Xi'an Shiyou Daxue Xuebao (Ziran Kexue Ban)/
Journal of Xi'an Shiyou University, Natural Sciences Edition
ISSN: 1673-064X
E-Publication: Online Open Access
Vol: 67 Issue 12 | 2024
DOI: 10.5281/zenodo.14354418

SYNTHESIS AND EVALUATION OF ACTIVATED CARBON FROM BROWN ALGAE FOR ENHANCED METHYL-ORANG DYE REDUCTION

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Abstract

This study explores the preparation, characterization, and adsorption capabilities of activated carbon derived from brown algae for water pollutant removal. The preparation involved chemical activation with phosphoric acid at 170°C for 2 hours, followed by physical activation at 600°C for 2 hours. Characterization revealed functional groups (e.g., OH, CH_2, carboxylic) aiding adsorption, with a density of 1.5 g/cm³ and low ash content indicating high purity. Adsorption capacities for iodine and methylene blue were 950 mg/g and 245 mg/g, respectively. The activated carbon efficiently adsorbed methyl orange, with optimal conditions of pH 3, 1 g adsorbent dosage, and 40-minute contact time, achieving a maximum adsorption capacity (Q_m) of 9.3 mg/g. Adsorption modeling with Langmuir and Freundlich isotherms validated its suitability for water treatment. The study highlights the potential of renewable brown algae-derived activated carbon as an effective, sustainable solution for removing organic pollutants.

Index Terms: Activated Carbon, Brown Algae, Adsorption Isotherm, Water Treatment, Sustainable Materials.

1. INTRODUCTION

The increasing global demand for clean water and the growing awareness of environmental sustainability have intensified efforts to develop innovative water treatment technologies [1-5]. Adsorption using activated carbon has emerged as one of the most effective methods for removing organic and inorganic pollutants from water due to its high surface area, porosity, and exceptional adsorption capacity [6-8].

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However, conventional activated carbons often derived from non-renewable sources such as coal [9]or wood [10], present sustainability challenges. This has spurred interest in bio-based alternatives, which utilize abundant, renewable, and low-cost raw materials.

Brown algae, a widely available marine biomass, has gained significant attention as a promising precursor for activated carbon production. Its high carbon content, natural abundance, and unique chemical composition, including alginate and fucoidan, offer excellent potential for developing efficient adsorbents.

Unlike terrestrial biomass, brown algae also contain minimal lignin, simplifying the activation process and enhancing yield. Novel adsorbents derived from brown algae have demonstrated remarkable efficiency in removing contaminants such as heavy metals, dyes, and organic pollutants from wastewater, making them a sustainable and eco-friendly alternative to traditional materials [11, 12].

This study focuses on the preparation and characterization of activated carbon from brown algae using a combination of chemical activation (with phosphoric acid) and physical activation at high temperatures [9, 13-15]. The adsorbent's physicochemical properties, including functional groups, surface area, and pore structure, are systematically evaluated.

Its performance in adsorbing methyl orange, a common dye pollutant in industrial effluents, is assessed under varying operational conditions, such as pH, contact time, and adsorbent dosage. Furthermore, the adsorption mechanism is analyzed using isotherm models [16-18], including Langmuir and Freundlich equations, to provide insights into the material’s potential for practical applications.

This work contributes to the growing body of research on bio-based adsorbents and highlights the potential of algae-derived activated carbon as an efficient and sustainable solution for water pollution challenges [19,12].

It also advances the understanding of adsorption mechanisms, paving the way for developing novel materials tailored for specific environmental applications.

2 MATERIALS AND METHODS

2.1. Choice of Biomaterial

Algae are living organisms capable of performing oxygenic photosynthesis, and their life cycle typically occurs in aquatic environments. Our focus is on brown algae collected from the beach of Stidia in the Mostaganem province (figure 1).

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Figure 1 : Algue pour la préparation du charbon

2.2. Choice of adsorbed molecule

We focused on removing a dye commonly used in the textile industry: helianthine (figure 2), also known as methyl orange (MeO) or methyl orange dye.

This dye is a pH indicator used in chemistry to signal the presence of an acidic medium (turning pink-red) or a basic medium (turning yellow-Orange).

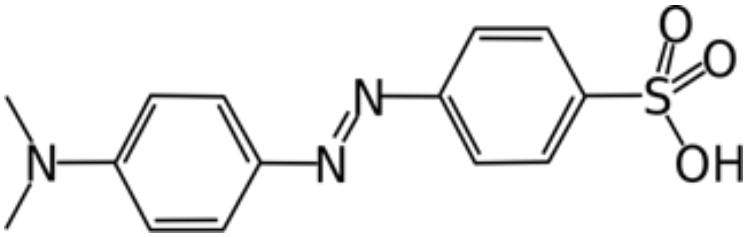

Figure 2: Structural Formula of Methyl Orange

2.3. Preparation of Activated Carbon

2.3.1 Raw Material Preparation

Brown algae, collected from Mostaganem Beach, Algeria, served as the raw material for the preparation of activated carbon. To eliminate impurities and surface debris, the algae were thoroughly washed with distilled water. The washed algae were air-dried under ambient conditions for 72 hours, followed by oven drying at 110°C for 6 hours.

This drying process resulted in a weight loss of approximately 30%, attributed to the removal of moisture and volatile compounds. The dried algae were then ground into a fine powder and sieved to obtain a uniform particle size of 125 µm, ensuring optimal activation efficiency and uniformity in subsequent processes

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2.3.2 Chemical and Physical Activation

The sieved algae powder was initially subjected to chemical activation using phosphoric acid (H_3PO_4) at a concentration of 40%. An impregnation ratio of 1:1 (w/v) between algae and acid was maintained to ensure effective activation. The mixture was heated at 170°C for 2 hours, promoting the breakdown of complex organic structures within the algae and facilitating the development of porosity. After the thermal treatment, the material was washed thoroughly with distilled water until a neutral pH was achieved (figure 3), ensuring the removal of residual acid. Subsequently, the material was oven-dried overnight at 110°C to prepare it for physical activation.

To enhance the porosity and adsorption efficiency of the activated carbon, the chemically treated material was subjected to physical activation. This step involved placing the material in a muffle furnace and heating it at 600°C for 2 hours. The high-temperature treatment eliminated any remaining volatile substances, further increased the specific surface area, and optimized the material's adsorption properties. The resultant activated carbon exhibited improved porosity and structural integrity, making it highly effective for pollutant adsorption applications.

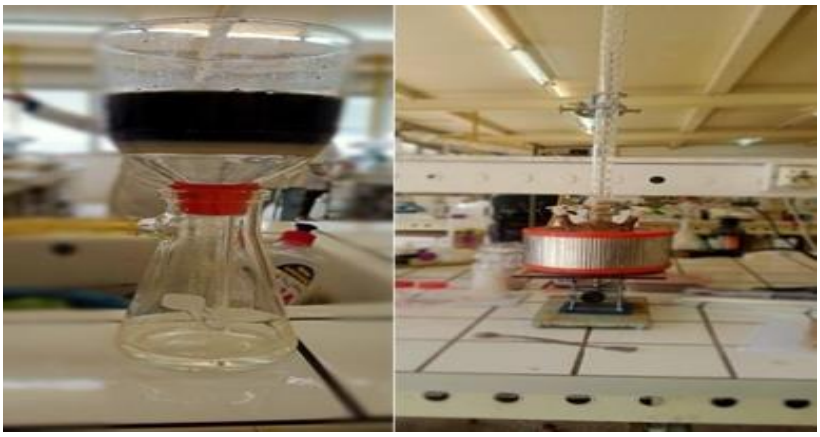

Figure 3: Washing and Filtration under Pressure Simultaneous

2.4. Characterization of Activated Carbon

To evaluate the physicochemical properties and adsorption potential of the activated carbon derived from brown algae, several characterization techniques FTIR, and pHpzc were employed.

3. RESULTS AND DISCUSSION

3.1 Characterization

The activated carbon derived from brown algae underwent extensive characterization to assess its physical and chemical properties, which are critical for its effectiveness as an adsorbent. Below is a detailed account of the findings (table 1):

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Table 1: physical and chemical properties of activated carbon derived from brown algae

Asch contient	%Asch	1,972%
humidity	%H	24%
Iodine Index	Id	945,26mg/g
MB Index	I_{BM}	609,794 mg/g

The low ash content, combined with high iodine and methylene blue indices, confirms the activated carbon's strong adsorption performance and suitability for diverse environmental applications. Although the humidity level suggests the need for pre-treatment or drying in some scenarios, it does not diminish the material's potential. These results position the activated carbon as a promising, sustainable, and efficient adsorbent for removing both small and large organic and inorganic pollutants, particularly in wastewater treatment processes [20].

3.1.1. pH at the point of zero charge pHpzc

pH at the point of zero charge is an important parameter in adsorption phenomena as it allows for the determination of the net charge on the surface of an adsorbent material. This information is essential for understanding adsorption mechanisms, especially when electrostatic forces are involved.

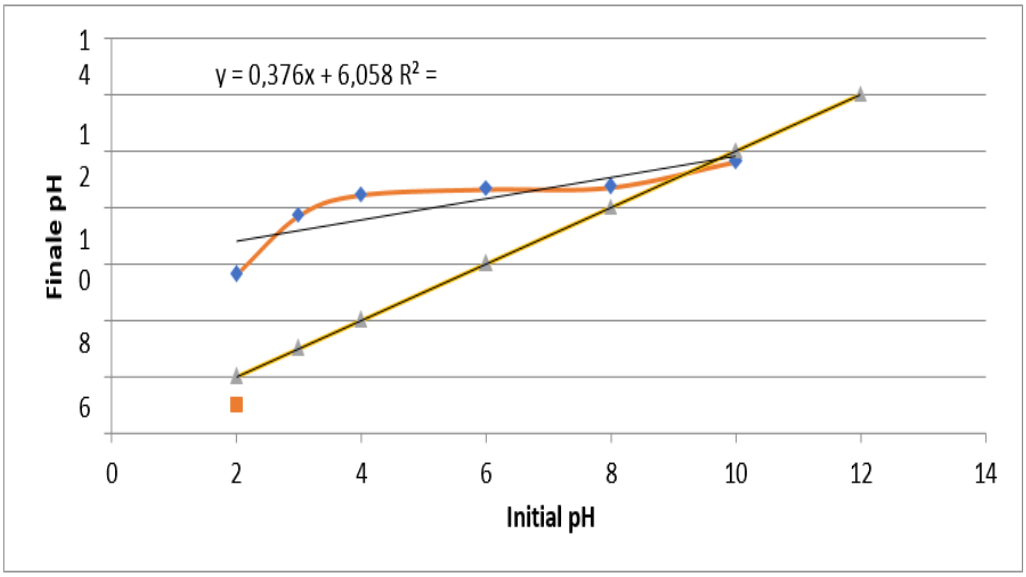

Figure 4: pH at the point of zero charge
(m=0,2 mg; V= 20 ml; C = 1N; t = 24h; T = 25 °C)

The pHpzc (point of zero charge) of activated carbon is 8 which is more favorable for cation adsorption, while anion adsorption would be more effective below these pH levels.[21].

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It is important to highlight that the adsorption of cations on all adsorbents will be favorable at pH values higher than the pHpzc, while the adsorption of anions will be favored at pH values lower than the pHpzc. This is because, at a pH below the pHpzc, the surface of the activated carbon is protonated (acidic), giving it a positive charge. Conversely, at a pH above the pHpzc, the surface of the activated carbon is deprotonated (basic), resulting in a negative charge.

Understanding the effect of pH on adsorption is essential, as it can have significant implications for selecting the appropriate adsorbent for a specific application. For example, if the goal is to adsorb cations, it is better to work at pH values higher than the adsorbent material's pHpzc. Conversely, if the goal is to adsorb anions, it is preferable to work at pH values lower than the pHpzc.

3.1.2. FTIR Spectroscopy

The infrared spectra were recorded using a BRUKER ALPHA IR spectrometer. All analyses were performed in the mid-IR range, specifically within a wavelength range of 500 to 4000 cm^{-1}. The figure below (figure 5) represents the IR spectrum of the prepared charcoal. The interpretation of the spectrum is provided in Table 2[22].

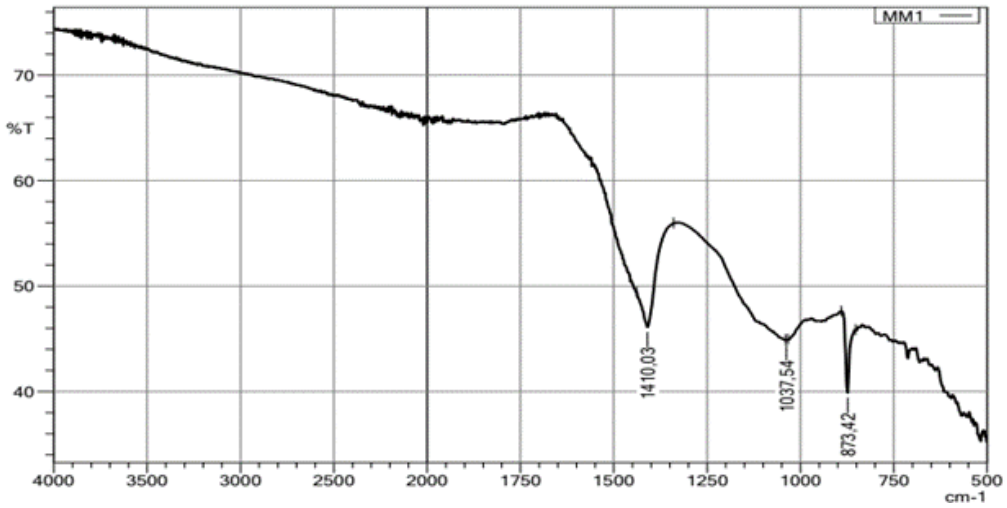

Figure 5: Brown algae activated carbon FTIR

Table 2: Characteristic Peak of activated carbon derived from brown algae

Peak in Spectrum (cm^{-1})	Characteristic Peak (cm^{-1})	Corresponding Bond or Vibration
1410.03	1410	C-H bending in alkanes
1037.54	1037	C-O stretching in esters
873.42	873	C=C bending in aromatic rings

The functional groups identified confirm the chemical activation process's success in creating active sites conducive to adsorption.

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3.2. Adsorption of Methyl Orange on the Prepared Carbon

Figure 6 illustrates an experimental process involving the adsorption of a substance (likely Methylene Orange, abbreviated as MeO) using Activated carbon derived from brown algae (AC) as the adsorbent.

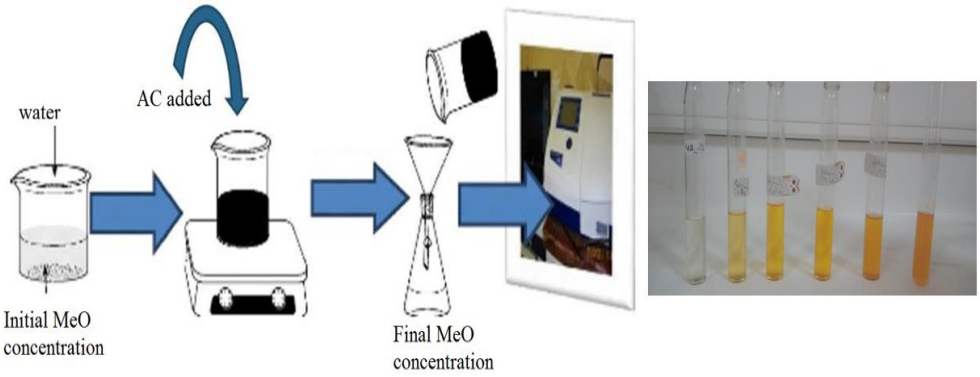

Figure 6: Application Process of Prepared Carbon for the Removal of Methyl Orange Dye

3.2.1. Parametric Study

a. Effect of Contact Time

The figure 7 shows the evolution of the amount of methyl orange adsorbed as a function of contact time. It is observed that the adsorption quantity increases over time until it reaches a maximum around 40 minutes, removing all pollutants. At this point, the amount of dye adsorbed by the carbon reaches approximately 8.960 mg/g. After 40 minutes, this amount remains constant with a slight decrease.

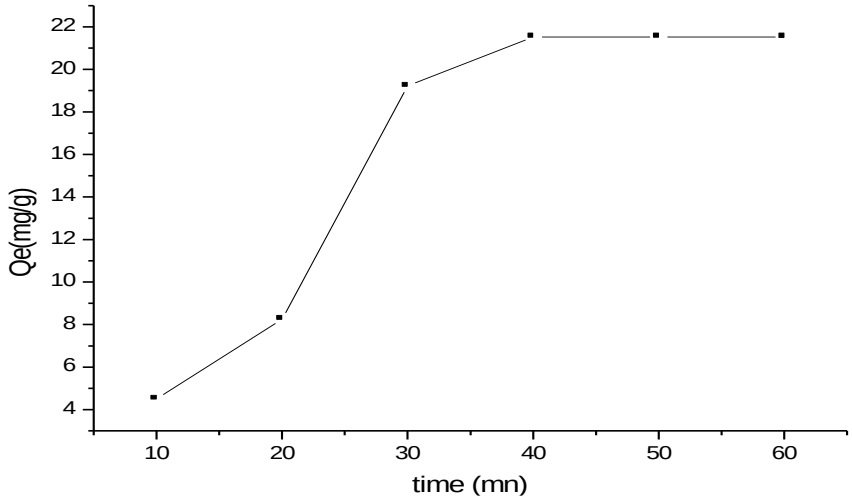

Figure 7 : Effect of contact time on methyl orange adsorption (m= 1g; V= 20 ml; C = 0.1N; T = 25 °C)

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At the start of the adsorption process, rapid reactivity is observed due to the significant number of active sites present on the surface of the biosorbent. When the contact time is extended, the dye has sufficient time to diffuse into the pores of the biosorbent.

The non-adsorbed portion can be attributed to the saturation of the biosorbent's surface, meaning all adsorption sites are occupied. Consequently, it is concluded that the optimal adsorption time for this study is approximately 40 minutes.

b. Effect of pH

We studied the effect of pH on the adsorption of methyl orange (MeO) following this protocol: the pH of a 20 ml methyl orange solution with a concentration of 0.1 N was adjusted to initial values ranging from 2 to 12. Then, 1g of porous activated carbon (PAC) was added, and the solution was stirred for 9 hours.

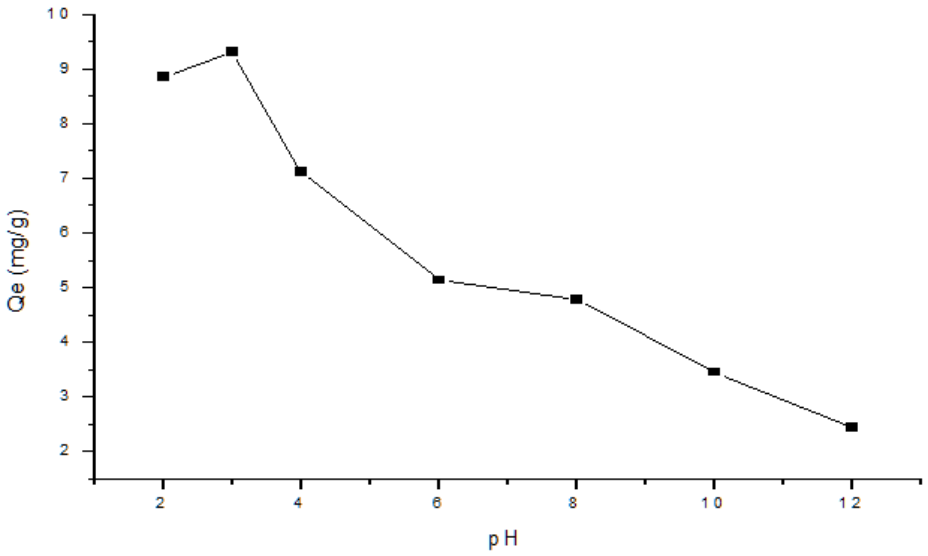

Figure 8 : Effect of pH on MeO adsorption (m=1g; V= 20 ml; C= 0.1 N; t = 40 min; T = 25 °C)

The figure 8 shows the effect of pH on methyl orange adsorption. It is observed that the maximum adsorption occurs at pH = 3.

The adsorption quantity decreases with increasing pH, with a value of 9.312 mg/g at pH 3, which decreases to 2.443 mg/g at pH 12. This can be explained by the fact that at acidic pH, the adsorption process of the dye by the algae-based carbon involves electrostatic interactions.

The protonated amine groups of the biosorbent interact with the anionic groups of the dye. At high pH levels, fewer adsorption sites on the carbon are available for methyl orange. In conclusion, the optimal pH for this study is considered to be 3.

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c. Effect of Initial Dye Concentration

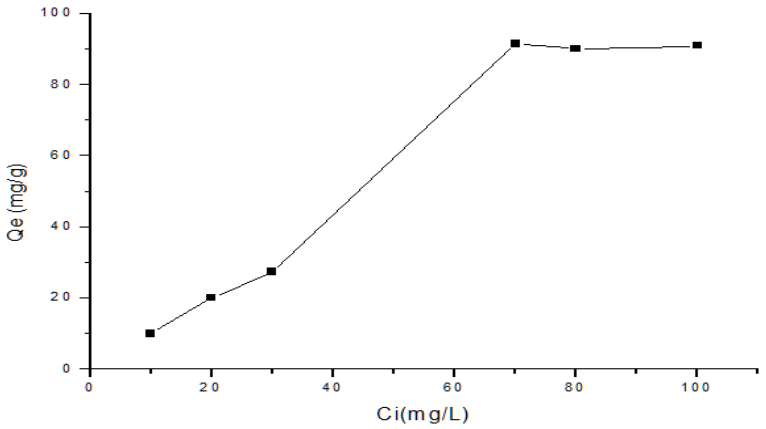

Figure 9: Effect of initial dye concentration on adsorption (m = 1g; V= 20 ml; pH = 3; t = 40 min; T = 25 °C)

The figure 9 shows the effect of the initial concentration of methyl orange on biosorption. The results indicate that the adsorption capacity increases with an increase in the initial dye concentration. Beyond a concentration of **70 mg/l**, a plateau is observed due to the saturation of active biosorption sites in the presence of a high dye content. This sorption characteristic indicates that surface saturation depends on the initial methyl orange concentration. At low concentrations, sorption sites quickly fix methyl orange. In conclusion, the optimal initial concentration for this study is **70 mg/l.**

d. Effect of Adsorbent Quantity

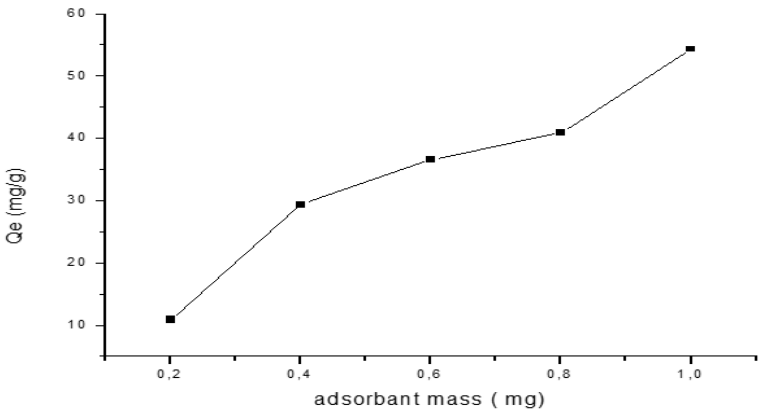

Figure 10: Effect of adsorbent mass on MeO adsorption (C = 75 mg/l; V = 200 ml; t = 45 min; pH = 3; T = 25 °C)

From the presented figure 10, it is observed that the efficiency of methyl orange biosorption increases as the biosorbent dose increases. In conclusion, the results of this study suggest that the optimal biosorbent mass for this application is 1g.

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3.2.2. Adsorption Isotherm Modeling

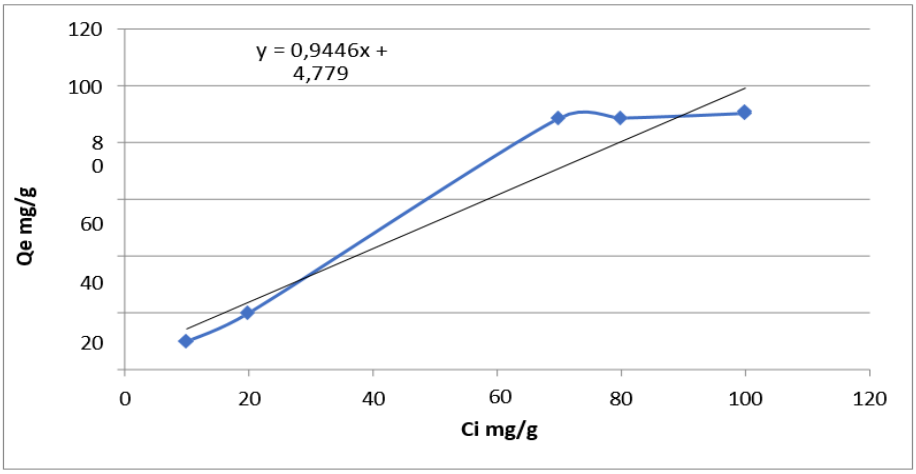

Figure 11: Equilibrium adsorption isotherm (m = 1g ; V = 20 ml ; pH = 3 ; t = 45 mn ; T = 25 °C)

The equilibrium adsorption isotherm follows a type L isotherm (figure11), which is often modeled using Langmuir and Freundlich models.

a. Langmuir model

The Langmuir adsorption isotherm assumes that adsorption occurs at specific homogeneous sites of the adsorbent and has been successfully applied for many monolayer adsorption processes. Plotting Ce/Qe as a function of Ce (figure 12) gives a straight line with a slope of 1/Qm and an intercept of 1/b, allowing for the determination of equilibrium parameters Qm and b.

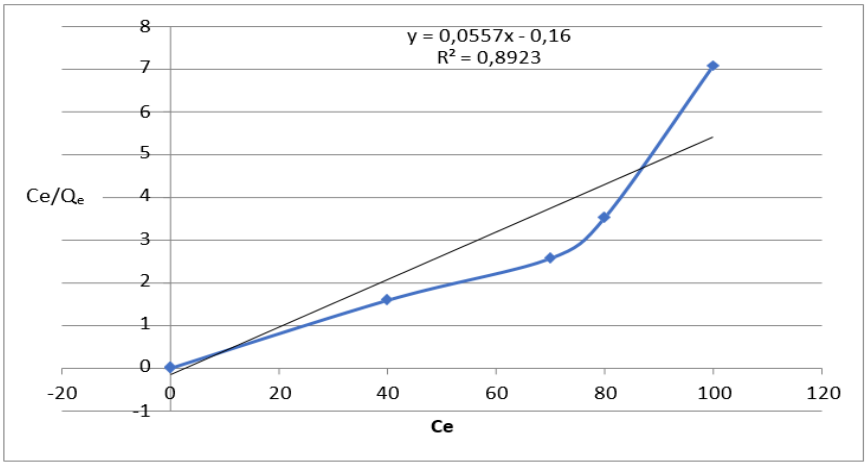

Figure 12: Langmuir adsorption isotherm modeling for methyl orange adsorption by carbon

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b. Freundlich Model

The Freundlich isotherm is an empirical equation based on an exponential distribution of adsorption sites and energies. The parameters Kf and n are determined from the plot of log Qe versus log Ce (figure 13).

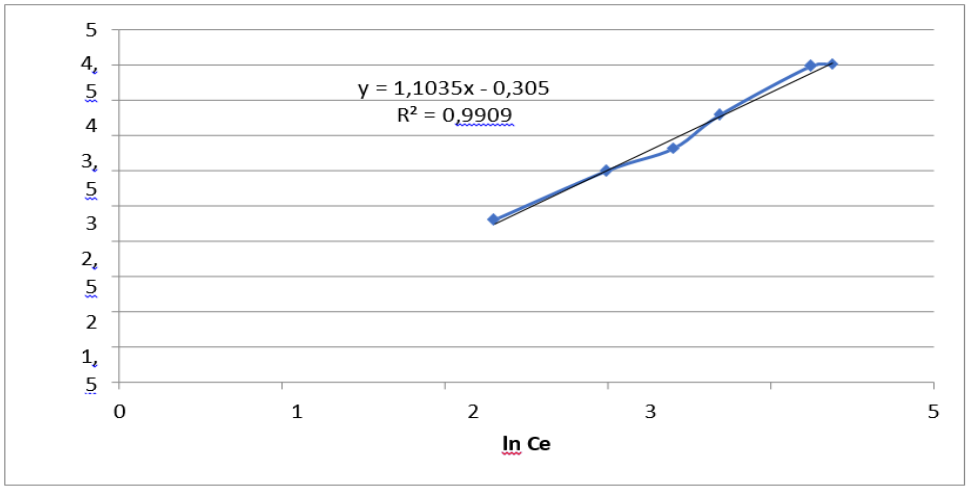

Figure 13 : Freundlich adsorption isotherm modeling for methyl orange adsorption by carbon

c. Summary of Modeling Results

The adsorption isotherm modeling of methyl orange by activated carbon from brown algae is well-described by the Freundlich model, with a correlation coefficient of **0.99.** This indicates that multilayer adsorption occurs on heterogeneous surfaces.

Table 3: Summary of Modeling Results

Model	Isotherm Constant	R2
Langmuir	Qm=11.547, b=−0.384	0.8244
Freundlich	K=0.7330, 1/n=1.1035	0.9909

6. CONCLUSION

This study successfully developed bio-based activated carbon from brown marine algae, specifically collected from Stidia Beach in the wilaya of Mostaganem, Algeria. The synthesis involved a two-step activation process: chemical activation using 40% phosphoric acid (H_3PO_4) at 170°C for 2 hours, followed by physical activation at 600°C for 2 hours. This methodology resulted in a material with a highly porous structure, characterized by abundant microspores and mesopores, which significantly enhanced its adsorption efficiency and specific surface area. The activated carbon was comprehensively characterized to evaluate its physicochemical properties and adsorption performance. The iodine index, measuring 945 mg/g, demonstrated a strong capacity for adsorbing small molecules, while the methylene blue index of 609 mg/g indicated

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effective adsorption of macromolecules. The point of zero charge (pHpzc) was determined to be 8, providing insights into the surface charge behavior of the material and its interaction with anionic and cationic species. Fourier-transform infrared spectroscopy (FTIR) analysis confirmed the presence of functional groups, including hydroxyl (OH) and carboxyl (COOH), which are critical for adsorption interactions.

A parametric study on the removal of methyl orange dye, a common industrial pollutant, identified optimal adsorption conditions: a contact time of 40 minutes, pH 3, an initial dye concentration of 70 mg/L, and an adsorbent dosage of 1 g. Under these conditions, the activated carbon achieved a maximum adsorption capacity of 9.3 mg/g. The adsorption behavior was modeled using isotherm equations, with the Freundlich model demonstrating superior fit (R^2 = 99.09%), suggesting multilayer adsorption on heterogeneous surfaces. In contrast, the Langmuir model, which assumes monolayer adsorption, provided a weaker correlation (R^2 = 82.44%).

These results establish the potential of brown algae-derived activated carbon as an effective, sustainable, and renewable adsorbent for environmental applications, particularly in water treatment. The material's high adsorption capacity for organic pollutants, including dyes, highlights its suitability for both aqueous and gaseous phase applications. Future work should focus on scaling up the production process, broadening the spectrum of pollutants addressed, and investigating regeneration methods to ensure the long-term sustainability and practicality of this bio-based adsorbent. This study contributes to the growing field of renewable materials for environmental remediation, leveraging abundant marine biomass to address critical pollution challenges.

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