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Effective Elimination of Methylene Blue by Biomass Activated Carbon of Pomegranate Peel: Nonlinear Adsorption Kinetics and Isotherms

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إزالة فعّالة للميثيلين الأزرق بواسطة الكربون المنشط من قشور الرمان: حركية الامتزاز غير الخطية والمتساويات الحرارية

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ABSTRACT

Background: In fact, pollution from industrial wastewater treatment plants has a negative impact on the aquatic environment. Accordingly, this study exhibits an easy separation of methylene blue (MB) dye from its aqueous solution using a biomass of activated from pomegranate peel.

Materials and Methods: Characterization studies were conducted to study the properties of the activated carbon using XRD, SEM and FTIR techniques. The experimental experiments were conducted to investigate the impact of several variables, the most important of which are: contact time (0-180 min), initial concentration (50-150 mg/L), adsorbent dose (0.02-0.1 g), pH (3-11), volume solution (25-100 mL), and temperature (20- 50 °C).

Results: Adsorption isotherms represented by Langmuir and Freundlich models have different correlation factors, 0.9979 and 0.9114 respectively. Langmuir model analysis has proven to be the appropriate model to explain the process of adsorption of MB dye to the surface of the adsorbent, which naturally tends to form a single layer of the adsorbate dye on the surface of the activated carbon adsorbent. The maximum adsorption capacity was 116 mg/g. As for the study of kinetics, the pseudo-second order (R2 = 0.9995) reaction was the closest to representing the kinetics of dye adsorption on the surface. Thermodynamic parameters were evaluated, indicating that the adsorption is spontaneous and endothermic. In which the Δ°G, Δ°H and Δ°S were as follow: (-9.196 to -18.058 kJ/mol), 77.363 kJ/mol, and 0.2954 kJ/mol K.

Conclusion: In conclusion, pomegranate peel activated carbon has been proven to be an effective and inexpensive adsorbent used to remove methylene blue from wastewater.

Key words: Pomegranate peel; Chemical activation; Adsorption; Methylene blue, Langmuir and Freundlich model.

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INTRODUCTION

Water pollution is caused by home waste and, more crucially, industrial effluent. The fast development of industrial and agricultural operations has resulted in the emission of massive quantities of pollutants to the environment, such as pharmaceuticals, pesticides and dyes, which is regarded as one of the greatest challenges in the globe [1]. These pollutants show the noticeable danger to the environment because they are not easily removed, they are "persistent" and also sometimes they are not affected by living organisms, so biological breakdown is very low [2-4]. Dyes are one of the forms of pollutants that must be eliminated from wastewater before releasing them into the environment due to their toxicity and harmful effect on photosynthetic activity [5]. Paying attention to studying the removal of pollutants, including dyes "methylene blue dye", pharmaceutical compounds, and other pollutants, is a shared responsibility to maintain a safe and clean aquatic environment. Several strategies have been investigated for dye removal, including chemical precipitation, photocatalysis, biological treatment, and advanced oxidation procedures. Biological methods for treating these dyes do not give satisfactory results due to the nature of the dyes' composition, which is difficult to break down biologically [6,7]. These procedures often fall short of completely removing methylene from the water. In order to find an alternative to inefficient biological and other treatment methods for removing toxins, efforts were intensified and several studies were issued that deal with more positive, more sustainable, less expensive and more effective methods for removing methylene blue dye and other manufactured dyes [8]. Adsorption is the most extensively used method for removing colors like MB from water. The main criterion for adsorption methods is their use of high-purity materials with low metal content and high carbon content, in addition to the high stability, ease of activation process, and continuous and permanent availability. In addition, the production process is low and no by-products occur during the treatment, and it is possible to store it for long periods without any cracking process occurring to it [9]. Several adsorbents used in the adsorption process, including activated carbon [10], zeolites [11], clay minerals [12], and carbon nanotubes [13], have shown us good and promising results in removing the methylene blue dye. The entire adsorption process is basically based on the attraction and binding of dyes on the surface of the adsorbent, which leads to their removal from their aqueous solutions. Activated carbon is a good example of such adsorbents, as its source is waste plants in all their parts. It is considered one of the distinguished materials due to its high ability to remove pollutants as well as in water treatment in general. Its large surface area and the presence of many pores on the surface enable it to remove pollutants and reduce pollution. However, several carbon sources have been used for adsorption of dyes from water. For example, tree leaves [14], fruit peels [15], pomegranate peel [16], plant roots [17], and inedible fruits "waste fig fruits" [18] have all been used for the same purpose. The reason is that these sources contain a high carbon content that allows, after chemical and physical processes, the formation of a suitable high surface for the purpose of adsorption of these dyes or other compounds. The main objective of this study is to prepare an adsorbent which is activated carbon prepared from pomegranate peels after activating using a concentrated potassium hydroxide solution for the removal of methylene blue dye from its aqueous solution. The adsorbent was characterized using FTIR, SEM, and XRD techniques. The effects of contact time, MB concentration, pH, adsorbent amount, and temperature were investigated, as well as PPAC's methylene blue (MB) adsorption capability. To assess the adsorption technique, research was conducted to investigate the isotherms, kinetics, and thermodynamics of methylene blue (MB) adsorption.

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MATERIALS AND METHODS

- **Materials**

In this work, pomegranate peels (PP) were gathered from the local markets in Al-Hilla, Babylon, Iraq. Merck provided KOH which acts as the activating agent. The adsorbate model utilized for the adsorption is Methylene Blue (molecular formula C16H18ClN3S, a molecular mass of 319.85 g/mol), which was bought from Dyestuffs and Chemicals Company, (China). Figure SI1 presents the molecular structure for methylene blue dye. All compounds utilized in this research were analytical grade and did not require additional purification. Methylene blue solution has been prepared as a stock solution (1000 mg/L), then serial dilutions have been prepared in distilled water.

- **Preparation of pomegranate peel**

The dry pomegranate peels (PP) have been gathered from a local market in Babylon city, Iraq. They are then gently cleansed with distilled water to eliminate dust particles and other contaminants. The gathered raw materials are next dried at room temperature until thoroughly dry, the dried bio waste was then processed in an electric mixer and sieved through a 75-mesh sieve to obtain uniform-sized particles.

- **Manufacture of PPAC**

Taking 3 g of produced pomegranate peel powder and placing it in a beaker with 7.5 mL of distilled water. Then, add 2.5 mL of concentrated sodium hydroxide (10M) while stirring continuously for 10 min until the powder is fully submerged. Leave it for one night and thoroughly wash it with water to drop the pH to 5.5-6.6. Then, we dried the sample under natural conditions for about 1-2 nights. The material is then carbonized in a burning oven at 350°C for two hours. Figure SI2 shows the full methodology scheme for synthesis of PPAC.

- **Characterization of PPAC**

The surface morphology for the active carbon content of pomegranate peel before and after adsorption was investigated using scanning electron microscope (SEM). The crystallinity test has been investigated using X-ray powder diffraction (XRD) analysis (Bruker D8 with Cu k radiation). FTIR spectroscopy was utilized to validate the existence of surface functional groups and chemical interaction in activated carbon treated with pomegranate peel, using a device (Shimadzu 84005-IRS) with a range of 400–4000 cm-1.

Nitrogen adsorption-desorption isotherms at 77 K were applied on the PPAC using a Japanese analysis device (company name). This device is used to analyze the surface texture, noting that before starting the analysis, the adsorbent was kept dried at 250°C for two nights.

- **Adsorption studies**

Practical experiments on dye adsorption on the surface of activated pomegranate peels were carried out under the following conditions: the volume of the solution was 50 mL and at different concentrations ranging from 50 to 150 mg/L. 0.08 g of activated carbon was placed in conical flasks and left for two hours in the shaker at room temperature. After adsorption, samples were separated in a centrifuge at 3000 rpm, the filters were examined using a UV-visible spectrophotometer, and calibration curves were obtained. Initial and residual dye concentrations in water were measured at the high adsorption wavelength (665 nm). The dye elimination

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effectiveness and capacity for adsorption were determined using equations 1 and 2 as shown below:

$$R\% = \frac{C_0 - C_t}{C_0} \times 100 \quad [1]$$

$$Q_t = \frac{(C_0 - C_t)}{M} \times V \quad [2]$$

Where C0 (mg/L) is the concentration of the starting dye in the solution, Ct (mg/L) is the concentration of MB at any given time (t). R% is the proportion of MB dye that was eliminated following adsorption; Qt is the adsorption ability (mg/g) at different times; V is the volume of solution and M is the mass of the adsorbent (g). Adsorption process experiments were conducted with different values of parameters. Several variables were chosen: contact time (0-180 min), initial concentration of dye (50-150 mg/L), pH (3-11), the amount of adsorbent (0.02-0.1 g), volume solution (25, 50 and 100 mL) and the temperature (20-50 °C). These experiments were conducted to obtain ideal conditions for adsorption strength by changing one parameter while keeping the other specified parameters constant.

RESULTS AND DISCUSSION

X-ray characterization of adsorbents

The X-ray device was used in a limited way, considering that the activated carbon is alone without any additives or fabrication of other combinations of other elements. Therefore, the goal of using the X-ray was to determine the degree of crystallinity or to prove the non-crystallinity of the activated carbon as shown in Figure 1. XRD spectrum of PPAC samples has a mostly amorphous structure, as evidenced by a wide diffraction background and the absence of a sharp peak. The results of analysis exhibited that the PPAC sample has non-crystalline form [19].

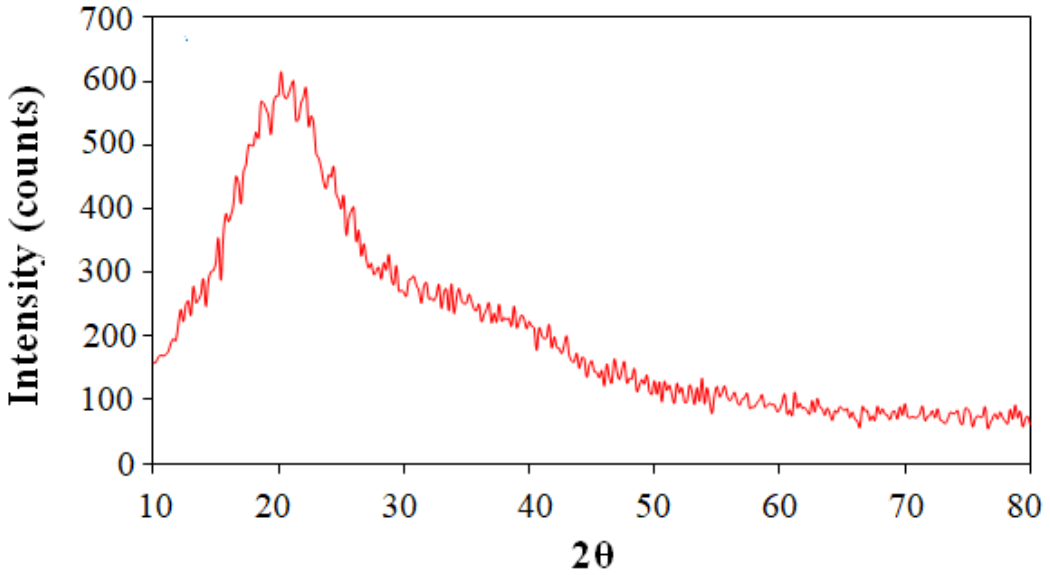

Figure 1. X-RAY pattern for pomegranate peel activated carbon

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SEM characterization of adsorbents

SEM study revealed that the surface morphology for pomegranate peel before and after adsorption is different clearly. PPAC before adsorption appear to have a surface characterized by the presence of voids of different sizes due to thermal activation, which led to a significant change in the morphological characteristics of pomegranate peel carbon. The outer surfaces of activated carbon after adsorption are characterized by a smooth surface and fully filled as shown in Figure 2. The existence of these holes may allow for surface diffusion and immobilization of a high number of MB molecules into the active sites of activated carbon.

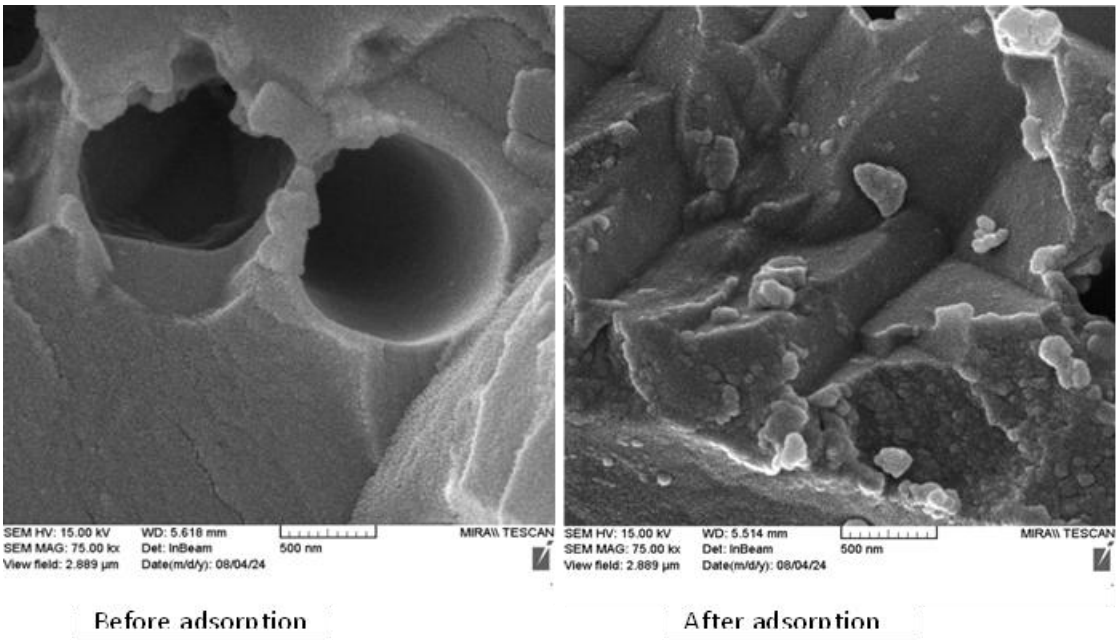

Figure 2. SEM morphology for pomegranate peel activated carbon before and after adsorption experiments at 15.0 kV and 500 nm

FTIR characterization of adsorbents

The surface functional aggregates of the prepared samples in the extent of 400-4000 cm-1 were analyzed by infrared spectrophotometry, such as hydroxyl, carbonyl or carboxyl groups, which can influence the adsorption behavior of PPAC before and after adsorption. In the sample (PPAC), the results of the FTIR analysis showed that the frequencies arising at 3426 cm-1 are due to hydroxide groups, while the frequencies at 1579 cm-1 are due to the carbon-oxygen polar carbonyl group (C=O), and the lowest frequency, 1433 cm-1, is due to the covalent bond between carbon and oxygen (C-O) [20]. The wide peak in the form of an inverted valley, which is located at a frequency of 3426 cm−1, is due to the vibrations of the OH groups in carboxylic organic acids, as well as alcohols of all kinds and phenols. The frequencies of stretching carbonyl (C=O) and carbon-oxygen groups (C-O) at 1579 cm-1 and 1433 cm-1, respectively, are

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the most bands that appear on activated carbon surfaces. These frequencies are of the stretching type and belong to ethers and carboxylic acids as presented in Figure 3. A pale peak at 1028 cm-1 was referred to the C–O bending vibrations [21].

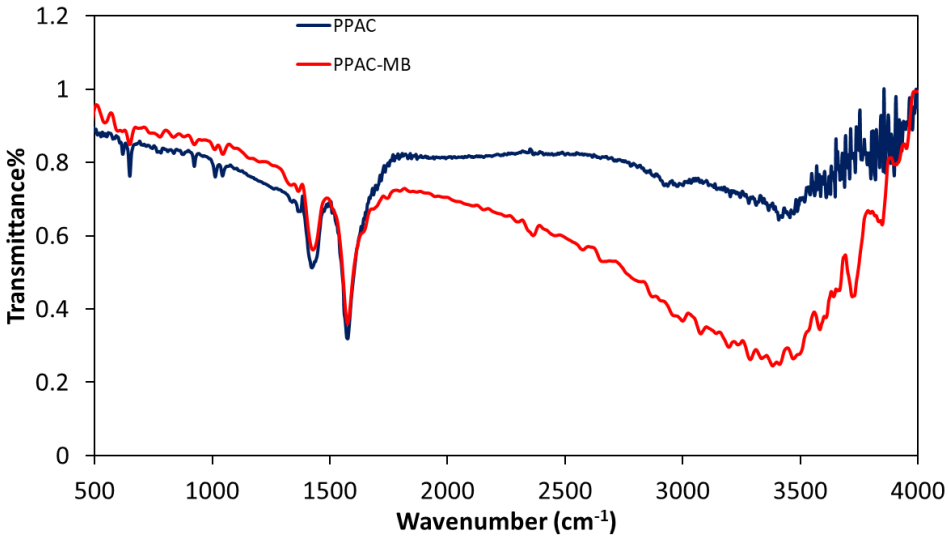

Figure 3. FTIR spectrum analysis for pomegranate peel activated carbon before and after adsorption.

BET characterization of adsorbents

Using the principle of nitrogen gas adsorption-desorption, the surface area of the activated carbon prepared from pomegranate peels was measured and shown in Figure 4. The temperature of the device for measuring the BET surface area was 77 K and the amount of activated carbon used to find the BET surface area was less than 0.02 g. It is simply possible that the ability to attract or bind to pollutants through what is called the adsorption process on the surface increases significantly with the increase in surface areas. This leads us to the possibility of the existence of many effective sites. The meaning is the existence of large and many cavities that can trap these pollutants. Usually, the surface area and the type of material have a major effect on the rate of adsorption and adsorption capacity. Therefore, it can be said that materials with a large surface area have more adsorption sites, which leads to an increase in the adsorption of a larger number of materials. It is well known that porous materials have a large surface area, which leads to an increase in the adsorption process in terms of adsorption capacity and adsorption removal. It is necessary to increase the surface area of the material through the "splitting" process or as called powdering, which is increasing the grinding process to obtain a very fine powder that works greatly to increase the adsorption efficiency. In this recent study, it was found that pomegranate peels, after being converted to activated carbon, may have a BET surface area estimated at 46 g/m2, which is considered a good surface area compared to other waste fig leaves in our previous study, which had a surface area of 18 g/m2.

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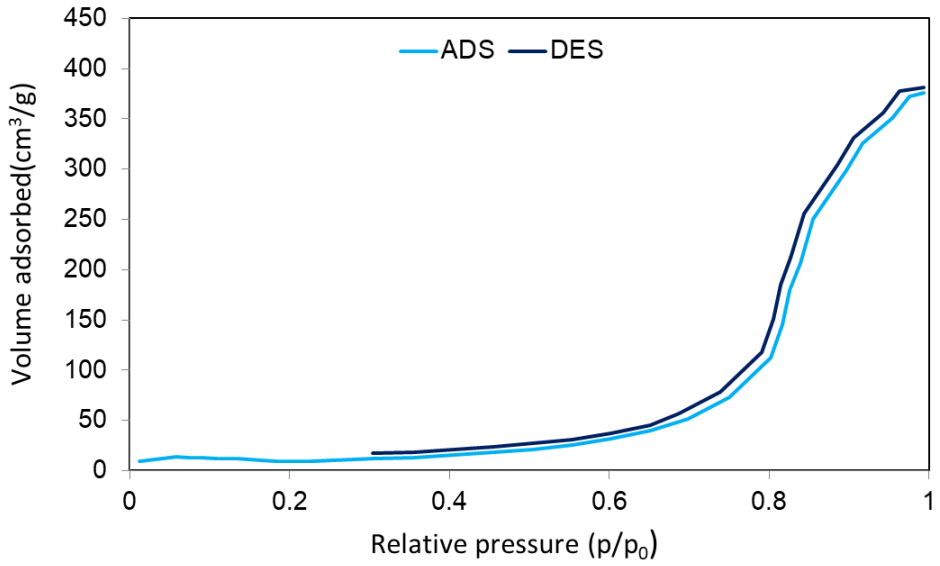

Figure 4. Nitrogen adsorption-desorption for pomegranate peel activated carbon

Adsorption studies

The effect of the initial concentration of MB and the contact time.

One of the most important criteria for evaluating the efficiency of the adsorption process is the initial concentration of the pollutant. The effect of a change in concentration between 50 and 150 mg/L on the adsorption of methylene blue on the 0.08 g PPAC surface is investigated on the adsorption capacity and removal% Table 1 and Figure SI3. The practical results showed that with increasing the dye concentration, the removal decreases, and this continues over time, i.e. at all times that were used from 30 to 180 min, but at the equilibrium time, which is about 120 min, the decrease is very small compared to the times before 120 min. The current study, like other previous studies, showed that an increase in the initial concentration of the dye leads to a decrease in the removal rate, as it was noted that the removal rate decreased from 100% to 35% in the first half hour due to insufficient active sites that are ready for adsorption. This indicates that a greater concentration of MB requires an extra time to get ready trap for other molecules. The results showed that the adsorption rate initially was relatively high as there were many active sites available on the surface of the PPAC material [22]. It is noted that with increasing time, the adsorption or removal rate increases until it reaches a certain time in which the adsorption decreases, which is called the equilibrium time. We note that after an hour has passed, the surface adsorption capacity decreases due to the lack of sufficient effective sites to adsorb the methylene blue dye. This indicates that when the concentration of MB increases, surface collisions occur between the adsorbed surface and the dye repeatedly, which leads to an increase in the elimination ability. Indeed, with the passage of time, the gap in the removal percentage decreases in adsorption of all concentrations, as the percentage of elimination was 30% in the

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first 30 min, but after three hours it became 80%. This is probably due to taking enough time to adsorb the remaining molecules of the adsorbate on the PPAC adsorbent. As for the surface adsorption capacity, it increases with time and also tends towards the path that removal is directed towards. It is clear that with increasing concentration and at constant time, the adsorption capacity increases significantly, reaching up to 83.96 mg/g at 150 mg/L after 180 min, except for the first half hour, which gave results that were the opposite of the results obtained at other times.

Effect of PPAC amount

The adsorbent dose is strongly related to the total cost of the adsorption process as well as the number of active sites, and is an important component of the adsorption technology [23]. It is well known that using large doses of the material leads to an improvement in increasing the adsorption rate, but in return it costs a lot of the adsorption process, and therefore finding the optimal weight to achieve the best adsorption rate with the less cost is necessary. However, in this recent study, different weights were used, ranging from 0.02 g to 0.1 grams Table 1 and Figure SI4, while maintaining the normal pH solution of 7, the equilibrium time of 100 minutes, and the initial concentration of the dye 100 mg/L. Increasing the weights used for the adsorbent certainly leads to an increase in the surface area, which leads to an increase in the potential effective sites for capturing or catching the pollutant, and thus the adsorption rate increases [24]. As can be observed, increasing the dosage of the adsorbent resulted in a drop in adsorption capacity from 124.3 mg/g to 50 mg/g when increasing from 0.02 to 0.1g.

Effect of PH

Another factor that is also considered the most important factor in affecting adsorption process is the acidity function (pH). Methylene blue dye is classified as a positive dye, meaning that when it is dissolved in water, it carries a positive charge. Therefore, the degree of ionization of this dye is closely related to the value of the solution's pH. Therefore, the process of changing pH solution requires intensive study in order to obtain the best results of adsorption of this dye on PPAC surface. [14]. This leads to the conclusion that the surface charge also plays a major role compared to the dye charge. Therefore, finding the surface charge is very important to know the appropriate acidic or basic medium to obtain the best adsorption process. Accordingly, the surface charge "the zero-point charge (pHZpc)" was studied according to this method [25] as presented in Figure SI5. The pHPZC of PPAC was found to be 6.7 which indicates that it is neutral in nature. Therefore, it was found that PPAC requires a basic or natural environment in order to achieve efficient adsorption. The effect of the pH of the initial solution on the adsorption of MB on PPAC was studied in the pH range (3, 5, 7, 8, 11) as shown in Table 1 and Figure SI6. We noticed in this study that in acidic conditions, the adsorption of MB is relatively low, ranging between 27 and 41% within 180 min. However, in basic conditions a high adsorption property is achieved (39-65%). In an acidic medium, the methylene blue dye shows repulsion forces against the H+ ions present in acidic media on the surface of the adsorbent. It is well known that the hydroxyl groups on the surface of PPAC were advantageous for the adsorption of adsorbent on the surface of activated carbon [26]. The cause for the decrease of MB adsorption at pH 3 may

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be the protonation of amino acid and carboxylic acid functional groups in the adsorbent, which increases the electrostatic repulsion of cationic MB dye. The adsorption of methylene blue on sorbents increases when the starting pH rises to a basic medium [27].

Effect of temperature

Another vital factor that directly affected the adsorption of dyes was temperature, which affects the solid-solute interface and the movement of contaminants during adsorption [28]. In this work, the MB adsorption removal effectiveness improved with increasing solution temperature because of the availability of more active sites as a result of the adsorbent surface activation at high temperatures [29]. From Table 1 and Figure SI7, it was observed that the absorption ability values increased as the temperature was raised because of the increased movement of large dye ions [30]. It was observed that the effect of temperature has positively affected the increase in the percentage of methylene blue removal, as the removal percentage increased from 75.65 to 90.15 with a temperature increase from 20 °C to 50 °C. At the same time, the adsorption capacity also improved significantly, as it increased from 94.57 to 112.68 mg/g with the same difference in temperature. This indicates that the increase in temperature resulted in an increase in the movement of the solute molecules and thus increases the speed of their adsorption diffusion, as proven in previous studies [18].

Effect of solution volume

Considering the previous studies, it was not noticed that there are many studies on the effect of the volume solution on the process of adsorption of pollutants, except for some research conducted by us [18], [31]. Therefore, in this study, the effect of the solution volume was adopted, and the extent of its effect on the removal% was studied, as well as the adsorption capacity of the dye using pomegranate peels activated carbon, where the dye concentration is fixed and then the volume is changed as presented in Table 1 and Figure SI8. Accordingly, the number of moles of the methylene blue dye will certainly change according to the change in volume. Therefore, increasing the number of moles will depend on increasing the volume because the concentration is fixed. Pomegranate peels activated carbon showed an actual effect in the adsorption of the methylene blue dye in the selected volume that were adopted, which are 25, 50, and 100 mL, noting that the large solution volume of 100 showed a little bit lower removal% than other volume but high adsorption capacity 93.56% and 116.95 mg/g, respectively. This is very clear because the number of moles, i.e. the number of molecules for the methylene blue dye has increased compared to the two previous volumes (25 and 50 mL), as shown in Figure SI8. In contrast, the small volumes, specifically volume 25 mL, gave a large removal% compared to the other volumes, as the removal rate reached 98.4%, but the adsorption capacity was greatly reduced compared to the two previous volumes, 30.75 mg/g. This is clear considering that the volume of the solution according to equation number 2 has a direct effect on the adsorption capacity, i.e. it is directly proportional to it. We conclude from this that small volumes give great removal and the reason is that the number of molecules in small volumes is few compared to the active sites present on the surface and therefore the surface is fully prepared

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to trap these pollutants. Through the study conducted, we conclude that the volume of 100 ml resulted the highest value of adsorption capacity reaches 116.9534 mg/g.

Table 1. Removal% and adsorption capacity (Q) under effect of all selected parameters for the adsorption of MB on PPAC.

	Effect of initial MB concentration [a]						
	50	**80**	**120**	**150**			
R%	100	100	99.4	89.5			
Q (mg/g)	31.25	50.00	74.55	83.96			
	Effect of amount PPAC [b]						
	0.02	**0.04**	**0.06**	**0.08**	**0.01**		
R%	35.5	68.5	95.9	99.3	99.4		
Q (mg/g)	59.6	74.5	95.9	102.8	106.5		
	Effect of pH [c]						
	pH 3	**pH 5**	**pH 7**	**pH 10**			
R%	42.2	62.3	94.7	99.4			
Q (mg/g)	52.8	77.9	118.4	124.2			
	Effect of temperature [d]						
	20 °C	30 °C	40 °C	50 °C			
R%	96.1	98.3	99.2	99.5			
Q (mg/g)	72.1	73.7	74.4	74.6			
	Effect of solution volume [e]						
	25 mL	50 mL	100 mL				
R%	100	99.3	89.33				
Q (mg/g)	37.5	74.5	134				

[a]180 min, 50 mL solution, 0.08 g PPAC; [b]180 min, 50 mL, 120 mg/L MB; [c]180 min, 0.08 g PPAC, 120 mg/L MB, 50 mL solution; [d]180 min, 50 mL solution, 120 mg/L, 0.08 g PPAC; [e]180 min, 50 mL solution, 120 mg/L MB, 0.08 g PPAC.

Adsorption isotherms

Analysis of the isotherms model of adsorbents is necessary as it assists to achieve great interpretation of the reaction between adsorbents and MB. Thus, this investigation was done to evaluate the adsorption isotherms of PPAC on MB at varied concentrations (80-400 mg/L), 180 min and 0.08 g. The MB adsorption isotherms data were nonlinearly fitted using the Langmuir and Freundlich isotherms models as shown in Table 2. The R2 values of the nonlinear isotherm models were 0.9979 and 0.9114, respectively. This study shows that the method of dye adsorption on the surface of pomegranate peels is closer to Langmuir model, that is, adsorption process involves the formation of a single layer on the PPAC surface. This is called homogeneous adsorption. The evidence that the adsorption is closer to Langmuir than Freundlich models is that the correlation factor is great [32]. To evaluate the fitting performance of the non-linear Langmuir and Freundlich models, the correlation coefficient (R^2) values were compared. The results indicate that both models exhibit nearly identical adsorption behavior for methylene

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blue (MB) on the adsorbent surface, as illustrated in Figure 5. The equilibrium constants obtained from the calculations are summarized in Table 2. Based on previous studies, it is evident that the adsorption of crystal violet dye onto activated carbon derived from different biomass sources typically follows the Langmuir isotherm model, which is in agreement with the results obtained in the current investigation [33]. According to the Langmuir model, PPAC has a maximum adsorption ability (Qmax) of 116.218 mg/g. This shows that the synthesized PPAC has an excellent ability to adsorb MB.

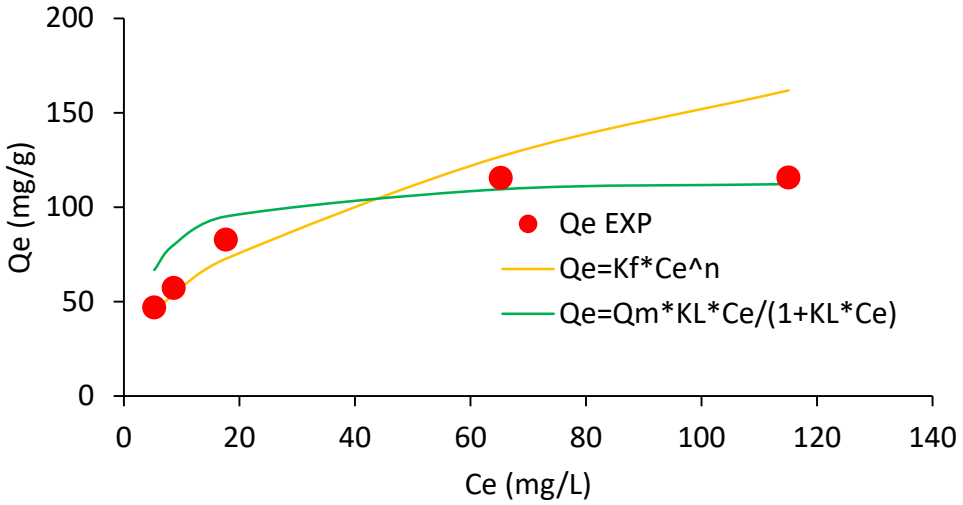

Figure 5. (Green solid line) Model of the Langmuir isotherm for MB adsorption on PPAC. (Yellow solid line) Model of Freundlich isotherm for MB adsorption on PPAC. under these conditions: 180 min, 0.08 g PPAC, MB dye solution concentration (80–300 mg/L).

Table 2. Non-linear Isotherm parameters for Langmuir and Freundlich models after adsorption of methylene blue on PPAC adsorbent under the following conditions: 0.08 g adsorbent, 50 mL, 180 min at 3 replications.

Non-linear Langmuir isotherm		
Qm ± SD (mg/g)	**KL (L/mg)**	**R^2**
116.218 ± 2.4	0.25476	0.9979
Non-linear Freundlich isotherm		
KF (mg/g) $(L/mg)^{1/n}$)	**n**	**R^2**
0.43±0.03	44.321	0.9114

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Thermodynamic studies

Studying the effect of temperature has an interesting connection in knowing the values of thermodynamic functions, which have a great impact in determining the mechanism of the adsorption reaction, as the change in Gibbs' energy (Δ°G) has the basic role in knowing the spontaneity of the reaction or its non-spontaneity. The more negative the value, the more spontaneous the reaction is, and the more positive it is, the more non-spontaneous the reaction is [34]. The effect of different temperatures (293, 303, 313 and 323 K) of adsorption MB on PPAC was studied. The results indicated a direct relationship between temperature and adsorption process of methylene blue dye. Thermodynamic parameters provide insight into the spontaneous and energetic feasibility of the adsorption process [35]. The temperature changes throughout the adsorption process were investigated using thermodynamic parameters. Equation (3) represents the Gibbs free energy change (Δ°G, KJ/mol), whereas Equations (4, 5) calculate the enthalpy change (Δ°H, KJ/ mol) and entropy change (Δ°S, J/ mol K) [36].

$$\Delta G^{\circ} = \Delta H^{\circ} - T\Delta S^{\circ} \quad [3]$$

$$\ln\left(\frac{C_s}{C_e}\right) = \frac{\Delta S^{\circ}}{R} - \frac{\Delta H^{\circ}}{RT} \quad [4]$$

$$K_d = \frac{C_s}{C_e} \quad [5]$$

where Kd is the coefficient of distribution; Cs is the equilibrium concentration in the solid phase, and Ce is the equilibrium concentration of MB in the liquid phase (mg/L). R (8.314 J/mol.K) represents the universal gas constant, while the T (K) refers to the absolute temperature. The Gibbs free energy is entirely negative, ranging from -9.196 to -18.058 kJ/mol. Adsorption of PPAC to MB at various temperatures is spontaneous and possible. Δ°H is also positive (77.363 kJ/mol), revealing an endothermic process during PPAC adsorption onto MB. Δ°S is equal to 0.295 J/K.mol . All thermodynamics data have presented in Table 3. It is a measure of the disorder or randomness present in the system." Positive Δ°S values suggest increased disorder and randomness at the solution interface of PPAC and MB during adsorption [37].

Table 3. values for the thermodynamic parameters for the methylene blue adsorption onto PPAC at various temperatures

Temperature (K)	**Thermodynamic parameters**		
	ΔG° (kJ/mol)	**ΔH° (kJ/mol)**	**ΔS° (kJ/mol.K)**
293	-9.19586	77.3626	0.295421
303	-12.1501		
313	-15.1043		
323	-18.0585		

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Kinetic studies

The study of reaction kinetics aims to understand the behavior of the reaction between the solute, which is meant here as the dye, and the surface, which is meant here as the adsorbent. As well known, there are two main types of kinetics, which are the Pseudo-first-order kinetic and pseudo-second-order kinetic.

The data on kinetics were investigated using PFO linear and PSO linear models, as indicated below in equations (6) and (7).

$$Q_t = Q_e\left(1 - e^{-Kad1t}\right) \quad [6]$$

$$Q_t = \frac{Q_e^2 . K_{ad2} . t}{(1 + Q_e . K_{ad2} . t)} \quad [7]$$

where Qe represents the adsorption capability at a state of equilibrium (measured in mg/g) and Qt represents the adsorption capacities at any moment t (min), (measured in mg/g). The rate constants for PFO (min^{-1}) and PSO (g/mg.min) adsorption processes are K_{ad1} and K_{ad2}, respectively. The results showed that the data fit both pseudo-first-order and pseudo-second-order model, as it gave a high value for the correlation coefficient "R^2" of 0.9839 and 0.9995, respectively (see Figure 6 and Table 4). From this, we can conclude that chemisorption and physisorption occur because it provides a good correlation for the adsorption of MB by PPAC.

Table 4 non-linear kinetic parameters of adsorption of methylene blue MB on PPAC adsorbent under these following conditions: 0.08 g PPAC adsorbent, 50 mL, 120 mg/L MB at 3 replications.

Pseudo-first order		**Pseudo-second order**	
K_{ads1} ± SD (min^{-1})	0.1168	K_{ads2} ± SD (g/min. mg)	0.00217
Qe ± SD (experimental)	70.96	Qe ± SD (experimental)	76.55
Qe ± SD (Calculated)	70.94	Qe ± SD (Calculated)	74.44
R^2	0.9932	R^2	0.9921

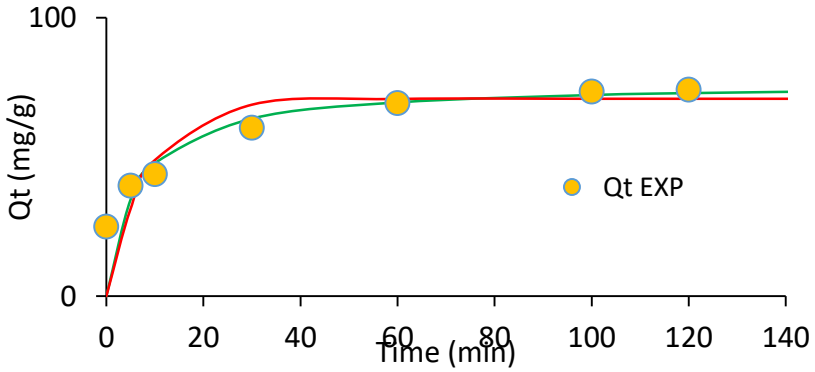

Figure 6. non-linear kinetic models for adsorption of methylene blue (MB) on PPAC adsorbent. (red color solid line) non-linear Pseudo first order model, and (green color solid line) non-linear Pseudo second order model. under these conditions: 120 mg/L MB solution, 50 mL, 0.08 g PPAC.

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Mechanism of adsorption

The adsorption mechanism occurs by oppositely charged ionic interactions such as dipole-dipole, induced dipole-dipole, hydrogen bonding, chemical bonding, and ion exchange [38]. The presence of many active functional groups on the surface is critical to dye adsorption in MB adsorption. Two major components influencing the adsorption process have been identified via research. The structure of the methylene blue dye is initially formed, followed by the appearance of functional groups on the leaf's surface. FTIR analysis reveals that several functional groups, including C=O, CH, CO, and OH, are accessible by the PPAC adsorbent. The functional groups present on the surface of the adsorbent may be the basis for the adsorption process, given that the dye carries a positive charge, and thus some of these groups carry a negative charge, which leads to the adsorption process via electrostatic interaction, H-bonding, π-π interaction, and n-π interaction [39].

Regeneration PPAC adsorbent

Regeneration of the adsorbent surface is an important and cost-effective approach in scientific studies, as it enables the assessment of the adsorbent's reusability with varying efficiencies. In this work, the regeneration capability of activated carbon prepared from pomegranate peels for the adsorption of methylene blue (MB) dye was examined. A chemical reagent–based column bed adsorption–desorption technique was employed due to its simplicity and low operational cost (Figure 7). In our previous study on the removal of malachite green dye using pomegranate peel activated carbon [40], four eluents with different physicochemical characteristics—water, 0.1 M HCl, methanol, and dilute acetic acid (1:1, v/v)—were tested, with dilute acetic acid showing the highest desorption efficiency. Based on these findings, an acetic acid–water mixture (1:1, v/v) was used in the current study for the regeneration of pomegranate peel activated carbon (PPAC). The experimental procedure involved passing 25 mL of a 20 mg/L MB solution through a column cartridge packed with 0.2 g of PPAC. The regeneration process demonstrated a high desorption efficiency of 97.55% during the first cycle, which decreased to 90.71% in the second cycle and gradually declined to approximately 73.9% by the fifth cycle.

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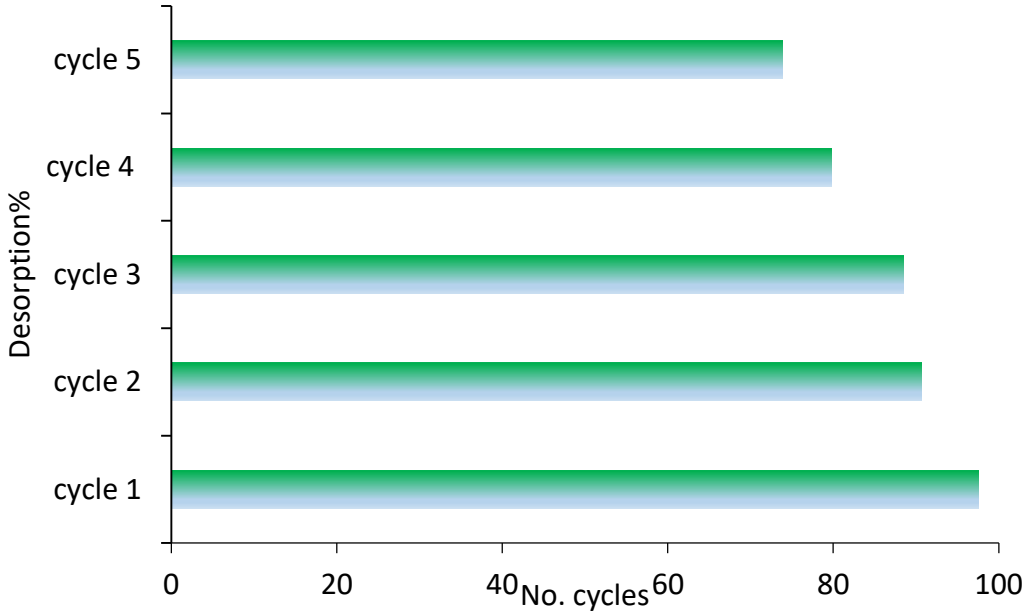

Figure 7. Evaluation the adsorption-desorption method for the sustainability of pomegranate peel activated carbon (PPAC) on methylene blue dye MB under these experimental conditions: 20 mL of the eluent (1:1, acetic acid, water); 25 mL of 20 mg/L MB; five cycles.

Matrix effect on adsorption MB

The effect of water matrix composition on the adsorption of methylene blue (MB) was investigated by evaluating the performance of PPAC in different water samples, including river water (RW), well water (WW), distilled water (DW), and saline water (SW, 1000 mg/L NaCl), as illustrated in Figure 8. All experiments were conducted under constant conditions: 0.08 g of PPAC, 50 mL of 120 mg/L MB solution, and a shaking time of 180 minutes. The findings showed that the highest removal efficiency occurred in distilled water (97.14%), whereas river water exhibited the lowest removal efficiency (84.1%), likely due to dissolved impurities that compete with dye molecules for the available adsorption sites. Saline water and well water demonstrated intermediate removal efficiencies of 96.0% and 96.5%, respectively, both slightly lower than that observed in distilled water. These results indicate that the composition of the water matrix significantly influences the adsorption efficiency of dyes onto activated carbon.

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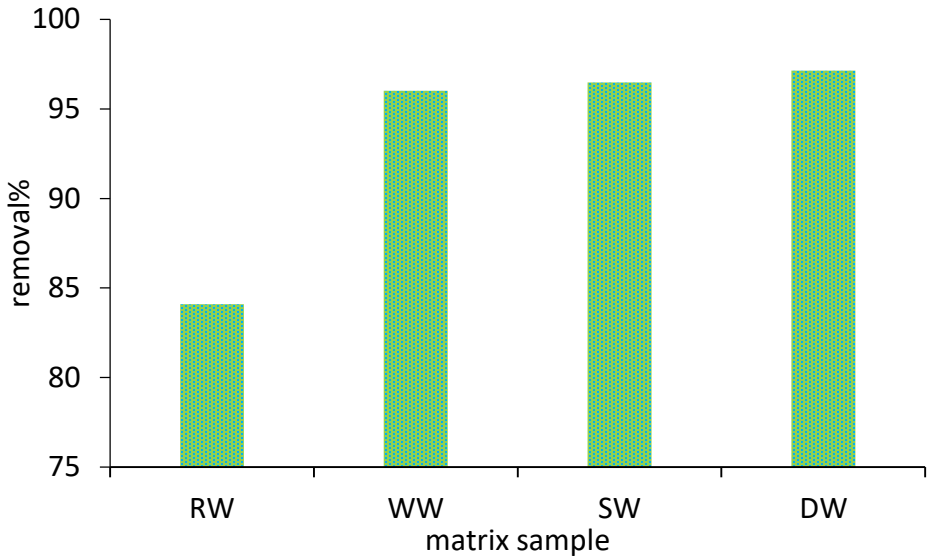

Figure 8. Different investigation of efficiency PPAC on removal of methylene blue MB from different matrix samples river water (RW), well water (WW), saline water (SW) and distilled water (DW) under these conditions: 0.08 g PPAC, 120 mg/L dye, 50 mL solution and 180 min shaking.

CONCLUSIONS

Based on the results obtained, we conclude that the surface of pomegranate peels activated by sodium hydroxide plays a major role in their ability to adsorb methylene blue dye from its aqueous solutions. The study of the effect of solution volume had a significant impact in obtaining visible and highly dynamic results, showing that large sizes for the same concentration give lower removal rates with high vibration amplitude. Regarding the effect of temperature, it was found that the adsorption process increases directly with the increase in temperature, as it was noted that the highest removal rate and adsorption capacity were 90.15% and 112.68 mg/g at a temperature of 50 C° compared to other temperatures under the same conditions. As for the adsorption isotherm models, the process of adsorption of dye on the surface of pomegranate peels activated carbon was more compatible with non-linear the Langmuir model, as the R2 =0.9979. The maximum adsorption capacity was 116 mg/g. The reaction kinetics intended for the Pseudo-first order and Pseudo-second order reaction in which the adsorption process of the MB dye on the surface of the pomegranate peels obeys the non-linear Pseudo-second order. The thermodynamic functions (ΔG, ΔH, and ΔS) were calculated according to the Vant' hof equation and it was shown that the desorption process is endothermic and spontaneous without any external influences. The Gibbs energy ranged between -9.196 and -18.0585 kJ/mol within the temperatures used, while the enthalpy of reaction and the entropy were 77.3626 kJ/mol and 0.2954 kJ/mol.K. Therefore, it can be suggested that PPAC is a potential low-cost adsorbent in removing the methylene blue dye from its aqueous solution.

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The study concluded that the prepared activated carbon (PPAC) is an effective adsorbent for the removal of methylene blue (MB) dye from various water matrices, including well water, river water, distilled water, and saline water. However, the composition of the water significantly affected the adsorption efficiency. The lowest removal efficiency was observed in river water, whereas the highest removal occurred in distilled water. In addition, PPAC exhibited good reusability, maintaining strong adsorption performance through several regeneration cycles, with desorption efficiencies reaching up to 88% by the third cycle. The resulting peels can be considered and taken into consideration in future concerns for water treatment and environmental protection, thus contributing to what is called sustainability.

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Conflict of interests.

There is no conflict of interests.

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الخلاصة

المقدمة: في الواقع، يُؤثر التلوث الناتج عن محطات معالجة مياه الصرف الصناعي سلبًا على البيئة المائية. وعليه، تُظهر هذه الدراسة طريقة سهلة لفصل صبغة الميثيلين الأزرق (MB) من محلولها المائي باستخدام الكربون المنشط المُستخلص من قشور الرمان.

طرق العمل: أُجريت دراسات توصيفية لدراسة خصائص الكربون المنشط باستخدام تقنيات حيود الأشعة السينية (XRD) والمجهر الإلكتروني الماسح (SEM) ومطيافية الأشعة تحت الحمراء بتحويل فوريه (FTIR). كما أُجريت تجارب عملية لدراسة تأثير عدة متغيرات، أهمها: زمن التلامس (0–180 دقيقة)، والتركيز الابتدائي (50–150 ملغم/لتر)، وجرعة المادة الماصة (0.02–0.1 غرام)، ودرجة الحموضة (3–11)، وحجم المحلول (25–100 مل)، ودرجة الحرارة (20–50 درجة مئوية).

النتائج: تختلف معاملات الارتباط بين منحنيات امتزاز لانغمير وفروندليش، حيث بلغت 0.9979 و0.9114 على التوالي. وقد أثبت تحليل نموذج لانغمير ملاءمته لتفسير عملية امتزاز صبغة الميثيلين الأزرق على سطح المادة المازة، والتي تميل بطبيعتها إلى تكوين طبقة أحادية من الصبغة الممتزة على سطح الكربون المنشط. وبلغت سعة الامتزاز القصوى 116 ملغم/غم. أما بالنسبة لدراسة الحركية، فقد كان تفاعل الرتبة الثانية الزائفة (R^2 = 0.9995) هو الأقرب لتمثيل حركية امتزاز الصبغة على السطح. وقد أشارت نتائج تقييم المعاملات الديناميكية الحرارية إلى أن الامتزاز تلقائي وماص للحرارة. حيث كانت قيم $\Delta G°$ و $\Delta H°$ و $\Delta S°$ كما يلي: (–9.196 إلى – 18.058 كيلوجول/مول)، 77.363 كيلوجول/مول، و 0.2954 كيلوجول/مول كلفن.

الاستنتاجات: في الختام، أثبتت الدراسات أن الكربون المنشط المستخلص من قشور الرمان مادة ماصة فعالة وغير مكلفة لإزالة الميثيلين الأزرق من مياه الصرف الصحي.

الكلمات المفتاحية: قشور الرمان؛ التنشيط الكيميائي؛ الامتزاز؛ الميثيلين الأزرق؛ نموذج لانغمير وفروندليش.

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