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Kinetic and thermodynamic study of the adsorption isotherm of methylene blue dye from aqueous solutions by activated carbon

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ABSTRACT

The efficiency of activated carbon in wastewater as a powerful blue dye remover has been studied. In this work, activated carbon manufactured from Sidr leaves was used to adsorb methylene blue dye. The factors affecting adsorption were studied, such as the weight of the adsorbent, temperature, contact time, and initial concentration of the dye, and the optimal conditions for adsorption were determined according to the experimental results. It was evident from the experimental results obtained that the removal efficiency increases with the increase of both the weight of the adsorbent material and the contact time. It was observed that the adsorption process was possible, spontaneous, and exothermic in the temperature range (293-333 K), and this is what determined the thermodynamic variables ΔG°, ΔH°, and ΔS°. The Langmuir, Freundlich, and Timken isotherms were used to examine the experimental equilibrium data. To follow the adsorption process, two logical models were chosen, represented by the Elovage equation and diffusion.

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دراسة حركية وثرموديناميكية لتساوي الحرارة لامتزاز صبغه المثلين الزرقاء من المحاليل المائية بواسطة الكربون المنشط

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الملخص

تمت دراسة كفاءة الكربون المنشط في مياه الصرف الصحي كمزيل قوي للصبغة الزرقاء. وفي هذا العمل تم استخدام الكربون المنشط المصنع من أوراق السدر لامتصاص صبغة الميثيلين الزرقاء. وتمت دراسة العوامل المؤثرة على الامتزاز مثل وزن المادة المازة، تم تحديد درجة الحرارة وزمن التلامس والتركيز الأولي للصبغة والظروف المثلى للامتزاز بناء على النتائج التجريبية. وتبين من النتائج التجريبية المتحصل عليها أن كفاءة الإزالة تزداد بزيادة كل من وزن المادة الممتزة. وقد لوحظ أن عملية الامتزاز كانت ممكنة وعفوية وطاردة للحرارة في نطاق درجات الحرارة (293–333كلفن)، وهذا يحدد المتغيرات الديناميكية الحرارية $\Delta G°, \Delta H°, \Delta S°$ تم استخدام متساوي الحرارة وفرندلخ وتيمكين ولانكماير لفحص بيانات التوازن التجريبية. لمتابعة عملية الامتزاز تم اختيار نموذجين منطقيين ممثلين بمعادلة الانتشار وايلوفج

1- Introduction

For a long time, dyes have been used in several industries, such as the plastic industry, leather, cosmetics, food industries, and the paper industry [1]. However, there are some negative effects released by these industries that pose major environmental problems and risks [2]. Synthetic dyes were used as coloring materials, as demonstrated by many of these industries. Synthetic dyes are difficult to analyze biologically when they are discharged into waste streams because these dyes have a synthetic origin and complex aromatic molecular structures [3]. Removal of dyes is a source of great concern, due to the characteristic and carcinogenicity of these dyes [4]. In the end, biological analysis cannot be relied upon alone in treating them. The study was conducted on ethylene blue dye, which often serves as a model compound for removing organic pollutants and colored objects from aqueous solutions. To remove the dyes, conventional wastewater was used, such as trickling filter, chemical coagulation, photolysis and activated carbon adsorption [5]. The adsorption method is considered the most optimal among the other chemical and physical methods that were tested [6]. Among other techniques, the adsorption process is considered the most suitable for removing organic and inorganic water pollutants from wastewater because it possesses great advantages such as low and profitable cost and high efficiency compared to other methods. Adsorption technology is considered one of the easy techniques for removing water pollutants, even at very low concentrations and high and low temperatures. The strength of adsorption performance is related to the unique properties of the designed absorbent materials, as adsorption is a surface phenomenon [7]. Adsorption is divided into two main parts: chemical adsorption, which involves the formation of chemical bonds between the adsorbent molecule and the surface of the adsorbent, and physical adsorption, which involves weak intermolecular forces [8]. To remove toxic substances from water, activated carbon is considered the most widely used because of its unique properties represented by its large surface area, its high absorption capacity, its porous structure, its high resistance to corrosion, its inert nature, and its surface reactivity[9].The development of activated carbon comes from relatively low cost sources, high efficiency, biodegradability, and the ability to remove dyes in more concentrated forms. Through the process of chemical activation or the process of physical activation, activated carbon is prepared. Activated carbon is a porous material that, during its production, suffers from a defect in its crystalline structure, which leads to the appearance of pores or gaps that are unstable in terms of their energy content, which lies behind the high ability of activated carbon to adsorption [10]. Activated carbon production consists of two stages: carbonization and activation [11]. The synthesis of activated carbon consists of two stages: activation and carbonization. By using activated carbon as an absorbent material in the study, the methylene blue dye was removed by adsorption technology [12]. In this article, the optimal conditions affecting the adsorption process were studied, such as (adsorbent concentration, initial dye concentration, and temperature). To get the best fit correlation, kinetic parameters were also computed to ascertain the adsorption process that was fitted into adsorption isotherms.

2- Materials and methods

Activated carbon prepared from Sidr leaves, methylene blue (MB) dye, and ion-free water were used.

2.1. Preparation of the adsorbent solution

Deionized water was used to prepare all solutions, and methylene blue dye of very high purity was used, the composition of which is shown in Figure 1.

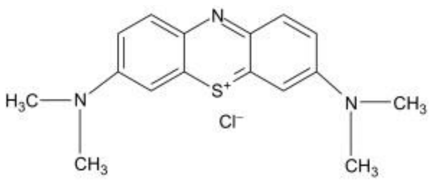

Fig. 1: Structure of methylene blue

3. Results and Discussion

3.1. Adsorption studies

After preparing different concentrations of methylene blue dye, the best concentration was determined to study the factors affecting adsorption (30ppm), as well as determining the molar absorption coefficient, by drawing the calibration curve represented in Figure 2.

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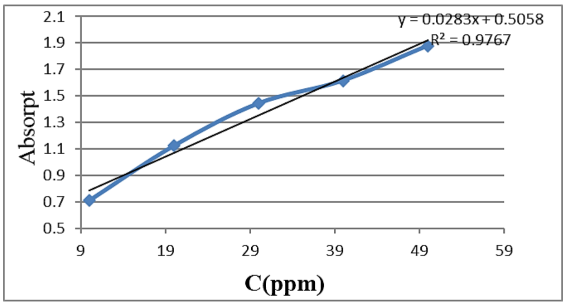

Fig. 2: Calibration curve of methylene blue dye

3.2. Effect of weight, Time, and temperature factor

A concentration of 30 ppm methylene blue dye was used in a volume of 30 ml in five 50 ml bottles. Different weights of activated carbon (0.001, 0.006, 0.007, 0.008, 0.009) were added, and the volumetric bottles were placed in a water bath at a temperature of 25 °C with a constant shaking speed of 90 r/min. The samples were then measured with a UV device, and the results shown in Table 1 and Figure 3 were obtained. A weight of 0.001 g was determined to study other variables. In the same way, the best time was determined after weighing 0.001 g of activated carbon. Each weight was placed in five volumetric bottles, each containing 30 ml of dye, and filtered at different times. The results shown in Table 2 and Figure 2 were obtained, where it was the best time required for the adsorption process. It is (100) minutes, where the removal rate reached 97.78 %. Finally, the best suitable temperature for adsorption was determined, as shown in Figure 3 and Table 3, where it reached 35 K, and the removal rate at that time was 97.66%.

Table 1: shows the effect of weight on the adsorption process

NO.	C^0 (ppm)	Wt(g)	λ (nm)	C_t (ppm)	%R
	30	0.001	0.0198	0.7006	97.665
		0.006	0.027	0.9554	96.815
		0.007	0.045	1.5924	94.692
		0.008	0.065	2.3001	92.333
		0.009	0.066	2.3355	92.215

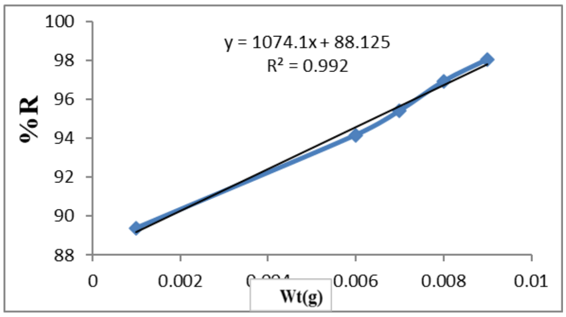

Fig. 3: shows the effect of weight on the adsorption process

Table 2 shows the effect of time on the adsorption process

NO.	C^0 (ppm)	Time (min)	λ (nm)	C_t(ppm)	% R
	30	20	0.782	27.672	7.76
		40	0.601	21.267	29.11
		60	0.098	3.4678	88.44
		80	0.0192	0.6794	97.73
		100	0.0188	0.6653	97.78

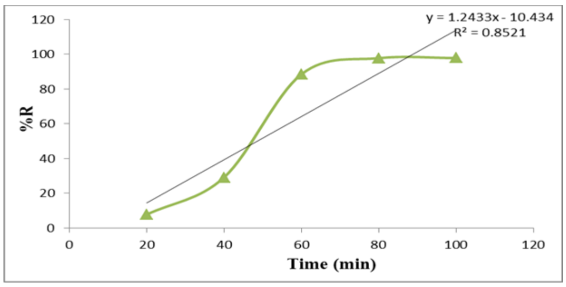

Fig. 4: shows the effect of time on the adsorption process

Table 3 shows the effect of temperature on the adsorption process

NO.	C^0 (ppm)	TC°	λ (nm)	C_t(ppm)	%R
	30	35	0.0198	0.7006	97.665
		45	0.027	0.9554	96.815
		55	0.045	1.5924	94.692
		65	0.065	2.3001	92.333
		75	0.066	2.3355	92.215

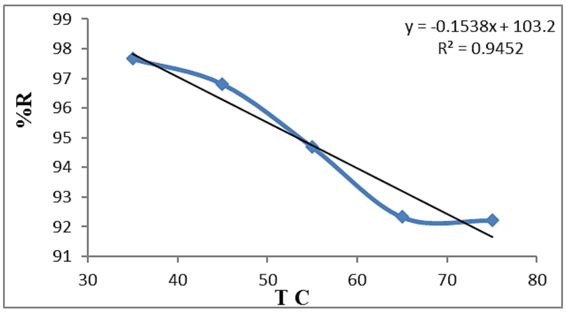

Fig. 5: shows the effect of temperature on the adsorption process

3.3. Adsorption Isotherms

Isotherms play a crucial role in improving the use of adsorbents, as they describe how adsorbents interact with adsorbents.

3.4. Langmuir Adsorption Isotherm

The Langmuir model assumes that monolayer formation occurs on the adsorbent surface, which reduces intermolecular forces with distance. This in turn indicates that only one formula can be adsorbed at one adsorption site. The adsorbent surface must be completely homogeneous, and possess energy-equivalent and identical adsorption sites. Equation 1 shows the Langmuir model. From Figure 6, we notice that most of the points lie on the straight line with a high correlation coefficient (R2), which confirms the consistency of the results with this isotherm. [13].

$$\frac{1}{qe} = \frac{1}{qm.Kl.Ce} + \frac{1}{qm} \text{........(1)}$$

In Fig. 6 the linear Langmuir adsorption isotherm isotherm. The value of (qm, KL) was calculated from the value of the slope and the linear intercept between (qe/ce)l versus (Ce) according to mathematical equation 1 and table 4.

Table 4: Methylene blue dye adsorption on Activated carbon surface: Langmuir isotherm constants

Adsorbent surfaces	K_l (L/mg)	q_m (mg/g)	R^2
AC	-3.83E-06	0.00011	0.98

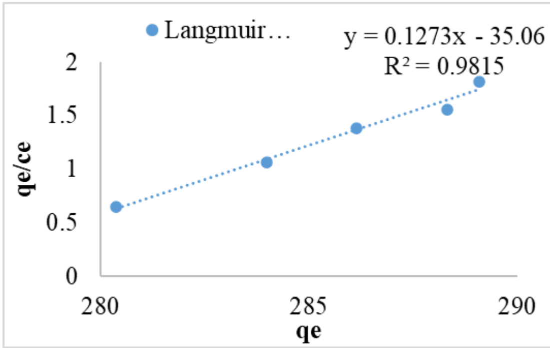

Fig. 6: Isotherm for linear Langmuir adsorption

3.5. Freundlich isotherm

The experimental Freundlich equation is based on adsorption on heterogeneous surfaces. It can be derived assuming a logarithmic decrease in the enthalpy of adsorption with an increase in the proportion of occupied sites on the adsorbent surface. Equation 2 shows the model of the experimental Freundlich equation. Table 5 shows the results obtained. Although a high correlation coefficient is obtained, most of the points do not lie on the straight line equation as is the case in the Langmuir isotherm. This is what Figure 7 shows [14].

$$\text{Ln}(qe) = \text{Ln}(K_f) + (1/n)\text{Ln}(C_e) \text{ ---- } (2)$$

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Table 5: Methylene blue dye adsorption on Activated carbon surface: Freundlich isotherm constants

Adsorbent surface	n	K_f (mg/g)	R^2
AC	32.74	283.801	0.98

From the results in Table 5, it is clear that the adsorption is physical. This is confirmed by the value, where when its value is greater than (1), the adsorption is considered physical.

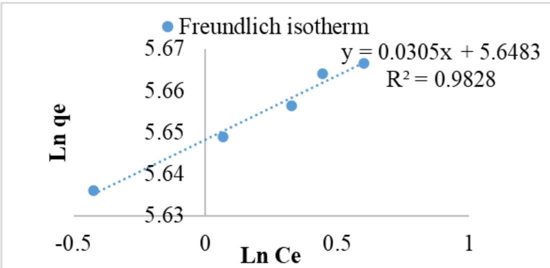

Fig.7: Isotherm for linear Freundlich adsorption

3.6. Temkin isotherm

The Temkin isotherm explains the effect of indirect adsorption/desorption interactions. This model assumes that the heat of adsorption for all molecules in the layer will decrease linearly with coverage. Equation 3 shows a Temkin model [15].

$$\mathbf{qe = \frac{RT}{bLnA} + RT/bLnCe} \text{ ------(3)}$$

Using this equation and by plotting between (qe) and (ln Ce), the values of the constants (b) and (A) were calculated from the slope and intersection of straight lines. The results obtained in Table 6 also show the Temkin constants that can be obtained from the slope of the straight line and its intersection from equations (3). When examining the results of the table, the success of applying the Temkin isotherm equation is proven through the Temkin constants that help in providing an explanation of the bonding energy between the surface molecules and the methylene blue dye. According to Figure 7, despite obtaining a high correlation coefficient (R^2), most of the points did not fall on the straight line, which shows that the results do not match this isotherm.

Table 6 shows the results obtained

Adsorbent surfaces	A (g / l)	b (J/mol)	R^2
AC	1.03	2.81	0.9809

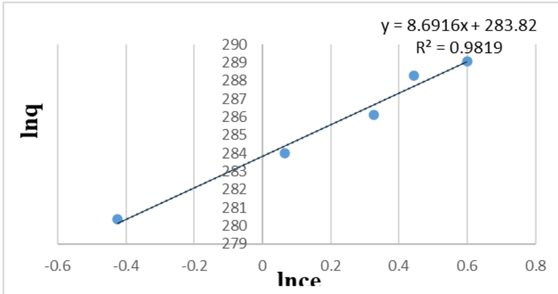

Fig. 8: Isotherm for linear Temkin adsorption

3.7. Adsorption Thermodynamic Study

In this study, the effect of temperature on the adsorption process of methylene blue dye by activated carbon was studied at different temperatures (308, 318, 328, 338, 348). This study aimed to study the thermodynamic variables (ΔG°, ΔH, ΔS°). The results in Table 7, and figure 8, Using mathematical equations (4,5,6) show that the adsorption process is nonspontaneous, endothermic physical adsorption. The Van der Hoff equation provides a thermodynamic explanation for the equilibrium of the adsorption constant, K [16].

$$K_{eq} = \frac{qmax * m(g)}{C_e * V(L)} \text{....}(4)$$

$$\ln K_{eq} = -\frac{\Delta H}{RT} + \frac{\Delta S}{R} \text{....}(5)$$

Where: Keq is the sorption distribution coefficient.

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qmax is the adsorption capacity of metals ion (mg/g), m is the quantity of nanomaterial's (g) ,Ce is the concentration at equilibrium (mg/L), V is Volume (L),ΔGo is the free energy change of sorption (kJmol-1),T is the temperature in K, R is the universal gas constant (8.314J $mol^{-1}K^{-1}$), ΔH is the enthalpy change (kJ mol^{-1}) ΔS° is the entropy change (kJ mol^{-1} K^{-1}).

The change in enthalpy and the change in entropy were calculated from the values of the slope and the intersection of straight lines between lnk versus 1/T through mathematical equations (7,8).

$$\text{Slop} = \frac{-\Delta H}{R} \ldots.(6)$$

$$\text{Intercept} = \frac{\Delta S}{R} \ldots.(7)$$

Table 7: The Values of thermodynamic function for the adsorption of Methylene blue on AC at different temperatures

Adsorbent surfaces	T (K)	ΔG° (kJ/mol)	ΔH (kJ/mol)	ΔS° (J / mol)
AC	308	11.086	42.045	-99.9161
	318	10.614		
	328	9.2042		
	338	8.0933		
	348	7.344		

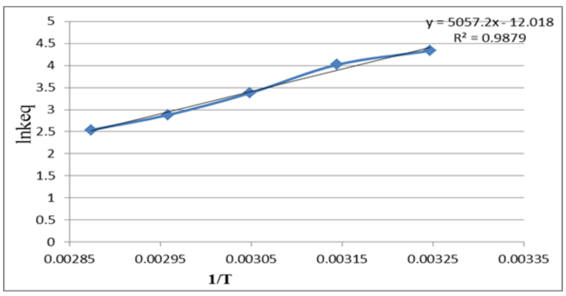

Fig. 9: Showing lnK versus 1/T to estimate the thermodynamic parameters for the adsorption of Methylene blue on the surfaces of activated carbon

3.8. The Kinetic Studies of Adsorption Process

First-order, second-order, eloage, and intraparticle diffusion kinetic models were applied to experimental data to investigate the adsorption process of methylene blue dye. The first-order, pseudo-second-order, diffusion, and Eloage kinetic model equations appear in Figures 9 to 12.

3.9. Pseudo First-order

The adsorption kinetics of blue dye at a concentration of 30ppm to the surface of activated carbon was studied using the pseudo-first order equation. From the value of the slope and the intersection of straight lines, the amount of adsorbed material was calculated(qe) and the adsorption velocity constant (K1). Equation 9 was used to obtain the results included in Table 7[17]. From the results of Table 7, it is clear that the theoretical adsorption (qe (cal)) capacity values do not match the practical adsorption (qe (exp)) capacity.

$$\text{Ln}\,(\text{qe} - \text{qt}) = \text{Ln qe} - K^1.t \;\ldots\ldots\ldots(8)$$

Table 7: The pseudo-first order kinetic parameters of activated carbon

Adsorbent surface	K_1(min^{-1} mg/g)	R^2	q_e (cal) mg/g	q_e (exp) mg/g
AC	0.098	0.79	1.466	1.2645

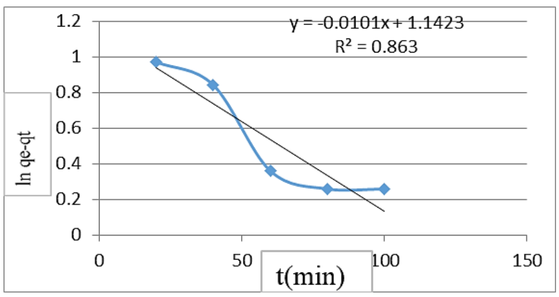

Fig. 10: The Pseudo- first order plots for methylene blue dye adsorption on activated carbon

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3.10. Pseudo Second Order

The adsorption kinetics of blue dye at a concentration of 30ppm to the surface of activated carbon was studied using the pseudo-second-order equation. From the value of the slope and the intersection of straight lines, the amount of adsorbed material was calculated(qe) and the adsorption velocity constant (K2). Equation 10 was used to obtain the results included in Table 8[18] .

$$\frac{t}{qt} = 1/K^2 qe^2 + (\frac{1}{qe})\, t \text{ ------- } (9)$$

Table 8: The Pseudo Second Order kinetic parameters of activated carbon

Adsorbent surface	K_2 $min^{-1}mg/g$	R^2	q_e (cal) mg/g	q_e (exp) mg/g
AC	0.021164	0.57	1.466	0.818

It is noted in Table 7 that the adsorption values gave a very weak linear relationship, and this was explained by the correlation coefficient (R2). In addition, The theoretical adsorption capacity values are completely different from the practical adsorption capacity.

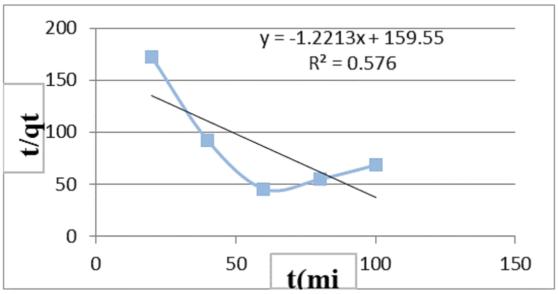

Fig. 11: The Pseudo- Second plots for methylene blue dye adsorption on activated carbon

3.11. Elovage Kinetic Equation

In this model, Equation 10 was used to obtain the results obtained in Table 9[19].

$$qt = \left(\frac{1}{\beta}\right) Ln\alpha\beta + \left(\frac{1}{\beta}\right) Ln\, t \text{ -----} (10)$$

Table 9: The Elovage Kinetic parameters of activated carbon

Adsorbent surfaces	α	β	R^2	q_e (cal) mg/g	q_e (exp) mg/g
AC	53.14	0.62	0.98	1.466	12.415

Although a high correlation coefficient (R2) was obtained as shown in Figure 11, according to Table 9, shwoing clear that the theoretical adsorption capacity values are completely different from the practical adsorption capacity.

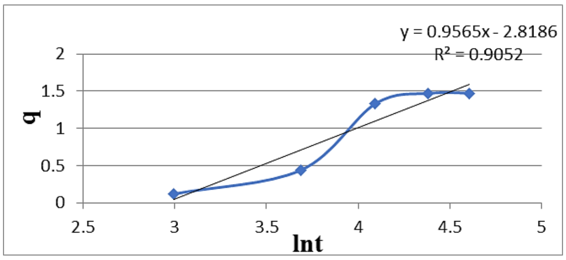

Fig 12: The elovage kinetic plots for methylene blue dye adsorption on activated carbon

3.12. The Intraparticle Diffusion

The adsorption kinetics of blue dye at a concentration of 30ppm to the surface of activated carbon was studied using the Intraparticle Diffusion equation. From the value of the slope and the intersection of straight lines, the amount of adsorbed material was calculated(qe) and the adsorption velocity constant (K2). Equation 11 was used to obtain the results included in Table 10[20].

$$qt = K_{diff}\sqrt{t} \quad . \quad + \quad C \quad \text{----} \quad (11)$$

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Table 10: The Intraparticle Diffusion parameters of activated carbon

Adsorbent surfaces	K_{diff} (mg/g.min$^{1/2}$)	C (mg/g)	R^2	q_e (cal) mg/g	q_e (exp) mg/g
AC	0.179	0.606	0.94	1.543	1.522

From Table 10 it is clear that the linear relationship obtained and according to the values of R2, it is noted that the straight line tends to deviate and stabilize after a short period of the adsorption time. The calculated values of qcal were quite close to the experimental values of qexp, so this model can be applied in the adsorption process. This is evidence that the results are consistent with this model.

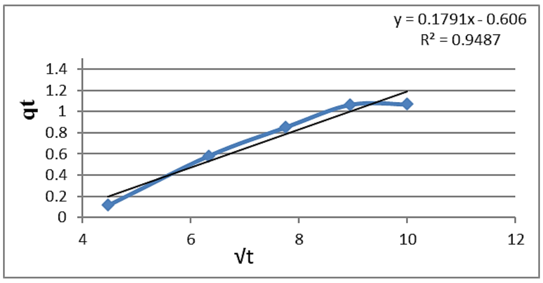

Fig. 13: The Intraparticle diffusion plots for methylene blue dye adsorption on activated carbon

4- Conclusion

In this article, it was explained that activated charcoal can be used as a powerful and inexpensive absorbent material to remove methylene blue dye from aqueous solution. It was shown from thermodynamic studies that the adsorption process was endothermic, and its spontaneity decreased with increasing temperature. Kinetic studies have proven that adsorption follows Elovage Kinetic, and according to the study of isotherms, the Freundlich isotherm has proven that adsorption is chemistry. This is demonstrated by the value of n.

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