



## Adsorption isotherm and kinetic modeling of 2,4-D pesticide on activated carbon derived from date stones

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### ABSTRACT

In this work, the adsorption of 2,4-dichlorophenoxyacetic acid (2,4-D) on activated carbon derived from date stones (DSAC) was studied with respect to pH and initial 2,4-D concentration. The experimental data were analyzed by the Freundlich isotherm, the Langmuir isotherm, and the Temkin isotherm. Equilibrium data fitted well with the Langmuir model with maximum adsorption capacity of 238.10 mg/g. Pseudo-first and pseudo-second-order kinetics models were tested with the experimental data, and pseudo-first-order kinetics was the best for the adsorption of 2,4-D by DSAC with coefficients of correlation  $R^2 \geq 0.986$  for all initial 2,4-D concentrations studied. The results indicated that the DSAC is very effective for the adsorption of 2,4-D from aqueous solutions.

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### 1. Introduction

2,4-Dichlorophenoxyacetic acid (2,4-D) is commonly used in agriculture sector. It is used for a control of wide range of broad leaf weeds and grasses in plantation crops, such as sugar cane, oil palm, cocoa and rubber. The 2,4-D is commonly preferred because of its low-cost and good selectivity. It is considered as moderately toxic and maximum allowable concentration is 100 ppb in drinking water. On the other hand 2,4-D is a poorly biodegradable pollutant. Consequently, it has been frequently detected in water bodies in various regions of the world [1]. The toxicity of pesticides and their degradation products is making these chemical substances a potential hazard by contaminating our environment [2]. Therefore, the removal of pesticides from water is one of the major environmental concerns these days.

Several methods are available for pesticides removal such as photocatalytic degradation [3,4], combined photo-Fenton and biological oxidation [5], advanced oxidation processes [6], aerobic degradation [7], nanofiltration membranes [8], ozonation [9] and adsorption [10–18]. Adsorption on activated carbon is the most widespread technology used to deal with purification of water contaminated by pesticides [19], dyes [20–22] and phenols [23], etc.

Commercially available activated carbons are still considered expensive due to the use of non-renewable and relatively expensive starting material such as coal. This has led a growing research interest in the production of activated carbons from renewable and cheaper precursors. To our knowledge, the adsorption of 2,4-dichlorophenoxyacetic acid in the solutions using date stones-based activated carbon was not studied.

In this work, we attempt to use date palm (*Phoenix dactylifera*) stones as low-cost, abundantly available and renewable precursor for production of activated carbon as adsorbent for the removal of pesticide from aqueous solutions. The date palm may be the world's oldest food-producing plant known to humans. Palm trees are abundant in several countries in the world such as Iraq, Saudi Arabia, Iran, Egypt, Algeria and other Mediterranean countries. The world annual production of the dates was more than 5 million tons in 2004 [24]. Pits of date palm (seed) are a waste product of many industries, after technological transformation of the date fruits [25–27] or their biological transformation [28,29]. Date stones represent about 10% of the date weight. Therefore, considerable amount of biomass is generated annually. Any attempt to reutilize this waste will be useful.

Therefore, the purpose of this work was to evaluate the adsorption potential of date stones-based activated carbon for 2,4-dichlorophenoxyacetic acid. The equilibrium and kinetic data of the adsorption process were then studied to understand the adsorption mechanism of 2,4-D molecules onto the prepared activated carbon.

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## 2. Materials and methods

### 2.1. Adsorbate

Technical grade 2,4-D of 95% purity obtained from Sigma–Aldrich (M) Sdn Bhd, Malaysia was used as an adsorbate and was not purified prior to use. The chemical structure of 2,4-D is shown in Scheme 1.

### 2.2. Preparation and characterization of activated carbon

The palm dates were initially scraped with a knife to remove all fibers present at surface. The collected date stones were washed and dried in an air oven at 70 °C for 48 h and then crushed and sieved to the desired particle size (2–3 mm). The resultant sieve cut was carbonized in a stainless steel vertical tubular reactor placed in a tube furnace at 700 °C under purified nitrogen (99.995%) flow of 150 cm<sup>3</sup>/min for 2 h. The heating rate was fixed at 10 °C/min. The char produced was then soaked in potassium hydroxide (KOH) solution with an impregnation ratio of 1:3 (Char:KOH wt.%). The mixture was then dehydrated in an oven overnight at 105 °C and then activated to a final temperature of 850 °C at heating rate of 10 °C/min. Once the final temperature was reached, the nitrogen gas flow was switched to carbon dioxide and activation was held for 2 h. The activated carbon was then cooled to room temperature under nitrogen flow of 150 cm<sup>3</sup>/min. Next the activated carbon was washed with 0.1 M hydrochloric acid and then with deionized water and until the pH of the washing solution reached 6.5–7 [21].

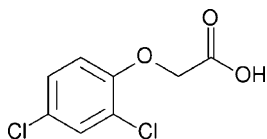
Textural characterization of the activated carbon was carried out by Accelerated Surface Area Porosimetry System 2000. Scanning electron microscopy (SEM) analysis was carried out for the prepared activated carbon to study the surface morphology.

### 2.3. Effect of solution pH

The effect of solution pH was studied by agitating 0.20 g of activated carbon and 200 mL of 2,4-D concentration 100 mg/L using water-bath shaker at 30 °C. The experiment was conducted at different pH from 2 to 11. Agitation was provided for 9 h and 30 min contact time at a constant agitation speed of 130 rpm. The pH was adjusted by adding a few drops of diluted 1.0N NaOH or 1.0N HCl and measured by using a pH meter (Ecoscan, EUTECH Instruments, Singapore).

### 2.4. Equilibrium studies

Batch equilibrium studies were carried out by adding a fixed amount of activated carbon (0.20 g) into 250-mL Erlenmeyer flasks containing 200 mL of different initial concentrations (50–400 mg/L) of 2,4-D solution. The flasks were agitated in an isothermal water bath shaker at 130 rpm and 30 °C for 9 h and 30 min. The initial and equilibrium 2,4-D concentrations were determined by absorbance measurement using double beam UV/vis spectrophotometer (Shimadzu, Model UV 1601, Japan) at 284 nm. It was then computed to 2,4-D concentration using standard calibration curve. The amount



Scheme 1. Chemical structure of 2,4-D.

of adsorption at equilibrium,  $q_e$  (mg/g), was calculated by:

$$q_e = \frac{(C_0 - C_e)V}{W} \quad (1)$$

where  $C_0$  and  $C_e$  (mg/L) are the liquid-phase concentrations of 2,4-D at initial and equilibrium, respectively.  $V$  (L) is the volume of the solution and  $W$  (g) is the mass of dry adsorbent used.

### 2.5. Sorption kinetics

For kinetic studies, 0.20 g of activated carbon was contacted with 200 mL of 2,4-D concentrations 50–400 mg/L using water-bath shaker at 30 °C. The agitation speed was kept constant at 130 rpm. At predetermined intervals of time, solutions were analyzed for the final concentration of 2,4-D. The amount of adsorption  $q_t$  (mg/g), at time  $t$  (h), was calculated by:

$$q_t = \frac{(C_0 - C_t)V}{W} \quad (2)$$

where  $C_t$  (mg/L) is concentrations of 2,4-D at time  $t$ .

## 3. Results and discussion

### 3.1. Characterization of DSAC

Table 1 summarizes the micropore size, pore volume and surface area data for DSAC. The surface morphologies of the date stone precursor char and activated carbon produced were determined using scanning electron microscopy. Fig. 1(a–c) shows the SEM photographs of the precursor, char and DSAC, respectively, at 500× magnifications. It can be seen from Fig. 1(a) that the surface precursor is very rough. The char surface shown in Fig. 1(b) indicates that the surface is not smooth with heterogenous pores. Fig. 1(c) shows the surface of activated carbon produced, which contains well-developed pores where there is a good possibility for 2,4-D to be trapped and adsorbed into the surface of the pores.

### 3.2. Effect of solution pH on 2,4-D adsorption

The effect of pH on 2,4-D adsorption was studied using 100 mg/L of initial concentration, pH 2–11 at 30 °C. The 2,4-D adsorption was significantly changed over the pH value of 2–11. The equilibrium adsorption ( $q_e$ ) was found to decrease with increasing pH as shown in Fig. 2. The  $q_e$  decreases from 100 to 30 mg/g for an increase in pH from 2 to 11. A similar result was reported for the adsorption of 2,4-D from aqueous solution by granular activated carbon [1]. The behavior clearly suggests that the adsorption was dominated by the interaction between pesticide and adsorbent surface.

### 3.3. Effect of contact time and initial concentration on 2,4-D adsorption

The adsorption of 2,4-D by DSAC was studied at different initial 2,4-D concentrations (50, 100, 200, 250, 300 and 400 mg/L). Fig. 3 shows the result for effect of initial concentration on adsorption of 2,4-D onto DSAC. As can be seen from Fig. 3, the amount of the adsorbed 2,4-D at low initial concentration (50–100 mg/L) achieve

Table 1  
Physical properties of DSAC

BET surface area (m <sup>2</sup> /g)	763.40
Langmiur surface area (m <sup>2</sup> /g)	1111.75
Total pore volume of pores (cm <sup>3</sup> /g)	0.424
Micropore volume (cm <sup>3</sup> /g)	0.215
Average pore diameter (Å)	26.184

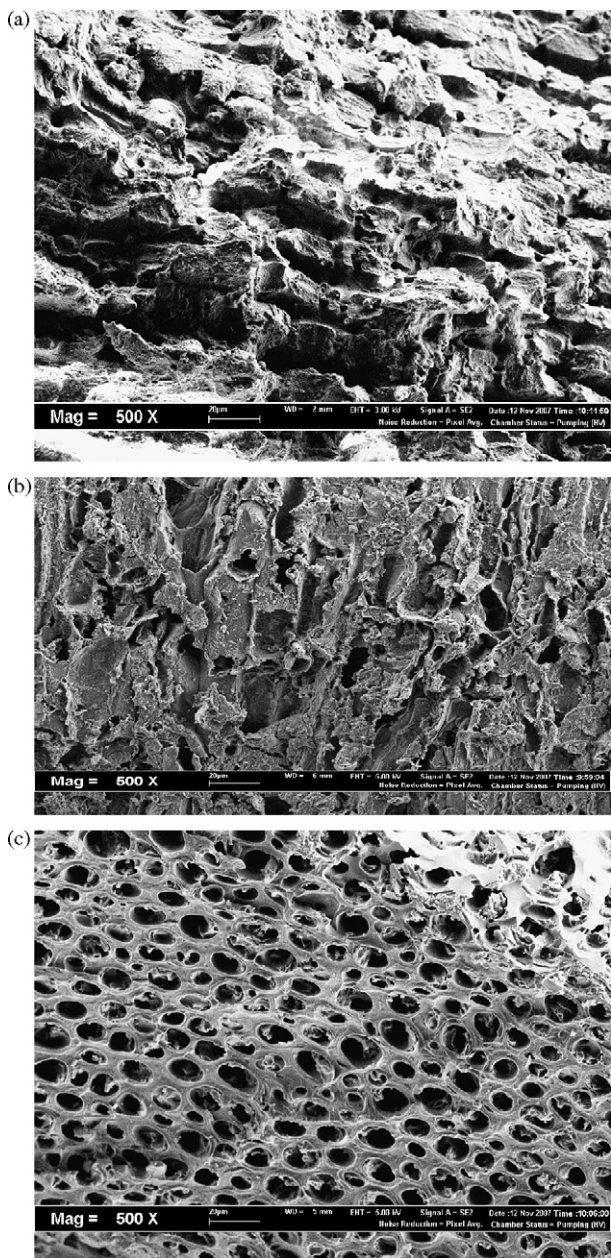


Fig. 1. SEM micrograph of (a) Precursor; (b) Char; (c) DSAC (magnifications: 500×).

adsorption equilibrium in approximately 4 h, while at high initial 2,4-D concentration (200–400 mg/L), the time necessary to reach equilibrium was 7 h and 30 min. However, the experimental data were measured at 9 h and 30 min to be sure that full equilibrium

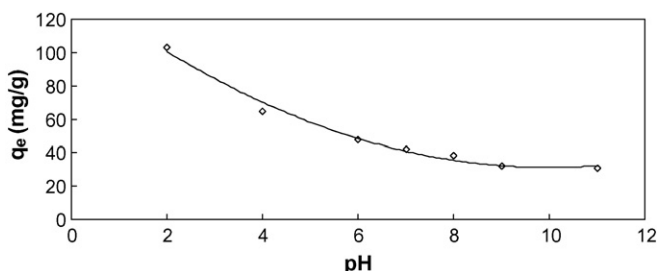


Fig. 2. Effect of pH on equilibrium uptake of 2,4-D.

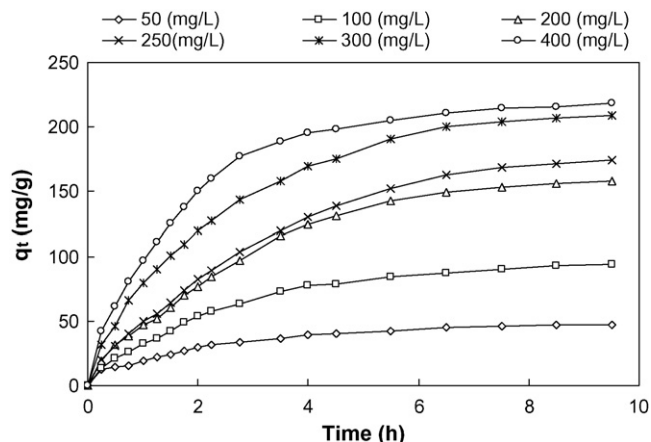


Fig. 3. Effect of initial concentration and contact time on 2,4-D adsorption ( $W=0.20$  g;  $V=0.20$  L;  $pH$  3.6;  $T=30$  °C).

was attained. It can be seen from Fig. 3 that an increase in initial 2,4-D concentration leads to increase in the adsorption of 2,4-D on DSAC. The % removals of 2,4-D at equilibrium were 91.83, 91.38, 78.73, 70.20, 68.07 and 54.14% for initial concentration of 50, 100, 200, 250, 300 and 400 mg/L, respectively.

The equilibrium adsorption increases from 50 to 200 mg/g, with increase in the initial 2,4-D concentration from 50 to 400 mg/L. This indicates that initial concentrations played an important role in the adsorption of 2,4-D on DSAC.

### 3.4. Equilibrium modeling

Three isotherms were tested for their ability to describe the experimental results, namely the Langmuir isotherm, the Freundlich isotherm and the Temkin isotherm.

The Langmuir adsorption model [30] is based on the assumption that maximum adsorption corresponds to a saturated monolayer of solute molecules on the adsorbent surface, with no lateral interaction between the sorbed molecules. The linear expression of the Langmuir model is given by Eq. (3)

$$\frac{C_e}{q_e} = \frac{1}{Q_0 b} + \left(\frac{1}{Q_0}\right) C_e \quad (3)$$

where  $q_e$  (mg/g) and  $C_e$  (mg/L) are the amount of adsorbed 2,4-D per unit mass of adsorbent and 2,4-D concentration at equilibrium, respectively.  $Q_0$  is the maximum amount of the 2,4-D per unit mass of adsorbent to form a complete monolayer on the surface bound at high  $C_e$ , and  $b$  is a constant related to the affinity of the binding sites (L/mg). The plot of specific adsorption ( $C_e/q_e$ ) against the equilibrium concentration ( $C_e$ ) (Fig. 4) shows that the adsorption obeys the Langmuir model. The Langmuir constants  $Q_0$  and  $b$  were deter-

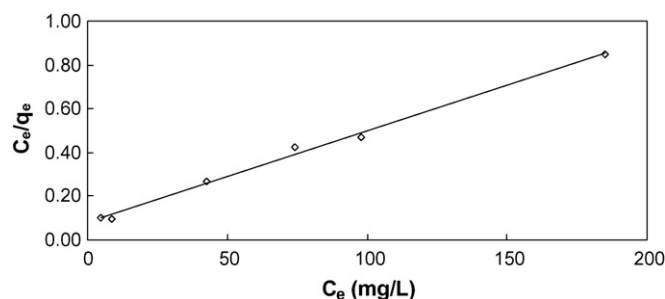


Fig. 4. Langmuir adsorption isotherm of 2,4-D on DSAC.

**Table 2**  
Isotherm parameters for removal of 2,4-D by DSAC

Isotherm	Parameters
Langmuir	
$Q_0$ (mg/g)	238.10
$b$ (L/mg)	0.052
$R^2$	0.9943
Freundlich	
$K_F$	2.529
$n$	32.063
$R^2$	0.9227
Temkin	
$A$	1.494
$B$	46.305
$R^2$	0.9805

mined from the slope and intercept of the plot and are presented in Table 2.

The essential characteristics of the Langmuir isotherm can be expressed in terms of a dimensionless constant separation factor  $R_L$  that is given by Eq. (4) [31]:

$$R_L = \frac{1}{1 + bC_0} \quad (4)$$

where  $C_0$  is the highest initial concentration of adsorbate (mg/L), and  $b$  (L/mg) is Langmuir constant. The value of  $R_L$  indicates the shape of the isotherm to be either unfavorable ( $R_L > 1$ ), linear ( $R_L = 1$ ), favorable ( $0 < R_L < 1$ ), or irreversible ( $R_L = 0$ ). The  $R_L$  values between 0 and 1 indicate favorable adsorption. The value of  $R_L$  in the present investigation was found to be 0.0459 indicating that the adsorption of 2,4-D on DSAC is favorable.

The Freundlich isotherm [32] is an empirical equation employed to describe heterogeneous systems. The linear form of Freundlich equation is expressed

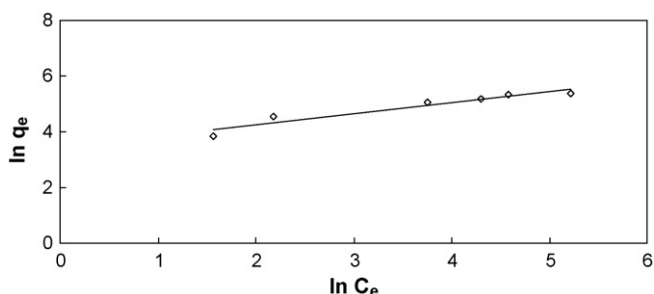
$$\ln q_e = \ln K_F + \left(\frac{1}{n}\right) \ln C_e \quad (5)$$

where  $K_F$  and  $n$  are Freundlich constants with  $K_F$  (mg/g (L/mg)<sup>1/n</sup>) is the adsorption capacity of the sorbent and  $n$  giving an indication of how favorable the adsorption process. The magnitude of the exponent,  $1/n$ , gives an indication of the favorability of adsorption. Values of  $n > 1$  represent favorable adsorption condition [33,34]. Values of  $K_F$  and  $n$  are calculated from the intercept and slope of the plot (Fig. 5) and listed in Table 2.

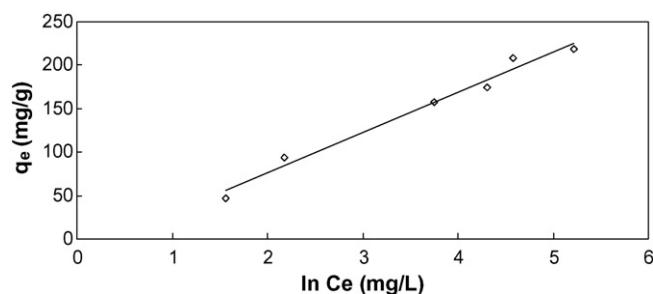
The Temkin isotherm [35] has generally been applied in the linear form:

$$q_e = B \ln A + B \ln C_e \quad (6)$$

where  $B = RT/b$ ,  $b$  is the Temkin constant related to heat of sorption (J/mol);  $A$  is the Temkin isotherm constant (L/g),  $R$  the gas constant (8.314 J/mol K) and  $T$  the absolute temperature (K). Therefore,



**Fig. 5.** Freundlich adsorption isotherm of 2,4-D on DSAC.



**Fig. 6.** Temkin adsorption isotherm of 2,4-D on DSAC.

by plotting  $q_e$  versus  $\ln C_e$  (Fig. 6) the constants  $A$  and  $B$  can be determined. The constants  $A$  and  $B$  are listed in Table 2.

As seen in Table 2, the Langmuir isotherm fits quite well with the experimental data (correlation coefficient  $R^2 > 0.99$ ). The monolayer adsorption capacity according to this model was 238.10 mg/g. The fact that the Langmuir isotherm fits the experimental data very well may be due to homogeneous distribution of active sites onto DSAC surface, since the Langmuir equation assumes that the surface is homogenous. A similar result was reported in literature for the adsorption of paracetamol dichloride from aqueous solution by activated carbon derived from used tires [19].

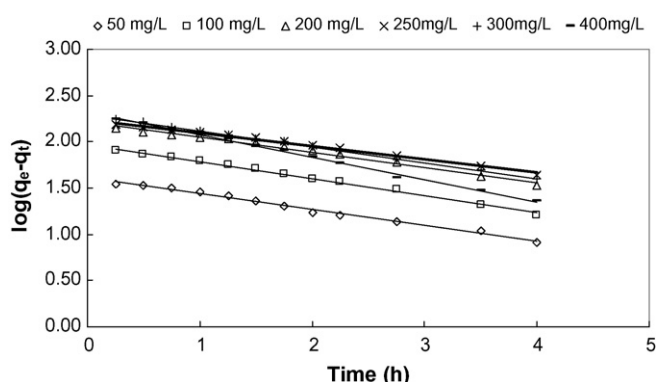
Table 3 compares the adsorption capacity of different types of adsorbents used for removal of pesticides. The value of  $Q_0$  in this study is larger than those in most of previous works. This suggests that 2,4-D could be easily adsorbed by DSAC.

### 3.5. Adsorption kinetics

The modeling of the kinetics of adsorption of 2,4-D on DSAC was investigated by two common models, namely, the Lagergren pseudo-first order model [41] and pseudo-second-order model [34,42]. Lagergren proposed a method for adsorption analysis which is the pseudo-first-order kinetic equation [41] in the linear form:

$$\log(q_e - q_t) = \log q_e - \left(\frac{k_1}{2.303}\right) t \quad (7)$$

where  $q_e$  and  $q_t$  are the amounts of 2,4-D adsorbed at equilibrium in mg/g, and at time  $t$  in h, respectively, and  $k_1$  is the pseudo-first-order rate constant (1/h). A linear plot of  $\ln(q_e - q_t)$  against time allows one to obtain the rate constant (Fig. 7). If the plot was found to be linear with good correlation coefficient, indicating that Lagergren's equation is appropriate to 2,4-D sorption on DSAC. So, the adsorption process is a pseudo-first-order process [41,43]. The Lagergren's first-order rate constant ( $k_1$ ) and  $q_e$  deter-



**Fig. 7.** Pseudo-first-order kinetic for adsorption of 2,4-D on DSAC.

**Table 3**  
Comparison of pesticide adsorption of this work and previous studies

Pesticide	Adsorbent	Q <sub>0</sub> (mg/g)	Reference
2,4-D	Date stone activated carbon	238.10	This work
Paraquat dichloride	Commercial activated carbon F300 (CAC)	68.0	[19]
Paraquat dichloride	Pyrolysis and activation of used tires (TAC)	27.8	[19]
Bentazon	Activated carbon-cloth	151	[36]
Propanil	Activated carbon-cloth	114	[36]
Bifenthrin	Norit GAC 1240 PLUS activated carbon	2.94 × 10 <sup>2</sup> ± 96 <sup>a</sup>	[37]
2,4-D	Carbonaceous adsorbent	277.7	[38]
Carbofuran	Carbonaceous adsorbent	277.7	[38]
Lambda-Cyhalothrin	Oil shale ash (OSA)	6.82	[39]
Deltamethrin	Oil shale ash (OSA)	10.74	[39]
Methyl parathion	Watermelon peels	24.3 ± 1.6 <sup>b</sup>	[40]

<sup>a</sup> μg/g.  
<sup>b</sup> μmol/g.

**Table 4**  
Comparison of the pseudo-first-order, pseudo-second-order adsorption rate constants and calculated and experimental q<sub>e</sub> values obtained at different initial 2,4-D concentrations

Initial concentration (mg/L)	q <sub>e,exp</sub> (mg/g)	Pseudo-first-order kinetic model			Pseudo-second-order kinetic model		
		k <sub>1</sub> (1/h)	q <sub>e,cal</sub> (mg/g)	R <sup>2</sup>	k <sub>2</sub> (g/mg h) 10 <sup>4</sup>	q <sub>e,cal</sub> (mg/g)	R <sup>2</sup>
50	47.32	0.915	40.89	0.992	0.0124	52.63	0.939
100	93.66	0.974	93.44	0.995	0.0027	129.87	0.924
200	157.70	0.866	162.73	0.986	0.0011	232.55	0.829
250	174.59	0.3316	175.186	0.9936	0.00104	250.45	0.8675
300	208.44	0.911	195.10	0.999	0.0017	263.15	0.974
400	218.33	1.290	209.39	0.997	0.0018	294.11	0.981

mined from the model are presented in Table 4 along with the corresponding correlation coefficients. It was seen that the pseudo-first-order model better represented the adsorption kinetics and the calculated q<sub>e</sub> values agreed with the experimental q<sub>e</sub> values (Table 4). This suggests that the adsorption of 2,4-D follows first-order kinetics. A similar result was reported for the adsorption of 2,4-dichlorophenoxy-acetic acid (2,4-D) from aqueous solution by granular activated carbon [1].

The pseudo-second-order kinetics may be expressed as [34,42]:

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \left(\frac{1}{q_e}\right) t \tag{8}$$

where k<sub>2</sub> (g/mg h) is the rate constant of adsorption, q<sub>e</sub> (mg/g) is the amount of 2,4-D adsorbed at equilibrium and q<sub>t</sub> (mg/g) is the amount of 2,4-D adsorbed at time t.

The equilibrium adsorption capacity (q<sub>e</sub>), and the second-order constants k<sub>2</sub> (g/mg h) can be determined experimentally from the slope and intercept of plot t/q<sub>t</sub> versus t (Fig. 8). The k<sub>2</sub> and q<sub>e</sub> determined from the model are presented in Table 4 along with the corresponding correlation coefficients. The values of the calculated

and experimental q<sub>e</sub> are represented in Table 4. It can be seen from Table 4 that there is no agreement between q<sub>e</sub> experimental and q<sub>e</sub> calculated values for the pseudo-second-order model.

### 3.6. Adsorption mechanism

In order to gain insight into the mechanisms and rate controlling steps affecting the kinetics of adsorption, the kinetic experimental results were fitted to the Weber’s intraparticle diffusion [44]. The kinetic results were analyzed by the intraparticle diffusion model to elucidate the diffusion mechanism, which model is expressed as:

$$q_t = k_{id} t^{1/2} + C \tag{9}$$

where C is the intercept and k<sub>id</sub> (mg/g h<sup>0.5</sup>) is the intraparticle diffusion rate constant, which can be evaluated from the slope of the linear plot of q<sub>t</sub> versus t<sup>1/2</sup> [44] as shown in Fig. 9. The intercept of the plot reflects the boundary layer effect. The larger the intercept, the greater the contribution of the surface sorption in the rate-controlling step. If the regression of q<sub>t</sub> versus t<sup>1/2</sup> is linear

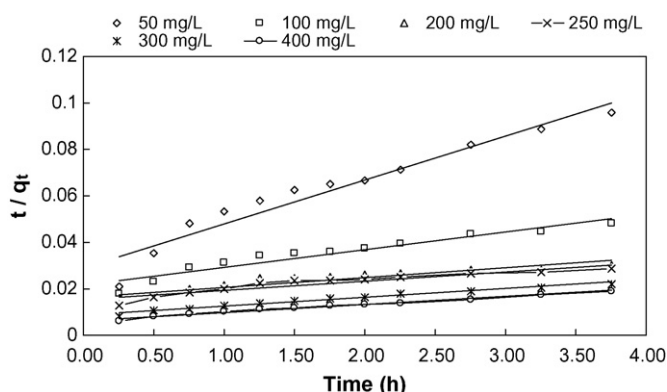


Fig. 8. Pseudo-second-order kinetic for adsorption of 2,4-D on DSAC.

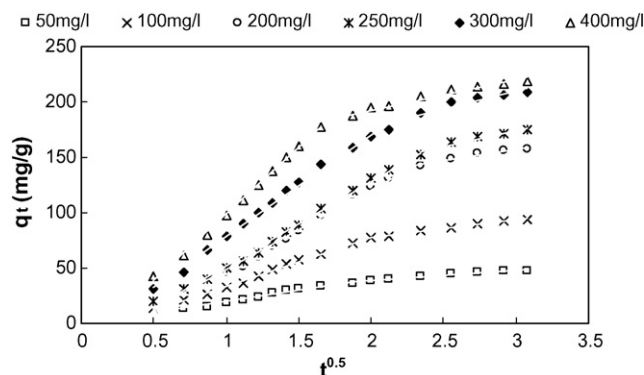


Fig. 9. Intraparticle diffusion plots for adsorption of 2,4-D on DSAC for different initial 2,4-D concentrations.

and passes through the origin, then intraparticle diffusion is the sole rate-limiting step. However, the linear plots (Fig. 9) at each concentration did not pass through the origin. This indicates that the intraparticle diffusion was not only rate controlling step. Ofomaja [45] noted the similar trend for dye adsorption on palm kernel fiber at various initial concentrations. He reported that the multiple nature observed in the intraparticle diffusion plot suggests that intraparticle diffusion is not solely rate controlling. External mass transfer of pesticide molecules on to sorbent particles is also significant in the sorption process, especially at the initial reaction period.

#### 4. Conclusions

The present study shows that activated carbon prepared from date stones can be used as an adsorbent for the removal of 2,4-D from aqueous solutions. The equilibrium data were fitted to the Langmuir, Freundlich and Temkin isotherm models, and the equilibrium data were best described by the Langmuir isotherm model. The suitability of the kinetic models for the adsorption of 2,4-D on the activated carbon was also discussed. It was found that the adsorption kinetics of 2,4-D obeyed pseudo-first-order adsorption kinetics.

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