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Kinetic and Equilibrium Studies of the Adsorption of Dichalcones on Activated Carbon

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Abstract

This work is involved with the evaluation of the adsorption efficiency of an activated carbon prepared in our labs for the removal of two dichalcones namely; 2,6-Bis(4-Nitrobenzylidene) cyclohexanone and 2,6-Bis (4-Hydroxybenzylidene) cyclohexanone from their aqueous solutions. These chalcones were synthesized from the reaction of appropriate aldehydes (4- Nitro (or Hydroxy) benzaldehyde) and ketone (Cyclohexanone) and were employed as adsorbate for accomplishing this study. Optimization of the conditions of the systems considered were investigated concerning factors influencing adsorption efficiency such as adsorbent dose, time of contact, and chalcone initial concentration. Concentration of the adsorbed chalcones were determined spectrophotometrically. Calibration curve was constructed at the (λmax) of each chalcone. A batch method with single component systems were carried out for achieving the adsorption processes. The Freundlich and Langmuir adsorption isotherms were fitted to the practical data of adsorption at equilibrium within a concentration range ($1*10^{-4}$ – $9*10^{-4}$M). The results showed a better fit of Freundlich isotherm to the experimental data of chalcone no.2, while, the Langmuir isotherm exhibited better fit to chalcone no.1. Kinetic study is performed by applying, pseudo first order and pseudo second order equations fitted to the practical data of the adsorption on activated carbon. The observed results indicated that the studied system are best followed the pseudo second order equation. This was determined by the values of R^2 close to unity (R^2 = 0.999 and 1.0) when compared with the first order (R^2 = 0.9169, 0.8829). In addition, the values of q_{ex} in the first order reaction are not consisted with the q_{cal} values.

Introduction:

Chalcones are aromatic compounds with two aromatic rings having diverse array of substituents. Chalcones have linear or approximately planar structures since they have aromatic rings connected by an electrophilic bridge consisted of three carbon α, β-unsaturated carbonyl system. These structure possesses conjugate double bonds with a delocalized π electron along both of the aromatic rings [1,2]. The synthesis of chalcones has created a broad attraction for organic and medicinal chemists since they are representing

starting materials for the synthesis of a large number of biologically important heterocyclic such as benzothiazepine, pyrazolines, 1,4-diketones, and flavones [2,3].

Chalcones and their derivatives have attracted increasing attention due to numerous pharmacological applications. They have displayed a broad spectrum of various biological activities such as anti-microbial [4], anti-inflammatory [5,6], analgesic [7], anti-malarial [8], anti-cancer [9-11], anti-oxidant [12-14], anti-filarial [15], antifungal [16-17] etc. In Another study a number of chalcone derivatives showed thermal diffusivities at room temperature measured by using photo acoustic technique [18]. Thermal diffusivity was found to vary with the number of methoxy groups conjugated in chalcones structure.

In a recent study [19], various substituted chalcones were used as corrosion inhibitors for carbon steel in HCl acid solution studied by different techniques relying on the adsorption efficiency of chalcone substituents on the surface of carbon steel which were found to follow Langmuir adsorption isotherm. The variation in inhibition efficiency is adopted by the type of the substituent groups in the benzene ring. The adsorption of chalcones was found to be dependent mainly on certain physico-chemical properties of the molecule and its substituents such as the electronic donation or withdrawing nature of the functional groups, its spatial distribution (steric effect), conjugation between electron density of functional groups and π orbital character of the aromatic rings [20,21]. Now days chalcones are being synthesized with wide choices of substituents, so they have acquired a great importance and being applied and used in various fields. For this reason, some chalcones and/or their degradation products could be toxic and carcinogenic, and their disposal into wastewater without prior treatment may create a source of pollution that damages the aquatic life. So, a proper treatment is needed and chalcones removal from wastewater became essential.

Adsorption has been proven to be a reliable method [22]. It is environmentally friendly, and economic with considerable potential for removal of many pollutants. Activated carbon is most widely used as adsorbent for the removal of biologically resistant organic pollutants from aqueous solutions. The high cost of the commercial activated carbon demanded the search for alternative and low cost adsorbent [22-23].

The aim of this study is: determination of the adsorbent (activated carbon) capacity of an adsorbate (dichalcone) by adsorption isotherm when present at equilibrium between adsorbate-adsorbent systems. The equilibrium is achieved when the adsorbent capacity is reached and the adsorption rate is equal the desorption rate. Significance of the adsorption isotherms can provide valuable information regarding the nature of intermolecular interactions involved at solid-liquid interface which helps in designing the adsorption system for effective removal of pollutants from the contaminated water since the equilibrium data are the fundamental requirements for this purpose [24,25]. Two of the well-established isotherms; Langmuir and Freundlich, will be applied onto the practical results of adsorption of the considered systems at constant temperatures. The isotherm parameters to be used for describing the nature of the adsorption systems, determination and investigating the kind of interactions between the chalcone and adsorbent surfaces.

Adsorption Isotherms:

Practically, the adsorption isotherm is the most conveniently and widely used method for determining the equilibrium in an adsorption system. The adsorption isotherm gives valuable information concerning the magnitude of the enthalpy of adsorption and the relative adorability of an adsorbate on a given adsorbent with respect to a chosen standard. The adsorption data can be represented by several isotherms cited in the literature [26]. The most important which being used in this research are Langmuir, and Freundlich, isotherms.

Langmuir Isotherm

It is the first theoretical adsorption isotherm derived by the American scientist Langmuir [27] by assuming a perfect smooth and homogeneous surface of adsorbent. On this surface only one adsorbate molecule reacts with each active site with no interactions among the adsorbate species that forming monolayer phase attached to the adsorbent surface.

The Langmuir isotherm can be expressed by several linear equations, the most popular one is the following equation:

$$\frac{Ce}{qe} = \frac{1}{bQ\max} + \frac{Ce}{Q\max} \text{.....(1)}$$

The Ce (mg/L) representing the adsorbate concentration in solution at equilibrium, where qe (mg/g) is the adsorption capacity at equilibrium and shows the amount of chalcone (mg) adsorbed per gram of carbon. Qmax (mg/g) is the theoretical maximum monolayer capacity of adsorbent and b is the Langmuir constant that related to the apparent energy of adsorption.

Freundlich Isotherm:

The equation of this isotherm is a modification achieved by Freundlich on the Boedecker isotherm [28]. It has been widely adopted to characterize the adsorption experiment. In contrast to Langmuir isotherm [29], it provides no information on the monolayer adsorption capacity. Freundlich isotherm is applicable to the adsorption on heterogeneous surfaces with the presence of interactions among the adsorbed molecules. The linear form of this isotherm can be expressed as follow:

$$\text{Log qe} = \log K_F + \frac{1}{n} \log Ce \quad \text{……(2)}$$

The K_F represent the Freundlich constant and its value is indicating to the adsorbent capacity that implicated with the bonding energy, n is called as heterogeneity factor and representing a measure of the deviation of the adsorption from linearity. A linear adsorption is obtained when n=1, while n<1 indicate to un-favoured adsorption or chemical adsorption, and when the value of n>1(n= 1-10) a favourable physical adsorption is obtained.

Kinetic study

Two of kinetic models were fitted to the experimental data of the studied systems namely, the pseudo first order eq(3) and second order eq(4) systems [30]. In the first order

$$\ln(q_e - q_t) = \ln q_e - k_1 t \text{ .....(3)}$$

Where q_e and q_t (mg/g) represent the adsorption capacity at equilibrium and at t time (min) respectively. Plotting $\ln(q_e - q_t)$ versus t should give a straight line of correlation

coefficient (R^2) close to unity and standard deviation (SD) less than 5% of the experimental error.

The value of experimental adsorption capacity (q_{exp}) must be consistent with calculated one (q_{cal}). If those two conditions or one of them are not applied, the system cannot be considered as pseudo first order.

In the pseudo second order adsorption: the rate constant of this kinetic model depends on the adsorption capacity of the solid adsorbed and not on the adsorbate concentration. The limited step of the rate constant in the adsorption mechanism according to this model include forces those resulted from electron participation or substitution between chalcones and activated carbon adsorbate.

The pseudo second order reaction can be presented by equation (4) as follow:

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} t \ldots\ldots\ldots\ldots (4)$$

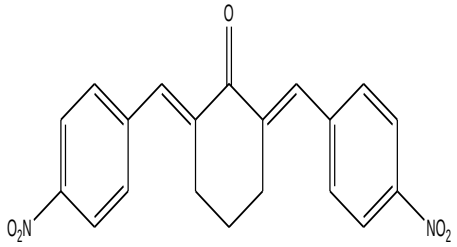
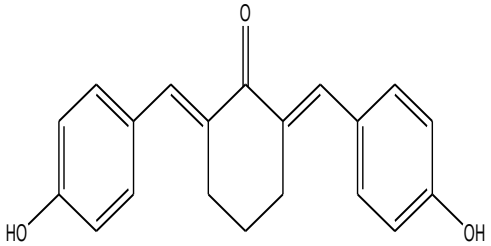
This model to be applicable, on the experimental data of adsorptions the plot of $\frac{t}{q_t}$ versus t should give a straight line and the calculated adsorption capacity (q_{cal})(from the plot) and agree with the experimental values (q_{exp}).

Experimental Part

Chemicals:

All chemicals employed in this study were supplied by the BDH, Fluka and Merck chemical companies and they were pure and used without needing further purification. The activated carbon as adsorbent, and dichalcones employed as model adsorbate in this work were synthesized by the reaction of appropriate aldehydes (4-Nitro Benzaldehyde and 4-Hydroxy Benzaldehyde) and ketone (cyclohexanone) and were supplied by another group of our colleagues [31, 32]. The chemical structures of the dichalcones are illustrated in Table 1.

Table 1: Name, structure, colour, melting point and λmax of the chalcones used in this study

Chalcone No.	Reaction between		Reaction Period (min)	Structure and Name	Yield%	mp°C color	λ_{max}
	Aldehyde	Ketone					
1	4-Nitro Benzaldehyde	Cyclo-Hexanone	20	 2,6-Bis(4-Nitrobenzylidine) Cyclohexanone	82	200-201 Broun	332
2	4-Hydroxy Benzaldehyde	Cyclo-Hexanone	60	 2,6-Bis(4-Hydroxybenzylidine) Cyclohexanone	76	190-192 Green	312

Adsorbents

Activated carbons [31] used as adsorbents for performing this study was prepared in our laboratory by treating asphalt (obtained from a city named as Beji located in the middle part of Iraq) with poly ethylene and KOH with ratio (1:2), and then heated up to 400^{o}C for 2 hours, then radiated by microwave with power of 630 watts for 16 min [31]. The obtained carbon has the properties listed in Table 2.

Table 2: The properties of the prepared carbon

Power of radiation (watt)	630	% Humidity	16.2
Time of radiation (min)	16	% Ash content	3.71
% Yield	51.3	Iodine number (mg/gm)	600
Density (gm/cm^3)	0.57	Methylene blue (mg/gm)	145

Chalcone Solution

The chalcones synthesized in this study are used as environmental pollutants. Solutions of chalcones (10^{-3}M) were prepared by dissolving certain amount in a solvent composed of %50 (v:v) mixture of ethanol-water, which were used as a blank during the measurements. Calibration curve was performed by serial dilutions [32].

Procedure of Adsorption:

The adsorption experiments were carried out as batch method. Certain amounts of adsorbent were added into several flasks, each of them containing 10 mL of chalcone solution and 0.02 gm of adsorbent. The flasks are then stirred at 100rpm using programmable water bath shaker (Julabo SW23) at the required temperature. The solution was then filtered and the concentration of chalcone at equilibrium was determined in the supernatant spectrophotometrically with UV-VIS spectro-photometer of CECIL CE-1021 type. The adsorption capacity is evaluated by the following equation:

$qe = (Co-Ce) \times V/m$ …….. (5)

The adsorption efficiency (of adsorption (Ads%)) is evaluated by the equation:

$Ads\% = (Cads/ Co) \times 100$ ……..(6)

Where Co is the initial concentration (mg/L) and Cads (mg/L) is the concentration of the adsorbed chalcone. V is the volume of solution (L) and m is the weight of adsorbent (gm).

Results and Discussion

Effect of dose

The aim of this study is to find a suitable dose that maintaining the system in an equilibrium and without total removal of the colour in order to achieve the thermodynamic study [33]. The results of Table 3 show that the adsorption capability declines with the raise of the quantity of adsorbent for a certain volume of solution. This is escorted by the elevation of adsorption efficiency (Adsorption%). This case could occur due to the increase of active locations on the solid adsorbent when increasing its amount. The adsorbent dose (2mg/L) is chosen to perform the further study.

Table 3: Effect of dose of adsorbent capacity to adsorb chalcone

Chalcone	Dose (gm/L)	qe(mg/gm)	Adsorption%
1	1	101.038	88.628
	2	84.788	92.108
	4	33.864	94.712

Influence of initial concentration

The investigation of primary concentration effect is carried out in the range of $1*10^{-4}$ to $9*10^{-4}$ Molar, at constant temperature and pH in a solvent of mixture %50 v:v ethanol–water. This investigation is aimed to determine the range of concentration that can be used later for the application of different isotherms on the adsorption data at various temperatures.

The results obtained are listed in Table 4. The results showed that, elevation of concentration decreasing the adsorption efficiency due to the increase of competition among the bulky chalcone molecules to be adsorbed on a certain numbers of active sites present on a certain weight of adsorbent [33].

Table 4: Effect of concentration on the adsorption efficiency on different adsorbents at 298K

Chalcone		Concentration (M)				
		1×10^{-4}	3×10^{-4}	5×10^{-4}	7×10^{-4}	9×10^{-4}
1	Adsorption efficiency (Adsorption%)	86.180	84.965	81.232	76.546	72.092
2		94.290	90.313	85.647	84.427	81.318

Effect of contact time

This investigation is aimed to find the time required for the studied system to reach equilibrium in order to achieve both the thermodynamic and kinetic studies. Table 5 indicates that, the rate of adsorption increases sharply in the first 10 minutes then decreases gradually and reaches equilibrium with 50-70 minutes in the studied chalcones. The time 70 min is selected for performing the remained studies.

Adsorption isotherm

Isotherm Freundlich:

The Freundlich equation is applied to the practical data of the two chalcones by plotting $\ln q_e$ versus $\ln C_e$ as shown in Figure 1. The constants of Freundlich isotherm are shown in Table 5. Chalcone (No2) is better fitted to this isotherm than chalcone (1) according to the R^2 values. The values of n indicate to favourable adsorption. The values of K_f which is related to the adsorption capacity has higher value for chalcones (No2).

Table 5: Time effect
on the adsorption ratio at 298K and $3*10^{-4}$ M

Chalcone No 1			Chalcone No 2		
Time (min)	Ct (mg/L)	qe (mg/g)	Time (min)	Ct (mg/L)	qe (mg/g)
10	5.47	36.57	10	10.92	43.74
20	5.13	36.74	20	9.65	44.38
30	4.11	37.25	30	8.48	44.96
40	3.60	37.50	40	7.62	45.39
50	3.26	37.67	50	7.36	45.52
60	2.85	37.87	60	6.96	45.72
70	2.72	37.94	70	6.90	45.74
80	2.75	37.93	80	6.85	45.77
90	2.72	37.94	90	6.95	45.72

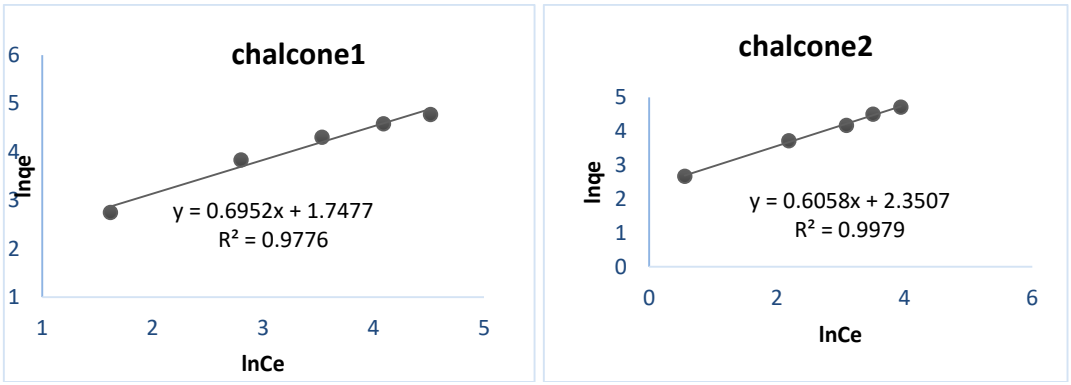


Figure 1: The Freundlich isotherm of the two chalcone

Table 6: The Freundlich isotherm constants of the two chalcones

Comp.	K_f	n	R^2
1	55.937	1.438	0.9776
2	224.233	1.650	0.9979

Langmuir isotherm

According to Langmuir equation, chalcone No1 is better fitted (R^2 =0.9965) than chalcone (No2) giving higher theoretical adsorption capacity (185.185 mg/g). The values of n (Freundlich constant) and value of b (Langmuir constant) which related to the adsorption energy are both higher values in chalcone 2 than 1 which is agree with adsorption capacity in Table 4.

Table 7: The Langmuir isotherm constants of the two chalcones

chalcone	Q(mg/g)	b (L/mg)	R^2
1	185.185	0.0192	0.9965
2	153.846	0.0442	0.9426

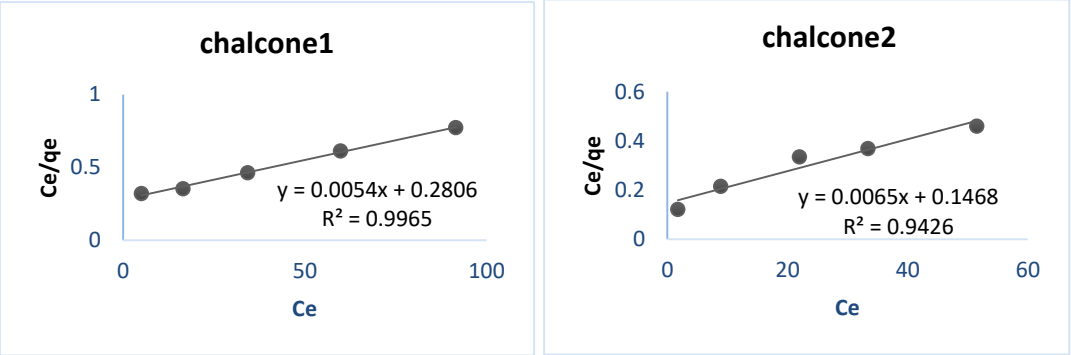


Figure 2: The Langmuir isotherm of the two chalcones

Kinetic study

Two kinetic model applied in this study are, the pseudo first and pseudo second order equations. Results of the fitting of the first model (eq.3) are given in Table 8 and shown in Figure 3. The results obtained indicating that, the studied system is not first order since weak linear relation are obtained (R^2= 0.9160 and 0.8829) in addition, there is no agreement between q_{exp} and q_{calc}.

The application of the second order eq (4) to fit the experimental data of the studied system gave the results listed in Table 9 and illustrated in Figure 4.
Better agreement between q_{exp} and q_{calc} are seen, higher values of R^2 is noticed (R^2 =0.9999 and 1.0). The above results proved that, the studied systems are applied to the pseudo second order equation.

The rate constant of the second order reaction can be used to estimate the initial rate of adsorption h ($mg.g^{-1}.min^{-1}$) by employing the following equation (7)

$$h = k_2 (q_e)^2 \quad \ldots\ldots\ldots\ldots (7)$$

The second order rate constant (k_2) obtained and the values of (h) for chalcones are given in Table 9. The results indicated that the initial rate (h) is higher than the rate constant (k_2) which agree with the results seen in Table 5 when studying the effect of time.

In the same time the values of the initial rate and the rate constant k_2 of chalcone 1 is higher than those of chalcone 2. These results are consistent with the experimental result illustrated earlier [34,35].

Table 8: Results of application the first order equation to fit the experimental data of the studied system

Chalcone	q_e(cala) mg/g	K_1 (min^{-1})	q_e(exp) mg/g	R^2
1	15.933	0.0566	37.94	0.9169
2	93.907	0.0836	45.74	0.8829

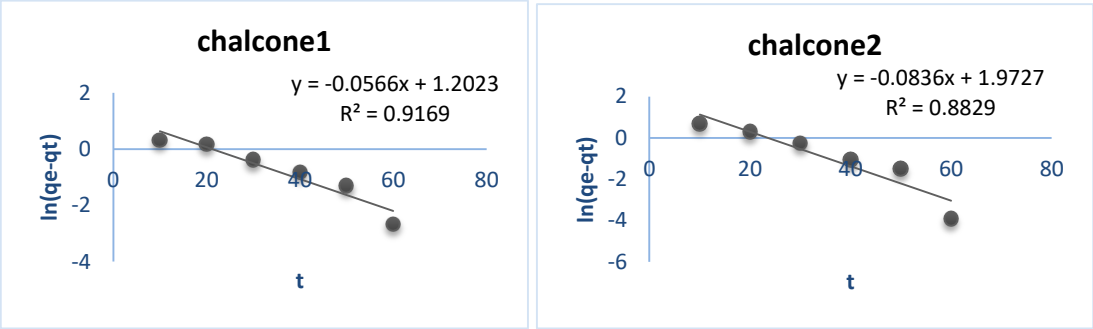


Figure: 3 The application of the first order equation to fit the experimental data of the studied system

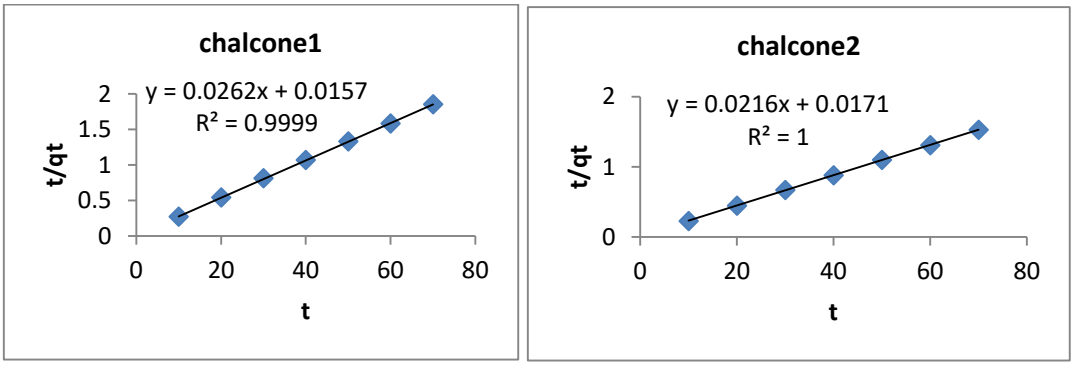


Figure: 4 The plot of t/q_t versus t when applying the second order equation to fit the experimental data of the adsorption system

Table 9: Results obtained from the application of the second order equation on the studied system

Chalcone	q_e(cala) mg/g	K_2 (g.mg^{-1}.min^{-1})	h (mg.g^{-1}.min^{-1})	q_e(exp) mg/g	R^2
1	38.167	0.04372	63.687	37.94	0.9999
2	46.296	0.02728	58.469	45.74	1

Conclusion

This research is a test of the possibility of removing chalcones or the products of its biological decomposition from their aqueous solutions by adsorption. Despite the low solubility in water, chalcones are removed by using a mixture of water and ethanol with 50% volume was used as a solvent. The optimum conditions for the adsorption system under study have been estimated. Two models of adsorption isotherms were also applied to the operation data of the adsorption system. The Langmuir isotherm showed greater applicability than Freundlich. The application of two kinetic models to fit the experimental data of adsorption showed that the adsorption of chalcone follows the pseudo-second order reaction. The negative charge on the carbon surface could be the reason for the increase of the rate constant as well as the initial rate in favour of chalcone no.1 due to the presence of the two electron withdrawing groups

(NO_2) which increasing the positive charge on this compound and its attraction to the carbon surface. The results obtained so far in this study agree with other studies found in the literature [31-38].

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We dedicate this research to the soul of our colleague, Professor Dr. Natiq Ghanem Ahmed, who left us with his body and still lives in our hearts.

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دراسات حركية واتزان لامتزاز الجالكونات على الكاربون المنشط

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الخلاصة:

يساهم هذا العمل في تقييم كفاءة امتزاز الكربون المنشط المحضر في مختبراتنا لإزالة اثنين من الجالكونات وهما؛ Bis (4-Nitrobenzylidene) -2,6 cyclohexanone و Bis (4-Hydroxybenzylidene) -2,6 cyclohexanone)من محاليلها المائية. تم تحضير هذه الجالكونات من تفاعل الألدهيدات المناسبة (4- Nitro (or Hydroxy) benzaldehyde) وكيتون (Cyclohexanone) وتم استخدامها كممتزات لإنجاز هذه الدراسة. تم ايجاد افضل الظروف للأنظمة المدروسة فيما يتعلق بالعوامل التي تؤثر على كفاءة الامتزاز مثل كمية المادة المازة ووقت التلامس والتركيز الأولي للجالكون. تم تحديد تركيز الممتز بالطيف الضوئي. تم عمل منحنى المعايرة عند (λmax) لكل جالكون. تم استخدام طريقة الدفعة الواحدة بأنظمة مكون واحد لتحقيق عمليات الامتزاز. تم تطبيق ايزوثيرم فرندلخ ولانكماير على البيانات العملية للامتزاز عند التوازن ضمن مدى من التراكيز (1*10-4 – 9*10-4 M). أظهرت النتائج التي تم الحصول عليها من ايزوثيرم فرندلخ انطباقا على البيانات العملية للجالكون رقم 2، بينما أظهرت نتائج ايزوثيرم لانكماير توافقًا أفضل مع الجالكون رقم 1. تم إجراء الدراسة الحركية عن طريق تطبيق معادلات الرتبة الأولى الكاذبة والرتبة الثانية الكاذبة على البيانات العملية لامتزاز الكربون المنشط. أشارت النتائج المحصل عليها إلى انطباق النظام المدروس على معادلة الرتبة الثانية الكاذبة. تم تحديد ذلك من خلال قيم R2 القريبة من الوحدة (R2 = 0.999 و 1.0) عند مقارنتها بالرتبة الاولى الكاذبة (R2 = 0.9169، 0.8829). بالإضافة إلى ذلك، فإن قيم qex المحصل عليها من الرتبة الأولى لا تتطابق مع قيم qcal.