Purification of Aqueous Solution from Ni (II) Ions Using Commercial and Bitter Orange Leaves Activated Charcoal

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Abstract

The purification of diluted aqueous solution from toxic Ni (II) ion was investigated using commercial and bitter orange leaves activated charcoal as adsorbents through batch method. The contact times between the adsorbents and adsorbate at 298 K and pH of 5 were studied. The percentage metal removal approached equilibrium within 75 min on Commercial and 90 min on prepared charcoal surfaces. The experimental data of the metal removal equilibrium were correlated by either the Langmuir or Freundlich equations. Results indicate that the Langmuir model gave a better fit to the experimental data than the Freundlich equation. Kinetic processes of Ni²⁺ adsorption on Commercial and prepared charcoal were described by applying pseudo-first-order and pseudo-second-order rate equations.

Keywords: Activated charcoal (AC); Nickel ion; Langmuir isotherm; Freundlich isotherm.

Introduction

Heavy metals are well identified contaminants for public health, plants and animals because of its toxicity [1]. Toxic heavy metals a part from being hazardous for living organism, when exceed the specific limits, have accumulation characteristic in nature as they cannot be biodegraded, causing health problems in animals, plants and human beings [2,3]. Inorganic effluent from the industries contains toxic metals such as Ni. Cu, Zn and Cd...etc., which tend to accumulate in the food chain [4]. Because of their high solubility in the aquatic environments, heavy metal can be adsorbed by living organism. Once they enter the food chain large concentrations of heavy metals may accumulate in the human body [5]. The increase in usage of heavy metals in industrial activities has caused the existence of them in wastewater [6]. As for most metals, the toxicity of nickel is dependent on the route of exposure and the solubility of the nickel compound [7]. Human nickel exposure originates from a variety of sources and is highly variable. It is normally present in human tissues and, under conditions of high exposure, these levels may increase significantly [8]. Human may be exposed to nickel by breathing air, drinking water, eating food or smoking cigarettes. Skin contact with nickel contaminated soil or water may also result in nickel exposure. In small quantities nickel is essential, but when the uptake is too high it can be a danger to human health [9]. The excessive exposure to nickel can lead to severe damage of lung, kidneys, skin dermatitis, and cancer [10]. In recent years, increasing awareness of the environmental impact of heavy metals has prompted a demand for the purification of industrial wastewater prior to discharge into natural waters. This has led to the introduction of more strict legislation to control water pollution [11]. Activated carbon adsorption has been a promising technique for the removal of heavy metal ions from water [12]. For examples, Kadirvelu K., et al [13] studied the adsorption of Cd^{2+} from aqueous solution by activated carbon prepared from coconut coirpith, Moreno J.C., et al [14] studied removal of Mn²⁺, Fe²⁺, Ni²⁺ and Cu²⁺ ions from wastewater using cow bone charcoal.

The objective of this study was to evaluate the feasibility of using the commercial and bitter orange leaves activated charcoal for the removal of nickel from aqueous solution. The adsorption process is studied from kinetic and isotherm standpoints.

Materials and Methods Adsorbent

Commercial and prepared activated charcoal were used as adsorbents in this study. The commercial activated charcoal (CAC) was supplied from the BDH, ANALAR, England. Prepared activated charcoal (PAC) was prepared from leaves of bitter orange tree (Citrus aurantium) [15]. The CAC and PAC were sieved using sieve nominal size 75 μ m (no.200). The adsorbents were characterized by X-ray diffraction (XRD) using instrument type BRUKER, D8 ADVANCE using Cu K α radiation of 1.54°A.

Adsorbate

A standard stock solution of Ni(II) ion (1000 mg/L) was prepared from Ni(NO₃)₂.6H₂O (supplied by BDH chemical) in deionized water. Several concentrations (25, 50, 70, 85, and 100 mg/L) were prepared from this standard stock. Absorbance values of these solutions were measured at the specified wave length of 232 nm.

Adsorption studies

(25ml) of Ni (II) solution of a known concentration ranged from (25 mg/L) to (100 mg/L)were added separately to volumetric flasks containing (0.5g) of CAC or (0.2g) of PAC, at temperature of $298K\pm 2$ and pH of 5 [15], the flasks were shaken in thermostatically controlled water bath shaker at a constant speed (180 cycles per min) for the required equilibrium time. The mixtures were then separated by centrifugation at (3000 rpm) for (15min) and then filtered using a whatman No.42 filter paper.

The metal equilibrium concentrations were measured using atomic absorption spectrometer (AAS) by comparing the experimental absorbance with the calibration curve.

The adsorption capacity of adsorbent was calculated using the equation below [10]:

 $Q_e = \frac{(C_o - C_e).V}{m} \qquad (1)$ Where:

- Q_e : Adsorption capacity of the adsorbent at equilibrium time t_e (mg/g).
- C_o: Initial concentration of adsorbate (mg/L).

- Ce: Equilibrium concentration of adsorbate after adsorption has occurred (mg/L).
- V: Volume of solution (L).
- m: Weight of adsorbent (g).

Results and Discussion

X-ray diffraction (XRD) analysis of the adsorbents.

The crystalline structure of the CAC and PAC were characterized using X-ray diffractometer, and the results are shown in Fig.(1) and Fig.(2) respectively. In Fig.(1) the angles 2Θ at 26.562 (003), 42.490 (101), and 45.672 (012) are the characteristic of CAC crystal and in Fig.(2) the angles 2Θ at 26.716 (003), 43.013 (101), and 46.421 (012) are those of PAC. The diffraction data were in good agreement with the standard spectrum.



Fig. (1) X-ray diffraction analysis of commercial activated charcoal.



Fig.(2) X-ray diffraction analysis of bitter orange leaves activated charcoal.

Contact time of adsorption

The contact times between the CAC and PAC with Ni(II) ion solution that is sufficient for the adsorption process to reach equilibrium using a fixed concentration ($C_0 = 85 \text{ mg/L}$) and pH of 5 at 298 K were studied at different periods (10, 30, 45, 60, 75, 90, 120 and 150) min.

The metal removal percentage was calculated using equation (2)[16].

Where:

R% : The metal removal percentage.

- C_o : The initial concentration of metal (mg/L).
- C_t : The residual concentration of metal after adsorption had taken place over a period of time **t** expressed as (mg/L).

The result of the effect of contact time on the adsorption of Ni(II) from aqueous solution is shown in Fig.(3). It was observed that the percentage metal removal increased rapidly with increase in contact time. The adsorption of Ni(II) ion by CAC and PAC was rapid for the first 10 min of contact time. The fast adsorption at the initial stage due to the initial concentration gradient between the adsorbate in solution and the number of vacant sites available on the adsorbent surfaces at the The progressive beginning. increase in adsorption and consequently the attainment of equilibrium adsorption may be due to limited mass transfer of the adsorbate molecules from the bulk liquid to the surface of adsorbent [16-18]. The time required for (Ni (II)-CAC) and (Ni (II)-PAC) systems to attain equilibrium to be 75, and 90 min respectively.



Fig.(3) Effect of contact time on adsorption of Ni (II) ion (C_o =85mg/L)on CAC and PAC Surfaces.

Kinetic study of adsorption (Ni²⁺) by CAC and PAC surfaces.

Kinetic studies were carried out with (0.5g) of commercial activated carbon and (0.2g) of prepared activated carbon at 298 K and pH of 5 from 10 to 150 min. The adsorption increased rapidly and reach equilibrium within 75 min for CAC and 90 min for PAC. The order of adsorbate – adsorbent interaction has been described using various kinetic models. Table (1) show the kinetic data for adsorption of Ni(II) ion on CAC and PAC surfaces which is being used in pseudo-first and pseudo-second order model.

Pseudo-first order rate expression, popularly known as the lagergren equation, is generally described by the following equation[14].

- $\ln(Q_e Q_t) = \ln Q_e K_1 t$ (3) Where:
- Q_e is the amount of the metal ions adsorbed at equilibrium per unit weight of sorbent (mg.g⁻¹).
- Q_t is the amount of the metal ions adsorbed at any time (mg.g⁻¹).
- K_1 is the rate constant (min⁻¹).

The slope and intercept of plots of $ln(Q_e-Q_t)$ versus t in Fig. (4) were used to determine the first order rate constant K_1 and equilibrium adsorption capacity Q_e . Pseudo first order model is used to described the reversibility of the equilibrium between liquid and solid phase [10].

The adsorption data was also analyzed in term of the pseudo-second order given by [5]:

$$\frac{t}{Q_t} = \frac{1}{K_2 Q_e^2} + \frac{1}{Q_e} t \dots (4)$$
Where:

K₂ is the equilibrium rate constant of pseudosecond order adsorption (g/mg. min.)

The plot of $\frac{t}{q_t}$ against t gives a linear relationship, and K₂ and Q_e can be calculated from the slope and intercept of the line in Fig. (5).

Results in Table (2) shows the kinetic rate constants, correlation coefficient R^2 , and calculated equilibrium sorption capacity (Q_e) of Ni(II) ions on CAC and PAC surfaces. From the results, it can be concluded that pseudo - second- order equation provides the best correlation coefficient R^2 and agreement between the calculated equilibrium sorption capacity (Q_e) and experimental (Q_e) values. The results suggest that the pseudo - second order sorption mechanism is predominant [5,10,19].

Table (1)Kinetic data for adsorption of Ni(II) ion on
CAC and PAC surfaces.

	CAC Qe = 2.8127 (mg/g)			
Time				
min	Q_t	ln(Q _e -	t/Q_t	
	mg/g	Q_t	min/mg.g ⁻¹	
10	2.4424	-0.9934	4.0943	
30	2.5801	-1.4584	11.6274	
45	2.6533	-1.8363	16.9600	
60	2.7301	-2.4937	21.9772	
		PAC		
Time	Qe = 7.9457 (mg/g)			
min	Q_t $ln(Q_e$ -		t/Q_t	
	mg/g	Q_t	min/mg.g ⁻¹	
10	6.4292	0.4164	1.5554	
30	6.7523	0.1768	4.4429	
45	6.9363	0.0093	6.4876	
60	7.2228	-0.3244	8.3070	
75	7.4764	-0.7565	10.0315	



Fig.(4) Pseudo - First order kinetic plot for the adsorption of Ni (II) ion from aqueous solution on CAC and PAC surfaces.



Fig. (5) Pseudo - Second order kinetic plot for the adsorption of Ni (II) ion from aqueous solution on CAC and PAC surfaces.

Table (2)The calculated parameters of the Pseudo-First order & Pseudo-Second order kineticmodels of (Ni 2+) ions from CAC & PAC

surfaces.					
	Pseudo - First order				
AC	K 1	Q_e	P ²		
	min ⁻¹	mg/g	Λ		
CAC	0.0293	0.5307	0.9741		
PAC	0.0176	1.9724	0.9531		
	Pseudo - Second order				
AC	K 2	Q_e	P ²		
	g/mg.min	mg/g	Λ		
CAC	0.1853	2.7925	0.9992		
PAC	0.0373	7.6628	0.9974		

The adsorption isotherm

Adsorption of Ni (II) ion from an aqueous solution on CAC and PAC was studied at four temperatures (298, 313, 333, and 353) K and pH of 5 as show in Table (3).

Adsorption isotherms are mathematical models that describe the distribution of the adsorbate species among liquid and adsorbent, based on a set of assumption that are mainly related to the heterogeneity/homogeneity of adsorbent, the type of coverage and possibility of interaction between the adsorbate species. Adsorption data are usually described by adsorption isotherms, such as Langmuir and Freundlich isotherms. These isotherm relate metal uptake per unit mass of adsorbent Q_e to the equilibrium adsorbate concentration in the bulk fluid face C_e [20].

The quantities adsorbed Q_e were plotted versus equilibrium concentration C_e to obtain

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the general case of the adsorption isotherms and shown in Fig.(6) and (7) for the prepared and commercial activated carbon at different temperatures.

 Table (3)

 Data of Ni(II) ion uptake by the CAC & PAC surfaces at different temperatures and pH of 5.

AC	Co mg/L	298 K		313 K		333 K		353 K	
		Ce mg/L	Qe mg/g	Ce mg/L	Qe mg/g	C _e mg/L	Qe mg/g	C _e mg/L	Qemg/g
	25	3.5471	1.0726	5.5283	0.9735	7.3396	0.8830	8.8773	0.8061
5	50	10.9245	1.9537	16.5094	1.6745	19.2264	1.5386	21.9150	1.4042
CA (70	18.9339	2.5533	27.9811	2.1009	30.7735	1.9613	35.0188	1.7490
	85	28.7452	2.8127	38.0943	2.3452	42.0094	2.1495	45.9433	1.9528
	100	38.0377	3.0981	50.3396	2.4830	54.1981	2.2900	60.3867	1.9806
PAC	25	1.2264	2.9717	2.0283	2.8714	3.0471	2.7441	4.5754	2.5530
	50	5.4150	5.5731	6.9528	5.3809	9.7924	5.0259	12.8018	4.6497
	70	12.6037	7.1745	16.6886	6.6639	19.4811	6.3148	20.9716	6.1285
	85	21.4339	7.9457	25.2924	7.4634	29.3018	6.9622	32.7264	6.5342
	100	29.2075	8.8490	33.3396	8.3325	39.5377	7.5577	42.7924	7.1509



Fig. (6) Adsorption isotherm of Ni (II) ion on CAC surface at different temperatures.



Fig. (7) Adsorption isotherm of Ni (II) ion on PAC surface at different temperatures.

The Langmuir isotherm

Langmuir isotherm describes monolayer adsorption based on assumption that all the adsorption sites have equal adsorbate affinity (the surface is homogeneous) and that adsorption at one site does not affect adsorption at an adjacent site [21,22].

In the case of adsorption from the solution, Langmuir isotherms could be fitted by the equation [11,23]:-

- $Q_e = \frac{abC_e}{1+bC_e} \dots (5)$ Where:-
- Q_e : The quantity adsorbed at equilibrium in (mg/g) .
- C_e : The equilibrium concentration of adsorbate in (mg /L).
- a and b: **a** is a Langmuir constant which is a measure of monolayer adsorption capacity in (mg/g) and **b** is also Langmuir constant in (L/mg)

Also, we can write the linear form of this equation as :-

$$\frac{C_e}{Q_e} = \frac{1}{ab} + \frac{C_e}{a} \qquad (6)$$

This form can be used as a linearization of experimental data by plotting C_e/Q_e against C_e as shown in Fig.(8) and (9). The Langmuir's constants (a) and (b) can be evaluated from the slope (1/a) and intercept (1/ab) of the linear equation [11, 23], and the results are shown in Table (4).



Fig. (8) Linear form of Langmuir isotherm of Ni (II) ion on CAC surface at different temperatures.



Fig. (9) Linear form of Langmuir isotherm of Ni (II) ion on PAC surface at different temperatures.

The Freundlich isotherm

Freundlich has proposed an empirical equation of adsorption in solution. Freundlich adsorption isotherm equation may be derived assuming a heterogeneous surface and is based on the idea that the adsorption depending on the energy of the adsorption sites [24,25]. This equation is as follows [20,26]:-

- Q_e : The quantity adsorbed at equilibrium in (mg/g).
- C_e : The equilibrium concentration of the adsorbate in (mg/L).
- K_f and n: The Freundlich experimental constants, K_f and n are indicators of the adsorption capacity and adsorption intensity, respectively.

Taking logarithms of equation (7) we get: -

If log Q_e is plotted against log C_e a straight line should be obtained as shown in Fig. (10) and (11). The slope of the line will give the value of 1/n and the intercept on the Y-axis gives the value of log K_f [20,26] and the results are shown in Table (5).

The Freundlich isotherm is widely used but provides no information on the monolayer adsorption capacity in contrast to the Langmuir model [20].



Fig. (10) Linear form of Freundlich isotherm of Ni (II) ion on CAC surface at different temperatures.



Fig. (11) Linear form of Freundlich isotherm of Ni (II) ion on PAC surface at different temperatures.

Table (4)

Langmuir empirical constants values and the correlation coefficients for the adsorption of (Ni²⁺) on CAC & PAC surfaces at different temperatures.

ed al		Langmuir model			
Activat charco (AC)	T K	a (mg/g)	b (L/mg)	R^2	
CAC	298	3.8505	0.1014	0.9973	
	313	3.1250	0.0759	0.9978	
	333	3.0845	0.0541	0.9988	
	353	2.7048	0.0499	0.9930	
PAC	298	9.6618	0.2718	0.9938	
	313	9.3370	0.1872	0.9921	
	333	9.0334	0.1226	0.9986	
	353	9.0252	0.0872	0.9959	

Table (5)

Freundlich empirical constants values and the correlation coefficients for the adsorption of (Ni²⁺) on CAC & PAC surfaces at different temperatures.

ed al		Freundlich model			
Activat charco (AC)	T K	n	K_{f}	R^2	
	298	2.2187	0.6331	0.9831	
CAC	313	2.3009	0.4769	0.9889	
	333	2.0496	0.3476	0.9817	
	353	2.0437	0.2915	0.9676	
PAC	298	2.9446	2.9093	0.9838	
	313	2.7225	2.3555	0.9740	
	333	2.5361	1.8732	0.9755	
	353	2.1556	1.3418	0.9641	

As presented in Table (4) and (5), The correlation coefficients for the linear Langmuir regression fits are larger than that for the Freundlich plot, so the Langmuir model could describe well the adsorption isotherm for the uptake of Ni(II) ion from aqueous solution on CAC and PAC surfaces thus, indicating to the applicability of monolayer coverage of the Ni²⁺ ion on the surface of adsorbent [27].

Conclusions

The two adsorbent which are used in the study [commercial and prepared activated charcoal from the dry leaves of bitter orange tree (Citrus aurantium)] have a high ability for removing the Ni(II) ion from aqueous solutions. however. prepared activated charcoal has the highest affinity. Equilibrium data obtained for the two adsorbents were fitted to the Langmuir more than Freundlich isotherms. The Langmuir model assumes that the uptake of metal ions occurs on a homogenous surface by monolayer adsorption. Adsorption of Ni²⁺ obeys pseudo-second order equation with good correlation.

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الخلاصة

يتناول البحث تنقية المحلول المائي من ايون النيكل الثنائي باستخدام الفحم المنشط التجاري والفحم المحضر من اوراق النارنج كسطوح امتزاز ودراسة زمن التماس بين المادة الممتزة وسطحي الامتزاز عند درجة حرارة ٢٩٨ كلفن والدالة الحامضية ٥ حيث بينت نتائج الدراسة ان النسبة المئوية لإزالة الفلز تصل الى حالة الاتزان عند ٥٧ دقيقة على سطح الفحم التجاري و ٩٠ دقيقة على سطح الفحم المحضر. حللت ايزوثيرمات الامتزاز وفق نموذجي لاتكماير و فريندلش اذ وجد ان ايزوثيرمات الامتزاز نتبع ايون النيكل الثنائي على سطحي الفحم التجاري والمحضر بتطبيق معادلات المرتبة الاولى والثانية حيث اظهرت النتائج ان عملية الامتزاز تطيع قانون المرتبة الثانية بشكل افضل وبمعامل ارتباط 20900 للفحم التجاري و 70900 للفحم المحضر من اوراق النارنج.

الكلمات المفتاحية: الفحم المنشط، ايون النيكل، لانكمايرايزوثيرم، فريندلش ايزوثيرم.