

Adsorptive Removal of Monocrotophos via Low Cost and Natural Adsorbent

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Abstract

Perlite was used for monocrotophos (MCP) adsorption from waste effluents. The surface and functional groups were characterized via spectrometric routes. The concentrations of 12, 20, 28, and 36 µg/mL were prepared, and equilibrium was reached in 120 minutes for the 12 µg/mL MCP-containing sample. The equilibrium took place in 220 min for the remaining samples. Langmuir, Freundlich, and Jovanovich were the isotherms used for explaining the adsorption. The monolayer capacity was 78.2 mg/g at 50°C. The n constant of the Freundlich isotherm (approximately 1.29 at 50°C) supported chemisorption ($n > 1$). Pseudo-first- and pseudo-second-order mechanisms were applied to the system. Comparing the experimental and calculated capacity deviations of the first- and second-order models, the first-order model showed better average values, with 0.1467 instead of 0.2224. The pseudo-first-order model governed the adsorption reaction, and the inner-pore adsorption mechanism (Weber-Morris kinetic model) was also investigated. The Weber-Morris constant was 0.33 mg/g.min^{1/2} at 50°C. The film and pore diffusion behavior were explained. The adsorption process was also analyzed. The Gibbs energy was calculated as -2276 J/mol. It was an indicator of the feasible adsorption nature.

Categories: Chemical Thermodynamics and Separation Processes, Environmental Engineering, Environmental Engineering and Sustainability

Keywords: boyd, ftir, perlite, kinetic, thermodynamic

Introduction

Fresh water resources of the world have been lessening because of the careless use by human beings. There are many reasons for this situation, including environmental and global changes. The contamination by various organic pollutants and heavy metals is rather dangerous because of their possible effects and carcinogenic nature [1,2]. The surface and ground waters of the world are contaminated due to the high toxicity and nonbiodegradability of these pollutants. Therefore, the importance of fresh water quality is essential for life [3].

Monocrotophos [dimethyl (E)-1-methyl-2-(methylcarbamoyl)vinyl phosphate, MCP] is an organophosphorus pesticide. It is employed for controlling insects in vegetable crops [4]. MCP is extremely toxic, causing severe effects on the eyes, nerves, and the human body [5,6].

Various techniques have been developed in order to get rid of these undesired effects [7-9]. These methods include physical or chemical treatments [10]. Another method is based on the effect of light [11].

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Various researches published about the removal of contaminants. Meimand et al. studied the sulfur dioxide adsorption by iron oxide nanoparticles@clinoptilolite/HCl composite [12]. Rajabş et al. published a paper related to the adsorption of petrochemical organic pollutants from industrial process streams [13]. Shi et al. studied the adsorption of MO and 2,4-D by green-synthesized bamboo hydrochar. The Langmuir isotherm supported the adsorption, and the monolayer capacity of the material was found to be 1088.51 and 731.73 mg/g for MO and 2,4-D Na, respectively [14]. Clebot et al. published a paper on the adsorption of two herbicides, 2,4-D and acetochlor, and three insecticides, namely, cypermethrin, dimethoate, and imidacloprid. The adsorption isotherms of the pesticides showed sigmoidal curves, revealing a dual adsorption effect. Vermicompost-containing biomixtures showed the highest adsorption capacity for acetochlor (7129-7394 mg/kg) and cypermethrin (5950-6190 mg/kg), and the lowest adsorption for dimethoate (520-1409 mg/kg) [15]. Yang et al. studied the adsorption of neonicotinoid pesticides on typical soil minerals. Those pesticides have the potential to pollute the ground and groundwater resources. The maximum adsorption capacity was found at a 0.005-40 solid-to-liquid ratio. The Na⁺, K⁺, and Ca soil minerals exert a substantial influence on the adsorption capacity of clayey soil. The adsorption followed a first-order kinetic model, and the Freundlich isotherm represented the data very well [16]. Aluminosilicates and silica-containing materials have a large surface area, pore width, and functional groups [17]. This paper presents a comprehensive study of the adsorption kinetics and thermodynamics of MCP under different experimental conditions using waste effluent.

Materials And Methods

Materials

Monocrotophos (C₇H₁₄NO₅P (CH₃O)₂PO-OC(CH₃)=CHCO-NHCH₃; 98%) was procured from the Agriculture Provincial Directorate of Forestry in Istanbul, Turkey. Perlite obtained from Bartın, Türkiye, has a clay composition. The particle size was 250-mesh. Clayey ingredients are beneficial in agricultural applications [18]. Some mineral components may be helpful for pollutant remediation. It was dried in an oven before the experimental trials. The structural composition is presented in Table 1.

Content	B ₂ O ₃	SO ₄ ²⁻	CaO	Na ₂ O	SiO ₂	MgO	SrO	Al ₂ O ₃	Fe ₂ O ₃	As ₂ O ₃	Heat loss
wt, %	14	2	13.53	6.87	13.8	11.8	1.15	0.94	0.35	0.0018	35.5582

TABLE 1: Perlite chemical constituent

The surface morphology of the clayey sample was examined using a JEOL JSM-5410LV scanning electron microscope (SEM). A UV spectrophotometer (UV-150-02) operating at 396 nm and a WTW pH meter were employed. FT-IR spectra were recorded using an Optima SP 300 over the wavenumber range of 4000-450 cm⁻¹.

Batch adsorption studies

A stock solution of 100 ppm was prepared, and the experimental samples were diluted with distilled water.

The experimental parameters were pH (1.0-7.0), initial MCP concentration (12-36 µg mL⁻¹), contact time (1-3 h) and temperature (20-50°C).

Adsorbent availability were determined below (Equation 1).

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$$q = \frac{(C_0 - C_e)V}{m} \quad (1)$$

where q is the sorption capacity (mg g^{-1}); C_0 and C_e are the initial and residual concentrations (mg L^{-1}); V is the volume (L); and m is the amount of the adsorbent (g)

Experimental data

Langmuir, Freundlich and Javanovich isotherms were tested.

Langmuir isotherm is (Equation 2) [19]:

$$\frac{C_e}{q_e} = \frac{1}{bq_0} + \frac{C_e}{q_0} \quad (2)$$

where q_e and C_e were defined. b is constant (g/mg).

Freundlich isotherm is represented as (Equation 3) [20]:

$$\log q_e = \log K_F + \frac{1}{n} \log C_e \quad (3)$$

where n and K_F values are isotherm constants. K_F is the adsorbent capacity.

Jovanovich [21] is at below Equation (4), The constants are found from the slope and intercept.

$$\ln q_e = \ln k_{\max} + K_f C_e \quad (4)$$

Where $q_{\max}$ (mg/g) and K_f (l/g) are the maximum adsorption capacity and the Jovanovich isotherm constant, respectively.

The pH of the solution was monitored using 0.1 N HCl or 0.1 N NaOH solutions. Some kinetic models and Boyd plot have been used.

Lagergren Equation (5) is as follows [22]:

$$\log(q_e - q_t) = \log q_e - \frac{k_1}{2.303} t \quad (5)$$

where k_1 is the first rate constant (min^{-1}).

Pseudo-second order model is presented as [23] (Equation 6):

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \quad (6)$$

where k_2 is the second rate constant ($\text{g mg}^{-1} \text{min}^{-1}$).

Intraparticle diffusion model [24] is given as Equation (7)

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$$q_t = k_{id}t^{0.5} + C \quad (7)$$

where k_{id} ($\text{mg/g min}^{0.5}$) and C (mg/g) are rate constant, and boundary layer constant, respectively.

Boyd plot was applied [25] to understand the inner particle diffusion mechanism.

$$F = 1 - \frac{6}{\pi^2} \exp(-B_t t) \quad (8)$$

To compute B_t , the related arrangements were performed, and the kinetic expression becomes:

$$F = \frac{q_t}{q_e} \quad (9)$$

where B_t is a constant and F is the fractional attainment of equilibrium at time t time (Equation 10).

$$B_t = -0.4977 - \ln \left(1 - \frac{q_t}{q_e} \right) \quad (10)$$

Thus, B_t vs time relationship gives Boyd plots.

Thermodynamic parameters

The thermodynamic parameters were shown shown below [26].

$$\log \frac{q_e}{C_e} = \frac{\Delta S^\circ}{2.303R} - \frac{\Delta H^\circ}{2.303RT} \quad (11)$$

$$\Delta G^\circ = \Delta H^\circ - T\Delta S^\circ \quad (12)$$

where q_e/C_e is called the adsorption affinity. ΔG° shows spontaneity in the case of negative values [27]. The constants are calculated from the defined values (Table 2).

Plot	Relationship	Slope	Intersect
First order	$\text{Log}(q_e - q)$ vs t	k_1	$q_e \text{ cal}$
Second order	t/q_t vs t	$q_e \text{ calc}$	k_2
Intraparticle diffusion	q_t vs $t^{0.5}$	k_{id}	C
Thermodynamic values	$\text{Log}(q_e/C_e)$ vs $1/T$	ΔH°	ΔS°

TABLE 2: Relationships of the axes of the values

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Results And Discussion

Clayey sample characterization

Fourier-transform infrared spectroscopy (FTIR) spectra were recorded using the Attenuated Total Reflectance (ATR) technique with an 8 cm^{-1} resolution and 100 scans. Figure 1 and Table 3 depict the functional groups.

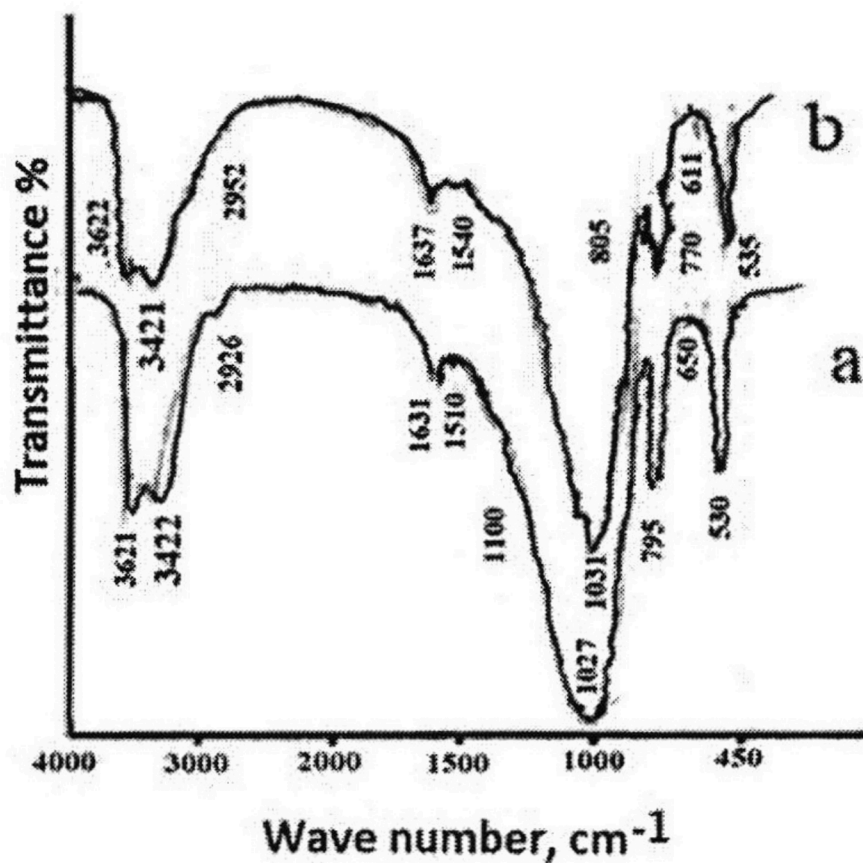


FIGURE 1: FTIR spectra of the samples (a) raw (P), (b) MCP-adsorbed P sample

FTIR, Fourier-transform infrared spectroscopy; MCP, Monocrotophos

Functional groups	Wave number
C-O stretching of mineral parts (MCP)	530, 650, 795 (a), 535 (b) cm^{-1}
CaO or MgO -with H [28]	611, 770 (b) cm^{-1}
[BO ₄]- interaction and B-O with B ₂ O ₃	805 (a) cm^{-1}
C-O stretching organic complex [29]	1027 (a) cm^{-1}
tetrahedral SiO ₄ -H complex [30]	1031, 1100 (b) cm^{-1}
CH ₂ /CH ₃ group of MCP	1540 cm^{-1} (b)
[BO ₃]-triangular complex	1510–1540 (a and b) cm^{-1}
C=C stretching MCP, B ₂ O ₃ group [31]	1631 (a), 1637 cm^{-1} (b)
Aliphatic CH [32]	2926 (a), 2952 cm^{-1} (b)
OH stretching of primer alcohols [33]	3621 cm^{-1} (a), 3622 cm^{-1} (b)
Amine interaction [34]	3421(a) cm^{-1} , 3422 (b) cm^{-1}

TABLE 3: Functional groups of the samples

SEM images of the (P) before (a), and after the experiment (b) are seen in Figure 2, respectively. The presence of more pores on the P surface in Figure 2(b) supports the adsorption process. The surface area and pore volume were 288.15 m^2/g and 78.24 cm^3/g , respectively.

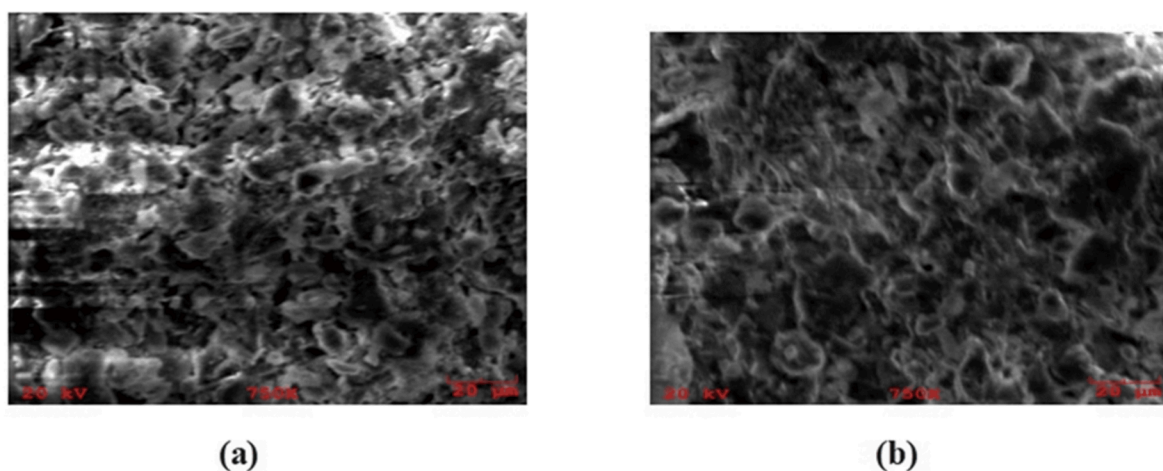


FIGURE 2: SEM images of the adsorbent: (a) raw P and (b) MCP-adsorbed P at a magnification of 350x

SEM, Scanning Electron Microscopy; MCP, Monocrotophos

Effect of pH

The pH results indicated that the maximum uptake was at pH 1 (Figure 3) between the positive clay surface and negative MCP molecules. The results were affected by the surface charge of the clay [35]. Adama and Al-Shammari reported similar behavior for 2,4-D adsorption by activated carbon [36].

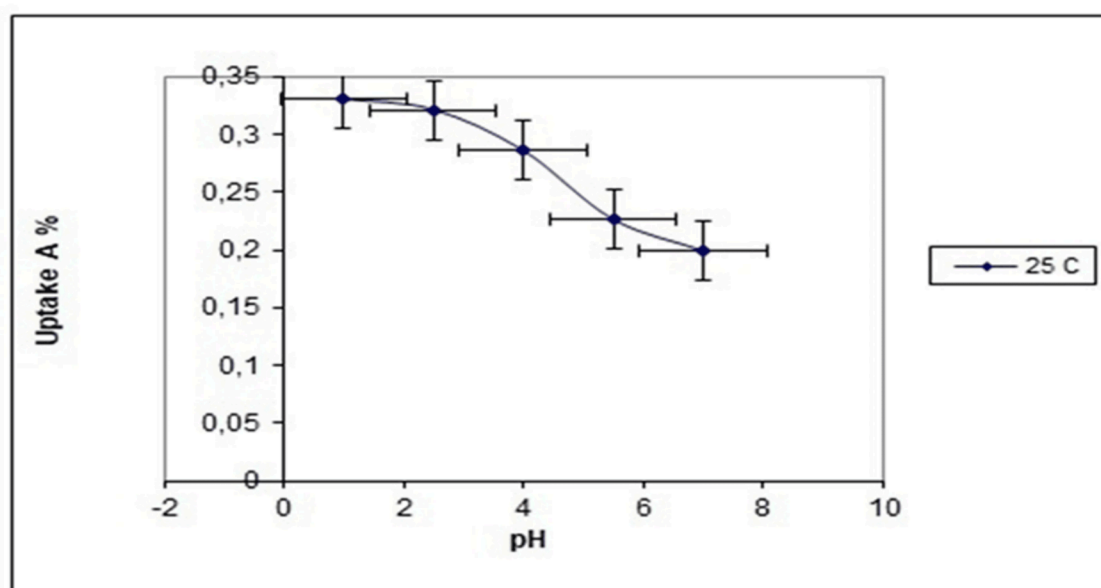


FIGURE 3: Relationship between pH and adsorption percentage

Stable state

The slight leachate of MCP (Figure 4) is due to the empty sites on the adsorbent. The available sites were filled with the MCP entities over time. During the first 30-40 min, MCP was transported to the macropores of the adsorbent. This is a surface reaction. Later, the molecules filled the inner and smaller pores of the clayey adsorbent. The results were similar to those reported in the organophosphate pesticide literature [37].

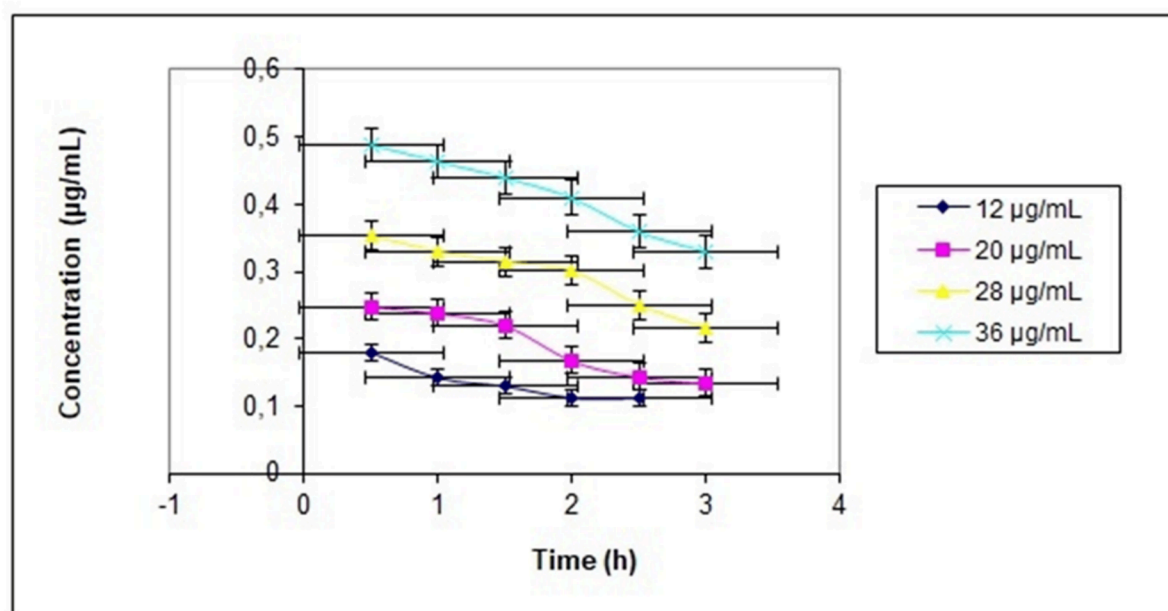


FIGURE 4: Contact time effect

Figure 5 shows the pesticide adsorption via perlite. As shown, the adsorption capacity was increased with the pesticide concentration from 12 to 36 µg/L at 20°C. The equilibrium times were 120 and 220 min for 12 µg/L and the remaining concentrations, respectively. The adsorbent capacity was increased from 0.3 mg/mL to 0.8 mg/mL. The concentration increase is important in the adsorption phenomenon. This is similar to the findings reported in [38].

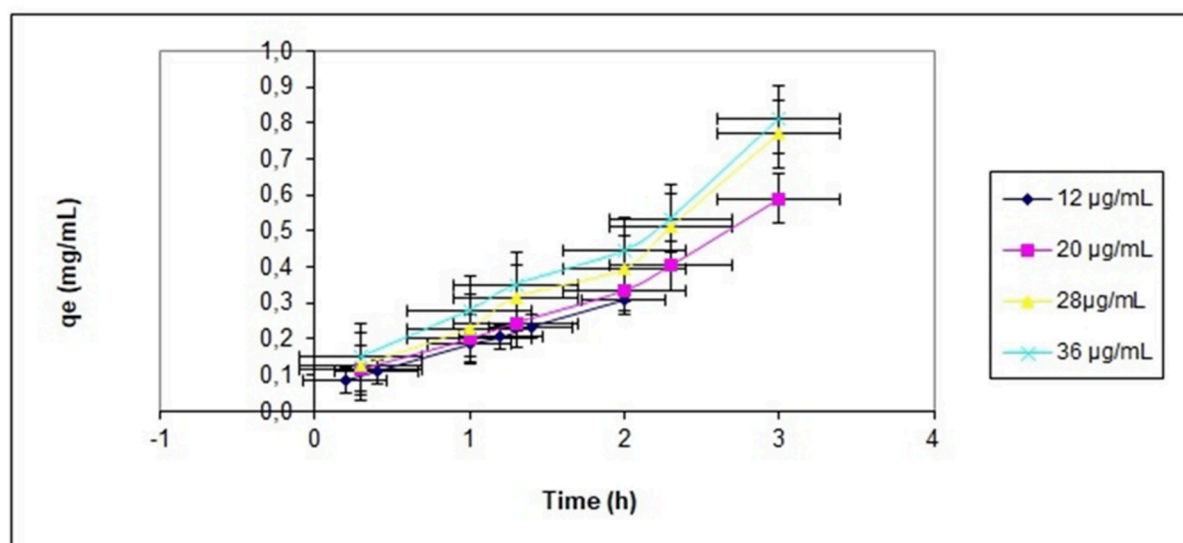


FIGURE 5: Steady state of experiments, pH = 1

Effect of temperature

The temperature effect was studied between 20°C and 50°C (Figure 6). The maximum adsorption capacity was at 50°C, but the increase in temperature was not significant. This is due to the endothermic nature of the adsorption process. The thermodynamic approach was in good agreement with this observation. This is similar to the findings reported in the study [38].

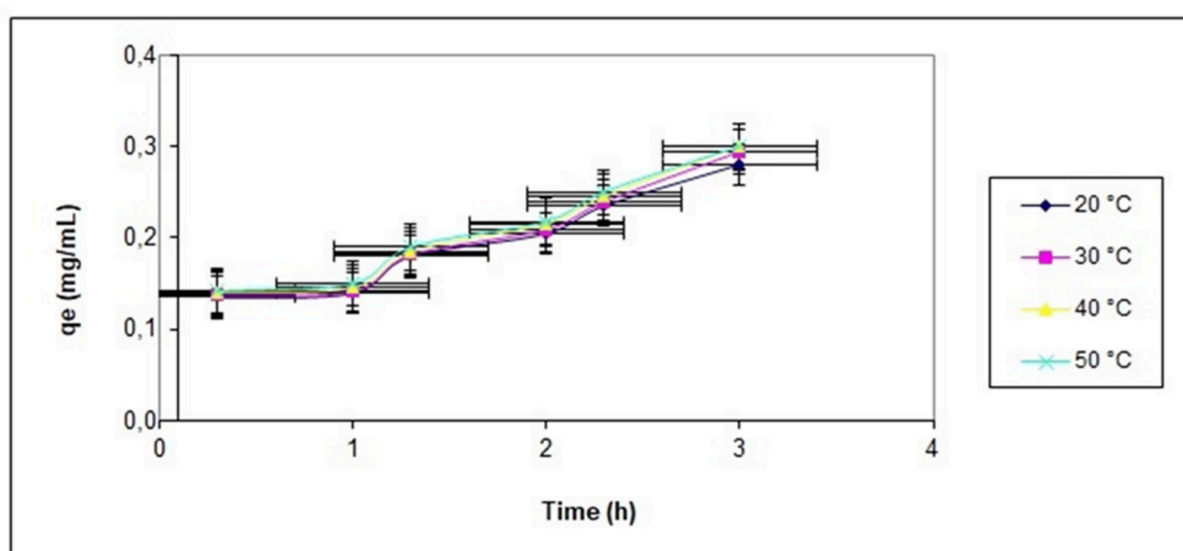


FIGURE 6: Temperature effect on MCP adsorption

MCP, Monocrotophos

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Equilibrium studies

Both Langmuir (Figure 7) and Freundlich isotherms (Figure 8) showed the best fit for the adsorbent (Table 4). The Freundlich isotherm assumes a heterogeneous nature, while the Langmuir isotherm exhibited a homogeneous structure of the adsorbent. Freundlich constants were indicators of physical adsorption due to the $n > 1$ factor. The monolayer adsorption capacity (q_0) increased with an increase in temperature. Langmuir and Freundlich constants were also in good agreement with the increase in temperature.

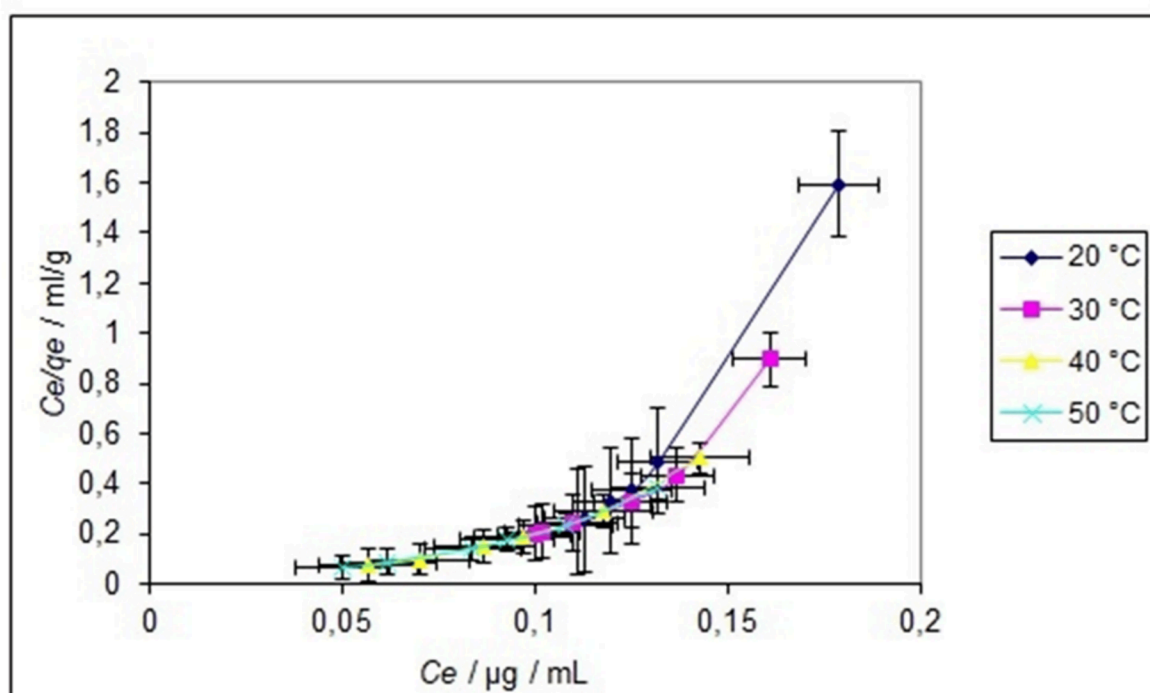


FIGURE 7: Langmuir plot

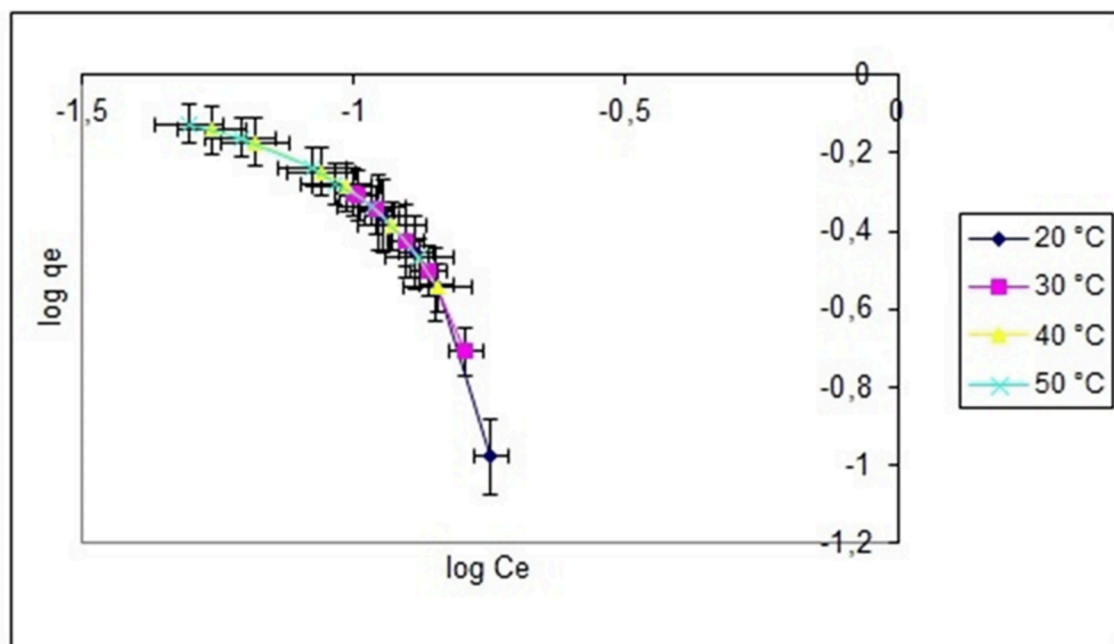


FIGURE 8: Freundlich plot

The Jovanovic isotherm (Figure 9) is very similar to the Langmuir, considering a monolayer and no lateral interactions. The high R^2 values (0.989 and 0.934 at 20°C for the Jovanovic and Langmuir isotherms, respectively) support this observation, as shown in Table 4 [39].

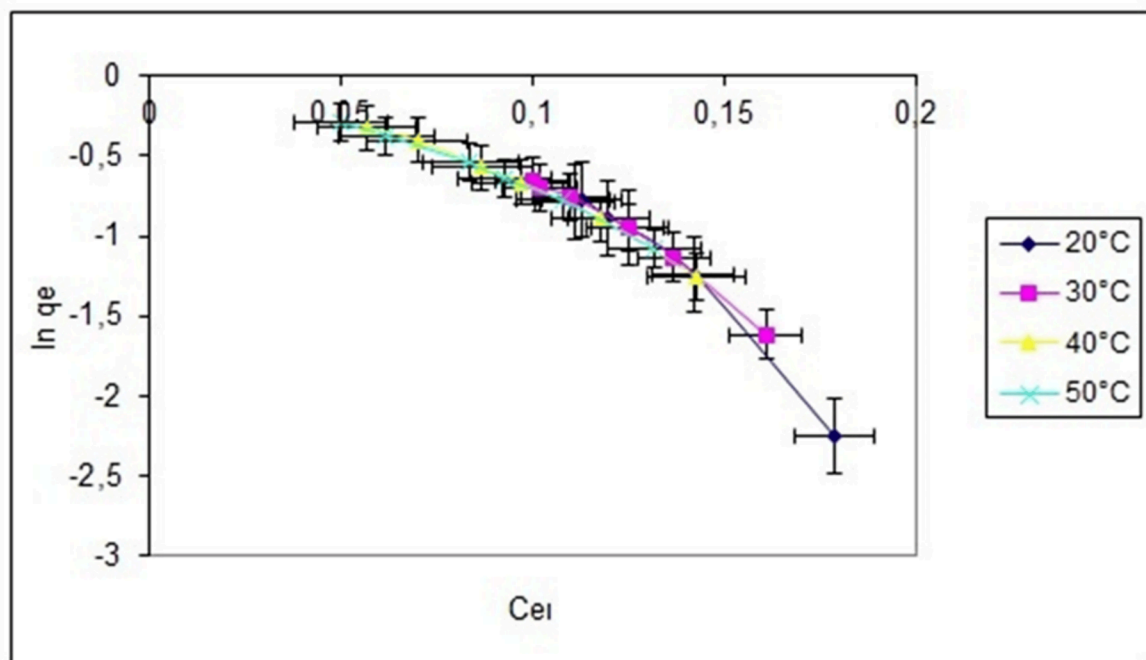


FIGURE 9: Javanovich plot

Langmuir isotherm	20°C	30°C	40°C	50°C
q_0 (mg/g)	0.1025 ± 0.0031	0.2065 ± 0.0040	0.5075 ± 0.0050	0.7821 ± 0.0053
b (L/mg)	9.6525	11.8920	17.8649	19.7620
R^2	0.934	0.985	0.993	0.990
Freundlich isotherm				
K_F mg/g (L/mg) ^{1/n}	7.2862 ± 0.1401	6.7858 ± 0.1344	2.0536 ± 0.1233	1.0782 ± 0.1122
n	0.3364	0.5290	1.0798	1.2864
R^2	0.986	0.987	0.993	0.932
Javanovich isotherm				
q_{max} (mg/g)	0.3092 ± 0.0035	0.3166 ± 0.0039	0.3501 ± 0.0042	0.3708 ± 0.0045
k_j	0.908	0.870	0.742	0.671
R^2	0.989	0.988	0.991	0.993

TABLE 4: Adsorption isotherm constants

Table 5 shows the maximum monolayer capacities of adsorbents. The clay (perlite) has a very large adsorption capacity compared with other adsorbents. It is a natural resource and is also relatively cheap.

Adsorbent	Q _{max} (mg/g)	Pollutant	Ref
Azadirachta Indica biochar	79.40 mg/g	Bentazone	[40]
P-dopant biochar	79.6 mg/g	Triazine	[41]
nnamon carbon	12.37 mg/g	chlorpyrifos	[42]
Eluthrilite Activated with ZnCl ₂ at 700°C	32.10 mg/g	MCPA	[43]
Bismuth oxide nanoparticle	27.38 mg/g	Quetiapine	[44]
Palygorskite OP1CEC	12.24 mg/g	2,4-D	[45]
Clinoptilolite rock	5.60 µg/g 4.99 µg/g	Lindane Aldrin	[46]
Micelle-clay Cloisite 30B	8.32 mg/g 3.47 mg/g	Chlorpyrifos Penconazole	[47]
Perlite	10.25-78.21 mg/g (20°C-50°C)	MCP	This study

TABLE 5: Adsorbent adsorption capacities

MCP, Monocrotophos

Kinetic studies

Table 6 represents the kinetic constants. The pseudo-first-order model (Figure 10) exhibited the best fit due to the high R^2 value and close adsorption capacities. The adsorption was rather rapid at the beginning of the adsorption process. This indicates the surface interaction between MCP and the adsorbent. As shown in Table 6, the deviation values of the second-order model are larger than those of the first-order model. There are complex interactions in the pores. Figure 11 shows the pseudo-second-order trend of MCP adsorption. Basic blue-3 adsorption and poly(sodium 4-styrenesulfonate) (PSS) plus ethyl-4-hydroxybenzoate (EP) adsorption followed pseudo-second-order adsorption kinetics [48], but 2,4-D removal and Congo red adsorption followed first-order kinetics [49,50]. High correlation coefficients for the intraparticle diffusion model validated the model for the adsorbent (Table 7). The MCP adsorption was found to occur very rapidly with intraparticle diffusion (Figure 12). The deviation was also closely related to the calculated data (Table 7).

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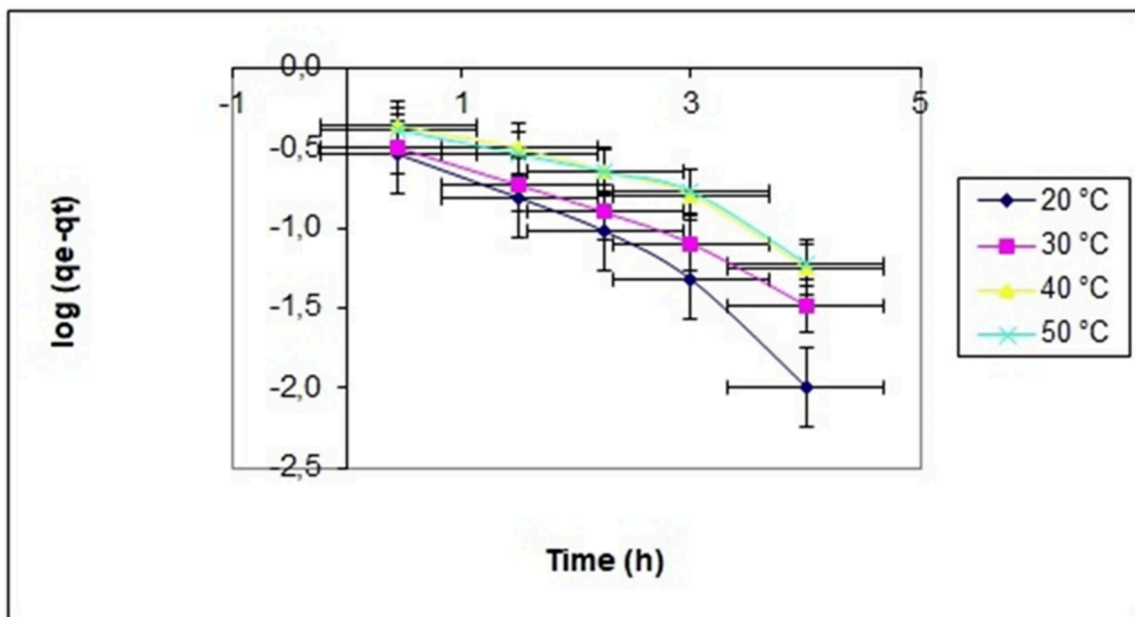


FIGURE 10: Pseudo first-order kinetic plots for 12 µg/mL MCP

MCP, Monocrotophos

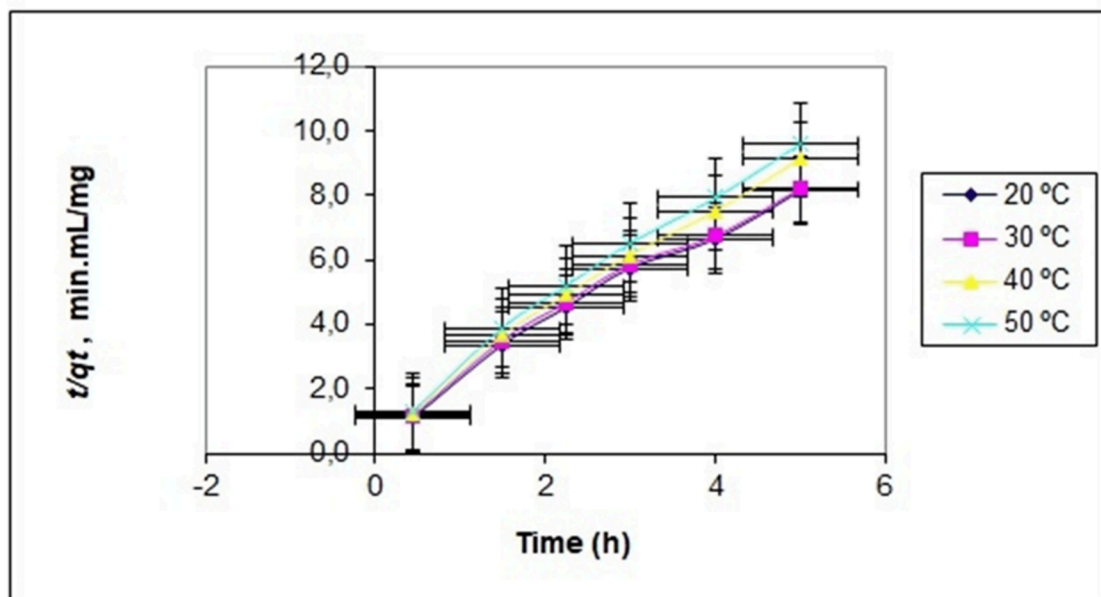


FIGURE 11: Pseudo second-order kinetic plots for 12 µg/mL MCP

MCP, Monocrotophos

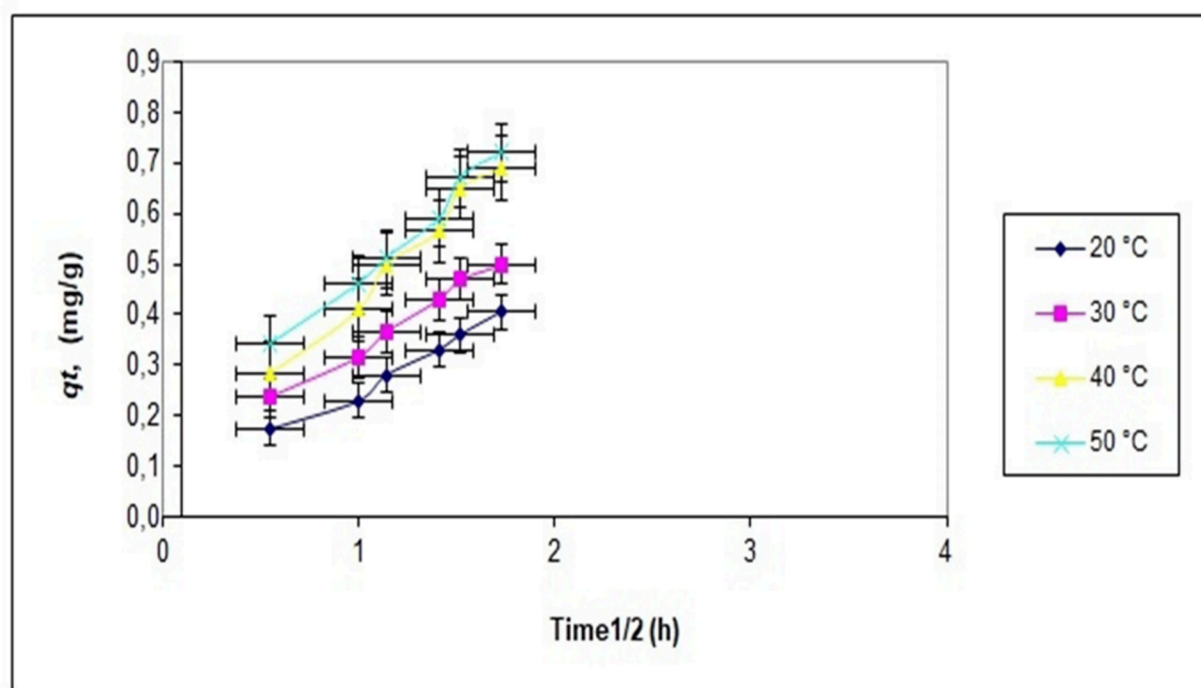


FIGURE 12: Weber Morris plot (intraparticle diffusion)

Co (µg/mL)	Pseudo first-order kinetic				Pseudo second-order kinetic		
	qe,exp (mg/g)	qe,cal (mg/g)	k ₁ (1/min)	R ²	qe,cal (mg/g)	k ₂ (g/mg min)	R ²
12	0.465	0.5750 ± 0.11	0.401	0.9468	0.06720 ± 0.3978	247.5248	0.9812
20	0.63	0.4631 ± 0.1669	0.271	0.9814	0.6686 ± 0.0386	2.2660	0.9778
28	0.795	0.6853 ± 0.1097	0.2449	0.9246	0.5916 ± 0.2034	3.3625	0.9904
36	0.81	0.6097 ± 0.2003	0.2224	0.9103	0.5603 ± 0.2497	11.3254	0.9891

TABLE 6: The kinetic parameters

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Co (µg/mL)	Pseudo first-order kinetic				Pseudo second-order kinetic		
	q _{e,exp} (mg/g)	q _{e,cal} (mg/g)	k ₁ (1/min)	R ²	q _{e,cal} (mg/g)	k ₂ (mg/g min ^{1/2})	R ²
12	0.465	0.5750 ± 0.11	0.401	0.9468	0.06720 ± 0.3978	247.5248	0.9812
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36	0.81	0.6097 ± 0.2003	0.2224	0.9103	0.5603 ± 0.2497	11.3254	0.9891

TABLE 7: Weber-Morris coefficients for 12 µg/mL MCP

MCP, Monocrotophos

Boyd plot (Figure 13) was also applied to investigate the intraparticle diffusion phenomenon [29]. A straight line through the origin is shown for IPC; otherwise, there is a film diffusion effect [51]. The plots did not pass through the origin (Figure 13) and showed both effective film and intraparticle diffusion mechanisms. The surface interaction (film diffusion-lateral line trend) and inner pore interactions (straight line trend) both supported this fact.

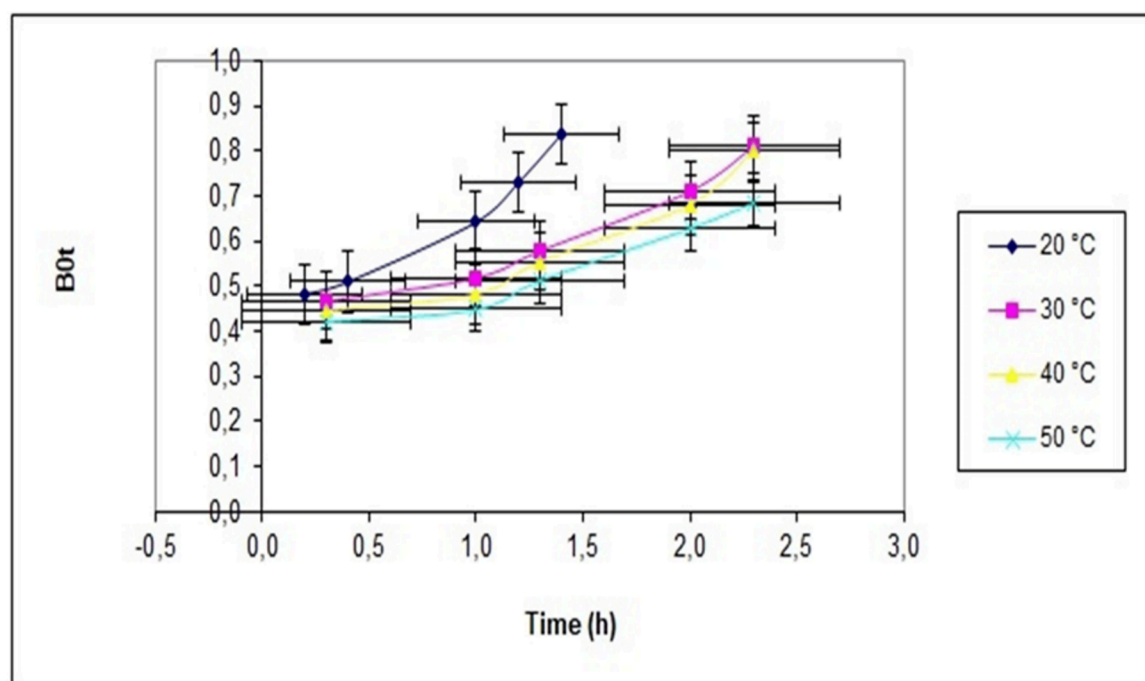


FIGURE 13: Boyd plot

Thermodynamic studies

Figure 14 shows the thermodynamic relationship. The process (Table 8) was spontaneous due to the negative ΔG° values. This fact indicated the feasibility of the clayey adsorbent for the adsorption process. It is quite effective and relatively cheap compared with other adsorbents such as activated carbon. The positive ΔH° values indicated endothermic adsorption. The adsorption reaction for the endothermic processes could be due to the increase in temperature, which increased the rate of diffusion of the adsorbate molecules across the external boundary layer and into the internal pores of the adsorbent particle, owing to the decrease in the viscosity of the medium [52]. The positive ΔS° values indicated increased randomness at the solid/solution interface. Entropy increased with increasing temperature. This is similar to the findings reported in [53,54]. The paper by Dhaif Allah et al. reports negative ΔH° and ΔS° values [55].

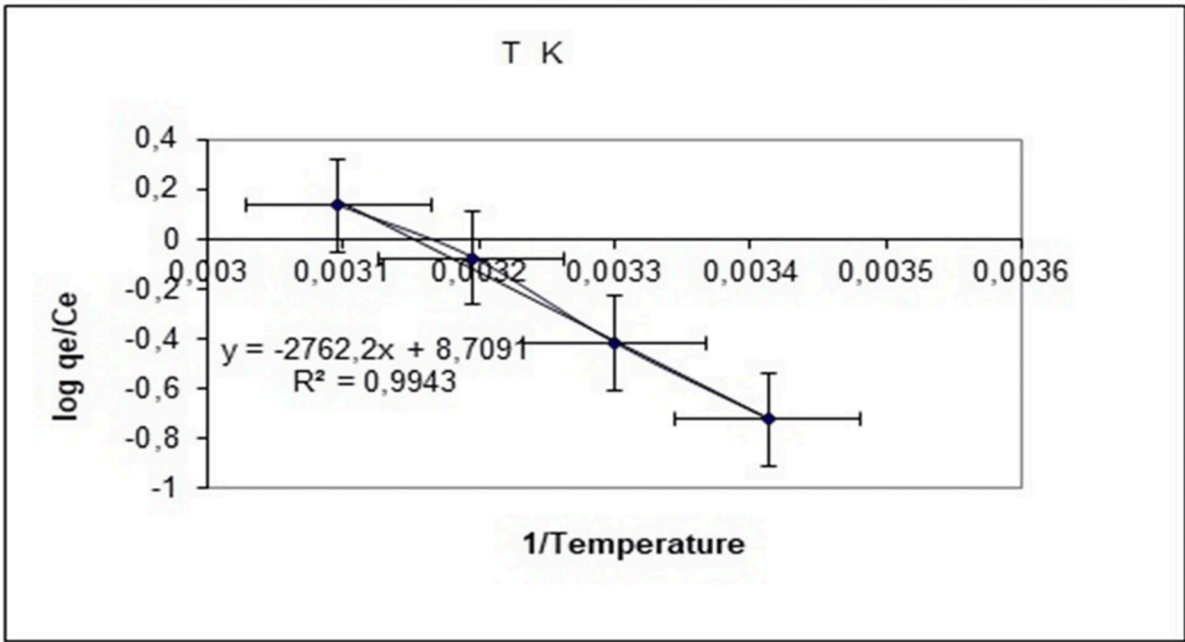


FIGURE 14: Plot of $\log(q_e/C_e)$ versus $1/T$

T(K)	ΔH° (J/mole)	ΔS° (J/mole)	ΔG° (J/mole)
293	47144.09	168.67	-2276.22
303	51999.19	174.31	-3962.92
313	56838.29	179.95	-5649.62
323	61685.39	185.59	-7336.32

TABLE 8: Thermodynamic values

Conclusions

MCP adsorption experiments were performed at temperatures of 20, 30, 40, and 50°C with 12, 20, 28, and 36 µg/mL concentrations. The adsorbent material was natural perlite, which has a porous nature. The maximum removal (55%) and uptake (0.465 mg g⁻¹) of MCP were achieved via perlite with a concentration of 12 µg/mL MCP at room temperature. Langmuir and Freundlich isotherms showed a good relationship. The Jovanovic isotherm also showed a high regression

coefficient. The monolayer capacity was 78.2 mg/g at 50°C. First- and second-order mechanisms were compared. First-order kinetic constants were found as 0.401 (0.9468), 0.271 (0.9814), 0.2449 (0.9246), and 0.2224 (0.9103) min⁻¹. The first-order kinetic model provided a better fit. IPD and film diffusion mechanisms were effective in the adsorption process. Film diffusion and pore diffusion were explained via the Boyd plot. The Boyd plot did not pass through the origin, which supported this behavior. The enthalpy, entropy, and Gibbs free energy values were found as 47,144.09, 168.67, and -2,276.22 J/mol, respectively. The thermodynamic calculations indicated that adsorption was feasible and spontaneous in nature. The outlook for perlite as an adsorbent is optimistic, suggesting its feasible applications and enhancing its widespread applications.

Additional Information

Author Contributions

All authors have reviewed the final version to be published and agreed to be accountable for all aspects of the work.

Concept and design: Jale Gülen, Sevda Karademir

Critical review of the manuscript for important intellectual content: Jale Gülen

Supervision: Jale Gülen

Acquisition, analysis, or interpretation of data: Ömer Karpuz, Sevda Karademir

Drafting of the manuscript: Ömer Karpuz, Sevda Karademir

Disclosures

Human subjects: All authors have confirmed that this study did not involve human participants or tissue.

Animal subjects: All authors have confirmed that this study did not involve animal subjects or tissue.

Conflicts of interest: In compliance with [the ICMJE uniform disclosure form](#), all authors declare the following:

- **Payment/services info:** All authors have declared that no financial support was received from any organization for the submitted work.
- **Financial relationships:** All authors have declared that they have no financial relationships at present or within the previous three years with any organizations that might have an interest in the submitted work.
- **Other relationships:** All authors have declared that there are no other relationships or activities that could appear to have influenced the submitted work.

Data Availability Statements

The datasets (and/or code) supporting this study are available from the corresponding author upon reasonable request.

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