Desalination and Water Treatment
www.deswater.com

doi: 10.1080/19443994.2014.978386

57 (2016) 1880–1889
January

Taylor & Francis
Taylor & Francis Group

Adsorptive removal of diazinon: kinetic and equilibrium study

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Received 18 December 2013; Accepted 11 October 2014

ABSTRACT

The adsorption of diazinon on bentonite acid treated with dilute H_2SO_4 solutions has been studied. Bentonite activated with 0.06 N sulfuric acid showed a higher efficiency in removing diazinon from aqueous solutions and was selected for further investigation. The effect of initial diazinon concentration, contact time, pH, and adsorbent dosage on the adsorption was studied in a batch mode process. Equilibrium data were analyzed by the Langmuir, Freundlich, Temkin, and Dubinin–Radushkevitch isotherm models. Langmuir isotherm provided the best fit to the equilibrium data with maximum adsorption capacity of 5.56 mg/g. The kinetic data were found to follow closely the pseudo-second-order model. The sorption process was found to be controlled by both film and pore diffusion with film diffusion at the earlier stages followed by pore diffusion at the later stages. Thermodynamics studies revealed the spontaneous and exothermic nature of the sorption process.

Keywords: Diazinon; Bentonite; Adsorption; Kinetic; Thermodynamic

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1. Introduction

Pesticides are intensively used against crop diseases in many agricultural areas [1–4]. Their use has increased during the last few years [5–8] despite their well-known hazards for human health [9]. Diazinon, an organophosphate insecticide, must be controlled seriously to protect human and environment from negative impact [10]. The European Union has set a maximum allowed concentration of 0.5 µg/L for the combination of all pesticides, and 0.1 µg/L for individual compounds, in drinking water [11].

One method for removing diazinon from aqueous solution is adsorption with the use of clay [12,13]. One type of clay mineral is bentonite. Bentonite, which is consisting essentially of montmorillonite group, is composed of two silica tetrahedral sheets with an octahedral alumina sheet [14,15]. Bentonite surface is negatively charged due to isomorphous substitution of Al^{3+} for Si^{4+} in the tetrahedral layer and Mg^{2+} for Al^{3+} in the octahedral layer [15,16]. This negative charge is balanced by exchangeable cations such as Na^+ in the lattice structure [15,16]. The layered structure of bentonite allows swelling after wetting. Ca^{2+} and Na^+ are hydrates in the presence of water; therefore, there is a hydrophilic environment at clay surface [16]. Very little information is available about using bentonite for the removal of organophosphate insecticides. To our knowledge, the adsorption of diazinon from aqueous

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solution using bentonite was not studied. Therefore, the objective was to determine the effects of pH, temperature, contact time, adsorbent dosage, and initial pesticide concentration. Also the adsorption efficiency of bentonite under batch mode [17] was examined. The data, thus, obtained may be helpful to environmental engineers for designing and establishing a continuous treatment plant for water and wastewater.

2. Materials and methods

2.1. Bentonite and pesticide

Bentonite (composition: SiO_2: 47%, Al_2O_3: 9.5%, MgO: 2.4%, Fe_2O_3 1%, K_2O: 1%, Na_2O: 0.39%, CaO: 0.33%, TiO_2: 0.18%, and loss of ignition: 38.15%) was purchased from ENOF (Ltd Algerian bentonite Company), Algeria. This result indicates the presence of silica and alumina as major constituents along with traces of magnesium, iron, potassium, sodium, calcium, and titanium oxides in the form of impurities.

Analytical grade diazinon (96–97% active substance) for the experiment was purchased from Cypermethrin, Jordan, and used as received (Table 1).

The concentration of diazinon was determined using a UV–vis spectrophotometer (Shimadzu UV-1240) at a wavelength of 247 nm.

2.2. Pretreatment, activation, and characterization of bentonite

Bentonite was washed thoroughly with distilled water, filtered, and dried at 110˚C for 24 h.

Five grams of this bentonite was soaked in 100 mL of 0.06 N H_2SO_4 at 87˚C for 2 h, washed with water several times, and dried at 110˚C overnight. The acid-activated bentonite (AAB) sample thus prepared was sieved and the sample of size less than 80 μm was used for studies. The BET surface area of AAB was determined from the N_2 adsorption isotherm with a surface area analyzer (Quantachrome Instruments, Nova 2000). The specific surface area of AAB was determined as 25.91 m^2/g.

The surface functional groups and structure were studied by FTIR spectroscopy. The FTIR spectra of the AAB were recorded between 400 and 4,000 cm^{-1} in a Shimadzu Iprir Thys.2 spectrometer.

Scanning electron microscopy (JOEL JEM 100 B) analysis was carried out on AAB to study its surface morphology before and after diazinon adsorption.

2.3. Adsorption experiments

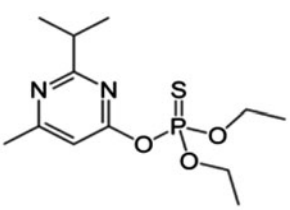
These adsorption tests were done in an Erlenmeyer flask as a batch reactor. For each adsorption test, 30 mL of the diazinon solution was transferred to the flask, and the solution pH level was adjusted to the desired value; the given amount of AAB was added to the solution, and the suspension was put on the magnetic device and stirred at 300 rpm for the predetermined time. Then, the suspension was filtered through a cellulose acetate filter (pore size of 0.2 μm) and the filtrate was analyzed for any residual diazinon. The selected variables and their limits were as follows; solution pH (3–7), AAB concentration (1–3 g/L), contact time (2–3 min), and solution temperature (11–30˚C). An equilibrium study was done using various concentration of AAB at the contact time of 1 h. The solution pH level was regulated using 1 N NaOH or HCl.

The diazinon uptake by acid treated bentonite was calculated by the following equation:

$$q_t = \frac{(C_0 - C_t)V}{m} \tag{1}$$

where q_t is the amount of adsorbed diazinon at time t (mg/g); V the volume of solution (L); C_0 and C_t are the initial and at time t diazinon concentrations, respectively (mg/L); and m is the weight of adsorbent (g).

Table 1
Characteristics of the employed pesticide

Pesticide	Molecular formula	Structural formula	Molar mass	Solubility in water
Diazinon	$C_{12}H_{21}N_2O_3PS$		304.35 (g/mol)	40 mg/L at 25˚C

The amount of adsorbed diazinon at equilibrium q_e (mg/g) was calculated by mass balance equation

$$q_e = \frac{(C_0 - C_e)V}{m} \tag{2}$$

where C_e is the equilibrium concentration of diazinon solution (mg/L).

3. Results and discussion

3.1. Effect of acid strength on diazinon adsorption

Bentonite treated with dilute H_2SO_4 was capable of the removal of diazinon compared with the ineffective adsorption behavior (lower than 2% removal) for untreated clay. Bentonite activated with 0.06 N H_2SO_4 solution (AAB) was proven the most effective possessing the highest adsorption removal capacity (77.3%), while treatment with sulfuric acid 0.02 and 0.1 N resulted in diazinon removal of 60 and 73.63%, respectively. Acid treatment has helped in transforming raw bentonite sample to a well-developed adsorbent; the pore diameters also increase [18].

3.2. Bentonite characterization by infrared spectroscopy and SEM

The FTIR spectra of acid-activated bentonite (b) and diazinon-loaded AAB (a) are shown in Fig. 1. The FTIR spectra of acid-activated bentonite is similar to that of diazinon-loaded AAB. A group of absorption peaks was observed between 3,448 and 3,630 cm^{-1}, which is due to H–O–H stretching vibration bands of water molecules, weakly hydrogen bonded to the Si–O surface in acid-activated bentonite. After adsorption of diazinon, the absorption peaks moved to 3,434 and 3,619 cm^{-1}, respectively. The band at 1,637 cm^{-1} also corresponds to the OH deformation of water (H–O–H bending vibration) observed at acid-activated bentonite. This band moved (1,638 cm^{-1}) after diazinon adsorption.

The bands that were observed at 1,382, 1,046, 917, 812, 798, and 778 cm^{-1} due to stretching vibrations of Si–O, Si–O–Si, OH attached to Al^{3+}, Fe^{3+}, and Mg^{2+} groups shifted slightly after the adsorption of diazinon. The bands at 520 and 466 cm^{-1} are due to Si–O–Al (octahedral) and Si–O–Si bendings, respectively [19]. These bands also shifted slightly after the adsorption of diazinon.

Scanning electron micrograph (SEM) of AAB before and after adsorption is shown in Fig. 2(a) and (b), respectively, (Magnification 300×). The surface features of the adsorbent after being coated with diazinon were different from that of the acid-activated bentonite. The obtained image revealed the coverage of bentonite surface with diazinon flakes.

3.3. Effect of pH

The pH value of the solution is an important controlling parameter in the adsorption process [20]. Hence, the batch adsorption experiments were conducted at different initial pH conditions ranging from 3 to 7. Fig. 3 shows the effect of pH on adsorption capacity at equilibrium, with initial diazinon concentration of 10 mg/L and adsorbent dosage of 3 g/L. First, the adsorption of diazinon increased slowly with increasing pH, reacted maximum for pH 5.2, and decreased. It was observed that the adsorption is highly dependent on the pH of the solution, which affects the surface charge of the adsorbent and the degree of ionization and speciation of the adsorbate. The decrease in adsorption with increase in pH may be explained on the basis of the point of zero charge pHpzc, at which the adsorbent is neutral [21]. The surface charge of the adsorbent is positive when the media pH is below the pHpzc value, while it is negative at a pH over the pHpzc [21]. In the present work, the obtained value of pHzpc of acid-activated bentonite is 5.5. The surface of bentonite is positively charged at a solution pH level below 5.5 (pHpzc) and would, therefore, be prone to electrostatically attract diazinon molecules which dissociate to anionic species in an acidic solution [22,23]. At pH 7.4, pH over pHpzc, the surface charge of the adsorbent is negative. The negatively charged sites reduce the adsorption of anionic diazinon due to electrostatic repulsion. This result is comparable to those obtained by other authors [23,24]. Maximum adsorption of diazinon occurs at pH 5.2 and, hence, was selected as the optimum and used throughout the study.

3.4. Effect of contact time and initial concentration

The results of adsorption of diazinon by AAB at various initial concentration of diazinon have been plotted as a function of contact time and are shown in Fig. 4. The amount of diazinon adsorbed onto bentonite increased with time at all initial concentrations and slowly reached saturation within 1 h. When the initial diazinon concentration increased from 10 to 20 mg/L, the amount adsorbed at contact time 15 min increased from 3 to 5 mg g^{-1}. Increasing the initial diazinon concentration would increase the mass transfer driving force. The rate of removal of diazinon onto AAB by

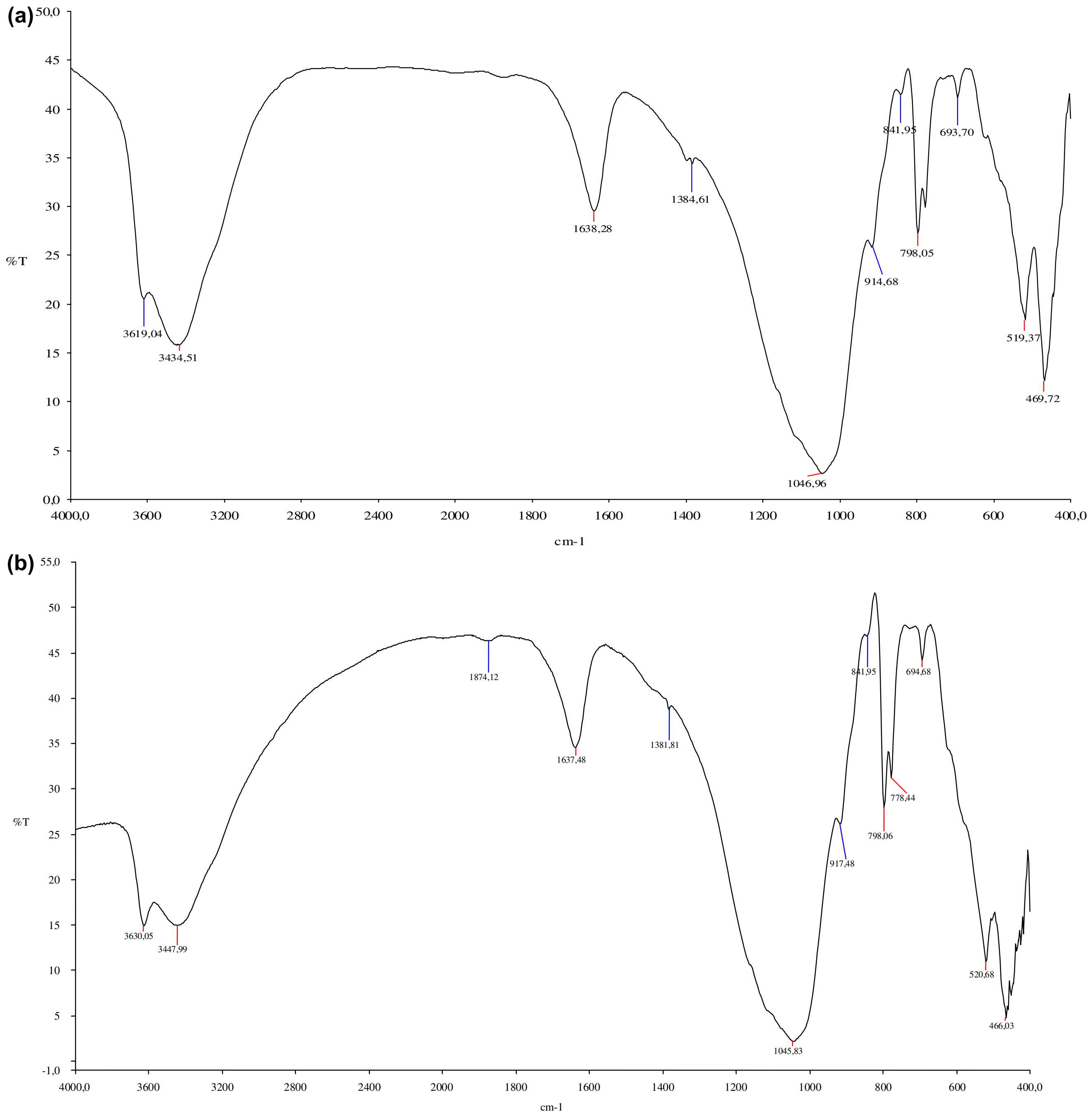


Fig. 1. Infrared spectra of acid activated bentonite (b) and diazinon-loaded AAB (a).

adsorption was rapid initially (0–15 min) and then slowed down gradually until equilibrium was attained beyond which there was no further adsorption. The initial uptake rate for diazinon was high as a large number of adsorption sites were available for adsorption at the onset of the process. Adsorption slowed down in later stages because after some time, the remaining vacant surface sites may be difficult to occupy due to the repulsive forces between diazinon molecules on the solid and liquid phases [23,25,26].

3.5. Effect of adsorbent dosage

Samples of acid-activated bentonite (1, 2, 3, and 4 g) were added to pesticide solution (1,000 mL) at an initial concentration of 10 mg/L at a constant

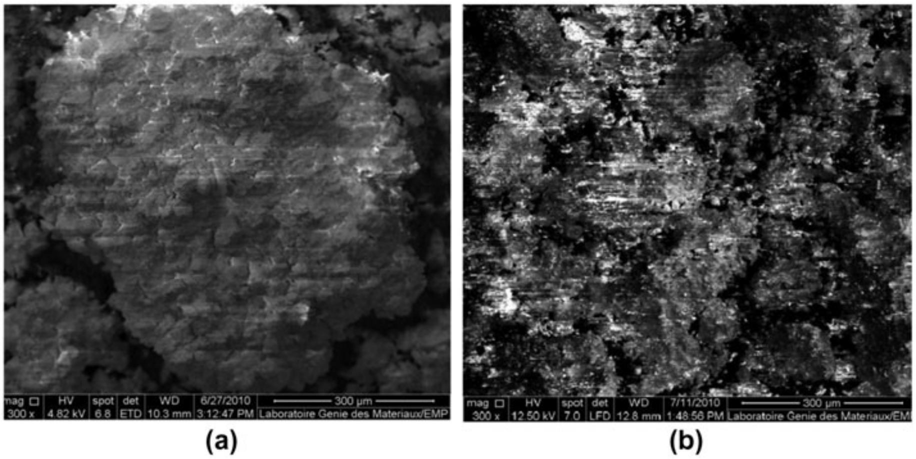


Fig. 2. (a) SEM of AAB before adsorption. (b) SEM of AAB after diazinon adsorption.

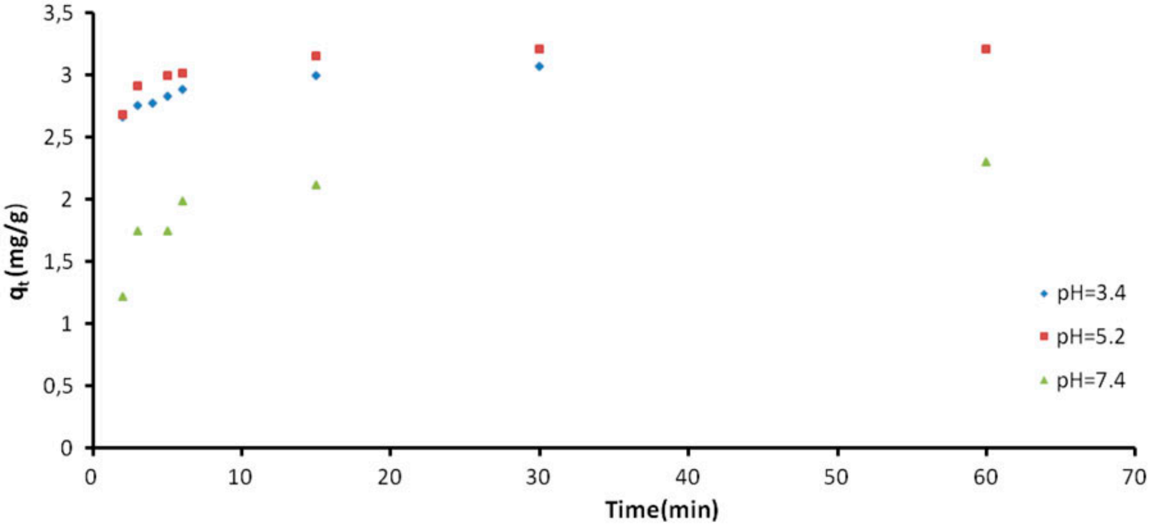


Fig. 3. Effect of initial pH on adsorption capacity of pesticide. Initial pesticide concentration 10 mg/L and Bentonite dosage 3 g/L.

temperature (20°C). From Fig. 5, it is evident that adsorbent dose significantly influences the amount of pesticide adsorbed. With an increase in bentonite dosage from 1 to 2 g/L the adsorption capacity decreases from 8 to 4.2 mg/g, whereas the percentage of removed diazinon increases from 75 to 85%. The increase in the percentage removed can be explained by the increasing surface area where the adsorption takes place [17]. However, further increase of adsorbent dose resulted in very less increase in adsorption; and hence, 1 g/L was considered the optimum dose.

3.6. *Adsorption isotherms*

In order to optimize the design of an adsorption system to remove diazinon from solutions, it is important to establish the most appropriate correlation for the equilibrium curve. Several models have been intensively used in the literature to describe experimental data of adsorption isotherms. The most widely used are the Langmuir, Freundlich, Dubinin–Radushkevich (D–R), and Temkin isotherms [27].

The calculated model constants including the regression coefficient R^2 along with model equation for the adsorption of diazinon by AAB are listed in Table 2.

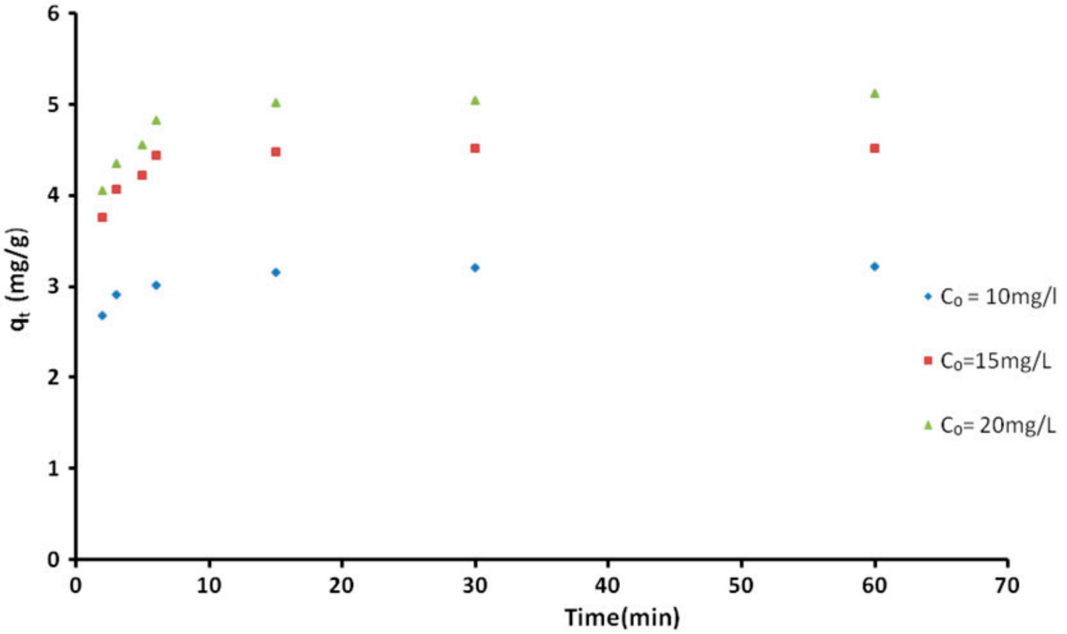


Fig. 4. Effect of contact time on adsorption capacity at different initial pesticide concentrations with pH 5.2 and bentonite dosage 3 g/L.

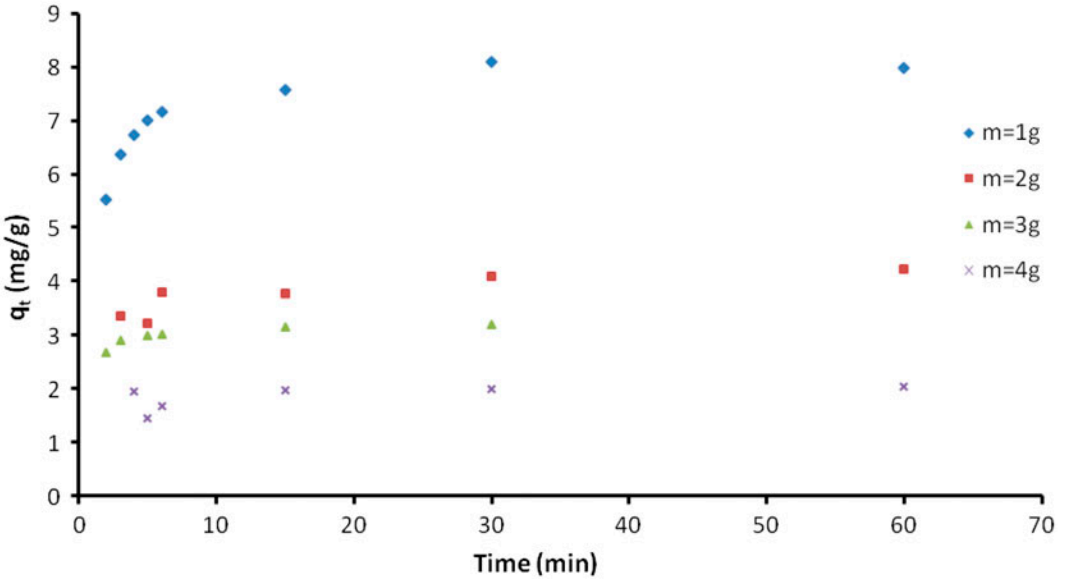


Fig. 5. Effect of bentonite dosage on adsorption capacity at pH 5.2 and temperature 20°C with contact time.

Fig. 6 depicts the comparison of experimental and predicted amount of diazinon adsorbed on AAB for all the isotherm models studied. Clearly, Langmuir isotherm best fits the experimental equilibrium data. This was confirmed by the robust fit of the equilibrium data to the Langmuir isotherm ($R^2 = 0.9963$). The equilibrium sorption of diazinon was shown to proceed by monolayer coverage of the AAB surface by diazinon. The maximum adsorption capacity of 5.563 mg/g for diazinon was observed from the Langmuir isotherm. The Langmuir separation factor, R_L, lies between 0 and 1, which represents that the removal of diazinon is favorable [28].

Table 2
Isotherm parameters for diazinon adsorption onto acid-activated bentonite

Models	Equations	Values
Langmuir	$q_e = \frac{bC_e q_{\max}}{1+bq_{\max}}$	
b (L/mg)	$R_L = \frac{1}{1+bC_0}$	3.107
$q_{\max}$ (mg/g)		5.563
R_L		0.031
R^2		0.9963
Freundlich	$q_e = K_F C_e^{\frac{1}{n}}$	
K_F (L/g)		3.583
n		3.57
R^2		0.7335
Dubinin–Radushkevich (D–R)		
q_m (mg/g)	$q_e = q_m \exp\left(-\beta\varepsilon^2\right)$	5.202
β (mol^2/J^2)	$\varepsilon = RT\ln\left(1+\frac{1}{C_e}\right)$	0.005
E (J/mol)		
R^2	$E = \frac{1}{\sqrt{-2\beta}}$	10
		0.797
Temkin	$q_e = \frac{RT\ln a_T C_e}{b_T}$	
a_T (L/g)		4.1969
b_T (J/mol)		2,613.51
R^2		0.8512

A few reports could be found on the adsorption of diazinon on solid adsorbents [23,29]. Lemic et al. [29] analyzed the adsorption of diazinon by organo-zeolites and reported a maximum adsorption capacity of 1.35 µg/g. Research by Moussavi et al. [23] observed maximum adsorption of 250 mg/g for diazinon onto activated carbon prepared from agricultural waste-based NAC. The adsorption capacity of diazinon obtained in this study is higher that obtained by Lemic et al. [29] and smaller than that obtained by Moussavi et al. [23]. A distinct advantage of using acid-activated bentonite as adsorbent is its lower cost and its economic feasibility in comparison to commercial carbon imported in Algeria (US $ 355 per ton). The bentonite granules costs US $ 10.0 per ton and considering the cost of transport, chemicals, electrical energy used in the process, and labor, the finished product would cost approximately US $ 32.0 per ton. Furthermore Algeria possesses bentonite reserves estimated at millions of tons that need to be valorized through the diversification of industrial prospects [30].

3.7. Thermodynamic analysis

The free energy change (ΔG^0), enthalpy change (ΔH^0), and entropy change (ΔS^0) were calculated using equations described in our earlier work [31]. The values of these parameters summarized in Table 3 indicate the sensitivity of the adsorption process toward temperature.

The enthalpy change, ΔH^0 is negative (exothermic) due to the decrease in adsorption with successive increase in temperature. Negative ΔG^0 values dictate spontaneous process, revealing increased randomness at the solid solution interface during the fixation of the pesticides on the active sites of the adsorbent. Since the adsorption process is exothermic, it follows that under these conditions, the process becomes spontaneous because of the negative entropy change.

3.8. Adsorption kinetics

The pseudo-first-order and pseudo-second-order models were used to test adsorption kinetics data in

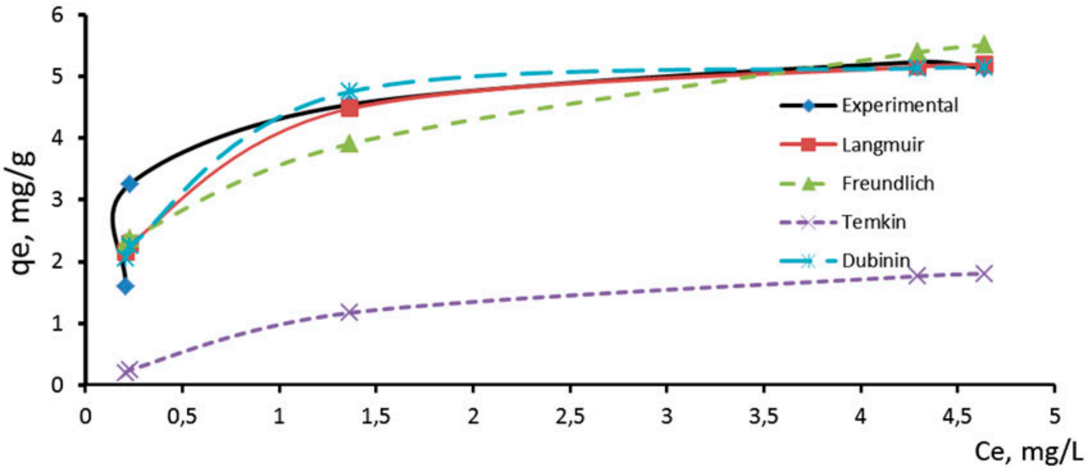


Fig. 6. Adsorption isotherm fitting for adsorption of diazinon onto AAB (20˚C, pH 5.2).

Table 3
Thermodynamic parameters for the adsorption of diazinon onto AAB

		ΔG° (kJ/mol)			
ΔH° (kJ/mol)	ΔS° (J/mol °K)	284 K	293 K	303 K	313 K
−45.28	−16.4	−6.339	−5.381	−4.511	−2.183

order to investigate the mechanism of adsorption and were analyzed as discussed earlier [21]. The values of the kinetic parameters as well as the regression coefficients (R^2) at three different temperatures are listed in Table 4.

The value of correlation coefficient R^2 for the pseudo-second-order adsorption model is relatively high (>0.99). However, the values of R^2 for the pseudo-first-order model are not satisfactory. Therefore, it has been concluded that the pseudo-second-order adsorption model is more suitable to describe the adsorption kinetics of the diazinon over AAB meaning the controlling rate step is chemisorption [26]. Fig. 7 indicates that pseudo-second-order kinetic model better approximates the kinetics of diazinon adsorption onto AAB.

The possibility of intra-particle diffusion was explored using the intra-particle diffusion model [28]:

$$q_t = k_{\text{int}} t^{0.5} + C \tag{3}$$

where k_{int} represents the rate constant (mg/g min$^{0.5}$) and C (mg/g) is a constant reflecting the significance of the boundary layer or mass transfer effect.

If the Weber and Morris [28] plot of q_t versus $t^{0.5}$ satisfies the linear relationship with the experimental data, then the sorption process is found be controlled by intra-particle diffusion only. However, if the data exhibit multi-linear plots, then two or more steps influence the sorption process. It is assumed that the external resistance to mass transfer surrounding the particles is significant only in the early stages of adsorption. This is represented by first sharper portion. The second linear portion is the gradual adsorption stage with intra-particle diffusion dominating. Fig. 8 presents the plots of q_t versus $t^{0.5}$ for the removal of diazinon. It is evident that from Fig. 8 that adsorption follows two phases, viz (I) a linear phase in which instantaneous extremely fast uptake takes place and (II) a quasi-stationary state. In phase (I), about 90% of diazinon was uptaken by AAB within a $t^{0.5}$ value of 2.5 min.

This is attributed to the instantaneous utilization of the most readily available adsorbing sites on the adsorbent surface. Phase (II) may be attributed to a very slow diffusion of the adsorbate from the surface film into the mesopores of the adsorbent. In Fig. 8, the data points are related by two straight lines. The first straight portion depicts boundary layer diffusion effects or external mass transfer effects [24] while the second linear portion is due to intraparticle or pore diffusion. Extrapolation of the linear portions of the plots back to the y-axis gives the intercepts which provide the measure of the boundary layer thickness. The deviation of straight lines from the origin (Fig. 8) may be due to difference in the rate of mass transfer in the initial and final stages of adsorption. Further, such deviation of straight line from the origin indicates that pore diffusion is not the sole rate-controlling step [32].

Kinetic data were further used to know about the slow step occurring in the present adsorption system using Bangham's equation [17].

Table 4
Kinetic model parameters and correlation coefficients for adsorption of diazinon at different temperatures onto AAB

	The pseudo-first-order model		The pseudo-second order model			Bangham model		
T (°C)	k_1 (min^{-1})	R^2	k_2 (g mg^{-1} min^{-1})	q_e (mg/g)	R^2	k_0 (ml/g/l)	α	R^2
11	0.703	0.470	0.561	3.953	0.999	491.216	0.096	0.844
20	0.216	0.164	0.759	3.247	1	333.636	0.067	0.946
30	0.709	0.470	1.477	2.873	0.999	304.419	0.035	0.834

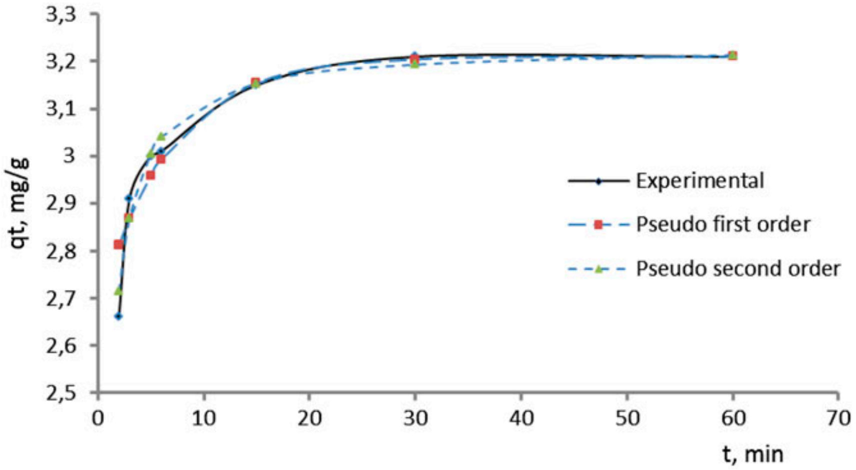


Fig. 7. Kinetic plots for diazinon adsorption onto AAB at 20˚C.

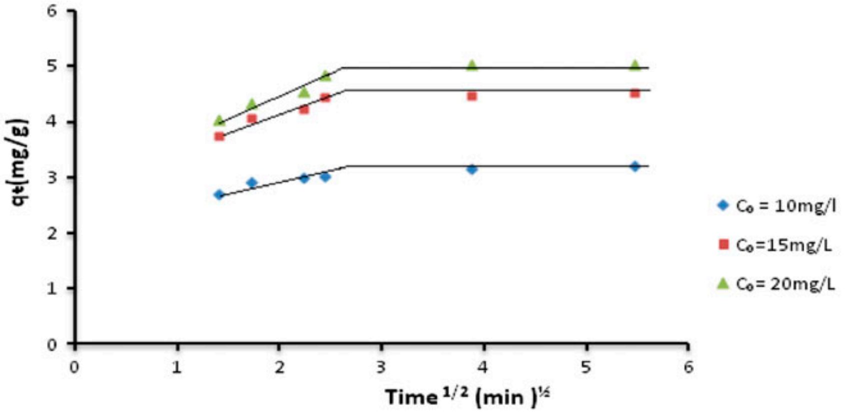


Fig. 8. Intraparticle diffusion plots for adsorption of diazinon at different initial concentrations.

$$\log\log\left(\frac{C_0}{C_0 - q_t m}\right) = \log\left(\frac{k_\circ m}{2.303V}\right) + \alpha\log(t) \quad (4)$$

where C_0 is initial concentration of adsorbate (mg/L), V the volume of solution (mL), m the weight of adsorbent used per liter of solution (g L^{-1}), q_t (mg g^{-1}) the amount of adsorbate retained at time t, and $\alpha(\prec 1)$ and k_0 are constants. $\log\log\left(\frac{C_0}{C_0-q_t m}\right)$ values plotted against $\log t$ (not shown here) did not yielded perfect linear curve for diazinon removal by AAB showing that the diffusion of adsorbate into pores of the sorbent is not the only rate-controlling step [17].

4. Conclusions

The present study shows that the AAB is an effective adsorbent for the removal of diazinon from aqueous solution. Batch adsorption studies revealed maximum adsorption occurring at a contact time of 60 min, low pH, and at room temperature. Adsorption kinetics followed pseudo-second-order model. The adsorption processes could well be described by two-stage diffusion. Langmuir isotherm showed favorable adsorptive removal of diazinon onto AAB.

The cost and adsorption characteristics favor AAB to be used as an effective adsorbent for the removal of diazinon pesticide from aqueous solution.

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