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Toward the discovery of an effective method for treating industrial effluents: Application of adsorption on activated carbon based on argan kernels

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Abstract: Water pollution caused by industrial effluents is a pressing issue in many countries, posing a threat to the environment. To combat this problem, scientists employ various methods, including physico-chemical processes like adsorption, to purify effluents. The present study focuses on the application of a naturally biodegradable adsorbent derived from argan trees located in southern Morocco, aiming to reduce the harmful effects of pollutants through a physico-chemical adsorption process. To highlight the purification power of the investigated natural adsorbent, an aqueous solution of dye methylene blue was prepared and used as synthetic effluent. The conducted experiments showed a significant removal rate within the first 15 minutes. In addition, the influence of adsorbent mass and concentration on the purification rate were also explored, and the results highlight the effectiveness of argan powder in adsorbing MB. By promoting pollution reduction, waste recovery, and water recycling, this research underscores the potential of argan kernels to benefit local communities.

Keywords: *Wastewater; Carbon, Water Treatment; Argan, Adsorption; Coloured Discharge; Isotherms.*

1. Introduction

Water is an essential part of our quality of life (Muhamed *et al.*, 2022). Its pollution causes environmental problems and limits agricultural, industrial and even tourist activities (El Attaoui *et al.*, 2023; Bouknana *et al.*, 2014; Kadirvelu *et al.*, 2003 ; Jain *et al.*, 2003). Above all, it reduces the quality and quantity of drinking water by contaminating groundwater. In many countries with more or less arid climates, water resources have become increasingly scarce over the last few decades. Most arid and semi-arid countries are now calling for better water conservation. Rainfall is scarce and irregular, evaporation is significant, and groundwater is largely exploited (Chaoua *et al.*, 2017).

Most of Morocco's water resources are limited. The total amount of rainfall over the whole country in an average year is estimated at around 150 billion m³, of which 121 billion goes to evapotranspiration and around 29 billion to runoff (Stenelvie *et al.*, 2019). Indeed, surface water flows are globally estimated at around 16 billion m³. While the potential of usable groundwater resources is estimated at around 4 billion m³ per year, more than 50% of these resources are distributed in the central and northern regions of the country (32 underground aquifers and more than 46 surface aquifers) (Stenelvie *et al.*, 2019). This means that many regions of Morocco are experiencing increasing water shortages, while water availability is limited and unevenly distributed. This water shortage is currently being exacerbated by the development of the industrial sector, which is leading to heavy use of water resources, as well as pollution and a reduction in the qualitative value of surface and groundwater.

In addition to water scarcity and the increase in wastewater production, which can lead to environmental pollution, there is the health problem of reusing untreated wastewater. In Morocco, household wastewater is often reused without prior treatment, with negative effects on human health, such as infectious diseases like typhoid and cholera, which are a real scourge for public health. Contributing to human health and protecting the environment are now key priorities for this country.

To do this, its wastewater generally undergoes several stages of treatment to purify the water for possible reuse and discharge into the natural environment. Numerous physical, chemical and biological techniques have been developed and tested for the treatment of dye-laden effluents. These include coagulation-flocculation (Hameed *et al.*, 2009), oxidation and ozonation (Malik *et al.*, 2003; Koch *et al.*, 2002), membrane filtration (Majouli *et al.*, 2012; Ciardelli *et al.*, 2001), and so on. Activated carbon is the most commonly used because of its high adsorption capacity for organic contaminants (Younssi *et al.*, 2015; Ahmad *et al.*, 2009). However, the high cost of activated carbon limits its use to the treatment of large volumes of polluted water. The search is therefore on for low-cost adsorbents, preferably derived from natural elements. Of all physical treatments, the adsorption process is considered the most effective method for decontaminating water (Thinakaran *et al.*, 2008; Bhattacharya *et al.*, 2005) and removing industrial effluents such as dyes (Gedam *et al.*,2019; Vallejo *et al.*, 2023; Id El Mouden *et al.*, 2012; Belaid *et al.*, 2011). At present, the sorption process is proving to be one of the most effective and interesting ways of treating wastewater containing dyes (Gupta *et al.*, 2003; McKay *et al.*, 1998; Walker *et al.*, 2003; Zeynolabedin *et al.*, 2015; Allen *et al.*, 1994). It has become the benchmark experimental method because of its performance and ease of use (Ramirez *et al.*, 2023; Dhedan *et al.*, 2012).

Today, the focus is on the use of biodegradable adsorbents that are inexpensive, locally available and of natural origin. Recently, activated carbon synthesised from agricultural residues has been widely exploited as the adsorbent for industrial effluent recovery, including coloured effluents, due to its highly porous structure, high specificity and high adsorption capacity. Recycling agricultural waste without generating pollutants is a major challenge for environmental protection (Soon *et al.*, 2011; Aaddouz *et al.*, 2023). In this study, we investigated the adsorption of methylene blue colorant on argan activated carbon. It's a colorant used mostly in the following industries: textiles, paper, rubber, plastics, leather, cosmetics, pharmaceuticals, and food. These industries reject waste containing dye residues. As a result, very low concentrations in these wastes are very visible (Alkan *et al.*, 2004; Turhan *et al.*, 2013).

The argan tree is endemic to Moroco, where it has an essential socio-economic and environmental role. It is the country's second largest forest species, covering an area of 828,000 ha (Charrouf *et al.*, 2002). Traditionally, Moroccans use argan fruits to extract edible oil (Miklavčič *et al.*, 2020). This oil has many uses. In nutrition, it has been on the market for a very long time as a basic ingredient and sometimes as the only source of vegetable fat in "Amazighdiand" (Khallouki *et al.*, 2017; Morton *et al.*, 1987). Over the past twenty years, argan oil gradually gained a reputation as the most expensive natural oil on the market, popular and sought-after in the cosmetics industry, where its human health benefits are widely recognized (El Abbassi *et al.*, 2014; Monfalouti *et al.*, 2010). As of 2022, the argan market accounted for USD 299.45 million and is projected to grow by USD 11.4 million by 2030 (Santoro *et al.*, 2023). Furthermore, argan oil plays essential role in corrosion inhibition via the action of numerous compounds at the metal surface to form a barrier for the arrival of aggressive ions as H^+ or dissolved gases as dissolved oxygen (Afia *et al.*, 2014; Laaroussi *et al.*, 2022)

The National Agency for the Development of the Oasis and Argan Zones reports a total area of approximately 2.5 million hectares in the Argan Biosphere Reserve, of which 830,000 ha are devoted to the argan tree. The argan tree is already making an active contribution to the economic and social development of the Souss-Massa, Essaouira and Guelmim Oued Noun regions.

Today, the argan industry is supported by two new national development programs: Green Generation and Moroccan National Forests (2020-2030). These strategies set out the programs and mechanisms needed to establish a new model for the development of the sector as a true economy, one that values people and preserves the ecosystem. At the same time, they are seeking, through proximity and a participative approach, to operate within the framework of a regional economy, a balanced human development where the gender approach will be privileged *("Réflexions Sur l'argan Du Maroc et Son Développement – AgriMaroc.Ma" n.d.*). These strategies and proportions are reasonable to facilitate their transformation into activated carbon production materials, the processing procedure is profitable, inexpensive. The activated carbon obtained is considered to be a high-performance adsorbent for water treatment and, above all, for the decontamination of effluents used in the textile industry.

The aim of this study is to valorize the use of agricultural residues for the treatment of industrial effluents. In this study, we present the experimental procedure followed, which involved the various stages of preparing adsorbent samples and activated carbons based on waste from natural sources such as argan kernels from the town of Essaouira. In addition, we prepared a solution of a dye that is widely used in the textile industry: methylene blue. We then carried out adsorption tests with this dye with the aim of eliminating the pollutants by adjusting several parameters such as the quantity of adsorbent used, the pH, the temperature and the concentration, and applying the two Langmuir and Freundlich adsorption isotherms, and presenting and discussing the various results obtained.

2. Materials and Methods

2.1. Effluent preparation

The Methylene blue is an organic dye in the form of a blue powder. It dissolves easily in water and is commonly used as a dye in the textile and paper industries. Its chemical structure is shown in **Fig. 1**.

Figure 1. Chemical structure of the used colorant (Chowdhury *et al.*, 2012).

To carry out the adsorption tests, a solution of methylene blue at a concentration of C_0 = 50 mg/l is prepared by dissolving 5 mg of the colorant in 100 ml of distilled water as shown in **Figure 2**. The resultant mixture is agitated over 3 minutes.

2.2. Preparation of the adsorbent material.

Before starting the charcoal black production process, the argan stones were pre-treated. First, they are rinsed in water to eliminate dirt as well as impurities. Next, they are then cured for 24 hours at 105°C using an oven. Drying extends the shelf life of these cores by reducing their moisture content, thus preventing the development of mold and bacteria. Once dried, the cores were crushed using an electric grinder to facilitate sieving. The cores were then sieved to retain only the 160 µm fraction for experimental purposes. the **Figure 3** illustrates the carbon preparation steps.

Figure 2. The mother dye solution (BM).

Figure 3. Adsorbent preparation steps.

2.3. Chemical activation of coal by phosphoric acid (H_3PO_4)

Amounts of 10 g of argan kernel charcoal were pyrolysed at 350°C in a muffle furnace for 2 hours. After calcination, a quantity of 1.25 g was impregnated with the activating agent solution (5 ml of H_3PO_4 + 95 ml of distilled water) for 15 min, under stirring at ambiant temperature as shown in **Figure 4**. The activated charcoal was filtered and rinsed using distilled water as well as sodium hydroxide until a pH of 7.6 was obtained, after which it is cured in the oven at 105°C over a period of 24 hours. The activated carbon obtained is stored in a hermetically sealed bottle until use.

2.4. Chemical activation of coal by caustic soda (NaOH)

A quantity of 2 g of argan charcoal is impregnated at room temperature with the activating agent solution (1 g of caustic soda (NaOH) + 100 ml of distilled water). The resulting mixture is stirred for 1 hour as **Figure 5** indicates. The activated carbon was filtered, washed in distilled water, and dried for 24 hours under oven conditions at 105°C. The activated carbon obtained is stored in a hermetically sealed bottle until use.

Figure 4. Chemical activation of carbon by phosphoric acid.

Figure 5. Chemical activation of carbon by caustic soda.

3. Results and Discussion

3.1. Characterization analysis

For the characterization of our carbon produced, we applied the metal characterization technique to reveal the structure of the metals. For this purpose, the XRD laboratory at the Faculty of Science in Ain Chok carried out an analysis of the prepared sample. **Figure 6** below gives the result of the analysis:

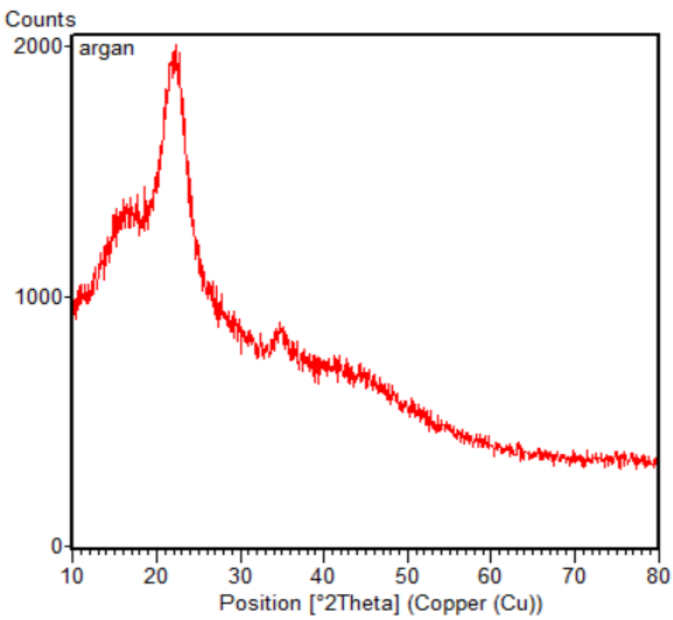

Figure 6. Diffraction of X-rays from unactivated carbon

Figure 6 illustrates the spectrum of carbon black not activated with activating agent (phosphoric acid). Its structure shows that the carbon black structure is amorphous and incomplete, which means that some particles have semi-amorphous phases. The peak at around $2\theta = 20°$ corresponds to the structure of carbon black.

3.2. Adsorption Experiments

We tested the adsorption capacity of dye (BM) by activated carbon, using 0.02 g of this carbon mixed into 100 ml of the colorant solution under stirring (300 rpm) for 15 minutes. After some time in contact, samples were taken and filtered. Their absorbances were then measured and the concentrations finally deduced using the calibration curve.

3.3. Analytical Methods

In accordance with standard methods for monitoring and analyzing water and wastewater, the absorbance of the methylene blue solution is determined using a spectrophotometer UV-visible at 665 nm, corresponding to the maximum absorption peak of the methylene blue. The concentration of colorant and the rate of elimination were determined from the colorant calibration curve.

Spectrophotometry is a procedure commonly used in chemistry for quantitative analysis, used to determine the amount of light that is absorbed in solution. We used a spectrophotometer (UV-6300PC) as illustrated on **Figure 7** to determine the transmittance or absorbance of a sample in a specific wavelength range, generally between 200 nanometres and 800 nanometres.

Figure 7. UV-visible spectrophotometer.

It is used to determine the residual concentrations of methylene blue. To do this, we prepared 5 diluted daughter solutions of the initial colorant solution at a concentration equivalent to C= 50 mg/l as shown in **Figure 8.** The corresponding absorbances were then measured using a UV-visible spectrophotometer, with the dye absorption wavelength (BM) set to 665 nm, whose results are presented in **Table 1**. The values in **Table 1** represent the calibration for various concentrations ranging from 1 to 15 mg/l of methylene blue. They take the form of a calibration curve plotting absorbance versus concentration, as illustrated on **Figure 9**. The amount of dye Q_e adsorbed and the percentage removal R% are calculated from the absorbances obtained by spectrophotometric analysis and the BM calibration curve using the following equations:

$$Q_e = ((C_0 - C_e) * V) / m$$

$$R\% = ((C_0 - C_e) * 100) / C_0$$

The **Table 2** shows the results obtained for the 5 solutions.

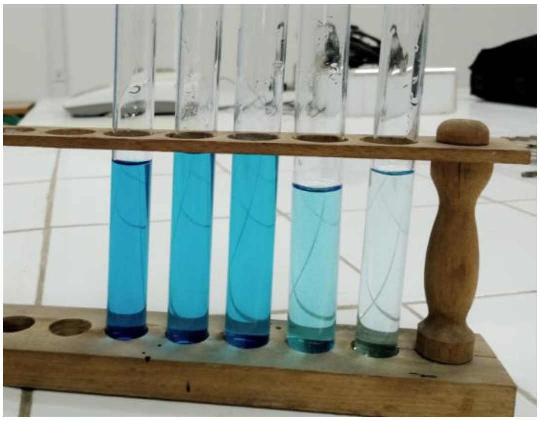

Figure 8. Diluted dye solutions (BM)

Table 1. Absorbance values for methylene blue by spectrophotometric analysis.

Concentration(mg/l)	0	1	3	9	12	15
Absorbance	0.0553	0.2253	0.5956	1.5309	1.9544	2.2543

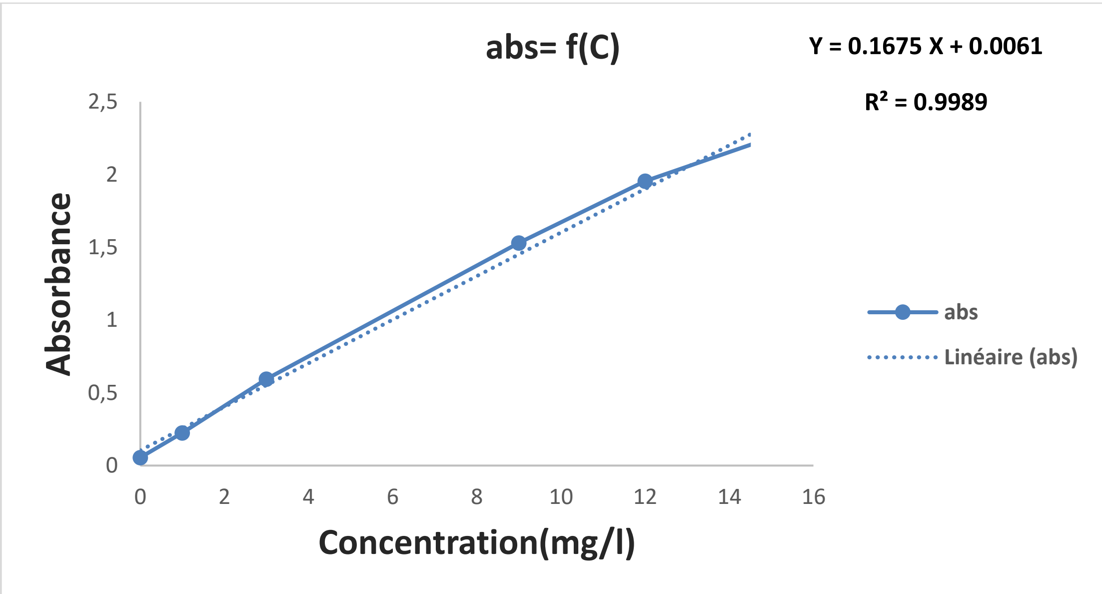

Figure 9. Calibration curve (BM)

Table 2. The results obtained with the five solutions of the dye used.

C_0 (mg/l)	10	20	30	40	50
Absorbance	0.946	1.007	1.021	1.023	1.026
C_e (mg/l)	5.611	5.975	6.059	6.071	6.088
% Elimination	43.89	70.125	79.803	84.822	87.824
Quantity adsorbed (mg/g)	21.945	70.125	119.705	169.645	219.56

3.4.Elimination Percentage

The curve on **Figure 10** shows that when the initial concentration of BM increases, the percentage of elimination decreases (keeping the mass of the adsorbent the same). For good elimination of methylene blue, the mass of the adsorbent must therefore be increased when the concentration of this dye increases, because by increasing the mass of the adsorbent, its specific surface area increases and so does the number of adsorption sites available, leading to an increase in the amount of dye adsorbed.

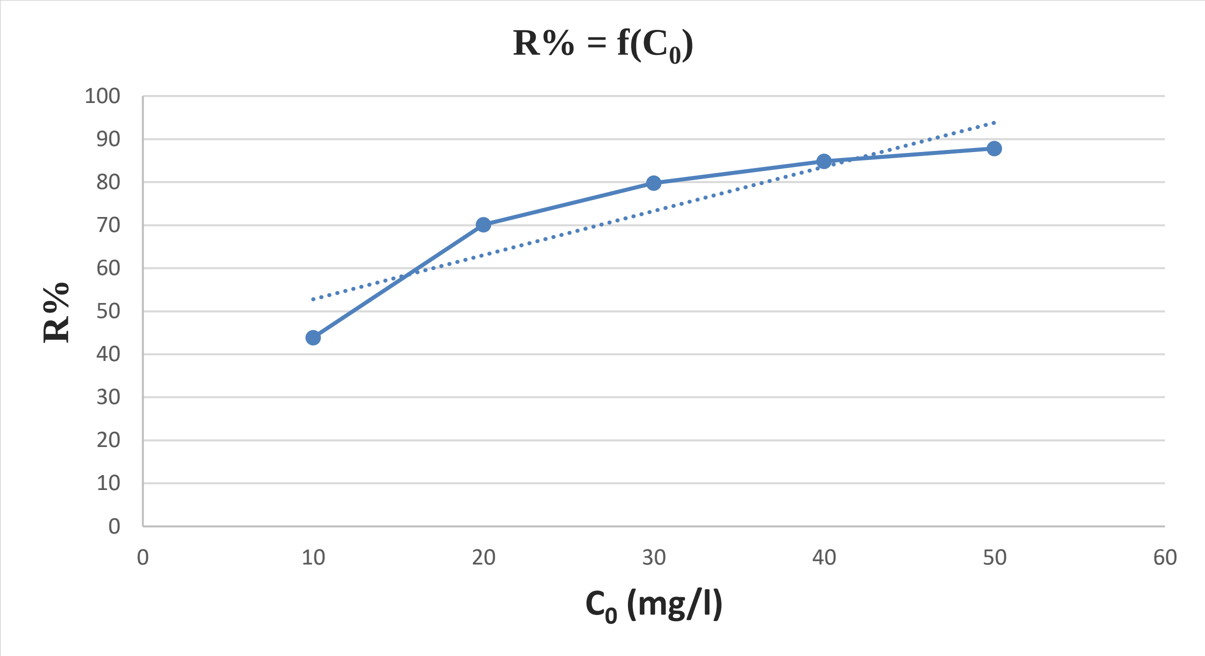

Figure 10. Elimination Percentage as a function of initial methylene blue levels

In fact, methylene blue absorption curves can be divided into two parts: the first part of the kinetics corresponds to a very short phase, where methylene blue absorption is very rapid, occurring in the first few minutes of adsorption, and a second phase of medium rapidity, where the adsorbed quantity evolves more slowly, and the adsorption rate is relatively low. This phenomenon can be explained by the existence of an initial phase of methylene blue adsorption on easily accessible sites, probably located on the external surfaces of solid supports, followed by molecular diffusion of the dyes to less accessible adsorption sites such as the interfoliar spaces of solids, or within organic matter, before reaching an adsorption equilibrium where all sites are occupied. The rapid fixation is explained by the high affinity of the supports for methylene blue retention, and by better diffusion of the substrate through the pores of these adsorbents. These results are similar to those obtained for the adsorption of dyes on different adsorbents such as: kaolinite (McKay *et al.*, 1980), aerobic granules (Ogunsile *et al.*, 2014) and other adsorbents (Kargi *et al.*, 2004),(Nejib *et al.*, 2015).

3.5. Adsorption isotherms

To analyze the adsorption equilibrium in this study, Langmuir and Freundlich models are used, which generally enable researchers to study the adsorption isotherms of adsorbent/adsorbate systems (Dabwan *et al.*, 2015; Zaka *et al.*, 2017). Adsorption isotherms play a very important role in determining maximum adsorption capacities and in identifying the type of adsorption. They result from knowing the contact time and from plotting adsorbed colorant quantity at equilibrium as a function of its concentration (Qe =f(Ce) as illustrated on **Figure 11**.

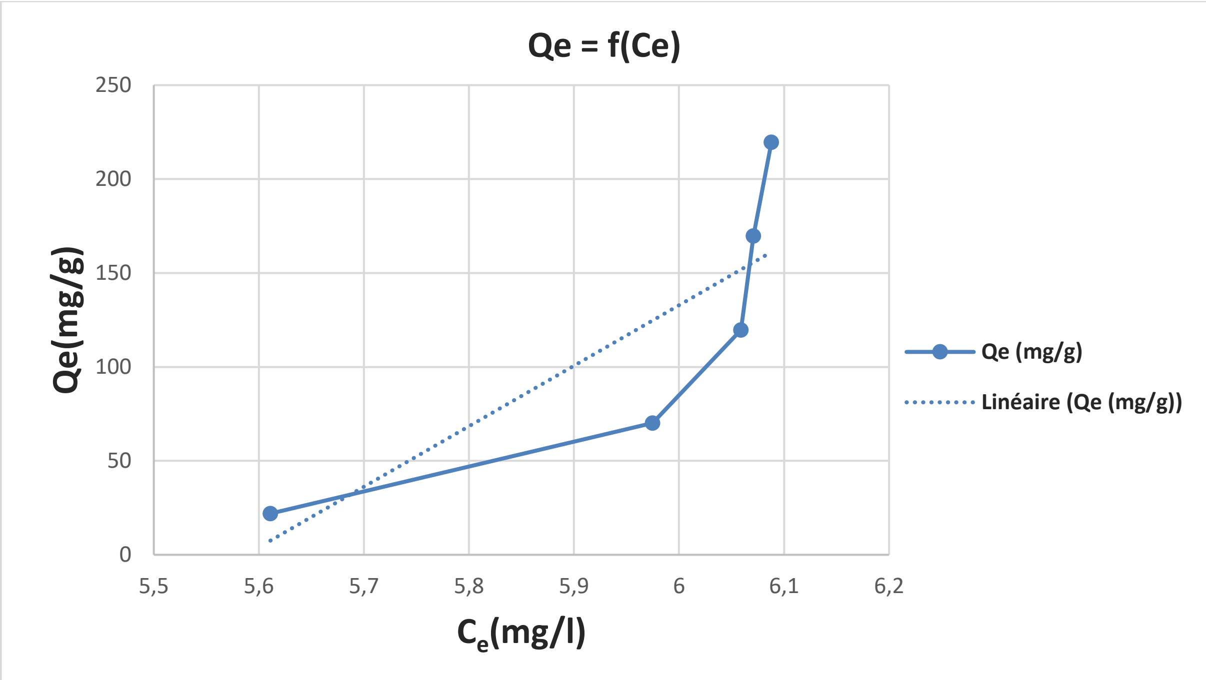

Figure 11. Evolution of equilibrium adsorption capacity of methylene blue as a function of its concentration

As the concentration increases, so does the strength of the concentration gradient, which promotes the circulation of dye molecules in solution across the carbon surface (Kiani *et al.*, 2021). The literature reports comparable results in terms of colorant removal(Kannan and Sundaram 2001; Lukubye and Andama 2017). Plotting linear isotherms according to the Langmuir and Freundlich models using experimental results:

3.5.1. Langmuir model

Langmuir's model is based on the assumption that the adsorbate forms a monolayer over the surface of the adsorbent without any interaction between the adsorbed molecules(Xia *et al.*, 2019; Vallejo *et al.*, 2023),whose results are presented in **Table 3**.

Table 3. Results of the Langmuir isotherm for methylene blue.

C_e (mg/l)	5.611	5.975	6.059	6.071	6.088
C_e/q_e	0.255	0.085	0.050	0.035	0.027

3.5.2. Freundlich model

The Freundlich model is applied to multilayer adsorption. According to this model, interactions between adsorbed molecules are likely(Karnib *et al.*, 2014),(Pandiarajan *et al.*, 2018),whose results are detailed in **Table 4**. It appears in **Figure 12** that the adsorption isotherm is suitably simulated by the linear Langmuir model, since this is the closest and most credible model that best describes our experiment and gives a good description of the BM adsorption process.

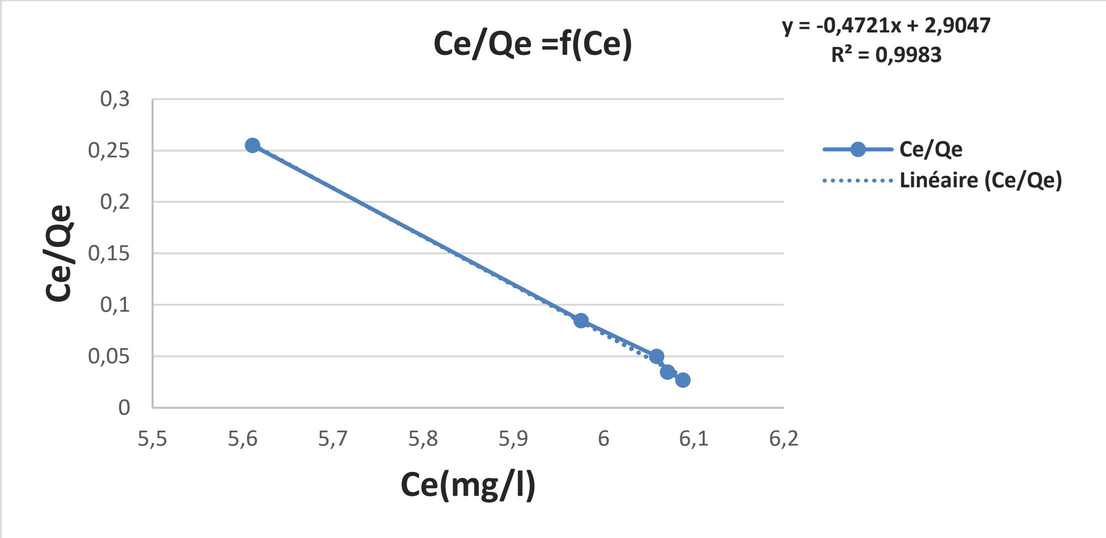

Figure 12. Graphical representation of the Langmuir isotherm for methylene blue.

Table 4. Results of the Freundlich isotherm for methylene blue.

Ln (C_e)	1.724	1.787	1.801	1.803	1.806
Ln (q_e)	3.088	4.250	4.785	5.133	5.391

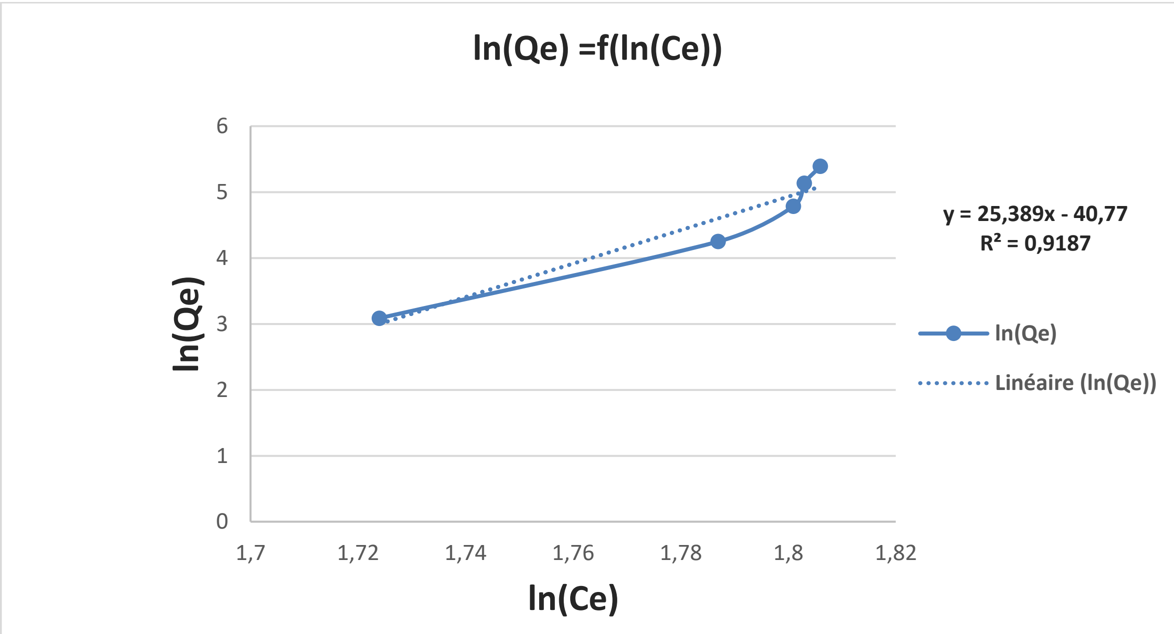

Figure 13. Graphical representation of the Freundlich isotherm for methylene blue.

3.5.3. Modeling constants :

Using the constants calculated in the previous part as a reference, we determine the parameters shown in **Table 5**. The **Figure 14** demonstrates that the adsorption isotherm is correctly simulated by the linear Langmuir model, insofar as it is the closest and most credible model that best describes our experimental results and well represents the methylene blue adsorption process.

Table 5. Experimental results according to Langmuir and Freundlich modeling of methylene blue.

C_e (mg/l)	5.611	5.975	6.059	6.071	6.088
Quantity adsorbed (mg/g)	21.945	70.125	119.705	169.645	219.56
q_e (Langmuir)	20.676	66.304	113.269	160.162	207.808
q_e (Freundlich)	0.0392	0.0121	0.007112	0.005016	0.003874

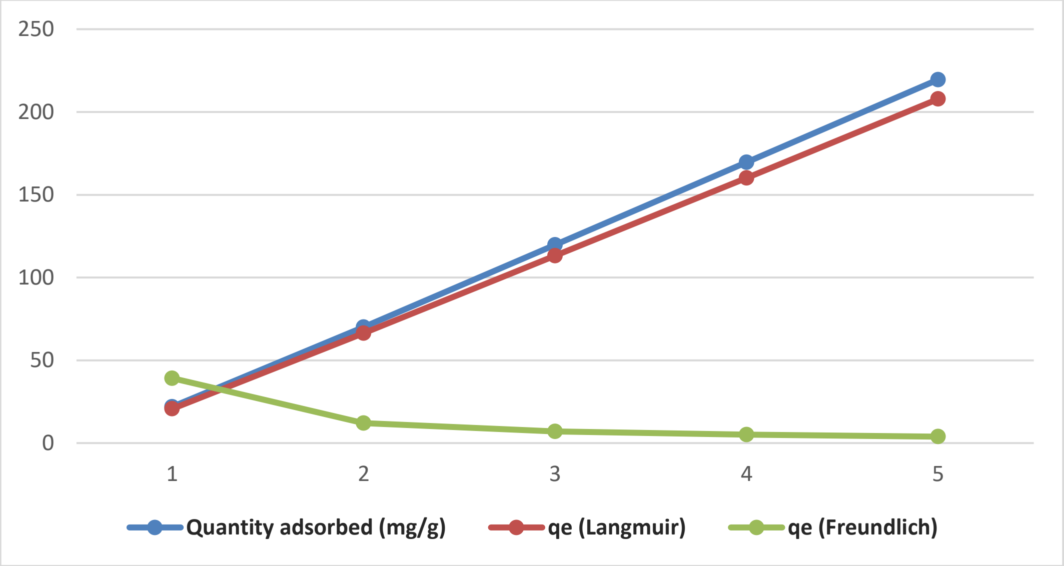

Figure 14. The comparison of the two adsorption isotherms of BM on activated carbon with the curve experimentally derived.

Conclusion

The aim of the present study is to examine the adsorption of a basic colorant (MB) on activated carbon using Langmuir and Freundlich adsorption isotherms.

The results of this study show that the decontamination of colored effluents is highly dependent on adsorption. It provides a test to establish the affinity of the activated carbon for the BM dye in order to optimize its removal.

- The kinetics as a function of BM concentration revealed an increase in the amount adsorbed with increasing solution concentration.
- The percentage of colorant removal increases with adsorbent mass, as increasing adsorbent mass increases the specific surface area and consequently the number of available adsorption sites, leading to an increase in the amount of adsorbed colorant.
- Finally, the linear Langmuir model is the model that best describes the adsorption process for methylene blue, allowing the adsorption isotherm for this dye to be traced more easily.
- The activated carbon produced from argan kernels is expected to be an economical absorbent for the removal of colorants from water and industrial wastewater.

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