies that there is a multilayer adsorption occurring on the PAC surface.

3.3. Thermodynamic study

The thermodynamic parameters of the adsorption process studied can be computed based on the temperature effect, comprising entropy (ΔS°), enthalpy (ΔH°), and free enthalpy (ΔG°). The difference between the free enthalpy of the products (solute adsorbed on the solid) and the total of the enthalpies of the reactants (solid + solute) is the variation in the free enthalpy of a system undergoing a transformation at constant temperature and pressure [C.D. Stoner, 2000, H. N. Tran et al., 2021]. It is expressed as follows:

$$\Delta G^\circ = \Delta H^\circ - T\Delta S^\circ \quad (7)$$

It is also possible to express the energy variation as a function of the equilibrium constant K_d .

$$\Delta G = \Delta G^\circ + RT \ln K_d \quad (8)$$

Given that $\Delta G = 0$ at equilibrium, it follows that:

$$\Delta G^\circ = -RT \ln K_d \quad (9)$$

We then obtain:

$$\ln K_d = \frac{\Delta S^\circ}{R} - \frac{\Delta H^\circ}{RT} \quad (10)$$

And

$$K_d = \frac{V(C_o - C_e)}{m \times C_e} \quad (11)$$

Figure 9 and table 5 bring together the results obtained from the thermodynamic study.

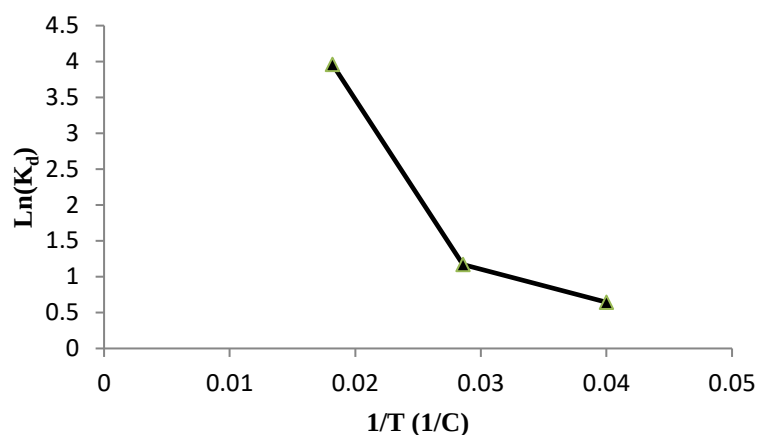


Figure 9: Study of thermodynamics

Table (5): Representation of thermodynamic parameters

Parameters		Values
ΔS^0 (J.mol ⁻¹ .K ⁻¹)		52.09539
ΔH^0 (kJ.mol ⁻¹)		124.8162
ΔG^0 (kJ.mol ⁻¹)	298 K	- 1596.356
	308 K	- 2998.853
	328 K	-10794.039
R ²		0.863

The positive values of entropy suggest that a medium disorder is present along with the adsorption. The process appears to be spontaneous based on the negative values of the free enthalpy ΔG^0 , indicating that the forces of attraction between the adsorbent and the adsorbate are strong enough to form on their own without the need for additional energy. Moreover, a drop in free energy values is observed to occur with rising temperature, which promotes the diffusion of adsorbed molecules into the interior pores of the adsorbent particles.

4. Conclusion

In conclusion, this work sheds light on both the kinetics and thermodynamics of methyl orange dye adsorption on powdered activated carbon, offering important insights into the mechanisms governing the adsorption process and its potential applications in water remediation. The study confirms that PAC is highly effective in removing dye from water, even at high concentrations.

The study of adsorption kinetics provides valuable insights into the adsorption mechanisms and helps in optimizing the design and operation of adsorption systems for wastewater treatment, the process being well described by the pseudo-second-order kinetic model. This suggests that the adsorption is

primarily controlled by chemisorption, with potential contributions from intraparticle diffusion. Understanding this kinetics is crucial for scaling up the process for industrial applications.

The adsorption isotherms are crucial in understanding how Methyl Orange interacts with powdered activated carbon. By analyzing these isotherms, one can determine the adsorption capacity, surface properties, and the nature of the adsorption process, which is vital for designing and optimizing water treatment systems.

Thermodynamic analysis clarifies the forces that propel the adsorption process, while kinetic modeling sheds light on the adsorption mechanisms and rate-limiting steps. In an environment where synthetic dye pollution is a major environmental concern, these results help to better understand the behavior of adsorption and offer useful information for the design and optimization of adsorption-based water treatment processes.

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