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Original Article

Kinetic and thermodynamic studies of malachite green adsorption using activated carbon prepared from desert date seed shell

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ABSTRACT

The negative effect of high concentration of dyes in the aquatic environment on humans and aquatic plants prompted this research. The adsorption of hazardous malachite green (MG) from aqueous solution using activated carbon derived from desert date seed shell (DDAC) was examined. Batch equilibrium technique was employed to study the effect of contact time (5-120 min), initial concentration (20-100 mg dm^{-3}) and temperature (303.15-333.15 K) on the adsorption capacity of the prepared adsorbent. Experimental data were analyzed using five kinetic models: pseudo-first-order, pseudo-second-order, Elovich, intraparticle diffusion and Boyd models and it was found that the pseudo-second-order model fitted the adsorption data most with the highest correlation (R^2 = 0.9999). The overall adsorption process appears to be jointly controlled by intraparticle diffusion and film diffusion mechanisms. Studies of thermodynamic behavior revealed negative values for ΔG (-11.45 to -13.42 kJ mol^{-1}), and a positive value for ΔH (8.39 kJ mol^{-1}) and ΔS (0.065 kJ mol^{-1} K^{-1}). These indicated the feasibility, endothermicity and spontaneity of the removal process. The results demonstrated that the adsorbent could be exploited in the removal of MG from aqueous solution.

1. Introduction

Industrial operations are associated with the disposal of huge amount of toxic substances such as synthetic dyes that lead to detrimental effects on the environment and human health. It was reported that 7 x 10^5 tonnes of synthetic dyes are produced yearly around the world and two-thirds of the dye consumption are dominated by the textile industries [1,2]. Due to the inefficiency of the dyeing process, customarily 30% of dyes are wasted in residual liquors through washing and exhaustion operations and up to 200,000 tonnes of dyes are disposed as effluent [3]. Among all categories of commercially available synthetic dyes, basic dyes are the brightest class of soluble dyes used by the textile industry [4].

Malachite green, (also known as Basic Green 4) is a water-soluble basic dye belonging to the triphenylmethane family. It is among the most widely employed colorant in several industrial applications. It has been extensively used in dyeing of wool, leather, silk, paper, and also in distilleries. It is also broadly used as a bactericide, fungicide and parasiticide in several aquaculture industries worldwide [5]. MG also finds application as a food coloring agent, food additive, a medical disinfectant, and anthelmintic [6]. However, high concentration of MG is toxic if indiscriminately disposed into the aquatic environment, with carcinogenic, and mutagenic effects on human beings and causing suffocation of aquatic plants [7]. Hence, the removal of MG from industrial effluent before been disposed into the water system is a management serious concern [8].

Numerous studies have reported the removal of MG from aqueous solution using various adsorbents such as pristine lignin [9], malt bagasse [10], kaolin [11], magnetic activated carbon [12] and coconut shell carbon [13]. However, the quest to enhance the adsorption efficiency are continuously been sought through new adsorbents with excellent characters and low-cost. Among the most

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promising adsorbent materials are those originating from plant biomass [14]. Desert date is one of the abundant forest-based resources available in Nigeria. Its fruits, kernel oil and leaves have been a basis of an active trade especially among rural folks for many years. Although, different parts of the plant are been utilized for different purposes, the wooden shell of the seed is traditionally discarded and thus constitutes a waste. A possible solution to its disposal problem is converting it into activated carbon, which has been widely employed over the years in the reclamation of pollutant-bearing wastewaters. To the best of our knowledge, no report has been documented on the removal of malachite green using desert date seed shell. Hence, the aim of this research is to probe the removal of MG from aqueous solution using activated carbon derived from desert date seed shell. .

2. Experimental

2.1. Chemicals

Malachite green dye was obtained from E. Merck (Mumbai, India). All other reagents used were of analytical grade. Distilled water was used throughout for the preparation of stock and experimental solutions. A stock solution of 1000 mg dm^{-3} was prepared by dissolving accurately weighed quantity of dye powder into 1000 cm^{-3} of distilled water in a standard volumetric flask. Experimental solutions were prepared by appropriate dilution of the stock solution. The pH of the aqueous solutions was adjusted using 0.1 mol dm^{-3} NaOH or HCl depending on the requirements.

2.2. Preparation of Activated Carbon

Activated carbon was prepared from desert date seed shells (DDSS) using a two-step chemical activation technique, as described by [15], but with modification in the activating agent used (NaOH instead of KOH). The dried DDSS were first carbonized at 700^{o}C in a muffle furnace (SXL-1008) for 90 minutes. The carbonized sample was then mixed with solution of the activating agent such that the mass ratio of NaOH to charcoal was 2:1. This was followed by further thermal treatment at 750^{o}C for 90 minutes. The sample was cooled, leached with 0.1 mol dm^{-3} HCl and then thoroughly washed with distilled water until a neutral pH (≈ 6.9) was attained. The desired activated carbon was obtained after drying in an oven at 110°C for 24 h.

2.3. Adsorption Experiment

Batch mode experiments were executed by agitating Erlenmeyer flasks in a temperature controlled incubator shaker (Innova 4000; New Brunswick Scientific) at constant agitation speed (150 rpm). The effects of solution temperature, contact time and initial concentration were studied. For the effect of contact time and initial concentration, a fixed amount of the adsorbent (0.1 g) was added into Erlenmeyer flask containing 50 cm^3 of dye solution (20-100 mg dm^{-3}). The pH of all the dye solutions was adjusted to predetermined optimum value of 8.0. The contents were then agitated for pre-defined time intervals (5-120 min) under room temperature (30 ± 2 °C). For the effect of solution temperature, 0.3 g of the adsorbent was added into 50 cm^3 of dye solution (100 mg dm^{-3}). The adsorption experiment was performed at different temperatures of 303.15, 313.15, 323.15 and 333.15 K. After each adsorption experiment, residual concentrations were analyzed using UV-vis spectrophotometer (Labda 35; Perkin Elmer) at the predetermined λ_{max} of 617 nm. A standard calibration curve was used to obtain the equilibrium dye concentration. All of the experiments were conducted in triplicate, and only the averaged values were reported. The maximum variation for the triplicates is usually less than 3%. The extent of dye removal from aqueous solution was calculated using the following equations:

$$qt = \left(\frac{Co - Ct}{m}\right)V \qquad (1)$$

$$qe = \left(\frac{Co - Ce}{m}\right)V \qquad (2)$$

where q_e and qt are the adsorption capacity (mg g^{-1}) at equilibrium and time t respectively, C_o is the initial dye concentration (mg dm^{-3}), C_e and C_t are the concentration (mg dm^{-3}) of dye at equilibrium and time t, V is the volume of dye solution (dm^{-3}) and m is the mass of the adsorbent (g).

2.4. Scanning Electron Microscopy (SEM)

The morphological features of DDAC before and after adsorption were analyzed using SEM analysis (PRO: X: Phenonm World 800-07334). The samples were placed on brass stubs using adhesive tape on both sides. SEM images were taken at an accelerating voltage of 15.00 kV and at 1000 times magnification.

3. Results and Discussion

3.1. Effect of Contact Time and Initial Concentration

Fig. 1 shows the dye uptake with respect to time at different initial concentrations (20-100 mg dm^{-3}) by DDAC. It is obvious that the amount of adsorbate adsorbed increased with the increase in initial dye concentration. As the initial concentration increased from 20 to 100 mg dm^{-3}, the adsorption uptake for MG was found to increase from 10 to 49.99 mg g^{-1}. This was ascribed to the increase in driving force to overwhelm mass transfer resistances of dye molecules from the solution onto the surface of the adsorbent [16]. A significant increase in adsorption capacity was observed at the early stage (5-20 min) of the adsorption which suggests that a substantial amount of vacant sites on adsorbent surface were occupied by adsorbate molecules. At the later stage of the process, there was marked reduction in the speed of dye uptake as reflected by small increase in adsorption capacity. This was attributed to a little number of unoccupied sites and repulsion between adsorbate molecules in liquid and solid phases. Similar results have been reported for the adsorption of MG dye on biochar and *L. leucocephala* biomass [17,18].

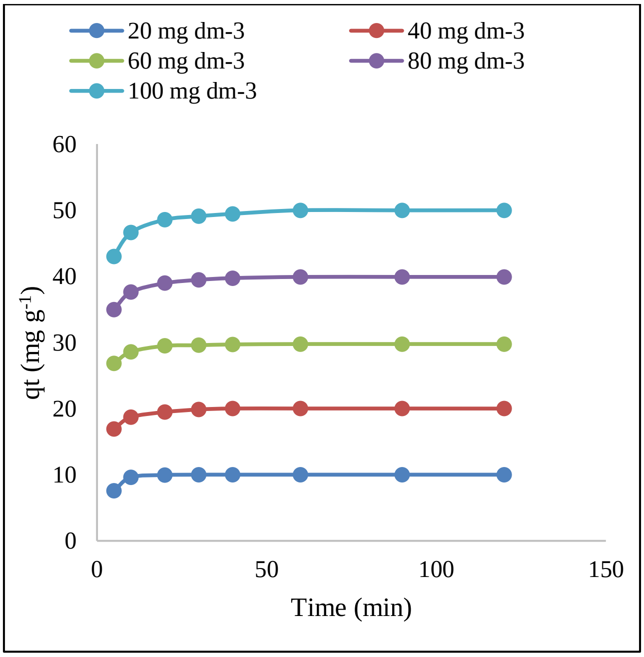

Fig. 1. MG dye uptake against time at various initial concentration

The contact time required for the attainment of equilibrium was observed to be dependent on initial dye concentration. For the initial concentrations of 20-40 mg dm^{-3}, equilibrium was attained around 30 min. However, for dye solutions of higher concentration (60-100 mg dm^{-3}), equilibrium time of 60 min was required. The variation in the contact time required to attain equilibrium is attributed to the fact that the dye molecules have to first overcome the boundary layer effect and then diffuse into the pores of the adsorbent [19]. Hence, MG solution with higher initial concentrations would take relatively longer time to attain equilibrium due to the higher amount of adsorbate molecules that will diffuse from the bulk liquid into the pores of the adsorbent. Similar behavior was observed in the removal of MG using walnut shell [20].

3.2. Effect of Temperature

The effect of temperature was investigated with different temperature ranges from 303.15 to 333.15 K. The results obtained are shown in Fig. 2. It is seen that the amount of MG adsorbed increased slightly from 16.49 to 16.56 mg g^{-1}. These indicate that the adsorption is favored at high temperatures which is true for an endothermic process. An increase in temperature normally enhances the diffusion rate of adsorbate molecules within the pores of the adsorbent due to the increased kinetic energy of the solute. Similar effect in temperature was previously reported on adsorption of MG onto eggshells [21].

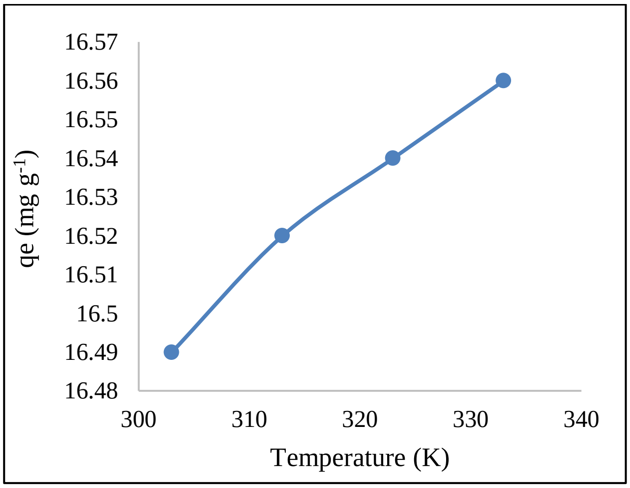

Fig. 2 MG dye uptake against temperature using 100 mg dm-3 dye concentration

3.3. Kinetic Studies

Kinetics is an important consideration in understanding the adsorption process as it provides information about the rate and controlling mechanism of the adsorption. In order to explain the kinetic behavior of MG adsorption onto DDAC, the pseudo-first-order, pseudo-second-order, Elovich, intraparticle diffusion and Boyd models were employed to fit the experimental data. The best fitting model was chosen based on conformity between the model predicted values and the experimental data as well as the linear correlation coefficient (R^2). The different kinetic parameters were evaluated and summarized in Table 1.

3.3.1. Pseudo-first-order kinetics

The liner form of pseudo-first order kinetic model described by [22] is expressed by the following equation:

$$\ln(q_e - q_t) = \ln q_e - k_1 t \qquad (3)$$

where; k_1 is the pseudo-first order adsorption rate constant (min^{-1}), q_e is the equilibrium amount of adsorbate adsorbed per unit mass of adsorbent (mg g^{-1}), q_t is the amount of adsorbate adsorbed per unit mass of adsorbent at time t (mg g^{-1}), and t is the time (min). A linear plot of ln (qe–qt) against t allows one to obtain the rate constant as slope while the intercept gives equilibrium adsorption capacity. From Table 1, the estimated q_{ecal} values of the pseudo-first-order model vary significantly with the experimental q_{eexp} values in all the concentration ranges studied. Also, the values of the correlation coefficient are relatively lower than those of pseudo-second-order model. This indicates that the pseudo-first-order model is not the most appropriate model to describe the MG adsorption kinetics. The reason can be attributed to the fact that pseudo-first-order model can only describe the adsorption kinetics of the initial stage and cannot give accurate description of the whole adsorption process [23].

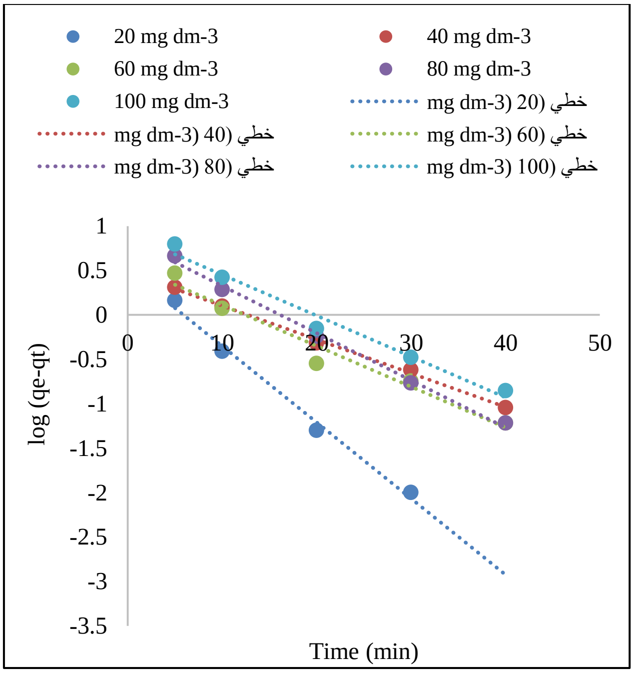

Fig. 3. Pseudo-first-order plot for MG adsorption onto DDAC

3.3.2. Pseudo-first-order kinetics

Pseudo-second order kinetic model is based on the assumption that chemisorption is the rate-controlling step. Its linear form as described by [24] is represented by the following equation:

$$\frac{t}{qt} = \frac{1}{k_2 qe^2} + \frac{t}{qe} \qquad (4)$$

Where; k_2 is the pseudo second order adsorption rate constant (g $min^{-1}mg^{-1}$), q_e is the equilibrium amount of adsorbate adsorbed per unit mass of the adsorbent (mg g^{-1}), q_t is the amount of adsorbate adsorbed per unit mass of adsorbent at time t (mg g^{-1}), and t is the time (min). The equilibrium adsorption capacity (qe), and the constant k_2 can be obtained from the slope and intercept of plot t/qt against t.

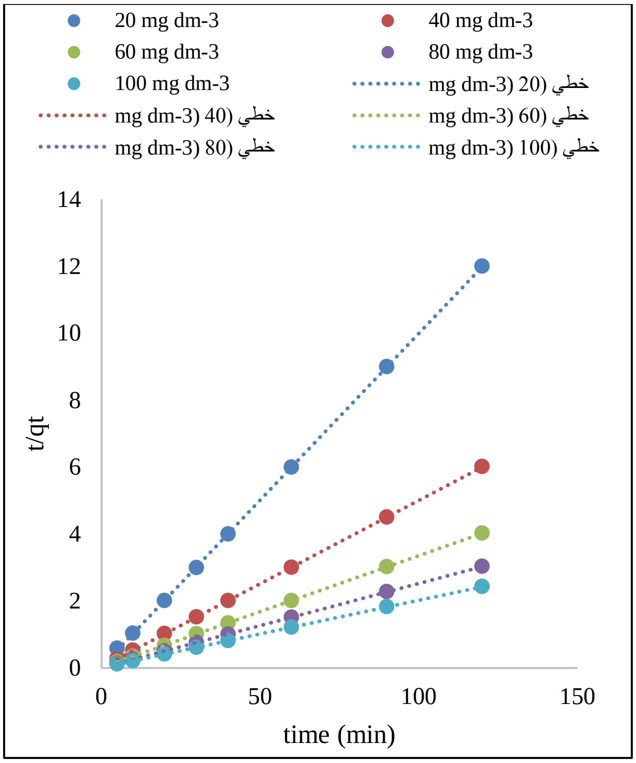

Fig. 4. Pseudo-second-order plot for MG adsorption onto DDAC

The parameters associated with this model are presented in Table 1. The q_{ecal} obtained from the pseudo-second-order for the sorption of MG were in conformity to the q_{eexp}. Also, the fitting degree of the pseudo-second-order model was extremely high. The R^2 values of the fitting line were greater than 0.998 in all the concentration ranges studied. These values were higher than those of the other kinetic models employed. The pseudo-second-order rate constant k_2, was found to decrease with increase in initial concentration. This suggest quicker adsorption rate at lower adsorbate concentration. This behavior was attributed to the lower competition for active sites at lower dye concentration. The more concentrated the solution, the more time it will take for the molecules to diffuse from bulk solution into the adsorbent pores and thus lower k_2 [25]. These results indicate that the pseudo-second-order model was the best model to describe the adsorption kinetics. It is important to mention that removal of MG by other adsorbents such as sawdust and banana peel have also been found to obey pseudo-second-order kinetics [26,27].

3.3.3. Elovich model

The linear form of this model may be represented by the following equation [28]:

$$q_t = \frac{\ln(\alpha \times \beta)}{\beta} + \frac{\ln(t)}{\beta} \qquad (5)$$

Where, α (mg g^{-1} min^{-1}) is the initial adsorption rate, β (g mg^{-1}) is the desorption constant of Elovich model and t (min) is time. Therefore, the constants α and β can be obtained from the slope and intercept of a plot of qt against ln t.

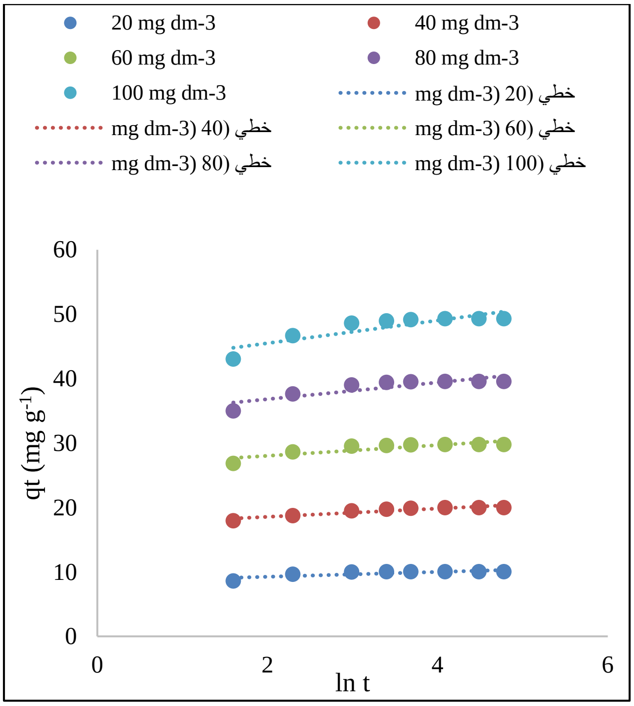

Fig. 5. Elovich plot for MG adsorption onto DDAC

The Elovich model shows a relatively low correlation coefficient for all the concentration ranges studied (Table 1). This indicates that the kinetic data of MG adsorption onto the adsorbent could not be appropriately modelled using this model. The Elovich constant β was observed to decrease with the increase in initial MG concentration. This suggest that desorption is more pronounced at lower adsorbate concentration.

3.3.4 Intraparticle diffusion model

Intraparticle diffusion model is based on the theory proposed by [29] which explains the mechanism of adsorption through diffusion and can be mathematically expressed as:

$$q_t = k_{id} t^{\frac{1}{2}} + C \qquad (6)$$

where; k_{id} is the intra-particle diffusion rate constant (mg $min^{-1/2}g^{-1}$), q_t is the amount of adsorbate adsorbed per unit mass of adsorbent at time t (mg g^{-1}), t is the time (min) and C is a constant. The intra-particle diffusion constant k_{id} can be obtained from the slope of the plot of qt against $t^{1/2}$. The intercept C, to this linear plot is a measure of thickness of the boundary layer.

The applicability of the intraparticle diffusion model is evaluated using a plot of q_t against $t^{1/2}$. The plots (Fig. 6) present a multilinearity and non-zero intercepts in all cases implying that intraparticle diffusion is not the sole rate-determining step. Also, the boundary layer thickness values are larger (Table 1) indicating the significant contribution of the surface adsorption in the rate controlling step [30]. Furthermore, the thickness of the boundary layer was found to be proportional to the initial concentration of MG in aqueous solution. The deviation from the origin may be attributed to the variation in the rate of mass transfer during the stages of adsorption [31]. So the overall adsorption process may be jointly controlled by film and intraparticle diffusion mechanisms.

3.3.5 Intraparticle diffusion model

Boyd model was also employed to investigate the rate controlling mechanism of the adsorption process. The model can be represented by the following form [32]:

$$B_t = -0.4977 - \ln\left(1 - \frac{q_t}{q_e}\right) \qquad (7)$$

where, q_t and q_e (mg g^{-1}) represent quantity of adsorbate adsorbed at time t (min) and at equilibrium, respectively;

$$B = \frac{\pi^2 D_i}{r^2}$$

(D_i is the effective diffusion coefficient of the adsorbate and r is the radius of adsorbent particles assumed to be spherical).

If the plot of Bt against time is a straight line passing through the origin, then the adsorption rate is governed by particle diffusion mechanism; otherwise, it is governed by film diffusion mechanism [33]. The linear plots in Fig. 7 do not pass through the origin, confirming the involvement of film diffusion in the uptake of MG by DDAC. However, the plots are not straight lines, indicating that film diffusion shows a relatively weak rate control for MG adsorption onto the adsorbent. Similar observations were previously reported for the adsorption of methyl orange and methylene blue by activated carbon nanotubes [34]

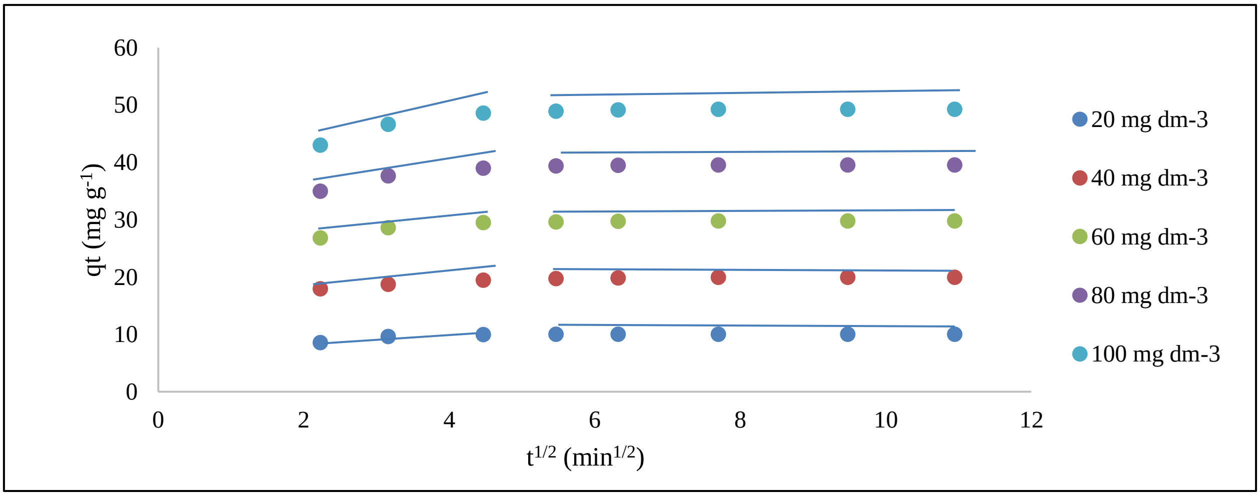

Fig. 6. Intraparticle diffusion plot for MG adsorption onto DDAC

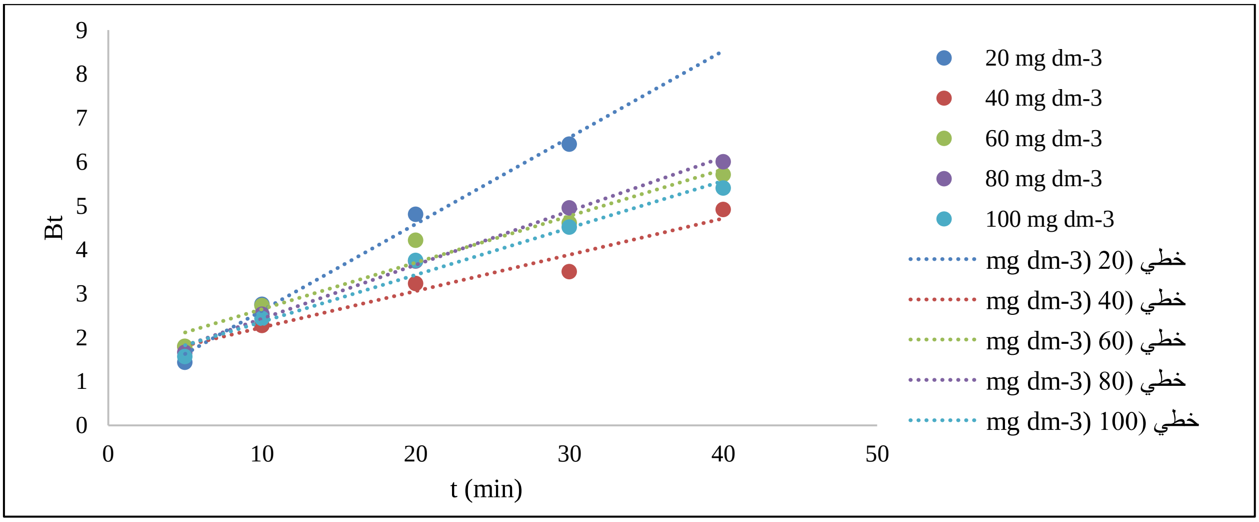

Fig. 7. Boyd plot for MG adsorption onto DDA

Table 1: Kinetic parameters for MG adsorption onto DDAC

Kinetic model	parameters	Initial MG concentration (mg dm^{-3})				
		20	40	60	80	100
Pseudo-first-order	k_1 (min^{-1})	0.197	0.087	0.105	0.122	0.106
	$q_{e\ exp}$ (mg g^{-1})	10.00	19.95	29.76	39.53	49.26
	$q_{e\ cal}$ (mg g^{-1})	3.20	3.02	3.69	7.19	8.19
	R^2	0.9916	0.9971	0.9638	0.9712	0.9311
Pseudo-second order	k_2 (g mg^{-1}min^{-1})	0.10	0.03	0.003	0.002	0.001
	$q_{e\ exp}$ (mg g^{-1})	10.00	19.95	29.76	39.53	49.26
	$q_{e\ cal}$ (mg g^{-1})	10.05	20.08	29.85	39.68	49.50
	R^2	0.9999	0.9999	0.9999	0.9999	0.9999
Intraparticle diffusion	K_{id} (mgg^{-1}min$^{-1/2}$)	0.11	0.20	0.23	0.39	0.54
	C	9.08	18.18	27.69	36.18	44.65
	R^2	0.4349	0.6724	0.5336	0.5439	0.5459
Elovich	β (g mg^{-1})	2.67	1.57	1.22	0.77	0.57
	R^2	0.6405	0.8642	0.7428	0.7546	0.7554

3.4 Thermodynamic Studies

To investigate thermodynamic behavior of MG adsorption onto DDAC, thermodynamic parameters of MG adsorption including Gibbs free energy (ΔG^{o}), standard enthalpy change (ΔH^{o}) and standard entropy change (ΔS^{o}) were evaluated using the following equations:

$$\Delta G^{\circ} = -RT\ln K_c \qquad (8)$$

where R (8.314 J mol^{-1} K^{-1}) is the universal gas constant, T (K) is the temperature, and K_c is the equilibrium constant of adsorption which is defined by the relation:

$$K_c = \frac{C_{ads}}{Ce} \qquad (9)$$

where C_{ads} and C_e (mg dm^{-3}) are equilibrium concentration of MG on the adsorbent and in the liquid phase, respectively.

The values ΔS^{o} and ΔH^{o} can be estimated from the slope and intercept of plot of ΔG against T, respectively as shown in Fig. 8.

$$\Delta G^{o} = \Delta H^{o} - T\Delta S^{o} \qquad (10)$$

The values of thermodynamic parameters for MG adsorption onto DDAC have been summarized in Table 2. The negative values of ΔG^{o} indicate that the adsorption process is feasible and spontaneous. Furthermore, the ΔG values become more negative with increasing temperature of the system. This indicates that the adsorption is favored at higher temperatures in the studied range. Also, the ΔG values are between -20 and 0 kJ mol^{-1} implying that the adsorption is governed by physical forces [35]. According to the data presented in Table 2, the positive value of ΔH confirms the endothermic nature of the MG adsorption process. The positive value of ΔS shows that the adsorption process is governed by entropy rather than enthalpy [36].

Table 2: Thermodynamic parameters of MG adsorption onto DDAC

Temp. (K)	ΔG(kJ mol^{-1})	ΔH(kJ mol^{-1})	ΔS(kJ mol^{-1}K^{-1})
303.15	-11.45	8.39	0.065
313.15	-12.18		
323.15	-12.84		
333.15	-13.42		

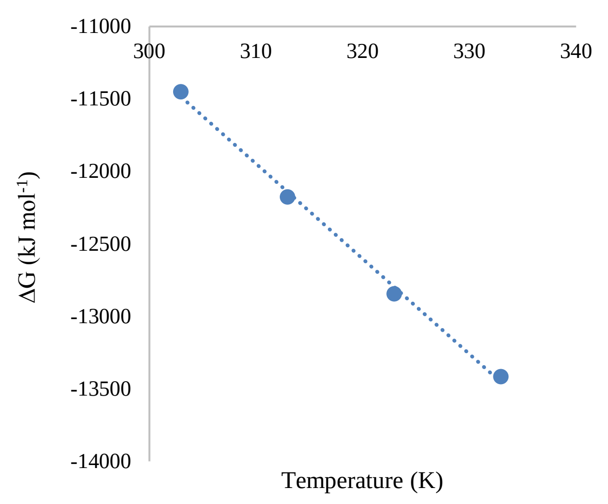

Fig. 8. Plot of Gibbs free energy change against temperature for MG adsorption

3.5 Surface Morphology of Activated Carbon

SEM is a very vital technique to observe the morphological features of adsorbent before and after adsorption. Fig. 9a depicts that the surface of the DDAC was irregular with uneven pore structure. The presence of pores on the surface of the adsorbent was attributed to the evaporation of some volatile organic compounds present in desert date seed shell during impregnation and thermal treatment. In contrast, Fig. 9b shows some distinguished white clusters and decline in the porosity of DDAC after the adsorption of dye molecules. These changes in morphology might be due to the attachment of MG molecules onto the surface of the adsorbent.

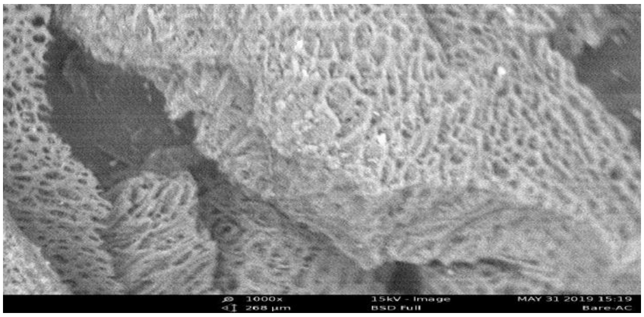

Fig. 9a SEM micrograph of DDAC before MG adsorption

Fig. 9b SEM micrograph of DDAC after MG adsorption

4. Conclusion

In this study, the ability of activated carbon derived from desert date seed shell to remove malachite green from aqueous solution was investigated. Experimental variables such as contact time, initial concentration and temperature were found to have effect on the adsorption process. Kinetic studies revealed that the pseudo-second-order model could well describe the removal of MG by DDAC. Studies on thermodynamic behavior showed that the adsorption process was feasible, spontaneous and endothermic in nature. The results demonstrate that the adsorbent could be exploited in the removal of malachite green from aqueous medium.

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