Equilibrium and Thermodynamic Studies on the Adsorption of an Organophosphorous Pesticide onto "Waste" Jute Fiber Carbon

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Adsorption of the monocrotophos pesticide from aqueous solution using "waste" jute fiber (JF) has been investigated. Adsorption equilibrium, kinetics, thermodynamics, and mass transfer studies were investigated as a function of agitation time, adsorbent dose, pH, and temperature. The adsorption kinetics were analyzed by using pseudofirst-order and pseudosecond-order kinetic equations. The adsorption isotherm data were fitted well by the Langmuir isotherm model, and the adsorption capacity was found to be 124 $\mathrm{mg \cdot L^{-1}}$. Thermodynamic parameters of the adsorption process, that is, ΔG, ΔS, and ΔH, were calculated, and their negative values indicate the adsorption was exothermic and had a spontaneous nature. The effective diffusion coefficients were calculated with respect to the variation in concentration and temperature. A single-stage batch adsorption process was also designed by using the adsorption isotherm data.

Introduction

Chemical substances have been used by humans to manage pests from the beginning of agriculture. The term pest comprises insects, weeds, mammals, and microbes, among others. A pesticide is any substance, natural or synthetic, invented to control any pest that fights with humans for food, demolishes property, or spreads illness.[1] At present, because of the potential toxic effects of pesticides on human health and on the environment, there are harsh policies for their registration and use all over the world, particularly in developed countries. The worldwide utilization of pesticides is about two million tons per year, of which 24 % is consumed in the United States alone, 45 % in Europe, and 31 % in the rest of the world. In addition many older, nonpatented, more toxic, environmentally persistent, and low-cost chemicals are used widely in developing nations, creating severe sensitive health problems and local and global environmental impacts.[2] The issue of pesticide use in agriculture is of particular importance as it has a major negative impact on farmers' health. Pesticide pollution not only affects human health but also affects multiple other environmental factors, for example soils, surface and ground water, crop productivity, micro-organisms, and flora and fauna, and so forth. Humans are exposed to pesticides by different routes of exposure such as inhalation, ingestion, and dermal contact. Pesticide introduction has also been associated with high cancer risks and reproductive problems in agricultural workers.[3] Once a pesticide is introduced into the environment, whether by application, disposal, or a spill, it can be influenced by many processes. Among the commonly employed pesticides, organophosphorous pesticides (OPPs) are generally favored over organochlorine pesticides.[4] They are generally more toxic than organochlorine compounds. OPPs are extensively used all over the world and are commonly found in surface and ground waters.[5] In general, it has been observed that OPPs are responsible for deaths in more than 70 % of pesticide death cases.[6]

OPPs, such as methylparathion, malathion, dimethoate, phosphamidon, and monocrotophos, are still extensively used worldwide because of their high toxicity. Monocrotophos (dimethyl(*E*)-1-methyl-2-(methylcarbamoyl) vinyl phosphate) is an OPP. It is very toxic to birds and is used as a bird poison. It is also very harmful to mammals. It is used to control a variety of pests on cotton, sugar cane, peanuts, ornamentals, and tobacco.[7] The Environmental Protection Agency (EPA) classifies monocrotophos as class I toxicity: highly toxic.[8] Cases of human poisoning are characterized by muscular weakness, unclear vision, generous perspiration, uncertainty, vomiting, pain, and small pupils. There is a risk of death due to respiratory failure.[9] Hence, it is necessary to reduce the usage of pesticides and remove them from the environment. Various treatment techniques and processes have been used to remove pesticides from contaminated water, including a membrane filtration method, photodegradation method, solid-phase extraction method,[10] ion-exchange method, chlorination and ozonation method, flocculation method, electrostripping voltammetric method, and adsorption.[11−13] Among all, adsorption is one of the most popular methods and is currently considered as an effective, efficient, and economic method for water purification.[14−17] Activated carbon has a special place among adsorbents.[18,19] Currently, activated carbons are manufactured from a variety of starting materials, that is, precursors, biowaste, steel industry waste,[20] baggasse, ricebran, rice husk,[13] bagasse fly ash,[17] jute fiber (JF),[21] and fly ash.[22]

In this paper, we study the adsorption of monocrotophos from aqueous solution using "waste" jute fiber carbon (JFC). JF is a

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10.1021/je1005906

best fiber having a meshy structure, which is basically cultivated in India and Bangladesh. Traditionally, it is used as a packaging material and for cheap decorative purposes. The availability of this waste material is wide in India, and basically it is made up of cellulosic material. Hence, it can be effectively used for the adsorption of pesticides. The prime objective of the present study is to check the feasibility and assess JFC as an adsorbent in the removal of monocrotophos from aqueous solutions. The effects of agitation time and initial pesticide concentration, temperature, pH of the pesticide solution, and adsorbent dose have been studied using batch adsorption experiments. Various kinetic, isotherm, thermodynamic, and mass transfer investigations and a batch adsorber system are used to study the adsorption process. UV adsorption spectroscopy was used to analyze the concentration of monocrotophos before and after adsorption onto JFC.

Methods and Materials

Preparation of Activated Adsorbent. "Waste" JF was collected from jute industries in Tamilnadu, India. Sun-dried "waste" JF was treated with sulfuric acid (SA) in the weight ratio 4:3 (JF/SA) and kept in a hot air oven, maintained at 160 °C for a period of 12 h. The resulting carbon was washed with distilled water until a constant pH of the slurry was reached. The carbon was then dried for 4 h at 100 °C. The dried material was ground and labeled as JFC.

Adsorbate. Commercially available monocrotophos was used without further purification, as a model pesticide. All chemicals used were analytical reagent grade and were obtained from Qualigens (Mumbai, India). A stock solution of 1000 mg·L^{-1} of the pesticide was prepared by dissolving the pesticides in doubly distilled water. Initially, the pesticide was dissolved in a small quantity of methanol, before the preparation of a buffered solution. The glassware used was of Scott Duran (Germany). In the absence of carbon, controlled experiments were also carried out for the adsorption of pesticides by the container walls. It was noted that there was no detectable degradation or absorption of pesticides by the container walls.

All experiments were done in a fume hood; also, polyethylene gloves and goggles were used for safety in the use of pesticides in the laboratory. Pesticides are packed and labeled properly in the local language with safety instructions.

Batch Adsorption Studies. Batch adsorption studies were performed at room temperature; 50 mL of different initial monocrotophos concentrations, (10, 20, 30, and 40) mg·L^{-1}, was taken in 100 mL conical flasks containing 0.25 g of "waste" JFC. The flasks were agitated at room temperature (28 °C) at 120 rpm for predetermined time intervals using a thermostatic rotary orbital shaker with an initial pH of 6.57. At time $t = 0$ and at equilibrium, the monocrotophos was measured using a Shimadzu UV−vis spectrophotometer (model: UV 1601). The maximum absorbance of pesticide was read at 230 nm, and it was used to calculate the amount of pesticide adsorbed, q (mg·g^{-1}). The adsorption values were measured before and after treatment at their respective adsorption maximum wavelength. The effect of adsorbent dose was studied by varying the carbon concentration [(0.2 to 4.5) g·L^{-1}] for different pesticide concentrations [(10, 20, 30, and 40) mg·L^{-1}]. Langmuir isotherm studies were carried out by agitating a fixed concentration of JFC (0.3 g·L^{-1}) of monocrotophos solutions of different initial monocrotophos concentrations, (60, 80, 100, 150, 200, 250, and 300) mg·L^{-1}, for 6 h. For the pH study, 50 mL of 20 mg·L^{-1} and 40 mg·L^{-1} monocrotophos solution was mixed with 0.25 g of JFC and agitated at different initial pH (2 to 10) values of monocrotophos solution for 3 h at 28 °C. Hydrochloric acid and sodium hydroxide solutions were used to adjust the pH. Temperature studies were also carried out with 50 mL of 40 mg·L^{-1} monocrotophos solution with the effect of agitation time for four different temperatures [(28, 33, 38, and 43) °C] to evaluate the effect of temperature on the adsorption process. Generally, the carbon concentration of 0.5 g·L^{-1}, pH 6.57, and room temperature, 28 °C, are maintained in all experiments, unless otherwise stated.

Table 1. Consolidated Table for the Kinetics of Monocrotophos Adsorption[a]

concentration	pseudofirst order			pseudosecond order		
mg·L^{-1}	$K_1\cdot10^{-2}$ min^{-1}	R^2	SSE	$k_2\cdot10^{-2}$ min^{-1}	R^2	SSE
10	3.045	0.9732	6.901	1.264	0.9375	8.520
20	2.853	0.9873	4.328	6.418	0.9746	6.141
30	1.716	0.9107	9.488	5.067	0.8968	16.225
40	2.087	0.9766	2.361	6.101	0.9976	5.573

[a] Conditions: adsorbent dose: 0.5 g·L^{-1}; adsorbent particle size: (0.25 to 0.35) mm; temperature: 28 °C.

Results and Discussion

Characterization of "Waste" JFC. The C/H/O/N/S present in the "waste" JFC was analyzed (carbon 52.66 %, hydrogen 3.23 %, oxygen 43.05 %, nitrogen 0.14 %, sulfur 0.93 %) by using an elemental analyzer (model CHNO-RAPID, Heraeus Co., Germany). The contents (moisture 12.22 %, ash 2.69 %, volatile matter 61.28 %, fixed carbon 23.81 %) were determined by the proximate analysis method according to ASTM 3172-3175 standards. A pH$_{\text{zpc}}$ value (6.56) was also measured.

Adsorption Kinetics. For evaluating the adsorption kinetics of OPPs on JFC, the pseudofirst-order $\log(q_e - q_t)$ versus t (figure not shown) and the pseudosecond-order t/q_t versus t (figure not shown) kinetic models were used, and the data are presented in Table 1. The amount of pesticide adsorbed at equilibrium, both experimental (q_e^{exp}) and calculated (q_e^{cal}), the linear regression coefficient, R^2, and sum of the squares of errors (SSE) values are considered for the evaluation of suitable model for adsorption kinetics. The linear fit is better when SSE values are low. The q_e^{exp} and q_e^{cal} values are not compliable for both pseudofirst-order and pseudosecond-order kinetic models (high SSE values). However, the linear regression coefficient values, R^2, for the pseudofirst-order kinetics are closer to unity compared to that of pseudosecond-order kinetics. Therefore, the pseudofirst-order kinetic model can explain the adsorption of monocrotophos more appropriately.

Effect of Contact Time and Initial Pesticide Concentration on Adsorption of Monocrotophos. The percent removal increases with increases in contact time of the pesticide studied and decreases with an increase in the initial pesticide concentration. The unit adsorption for monocrotophos was increased from (15.21 to 39.84) mg·g^{-1} as the monocrotophos concentration increased from (10 to 40) mg·L^{-1}. The curves in Figure 1 are smooth and continuous, suggesting possible monolayer coverage of pesticide on the JFC surface. The initial concentration affects both the equilibrium uptake capacity of the adsorbent and the adsorption rate.[21]

Effect of Carbon Dose. In this study the amount of carbon dose was varied from (0.2 to 4.5) g·L^{-1}. The adsorption increased from (56.32, 40.64, and 36.32 to 100) % for the initial concentrations of (10, 20, and 30) mg·L^{-1}, respectively, and (33.32 to 97.92) % for 40 mg·L^{-1} for the carbon dose of (0.2 to 4.5) g·L^{-1}. The percentage of pesticide removal increased with an increase in carbon dose. It is expected that the increase

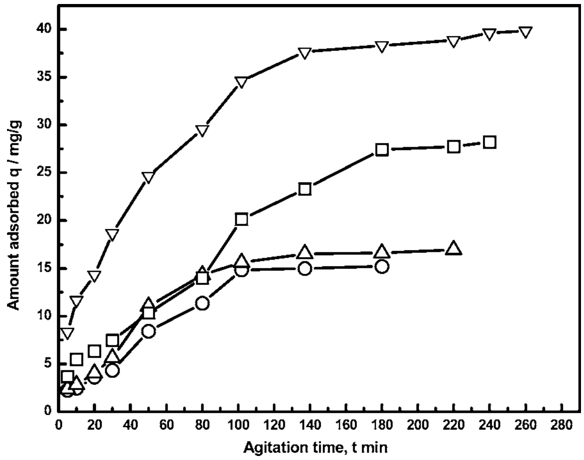

Figure 1. Effect of contact time and initial monocrotophos concentration. ○ = 10 mg·L^{-1}; △ = 20 mg·L^{-1}; □ = 30 mg·L^{-1}; ▽ = 40 mg·L^{-1}. Conditions: initial pH = 6.25; JFC dose = 0.5 g·L^{-1}; temperature = 28 °C.

in the concentration of JFC results in an increase of surface area with more adsorptive sites for the adsorption to occur.

Effect of pH. The percent removal of monocrotophos decreased from (43.32 to 33.01) % for the initial pesticide concentrations of 20 mg·L^{-1} and (40.24 to 30.42) % for the initial pesticide concentrations of 40 mg·L^{-1} in the pH range (2.0 to 10.0) studied. At a very low pH, an attractive force is raised between the adsorbent and the adsorbate molecule. Kyriakopoulos et al.[23] also reported a similar observation for the adsorption onto resin. This attractive force is due to surface functional groups that may be surrounded by the hydronium ion obtained from pesticide solution. At a higher pH, a portion of surface functional groups should become deprotonated. This makes interaction between the adsorbent and the adsorbate molecules weak. $\mathrm{pH_{zpc}}$ can be used as an index of the ability of a surface to become either positively or negatively charged. This ability is controlled by the pH of the surrounding solution. At this pH the net charge is zero, while above that the surface will carry a net negative charge and vice versa. On the basis of the definition of $\mathrm{pH_{zpc}}$, the activated carbon adsorbent will react as a positive surface when $\mathrm{pH_{solution}} < \mathrm{pH_{zpc}}$ and as a negative surface when $\mathrm{pH_{solution}} > \mathrm{pH_{zpc}}$. The $\mathrm{pH_{zpc}}$ of the JFC is equal to 6.56. The adsorption was significantly enhanced in the pH range 3.0 to 6.0. The adsorption capacity increased when $\mathrm{pH} < \mathrm{pH_{zpc}}$.

Adsorption Isotherm. The value Q_o (124.07 mg·g^{-1}), monolayer capacity, and R^2 (0.922) values for the concentration of pesticide from (60 to 300) mg·L^{-1} were calculated by using the Langmuir isotherm equation. The formation of a monolayer coverage of adsorbent on the surface of carbon can be clearly confirmed by the Langmuir isotherm data. The separation factor (R_L) lies between 0 and 1 ($0 < R_L < 1$), suggesting the adsorption of pesticide on the JFC is favorable.

Effect of Temperature. Monocrotophos adsorbed decreased from (39.84 to 27.49) mg·g^{-1} with an increase of temperature from (28 to 43) °C for an initial pesticide concentration of 40 mg·L^{-1} with a fixed JFC concentration (0.5 g·L^{-1}). The temperature increase makes the pesticide more soluble and lowers its affinity for the adsorbent surface.

Thermodynamic Parameters. The thermodynamic parameters, ΔG, ΔH, and ΔS, for the adsorption process were calculated using Eyring's plot,[19] and the values are presented in Table 2. The negative value of ΔS suggests that there is decrease in the state of disorder in the molecule during the adsorption process, which in turn is due to the binding of molecules with the adsorbent surface. A negative value of the enthalpy change, ΔH, indicates that the pesticide−exchanger interaction is exothermic in nature and a weak force of attraction.[24] The value of ΔH less than 40 K·J·mol^{-1} indicates physical adsorption.[25]

Table 2. Thermodynamic Data for the Removal of Monocrotophos

temperature	$-\Delta G^{o}$		$-\Delta S^{o}$	$-\Delta H^{o}$
°C	kJ·mol^{-1}	K_c	J·K^{-1}·mol^{-1}	kJ·mol^{-1}
28	3.816	0.662	85.726	29.620
33	3.388	0.605		
38	2.959	0.495		
43	2.531	0.397		

The negative value of ΔG indicates that the adsorption of the pesticide is spontaneous and feasible.[14] It may also be noted that the Gibbs energy values obtained for the system increase with temperature, which shows that the adsorption of pesticides is more favorable at lower temperatures.[24] The ΔG for physical adsorption is generally in the range of (0 to 20) kJ·mol^{-1}, and that for chemical adsorption is in the range of (80 to 400) kJ·mol^{-1}.[26] From Table 2, the Gibbs energy change during the adsorption process was in the range of (2.531 to 3.818) kJ·mol^{-1}.

Mass Transfer Studies. On the basis of the Weber and Morris theory, the intraparticle diffusion coefficient K_{id} (mg·g^{-1}·min$^{-0.5}$) was calculated. A good correlation of q_e experimental with q_e calculated, $R^2 > 0.9$ (near to one), and low SSE values of this model justifies the mechanism for the diffusion of pesticides. The calculated rate constants for monocrotophos varied from (1.35 to 3.30) mg·g^{-1}·min$^{-0.5}$. The rate constants for the intraparticle diffusion were found to increase with the concentration of pesticide.

To determine the actual rate-controlling step involved in the monocrotophos adsorption process, the adsorption data were further analyzed using the kinetic expression given by Boyd.[27] This is in accordance with the observations of Reichenberg.[28] For every calculated value of the factor, F, a fraction of solute adsorbed at different times t, corresponding values of B_t are obtained from the Reichenberg table, the values of the effective diffusion coefficient of adsorbate in the adsorbent phase, D_i, are given in Table 3. In each case, with respect to the variation in concentration and also with the variation in temperature for a fixed pesticide concentration (40 mg·L^{-1}), the plot of B_t versus t (Figures 2 and 3) distinguishes between the film-diffusion and particle-diffusion-controlled rates of adsorption. The B_t versus t plots for the adsorption of the pesticide onto JFC shows linearity in the entire concentration range under study, but the straight lines do not pass through the origin, revealing thereby that the rate-determining process is film diffusion for JFC.

The diffusion coefficient, D_i, decreases with an increase in initial concentrations of pesticide. Initially the amount of pesticide adsorbed onto the carbon surface increases rapidly, but then the process slows down and reaches equilibrium. According to Atun et al.,[29] this behavior is attributed to the fact that there is a reduction in immediate solute adsorption because of the lack of enough available open sites to adsorb high initial concentrations of pesticide which in turn supports film diffusion. The simultaneous diffusion of the ingoing pesticides through the pores of different widths and different electronic fields along the diffusion path determine the effective diffusion coefficient D_i. The diffusion within the pores of wider path and weaker retarding forces of electrostatic interaction accounts for the faster component of D_i, and the diffusion within the pores of narrow path and stronger retarding forces accounts

Table 3. Data for the Effect of Concentration and Temperature of Monocrotophos on the Diffusion Coefficient

diffusion coefficient, $D_i/10^{-12}$ m^2·s^{-1}				diffusion coefficient, $D_i/10^{-12}$ m^2·s^{-1}			
10 mg·L^{-1}	20 mg·L^{-1}	30 mg·L^{-1}	40 mg·L^{-1}	28 °C	38 °C	43 °C	48 °C
4.499	4.110	2.099	2.577	2.576	2.117	2.111	1.728
D_o/m^2·s^{-1}		$1.024 \cdot 10^{-15}$		energy of activation, E_a/kJ·mol		17.967	

for the slower component of D_i. As the temperature increases, the contribution of the faster component of D_i decreases (Table 3). This is due to the decreasing mobility of ingoing species at higher temperature which to some extent overcomes the effluence of retarding forces.[14] The energy of activation E_a and pre-exponential factor (D_o) analogous to the Arrhenius frequency factor were also evaluated.

The values of E_a and D_o for the diffusion of the pesticide into JFC are calculated from the slope and intercept of the plot, log D_i versus $1/T$ (plot not given), and are presented in Table 3. Similar results are obtained for the diffusion coefficient, D_i, in the adsorption of dyes onto an agricultural based activated carbon.[21]

Designing of a Single Stage Batch Adsorber. An adsorption isotherm can be used to predict the design of single-stage batch adsorption systems.[30,31] A schematic diagram is shown in Figure 4, where the solution contains V (L) of water and an initial concentration C_o, which is to be reduced to C_1 in the adsorption process. In the treatment stage M (g) JFC is added, and the pesticide concentration on the JFC changes from q_o (initial) to q_1. The mass balance that the pesticide removed from the solution to that accumulated by the solid is

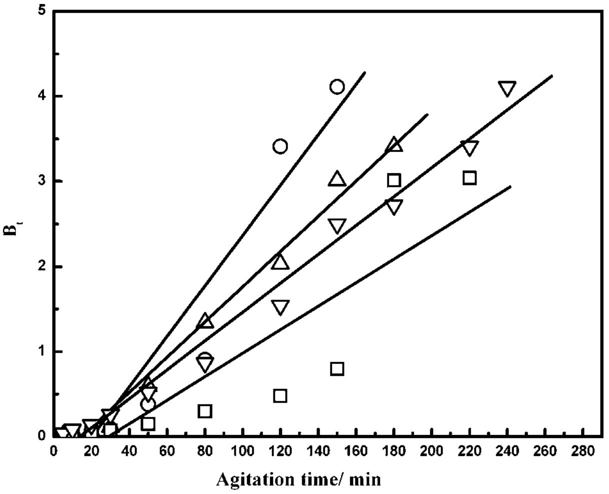

Figure 2. Effect of concentration of monocrotophos on the diffusion coefficient. ○ = 10 mg·L^{-1}; △ = 20 mg·L^{-1}; □ = 30 mg·L^{-1}; ▽ = 40 mg·L^{-1}. Conditions: initial pH = 6.25; JFC dose = 0.5 g·L^{-1}; temperature = 28 °C.

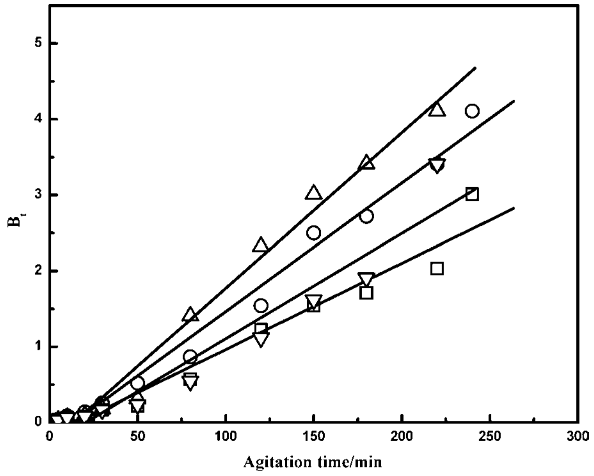

Figure 3. Effect of temperature on the diffusion coefficient of monocrotophos adsorption. ○ = 28 °C; □ = 38 °C; ▽ = 43 °C. Conditions: initial pH = 6.25; JFC dose = 0.5 g·L^{-1}; monocrotophos concentration = 40 mg·L^{-1}.

$$V(C_o - C_1) = M(q_o - q_1) = Mq_1 \tag{1}$$

If the system is allowed to come to equilibrium, then, $C_1 \rightarrow C_e$, and $q_1 \rightarrow q_e$. In the case of the adsorption of pesticides onto JFC the Langmuir isotherm gives the best fit to the experimental data. Consequently, the Langmuir equation can be best substituted for q_1, giving the adsorbent or solution for a given change in pesticide concentration, $C_o - C_e$, in this particular system.

$$\frac{M}{V} = \frac{C_o - C_e}{q_1} = \frac{C_o - C_e}{q_e} = \frac{C_o - C_e}{\dfrac{K_L C_e}{1 + a_L C_e}} \tag{2}$$

where K_L and a_L are the Langmuir constants. From the plot of M versus V (plot not given) the required amount of JFC to reduce the pesticides content by 90 % at various volumes of solution are derived. For example, if 10 L of the solution is to be treated, the required mass of JFC is 8.925 g for 90 % pesticide removal.

Conclusion

An agricultural waste byproduct, JFC, was found to be effective for the removal of monocrotophos from aqueous solution. The kinetics of sorption follows a first-order rate equation. On increasing the pH of the solution, adsorption decreases. Thermodynamic data reveal that the adsorption was exothermic and spontaneous in nature and also follows the Langmuir model. The film diffusion mechanism was confirmed by a mass transfer study. As the temperature increases, the

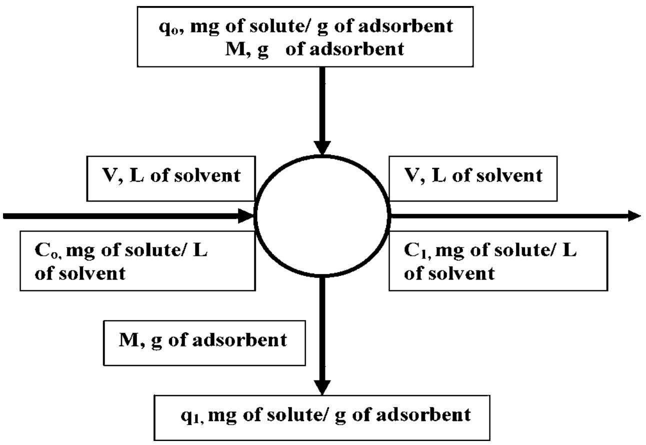

Figure 4. Single stage batch adsorber design.

contribution of the faster component of D_i decreases. This is due to the decreasing mobility of the ingoing species at higher temperatures. A single stage batch adsorber was successfully designed for the removal of monocrotophos.

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Received for review May 29, 2010. Accepted October 31, 2010. The authors sincerely thank the Quantum Functional Semiconductor Research center (QSRC), Dongguk University-Seoul, South Korea for its research funds.

JE1005906