Equilibrium, Mechanism, and Mass Transfer Studies of Nickel(II) Adsorption by Sewage Sludge-Derived Activated Carbon

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ABSTRACT: *The present study was undertaken to evaluate the equilibrium, mechanism, and mass transfer of nickel(II) ions adsorption on activated carbon derived from sewage sludge. Batch adsorption experiments were performed as a function of initial Ni(II) ions concentration (10-50 mg/L) and contact time (10-120 min). The experimental data were analyzed by different models and Freundlich model showed a better representation of equilibrium data (R^2>0.99) and the mean adsorption energy was found to be E= 3.98 kJ/mol. Mechanism study indicates that both external mass transfer (D_2) and mass transfer diffusion coefficient (k_L) are important in determining the adsorption rates (5.50 × 10^{-5} cm/s and 7.30 × 10^{-7} cm/s). We also found that nickel(II) adsorption onto activated carbon would be attributed to a Physico-chemical adsorption process. The results suggest that sewage sludge-derived activated carbon could be used beneficially as an effective and alternative adsorbent for the removal of nickel(II) ions from aqueous solutions.*

KEYWORDS: *Equilibrium, Mechanism, Mass transfer, Ni(II) adsorption, Sewage sludge, Activated carbon.*

INTRODUCTION

In recent years, water pollution has become a global crisis as a result of the development of modern industry [1, 2]. Heavy metals are a class of toxic pollutants released to surface water and groundwater due to various natural and human activities [3]. Nickel is one of the toxic heavy metals that is widely used in silver refineries, electroplating, zinc base casting, and storage battery industries [4]. Nickel toxicity to humans has caused cancer of the lungs, nose, and bones. For this, it is essential to remove nickel from wastewater before entering the environment [5].

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The maximum permissible limit of nickel in water is 0.2 mg/L [6] and according to OJPDRA (Official Journal of the People's Democratic Republic of Algeria), the permissible limit of nickel in effluent wastewater is 5 mg/L[7]. For the removal of nickel ions, different processes have been employed include chemical precipitation, ion exchange, flotation, membrane filtration, electrochemical treatment, coagulation-flocculation, adsorption, and other processes [8]. Adsorption is accepted as the cheapest, simple, most effective method and adsorption is an efficient process that has a wide application in the removal of pollutants and metal ions from water solutions [9-12].

Various effective adsorbents for large-scale use in water decontamination (removal of various micropollutants) have been developed from industrial and agricultural wastes. Certain adsorbents such as activated carbon, silica gel, activated alumina, zeolites, and ion exchange resin have higher capacity in the removal of toxic heavy metals. So, their utilization is not common and confined to the special treatment due to high installation and operating costs [13]. Adsorption by activated carbon is widely used for the elimination of pollutants in water and has been studied extensively [9]. Activated carbon demonstrated significant adsorption in gas and liquid phases due to its large specific surface area, high micropore volume, favorable pore size distribution, capability for rapid absorption, and thermal stability [14]. However, due to their high production cost and loss during the regeneration, the commercial activated carbon is a very expensive material than other adsorbents, which limits its use as an adsorbent in developing countries [15]. Currently, several reports showed that the activated carbon can be prepared from different carbonaceous materials such as almond husk [5], scrap tire [9], coir pith [16], cashew nutshell [17], coffee ground [18], coffee husks [19], apricot stone [20], corncob [21], rice stems [22] and sewage sludge [8].

Sewage sludge is carbonaceous material produced by wastewater treatment plants [23]. The increasing generation of sewage sludge demands the development of new ways of valorization [24]. Currently, the traditional sludge disposal routes include incineration, land application, and composting [25]. However, the incineration and landfill of sewage sludge are hampered by their poor public image, moreover, incineration is both expensive and gives rise to significant quantities of potentially hazardous ash. The utilization of sewage sludge as a fertilizer has been limited by the imposition of legislation in recent years. Disposal by landfills is further inhibited by its rising cost and the competing pressures for an ever-diminishing supply of suitable landfill sites [26]. Thanks to their high carbon content, sewage sludge can be a precursor for the production of activated carbon [7]. Activated carbon prepared from sewage sludge is more important and very interesting because it gives a reusable byproduct and reduces sewage sludge volume [8].

According to the best of our knowledge, there are not many reports in the literature about the mechanism and mass transfer of nickel(II) adsorption onto activated carbon prepared from sewage sludge. The consequent activated carbon prepared from sewage sludge was used as an adsorbent to remove nickel(II) ions from aqueous solutions. The parameters of equilibrium, mechanism, and mass transfer involved in this process were investigated herein.

EXPERIMENTAL SECTION

Chemicals reagents

Nickel(II) ions solutions were prepared from hexahydrate nickel sulfate ($NiSO_4 \cdot 6H_2O$) obtained from Sigma Aldrich® of analytical reagent grade. The concentration of Ni(II) ions was analyzed using a flame atomic absorption spectrometer with nickel hollow-cathode lamp and air acetylene flame (Perkin Elmer AAnalyst 400) with a precision better than ±2 % at a wavelength of 232.0 nm.

Preparation and characterization of activated carbon

Sewage sludge used in this work was collected from Waste Water Treatment Plant (WWTP) located in Guelma city (Algeria). The details of procedure preparation and Physico-chemical characterization results (iodine number, methylene blue number, specific surface area (S_{BET}), XRD analysis and Fourier Transform InfraRed (FT-IR) spectra have been recently reported and are presented in our previous article [8].

Batch adsorption experiment

The effect of selected operating parameters: initial Ni(II) concentration (10-50 mg/L) and contact time (10 - 120 min) on the adsorptive removal of Ni(II) ions were studied in a batch mode. The equilibrium adsorption

capacity of Ni(II) ions, q_e(mg/g), is calculated from the following equation [27]:

$$q_e = \frac{(C_0 - C_e)}{m} V \quad (1)$$

Where C_0 (mg/L) is the initial Ni(II) ions concentration; C_e(mg/L) is the equilibrium Ni(II) ions concentration; V (L) is the volume of the solution and m (g) is the mass of the adsorbent.

For mechanism and mass transfer studies, the procedure of the experiment was basically identical to those of the equilibrium study. The adsorption capacity of Ni(II) ions at any time t, q_t (mg g^{-1}), was calculated by the following equation [28]:

$$q_t = \frac{(C_0 - C_t)}{m} V \quad (2)$$

Where C_t (mg/L^{-1}) is the concentration of Ni(II) ions solution at time t.

All experiments were repeated three-time and average values were reported. Standard deviations were found to be ± 2 %. Further, the error bars for the figures were so small as to be smaller than the symbols used to plot the graphs.

Equilibrium isotherms study

In order to clarify the phenomenon of adsorption of Ni(II) ions on the prepared activated carbon, an equilibrium isotherms study was carried out. Langmuir, Freundlich, and Temkin models expressions are found in our previous manuscript [8].

The linear and logarithmic form of the Dubinin-Radushkevich isotherm model can be written as the following expression [29]:

$$\ln q_e = \ln Q_{max} - \beta\varepsilon^2 \quad (3)$$

Where β (mol^2/J) is the activity coefficient related to the mean free energy of adsorption and ε (J/mol) is the Polanyi potential that can be written as:

$$\varepsilon = RT \ln\left(1 + \frac{1}{C_e}\right) \quad (4)$$

The Redlich-Peterson isotherm model can be written as following [30]:

$$\frac{q_e}{q_m} = \frac{K_L C_e}{1 + (K_L C_e)^n} \quad (5)$$

RESULTS AND DISCUSSIONS

Equilibrium isotherms study

The different equilibrium adsorption isotherms of Ni(II) ions onto prepared activated carbon are graphically illustrated in Fig. 1. From the figure, we can notice that the adsorption models are consistent with the experimental data in the following order: Freundlich, Dubinin-Radushkevich, Langmuir, Temkin, and Redlich-Peterson. The adsorption parameters were evaluated from the isotherms and the values of correlation coefficient (R^2) for the different isotherm models are listed in Table 1.

Based on the results of our previous study [8] and the results of Table 1, the Freundlich model provides the best correlation of experimental data with a high correlation coefficient (R^2= 0.9938). This result indicates that the adsorption of Ni(II) ions on the prepared activated carbon is multilayer. The maximum monolayer adsorption capacity (Q_{max}) was 11.52 mg/g. The mean adsorption energy calculated from Dubinin-Radushkevich isotherm, 3.98 kJ/mol, indicates that the adsorption process may be carried out *via* physical process.

Mechanism and mass transfer study

For a solid-liquid adsorption process, analyzing the rate-controlling steps such as mass transport and chemical reaction processes is very beneficial for elaborating the adsorption mechanism. The adsorption reaction is usually divided into the following steps [31, 32]:

- Metal ion from the bulk liquid to the liquid film or boundary layer surrounding the adsorbent.
- Transport of solute ions from the boundary film to the external surface of the adsorbent (film diffusion).
- Transfer of ions from the surface to the intra-particular active sites (particle diffusion).
- Adsorption of ions by the active sites of adsorbent.

The uptake of nickel(II) ions can be controlled by either the intra-particle diffusion or the mass transfer through the boundary film of liquid [31].

The intra-particle diffusion model is determined by using the following equation [33]:

Table 1: Different isotherms model parameters at 25 °C.

Models	Parameters		
Dubinin-Radushkevich model	Q_{max} (mg/g)	E (kJ/mol)	R^2
	19.999	3.9800	0.9804
Redlich-Peterson model	K_F (L g^{-1})	Q_{max} (mg/g)	R^2
	11.482	11.520	0.8474

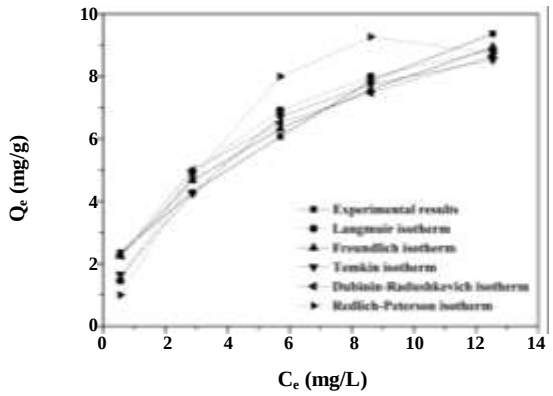

Fig. 1: Adsorption isotherms of nickel(II) adsorption by prepared activated carbon.

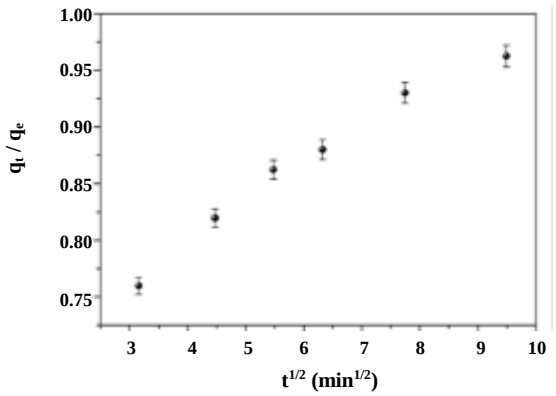

Fig. 2: Plot for determination of film diffusion coefficient (D_1).

$$q_t = k_{id}\, t^{1/2} + C_i \tag{6}$$

Where k_{id} (mg/g.$min^{1/2}$) is the intra-particle diffusion rate constant and C_i is the intercept at stage *i*, which k_{id} and C_i were calculated from the slope and intercept of the linear plot of q_t versus $t^{1/2}$, respectively. The corresponding model parameters based on the above equation are presented in our previous study [8].

The processes of film diffusion (D_1, cm^2/s) and pore diffusion (D_2, cm^2/s) control the intra-particle diffusion process. Assuming that the adsorbent particle was a sphere of radius '*a*' and that the diffusion follows Fick's law, the relationship between the uptake and the time is taken for it is given by the following equation [34]:

$$\frac{q_t}{q_e} = 6\left(\frac{D_1}{a^2}\right)^{1/2}\left\{\pi^{-1/2} + 2\sum_{n=1}^{\infty} \text{ierfc}\frac{na}{Dt^{1/2}}\right\} - 3\frac{Dt}{a^2} \tag{7}$$

At the small periods, *D* is replaced by D_1, and Eq. (10) reduces to:

$$\frac{q_t}{q_e} = 6\left(\frac{D_1}{na^2}\right)^{1/2} t^{1/2} \tag{8}$$

A plot of q_t/q_e versus $t^{1/2}$ represents a linear relationship from which the value of the film diffusion coefficient (D_1) for the activated carbon was calculated from the slope (Fig. 2).

For large time periods, the relation between the weight uptake and the diffusion is given by the equation:

$$\frac{q_t}{q_e} = 1 - \frac{6}{\pi^2}\sum_{n=1}^{\infty}\frac{1}{n^2}\exp\left(\frac{-Dn^2\pi^2 t}{a^2}\right) \tag{9}$$

As *t* tends towards larger periods of time, Eq. (12) can be written in the form:

$$\left(1 - \frac{q_t}{q_e}\right) = \frac{6}{\pi^2}\exp\left(\frac{-D_2\pi^2}{a^2}t\right) \tag{10}$$

If $B = (D_2\pi^2/a^2)$, Eq. (13) can be simplified as:

$$\left(1 - \frac{q_t}{q_e}\right) = \frac{6}{\pi^2}\exp(-Bt) \tag{11}$$

$$Bt = -0.4799 - \ln\left(1 - \frac{q_t}{q_e}\right) \tag{12}$$

The values of *Bt* were calculated by using Eq. (15). The calculated values of *Bt* are plotted against *t* as shown

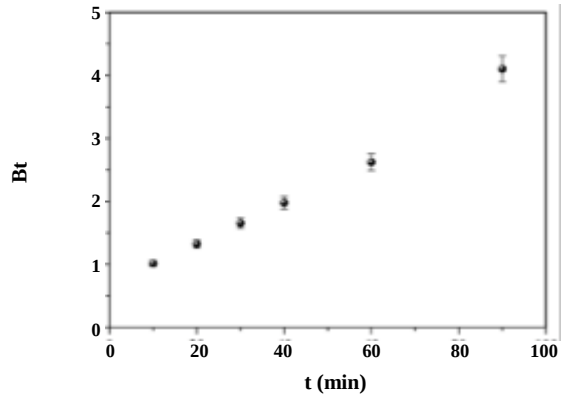

Fig. 3: Plot for determination of pore diffusion coefficient (D_2).

in Fig. 3. We can see that the plot is a straight line not passing through the origin, which means that the adsorption rate is governed by the film diffusion mechanism [35]. The value of B is calculated by the plot slope, this latter is used to calculate the coefficient of pore diffusion (D_2).

External diffusion across the boundary layer surrounding each adsorbent particle and internal diffusion into the porous particle are the two main mediums of mass transfer resistances. The external mass transfer is given by the following equation [36]:

$$N_t = k_L A(C_t - C_e) \tag{13}$$

Where N_t is the diffusion rate across the film layer surrounding the adsorbent particle, k_L is the coefficient of external mass transfer, A is the external surface area of adsorbent, C_t and C_e are the adsorbate concentration at time t and at the equilibrium time, respectively.

Diffusion rate can be also written by using the Eq. (16) as:

$$N_t = -V\frac{dCt}{dt} = m\frac{dq_t}{dt} \tag{14}$$

At the initial conditions (i.e., $C_t = C_0$ and C_e=0 at t = 0), therefore:

$$(N_t)_{t\to 0} = k_L A C_0 \tag{15}$$

Combining the previous equations, the external mass transfer coefficient, k_L, can be written as follows:

$$k_L = \frac{mk_2 q_e^2}{C_0 A} \tag{16}$$

Where m is the mass of the adsorbent (g), k_2 (g/mg.min) is the second-order rate constant, q_e(mg/g) is the equilibrium capacity of adsorbed nickel ions, C_0 (mg/m^3) is the initial concentration of nickel ions and A is the external surface area of the adsorbent.

The diffusion coefficient of nickel ions in an aqueous solution, D_{AB}, was estimated by the Wilke-Chang correlation [37]:

$$D_{AB} = \frac{7.4\times10^{-8}\ (\phi\ M_B)^{1/2}\ T}{\eta_B\ V_A^{0.6}} \tag{17}$$

Where ϕ is the association parameter of the solvent (in the case of water ϕ=2.6), M_B is the molecular weight of water (M_B=18 g/mol), η_B is the viscosity of water (η_B = 0.89 cp), and V_A is the volume molar of nickel (V_A= 6.59 10^{-6} m^3/mol).

The coefficient values of film diffusion (D_1), pore diffusion (D_2), external mass transfer coefficient (k_L), and coefficient diffusion (D_{AB}) are given in Table 2.

The adsorption data in Table 2 shows that the diffusion coefficient (D_{AB}) and external mass transfer (k_L) have a good value this may be due to the increase in the rate of mobility of nickel(II) ions. Chemical interaction, ionic interaction, and other physical forces may be responsible for the diffusion of nickel(II) ions into prepared activated carbon.

The coefficient diffusion parameters found in this study were compared with another coefficient diffusion of other pollutants adsorbed onto activated carbon prepared from different carbonaceous materials.

The comparison of mass transfer coefficient values are listed in Table 3, it can be seen that the value of the external mass transfer coefficient of nickel has the same order as Naproxen Sodium and is different from the other adsorbates. The film diffusion of nickel has the same order of other adsorbates and the pore, diffusion is greater than all of the other adsorbates. They have shown that the mass transfer coefficients increase with the increase in temperature and decrease with the increase in the initial concentration [38, 41].

CONCLUSIONS

Batch experiment studies of equilibrium, mechanism, and mass transfer for nickel(II) ions adsorption have been carried out by using sewage sludge-derived activated carbon. Equilibrium study showed that the Freundlich model provides the best correlation (R^2>0.99) for the adsorption process, which indicates the heterogeneous and multilayer

Table 2: Coefficients diffusion parameters and external mass transfer values.

Parameters	Values
Film diffusion D_1 (cm^2s^{-1})	3.55×10^{-9}
Pore diffusion D_2 (cm^2s^{-1})	3.38×10^{-7}
External mass transfer k_L (cm/s)	7.30×10^{-7}
Diffusion coefficient D_{AB} (cm^2s^{-1})	5.50×10^{-5}

Table 3: Comparison of Coefficients diffusion parameters and external mass transfer.

Adsorbents	Adsorbates	Mass transfer coefficients			References
		k_L (cm/s)	D_1 (cm^2s^{-1})	D_2 (cm^2s^{-1})	
Lignite	Malachite Green	1.32×10^{-5}	3.41×10^{-9}	7.46×10^{-9}	[38]
Polymeric wastes	Naproxen Sodium	2.43×10^{-7}	0.66×10^{-9}	4.75×10^{-11}	[39]
Van apple pulp	Lead	6.19×10^{-8}	0.83×10^{-9}	0.8×10^{-8}	[40]
Van apple pulp	Zinc	3.58×10^{-8}	0.79×10^{-9}	0.51×10^{-8}	
Sewage sludge	Nickel	7.30×10^{-7}	3.55×10^{-9}	3.38×10^{-7}	Present study

adsorption. The maximum monolayer adsorption capacity, Q_{max}, was found to be 11.52 mg/g and the mean adsorption energy (E= 3.98 kJ/mol) indicated that the adsorption process is controlled by physical process. We found that both external mass transfer (D_2) and diffusion coefficient (k_L) are important in determining the adsorption rates (5.50×10^{-5} cm/s and 7.30×10^{-7} cm/s). The value of adsorption energy and kinetic study indicate that nickel(II) adsorption would be attributed to a Physico-chemical adsorption process rather than a purely physical or chemical adsorption process. Based on the results of the present study, activated carbon prepared from sewage sludge can be used as a cost-effective and potential adsorbent for the treatment of waters containing nickel(II) ions.

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