Adsorption of Ciprofloxacin Hydrochloride from Aqueous Solution by Iraqi Porcelinaite Adsorbent

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Abstract

Pharmaceuticals are widely distributed in different applications and also released into the environment. Adsorption of Ciprofloxacin HCl (CIPH) on Porcelinaite was studied at ambient conditions. The adsorption isotherms can be well described using the Freundlich and Temkin equations. The pH of the solution influences significantly the adsorption capacity of Porcelinaite, the adsorption of CIPH increased from the initial pH 1.3 and then decreased over the pH rang of 3.8-9. The adsorption is sensitive to the change in ionic Strength, which indicate that electrostatic attraction is a significant mechanism for sorption process. The enthalpy change (ΔH) for the adsorption of CIPH onto Porcelinaite signifies an endothermic adsorption. The ΔG value is negative at all studied temperatures, inferring that, the adsorption of CIPH onto porcelinaite will follow a spontaneous trend. The ΔG value decreased when the temperature increased from 15 ^{o}C to 37.5 ^{o}C, suggesting increase in adsorption of CIPH with increasing temperature. The positive value of ΔS reflects the affinity of porcelinaite towards drug and also suggests increased randomness at the solid-solution interface.

Keywords**:** Adsorption, Ciprofloxacin HCl, Freundlich and Temkin isotherms, Porcelinaite, Thermodynamic parameters.

Introduction

The impact and removal of conventional pollutants, such as persistent organic pollutants was in focus for the last decades and so their behavior is well understood [1]. On the other side very little is known about the behavior of trace pollutants yet, which are present in the environment in extremely low concentrations.

Pharmaceuticals are an example for the variety of man-made trace pollutants that are introduced in surface or subsurface water bodies [2]. Pharmaceuticals have been identified in the environment, including antibiotics, analgesics, psychiatric drugs, and natural and synthetic hormones [3]. Unused human pharmaceuticals may also enter the environment through landfill leachate [4-7].

Many types of adsorbents such as kaolin [8,9], charcoal [10], polymers [11] attapuligate [12] and bentonite [13-17] in adsorption of drug, are recognized in clinical practice and environmental treatment. The safety, high adsorptive capacity, there low density and the high specific surface, have been accepted for a long time, and they account for most of the current uses of clay.

Pharmaceuticals poisoning also has been defined as a condition produced by any substance which when swallowed, inhaled, injected or absorbed precutaneously is capable of causing death, injury, toxic or untoward reactions [18].

Ciprofloxacin hydrochloride is a synthetic chemotherapeuticantibiotic (1-cyclopropyl-6-fluoro-1,4-dihydro-4- oxo-7-(1-piperazinyl)-3-quinoline carboxylic acid), Its empirical formula is $C_{17}H_{18}FN_3O_3$ and its molecular weight is 331.4 g/mol Fig.1, Ciprofloxacin hydrochloride is a broad-spectrum antimicrobial agents belonging to the fluoroquinolone group Its mode of action depends upon blocking bacterial DNA replication by binding itself to an enzyme called DNA gyrase, thereby preventing the enzyme's ability to untwist the DNA double helix, which is required for DNA replication [19].

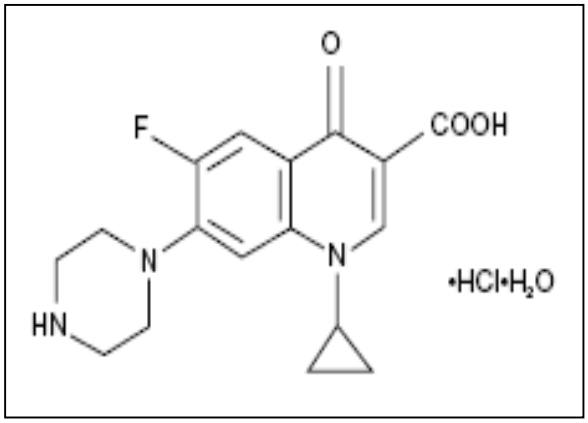

Fig. (1) Formula of Ciprofloxacin Hydrochloride.

The adsorption capacity of ciprofloxacin hydrochloride (CIPH) was determined on three types of carbon-based materials: activated carbon (commercial sample), carbon nanotubes (commercial multi-walled carbon nanotubes) and carbon xerogel [20]. also studied adsorption capacities on montmorillonite, rectorite, and illite [21], charcoal and talc [22], Chitosan [23]. Various studies have shown that CIPH is susceptible to direct photochemical transformations by exposure to direct ultraviolet (UV) light and by adding photocatalytic reagents such as hydrogen peroxide (H_2O_2), and titanium dioxide(TiO_2), to aqueous solutions [24].

In this study for the first time we investigate the adsorption of Ciprofloxacin Hydrochloride on the surface of Iraqi porcelinaite. The system variables studied include sorbent dose, contact time, FTIR analysis, initial concentration of the drug, PH, ionic strength and temperature.

Experimental Process

Materials and apparatus

The drug employed in this research (Ciprofloxacin Hydrochloride) was obtained from (state company for drug industries and (porcelinaite) employed in this study were obtained from open mine in area of the western desert- Iraq supplied from (The general company for Geological survey and mining), Baghdad, The mineralogical composition of the adsorbent is shown in the Table (1). Sodium Chloride (fluka) and Hydrochloric acid (BDH). The clay in powder forms were washed several times with excessive amounts of distilled water. The adsorbent were dried at (120 ^{O}C) in an oven (D-6450) hand (160 ^{O}C) for 3h and then kept in airtight containers. The clay was then ground and sieved by using a test sieves (Retsoh Gmb& Co. KG, Germany) sieve. The particle size of 75 μm was used for the clay in the experiments of this work, Thermostated shaker bath (Lab tech-Korea), pH meter (HI98107, Hanna Instruments) then filtered by using filter paper Wattman No. 42 and centrifuge(Hettich EBA20) The spectrophotometric absorbencies did by (SHIMADZU 1800) double beam UV- Visib-spectrophotometer, FTIR, IR Affinity (SHIMADZU, Japan).

Table (1)
Chemical composition of Iraqi Porcelinaite minerals.

Element	*% wt.*
$O_{(total)}$	44.55
Si	25.04
Fe	0.30
Al	0.77
Ca	4.29
Ti	0.07
Na	12.97
L.O.I	12.00

Methods

Standard solution of Ciprofloxacin Hydrochloride drug made in distilled water by dissolving 1gm of drug in 1000 ml. UV-Visible scanning spectrum has been recorded and wavelength value corresponding to the maximum absorption found to be at (275 nm), this value utilized for measurements of estimation throughout this research. To determine adsorption isotherms for the drug with porcelinaite surface, solution of different concentrations for this drug were prepared by serial dilutions in the range of (2-14 mg/L). Adsorbent surface samples 0.1 g was weighed by using electrical balance. Each sample was then placed in a screw cap bottle and 10 ml of serial drug solution was added to each sample. The bottles were put in thermo stated shaker at different temperature (15, 25 and 37.5^{o}C). The shaking was continued for a period exceeding the time to attain equilibrium for the adsorbents. The pH of solution was adjusted

with HCl and NaOH solutions and ionic strengths (0.2, and 0.4M Sodium Chloride solution). At the end of the adsorption period, the rotation was stopped and the solution was filtered by using Filter paper then centrifuged. The clear supernatants were assayed for drug, spectrophotometrically. The adsorbed amount of the drug was calculated from the concentration in solutions before and after adsorption according to the equation (1):

$$Qe = x/m = (Co-Ce)V/W \quad \text{..... (1)}$$

Where Co and Ce are the initial and equilibrium liquid-phase concentrations of drug solution(mg/L), respectively, Qe is equilibrium drug concentration on adsorbent (mg/g), V is the volume of drug solution (L), and W is the mass of clay sample used (g).

The percentage of drug removal was determined using the equation (2):

$$\%removal=(C_o-C_e)x100/C_o \quad \text{..... (2)}$$

From the Beer's plot for the Ciprofloxacin Hydrochloride drug previously made, the amount of free drug in solution was determined. From the results, the time to attain equilibrium for adsorbent was determined.

Result and Discussion

Effect of initial drug concentrations

The adsorption capacity of porcelinaite for CIP was determined at different initial drug concentrations (2 - 14 ppm). The results were represented in Fig. (2) show that the drug amount sorbed increase with the increase of drug concentration but the percent of drug removal decrease at high concentration. The increase in adsorption with the increase in CIP concentration is due to the driving force that initial concentration provides to overcome the mass transfer resistance between the aqueous and solid phases.

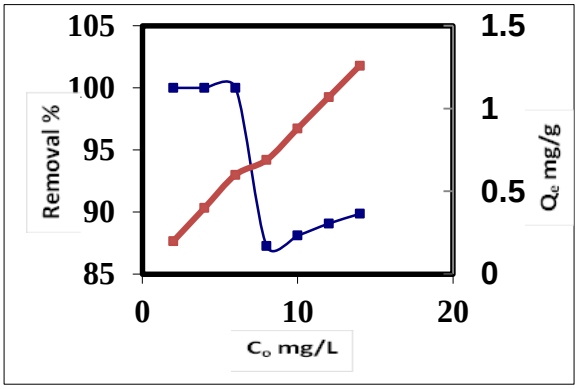

Fig.(2) The influence of initial drug concentration on the adsorption of CIPH on porcelinaite (T=37.5Co, pH= 3.8 ,P. size 75µm).

Effect of adsorbent dose

The dependence of adsorption of the drug on the amount of porcelinaite was studied by varying the adsorbent dose from 0.05 to 1 g at temperature (37.5°c) and at their optimal pH, while keeping the volume and concentration of the drug solution constant. The results are given in Table (2) and graphically represented in Fig. (3). The Fig. indicates that maximum sorption at 0.1g and then there was no further increase of sorption. It is evident that the maximum removal of drug 84.716% at 10 mg/L concentration was obtained with 0.1 g of Porcelinaite. The adsorption of the drug decreased rapidly with increase in the dose of the adsorbent.

Table (2)
Effect of adsorbent dose on adsorption of CIPH at 10 mg/L.

Wt. (g)	*Ce(mg/L)*	*Removal %*
0.05	1.6133	83.867
0.1	1.5284	84.716
0.2	1.6860	83.140
0.3	1.7709	82.291
0.4	1.8193	81.807
0.5	1.8921	81.079
0.6	1.9406	80.594
0.8	2.1466	78.534
1.0	2.2921	77.079

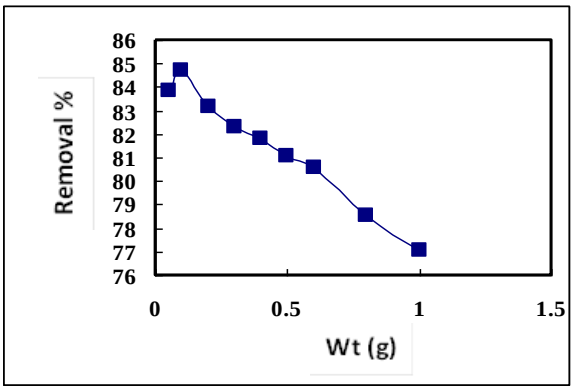

Fig. (3) Effect of adsorbent dose on adsorption of CIPH at 10 mg/L.

Effect of contact time

The equilibrium time is one of the characteristics, defending efficiency in the removal of drug. The effect of contact time and the percent removal of drug form aqueous solution by porcelinaite is shown in the Fig. (4). It has been observed from the data that over 78.29 % of the adsorption of drug form aqueous solution of porcelinaite was completed within first 5 minutes and equilibrium was reached at 20 minutes. In case of adsorption this was because of rapid diffusion of drug from solution to the external surface of adsorbents where the drug sorbs at the active surface of the adsorption.

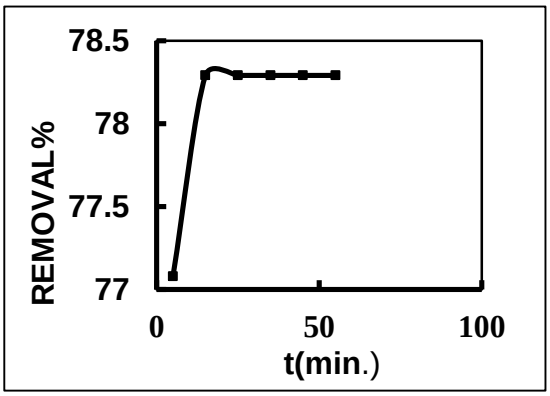

Fig.(4) Effect of contact time at 37.5 ^{0}C.

FTIR analysis

The FTIR spectrum obtained Fig. (5) for the adsorbent displayed the following major bands: 3630 cm^{-1}: Si-OH, Al-OH stretch; 3539.38-1637.56 cm^{-1}: H-OH stretch; 1637.56-1045cm^{-1}:Si-O-Si, Si-O stretch; 472 cm^{-1} :Si-O ,Si-O-Fe stretch It is reflecting the complex nature of adsorbent and shows significant band shifting and intensity changes due to ciprofloxacin hydrochloride sorption Fig. (5 and 6).

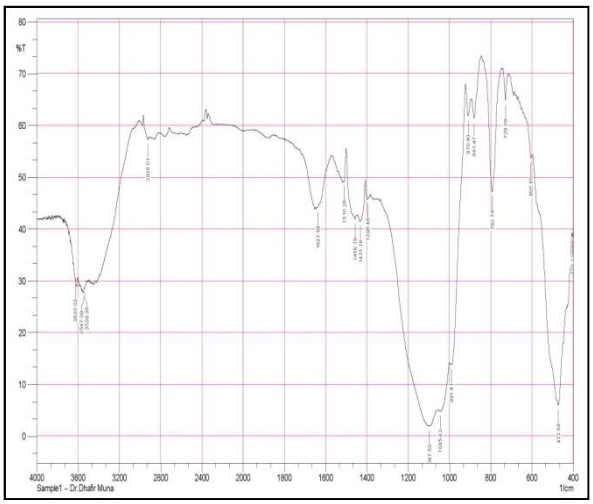

Fig. (5) FTIR spectrum of fresh Porcelinaite adsorbent.

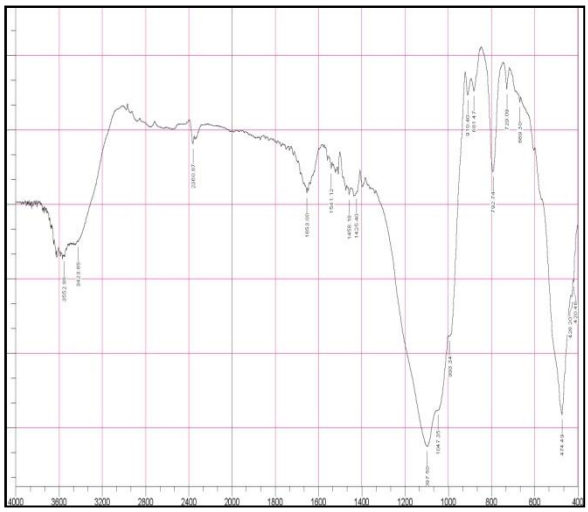

Fig. (6) FTIR spectrum of drug loaded adsorbent.

Effect of pH on adsorption

The pH is one of the important factors in controlling the adsorption of drug on adsorbent. The adsorption of CIPH from 10 mg/L concentration on given adsorbent was studied at pH (1.3, 3.8,5,6,7 and 9). The result shown in Table (3) Fig. (7) shows that the adsorption of CIP increased from the initial pH 1.3 and then decreased over the pH rang of 3.8-9.Therefore, In this study we found that pH(1.3)as the optimum pH. At low pH region the surface of the clay will be largely protonated. The positive ions (H+) provide an electrostatic attraction between the clay surface and the drug molecules leading to maximum adsorption on the other hand, at pH

above 1.3 the degree of protonation of The surface of the clay will be less which result in the decrease in diffusion and adsorption thereby due to electrostatic repulsion Furthermore, lower adsorption of the Drug molecules in alkaline medium can be attributed to the competition from excess Hydroxide ions (OH^-) with the drug molecules for the adsorption sites.

Table (3)
Effect of pH on adsorption of CIP at 10 mg/L.

pH	*Ce(mg/L)*	*Co - Ce*	*Removal%*
1.3	0	10	100
3.8	1.189	8.811	88.11
5	1.286	8.714	87.14
6	1.4072	8.5928	85.928
7	1.6012	8.3988	83.988
9	1.8315	8.1685	81.685

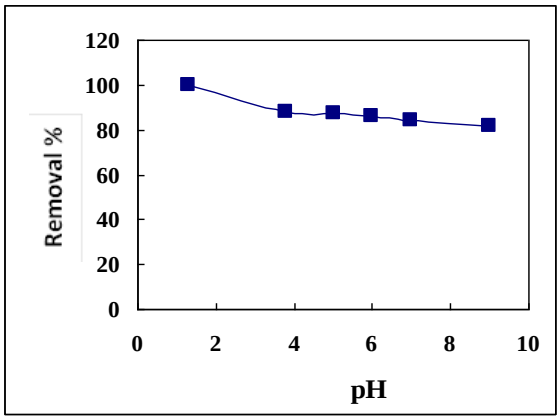

Fig.(7) Effect of pH on adsorption of CIPH at 10 mg/L.

Effect of Ionic Strength

The increase in the amount of adsorbate with increase in the ionic strength of electrolyte.

(NaCl) for the adsorbent CIPH Table (4) and Fig. (8) indicate that the adsorption is sensitive to the change in ionic Strength, which indicate that electrostatic attraction is a significant mechanism for sorption process. The increase in adsorption with ionic strength may be due to the Compression of the thickness of the diffused double layer. Such compression may help the sorbent particle and sorbate species to approach each other more closely, by then the attractive forces become significant, leading to increased adsorption. These results indicate that electrostatic attraction plays a significant role in the removal of CIP.

Table (4)
Effect of ionic strength on adsorption of CIPH at 37.5°C.

C_0 mg/L	0.2 M NaCl		0.4 M NaCl	
	C_e mg/L	Q_e mg/g	C_e mg/L	Q_e mg/g
2	1.5527	0.0447	1.4096	0.0590
4	2.0996	0.1950	1.5284	0.2471
6	2.0981	0.3901	1.5939	0.4406
8	2.2557	0.5744	1.7927	0.6207
10	2.4618	0.7538	1.8690	0.8131
12	2.5224	0.9477	1.9418	1.0058
14	2.5587	1.1441	1.9406	1.2059

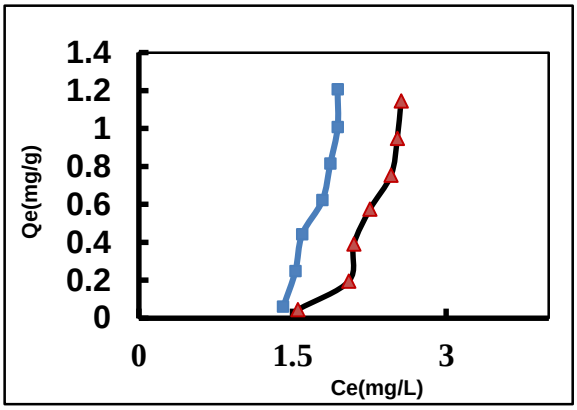

Fig. (8) Effect of ionic strength on the Adsorption CIPH on porcelinaite (■ 0.4 M and ▲0.2 M NaCl) T=37.5°C, pH=3.8 and p.size 75µm.

Isotherms study

The adsorption isotherm indicates how the adsorbed molecules distribute between the liquid phase and the solid phase when the adsorption process reaches an equilibrium state. The analysis of the isotherm data by fitting them to different isotherm models is an important step in finding a suitable model that can be used for design purpose. The adsorption capacity of this system was investigated with Freundlich and Temkin adsorption isotherms. The drug sorption isotherm followed the Freundlich model, as shown in Fig. (9). The relation between the drug uptake capacity Qe (mg/g) of adsorbent and the residual drug concentration C_e (mg/L) at equilibrium is given by

$LogQ_e=logK_F+(1/n)logC_e$ ..... (3)

Where the intercept, log K_F, is a measure of adsorbent capacity, and the slope 1/n is the sorption intensity.

The Temkin isotherm (25) is expressed as shown below.

$Q_e= BlnK_T + B\ lnC_e$ ..... (4)

Where K_T (L/g) is the equilibrium binding constant, corresponding to the maximum binding energy and constant B= (RT/b) is related to heat of adsorption. The Temkin isotherm plot between lnC_e and Q_e is shown Fig. (10) enables the determination of the B and $K_T$from the slope and intercept Table (5). These results in Table (5) show the Freundlich model was found to fit data significantly better than the Temkin show the more heterogeneous nature of Porcelinaite powder.

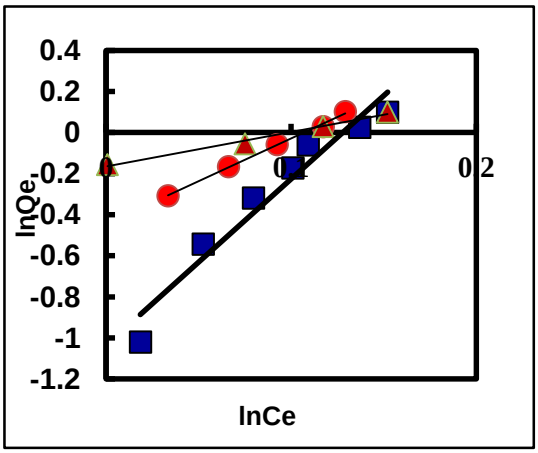

Fig.(9) Freundlich isotherm for adsorption of CIPH on Porcelinaite at different temperature (■ 15º C, ●25º C and ▲37.5º C).

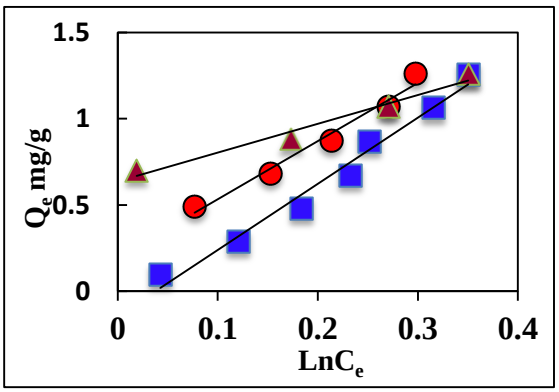

Fig.(10) Temkin isotherm for adsorption of CIPH on Porcelinaite at different temperature (■ 15º C, ●25º C and ▲37.5º C).

Table (5)
Isotherm constants for adsorption of drug on Porcelinaite at 37.5 ºC.

Freundlich			Temkin		
constants		Correlation coefficient (R^2)	constants		Correlation coefficient (R^2)
K_f	n		A	B	
1.47	0.59	0.9889	1.46	1.67	0.9739

Effect of temperature

The effect of temperature was investigated at three different temperatures (15, 25 and 37.5ºC). In present investigations, the amount of adsorption of Ciprofloxacin Hydrochloride increased with temperature increase from 15 to 37.5^0 C Fig. (11). Most of the adsorption processes are reported to be exothermic in nature, but present study has been found to be an example of endothermic adsorption.

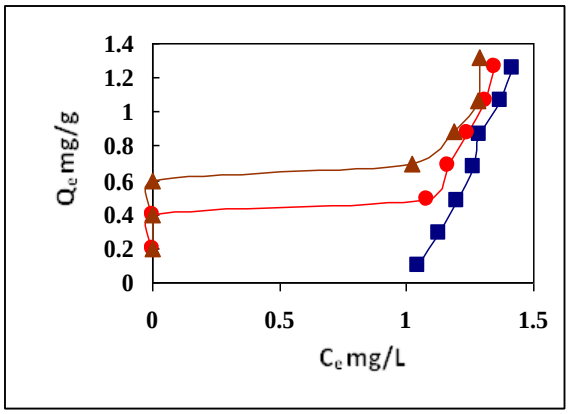

Fig.(11) Effect of temperature on adsorption of CIPH at different temperature (■ 15º C, ●25º C and ▲37.5º C).

Thermodynamic studies

Thermodynamic parameters such as Gibb's free energy (ΔG) (J/mole), enthalpy (ΔH) (J/mole) and entropy (ΔS) (J/mole.K) changes can be determined by the following equations:

$\Delta G = -RT \ln K_0$ ..... (5)
$\ln K_0 = -\Delta G/ RT$ ..... (6)
$\ln K_0 = \Delta S/R - \Delta H/RT$ ..... (7)

Where R is universal gas constant (8.314 J. $mole^{-1}.K^{-1}$) and T is absolute temperature in Kelvin. Gibb's free energy change ΔG is calculated using K_0. The equilibrium constant $K_o$can be defined as:

$$Ko = \frac{Co-Ce}{Ce} = \frac{Co}{Ce} - 1 \quad \text{..... (8)}$$

The estimated thermodynamic parameter such as ΔH,ΔS and ΔG and equilibrium constants at each temperature are summarized in Table (6). The enthalpy change (ΔH) for the adsorption of CIPH onto Porcelinaite signifies an endothermic adsorption. The ΔG value is negative at all studied temperatures, inferring that, the adsorption of CIPH onto Porcelinaite will follow a spontaneous trend. The ΔG value decreased when the temperature increased from 15°C to 37.5°C, suggesting increase in adsorption of CIPH with increasing temperature [17]. The positive value of ΔS reflects the affinity of Porcelinaite towards drug and also suggests increased randomness at the solid-solution interface.

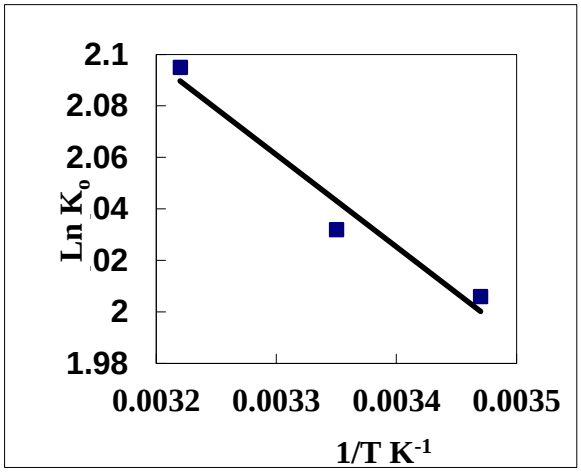

Fig.(12) Plot ln K_0 versus 1/ T for the estimation of thermodynamic parameters.

Table (6)
Thermodynamic parameters for CIPH adsorption on Porcelinaite.

Temperature °C	*K_o*	*ΔG KJ mol^{-1}*	*ΔH KJ mol^{-1}*	*ΔS J mol^{-1} K^{-1}*
15	7.43	-4.80	2.97	26.95
25	7.63	-5.04		
37.5	8.13	-5.41		

Conclusion

Following conclusions are drawn from above discussed results:

Porcelinaite has a good adsorption capacity at 37.5 °C for the adsorption of CIPH, the percent of drug removal decrease at high concentration, equilibrium time for the adsorption of CIPH on porcelinaite from aqueous solutions is estimated 20 minute also the maximum adsorption of CIPH took place in the pH 1.3, the maximum removal of drug 84.716 % at 10 mg/L concentration was obtained with 0.1 g of Porcelinaite, The increase in the amount of adsorbate with increase in the ionic strength and the adsorption process of CIPH can be described by Freundlich and Temkin isotherm model.

Acknowledgments

The authors are highly thankful to the Department of Chemistry, College of Education for Pure Science Ibn Al- Haitham, University of Baghdad.

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الخلاصة

تعد المستحضرات الصيدلانية من المركبات ذات الاستعمال الواسع والتي من الممكن ان تنتشر في البيئة. في هذا البحث تمت دراسة امتزاز عقار السيبروفلوكساسين هيدروكلورايد على سطح ماز هو البورسيلاين العراقي عند ظروف محددة ولوحظ من خلال الدراسة ان ايزوثيرمات الامتزاز تتبع كل من معادلة فريندلش وتمكن على التوالي كما تم دراسة تاثير الاس الهيدروجيني على قابلية الامتزاز عند مدى من ١,٣-٩ كما تم ايضا دراسة تاثير الشدة الايونية على عملية الامتزاز كما تم ايضا دراسة الانثالبي لعملية الامتزلز حيث اوجدت القيم ان العملية تعتبر ماصة للحرارة وكذلك تمت دراسة كل من الانتروبي والتغير في طاقة كبس وعند درجات حرارية متعددة.