

## Adsorption of Nickel Ions From Aqueous Solutions by Green Tea Waste

Mohanad Hazim Najj HALBOOS\* Aayad Ammar SAYHOOD\*\* and  
Hayder Mustafa KAMAL\*

\*Department of Chemistry\ College of Science\ Kufa University

\*\*Department of Basic Sciences\ College of Dentistry\ Kufa University

### Abstract:

In this research, the green tea waste (GTW) was utilized for the removal of Ni(II) ion from aqueous solutions. Adsorption experiments were conducted under various conditions where the batch method was used to determine the best adsorption ratio and the adsorption capacity for the concentration used of Ni(II) ion between 10-100 mg/L, the results show that the best concentration was 10 mg/L where the adsorption ratio was 92% and the adsorption capacity was 2.3 mg/g. The adsorbent dose was 4 g/L, and the shaking time found that it equal to 60 min and the best pH value between (3-8). The effect of temperature and thermodynamic functions wear also studied, the adsorption capacity was increased with increased the temperature and the reaction was endothermic. The equilibrium isotherm data was analyzed using the Langmuir, Freundlich and Tempkin equations. The rate constant  $k_2$  for the adsorption of Ni(II) ion is equal to 0.0571 g/mg.min.

**Keywords:** The green tea waste (GTW), nickel ions, adsorption, batch method, Langmuir, Freundlich and Tempkin isotherm and the rate constant.

### Introduction:

Contamination of water by toxic heavy metals through wastewater discharge by industrial activity is one of the major environmental issues. Rapid industrialization has seriously contributed to the release of toxic heavy metals in the water streams <sup>(1)</sup>. Mining, electroplating, metal processing, textile and battery manufacturing industry are the main source of heavy metals contamination <sup>(2)</sup>. Those activities polluted the water streams especially rivers and made them lose their potential value and beneficial use <sup>(3)</sup>. Heavy metals cannot be metabolized and bio-accumulate in organism body. These toxic metals can move through the biological chain thereby reaching human being and leading to chronic and acute ailments. Heavy metal toxicity can result in damage or reduced mental and central nervous function, lower energy levels and damage to blood composition, lungs, kidneys liver and organs <sup>(4)</sup>.

The most common adverse health effect of nickel in humans is an allergic reaction; large amounts of nickel can cause lung and nasal sinus cancers <sup>(5)</sup>. Nickel is also toxic, especially to activated sludge bacteria. The presence of Ni (II) is detrimental to the operation of anaerobic digesters used in wastewater treatment plants. Hence, it is essential to removal Ni from industrial waste water before transport and cycling into the nature environment <sup>(6)</sup>. Removal of heavy metals from wastewater is usually achieved by physical and chemical processes which include precipitation, coagulation, reduction, membrane procession change and adsorption <sup>(7)</sup>. All the chemical methods have proved to be much expensive and less efficient than the adsorption process. In addition, chemical methods increase the pollution load on the environment <sup>(8)</sup>. Adsorption has advantages over the other methods because of simple design with a sludge free environment and can involve low investment in term of both initial cost and land required <sup>(9)</sup>.

In recent years, Different adsorbents for nickel ions like loofa sponge immobilized<sup>(10)</sup>, aspergillus niger<sup>(11)</sup>, sphaerotilus natans<sup>(12)</sup>, grape bagasse<sup>(13)</sup>, peat moss<sup>(14)</sup>, activated sludge<sup>(15)</sup>, activated carbon<sup>(16)</sup>, rice husk<sup>(17)</sup>, bacterium *Bacillus thuringiensis*<sup>(18)</sup> and yeast<sup>(19)</sup>.

In our study, used green tea waste (GTW) for removal of Ni(II) from aqueous solution by adsorption under various conditions.

## Experimental Part:

### Apparatus:

Flame Atomic Absorption Spectrophotometer model (AA-6200 Shimadzu, Japan) with an air-acetylene flame was used throughout this research work. Shaker water bath SB-16, England, Oven, Memmert 30C<sup>0</sup>-220C<sup>0</sup>, W. Germany, Centrifuge machine, Hitachi EBA35, Japan and pH-meter model (Ionlop-720).

### Adsorbent preparation:

15 g of green tea waste (GTW) used in this study was washed and treated with 100 ml of 0.1 M H<sub>2</sub>SO<sub>4</sub> to remove tannin. The resulting was filtered and washed with water several times till it was free of SO<sub>4</sub><sup>-2</sup> and dried at 80C<sup>0</sup> in oven until constant weight was attained for (6h)<sup>(20-22)</sup>. The green tea waste mainly consists of carbon 48.60 % wt/dm, hydrogen 5.50 % wt/dm, and nitrogen 0.50 % wt/dm<sup>(23)</sup>.

### Preparation stock solution of Ni(II) (100 mg/L):

A standard solution of 100 mg/L of nickel ion was prepared by dissolving 0.0495 g of Ni(NO<sub>3</sub>)<sub>2</sub>.6H<sub>2</sub>O in a minimum amount of distilled water in a 100 ml volumetric flask and the volume was made up to the mark with distilled water. Solutions of different concentrations were prepared by serial dilutions for nickel ion between (100-10 mg/L). The pH of the solution was maintained by adding dilute NaOH or HNO<sub>3</sub> solution

### Adsorption studies:

Adsorption experiments were performed in a shaker water bath at 180 rpm using 100 ml Erlenmeyer flasks containing 25 ml of different nickel concentrations. After one hour of contact (according to the preliminary sorption dynamics tests), with 0.1 g of green tea waste (GTW), equilibrium was reached and the reaction mixture was centrifuged for 5 min. The metal content in the supernatant was determined using Atomic Absorption Spectrophotometer (AAS) at wavelength 232.0 nm after filtering the adsorbent with Whitman filter paper. From the metal concentrations measured before and after the adsorption (C<sub>i</sub> and C<sub>e</sub>, mg/L, respectively) and weight of (GTW) (m, g), as well as volume of aqueous solution (V, L), the percentage of adsorption (%Adsorption) and the equilibrium adsorption capacity of metal ion (q<sub>e</sub>, mg/g) were calculated according to equations (1) and (2):

$$\% \text{ Adsorption} = \frac{C_i - C_e}{C_i} \times 100 \quad (1)$$

$$q_e = \frac{(C_i - C_e)V}{m} \quad (2)$$

## Results and Discussion:

### Effect of nickel ion concentration:

Fig. 1 and Table 1 show the effect of nickel ion concentration on the adsorption of nickel by (GTW). The data shows that the metal uptake increases and the percentage adsorption of nickel decreases with increase in metal ion concentration. This increase (2.3 to 18.78 mg/g) is a result of increase in the driving forces i.e. concentration gradient. However, the percentage adsorption

of nickel ions on (GTW) was decreased from 92 to 75.12%. Though an increase in metal uptake was observed, the decrease in percentage adsorption may be attributed to lack of sufficient surface area to accommodate much more metal available in the solution. The percentage adsorption at higher concentration levels shows a decreasing trend whereas the equilibrium uptake of nickel displays an opposite trend. At lower concentrations, all nickel ions present in solution could interact with the adsorbed and thus the percentage adsorption was higher than those at higher nickel ion concentrations. At higher concentrations, lower adsorption yield is due to the saturation of adsorption sites.

#### ***Effect of the adsorbent dose:***

Batch experiments were carried out by varying the adsorbent dose between (4-1) g/L at the best initial concentration (10 mg/L). The percentage of adsorption increases from 66.3 to 92% and the adsorption capacity decreases from 6.63 to 2.3 mg/g, are illustrated in the Table 2. The increase in the percentage of adsorption is due to the increase in amount of free surface available<sup>(24)</sup>.

#### ***The effect of contact time:***

Adsorption of Ni(II) on (GTW) as a function of time at the best initial concentration 10 mg/L was studied, where shaken it to different times between (1-200) min. The data obtained from the adsorption of nickel ions on (GTW) showed that a contact time of 60 min. was sufficient to achieve equilibrium and the adsorption did not change significantly with further increase in contact time. Therefore, the uptake and unadsorbed nickel concentrations at the end of 60 min. are given as the equilibrium values ( $q_e$ , mg/g;  $C_e$ , mg/L), respectively (Fig. 2).

#### ***Effect of pH:***

Adsorption experiments were performed over a range of pH (1-10) for the best initial Ni(II) concentration 10 mg/L and shaken time to 60 min. From Fig. 3 it is observed that the percentage of adsorption increase from pH (3-8). At low pH, i.e., higher hydrogen ion concentration, it occur to decrease the percentage of adsorption. Above pH 8 the percentage of adsorption decrease, due to the effect of the hydroxyl ion, ( $\text{Ni}(\text{OH})^+$ ) may be formed<sup>(25)</sup>.

#### ***Effect of temperature:***

Experiments were performed at different temperatures, from 288 to 328 K<sup>0</sup> for the best initial Ni(II) concentration (10 mg/L) at pH equal to 6 and shaken time to 60 min. The adsorption capacity of Ni(II) increased from 2.2875 to 2.3322 mg/g when the temperature increased from 288 to 328 K<sup>0</sup>, are shown in the Table 3 and Fig. 4. The increase in the adsorption capacity showed the endothermic nature of the process.

The change in enthalpy ( $\Delta H$ ) and entropy ( $\Delta S$ ) were calculated from the plot of  $\log (q_e/C_e)$  versus  $1/T$  for Ni(II) by using the Van't Hoff equation<sup>(26)</sup>, is shown in Fig. 5.

$$\log \frac{q_e}{C_e} = \frac{\Delta S}{2.303R} - \frac{\Delta H}{2.303RT} \quad (3)$$

$$\text{Slope} = -\left( \frac{\Delta H}{2.303R} \right) \quad (4)$$

$$\text{Intercept} = \left( \frac{\Delta S}{2.303R} \right) \quad (5)$$

The adsorption enthalpy of the Ni(II) calculated from slope of the plot is 5.0282 KJ/mol and entropy ( $\Delta S$ ) was also calculated from intercept of the plot is 25.555 J/mol.K<sup>0</sup>. These values of

the heat of adsorption show that the type of adsorption onto (GTW) is physical adsorption, because the adsorption with the heat that is lower than 20 KJ/mol<sup>(27)</sup>. The change in free energy ( $\Delta G$ ) was calculated from Gibbs equation<sup>(28)</sup>:

$$\Delta G = \Delta H - T\Delta S \quad (6)$$

The increase in the adsorption capacity of (GTW) with temperature indicates an endothermic process. The positive value of ( $\Delta S$ ) suggested some structural changes in adsorbent and adsorbate and also reflects the affinity of the adsorbent material under consideration towards Ni(II) ions. The negative values of free energy ( $\Delta G$ ) show the spontaneous adsorption of metal ion on the adsorbent, is illustrated in the Table 4. The value of ( $\Delta G$ ) decreases with increasing temperature, demonstrating an increase in the feasibility of adsorption at higher temperatures.

#### **Adsorption isotherm:**

The equilibrium adsorption of nickel on (GTW) as a function of the initial concentration of nickel is shown in Fig. 6. There was a gradual increase of adsorption for nickel ions until equilibrium was attained. The Langmuir, Freundlich and Temkin models are often used to describe equilibrium adsorption isotherms.

The Langmuir<sup>(29)</sup> adsorption model was chosen for the estimation of maximum nickel sorption by the adsorbent. The Langmuir isotherm can be expressed as,

$$\frac{C_e}{q_e} = \frac{1}{q_m b} + \frac{C_e}{q_m} \quad (7)$$

where  $q_e$  is the amount of adsorbate retained per unit weight of the adsorbent (mg/g) at equilibrium,  $C_e$  the equilibrium concentration of Ni(II) (mg/L),  $q_m$  Indicates the monolayer adsorption capacity of adsorbent (mg/g) and the Langmuir constant  $b$  (L/mg) is related to the energy of adsorption. The Langmuir constants ( $q_m$  and  $b$ ) were calculated from the plots of  $C_e/q_e$  versus  $C_e$ , using linear least-squares fitting where the slope equal to  $(1/q_m)$  and the intercept equal to  $(1/q_m b)$ . The values of the constants of this model with correlation coefficients ( $R^2$ ) are given in Fig. 7 and Table 5.

The Freundlich<sup>(30)</sup> model is represented by the equation,

$$\ln q_e = \ln K_f + \frac{1}{n} \ln C_e \quad (8)$$

where  $K_f$  (mg/g) is the Freundlich constant related to adsorption capacity of adsorbent and  $n$  is the Freundlich exponent related to adsorption intensity (dimensionless). The Freundlich constants ( $K_f$  and  $n$ ) were calculated from the plots of  $\ln q_e$  versus  $\ln C_e$ , using linear least-squares fitting where the slope equal to  $(1/n)$  and the intercept equal to  $(\ln K_f)$ . The values of the constants of this model with correlation coefficients ( $R^2$ ) are given in Fig. 8 and Table 5.

The Temkin<sup>(31)</sup> isotherm has generally been applied in the following form,

$$q_e = \frac{RT}{b_T} \ln K_T + \frac{RT}{b_T} \ln C_e \quad (9)$$

where  $K_T$  (L/mg) is Tempkin adsorption potential and  $b_T$  (kJ/mol) is heat of sorption. The Tempkin constants ( $K_T$  and  $b_T$ ) were calculated from the plots of  $q_e$  versus  $\ln C_e$ , using linear least-squares fitting where the slope equal to  $(RT/b_T)$  and the intercept equal to  $([RT/b_T] \ln K_T)$ . The values of the constants of this model with correlation coefficients ( $R^2$ ) are given in Fig. 9 and Table 5.

#### **Adsorption kinetic studies:**

The prediction of adsorption rate gives important information for designing batch adsorption systems. The kinetics of the adsorption data was analyzed using two kinetic models, pseudo-first order and pseudo-second order kinetic model for Ni(II) adsorption <sup>(20)</sup>. The pseudo-first-order rate equation is expressed as:

$$\log(q_e - q_t) = \log q_e - \frac{k_1 t}{2.303} \quad (10)$$

where  $q_t$  and  $q_e$  are the adsorption capacity at time,  $t$ , and at equilibrium, respectively, and  $k_1$  is the rate constant of pseudo-first-order ( $\text{min}^{-1}$ ). The values of  $k_1$  at different adsorption parameters were calculated from the plot of  $\log(q_e - q_t)$  as a function of the time for the best initial Ni(II) concentration (10 mg/L) at pH equal to 6 and temperature equal to 328 K<sup>0</sup> should give a linear relationship where the slope equal to  $(-k_1/2.303)$  and the intercept equal to  $(\log q_e)$ , is given by Fig. 10 and Table 6.

The pseudo-second-order rate equation is expressed as:

$$\frac{t}{q_t} = \frac{1}{(q_e)^2 k_2} + \frac{t}{q_e} \quad (11)$$

where  $k_2$  is the rate constant of pseudo-second-order (g/mg.min),  $q_t$  and  $q_e$  are the adsorption capacity at time  $t$  and at equilibrium, respectively. The values of  $k_2$  were calculated from the plot of  $(t/q_t)$  versus  $(t)$  for the best initial Ni(II) concentration (10 mg/L) at pH equal to 6 and temperature equal to 328 K<sup>0</sup>, should give a linear relationship where the slope equal to  $(1/q_e)$  and the intercept equal to  $[1/k_2.(q_e)^2]$ , is given by Fig. 11 and Table 6. It was noticed that the correlation coefficient value for the pseudo-second-order were found to be higher than that of the correlation coefficient value for the pseudo-first-order. The theoretical  $q_e$  value was closer to the experimental  $q_e$  value. In the view of these results, it can be said that the pseudo-second-order kinetic model provided a good correlation for the adsorption of Ni(II) on GTW in contrast to the pseudo-first-order model.

## Conclusion:

The green tea waste (GTW) was found to be a good adsorbent for the removal of Ni(II) ion from aqueous solutions. The Ni(II) ion removal efficiency of the adsorbent is found to be increase with the increase in time, temperature and adsorbent dose; however the removal of nickel was decrease with increase adsorbate concentration. The increasing percentage adsorption with increasing temperature, the adsorption mechanism is endothermic. The experimental values were well fitted to the Langmuir, Freundlich and Tempkin isotherm equations. The adsorption kinetic followed a pseudo-second-order rate equation where  $k_2$  is equal to 0.0571 g/mg.min.

**Table 1** The effect of initial Ni(II) concentration on the percentage of adsorption and the adsorption capacity

$C_i$ , mg/L	$C_e$ , mg/L	% Adsorption	$q_e$ , mg/g
10	0.8	92	2.3
20	1.64	91.8	4.59
30	2.51	91.6	6.8725
40	3.84	90.4	9.0375
50	5.12	89.76	11.22
60	7.47	87.55	13.1325
70	9.36	85.63	15.16
80	14.53	81.83	16.36
90	19.17	78.7	17.7
100	24.88	75.12	18.78

**Table 2** The effect of the adsorbent dose on the percentage of adsorption and the adsorption capacity for the best initial concentration (10 mg/L)

adsorbent dose, g/L	$C_e$ , mg/L	% Adsorption	$q_e$ , mg/g
4	0.8	92	2.3
2	1.64	83.2	4.16
1	3.37	66.3	6.63

**Table (3).** The effect of temperature on the adsorption capacity

K	1/ K	$C_e$ , mg/L	% Adsorption	$q_e$ , mg/g	$\log q_e/ C_e$
288	0.00347	0.85	91.5	2.2875	0.4299
298	0.00335	0.812	91.88	2.297	0.4516
308	0.00324	0.774	92.26	2.3065	0.4742
318	0.00314	0.714	92.86	2.3215	0.512
328	0.00304	0.671	93.29	2.3322	0.541

**Table 4.** The thermodynamic parameters for removal of Ni(II) on (GTW)

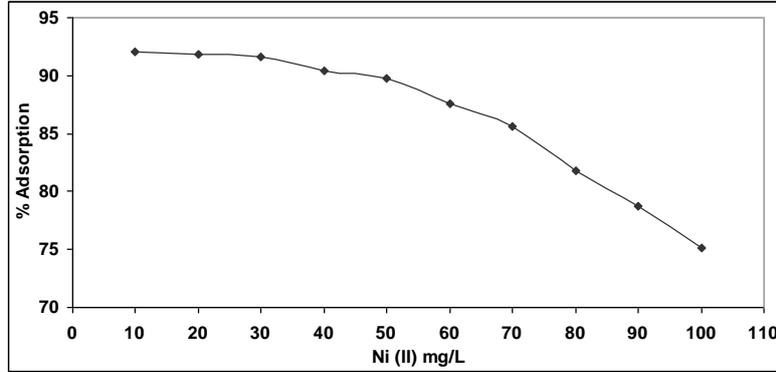
K	1/ K	$\Delta H$ , KJ/ mol	$\Delta S$ , J/mol.K	$\Delta G$ , KJ/ mol
288	0.00347	5.0282	25.555	-2.3318
298	0.00335			-2.5872
308	0.00324			-2.8427
318	0.00314			-3.0983
328	0.00304			-3.3538

**Table 5.** Langmuir, Freundlich and Temkin isotherm constants

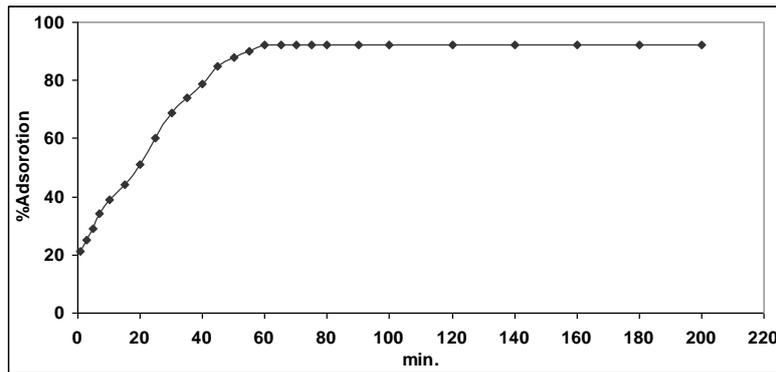
Langmuir constants			Freundlich constants			Tempkin constants		
$q_m$ , mg/g	$b$	$R^2$	$K_f$ , mg/g	$n$	$R^2$	$K_T$ , L/mg	$b_T$ , kJ/mol	$R^2$
23.8095	0.1566	0.9945	3.2181	1.6007	0.9716	1.7015	485.6	0.9905

**Table 6 Pseudo-first-order and Pseudo-second-order kinetic constants for adsorption Ni(II) on GTW**

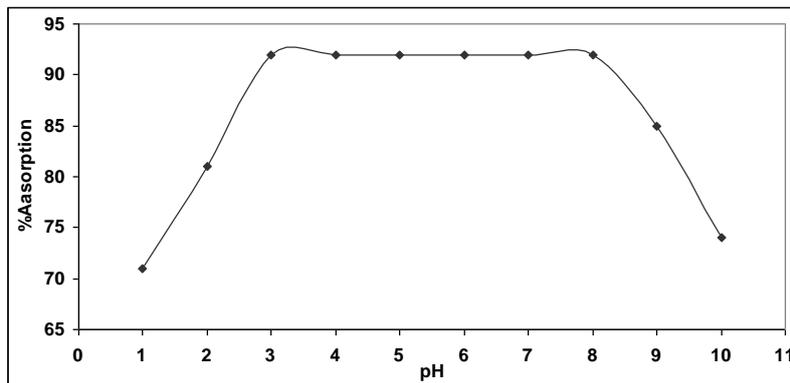
Pseudo-first-order				Pseudo-second-order			
$q_{e, exp.}$ mg/g	$q_{e, cal.}$ mg/g	$k_1$ ( $min^{-1}$ )	$R^2$	$q_{e, exp.}$ mg/g	$q_{e, cal.}$ mg/g	$k_2$ (g/mg.min)	$R^2$
2.3	2.5176	0.0594	0.9267	2.3	2.4248	0.0571	0.9979



**Fig. 1 The effect of initial Ni(II) concentration on the percentage of adsorption**



**Fig. 2 The effect of contact time on the percentage of adsorption**



**Fig. 3 The effect of pH on the percentage of adsorption**

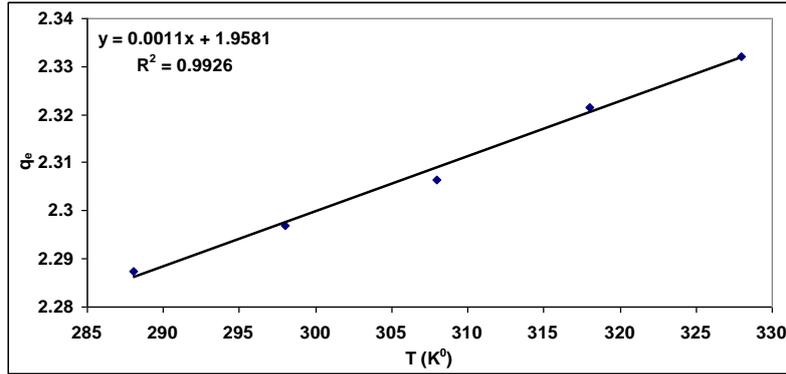


Fig. 4 The effect of temperature on the adsorption capacity

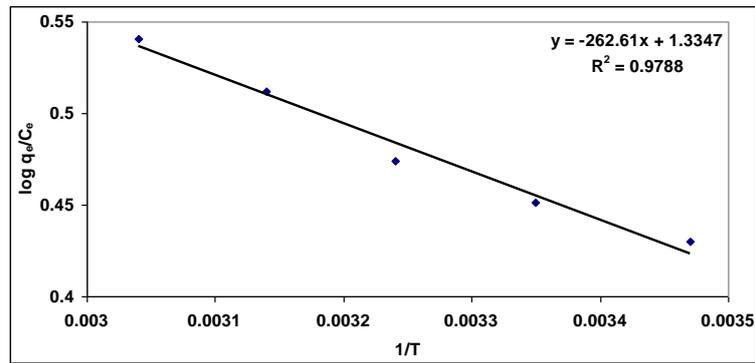


Fig. 5 The plot of  $\log (q_e/C_e)$  versus  $1/T$

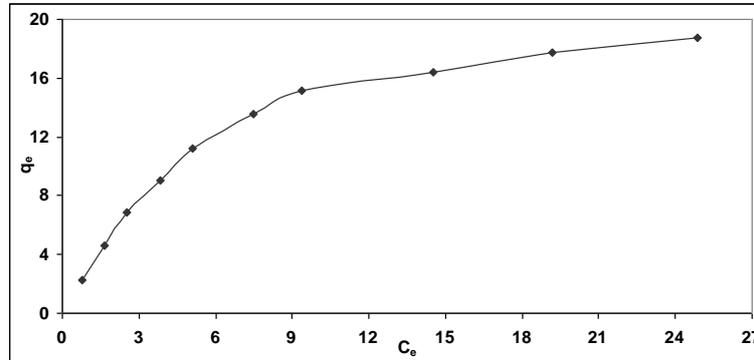


Fig. 6 The adsorption isotherm of Ni(II) on GTW

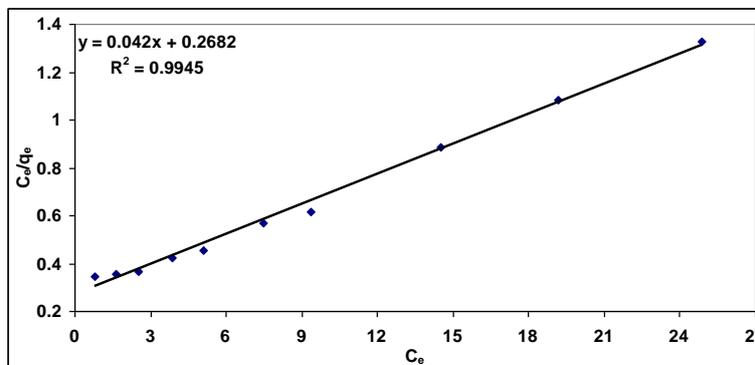


Fig. 7 Linear form of Langmuir equation for adsorption Ni(II) on GTW

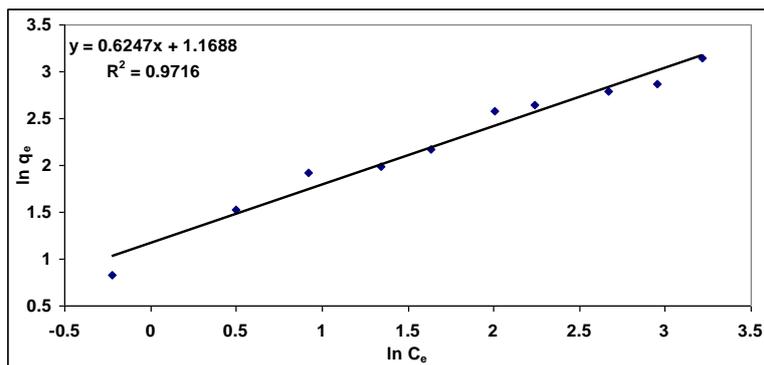


Fig. 8 Linear form of Freundlich equation for adsorption Ni(II) on GTW

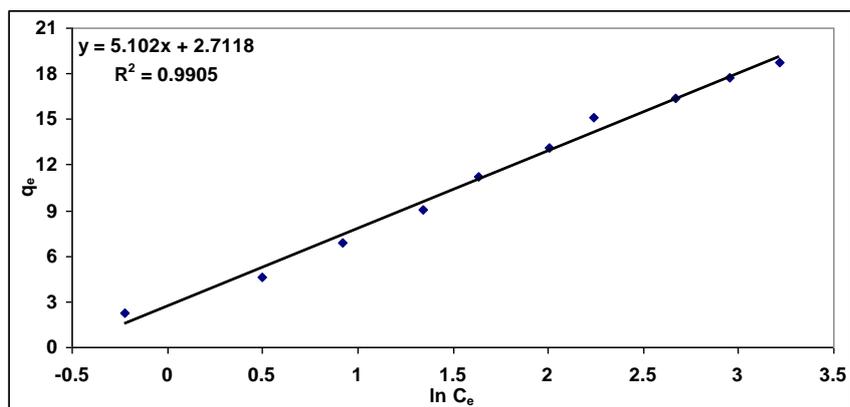


Fig. 9 Linear form of Tempkin equation for adsorption Ni(II) on GTW

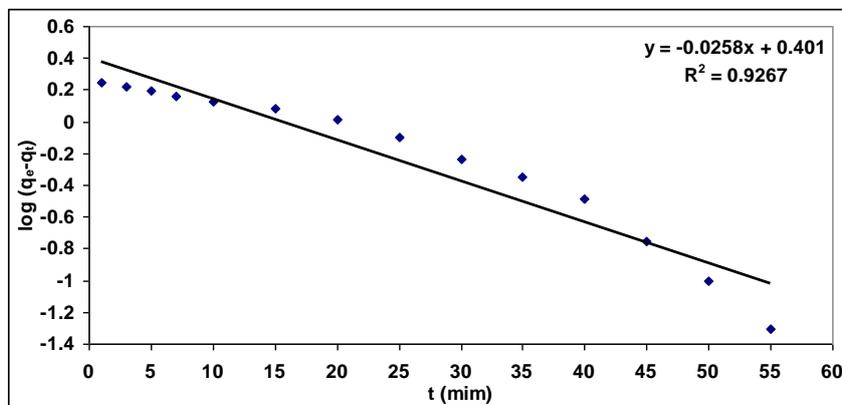


Fig. 10 Pseudo-first-order kinetic for adsorption Ni(II) on GTW

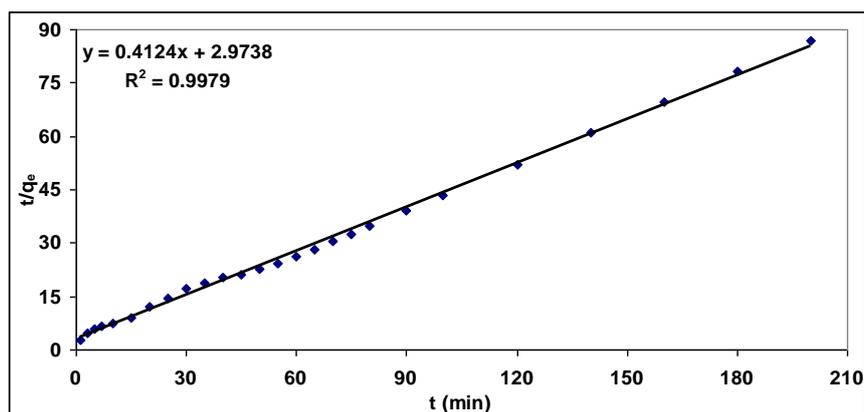


Fig. 11 Pseudo-second-order kinetic for adsorption Ni(II) on GTW

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### امتزاز ايونات النيكل من محاليله المائية بواسطة فضلات الشاي الأخضر

مهند حازم ناجي حلبوص\* ، اعياد عمار صيهود\*\* ، حيدر مصطفى كمال\*  
\* قسم الكيمياء \ كلية العلوم \ جامعة الكوفة  
\*\* فرع العلوم الأساسية \ كلية طب الأسنان \ جامعة الكوفة

#### الخلاصة:

في هذا البحث، تم استعمال فضلات الشاي الأخضر لإزالة أيونات النيكل الثنائي من محاليله المائية. ان تجارب الامتزاز أجريت تحت الظروف المختلفة حيث استعملت طريقة الدفعات لتقدير أفضل نسبة امتزاز وسعة امتزاز للتركيز المستعملة لأيون النيكل الثنائي والتي تراوحت ما بين 10-100 ملي غرام لتر، أظهرت النتائج ان أفضل تركيز كان 10 ملي غرام لتر حيث كانت نسبة الامتزاز 92% وسعة الامتزاز 2.3 غرام ملي غرام وجرعة الامتزاز كانت 10 غرام لتر ، ووجد أن زمن الرج مساوي إلى 60 دقيقة وأفضل قيمة pH كانت ما بين (3-8). كما تمت دراسة تأثير درجة الحرارة والدوال الترموديناميكية وقد لوحظ إن سعة الامتزاز تزداد بزيادة درجة الحرارة والتفاعل من النوع الماص للحرارة . تم تحليل بيانات أيزوثيرم الاتزان باستعمال معادلات لانكمير، فراندش وتيمبكين. ووجد ان ثابت السرعة  $k_2$  لامتزاز أيون النيكل الثنائي يساوي 0.0571 غرام ملي غرام دقيقة .