Adsorption of Metoclopromide Hydrochloride onto Burned Initiated Iraqi Bentonite

Omar S. Alkhazrajy, Mohammed H. Abdul Latif and Maha A. Al – Abayaji
Department of Chemistry, College of Education Ibn Al Haitham, University of Baghdad, Baghdad-Iraq.
E-mial: omersabih@yahoo.com.

Abstract

Bentonite is widely used as an adsorbent for the management of patients with drug overdoses, poisonings and environment treatment. Multiple oral doses of bentonite increase the elimination of several, but not all, drugs and poisons. Adsorption of Metoclopromide hydrochloride onto initiated burned bentonite from aqueous solution has been investigated, an adsorption isotherms and FTIR spectroscopy characterization were studied. From the adsorption studies may be deduced that amount of Metoclopromide hydrochloride adsorbed by initiated burned bentonite slightly increases with the decrease in the pH of the solution and the increase in solution temperature caused a simple increase in the adsorption capacity values found from Freundlich model (R^2=0.975). Thermodynamic functions, the change of free energy (ΔG= -7.924 K J mol^{-1}), enthalpy (ΔH=9.612 J mol^{-1}) and entropy (ΔS=2.498 J $mol^{-1}k^{-1}$) of sorption were also calculated. These parameters show that the sorption process is spontaneous and endothermic at 37 °C. The effect of contact time, clay dosage and ionic strength also studied.

Keywords: Adsorption, Metoclopromide HCl, Burned initiated Bentonite .

1. Introduction

Drugs are substances used to relieve pain and treat illness to achive this aim drug doses must be delivered to the targeted tissues so that is therapeutic, yet non toxic levels are obtained[1]. Drug poisoning 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 [2] Reaction to a drug caused by an allergic sensitivity is not considered drug poisoning. Virtually all drugs, especially in large doses or when taken over long periods of time, can initiate a toxic condition [3]. The major principles applied in the emergency treatment of accidental poisoning by drug are dilution, emesis and adsorption [4,5]. In cases where no specific antidotes exist, prevention of further adsorption of a drug from the oral route is by use of oral adsorbents. This could be of immense benefit in the management of drug overdose or poisoning. On other hand pharmaceuticals have been identified in the environment, including antibiotics, analgesics, psychiatric drugs, and natural and synthetic hormones . Human pharmaceuticals enter the environment through incomplete wastewater treatment of drugs either not absorbed by the body or intentionally discardeddownthe drain. Unused human pharmaceuticals may also enter the environment through landfill leachate [6-10].

Many types of adsorbents such as kaolin [11,12], charcoal [13-15]), polymers [16] attapuligate [17] and bentonie [18-22] in the prevention of further 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 along time, and they account for most of the current uses of clay. Bentonite is a natural clay consist mainly of montmorillonite a complex of colloid magnesium aluminum silicate with small amount of minerals, the adsorption of tir cyclic drugs from solution on bentonite surface at different conditions was studied.[23],

investigation the activity of bentonite as antidote in treatment of poisoning by some GTI drugs [24] , also studied Cation exchange interaction between antibiotic ciprofloxacin and Montmorillonite [25]**.**

MetoclopromaideHydrochloride (Fig.(1)) (4-amino-5-chloro-N- (2- (diethylamino) ethyl) -2-methoxybenzamide HCl is an antiemetic and gastroprokinetic agent [26] . Thus it is primarily used to treat nausea and vomiting, and to facilitate gastric emptying in patients with gastroparesis [27]. It is also a primary treatment for migraine headaches. Common adverse drug reactions (ADRs) associated with metoclopramide therapy include: restlessness, drowsiness, dizziness, lassitude, and/or dystonia. Infrequent ADRs include: headache, extrapyramidal effects such as oculogyric crisis, hypertension, hypotension, hyperprolactinaemia leading to galactorrhoea, constipation, and/or depression. The risk of extrapyramidal effects is increased in young adults (<20 years) and children, and with high-dose or prolonged therapy. Tardive dyskinesias may be persistent and irreversible in some patients [28,29]. In this study for the first time we i nvestigate the adsorption of Metoclopromaide Hydrochloride on the surface of burned initiated Iraqi bentonite and calculate the the effect of factors like pH, ionic strength contact time and weight of clay on percentage of removal of the drug, also calculate the isothermic factors of adsorption process.

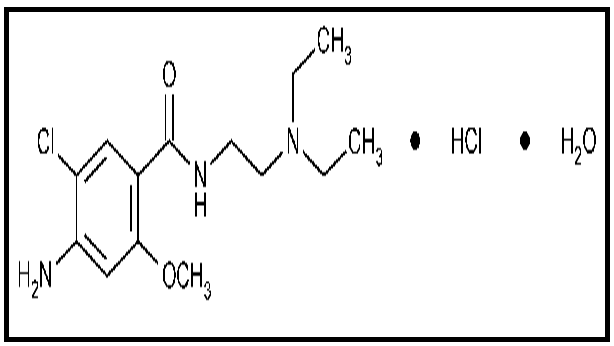

Fig.(1) Structure of Metoclopromaide Hydrochloride.

2. Experimental Process

Materials and Apparatus

The drug employed in this research (Metoclopromaide hydrochloride) was obtained from (state company for drug industries and medical appliances Samara – Iraq. The clay (bentonite) employed in this study were obtained from open mine in (Trifawi) area of the western desert- Iraq supplied from (The general company for Geological survey and mining), Baghdad, The mineralogical composition, granulometry 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) and burned at 600 ^{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 250 μm was used for the clay in the experiments of this work, Thermostated shaker bath (albaTech), pH meter (HI98107, Hanna Instruments). Cintra5 double beam UV-Visible spectrophotometer, FTIR spectro-photometer (SHIMADZU 8400S) used for the characterization of bentonite used in this study befor and after adorption of drug.

2.2. Experimental work

Standard solution of Metoclopromaide 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 (214 nm), this value utilized for measurements of estimation throughout this research. Two types of experiments were carried out. The first was to investigate the time to attain equilibrium; the second was to find out the extent of adsorption of the drug on the adsorbent.

For the time to reach equilibrium study, 25 ml of an initially fixed concentration 20mg/L of drug solution was added separately into 25 ml volumetric flasks to which (0.5 g) of each adsorbents were put. Similarly, 25 ml of distilled water was added to equal graded amounts to adsorbents in 25 ml volumetric flask (without drug used as blanks for the absorbance measurements). The slurries were mixed and put in the shaker bath at 37 ^{o}C Rotation was stopped and the solution was then filtered by using nylon filer with syringe (0.45µm). The concentrations of the clear supernatants of the drug were assayed spectrophotometrically at different time intervals from (5 to 150 minutes). From the Beer's plot for the Metoclopromaide Hydrochloride drug previously made, the amount of free drug in solution was determined. From the results, the time to attain equilibrium for adsorbent have been determined.

Table (1)
The components of crude bentonite.

compound	SiO_2	Al_2O_3	CaO	Fe_2O_3	MgO	Na_2O	SO_3	**Lose on ignition**	Total
Wt. %	54.66	14.65	4.77	4.88	6.00	0.65	1.20	12.56	99.37

Adsorption isotherm

In the present investigation, batch mode operation was selected in order to measure the progress of adsorption. To determine adsorption isotherms for the drug with bentonite surface, solution of different concentrations for this drug were prepared by serial dilutions in the range of (5, 10, 15, 20, 25, 30 mg/L).

Adsorbent surface samples 0.5 g was weighed by using electrical balance Each sample was then placed in a screw cap bottle and 25 ml of serial drug solution was added to each sample. The bottles were put in thermostated shaker at different temperature (25, 37 and 45 ^{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 solution.

At the end of the adsorption period, the rotation was stopped and the solution was filtered by using nylon filer with syringe (0.45µm). 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):

$$Qe = (C_o - C_e)\ V/W \quad \text{.....(1)}$$

Where C_o and C_e are the initial and equilibrium 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 :

$$\% \text{ of removed} = (C_o - C_e) \times 100/C_o \quad \text{.....(2)}$$

Result and Discussion

Characterization of Clay

Natural Iraqi bentonite FTIR spectrum Fig.(2) showed adsorption band at 3628.10 cm^{-1} (Al-Al-OH) (Mg-OH-Al) corresponding to starching vibration of structural OH groups coordinating to Al-Al pair or Mg-OH-Al. Adsorbed water gives a broad bands from 4306.29 cm^{-1} to 3533.59cm^{-1} corresponding to H_2O-stretching vibration. Al, Mg bound water molecules gives H-O-H stretching vibration band at 1643cm^{-1}. Also three bands at 1546.91, 1427.32 and

1384.89 cm^{-1}corresponding to H...O...H are weak. The complex broad band around 1033 cm^{-1} belongs to Si-O stretching vibration. Two bands at 914.26 cm^{-1} and 837.11 cm^{-1} are most characteristic for quartz. Finally the bands from 420.00 $cm^{-1}$to 516.93 cm^{-1} are related to Al-O-Si , Si-O-Si deformations.

Initiated bentonite FTIR spectrum Fig.(3) showed the same bands of Fig.(2) but with higher transmittance percent and sharper than bands of FTIR spectrum of natural bentonite. Nevertheless weak H..O..H disappear in this spectrum. Adsorbed water band appear at 4321.72 cm^{-1}, two bands belong to Al, Mg bound water molecules observed at 1654.92 cm^{-1} and 1641.42 cm^{-1}. The broad complex band becomes single band at 1039 cm^{-1} belongs to Si-O stretching vibration. Also we observe two bands belongs to Al…OH stretching vibration.

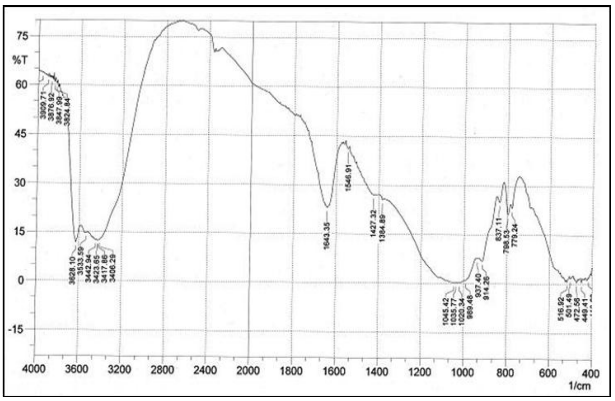

Fig.(2) FTIR spectrum for crude Iraqi bentonite (Trifawi).

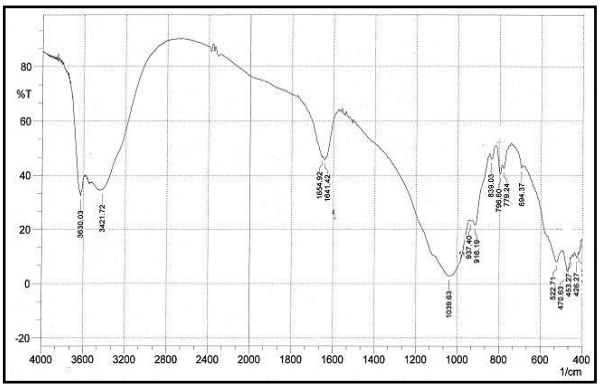

Fig.(3) FTIR spectrum for initiated Iraqi bentonite (Trifawi).

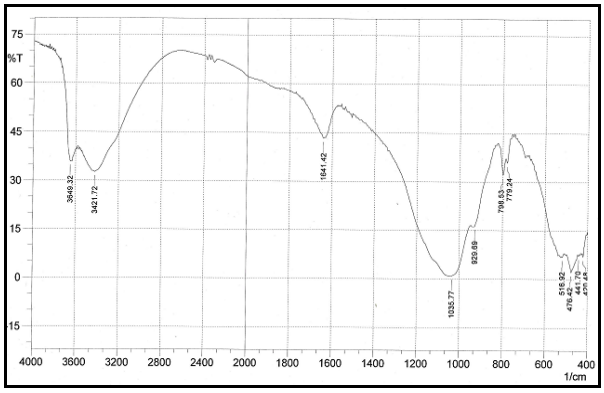

Fig.(4) FTIR spectrum for burned initiated Iraqi bentonite (Trifawi).

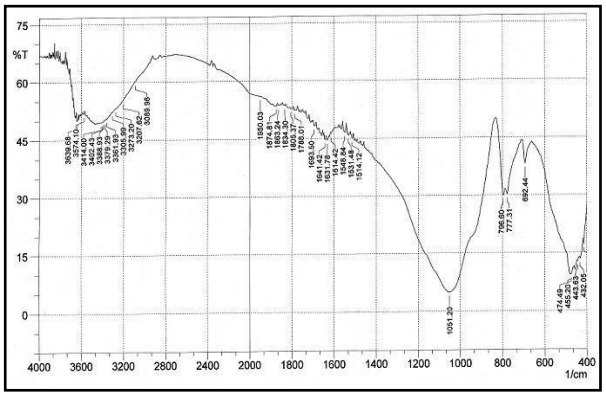

Fig.(5) FTIR spectrum for adsorption of Metoclopromide HCl on burned initiated Iraqi bentonite (Trifawi).

at 937.04 cm^{-1} and 916.19 cm^{-1} with higher transmittance percent .the quartz characteristics band from 694.37 cm^{-1} to 839.03 cm^{-1} become boarder. Finally Al-O-Si, Si-O-Si and Si-O stretching vibration bands from 426.27 cm^{-1} to 522.71 cm^{-1} become sharper and triplet bond[30,32]. Burned initiated Iraqi bentonite FTIR spectrum Fig.(4) showed the same bands of that in Fig.(4) but sharper and with higher transmittance percent , also the band of adsorbed water become a single band at 3421.7 cm^{-1} mostly due to burning process and the same thing happen with band Al, Mg around water molecule stretching vibration also H…O...H weak bands disappear in this spectrum.

Fig.(5) showed FTIR for Metoclopromide HCl adsorbed on burned initiated bentonite, comparing this with FTIR spectrum of

initiated burned bentonite, we observe that the major bands of burned form exist at the same position and the drug FTIR bands are distributed between them ,exactly in range of 1514.12 cm^{-1} to 3639.68 cm^{-1}. Drug FTIR band are distributed between two regions, the first from 1550 cm^{-1} to 1850 cm^{-1} corresponding to bending C=O, band at 1693.50 cm^{-1} and 1641.41 cm^{-1}, also N-H bonding band at 1631.78 cm^{-1} and 1614.42 cm^{-1}. The R-(CO)-NH-R amide group of metoclopromide HCl drug exist at 1541.12 cm^{-1} and 1531.48 cm^{-1}. The band at 1541.12 corresponding to N-H bending primary aromatic band. Bands from 1788.01 cm^{-1} to 1950.03 cm^{-1} belongs to combination or over tone bands. The second region from 2700 cm^{-1} to 3700 cm^{-1} corresponding to N-H stretching solid primary solid symmetric vibration, at 3402.43 cm^{-1} and 320.62 cm^{-1} N-H stretching solid primary asymmetric vibration at 3574.10 cm^{-1} and 3361, 93 cm^{-1} [33].

Effect of pH on Adsorption

In order to optimize, the pH of maximum removal efficiency experiments were conducted by using pH 1.2 ,2.6 and 3.6 (we get it by using Phthalate buffer which was prepared by dissolving 2.04 g of potassium hydrogen phthalate in 100 ml of water and the pH was adjusted by using 0.1 M hydrochloric acid and NaOH) by adding 0.5 g of burned bentonite adsorbent with 25 mL of 20 mg/L drug solutions at temperature (37^0C). The results we included in the Table (2) and are graphically represented in Fig.(6), from the graphs it is clear that adsorption of drug did not basically depends on pH of the solution. Sorption decrease slightly with decrease in pH of the adsorbent. It is well known that surface charge of adsorbent can be modified by charging the pH of the solution and the chemical species in the solution depends on this parameter the percent adsorption of drug decreased with the decrease in pH, because protons compete with drug for sorption sites on the adsorbent surface [34].

Effect of Adsorbent dose

The dependence of adsorption of the drug on the amount of bentonite was studied by varying the adsorbent dose from 0.1 to 1 g at temperature (37 ^{o}C) and at their optimal pH, while keeping the volume and concentration of the metal solution constant. The results are given in Table (3) and graphically represented in Fig.(7). The figure indicates that sorption increased with increasing sorbent dose up to 0.5 g and then there was no further increase of sorption. It is evident that the maximum removal of drug 98.82% at 20 mg/L concentration was obtained with 0.5 g of burned initiated bentonite. The adsorption of the drug increased rapidly with increase in the dose of the adsorbent due to greater availability of the exchangeable sites or surface area.

Table (2)
Effect of pH on adsorption on percentage of removal of Metoclopromaide HCl on bentonite (C_o 20 mg/L).

pH	*Ce mg/L*	*Qe mg/g*	*% removed*
1.2	0.9781	0.9510	95.109
2.6	0.9645	0.9517	95.177
3.6	0.9316	0.9536	95.360

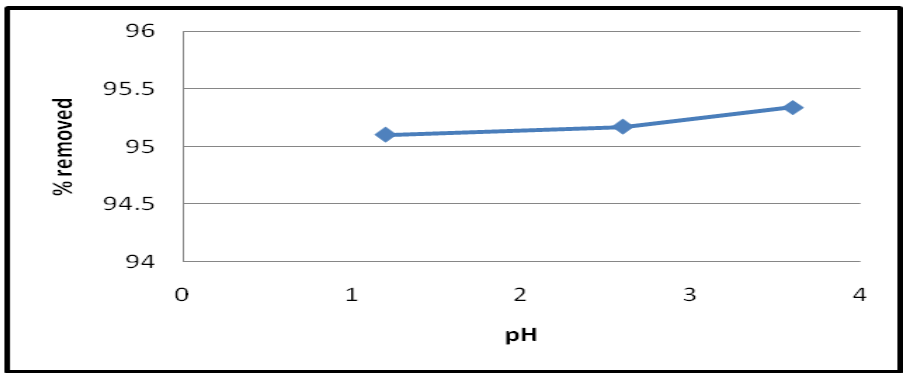

Fig.(6) Effect of pH on adsorption uptake(percentage of removal) of Metoclopromaide HCl on bentonite (C_o 20 mg/L).

Table (3)
Effect of weight of clay on adsorption uptake (percentage of removal) of Metoclopromaide HCl on bentonite (C_o 20 mg/L).

Dose of clay gm	*Ce mg/L*	*Qe mg/g*	*% removed*
0.1	3.4884	4.1279	82.55
0.2	0.7450	2.4068	96.27
0.3	0.4811	1.6265	97.59
0.4	0.3058	1.2309	98.40
0.5	0.2356	0.9882	98.82
0.6	0.2770	0.8217	98.60
0.7	0.3072	0.7033	98.40
0.8	0.3333	0.6145	98.30
0.9	0.3550	0.5456	98.20
1	0.3169	0.4920	98.36

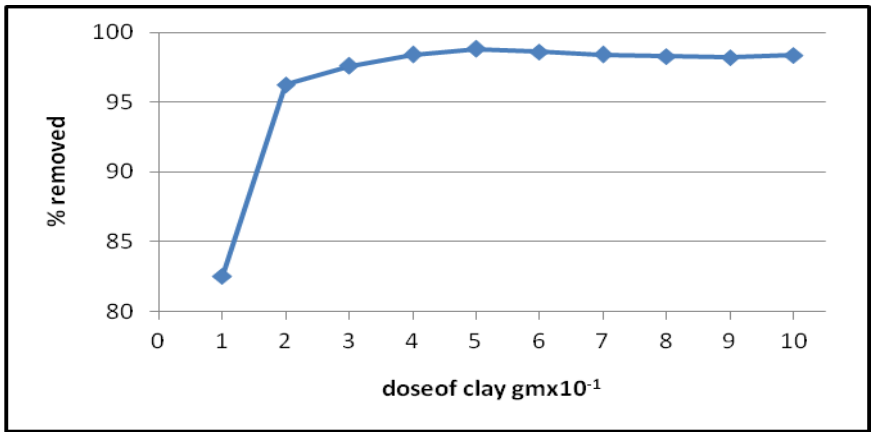

Fig. (7) Effect of weight of clay on adsorption uptake (percentage of removal) of Metoclopromaide HCl on bentonite (C_o 20 mg/L).

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 burned initiated bentonite is shown in the Fig.(8) and in Table (4). It has been observed from the data that over 80 % of the adsorption of drug form aqueous solution by burned initiated bentonite was completed within first5 minutes and equilibrium was reached at 30 minutes. In case of adsorption this was because of rapid diffusion of ions form solution to the external surface of adsorbents where the drug sorbs at the active surface of the adsorption.

Effect of temperature

Temperature study on adsorption of Metoclopromaide Hydrochloride on burned bentonite at three different temperatures i.e., 25,37 and 45^{o}C. The results obtained are listed in Table (5) and Fig.(9) the equilibrium adsorption capacities slightly increased with an increase of temperature from 25 to 45 ^{o}C. This increasing indicates that adsorption of Metoclopromaide Hydrochloride is controlled by an endothermic reaction.

Table (4)

Effect of contact time on adsorption uptake (percentage of removal) of Metoclopromaide HCl on bentonite(C_o 20 mg/L).

Time(min)	*Ce mg/L*	*Qe mg/g*	*% removed*
5	5.9884	0.7006	70.000
10	2.5791	0.8710	87.100
15	1.6001	0.9200	91.99
20	1.0980	0.9450	94.510
25	0.9275	0.9536	95.362
30	0.5666	0.9716	97.167
60	0.6768	0.9666	96.663
90	0.6724	0.9664	96.664
150	1.0130	0.9493	94.935

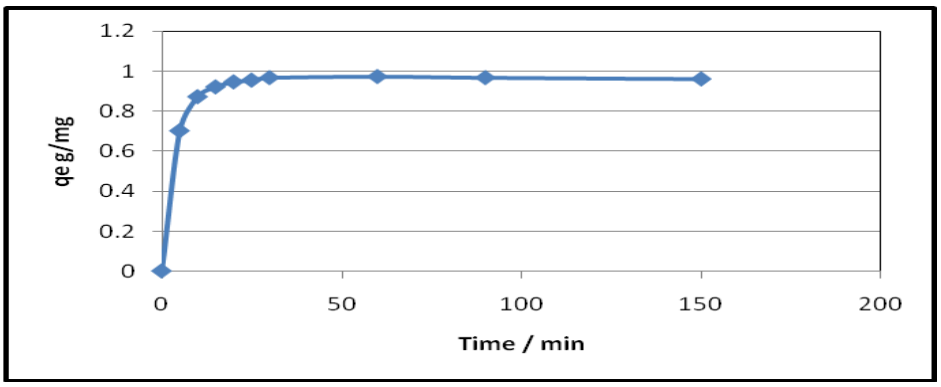

Fig.(8) Effect of contact time on adsorption uptake of Metoclopromaide HCl on bentonite (C_o 20 mg/L).

Table (5)
Effect of temperature on adsorption uptake (percentage of removal) of Metoclopromaide HCl on bentonite(C_o 20 mg/L).

conc. mg/L	25°C		37°C		45°C	
	C_e mg/L	q_e mg/g	C_e mg/L	q_e mg/g	C_e mg/L	q_e mg/g
0	0	0	0	0	0	0
5	0.3010	0.2340	0.2998	0.2350	0.1923	0.2404
10	0.5981	0.4701	0.4821	0.4758	0.4012	0.4799
15	0.8746	0.7062	0.8297	0.7085	0.7001	0.7151
20	0.9638	0.9518	0.9681	0.9516	0.7834	0.9608
25	1.0863	1.1956	1.0698	1.1965	0.8572	1.2071
30	1.2083	1.4000	1.1933	1.4400	0.9112	1.4545

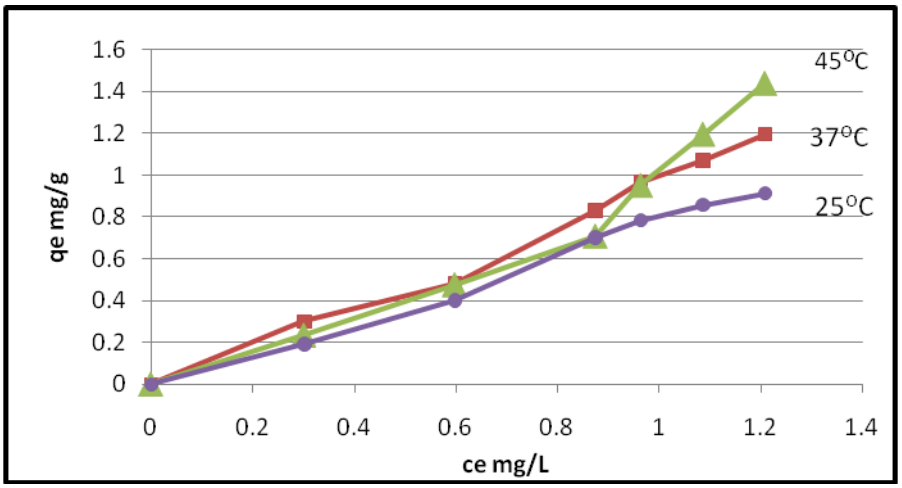

Fig. (9) Effect of temperature on adsorption uptake of Metoclopromaide HCl on bentonite (C_o 20 mg/L).

Effect of Ionic Strength

The result obtained for both free and supported catalyst are given in Fig.(6). The clay supported catalyst appears significantly less active at high ionic strengths than the free salt solution (aqueous solution) and the percentage of removal will decrease Table (6). Hence, the adsorption of catalyst given less stabilization to the active site against electrostatic interactions. The influence of ionic strength on bond and free salt solution was determined by adding (0.1, 0.2and 0.3 M sodium chloride) to the reaction medium at constant pH and temperatures. These results show that when the ionic strength was increased, the activity of the immobilized catalyst reduced more than the activity of the free catalyst.

Table(6)
Effect of Ionic strength on adsorption uptake (percentage of removal) of Metoclopromaide HCl on bentonite(C_o 20 mg/L).

Conc. M	Ce mg/L	Qe mg/g	% removed
With out	0.9681	0.9516	95.16
0.1	2.6811	0.8659	86.60
0.2	3.471	0.82645	82.64
0.3	3.8492	0.80745	80.74

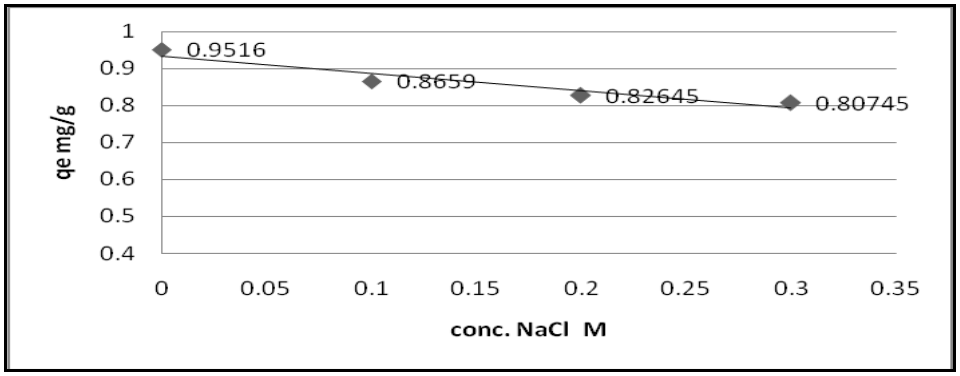

Fig.(10) Effect of Ionic strength on adsorption uptake of Metoclopromaide HCl on bentonite (C_o 20 mg/L).

Adsorption Isotherms

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 the Freundlich, Langmuir and Temkin adsorption isotherms [29]. The drug sorption isotherm followed the linearized Freundlich model, as shown in Fig.(11). 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

$Logq_e = logK_F + (1/n)\ logC_e$

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

Table (7)
Goodness of fit of the Freundlich ,Langmuir and Temkin isotherm to the sorption experimental data. Values corresponding to best fit isotherm (37^o).

Adsorbent	R^2		
	Freundlich	*Langmuir*	*Temkin*
Bentonite	0.975	0.579	0.876

Table (8)
Calculated thermodynamic parameters of Metoclopromide Hydrochloride adsorption on clays surface ($37c^o$).

Surface	*ΔH* ($J\ mol^{-1}$)	*ΔG* ($k\ Jmol^{-1}$)	*ΔS* ($J\ mol^{-1}k^{-1}$)
Bentonite	9.612	-7.924	2.498

The isotherm data fit the Freundlich model well (R^2=0.975). The values of the constants K_f and1/n were calculated to be 1.0917 and 0.82, respectively. Since the value of 1/n is less than 1, it indicates a favorable adsorption. The Freundlich isotherm is more widely used, but provides no information on the monolayer adsorption capacity, in contrast to the Langmuir model.

The Langmuir equation relates the solid phase adsorbate concentration (q_e) or uptake to the equilibrium liquid concentration (C_e) as follows: $q_e = abC_e/1+bC_e$ where a and b are the Langmuir constants, representing the maximum adsorption capacity for the solid phase loading and the energy constant related to the heat of adsorption, respectively.

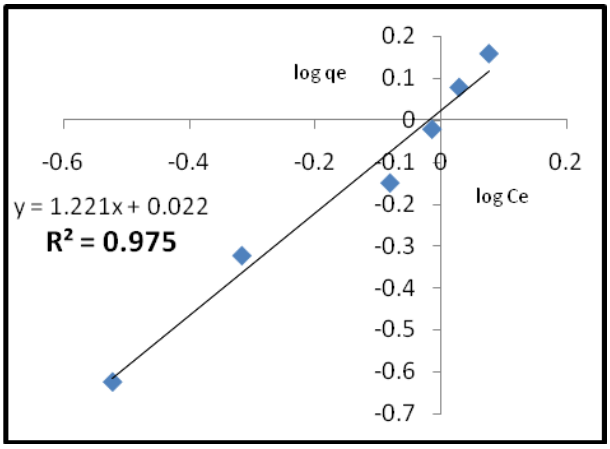

Fig.(11) Freundlich adsorption isotherm.

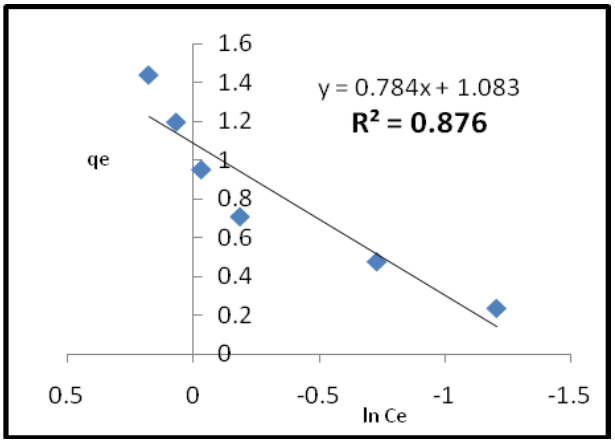

Fig. (12) Temkin adsorption isotherm.

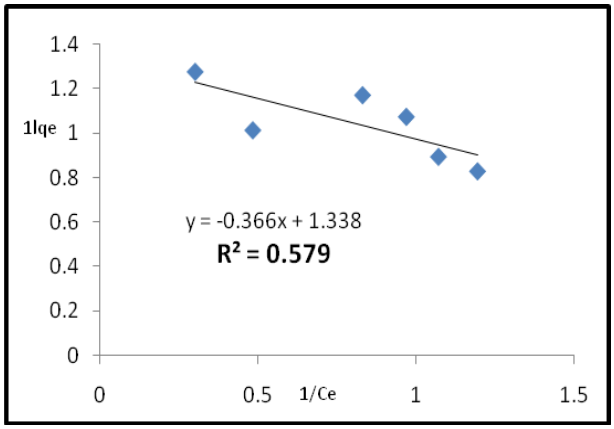

Fig.(13) Langmuir adsorption isotherm.

Conclusion

The sorption of drug on the adsorbents was affected by the parameters such as pH, contact time and adsorbent dosage. Initiated burned Iraqi bentonite is strongly and rapidly adsorbed Metoclopromide HCl in acid medium *in-vitro*. Thus, it could be effectively used to prevent drug absorption from the gastro-intestinal tract in cases of overdose or poisoning. Though bentonite had a high adsorbing capacity for the drug . It should not therefore be administered concurrently with the fluoroquinolones in the treatment this would lead to therapeutic failure. The equilibrium sorption data fitted the Freundlich isotherm model better than the Langmuir and Temkin models, the thermodynamic study of this work relieved that the adsorption of this drug was found to exhibit an exothermic process on the clays surfaces

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الانثالبي (ΔH=9.612 J mol^{-1}) والعشوائية (ΔS=2.498 J $mol^{-1}k^{-1}$) من خلال هذه المؤشرات تبين ان عملية الامتزاز تحدث بصورة تلقائية وماصة للحرارة كذلك تم دراسة بعض المتغيرات الاخرى مثل زمن التماس و الشدة الايونية على عملية الامتزاز.

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الخلاصة

يستعمل البنتونايت بشكل واسع كسطح ماز لمعالجة المرضى الذين يتناولون جرعات زائدة من الدواء وفي حالات التسمم وكذلك في المعالجات البيئية، ويتم معالجة التسمم من خلال تناول جرعات متعددة من البنتونايت. تم دراسة امتزاز دواء الميتوكلوبرومايد هيدروكلورايد على سطح البنتونايت العراقي المنشط المحروق وتم دراسة ايزوثيرمات الامتزاز واستخدام تقنية الاشعة تحت الحمراء لمعرفة ومتابع عملية التنشيط والامتزاز. من خلال الدراسة تبين ان امتزاز دواء الميتوكلوبرومايد على البنتونايت المنشط المحروق يزداد زيادة طفيفة مع الزيادة في مستوى الاس الهيدروجيني للمحلول كذلك ارتفاع درجة الحرارة يودي الى زيادة طفيفة في كمية المادة الممتزة , ايزوثيرمات الامتزاز تطابقت مع معادلة فريندلش للامتزاز بمعامل ترابط (R^2=0.975) تم حساب الدوال الثرموداينمكية مثل التغير في الطاقة الحرة (ΔG= -7.924 K J mol^{-1})