Adsorption of malachite green from waste water using prosopis juliflora bark carbon

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ABSTRACT

Adsorption behavior of malachite green from waste water using prosopis juliflora bark carbon was investigated. The effect of contact time, adsorbent dose, solution pH, initial concentration of malachite green and temperature are investigated. Freundlich and Langmuir isotherms are used to determine the adsorption capacity of prosopis juliflora bark carbon as an adsorbent. The entropy change (ΔS^0) values and enthalpy change (ΔH^0) values are calculated from the slope and intercept of the linear plot of $\ln K_0$ versus $1/T$. The free energy change (ΔG^0) is calculated from the equilibrium constant (K_0) value. The equilibrium constant (K_0) for the adsorption process is determined from the slope of the plot of $\ln(q_e/c_e)$ against c_e. The experimental values of entropy, enthalpy and free energy indicate that the adsorption of malachite green is a physical adsorption. The kinetic studies of malachite green follows pseudo second order and intra-particle diffusion model.

Keywords: Adsorption; kinetics; malachite green; PJBC; thermodynamic.

INTRODUCTION

Rapid growth of population, modern agriculture, development of new industries and domestic wastes are responsible for discharge of waste water. Generally, waste water is directed to the nearest water sources such as rivers, lakes and seas. Colour is an important quality of water from an aesthetic point. Dyes present in water can decrease the quality of water. The textile and printing industries discharge large amount of dyes and pigments to the water bodies. Colour inhibits the light penetration and retards the photosynthetic activities that prevent the growth of biotic systems. The concentration of dyes even at 1.0 mg/L is unfit for human consumption. Many dyes reaching the water sources are difficult to decompose and may cause carcinogenicity (Ramakrishna & Viraraghvan, 1997). Most of the dyes are stable to photo degradation, bio-degradation

and oxidizing agents. Consequently, it is important to remove these dyes from waste water before its final disposal to water bodies. Currently, several physical or chemical processes are used to treat the waste water. However, these processes are costly and cannot be used effectively to treat the waste water (Malik *et al*., 2007). Activated carbons are widely used as efficient and useful adsorbents for the removal of colour and odour in waste water. This paper reports the adsorption of malachite green dye from waste water using prosopis juliflora bark carbon (PJBC) as an adsorbent. This PJBC adsorbent is also used to remove Victoria blue from aqueous solution (Tamilarasan & Kumar, 2014). The adsorption behavior of the malachite green dyes as a function of contact time, PJBC adsorbent dose, initial solution pH, initial MG concentration and temperature are studied. Adsorption data is fitted to Freundlich and Langmuir isotherms. The thermodynamic parameters such as ΔG^0, ΔH^0, ΔS^0 are calculated using Van't Hoff equation. The batch adsorption experimental values such as q_e, q_t, C_e and C_0 are used fitted with pseudo second order and intra-particle diffusion kinetic model.

MATERIALS AND METHODS

Adsorbate

Malachite Green (MG) dye used in this study was purchased from Sigma-Aldrich company. Its molecular formula is $C_{50}H_{52}N_4O_8$. The λ_{max} for this MG dye is 618 nm. The dye stock solution was prepared by dissolving accurate weight of 1 g/L dye in distilled water. The batch method experimental solutions of desired concentration was obtained by diluting the dye stock solution.

Preparation of prosopis juliflora bark carbon adsorbent

The raw prosopis juliflora bark carbon was purchased from local market in India. The raw PJBC was finely powdered with grinding machine. Then PJBC powder was treated with concentrated sulphuric acid for 2 hours, filtered and then washed with distilled water. The residue was dried and placed in a muffle furnace for about 24 hours at 800ºC, then the powder sample was used for batch experiments.

Effect of contact time

The effect of contact time between adsorbent PJBC and adsorbate (MG**)** was determined by keeping initial MG concentration, adsorbent dosage, initial solution pH and temperature as kept constant.

Effect of PJBC adsorbent dose

To study the effect of PJBC dose on the MG adsorption, different amounts of PJBC powder (5-500 mg) were added into different 250 ml stopper glass flasks containing a definite volume (50ml in each-flask) of (50mg/L) dyes solution without changing the solution pH at 30ºC. The flasks containing experimental solutions were placed in

a thermostatic water bath shaker for 60 minutes, then the residual MG concentrations were measured by using spectrophotometer.

Effect of solution pH

The effect of solution pH on the adsorption of MG was studied by mixing 25 mg of PJBC and 50 mg/50 ml of MG dye solution with different pH values (2.0 -11.0) at 30°C. The pH of solutions was adjusted by using 0.1N NaOH or 0.1N HCl solution. The flask containing the experimental solutions was shaken for 60 minutes.

Effect of temperature

The effect of temperature was studied at four different temperatures such as 30°C, 40°C, 50°C and 60°C in a thermostatic water bath shaker and all other variable parameters were kept constant.

Effect of Initial MG dye Concentration

Effect of initial dye concentration was studied by using different concentrations (such as 50, 100, 150, 200 and 250 mg/L) of MG dyes solution with 25 mg of PJBC absorbent and keeping all other variable parameter as constant.

Batch method

The batch adsorption (Hameed, 2009a) experiments were carried out by adding a fixed amount of adsorbent (25 mg/50ml) into a number of 250 ml stopper glass flasks containing a definite volume (50 ml in each case) of different initial concentrations (50, 100, 150, 200 and 250 mg/L) of dye solutions without changing pH of the solution and at temperatures 30, 40, 50 and 60°C. The flasks containing experimental solution were placed in thermostatic water bath shaker for about 60 minutes. The residual MG dye concentrations were measured. The amount of adsorption q_t (mg/g) at time "t", amount of adsorption at equilibrium q_e (mg/g) and percentage removal of MG dye were calculated using the following relations

$$q_t = \frac{(C_O - C_t)V}{W} \tag{1}$$

$$q_e = \frac{(C_O - C_e)V}{W} \tag{2}$$

$$\% \text{ Removal} = \frac{C_o - C_t}{C_o} X 100 \tag{3}$$

Where C_o, C_t (mg/L) and C_e (mg/L) are the initial, at time "t" and at equilibrium of the liquid phase concentrations of MG dye respectively. V (L) is the volume of the solution and W (g) is the mass of dry adsorbent used.

ADSORPTION ISOTHERM

The adsorption isotherms showed how the adsorbate molecules were distributed between the adsorbent and solution. The Freundlich and Langmuir isotherms were used to measure the adsorption capacity of the PJBC absorbent for the removal of MG dyes.

Freundlich isotherm

The linear form of Freundlich isotherm (Freundlich, 1906) is represented by the equation

$$\log q_e = \log K_f + \frac{1}{n} \log C_e \tag{4}$$

Where q_e is the amount of dyes adsorbed per unit weight of the adsorbent, (mg/L), K_f is [mg/g (mg/L)$^{-1/n}$] the adsorption capacity and 1/n is the adsorption intensity. In general as K_f value increases, the adsorption capacity for the chosen adsorbent increases. The values of "n" in the range of zero to one indicate that the adsorption is favorable (Hameed, 2009b).

Langmuir isotherm

The Langmuir (Langmuir, 1918) isotherm equation can be described by

$$C_e/q_e = \frac{1}{Q_m b} + \frac{C_e}{Q_m} \tag{5}$$

Where C_e (mg/L) is the concentration of the MG at equilibrium, q_e (mg/g) is the amount of adsorbate (MG) per unit mass of adsorbent, Q_m is the Langmuir adsorption capacity of an adsorbent and b (L/mg) is the energy of adsorption (or rate of adsorption). The linear plot of specific adsorption capacity (c_e/q_e) against the equilibrium concentration (C_e) shows the fitness of the Langmuir model. The Langmuir constant Q_m and b were determined from the slope and intercept of the plot. The equilibrium parameter R_L (McKay *et al.*, 1982) was used to find out the feasibility of the Langmuir isotherm

$$R_L = \frac{1}{1 + bC_0} \tag{6}$$

Where C_o (mg/L) is the initial concentration of adsorbate and b (L/mg) is Langmuir isotherm constant. The R_L values between 0 to 1 indicate that the adsorption is favorable.

THERMODYNAMIC TREATMENT OF THE ADSORPTION PROCESS

Thermodynamic parameters such as ΔG^0, ΔH^0, ΔS^0 were calculated using the relation

$$\Delta G^0 = -RT \ln K_0 \tag{7}$$

$$\ln K_0 = -\frac{\Delta H^0}{RT} + \frac{\Delta S^0}{R} \tag{8}$$

Where ΔG^0 is the free energy change of adsorption (KJ/mol), T is the temperature in Kelvin, R is the gas constant (8.314 Jmol^{-1}K^{-1}), ΔH^0 is the standard heat change of adsorption (KJ/mol) and ΔS^0 is standard entropy change (J/mol/K). The equilibrium constant (K_0) for the adsorption process was determined from the plot of $\ln(q_e/c_e)$ against c_e and the plot was extrapolated to zero c_e value (Khan & Singh, 1987). The value of ΔH^0 and ΔS^0 can be calculated from the slope and intercept of plot of $\ln K_0$ against 1/T.

ADSORPTION KINETICS

The equilibrium parameters such as q_e, q_t, C_e, C_0 and time were used to fit with pseudo second order and intra-particle diffusion kinetic model.

Pseudo second order

Pseudo second order adsorption kinetic rate equation (Ho & McKay, 1998; 2000) is

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} t \tag{9}$$

Where k_2 is the rate constant of pseudo second order (g mg^{-1}min^{-1}). The q_t (mg/g) is the amount of dye adsorbed at time "t" and q_e(mg/g) is the amount of dye adsorbed at equilibrium. The linear fit method was used to calculate the slope, intercept and correlation coefficient value. The q_e values were calculated from slope and k_2 values were calculated from intercept.

Intra-particle diffusion model

The intra-particle diffusion equation (Weber & Morris, 1964) is

$$q_t = k_{id} \, t^{1/2} + C \tag{10}$$

Where k_{id} is the intra-particle diffusion rate constant (mg g^{-1}min$^{-1/2}$) and C is constant. The linear fit method of q_t against $t^{1/2}$ was used to calculate intraparticle diffusion rate constant (k_{id}) and correlation coefficient (R^2) values. The fitness of the model was explained on the basis of high correlation coefficient (R^2) values. The high correlation coefficient (R^2) value indicates that the model successfully describes the kinetics of MG dye adsorption. If the intra-particle diffusion was rate determining step, the plot of q_t versus square root of contact time ($t^{1/2}$) should yield a straight line passing through the origin, otherwise some other process accompanied along with intra-particle diffusion model.

RESULTS AND DISCUSSION

Effect of PJBC adsorbent dose

The effect of PJBC adsorbent dose was studied by varying the adsorbent dose from 5–500 mg/50 ml and the results are shown in Figure 1. From the Figure 1, it can be seen that the % removal of MG dye increases with increase in adsorbent dose, which was due to increase in surface area of the adsorbent. The % removal of MG dye suddenly increases in the range of 25 - 50 mg/50ml and then reaches to a limiting value. However 91% of MG dye removal takes place using 25-50 mg/50ml of adsorbent dose. So the optimum adsorbent dose of 25 mg/50 ml was used for batch experiments.
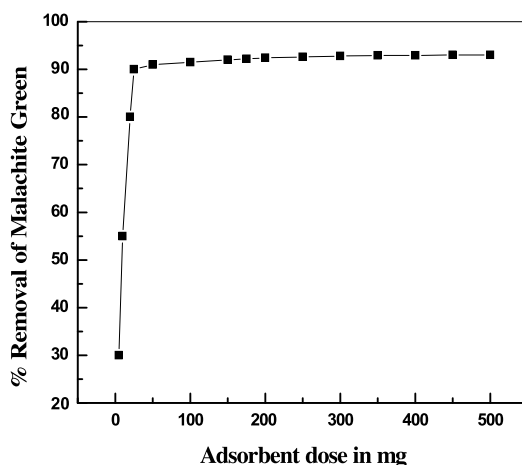
Fig. 1. Effect of adsorbent dose for the removal of MG dye

[MG] = 50 mg/L; pH = 6.5; Temp = 30°C; Contact time = 60 minutes

Effect of contact time

The effect of contact time determined by using initial concentration of MG is 50 mg/L, adsorbent dose is 25mg/50ml and pH is 6.5 at 30°C with different time interval such as 10, 20, 30, 40, 50, 60, 70, 80 and 90 minutes as shown in Figure 2. The % removal of MG increases initially and reaches almost constant value. So the optimum contact time for the MG adsorption was taken as 60 minutes. Similar experiments were also repeated by using different MG concentrations (100, 150, 200 and 250 mg/L) at different temperatures (30, 40, 50 and 60°C). The results of contact time studies show that 60 minutes was sufficient for batch adsorption experiments.
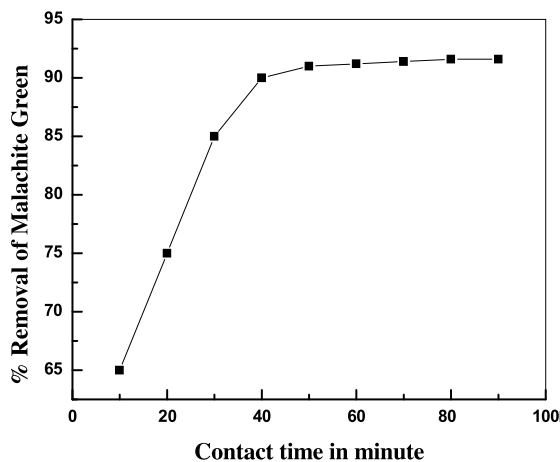
Fig. 2. Effect of contact time for the removal of MG dye

[MG] = 50mg/L: pH = 6.5; Temp = 30°C; Adsorbent dose = 25mg/50ml.

Effect of solution pH

The pH of solution was one of the most important factors that controls the adsorption of MG on the PJBC adsorbent material. The adsorption capacity can be attributed to the chemical form of MG dye in the solution and adsorbent surface at a specific pH. Therefore, an increase in solution pH may cause an increase or decrease in the adsorption capacity of the adsorbent. To study the effect of solution pH, the initial solution pH was tested in varying ranges from 2.0 to 11.0 as shown in Figure 3. The % removal of MG increases from pH 2.0 to 7.0 and then decreases. This might be due to the replaceable positive ions present on the surface of the adsorbent and that may be responsible for adsorption of MG. The pH_{zpc} of the PJBC was 4. The lower pH values (pH < 6), the overall adsorbent surface and adsorbent solution interface become positive, that hold back the adsorption of positively charged MG. In the pH range of 6.0 to 7.5, the maximum amount of MG dye molecules were entered in to the adsorption sites, which was responsible for the maximum % removal of dye. On the other hand, the pH above 7 was alkaline, so the cationic dye was strongly associated to the alkaline solution, not to the adsorbent surface that decreases the % removal of dyes above the pH 7. From the experimental results, it was observed that more than 90% of MG dye removal has taken place in the range of 6.0 to 7.5. The maximum % removal of MG dye has taken place at a pH of 7.0.
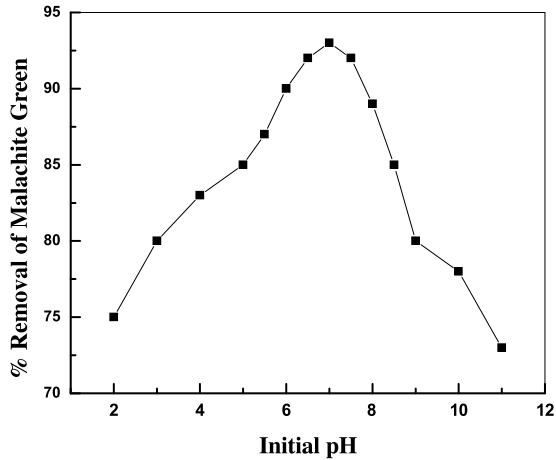
Fig. 3. Effect of initial solution pH for the removal of MG dye

[MG] = 50 mg/L; Temp = 30°C; Adsorbent dose = 25mg/50ml; Contact time = 60 minutes

Effect of Temperature

The extent of adsorption depends on the temperature of the solid-liquid interface. Thermodynamic parameters and kinetic process were depending on the structure of MG and surface functional groups of an adsorbent. The effect of temperature was studied at 30, 40, 50 and 60°C and the results were summarized in Figure 4. The results show that the % removal of MG dye in the range of 50 to 150 mg/L slightly increases with the raise in temperature. When the % removal of 200 mg/L and 250 mg/L of dyes concentration increases up to 50°C and above 50°C, then the % removal of MG decreases.
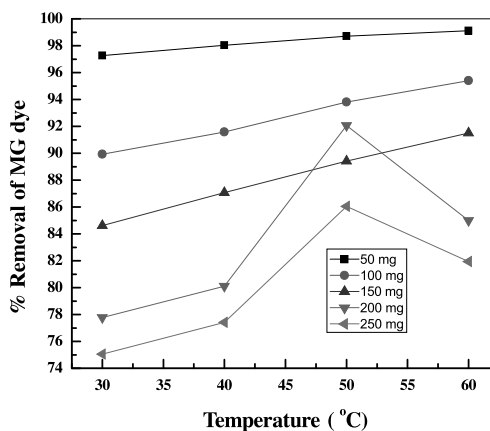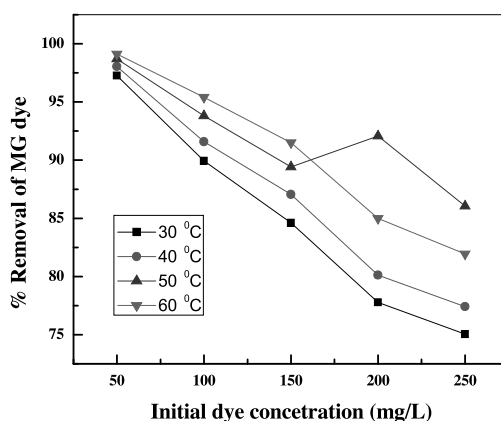

Fig. 4. Effect of temperature for the removal of MG dye

[MG] = 50-250 mg/L; pH = 7.0; Adsorbent dose = 25mg/50ml; Contact time = 60 minutes.

Effect of Initial dye Concentration

Effect of initial dye concentration was carried out with different initial concentrations such as 50, 100, 150, 200 and 250 mg/L of MG dye solutions using 25 mg/50 ml of absorbent. These results are shown in Figure 5. The % removal of MG decreases with an increase in initial concentrations of dye. The same amount of adsorbent contain fixed number of active sites. But the dye concentration increases, then the % removal of dye proportionately decreases. As the temperature increases above 50°C, the % removal of dye decreases. This also supports a physical adsorption.

Fig. 5. Effect of initial concentration of MG dye [MG] = 50-250 mg/L;

Temp = 30, 40, 50 and 60°C; Adsorbent dose = 25mg/50ml; Contact time = 60 minutes.

ADSORPTION ISOTHERM

The Freundlich and Langmuir models were used to fit with experimental data and these parameters are shown in Table 1. From Table 1, it was clear that, the Langmuir adsorption capacities (Q_m) value increases up to 50°C and then decreases with a raise in temperature. But adsorption intensities (b) value increases with an increase in temperature. The Freundlich adsorption capacities value increases with raise in temperature, this was due to multilayer adsorption. The adsorption intensity can also be expressed in terms of n values. The n values between 1 to 10 indicate the adsorption was favorable. The n values calculated from Freundlich isotherm was also lying in between 1 to 10. This indicates that the Freundlich adsorption was a favorable process. Adsorption capacities of these two isotherms indicate that the adsorption was a physical adsorption. The R_L values at different temperature were calculated to find the feasibility of Langmuir isotherm and shown in Table 2. The R_L values between 0 and 1 also support the Langmuir isotherm for MG adsorption.

Table 1. Langmuir and Freundlich parameters for adsorption of MG.

Temp (°C)	Langmuir		Fruendlich	
	Q_m	b	K_f	n
30	410.26	0.0971	84.4112	2.8687
40	418.15	0.1181	95.7856	2.9901
50	491.17	0.1387	109.8753	2.6895
60	429.87	0.2030	124.7383	3.2973

Table 2. Equilibrium parameter (R_L)

C_0 mg/L	Temperature			
	30°C	40°C	50°C	60°C
50	0.1708	0.1448	0.1260	0.0897
100	0.0934	0.0781	0.0672	0.0470
150	0.0642	0.0534	0.0458	0.0318
200	0.0490	0.0406	0.0348	0.0240
250	0.0396	0.0328	0.0280	0.0193

THERMODYNAMIC TREATMENT OF THE ADSORPTION PROCESS

Thermodynamic parameters such as ΔG^0, ΔH^0, ΔS^0 are shown in Table 3. The ΔG^0 values calculated from equilibrium constant was a negative. This negative value of ΔG^0 indicate that the adsorption of MG using PJBC adsorbent was spontaneous process. The negative values of ΔH^0 indicate that the adsorption was exothermic. The magnitude of ΔH^0 value suggests that the adsorption was physical adsorption (If ΔH^0 < 40 KJ/mol is physical adsorption). The positive ΔS^0 shows increased randomness at the PJBC adsorbent surface. In this condition the adsorbed water molecules, which were displaced by the MG species, gain more translational entropy than lost by MG molecules, thus allowing the predominance of randomness in the system. This was also further supported by the positive values of ΔS^0, which suggest that the freedom of MG was not too restricted in the adsorbent surface and this confirms the adsorption was physical adsorption. However, the value of ΔS^0 was found to be decreased as the initial concentration of MG increases due to the decrease in the randomness of MG. The ΔG^0 value increases with an increase in temperature due to the increase or enlargement of pore size or activation of the adsorbent surface.

Table 3. Thermodynamic parameters

C_0 mg/L	ΔG^0 (KJ/mol)				ΔH^0 (KJ/mol)	ΔS^0 (J/mol/K)
	30°C	40°C	50°C	60°C		
50	-8.99	-10.17	-11.63	-13.06	-32.40	136.35
100	-5.51	-6.21	-7.30	-8.39	-23.87	96.62
150	-4.29	-4.96	-5.73	-6.58	-22.77	85.97
200	-3.16	-3.63	-6.58	-4.80	-21.40	81.58
250	-2.78	-3.21	-4.89	-4.19	-15.48	60.50

ADSORPTION KINETICS

The adsorption kinetics of MG using PJBC was studied, the initial concentrations of MG were 50, 100, 150, 200 and 250 mg/50 ml with the time interval of 15, 30, 45 and 60 minutes. The pseudo second order rate constant (K_2) values, intra particle diffusion constant (K_{id}) and correlation coefficient (R^2) values were calculated with linear fit method and given in Table 4. From Table 4, it was clear that both the rate constant values were having high correlation coefficient (R^2) values (R^2 > 0.9800). However the q_e values calculated from the pseudo second order were nearly equal to experimentally calculated q_e values. This also further supports the pseudo second order. The intra particle diffusion model was not only having high correlation coefficient (R^2) values but also having intercept value (intercept value not given in table). This intercept value indicates that the adsorption not only depends on intra particle diffusion, but also depends on some other process. These might be boundary layer or surface adsorption.

Table 4. Kinetic parameters

C_0 mg/L	Temp (°C)	Pseudo second order			Intraparticle diffusion	
		q_e	K_2 x 10^{-3}	R^2	K_{id}	R^2
50	30	91.66	2.284	0.9938	9.9304	0.9914
	40	92.44	2.132	0.9933	9.8517	0.9849
	50	93.10	1.994	0.9936	9.6288	0.9821
	60	93.51	1.955	0.9939	9.8585	0.9853
100	30	168.66	2.776	0.9930	9.9304	0.9903
	40	171.96	2.670	0.9923	9.8517	0.9835
	50	176.40	2.474	0.9939	9.6288	0.9889
	60	179.59	2.382	0.9948	9.8585	0.9941
150	30	231.44	3.095	0.9916	14.8280	0.9864
	40	238.79	2.959	0.9928	14.5729	0.9899
	50	245.85	2.866	0.9946	14.5273	0.9920
	60	252.09	2.732	0.9630	14.1822	0.9260
200	30	231.44	3.387	0.9901	19.8937	0.9885
	40	275.65	3.309	0.9901	19.6638	0.9850
	50	345.85	1.535	0.9911	19.6425	0.9863
	60	295.16	3.125	0.9940	19.1915	0.9942
250	30	285.69	3.479	0.9895	24.2940	0.9870
	40	297.48	3.494	0.9892	24.7175	0.9854
	50	385.44	2.157	0.9876	25.8468	0.9805
	60	320.10	3.461	0.9908	25.1603	0.9891

CONCLUSION

The adsorption of MG from waste water using PJBC was investigated under different experimental conditions by batch process. The optimum solution pH for adsorption of MG was 7.0. The Langmuir adsorption capacities increase up to 50°C and then decrease. The Freundlich adsorption capacities increase up to 60°C due to multilayer adsorption. The free energy values indicate that the adsorption process was spontaneous. Enthalpy values indicate that the adsorption of MG was exothermic. The entropy values indicate the randomness of MG dye adsorption. The kinetic studies indicate that the adsorption follows pseudo second order and intraparticle diffusion models. The % removal of PJBC adsorbent was greater than 90%. So the results of kinetic studies indicate that the PJBC adsorbent can be used for the removal of malachite green from waste water.

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امتزاز أخضر الملاكيت من مياه الصرف باستخدام الكربون من لحاء شجر *"الجوليفلورا بروسوبيس"*

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خلاصة

تم دراسة سلوك امتزاز أخضر الملاكيت من مياه الصرف باستخدام الكربون من لحاء شجر *"الجوليفلورا بروسوبيس"*. تم دراسة تأثير زمن التعرض و جرعة الإمتزاز و درجة حموضة المحلول و تركيز أخضر الملاكيت و درجة الحرارة. تم استخدام المتعادلات الحرارية "لفروينليدش" و "للانجمير" لتحديد السعة الأمتزازية للحاء شجر *"الجوليفلورا بروسوبيس"*. كما تم حساب التغير في "الأنتروبيا" ($\Delta S°$) والمحتوى الحراري ($\Delta H°$) من الميل والتقاطع لعلاقة الخط المستقيم بين $\ln K°$ و $1/T$. تم حساب قيم التغير في الطاقة الحرة ($\Delta G°$) من قيمة ثابت الأتزان $K°$. تم تقدير قيمة ثابت الأتزان $K°$ لعملية الأمتزاز من الميل لعلاقة الخط المستقيم بين $\ln(q_e/c_e)$ وc_e . القيم التجريبية "للأنتروبيا" و المحتوى الحراري والطاقة الحرة تشير الى أن الأمتزاز لأخضر الملاكيت من نوع الأمتزاز الفيزيائي. كما أن الدراسة الكيناتيكية لأخضر الملاكيتهو من الدرجة الثانية ومن نموذج الأنتشار الجسيمي البيني.