Kinetic and Thermodynamic Study of the Adsorption of some Organic Acids by MnO_2

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Abstract
In this research work, the adsorption efficiency of three structurally related acids namely, glycolic, mandelic and benzylic acids by MnO_2 from their aqueous solutions is investigated.The study is aimed at developing a better understanding of the adsorption mechanism regarding the molecular level. The experiments were achieved as batch methods.The factors affecting the adsorption efficiency such as; contact time, initial concentration, initial pH of the acid medium and temperature are studied. The results obtained showed that, the increase of initial concentration increases the adsorption efficiency. The initial pH of the medium plays an important role in controlling the attachment of acid molecules on the solid surface of MnO_2. The highest adsorption efficiency is observed in the acid medium (natural pH of the acid solutions).This study proved that, the acid is connected to the solid surface of absorbent in its anion form.The thermodynamic functions of adsorption are estimated at different initial concentrations (0.005-0.05 N). The study also included the application of the two kinetic models on the adsorption data namely; the pseudo first order and pseudo second order equations. The investigation is performed at certain concentration and various temperatures. The results denoted that, the studied systems are better fit the second order model.

Key words: Adsorption, Organic acids, Manganese dioxide, Kinetic study, Thermodynamic of adsorption.

Introduction
The adsorption process is a very complicated phenomenon, depending on the nature of adsorbate and adsorbent surfaces and type of functional groups present on their structures. The accumulation of the adsorbate materials on the solid surface of adsorbent is resulted in various types of molecular and ionic interactions which are still not well known. The exceedingly large number of organic pollutants found in the aquatic environments created an important challenge that, promoted a demand for effective treatment [(1)]. Organic acids are among the pollutants that attracted the attention of several searcher groups because of their adsorptive, complexant, and reductive properties [(2-5)]. The metal oxides have been proved to be good adsorbents [(6)]. They were used for the removal of organic acids from their aqueous solutions by adsorption [(3)]. Manganese (IV) oxide is blackish or brown inorganic compound with the formula of MnO_2. It occurs naturally as mineral pyrolusite, which is the main ore of manganese, and represents the most reactive oxidants found in the soil and sediments[(7)]. The most important reactions of MnO_2 are associated with its redox, both oxidation and reduction. Its adsorptive properties are responsible for the formation of surface complexes, which are the prerequisite of all reactions at oxide surfaces. Such characteristics encouraged many researcher groups for the use of MnO_2 especially as adsorbent for the removal of organic acids [(5, 6)]. The acid molecule carrying a negative charge is weakly adsorbed on the MnO_2 surface. Such behavior is observed in the adsorption of organic acids from their aqueous solution [(3)].The MnO_2 has also been applied for the oxidation of aromatic organic compounds such as phenol[(4)] dihydroxyl benzene[(5)] , aniline[(8)] and more complex aromatic structures[(9)] . The mechanism of the reaction of organic substrate with MnO_2 can possibly pass through different pathways [(10)]; the acid molecule can simply adsorbed on the solid surface of MnO_2 as in the following equation:

$$Mn(IV) + L\ (aq) \rightleftharpoons Mn^{(II)}\ L \quad ----(1)$$

Where L, denote to the organic substrate. The reductant properties could enable the conversion of $Mn^{(IV)}$ to a much more soluble $Mn^{(II)}$, in a process termed as reductive properties, as in the equation:

$$Mn^{(IV)}L \longrightarrow Mn^{(II)}(aq) + L_{ox}(aq)\ (+ > Mn^{(IV)}) \quad ----(2)$$

Where L_{ox} is the oxidized product of substrate and (+ > Mn (IV)) represents the unoccupied surface sites.The reaction mechanism may involve other pathways [(10)], where there is no room to mention here. Whatever was the mechanism pathway, the formation of complex by adsorption is the first step in any surface chemical reaction [(6, 11)]. The exact nature of this complex is not known at present.The kinetic of each pathway depends on adsorption, complexant, and reductant properties of organic structure, the identity of the MnO_2 surface, and the conditions of the environmental media (pH, major ion composition, and so on). Because of the importance of MnO_2 as a catalyst, and since the catalytic behavior pass through adsorption of the reacting species on its surface, this research work, is aimed at developing a deeper understanding of the molecular level adsorption mechanism by investigating the adsorption of some structurally related acids on the MnO_2

Kinetic model
In this research work, two kinetic models were employed to fit the experimental data, namely; the Lagergren pseudo first order and pseudo second order models.

1- Pseudo first order equation
This equation is described by Lagergren [(12, 13)] as:

$$\ln(qe - qt) = \ln qe - k_1 t \quad ----(3)$$

Where qe and qt (mg. g^{-1}), are the adsorption capacity at equilibrium and at time (t) respectively. k_1 (min^{-1})

is the rate constant of the pseudo first order adsorption.By plotting the values of ln (qe – qt) versus t, a straight line should be obtained if the adsorption data fit the first order model and the calculated value of qe that obtained from the intercept of the line must be equal or close to the experimental qe. The value of k_1 can be calculated from the slope of the straight line.

2- Pseudo second order equation [13, 14]

The pseudo second order kinetics may be expressed as follow:

$$\frac{t}{qt} = \frac{1}{k_2 q^2 e} + \frac{1}{qe} t \quad ---(4)$$

By plotting the value of t/qt versus time (t), the equilibrium adsorption capacity (qe) and the second order constant (k_2) ($g.mg^{-1}. min^{-1}$) can be determined from the slope and intercept of the straight line respectively.The adsorption system can be considered as second order when the correlation coefficient (R) of the line is close to unity and the calculated qe is consistent with the experimental value.

Adsorption thermodynamic:

The calculation of the apparent thermodynamic parameters can provide a clue for the elucidation of the adsorption mechanism of the studied acids on MnO_2.The enthalpy $(\Delta H, kJ.mol^{-1})$ and entropy (ΔS^o, J. mol^{-1} K^{-1}) of the acids adsorption on MnO_2 can be estimated from the slope and intercept of the straight line resulted from plotting lnK against 1/T according to Vant Hoff's equation:

$$\ln K = (\Delta S^\circ / R) - (\Delta H / RT) \quad ----(5)$$

Where K is the distribution coefficient, and calculated from the ratio of adsorbed acid on the remained (Free acid) in the solution at equilibrium. R is the gas constant (8.314 J. mol^{-1} K^{-1}) and T is temperature in Kelvin.The Value of ΔG° could be evaluated by using the following equations:

$$\Delta G^\circ = -RT \ln K \quad ----(6)$$

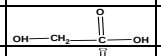
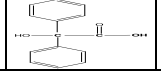
$$\Delta G^\circ = \Delta H - T\Delta S^\circ \quad ----(7)$$

Experimental

1- Adsorbate:

Three acids are used for this investigation, namely; glycolic, mandelic and benzylic acids. These acids vary in their structures by substituting the H (α – to the carboxylic group) in the glycolic acid by one and two aromatic rings in the mandelic and benzylic acids respectively (Table (1)). Those acids are supplied by Merck- Schachardn, BDH Company, Riedel- DE Haenag Seelze. They are of analytical grade and were used without further purification.

Table (1): Abbreviations, structures and molecular weight of the studied acids

Acids	Abbreviation	Structures	M. Wt.
Glycolic acid	Glyc		75
Mandelicacid	Mand		152.15
Benzylic acid	Benzy		228.25

2- Adsorbent

Manganese dioxide (MnO_2) is used as an adsorbent in this study. It is supplied by Fluka. It was used as a colloidal and without any purification.

3- Effect contact time:

0.02 g of MnO_2 was added into a 50 mL of acid solution of initial concentration 0.05 N at 20 $^\circ C$ .Different samples were shaken for a period of 90 minutes at a time intervals of 5 and 10 minutes with continuous stirring at 90 rpm. The sample was then filtered rapidly and the acid content of the filtrates was determined by titration with 0.1 N NaOH. The experiment was repeated at different temperatures (25, 30, and 40 $^\circ C$).

4- Effect of initial concentration

The effect of initial concentration is conducted at equilibrium conditions within the range of (0.005 - 0.05 N). Four samples of different concentrations of the tested acids are prepared using distilled water as a solvent (few mLs of ethanol was used for the completion of benzylic dissolution). 0.25 g of MnO_2 was added into a 50 mL of the acid solution, shaken for 90 minutes, filtered and the amount of adsorbed acid is estimated by titration with 0.1N NaOH and using phenolphthalein as indicator.The removal efficiency is expressed in terms of adsorption percentage and calculated as follows:

$$\%Adsoption = \frac{C_i}{C_o} \times 100 \quad ---(8)$$

Where C_i, is the concentration of the adsorbed acid and C_o, is the initial concentration.

5- Estimation of thermodynamic functions of adsorption

The value of the distribution coefficient of adsorption (K) is determined at various temperatures using the following equation:

$$K = \frac{C_i}{C_o - C_i} \quad -----(9)$$

Where, (C_o- C_i) is the concentration of the free acid in solution at equilibrium. The values of ΔG° , ΔH and ΔS° are calculated by using equations (7) and (5) respectively.

6- Kinetic Study:

The kinetic study is achieved as a batch mode at the natural pH of each acid solution of concentration (0.05 N) and at 20 $^\circ C$, within a period of time ranging from 5- 60 minutes. This period is determined from the study of the effect of contact time. The experiments were carried out by adding 0.25 g of MnO_2 into 50 mL of acid solution of concentration (0.05 N). The acid solution was continuously stirred at 90 rpm with appropriate time intervals and then filtered, and the supernatant was titrated with 0.1N NaOH for the determination of the remained acid concentration. The value of the adsorbed acid concentration is calculated by

difference. The above method is repeated at different temperatures (25, 30 and 40 $^{\circ}C$).

Results and Discussion

1- Effect of contact time

The adsorption of glycolic, mandelic and benzylic acids on MnO_2 is conducted as batch experiments at initial concentration (0.05 N) and at different temperatures (20, 25, 30, and 40 $^{\circ}C$). The rate of uptake was estimated by titration with 0.1 N NaOH. The results obtained are shown in Figure (1).

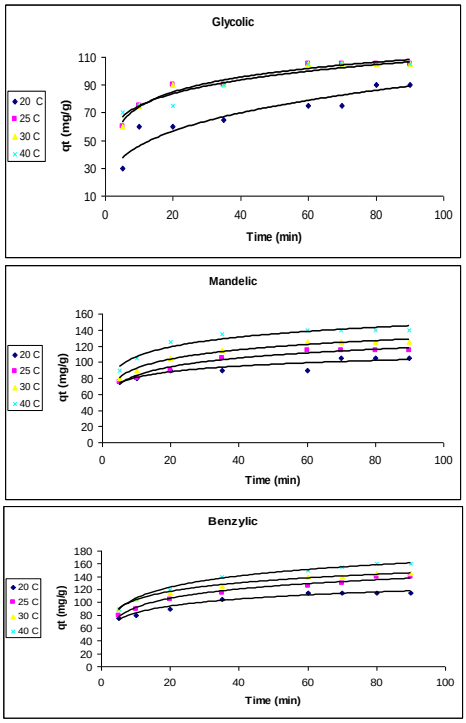


Figure (1): The variation of the adsorption efficiency with time

The results of Figure (1) indicate that, the uptake level is very fast within the first 5 minutes, being very sharp, showing high affinity between the MnO_2 surface and the acids. The adsorption process is then gradually decreased and becomes slower with the time going on. Finally, saturation is reached, beyond which, no further adsorption can take place. The results suggest that, equilibrium could be reached in about 60 minutes in all of the considered acids.

2- Effect of initial concentration

The effect of initial concentration is tested at various concentrations in the range of 0.005 to 0.05 N, while keeping all other parameters constant. The experiments were repeated at different temperatures. The adsorption efficiency is calculated using equation (8). The distribution coefficients (estimated by eq. 9) and the adsorption efficiencies are listed in Table (2).

Table (2): The values of distribution coefficients and adsorption efficiencies of the considered acids at various temperature

Conc. (N)	Temp $^{\circ}C$	Glycolic		Mandelic		Benzylic	
		K	% Ads.	K	% Ads.	K	% Ads.
0.05	20	1.941	66	4.000	80	3.166	76
	25	1.500	60	3.166	76	2.846	74
	30	1.174	54	2.846	74	2.125	68
	40	0.923	48	1.941	66	2.111	64
0.03	20	1.500	60	2.333	70	1.727	63
	25	1.307	56.6	2.000	66	1.500	60
	30	0.666	40	1.727	63.3	1.142	53
	40	0.500	33.3	1.307	56	0.875	46
0.01	20	1.000	50	1.500	60	1.000	50
	25	0.666	40	1.000	50	0.818	45
	30	0.428	30	0.666	40	0.666	40
	40	0.250	20	0.428	30	0.666	30
0.005	20	0.666	40	1.000	50	0.666	40
	30	0.250	20	0.666	40	0.428	30
	35	0.250	20	0.428	30	0.428	30
	40	0.250	20	0.250	20	0.250	20

The results of Table (2) show that, the adsorption of acids by MnO_2 is highly dependent on the acid concentration. The amount of adsorbed acid by MnO_2 is increased by the increase of acid concentration at definite temperature. Comparing the adsorption efficiencies of the three acids considered in this investigation, the results indicated that, the adsorption efficiency increased in the following order at certain temperature: Glyc. < Benzy < Mand .Since the acids are expected to exist as anions in their aqueous solutions due to the ionization processes. The attachment of acids onto the MnO_2 surface will be by their anion forms, therefore, the more stable anion is the more liable acid to be adsorbed. Due to the ability of the negative charge of the benzylic anion to be spread on two aromatic rings and that of mandelic anion to be distributed on one aromatic ring, the stability of the anion forms of the studied acids, are in the following order: Glyc < Mand < Benz. Accordingly, the adsorption efficiency of the benzylic acid is expected to be higher than that of mandelic acid. The experimental investigation showed results of opposite direction. This could be due to the steric effect caused by the two rings present in the benzylic structure.

3- Effect of the initial pH

An attempt was made to investigate the influence of the initial pH solution on the removal of acid by adsorption on MnO_2. Three different pH's; at natural pH of acid solutions (2.27 of glycolic, 2.63 of mandelic and 2.81 of benzylic acids), neutral (pH=7) medium and at basic (pH= 9) medium were investigated. The results obtained are listed in Table (3)

Table (3): Effect of initial pH solution on adsorption of acid by MnO_2 at 20 °C

Compd.	pH	%Ads.	K
Glycolic	2.27 *	66	1.941
	7.00	60	1.500
	9.00	46	0.851
Mandelic	2.63 *	80	4.000
	7.00	68	2.125
	9.00	58	1.381
Benzylic	2.81 *	76	3.166
	7.00	62	1.631

* Natural pH

The results of Table (3) indicate that, the three acids vary in the same trend in which the percentage of the acid removal is decreased when increasing the initial pH of the acid solution from acidic (natural pH) to neutral (pH=7) medium. This can be attributed to the decrease of the ionization abilities of the acids in the neutral medium, which in turn lowers the adsorption efficiency, since they are attached to the MnO_2 surface as anions.

The lowest adsorption efficiency is observed in the basic (pH=9) medium, which may be due to the presence of excess of OH^- ions competing with the acid anions to be attached to the adsorption sites. The results of this study may conclude that, the initial pH of the acid solution plays an important role in such investigation.

4- Effect of temperature

The effect of varying temperatures, when keeping other parameters constant, on the removal of the acids considered in this study by MnO_2 is investigated in the range of 20-40 °C at different initial concentrations. The results obtained are portrayed in Table (2). The results of Table (2) showed that, the amount of adsorbed acid decreased with the increase of temperature for all of the studied acids and at all of the concentration selected for this investigation. These observations suggest that, the adsorption of the studied acids is exothermic process and is physical in nature in which the increase of temperature increases the desorption process.

5- Calculation of thermodynamic functions:

The various intermolecular forces between the acids and MnO_2 can specify the direction of the adsorption process, the nature of these forces and the order of the adsorptions system. The thermodynamic functions are a good measure for such determination. The estimation of the heat of adsorption is conducted depending on the adsorption isotherm knowledge at various temperatures. The values of the adsorption distribution coefficients (K) at equilibrium determined at different temperatures according to equation [9] are shown in Table (2). The values of ΔH and $\Delta S°$ evaluated from the slope and intercept of the straight line obtained from plotting ln K versus 1/T by employing equation (5) respectively. This straight line is shown in Figure (2).

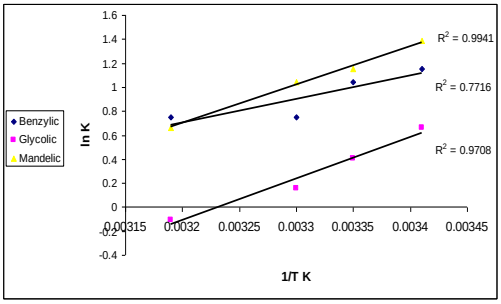


Figure (2): The relation between ln K versus 1/T for the three acids considered is this study.

The values of $\Delta G°$ are estimated by using equations (7).The values of $\Delta S°$, ΔH and $\Delta G°$ for the adsorption of the studied acids on MnO_2 at two different concentrations (0.05 and 0.05 N are listed in Table (4)).

Table (4): The adsorption thermodynamic functions of considered acids at different concentrations

	0.005 N			0.05 N		
	$-\Delta G^o$ $kJ.mol^{-1}$	$-\Delta H$ $kJ.mol^{-1}$	$-\Delta S^o$ $J.mol^{-1}.K^{-1}$	$-\Delta S^o$ $J.mol^{-1}.K^{-1}$	$-\Delta H$ $kJ.mol^{-1}$	$-\Delta G^o$ $kJ.mol^{-1}$
Glyc	1.61556	27.940	90.165	110.401	30.465	990.15
Mand	3.3771	26.84	79.952	179.923	52.660	0
Benzy	2.80741	16.129	45.746	122.37	34.754	990.15

Looking at the results of Table (4) one may conclude that, the negative value of $\Delta G°$ confirms that, the acids adsorption on MnO_2 surface is a spontaneous process, while the negative sign of ΔH is an indication to the exothermic nature of adsorption system under consideration, and the value of ΔH refers to the type of forces that control the adsorption process which are physical in nature (< 40 kJ .mol^{-1}). The negative value of ΔS^o explains the increase in the system order due to adsorption process. Comparing the values of ΔH and ΔS^o obtained for the three acids at the highest (0.05 N) and lowest (0.005 N) concentrations chosen for this study showed an increase in the opposite direction.

At (0.05 N)	**Mand**	>	**Benzy**	>	**Glyc**	
ΔH	-52.66		- 34.75		-30.46	$k\ J\ .\ mol^{-1}$
ΔS^o	-179.92		-122.37		-110.40	$J.\ K^{-1}.\ mol^{-1}$
At (0.005 N)	**Benzy**	<	**Mand**	<	**Glyc**	
ΔH	-16.129		-26.84		-27.94	$k\ J\ .\ mol^{-1}$
ΔS^o	-45.74		- 79.95		-90.16	$J.\ K^{-1}.\ mol^{-1}$

These results may indicate that, these values are significantly affected by the initial concentration. At higher concentration, the competition among the acid molecules to be attached on a certain number of

active sites present on a specific amount of the MnO_2 surface is increased. This, in turn, is affected by the size of molecule that elevates the steric interactions. The effect of steric congestion is seemed to be weakened at lower concentration.

6- Kinetic study:

The kinetic of acids adsorption onto MnO_2 was investigated using two different models; the pseudo first order (eq.3) and pseudo second order (eq.4) kinetics. The study is achieved as batch experiments at certain conditions of initial concentration (0.05 N), natural pH of each acid and at different temperatures. According to the pseudo first order model, the value of k_1 and calculated qe are evaluated from the slope and intercept of the straight line resulted from plotting log (qe- qt) versus time respectively (Figure (3)).

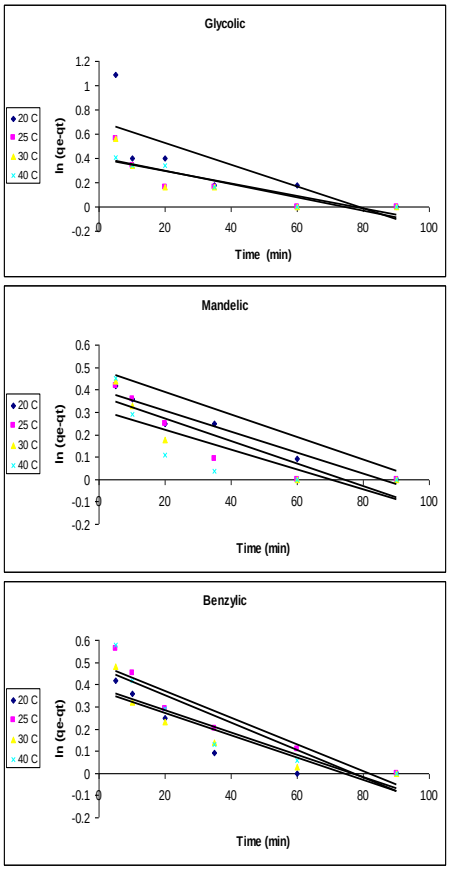


Figure (3): lines obtained from the application of first order model of the adsorption system

In a similar way, the application of the pseudo second order model is carried out by plotting t/qt versus time. The values of k_2 (g mg^{-1}. min^{-1}) and calculated qe (qe thero) are estimated from the intercepts and the slopes of the obtained straight lines respectively (Figure (4)).

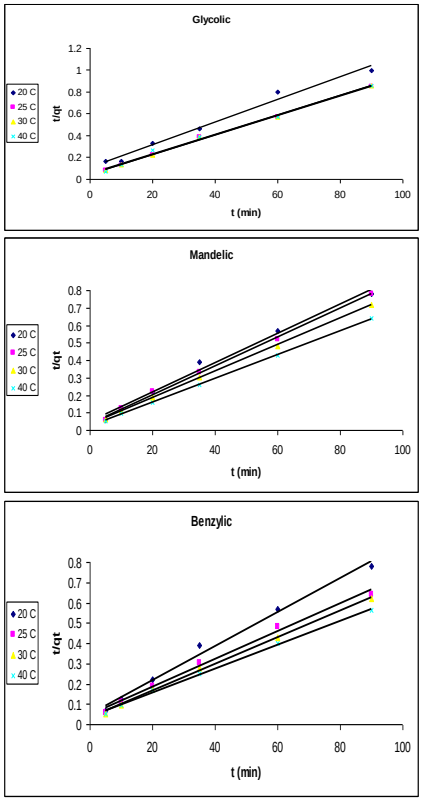


Figure (4): The fitting of the adsorption data to the second order model

Comparison between the experimental values of qe and the calculated ones obtained from the fitting of first and second order kinetic models on the adsorption data are given in Tables (5) and (6) respectively.

Table (5): Results of the kinetic study obtained from the application of first order model on adsorption data

Temp °C	k_2 (g.mg^{-1}.min^{-1})	R^2	q_e theo (mg/g)	q_e exp (mg/g)
Glycolic acid				
20	0.001060	0.9847	96.10	90
25	0.001770	0.9975	111.11	105
30	0.001788	0.9976	111.11	105
40	0.001494	0.9935	121.35	105
Mandelic acid				
20	0.001380	0.9870	119.00	115
25	0.001776	0.9970	121.95	115
30	0.001799	0.9993	131.57	125
40	0.002080	0.9997	147.05	140
Benzylic acid				
20	0.001320	0.9905	121.95	115
25	0.000979	09931	147.05	140
30	0.001280	0.9978	151.51	145
40	0.000923	0.9978	169.49	160

Table (6): Application of second order kinetic model on adsorption data

Temp °C	k_1 (min^{-1})	R^2	q_e theo (mg/g)	q_e exp (mg/g)
Glycolic acid				
20	0.02049	0.5857	5.030	90
25	0.01266	0.7068	2.546	105
30	0.01266	0.7068	2.546	105
40	0.01197	0.884	2.506	105
Mandelic				
20	0.01082	0.946	2.525	115
25	0.01174	0.8213	2.361	115
30	0.01174	0.2485	3.129	125
40	0.01013	0.6274	2.045	140
Benzylic acid				
20	0.01174	0.8202	2.316	115
25	0.01381	0.8816	3.080	140
30	0.01150	0.8222	2.426	145
40	0.01427	0.812	2.970	160

According to the results presented in Tables (5 & 6), the second order model provided best fit with the experimental data of adsorption. High correlation coefficients (R^2 close to unity) are obtained. The calculated values of adsorption capacities (qe theo) are consistent with the experimental values of (qe exp). Meanwhile, the application of Lagergren equation on adsorption data gave weak correlation coefficients. The calculated values of qe are too low compared to the experimental qe values, for all of the adsorption data of the three acids involved in this study.The above results confirm that, the adsorption of the three acids considered in this study on the surface of MnO_2 obeys and follows the pseudo second order kinetic model.

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دراسة حركية وثرموداينمكية لامتزاز بعض الحوامض العضوية باستخدام MnO_2

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(تاريخ الاستلام: ٨ / ٥ / ٢٠١١ ---- **تاريخ القبول:** ٢٦ / ١٠ / ٢٠١١)

الملخص

تضمن هذا البحث دراسة كفاءة امتزاز بعض الأحماض العضوية (الكلايكوليك و المندليك و البنزيليك) والتي تتفاوت بصيغها التركيبية من محاليلها المائية.وقد هدفت الدراسة التوصل إلى فهم عميق وواضح لميكانيكية امتزاز هذه الأحماض وعلاقتها بالتركيب ألجزيئي لها. وقد أنجزت التجارب العملية باستخدام طريقة الدفعة الواحدة. لقد أشتمل البحث على دراسة العوامل المؤثرة على الامتزاز مثل زمن التماس والتركيز الابتدائي والدالة الحامضية والامتزاز ودرجة الحرارة.أظهرت النتائج إن زيادة التركيز الابتدائي يزيد من كفاءة الامتزاز وان الدالة الحامضية الابتدائية لوسط الامتزاز تلعب دورا أساسي في التحكم بطريقة ارتباط الجزيئات الممتزة بالسطح ألماز . وتبين النتائج إن أعلى كفاءة امتزاز تم ملاحظتها في الوسط ألحامضي (عند الدالة الطبيعية لمحاليل الحوامض) .وقد أثبتت الدراسة إن الأحماض العضوية المدروسة ترتبط مع سطح الـ MnO_2 بشكلها ألايوني السالب . وقد حسبت الدوال الثرموداينميكية لنظام الامتزاز بالاعتماد على النتائج المحصل عليها من دراسة تأثير درجة الحرارة عند مدى من التراكيز (٠,٠٠٥-٠,٠٥)N.أشتمل البحث أيضا على دراسة حركية امتزاز هذه الأحماض على سطح الـ MnO_2 وذلك من خلال تطبيق نموذجي معادلتي المرتبة الأولى الكاذبة والثانية الكاذبة في عدد من درجات الحرارة وعند تركيز ثابت . أظهرت النتائج إن النظام المدروس يتبع نموذج المرتبة الثانية الكاذبة.