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Using Moringa Seed Peel Powder to Remove Dyes from Aqueous Solutions

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Abstract: Moringa seed peel powder was used to remove methylene blue dye from the aqueous solution. The surface of the Moringa seed peel powder was characterized using (FT-IR) infrared spectroscopy, (BET) surface specification, and (SEM) scanning electron microscopy. The influencing factors were studied. On adsorption, which is the amount of adsorbent, contact time, initial concentration of the dye, temperature, and acid pH to predict the optimal conditions under which the adsorption process takes place. Adsorption equilibrium and adsorption rate data were evaluated using different isotherm and kinetic models. The isotherm results showed that the Langmuir model gave better results for the studied equilibrium data. The adsorption kinetic data better follow the pseudo-second order model. Thermodynamic variables were also evaluated and the calculated parameters indicated that the adsorption process was spontaneous and exothermic in nature.

Key words: Adsorption, Methylene blue, Moringa seeds, Dyes.

1. INTRODUCTION:

The aquatic environment contains many different pollutants, among which the most dangerous and toxic are dyes. The obvious dangers caused by these dyes are lack of oxygen, color and odor changes, in addition to mutations that organisms may suffer from, which may lead to cancer. Dyes are considered one of the pollutants that most affect the ecosystem because they contain many harmful chemical components[1],[2]. Methylene blue is one of the most important and most widely used dyes in silk, cotton, and wool. However, this dye causes many harmful health effects, such as vomiting, nausea, skin and eye irritation, and increased heart rate[3],[4]. There are many methods used to treat dyes present in aqueous solutions, such as coagulation, photolysis, nanofiltration, biological analysis, oxidation, etc., but the adsorption method compared to other methods has many advantages. Adsorption is considered a simple, effective and versatile process, due to its low economic cost and high potential for removing organic waste from aqueous solutions[5],[6]. Agricultural waste is considered an environmentally friendly material due to its abundant availability in nature and its unique chemical composition, in addition to its lower economic cost, which makes it a viable option for treating polluted water[7],[8]. Recently, adsorbents made from agricultural wastes such as marula seed hulls[9], coffee waste[10], wheat hulls[11], and Moringa seed pulp [12] have been used. Knowing that Moringa is the miracle tree and its original habitat is in the areas under the Himalayas in Pakistan, Africa and India, it is considered the most widely cultivated among (13) species of the (Moringaceae) family because it has a high ability to grow in hot environments. And wet and dry. This plant has many applications, including in the food, cosmetics and pharmaceutical industries due to its phytochemical and medicinal properties [13],[14],[15]. The aim of this study is to evaluate the adsorption behavior in removing methylene blue dye from aqueous solution using Moringa seed peel powder under different conditions of pH of the solution, temperature, contact time, and amount of adsorbent.

2. Experimental

2.1. Raw materials and chemicals:

Morinca seed husks were obtained from local markets, and a methylene blue dye solution was prepared with distilled water. After that, all working solutions were prepared by diluting the dye solution prepared before conducting the experiments. Laboratory reagents were used without purification: NaOH, HCl.

2.2. Preparation of the adsorbent:

The husks of the Moringa seeds were washed with distilled water to remove contaminants, then left to dry. They were then dried in an electric oven at a temperature of (100°C) for 3 hours, then ground and used a molecular sieve (Retsch, Germany) to obtain particles of a size of (100 µm).) and stored in a plastic box for use in practical experiments.

2.3. Preparation of the standard methylene blue dye solution:

The standard dye solution was prepared at a concentration of (500 mg/L) by dissolving (0.05 g) of the dye in a 100 ml volumetric vial, and the volume was completed to the mark with distilled water. The rest of the concentrations were prepared by diluting this solution, and the dye adsorption was studied using an ultraviolet-visible spectrophotometer (UV-1800, Shimadzu) at a wavelength of 665 nm.

3. Results and discussion

3.1. Analysis and characterization

3.1.1. Fourier transform infrared (FT-IR)

The results of the FT-IR spectrum in (Figure 1) showed the presence of the main absorption bands, as we see bands confined between (3410-3389 cm^{-1}) representing bonds of OH groups. It appears that the absorption peaks at 2854 cm^{-1} and 2926 are long and sharp and may be due to the C-H stretching of CH_3 or the functional group C=O. The absorption peak is confined between (1647-1637 cm^{-1}) and there is an elongation due to the expansion of the functional group of carboxylic acids, C=O. The peak observed at 151 cm^{-1} corresponds to the secondary amine group, and we also see that the peak at 1458 cm^{-1} has transformed into 1425 cm^{-1} and the peak at 1267 cm^{-1} has transformed into 1230 cm^{-1}, and the reason for this is due to the expansion of C-O. For functional groups. The peaks at 1377 cm^{-1} and 1319 cm^{-1} also have an elongation due to the bending N=O in the functional groups. Also, the peak observed at and corresponds to expansion, and 1055 cm^{-1} and 1035 cm^{-1} correspond to C=O of phenol, ester, or ether. At 653 cm^{-1} and 617 cm^{-1} corresponds to C-N stretching, while the peak observed at 590 cm^{-1} corresponds to S-O[16],[17],[18], as shown in Figure (1).

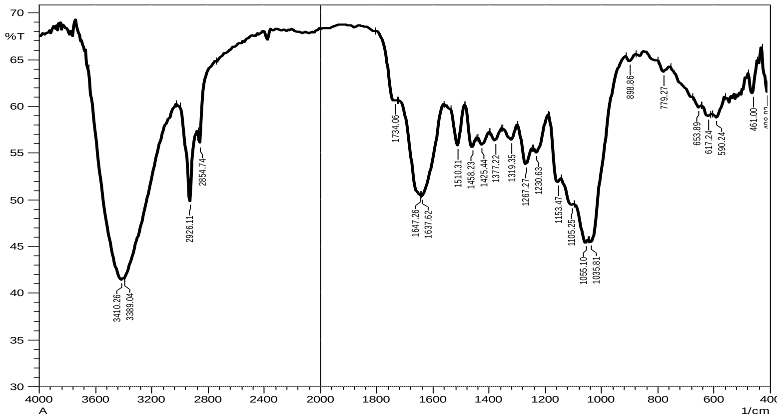

Figure (1): FTIR spectroscopy of moringa seed peels

3.1.2. Scanning electron microscope (SEM):

The scanning electron microscope (SEM) is a type of electron microscope. It may produce images of the sample by scanning the surface of the sample with a focused beam of electrons. The electrons interact with the molecules on the surface of the sample, producing various signals containing information about the surface shape of the sample. It is shown in (Figure 2) that the surface structures of the moringa seed shells were rough and uneven. It has been observed that the surface particles are more like stick-like and rectangular shapes with unequal length. Physical treatment was able to produce a porous adsorbent and thus increase the surface area[18],[19].

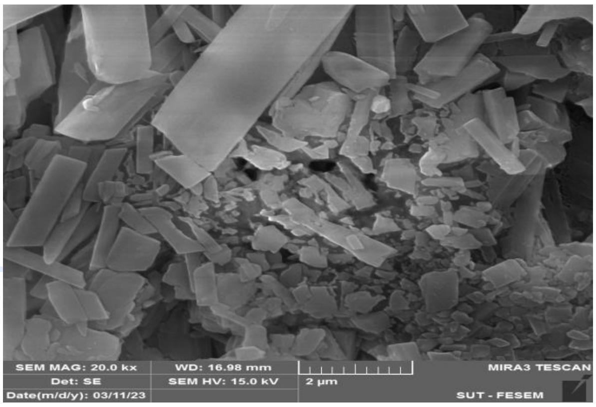

Figure (2): Electronic scanning of raw Moringa seed husks

3.1.3. Characterization of BET:

BET theory serves as the basis for an important analysis technique to measure the surface area of a sample and aims to explain the physical absorption of gas atoms on a solid surface. It has been used to determine the surface area and evaluate the porosity of Moringa seed husks. We note that the value of the surface volume is 1.507 cm3/g and the surface area is 1 m2. /g 6.56, respectively, which are moderate and somewhat acceptable values[20]. As shown in Table (1).

Table (1) Pore size and surface area of Moringa seed hulls

Examination	surface size(cm^3/g)	surface area(m^2/g)	Pore size(cm^3/g)	Pore diameter rate(nm)
Moringa	1.507	6.56	0.01091	11.664

3.2. Batch adsorption studies

3.2.1. Effect of the amount of adsorbent

The amount of adsorbent used varied from 0.01 g to 0.2 g. It was noted that the removal of methylene blue increased with increasing quantity, reaching a maximum capacity of 0.1 g of the amount of adsorbent (Figure 3). It is clear that a higher amount of adsorbent material leads to a higher surface area and provides a greater number of active sites[21]. It was noted that after a dose of 0.1 g there was no change that may be due to the overlap of the active sites at higher doses, so 0.1 g was taken. As an ideal weight.

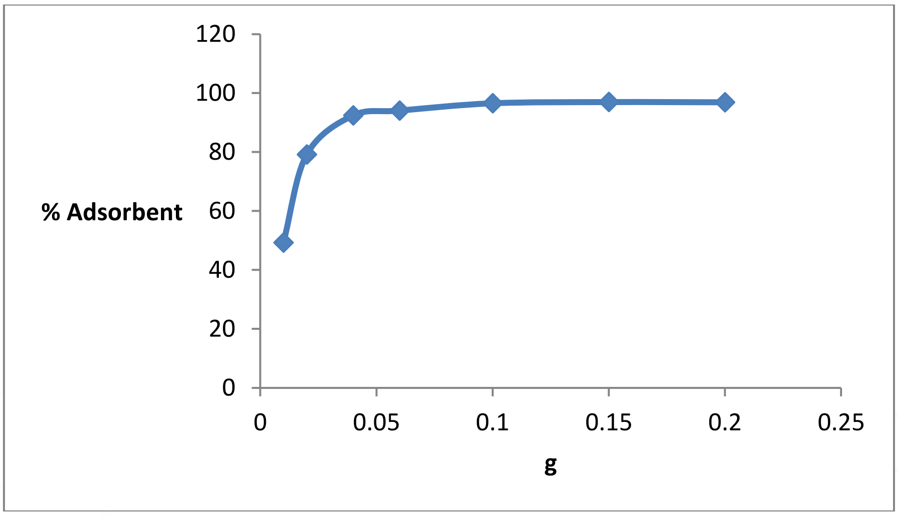

Figure 3: Effect of the amount of Moringa seed peel powder on removing methylene blue

3.2.2. Effect of contact time

The effect of different times on the adsorption capacity of methylene blue dye on the surface of Moringa seed peel powder was studied, ranging from (5-180 min). It can be seen from Figure 4 that at first the rate of removal of the methylene blue dye is very fast, which is due to the availability of many empty active sites at the beginning of the process. As the adsorption process continues, the speed begins to gradually slow down as a result of the decrease in the number of unoccupied active sites until reaching an almost constant value. At about 60 minutes, which indicates reaching a state of equilibrium. The equilibration time was determined as 60 minutes and was used in subsequent studies.

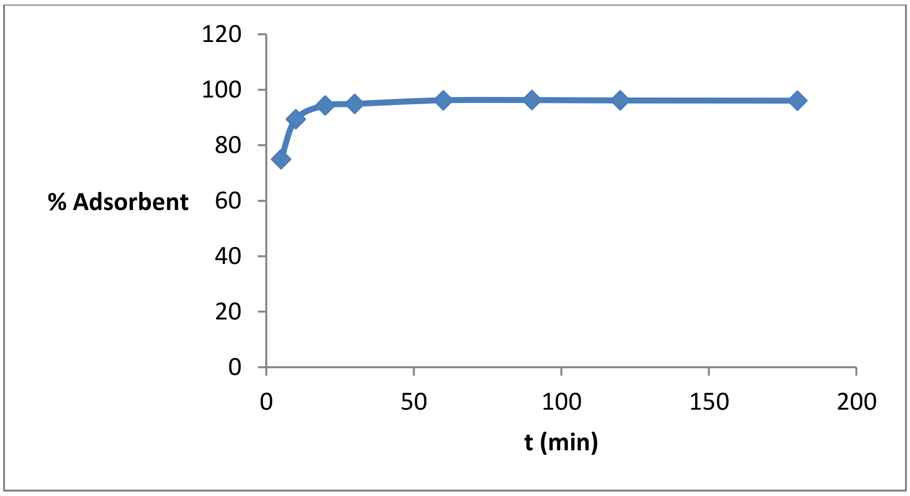

Figure 4: Effect of contact time on the removal of methylene blue from the surface of Moringa seed peel powder

3.2.3. The acid function pH:

This study was conducted in a range of pH ranging from 3 to 10. Figure 5 shows the effect of pH on the removal of methylene blue dye from the surface of Moringa seed peel powder. It was found that the process of adsorption of methylene blue dye increases gradually with increasing pH, so the efficiency may decrease. Adsorption under acidic conditions. The reason is that hydrogen ions make the surface of the adsorbent material a positively charged surface, while under basic conditions the maximum adsorption rate will appear at pH 8 and 9 due to the negative surface charge of the adsorbent molecules[22].

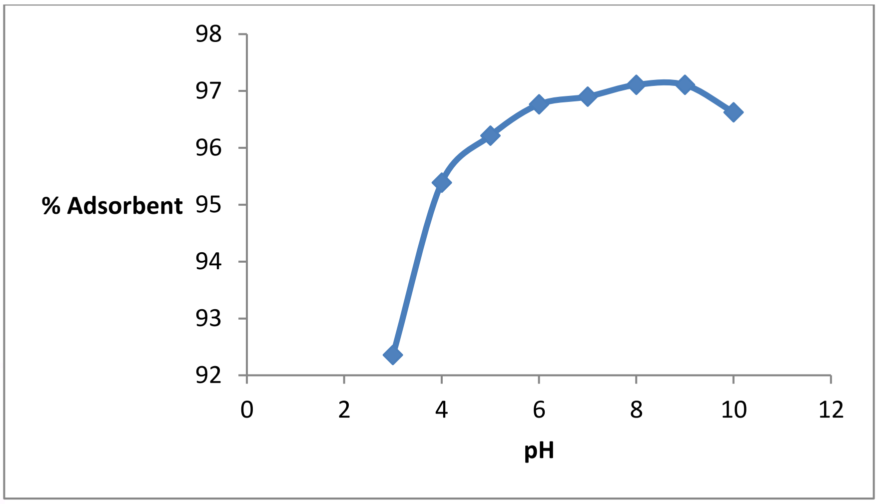

Figure 5: The effect of acid on the removal of methylene blue from the surface of Moringa seed peel powder

3.2.4. The effect of the initial concentration of the dye

The effect of the initial concentration of methylene blue dye on the adsorption process was studied, using different concentrations that ranged between (20-100 mg/L), and using a specific amount of Moringa seed peel powder (0.1 g). It was observed in Figure 6 that the highest adsorption efficiency, about 96%, was obtained at the low concentration of 20 mg/L. As the initial concentration of methylene blue dye increased, the efficiency began to decrease. The reason for the decrease in the percentage is due to a decrease in the number of unoccupied active sites necessary for adsorption. Methylene blue molecules, as the concentration of the dye increases, the more dye molecules are in the solution and thus the adsorption efficiency decreases[23].

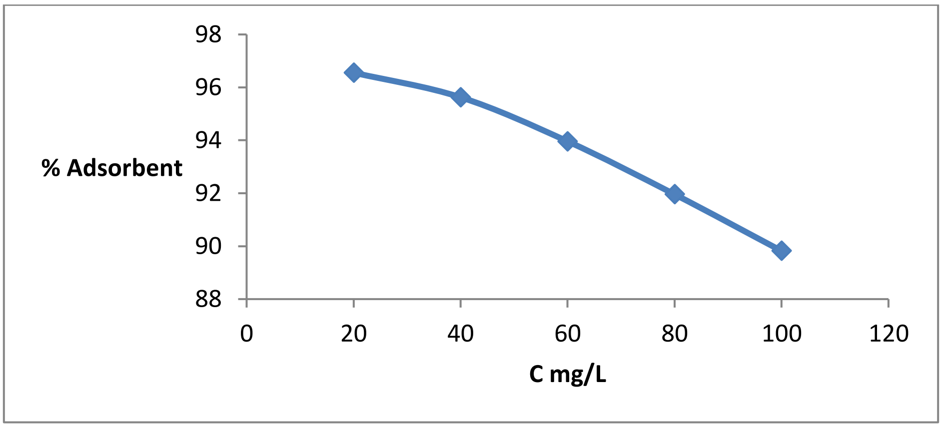

Figure 6: Effect of the initial concentration of removing methylene blue on the surface of Moringa seed peel powder

3.2.5. Effect of temperature

Temperature is one of the most important factors that affect the adsorption process. The results of this effect showed that the effectiveness of adsorption decreases with increasing temperature, as shown in (Figure 7). This behavior indicates the weakness of the physical bonds between the methylene blue dye molecules and the active sites of raw moringa. The adsorption capacity increased to a maximum capacity of 96% at temperature. 20 °C, and after this temperature the removal percentage gradually decreased from 96% to 95%. Adsorption is preferred at a low temperature[5], so the best results were achieved at 20 degrees Celsius.

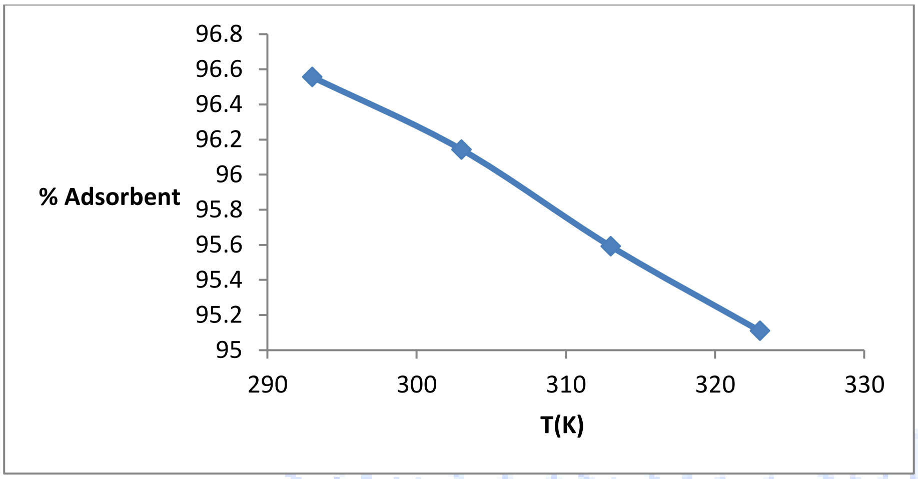

Figure 7: Effect of temperature to remove methylene blue on the surface of Moringa seed peel powder

3.3. Study of adsorption isotherms

Adsorption equilibrium data of methylene blue dye on the surface of Moringa seed peel powder were evaluated using the Langmuir and Freundlich models.

Langmuir assumed that adsorption is monolayer on a homogeneous surface and without interference between the molecules of the adsorbent, while Freundlich isotherm assumed that adsorption is multilayer on a heterogeneous surface.

The Langmuir and Freundlich isotherm equations can be expressed in the following formulas[24]:

$$q_e = \frac{q_m K_L C_e}{1+K_L C_e} \quad (1)$$

$$q_e = K_F C_e^{1/n} \quad (2)$$

Where C_e represents the equilibrium concentration (mg/L), and q_m represents the amount of adsorption at equilibrium (mg/g). q_e represents the adsorption capacity (mg/g), n represents the Freundlich intensity constant, and K_L and K_F are the Langmuir and Freundlich constants.

Figures (10 and 9) show the Langmuir and Freundlich diagrams. It was noted from the figures that the adsorption temperature of methylene blue on the peels of raw Moringa seeds was well described by the Langmuir equation. The parameters of the Langmuir and Freundlich isotherms are mentioned in Table (2). By comparing the values of the correlation coefficients R^2, it was found that the Langmuir equation gives a better fit to the experimental equilibrium data than the Freundlich isotherm. Which indicates that the adsorption is monolayer on the homogeneous surface, The dimensionless separation factor can also be used to express the Langmuir isotherm to predict whether the adsorption process is favorable or unfavorable. This can be evaluated through the relationship in Equation (3)[25]:

$$R_L = \frac{1}{1+K_L C_o} \quad (3)$$

Where C_o represents the initial concentration, and K_L represents Langmuir constant. The value of the dimensionless separation coefficient R_L was less than one, which means that the adsorption process favored taking place.

Table (2) Langmuir and Freundlich isotherm parameters for the removal of methylene blue on Moringa seed peel powder

Langmuir				Freundlich		
$q_e cal$	K_L	R_L	R^2	K_F	n	R^2
19.53125	5.33333	0.009288	0.8828	12.52717	7.092199	0.8451

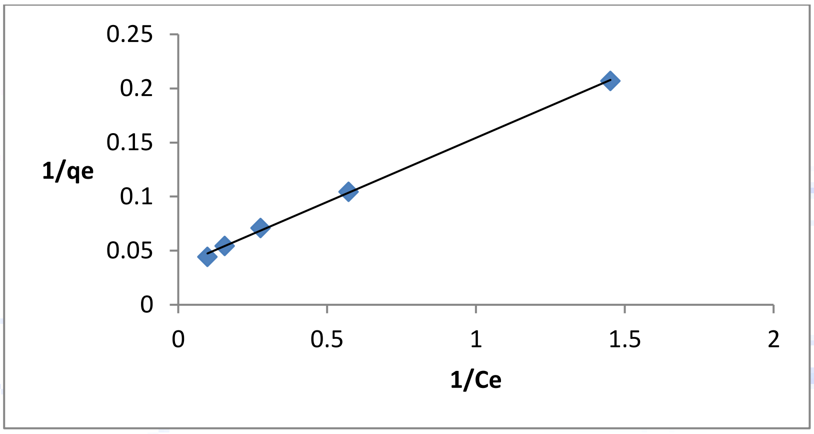

Figure 8: Langmuir isotherm for removing methylene blue on Moringa seed peel powder

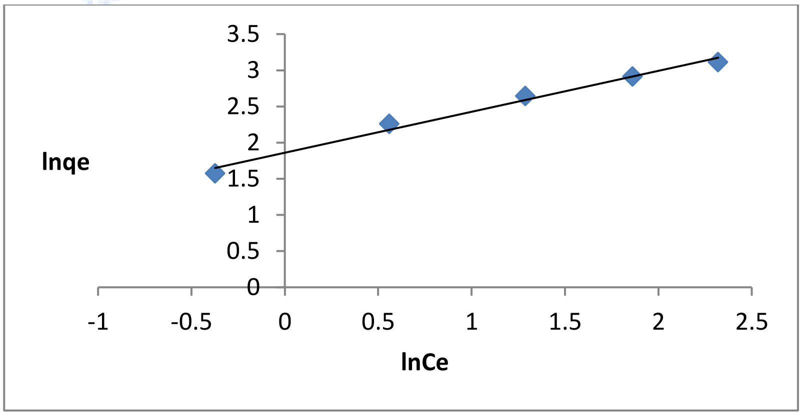

Figure 9: Freundlich isotherm for removing methylene blue on Moringa seed peel powder

3.4. Study of adsorption kinetics

The kinetics of adsorption of methylene blue dye on the surface of raw moringa is required to test the optimal operating conditions for the adsorption process on a large scale. However, the adsorption kinetics helps predict the rate of adsorption and also provides important information for designing adsorption processes. In this study, four kinetic models were used, which is the first-order model. The pseudo-second order model, the Elovich model, and intraparticle diffusion to describe the adsorption process, as in the following equations:

The linear equation of the pseudo-first-order kinetic model can be written as follows:

$\ln(q_e - q_t) = ln q_e - K_1 t$ (4)

Where $q_t$and $q_e$are the amount of dye adsorbed at equilibrium and at time t (mg/L) and $K_1$is the adsorption rate constant, so $q_e$and K_1 are calculated from the slope and intercept of the line graph between ln$(q_e$ - $q_t)$ and t.

The pseudo second-order adsorption kinetics can be expressed as follows[26],[27]:

$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e}$ (5)

where q_e and q_t are the amount of dye adsorbed and at time t (mg/L) respectively, K_2 is the rate constant of adsorption, and q_e and K_2 are calculated from the slope and intercept of the linear plot between t/ q_t versus t.

The Elovich equation can be written in the following form:

$q_t = \frac{1}{\beta} ln\alpha\beta + \frac{1}{\beta} ln t$ (6)

Where α represents the initial adsorption rate (mg/g.min), and β represents the adsorption constant (g/mg), it is calculated from the slope and intercept of the line plot between q_e versus ln t.

The intraparticle diffusion model is also expressed by the following equation[28],[29]:

$q_t = k_i t^{0.5} + C$ (7)

Where C represents the intersection (mg/g), K_{int} represents the diffusion rate constant within the particles (mg.g^{-1}.min$^{-1/2}$), and K_{int} can be calculated from the slope of the straight line resulting from plotting the relationship between q_e versus the square root of time t $^{1/2}$.

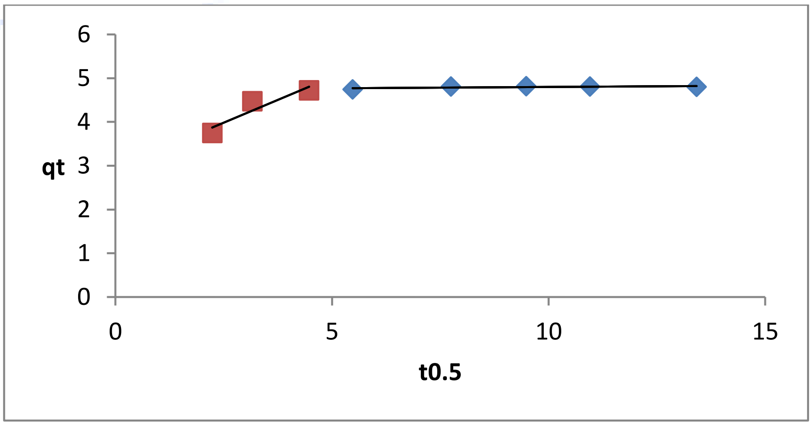

Figure 10: Intra-particle diffusion model for the removal of methylene blue on the surface of Moringa seed peel powder.

Table (3): Values of kinetic parameters for the removal of methylene blue on the surface of Moringa seed peel powder

Kinetic model	Parameter	Value
Pseudo-first order	$q_e\ exp. (mg/g\ )$	4.810606
	$q_e\ cal. (mg/g\ )$	1.281076
	k_1 (min^{-1})	-0.1094
	R^2	0.9047
Pseudo-second order	$q_e cal.\ (mg/g\ )$	4.833253
	k_2(g/mg.min)	0.291406
	R^2	1
Elovich	α (mg/g.min)	1735468
	β (g/mg)	4.206984
	R^2	0.6593
Intraparticale diffusion	$k_{int(1)}$ (mg/g.min)	0.1466
	)mg/g.min ($k_{int(2)}$	0.0061
	$R^2\ _{(1\ )}$	0.8671
	$R^2{}_{(2\ )}$	0.4179

It was shown from the table above that the false second-order model gave a higher applicability compared to other kinetic models, and this was reached through the correlation coefficient (R2), which has a value of (1 = R2), where we note that the value of the theoretically calculated adsorption capacity ($q_e$cal) is very close to the experimental adsorption capacity ($q_e$exp). This confirms that the adsorption process of methylene blue dye on the surface of raw mornga is subject to the false second-order model, which indicates the presence of chemical interactions of valence electron strengths between the molecules of methylene blue dye and raw mornga, as indicated by the value of the correlation coefficient. (R2) of the ELovich model indicates that it is insufficient to describe the relationship between the methylene blue dye molecules and the adsorbent surface (raw moringa). However, the intraparticle diffusion model usually includes three steps. The first step represents a rapid diffusion of the methylene blue dye on the outer surface of raw moringa. In the second step, the diffusion of methylene blue molecules inside the particles is the rate-determining step, and in the third step (the last stage is the equilibrium stage), in which the diffusion inside the particles is slower due to the lower concentration of methylene blue in the aqueous solution[30],[31]. It was observed from the intra-particle diffusion diagram presented in Figure (10) that there is a non-linear line that does not pass through the origin, meaning that the intra-particle diffusion process includes only two steps. Where the first step is missing, and this indicates the speed of adsorption of the external surfactant, it may make reaching the second stage quickly.

Study of thermodynamic functions

The values of the thermodynamic variables, including Gibbs free energy ΔG°, enthalpy ΔH°, and entropy ΔS°, were determined by using the following equations[32]:

$$\Delta G° = -RT lnK \quad (8)$$

$$lnK_e = \frac{\Delta S°}{R} - \frac{\Delta H°}{RT} \quad (9)$$

Where R represents the gas constant (8.314 $J.K^{-1}.mol^{-1}$), T represents the absolute temperature (K), and K_e represents the thermodynamic equilibrium constant. The thermodynamic functions were calculated from the slope and intercept of the linear graph resulting from the relationship between lnK versus 1/T, and the values of the thermodynamic functions were mentioned in Table (4).

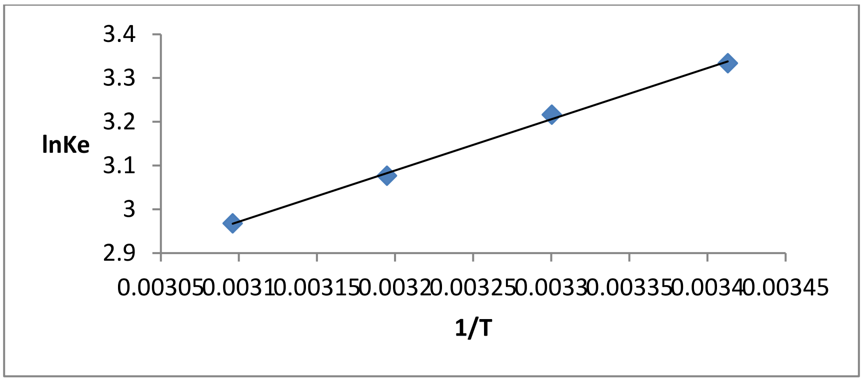

Figure 11: Van't Hoff diagram for the adsorption of methylene blue on the surface of Moringa seed husk powder

Table (4): Thermodynamic parameters for removing methylene blue from the surface of Moringa seed peel powder

ΔS°	ΔH°	ΔG°				R^2
		298 K	303 K	313 K	323 K	
-5.4473328	-9727.38	-8120.734371	-8101.597433	-8006.534862	-7970.019044	0.998

Table (4) shows that the values of ΔG° are negative at all temperatures, which indicates the spontaneous adsorption of methylene blue on the surface of raw Moringa seed peel powder, while the negative value of ΔS° indicates a decrease in randomness as a result of the adhesion of methylene blue molecules to the active sites. The active substance present on the surface of the raw moringa, while the negative value of ΔH° indicates that the adsorption is heat-emitting.

4. Conclusions

In this study, Moringa seed peel powder was used to remove methylene blue dye from aqueous solution. Methylene blue was efficiently adsorbed from the solution through the bioadsorbent. The adsorption of methylene blue was studied as a function of time, temperature, pH, adsorbent dose, and initial concentration of methylene blue. The equilibrium time for removing methylene blue was achieved within 60 min. It was also found that the maximum removal of methylene blue occurs at a pH between 9.8. The optimal dose of raw moringa was chosen as 0.1 g in all experiments. The adsorption isotherm data were well explained by the Langmuir adsorption isotherm while the adsorption kinetics data were better explained by the pseudo-second order kinetic model. The thermodynamic variables were calculated as it was found that the adsorption process is spontaneous and heat-emitting. Moringa seed husk powder played an effective role in removing the selected dye.

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