

# Kinetic and Thermodynamic Study of the Adsorption of Crystal Violet by Activated Carbon Based on *Azadirachta indica* Seed Cakes

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## Abstract

Activated carbon prepared from *Azadirachta indica* seeds oil cake was effectively used to remove the cationic dye crystal violet (CV) from aqueous solutions. In this study, activated carbons prepared by activation with orthophosphoric acid followed by carbonization at different temperatures were used as adsorbents to remove crystal violet, a basic dye, from aqueous solutions. The results show that the adsorption rate increases slightly with increasing particle size (from 87.6% to 89.9%) for a size range from 160 to 500  $\mu\text{m}$ . Beyond 500  $\mu\text{m}$ , a reduction in the rate was observed down to 69.96%. The results show that the maximum adsorption rate of the purple crystal obtained is 89.87% for an optimal particle size of 500  $\mu\text{m}$ , 90.39% for a mass of 2 g, 90.50% for a time of 50 minutes, 92.51% for an agitation speed of 250 rpm, 99.04% for a volume of 200 mL, and 93.53% for a CV concentration of 8 mg/L. In addition, the pH and temperature have a considerable influence. The model that best describes the adsorption isotherm is that of Freundlich with pseudo-second-order kinetics. Results show the effectiveness of Neem seed meats for the removal of the purple crystal from aqueous solution.

## Keywords

*Azadirachta indica* Seeds Oil Cake, Activated Carbon, Adsorption, Isotherm

## 1. Introduction

Heavy metals and dyes constitute the most persistent and dangerous pollutants for humans and their environment. Larger concentrations of heavy metals or dyes in the effluent stream cause serious health problems in the brain, kidney, and ane-

nia. Copper consumption at high dosages leads to serious toxicological concerns since it could be deposited in the brain, skin, liver, and kidney (Al Moharbi et al., 2020; Costa et al., 2020; Sulaiman & Garba, 2014). Higher concentrations of copper cause vomiting, cramps, convulsions, or even death (Paulino et al., 2006; Petrović et al., 2017; Reddy, Lee, & Sessaiah, 2012). While purple crystal causes moderate eye irritation, painful sensitization to light, and permanent injury to the cornea and conjunctiva.

Because many organic dyes and heavy metals are harmful to humans, their removal from effluents is important for the environment. There are several methods for removing dyestuffs and heavy metals from wastewater, such as chemical and electrochemical oxidation, membrane separation process, photodegradation, coagulation, adsorption, and biological processes. Of them, adsorption seems to be the most efficient method. Adsorption is a widely used method for the treatment of industrial wastewater containing color, heavy metals, and other inorganic and organic impurities (Hamidzadeh, Torabbeigi, & Shahtaheri, 2015; Khan et al., 2015; Li et al., 2018). The advantages of adsorption are its simplicity of operation, low costs, and absence of sludge formation. Conventional adsorbents such as activated carbon, biowaste, and clay have been used to remove dyes and heavy metals from water, but over the last few years we have increasingly observed the use of activated carbons based on agricultural residues (Ukanwa et al., 2019; Ndoye et al., 2023; Tamizh Selvi & Silambarasan). Recent studies have shown the effectiveness of these materials for the removal of certain pollutants such as dyes and metals (Li et al., 2022; Buhani, Luziana, Rilyanti, & Sumadi, 2019).

The most widely used carbonaceous materials for the industrial production of activated carbons are coal, wood, and coconut shell. These types of precursors are expensive and are often imported, making it necessary for developing countries to find a cheap and available feedstock for the preparation of activated carbon for use in industry, drinking water purification, and wastewater treatment. Several suitable agricultural by-products (lignocellulosics), including peach stones, date stones, waste apple pulp in cider production, rice husks, pistachio-nut shells, and grain sorghum, have been investigated in recent years as activated carbon precursors and are still receiving renewed attention. In comparison, olive-waste cakes have received much less consideration as a lignocellulosic material for activated carbon production (Baccar et al., 2009; Kielbasa et al., 2022; Li et al., 2016).

Indeed, neem seed is a very abundant product in Senegal, but its usefulness remains very low. Significant quantities of *neem* seeds are generated each year and constitute a significant source of waste. Such products corresponding to this loss can be of significant economic interest (Mishra & Mohanty, 2018; Adepoju, 2020). It is therefore important to recover such waste. However, neem seeds contain a large amount of oil, 45% of which is extracted by wooden mills, and neem oil has been found to be a potential source of the natural insecticide azadirachtin (Agbo, Nta, & Ajaba, 2019; Bhattacharyya, Chutia, & Sarma, 2007). The cake from seeds after oil extraction is a good source of nutrients (CP: 35% - 38%; EE: 4.5% - 5.5%;

CF: 12% - 15%; Ca: 0.75%; P: 0.45% on DM), and in particular, the one out of its kernel is proteinaceous and is relatively balanced in its amino acid and mineral profile. But the cake is toxic and bitter to taste owing to triterpenoids (*nimbin*, *salannin*, *azadirachtin*), which restricts its safe inclusion in livestock diets (Bhattacharyya, Chutia, & Sarma, 2007; Singh, Sharma, & Singh, 2017).

These seeds, once removed from their oils, have a very large specific surface area. It is in this context that our study takes place. The aim of this study is to valorize *neem* seed oil cakes in the form of activated carbon for the removal of dyes in wastewater. For this, it will also be necessary to explore the influence of the activation temperature on the properties of the alternating current produced, as well as to optimize the operating conditions for the kinetics and thermodynamics of the adsorption of crystal violet.

## 2. Material and Methods

### 2.1. Preparation of the Absorption Material

The adsorbent material used in this study is activated carbon made from *neem* seed cake. The *neem* seed cakes were washed several times with tap water before being rinsed with distilled water; they were then placed in an oven at 105 °C for one hour. After drying, the samples obtained were carbonized in an oven at 400 °C. A mass of the charcoal obtained was brought into contact with orthophosphoric acid in a 200 mL beaker with a mass ratio equal to 1. The mixture was stirred for one hour, then neutralized with sodium carbonate (Na<sub>2</sub>CO<sub>3</sub>). The mixture thus obtained was rinsed with distilled water to remove traces of remaining acid or base and then placed in an oven at 105 °C for one hour. The material obtained constitutes activated carbon based on *neem* seed cake (Figure 1).



**Figure 1.** (a) Neem seed cakes after washing; (b) Activated carbon obtained from neem cake.

### 2.2. Experimental Procedure

All experiments were carried out in 100 mL beakers. For each test, a certain mass

of activated carbon was added to the crystal violet. The effectiveness of the activated carbon is determined by its adsorption capacity, denoted as  $q_t$  and expressed in (mg/g), or by its adsorption rate, denoted as  $R$  and expressed in (%), given respectively by the following relationships.

$$q_t = \frac{(C_0 - C_e) * V}{m} \quad (1)$$

With:

$C_0$ : initial solute concentration (mg/L);

$C_e$ : residual concentration of the solute at equilibrium (mg/L);

$V$ : volume of the solution (L);

$m$ : mass of the adsorbent (g).

$$R = 100 * \frac{(C_0 - C_e)}{C_0} \quad (2)$$

The adsorption experiments of crystal violet onto activated carbon derived from neem seed cakes were conducted using UV-visible spectrophotometry to determine the dye concentration before and after adsorption, at a maximum absorbance wavelength of 585 nm. A comprehensive set of parameters was investigated to optimize the adsorption process and understand the underlying mechanisms. These parameters include:

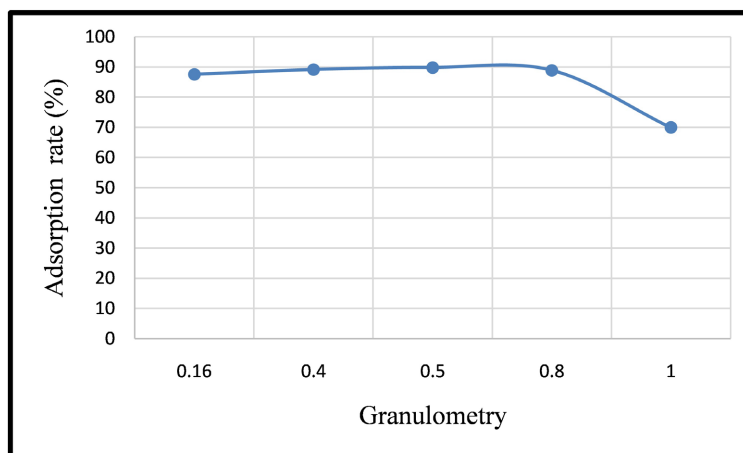
- Effect of particle size of the activated carbon, which influences the available surface area and porosity;
- Effect of adsorbent mass, to evaluate the relationship between the quantity of material and the adsorption efficiency;
- Effect of contact time, to assess the kinetics of adsorption and determine the equilibrium time;
- Effect of stirring speed, which affects external mass transfer and the homogeneity of the system;
- Effect of solution volume, to analyze the dilution effect and adsorption capacity per unit volume;
- Effect of initial dye concentration, to understand the influence of the concentration gradient and site saturation;
- Effect of pH, given its impact on the ionization state of the adsorbate and the surface charge of the adsorbent;
- Effect of temperature to evaluate the thermodynamic behavior of the adsorption process (endothermic or exothermic nature).

### 3. Results and Discussion

#### 3.1. Adsorption of Crystal Violet (CV) by Activated Carbon

##### 3.1.1. Effect of Coal Particle Size

Five types of adsorbent particle sizes were used to study their effects on the adsorption of crystal violet (CV). The results obtained are shown in **Figure 2**.

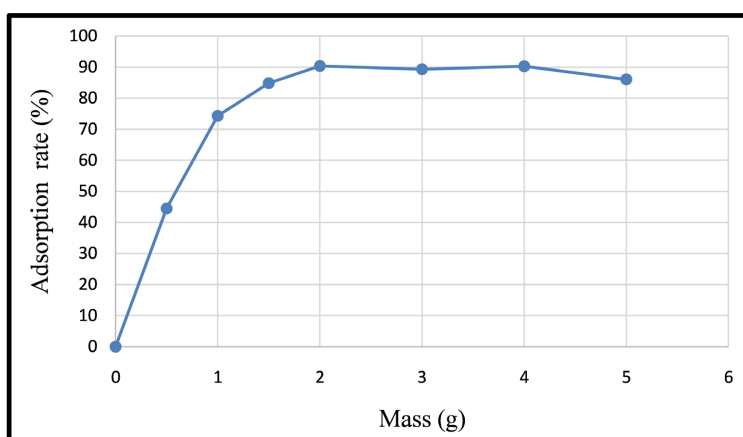


**Figure 2.** Evolution of the adsorption rate as a function of particle size:  $m = 2$  g,  $V = 50$  mL,  $T_s = 60$  min,  $[CV] = 5$  mg/L,  $\Omega = 750$  rpm.

The results obtained show that the adsorption rate increases slightly with increasing particle size (from 87.6% to 89.9%) for a size range from 160 to 500  $\mu\text{m}$ . Beyond 500  $\mu\text{m}$ , a reduction in the rate was observed, down to 69.96%. The best rates are therefore obtained with small particle sizes. This is due to the fact that small particles have much larger specific surface areas than large particles (Sahoo & Prelot, 2020; Motitswe, Badmus, & Khotseng, 2022).

### 3.1.2. Effect of Adsorbent Mass

Figure 3 describes the evolution of the adsorption efficiency of crystal violet and the adsorption capacity of the material as a function of the mass of the adsorbent.



**Figure 3.** Effect of mass on the adsorption of crystal violet:  $[CV] = 5$  mg/L,  $V = 50$  mL,  $T_s = 60$  min,  $\Omega = 750$  rpm.

The adsorption rate of crystal violet increases significantly with the mass of activated carbon. In fact, the rate increases until it reaches a value of 90.40%, from which it stabilizes. This increase from 44.53% to 90.40% for masses ranging from 0.5 to 2 g could be attributable to the increase in the specific surface area of the adsorbent and the availability of a greater number of adsorption sites. Beyond 2 g,

the number of accessible free sites becomes stable (Abbas, Harrache, & Trari, 2020; Amin, 2009; Mehr et al., 2020).

### 3.1.3. Effect of Contact Time

Figure 4 represents the evolution of the adsorption capacity of the adsorption material and that of the removal rate of crystal violet as a function of contact time.

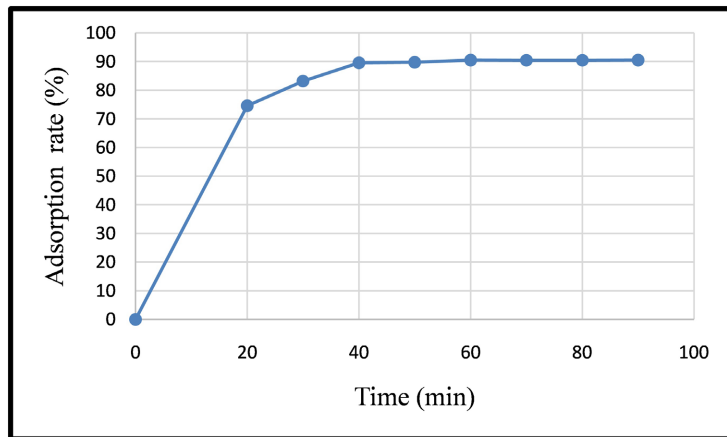


Figure 4. Evolution of the adsorption rate as a function of contact time: [CV] = 5 mg/L,  $V = 50$  mL,  $m = 2$  g,  $\Omega = 750$  rpm, particle size = 500  $\mu\text{m}$ .

The results obtained in this study show that activated carbon based on *neem* seed cake is an effective adsorbent for eliminating CV dye from aqueous solutions because, in a very short time (20 minutes), the rate is around 80%. Indeed, it increases rapidly during the first 40 minutes depending on the contact time. Beyond this value, it remains constant. This can be explained by molecular diffusion (Park et al., 2020; Qiu et al., 2022; Baccar et al., 2010) towards the adsorption sites until an adsorption equilibrium is reached, where all sites become occupied.

### 3.1.4. Effect of Stirring Speed

The results of the variation in the adsorption rate of crystal violet by CA as a function of the stirring speed are shown in the following Figure 5.

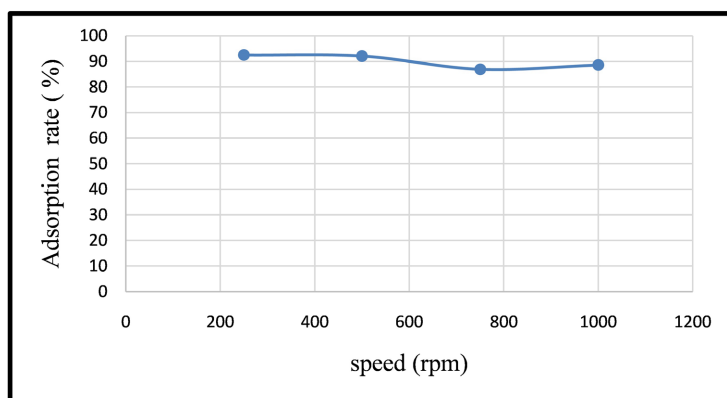
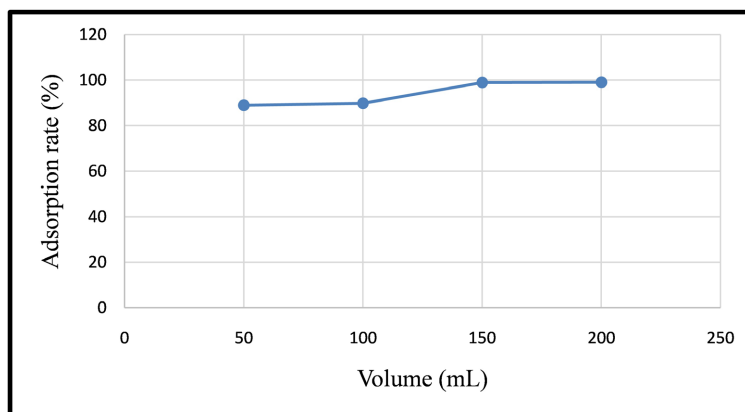


Figure 5. Effect of stirring speed on the adsorption of crystal violet: [CV] = 5 mg/L,  $V = 50$  mL,  $m = 2$  g,  $T_s = 60$  min, particle size = 500  $\mu\text{m}$ .

Increasing the stirring speed leads to a slight decrease in the adsorption rate. The rate values found are approximately equal to 90%. This means that it has little influence on the adsorption rate. This small reduction observed could be due to the fact that for very high stirring speeds (Sulyman et al., 2021), the crystal violet molecules will have difficulty attaching to the activated carbon, but also due to the agglomeration of the support particles (Al-Shehri et al., 2021; Buhani et al., 2024; Algarni et al., 2022).

### 3.1.5. Effect of Solution Volume

The results of the volume variation on the retention of crystal violet by the CA are represented in the following **Figure 6**:



**Figure 6.** Evolution of the adsorption rate as a function of the volume of the solution. [CV] = 5 mg/L,  $m = 2$  g,  $T_s = 60$  min,  $\Omega = 750$  rpm, particle size = 500  $\mu\text{m}$ .

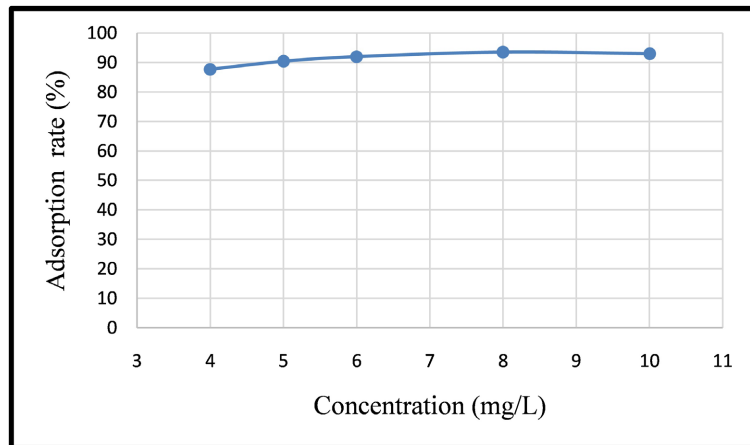
These results reveal that the adsorption rate increases from 88.93% to 99.04% for volumes ranging from 50 to 200 mL. This increase is much greater with the 100 and 150 mL volumes. This can be explained by the fact that the increase in volume causes a dispersion of the activated carbon particles in solution (Aniagor et al., 2023; Wu et al., 2021), facilitating their encounter with the crystal violet molecules.

### 3.1.6. Effect of Initial Concentration

The results of the variation in the adsorption rate of crystal violet by CA as a function of the initial concentration are shown in **Figure 7**.

It shows that an increase in the initial concentration of CV leads to an increase in the quantity adsorbed by CA. Indeed, if the initial concentration of the crystal violet becomes greater, there will consequently be more molecules that will diffuse towards the sites of the particles of the support, so retention becomes greater. Saturation is reached for a concentration of 8 g/L, corresponding to an adsorption rate of 93.53%. This can therefore be explained by the occupation of all the adsorption sites of this CA (Foroutan et al., 2021; Cruz et al., 2020). Moreover, the adsorption mechanism of crystal violet (CV) onto activated carbon involves electrostatic interactions,  $\pi$ - $\pi$  stacking, and van der Waals forces. At low concentrations, the active sites are readily accessible. At higher concentrations, competition between

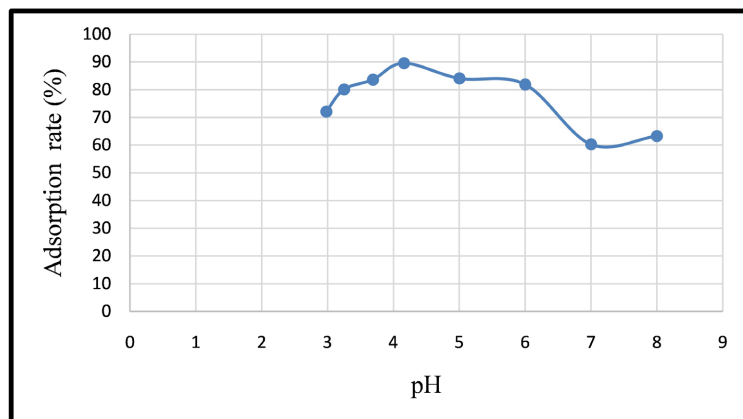
molecules increases until saturation is reached. Adsorption then reaches a plateau corresponding to the maximum capacity of the material (Kumar et al., 2010).



**Figure 7.** Effect of initial concentration on the adsorption of crystal violet:  $V = 50$  mL,  $m = 2$  g, contact time = 60 min, speed = 750 rpm, particle size = 500  $\mu\text{m}$ .

### 3.1.7. Influence of the pH of the Solution on Adsorption

**Figure 8** shows the test results on the effect of pH versus CV removal in solution on the absorption material.



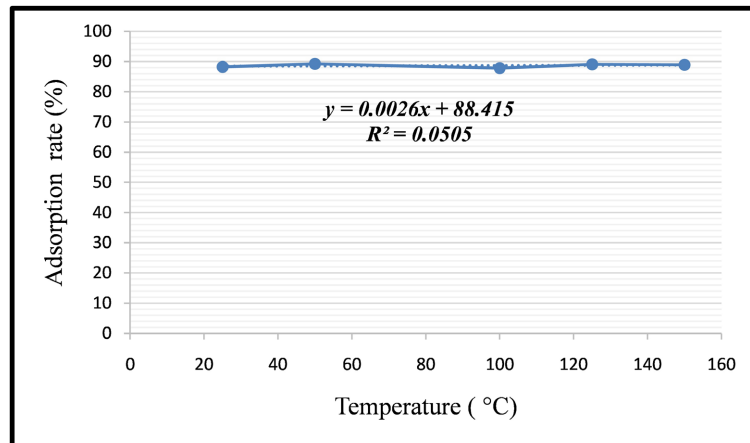
**Figure 8.** Evolution of the adsorption rate as a function of the pH of the solution.  $[CV] = 5$  mg/L,  $V = 50$  mL,  $m = 2$  g, contact time = 60 min, speed = 750 rpm, particle size = 500  $\mu\text{m}$ .

Acidic conditions ( $\text{pH} < 4.16$ ) resulted in adsorption rates ranging from 72.08% to 90%. This behavior could be attributed to structural modifications of the crystal violet molecules in strongly acidic media, as suggested by the visible change in color of the solution during the process. At higher pH values (between 4.16 and 8), similar trends were observed, although the adsorption efficiency varied depending on the degree of ionization of the dye and the surface charge of the adsorbent (Qiu et al., 2022; Elsellami et al., 2010; Kuo et al., 2017). These findings confirm that pH has a direct and significant impact on the adsorption capacity. Consequently, optimal removal of crystal violet should preferably occur under

near-neutral pH conditions, where the dye remains stable and the surface charge of the activated carbon favors adsorption (Chen et al., 2022; Tan et al., 2023).

### 3.1.8. Influence of Temperature

The effect of temperature on the adsorption properties of the dye (CV) on activated carbon is shown in Figure 9.



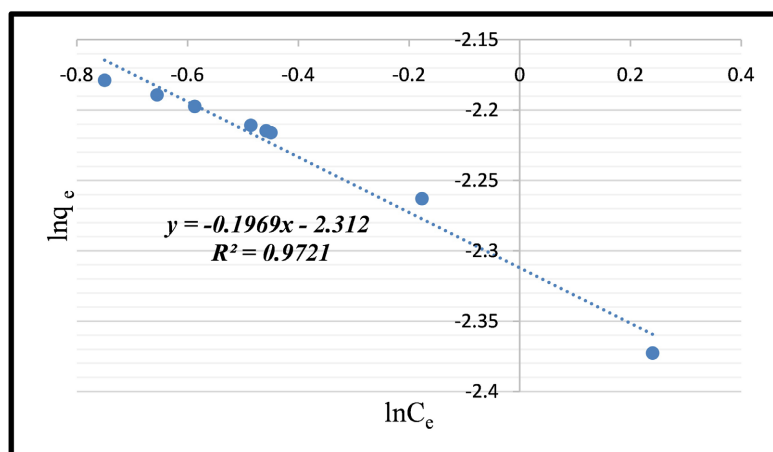
**Figure 9.** Evolution of the adsorption rate as a function of temperature: [CV] = 5 mg/L,  $V = 50$  mL,  $m = 2$  g, contact time = 60 min, speed = 750 rpm, particle size = 500  $\mu\text{m}$ .

The adsorption rate exhibits only slight variations with temperature, suggesting that temperature has a limited influence on the retention of crystal violet under the conditions tested. This weak dependence may indicate that the adsorption process is not strongly thermally activated and that physical interactions dominate the adsorption mechanism (Ziegler & Handy, 1981; Cai et al., 2019).

## 3.2. Simple Adsorption Isotherms

### 3.2.1. Freundlich Modeling

The adsorption results (Figure 10) are modeled here based on Freundlich assumptions. The model equation is (Khayyun & Mseer, 2019).



**Figure 10.** Freundlich model for CV adsorption by activated carbon.

$$\ln q_e = \ln K_f + \left(\frac{1}{n}\right) \ln(C_e) \quad (3)$$

The modeling is shown in the figure below:

### 3.2.2. Langmuir Modeling

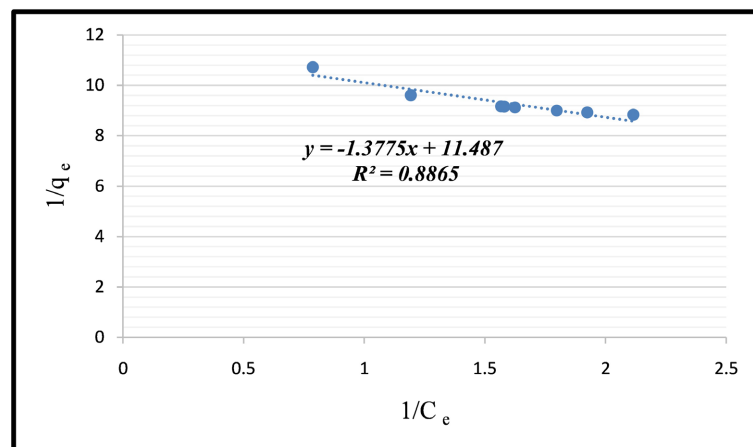
We also used the Langmuir isotherm to analyze the results. The Langmuir equation is:

$$\frac{1}{q_e} = \frac{1}{Q_0 K_L} \left(\frac{1}{C_e}\right) + \frac{1}{Q_0} \quad (4)$$

The Langmuir linear is:

$$C_e/q_e = f(C_e) \quad (5)$$

The modeling of the experimental results by the isotherm, exploited in its linear form, is represented in **Figure 11**.



**Figure 11.** Langmuir model for CV adsorption by activated carbon.

The values of the Langmuir and Freundlich parameters, as well as the correlation coefficients, are mentioned in **Table 1**.

**Table 1.** Parameter values of the Langmuir and Freundlich models.

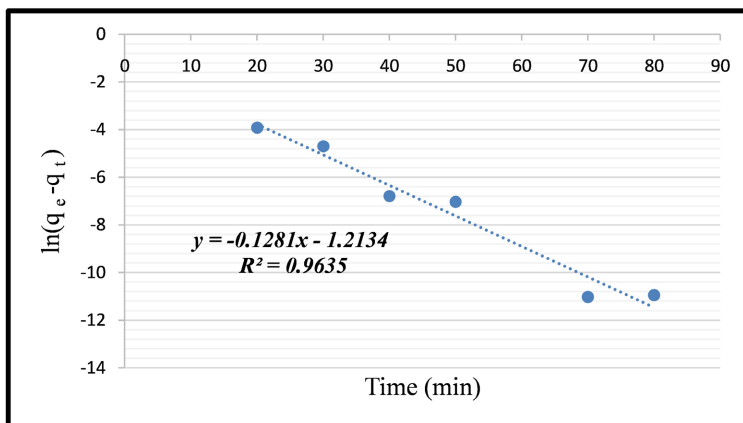
Langmuir			Freundlich		
$Q_0$	$K_L$	$R^2$	$K_f$	$K_L$	$R^2$
0.087	8.344	0.8865	0.099	5.0787	0.9721

According to the graphic representation of each isotherm and the correlation factors, as well as the constants of each model, we noticed that the Freundlich isotherm seems to be best suited to the modeling of the adsorption isotherm of crystal violet by the adsorbent material (linear regression coefficient close to 1).

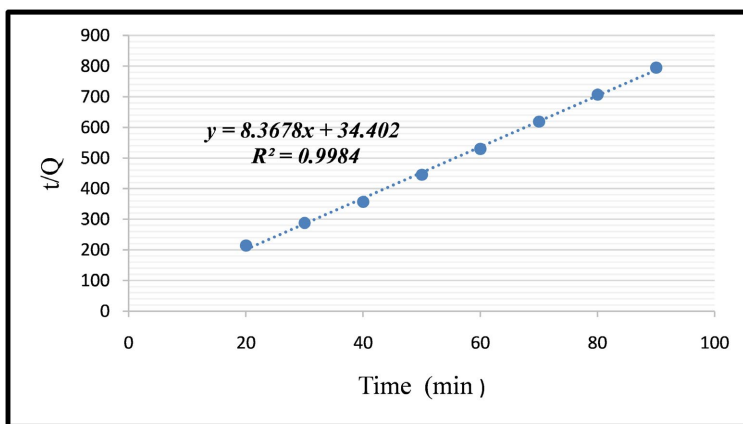
### 3.3. Adsorption Kinetics

The kinetic data obtained during the adsorption process of crystal violet on the

adsorbent material were analyzed by two kinetic models: pseudo-first order kinetics and pseudo-second order kinetics. The plotting linear equations are given respectively by **Figure 12** and **Figure 13**.



**Figure 12.** Plot of the linear form of the pseudo-first-order kinetic model.



**Figure 13.** Plot of the linear form of the pseudo-second-order kinetic model.

**Table 2** groups together the values of the rate constants  $k_1$  and  $k_2$ , the adsorption capacities, and the determination coefficients of the two kinetic models.

**Table 2.** Kinetic parameters of the adsorption of crystal violet by the adsorbent.

Pseudo first order			Pseudo second order			
$R^2$	$k_1$ ( $\text{min}^{-1}$ )	$q_e$ (mg/g)	$R^2$	$k_2$ (g/mg.min)	$q_{e\text{ cal}}$ (mg/g)	$q_{e\text{ exp}}$ (mg/g)
0.9635	0.1281	0.2972	0.9984	2.0355	0.1195	0.1131

These results show that the adsorption of crystal violet by activated carbon based on neem seed cake follows a pseudo-second-order kinetic law because the coefficient of determination  $R^2$  given by the second-order model is much higher than that of the pseudo-first-order. Furthermore, the adsorption capacity calculated from the pseudo-second-order kinetic model (0.1179 mg/g) is in good agreement with the adsorption capacity determined experimentally (0.1131 mg/g).

### 3.4. Thermodynamic Study

The thermodynamic study informs us about the feasibility and spontaneous nature of the adsorption process. Parameters such as free energy ( $\Delta G^\circ$ ), enthalpy change ( $\Delta H^\circ$ ), and entropy change ( $\Delta S^\circ$ ) can be estimated from equilibrium constants at different temperatures. They were determined from the experimental data, in order to evaluate and understand whether the adsorption processes studied were spontaneous or not; exothermic or endothermic. We have the following formulas:

$$\Delta G^\circ = \Delta H^\circ - T\Delta S^\circ \quad (6)$$

$$\Delta G^\circ = -RT \ln K_C \quad (7)$$

$$K_C = \frac{q_e}{C_e} \quad (8)$$

$$\ln K_C = -\frac{\Delta H}{RT} + \frac{\Delta S^\circ}{R} \quad (9)$$

$K_c$ : The distribution constant of the solute in the adsorbent and in the solution;

$q_e$ : The quantity adsorbed at equilibrium (mg/g);

$C_e$ : Solute concentration;

$R$ : the ideal gas constant ( $8.314 \text{ J}\cdot\text{mol}^{-1}\cdot\text{K}^{-1}$ );

$T$ : absolute temperature (K).

The plot of  $\ln K_c$  versus  $1/T$  (Figure 14) is a line with slope ( $\Delta H^\circ/R$ ) and ordinate at the origin ( $\Delta S^\circ/R$ ).

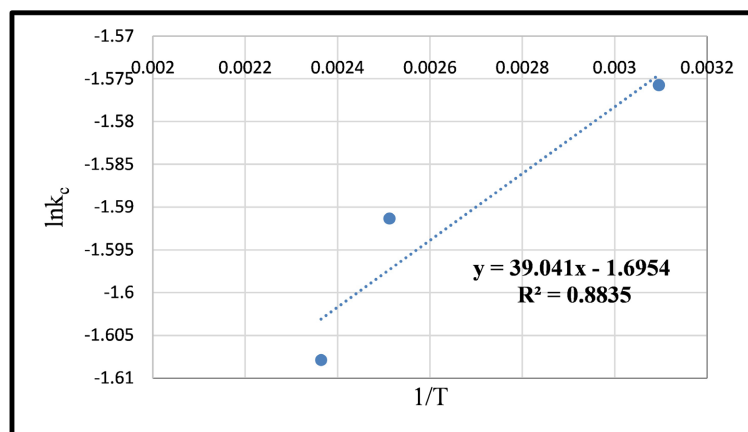


Figure 14. Representation of  $\ln(K_c)$  as a function of temperature ( $1/T$ ).

Table 3. Thermodynamic parameters of the adsorption of crystal violet on CA based on neem seed cakes.

Settings	$\Delta G^\circ$ ( $\text{J}\cdot\text{mol}^{-1}$ )	$\Delta H^\circ$ ( $\text{J}\cdot\text{mol}^{-1}$ )	$\Delta S^\circ$ ( $\text{J}\cdot\text{mol}^{-1}\cdot\text{K}^{-1}$ )
25	3875.523		
50	4228.098		
125	5285.223	-324.587	-14.095
150	5637.598		

The thermodynamic parameters, namely  $\Delta H^\circ$ ,  $\Delta S^\circ$ , and  $\Delta G^\circ$ , were determined from the experimental results obtained at different temperatures (Table 3).

The negative value of  $\Delta H^\circ$  shows that the adsorption of crystal violet on the adsorbent is an endothermic process. It is a physical type of adsorption because  $\Delta H^\circ < 40$  kJ/mol. The negative value of the entropy change indicates that crystal violet is more organized at the solid–liquid interface than in the liquid phase. The positive values of  $\Delta G^\circ$  (free enthalpy) indicate that the adsorption of crystal violet on the adsorbent is a non-spontaneous process. Increasing the temperature does not favor the adsorption of this dye.

### 3.5. Application to Copper Removal and COD Reduction

Figure 15 presents a comparative analysis of copper adsorption using two different masses of activated carbon (2 g and 4 g), each calcined at two distinct temperatures (400 °C and 700 °C). All experiments were conducted under identical conditions.

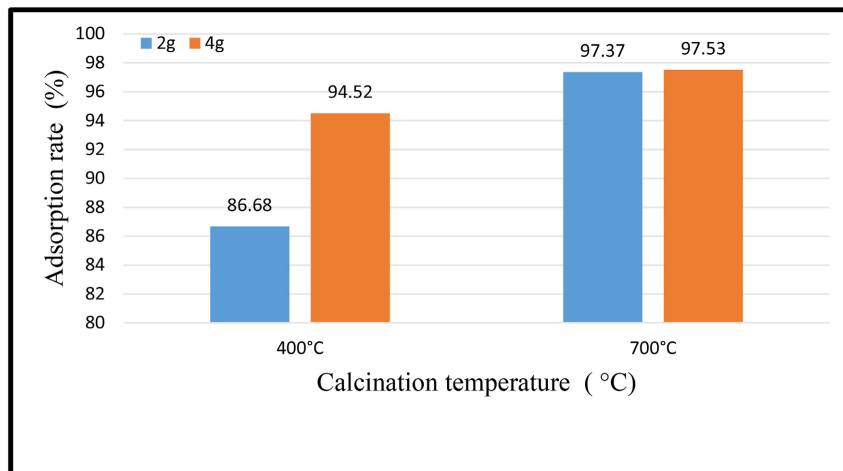


Figure 15. Effect of calcination temperature on adsorption Rate (%).

Activated carbon at 700 °C has better adsorption rates (97.37% and 97.53%) than that calcined at 400 °C (86.69% and 94.52%) for the two masses of carbon (2 and 4 g, respectively). This could be explained by the fact that for the carbon calcined at 400 °C, there remained a quantity of degradable organic matter. As the calcination temperature increases, this organic matter degrades more and more, leading to the existence of a significant number of active sites on the carbon. We can therefore conclude that the calcination temperature of the activated carbon influences the adsorption of copper.

Domestic wastewater is treated with activated carbon based on neem seed cake following pretreatment and decantation over a few days. The COD decreased from 750 to 296 mg/L, representing a reduction rate of 60.5%.

## 4. Conclusion

Water pollution by dyes and heavy metals poses a serious problem for the envi-

ronment and human health. Their removal using natural adsorbents constitutes an effective means in the case of water contaminated by these pollutants. This work was part of the valorization of a local product in Senegal in the form of an adsorbent, namely neem seed. Its objective was to demonstrate the adsorbent potential of activated carbon based on neem seed cake.

The adsorbent used in this study was found to be an effective adsorbent for the adsorption of crystal violet and copper from aqueous solutions. The adsorption rate of crystal violet increases as a function of the particle size of the CA for particle sizes ranging from 160 to 500  $\mu\text{m}$  and decreases from 500  $\mu\text{m}$ , while for copper, it decreases as a function of the particle size. The adsorption rates of copper and crystal violet increase with the mass of the activated carbon and reach saturation at 2 g for crystal violet. They increase with the volume of the solution. Increasing the concentration of the dye (CV) causes an increase in the adsorption rate until saturation is reached, after which it decreases. Increasing the contact time leads to an increase in the CV adsorption rate until equilibrium is reached. Increasing the stirring speed leads to a decrease in the adsorption rate. For pH, the best performance is obtained at the normal pH of the solution. Temperature has little influence on the adsorption rate. According to the two models studied, the Freundlich isotherm seems to be best suited to modeling the adsorption isotherm of crystal violet by the adsorbent. The adsorption process is not spontaneous. It is an exothermic and physical phenomenon. Adsorption follows a pseudo-second order kinetic law.

## Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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