

original article

Isotherms and kinetics studies of biosorption nickel (II) and chromium (VI) from aqueous solution by dried activated sludge

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ABSTRACT

Aims: The aim of this study is the recovery of municipal refuse and using it as a biosorbent for Nickel (II) and Chromium (VI) (Ni^{2+} and Cr^{6+}) removal from aqueous solutions.

Materials and Methods: Activated sludge was obtained from the wastewater treatment plant in a dairy industry. All experiments were performed in the batch system and effective parameters such as the pH, adsorbent dosage, and the initial concentration and contact time of Ni^{2+} and Cr^{6+} were investigated. The adsorption isotherms and kinetics were evaluated to describe the metal uptake and dynamic reactions.

Results: The results of this study showed that with an increasing adsorbent dose and contact time, Ni^{2+} and Cr^{6+} removal efficiency increased. The maximum adsorption of Ni^{2+} and Cr^{6+} was obtained in pH 6 and 2, respectively. Meanwhile, with increasing Ni^{2+} and Cr^{6+} concentration, the removal efficiency decreased. The results best fitted the Langmuir isotherm and the maximum absorption capacity of Ni^{2+} and Cr^{6+} onto dry activated sludge (DAS) were 2.17 and 2.23 mg/g, respectively. Analysis of the adsorption kinetics showed that the intraparticle diffusion kinetic had been good and presented Ni^{2+} and Cr^{6+} uptake onto DAS, and the intraparticle diffusion rate constant of Ni^{2+} and Cr^{6+} were 0.044 and 0.042 ($\text{mg/g min}^{0.5}$), respectively.

Conclusions: According to the results, dry activated sludge is suggested as a low cost and available adsorbent for removing Ni^{2+} and Cr^{6+} from aqueous solutions.

Key words: Adsorption isotherms, adsorption kinetics, biosorbent, heavy metals

INTRODUCTION

Today, water body pollution to organic and inorganic matters such as heavy metals and chlorinated organic compounds is one of the most significant problems.^[1,2] Pollutants can lead to changes in the physical, chemical,

and biological quality in water reservoirs.^[1] Heavy metals such as cobalt, nickel, and chromium have been identified in mines, plating, tannery, electronics, petrochemical, and melting industry wastewaters.^[2-6] Chromium and nickel metals are a cause of major concern in developing countries, due to their excessive usage and their nondegradable nature.^[4] Chromium(VI) is largely soluble in water and carcinogenic.^[2,7,8] Nickel(II) is a more toxic and carcinogenic metal than nickel(IV).^[1,2,4] According to the standard, the industrial effluent permissible discharge level of Cr(VI) and Ni(II) into a water body is 0.1 and 3.0 mg/L, respectively.^[4] In recent years, different methods such as ion exchange, reverse osmosis and chemical precipitation,^[7,9] electrochemical treatment and adsorption,^[4,10] solvent extraction, membrane

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processes, and electro dialysis^[6,11] have been used for heavy metals removal. The disadvantages of these methods are: The high investment costs, lack of usage in a small industry, operation problems, high energy consumption, and sludge production.^[11-13] At present, studies on the biosorption mechanisms of heavy metal removal have increased due to their low cost, high removal efficiency, non-use of chemicals and nutrients, biosorbent regeneration, and the recovery of heavy metals.^[3,5] The aim of present study is the recovery of municipal refuse, to investigate the capacity of dried sludge to adsorb the heavy metals Ni (II) and Cr (VI) from aqueous solutions. The results provide a useful insight into the metal uptake by dried activated sludge at a low cost, and it is a natural adsorbent.

MATERIALS AND METHODS

Preparation of dried activated sludge

Activated sludge was collected as slurry from the sludge return line of the wastewater treatment facility in a dairy industry (Tehran, Iran). The performance characteristics of the sludge used in the experiment are shown in Table 1. Activated sludge was dried at 105°C for 12 hours in a hot air oven to reach a constant weight and then sieved through a 70 – 60 standard mesh (0.21 – 0.25 mm). Following this, the dried activated sludge (DAS) was loaded with H⁺ in a solution of 0.1 M HCl (DAS concentration of 50 g/L) for 30 minutes, under slow stirring (HANA-HI 190 model). Subsequently, the DAS was washed with deionized water to remove the excess hydrogen ions. Finally the DAS was again dried at 105°C for 12 hours.

Chemicals

A stock solution of Ni²⁺ and Cr⁶⁺ were prepared by dissolving Ni (NO₃)₂·6H₂O and K₂Cr₂O₆, in double distilled water. Before performing the experiment, the Ni²⁺ and Cr⁶⁺ concentration in the stock solution was measured. All the chemicals in this study were of extra pure or analytical grade. Ni²⁺ and Cr⁶⁺ concentrations in solution were measured using VARIAN 240 Atomic absorption. All the adsorption experiments were carried out at room temperature (25 ± 2°C). The removal efficiency and Ni²⁺ and Cr⁶⁺ adsorption rate with DAS were calculated as depicted in equations 1 and 2, respectively.

$$1) R = [(C_0 - C_e) / C_0] \times 100$$

$$2) q_e m = (C_0 - C_e) V$$

Batch biosorption experiments

Effect of pH

The effect of pH on the adsorption of Ni²⁺ and Cr⁶⁺ on DAS was carried out using 100 ml of the metal ion solution containing a concentration of 5 mg/L. 0.1 N HCl or 0.1 N NaOH was used to adjust the pH in the range of 2 to 7 (2, 3, 4, 5, 6, and 7) and 0.5 g of DAS was added to each container. After 240 minutes, the remaining concentrations of Ni²⁺

and Cr⁶⁺ in the solution were determined. In all stages, the suspensions were stirred using an agitation speed of 100 rpm.

Effect of contact time

The effect of contact time was performed by shaking 2 g DAS in 100 ml solution with 5, 10, and 20 mg/L Ni²⁺ and Cr⁶⁺ concentration and a constant pH (6 ± 0.2). Next, the remaining concentrations of Ni²⁺ and Cr⁶⁺ were determined at predetermined time intervals (15 minutes) up to 240 minutes. At this stage, for any time was considered one container in order to fix the adsorbent/solution ratio.

Effect of Ni²⁺ and Cr⁶⁺ initial concentration and adsorbent dose

This stage was conducted using 0.5, 1, and 2 g DAS in 100 ml solution of Ni²⁺ and Cr⁶⁺ with initial concentrations of 2 to 35 mg/L (2, 5, 8, 11, 14, 17, 20, 23, 26, 29, 32, 35 mg/L). The remaining amounts of Ni²⁺ and Cr⁶⁺ were analyzed after 180 minutes (equilibrium time).

RESULTS

Effect of pH on Ni²⁺ and Cr⁶⁺ removal

Figure 1 illustrates the results of the pH effect on Ni²⁺ and Cr⁶⁺ removal. Maximum Ni²⁺ and Cr⁶⁺ removal was obtained with pH 6 and 2, respectively. According to Figure 1, by increasing pH from 2 to 6, Ni²⁺ removal efficiency increased from 31 to 49% then dropped after 6.

Effect of contact time on Ni²⁺ and Cr⁶⁺ adsorption

Figures 2 and 3 present the effect of contact time on Ni²⁺ and Cr⁶⁺ adsorption. According to the results, Ni²⁺ and Cr⁶⁺ adsorption increased with an increase in contact time, and 180 minutes was the equilibrium time.

Table 1: The sludge characteristics used for experiment performance

Parameter	Characteristics
Sludge type	Return line
Suspended solids	2.6 g/L
Settleable solids after 30 minutes	318 ml/L
Sludge age	16 days

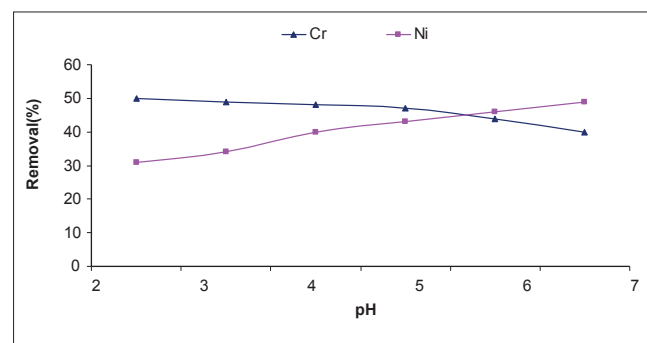


Figure 1: Effect of pH on Ni²⁺ and Cr⁶⁺ removal (initial concentration 5 mg/L, 5 g/L DAS, 240 minutes contact time)

Effect of the Ni²⁺ and Cr⁶⁺ initial concentration and the DAS dose on the adsorption rate

The adsorption capacity (mg/g) ascended with increasing the metal initial concentrations and decreasing the adsorption sites to less than the number of input metal ions [Figures 4 and 5]. In addition, when the DAS dose increased, the removal efficiency increased, but the adsorption capacity decreased. The maximum absorption capacity and removal efficiency were obtained at the Ni²⁺ and Cr⁶⁺ initial concentrations of 35 mg/L and 2 mg/L, respectively.

DISCUSSION

Effect of pH on Ni²⁺ and Cr⁶⁺ removal

The pH of the solutions in the biosorption process should be below 7, as metal ions precipitated at a pH above 7.^[14] According to the previous studies, the adsorption capacities for heavy metals was strongly dependent on the pH of the solution.^[15,16] At low pH, the dominant species of Cr⁶⁺ included HCrO⁺, Cr₂O₇²⁻, Cr₃O₁₀²⁻, and Cr₄O₁₃²⁻, which could be adsorbed primarily because of the electrostatical nature.^[15] At very low pH, the adsorbent surface was coated with hydromium ions, which increased Cr⁶⁺ that interfered with the binding sites on the dried activated sludge. By increasing the pH, the surface charge on DAS became negative and Cr⁶⁺ absorption was reduced. The results of this study for Cr⁶⁺ removal was significant with pomegranate

husk carbon (pH: 1),^[15] wheat bran (pH: 2),^[17] activated carbon (pH: 1),^[7] Aspergillus Niger (pH: 2),^[16] and marine algal mass (pH: 2).^[18]

According to Figure 1, by increasing the pH, the removal efficiency increases and then drops after 6. This can be attributed to the ion exchange. The results of this study are consistent with Aspergillus Niger (pH: 5),^[16] Tithonia diversifolia (pH: 5),^[19] Chlorella vulgaris (pH: 5),^[20] and activated sludge (pH: 4.5)^[12] on Ni²⁺ removal.

Effect of contact time on Ni²⁺ and Cr⁶⁺ absorption

The results of this study showed that as the time increased, the removal efficiency and adsorption capacity increased. The rate of Ni²⁺ and Cr⁶⁺ removal was initially rapid and then decreased gradually to reach an equilibrium, which at that time was not observed to be a significant increase in the removal rate. Perhaps the initial rapid sorption was due to the participation of specific functional groups and active surface sites.^[21] The association between the contact time and Ni²⁺ and Cr⁶⁺ removal efficiency was statistically significant (*P* value < 0.001).

Effect of Ni²⁺ and Cr⁶⁺ initial concentration and the DAS dose on the adsorption rate

In a batch system, the initial concentrations provide an important driving force to overcome all mass transfer resistances of the metal between the aqueous and solid

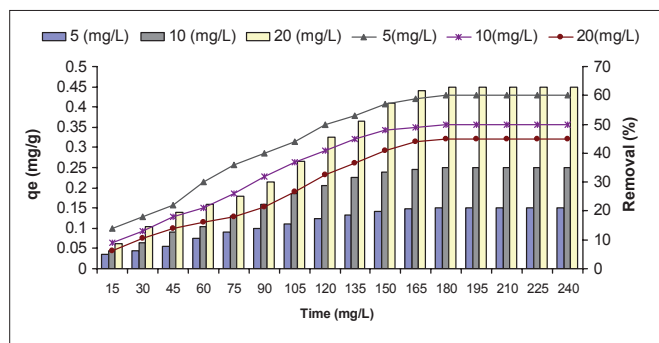


Figure 2: Effect of contact time on Ni²⁺ adsorption (initial concentration 5, 10, and 20 mg/L, 20 g/L DAS, pH: 6)

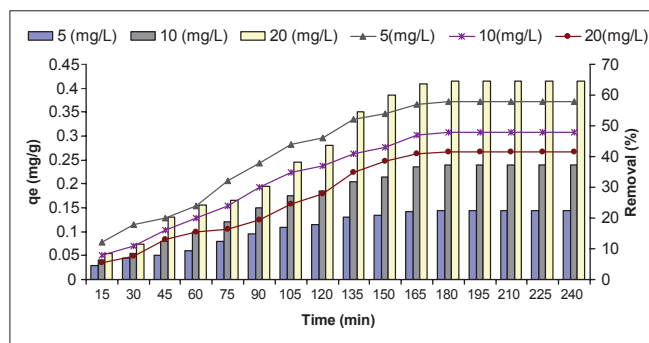


Figure 3: Effect of contact time on Cr⁶⁺ adsorption (initial concentration 5, 10, and 20 mg/L, 20 g/L DAS, pH: 6)

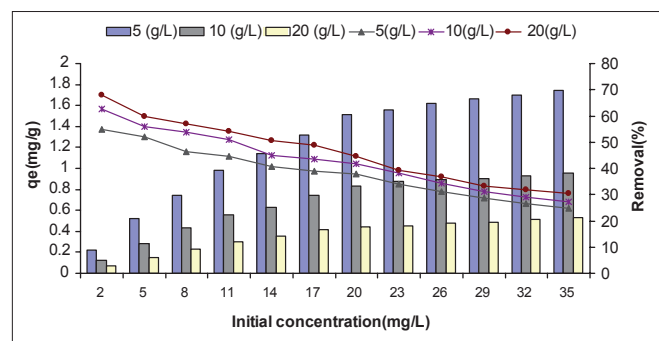


Figure 4: Effect of Ni²⁺ initial concentration and DAS dose on adsorption rate (Ni²⁺ initial concentration 2-35 mg/L, 5, 10, 20 g/L DAS and pH: 6)

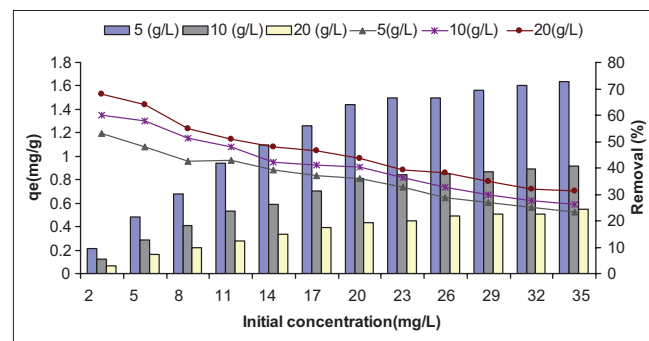


Figure 5: Effect of Cr⁶⁺ initial concentration and DAS dose on adsorption rate (Cr⁶⁺ initial concentration 2-35 mg/L, 5, 10, 20 g/L DAS and pH: 6)

phases. On the other hand, the adsorption capacity (mg/g) ascends with increasing the metal initial concentrations and decreases the adsorption sites to less than the number of input metal ions [Figures 4 and 5]. In addition, when the DAS dose is increased, the removal efficiency increases, but the adsorption capacity decreases.

Isotherms study

The equilibrium of sorption is one of the significant physicochemical aspects for the evaluation of the sorption process as an operation unit.^[22] The correlation of the isotherm information with the theoretical or empirical equation is appropriated for practical operation. In this study, the conventional isotherms in water and wastewater have been analyzed. Tables 2 and 3 show the isotherm equation and results of the isotherm calculations, respectively.

The most appropriate isotherm to describe the results is the Langmuir isotherm. The maximum adsorption capacities for Ni²⁺ and Cr⁶⁺ adsorption onto DAS are 2.17 and 2.23 (mg/g), respectively.

According to Table 3, in the Freundlich isotherm model, K_f and n indicate the Freundlich isotherm constants and adsorption intensity, respectively. The value of these parameters for Ni²⁺ adsorption is higher than Cr⁶⁺ adsorption. In the Langmuir isotherm model, Q_m depicts the required amount of metal ions for the monolayer formation. The Q_m in Cr⁶⁺ adsorption is higher than in Ni²⁺ adsorption, which shows that higher amounts of Cr⁶⁺ are needed to form a monolayer. On the other hand, the absorption capacity for Cr⁶⁺ removal is higher than that of Ni²⁺ removal. Also, the K_L constant represents

the adsorption energy. K_L in Ni²⁺ adsorption is more than in Cr⁶⁺ adsorption, so Ni²⁺ is adsorbed with higher adsorption energy than Cr⁶⁺. The Langmuir and Freundlich isotherms represent monolayer adsorption and the BET isotherm reveals double-layer adsorption. The Langmuir isotherm best fits the experimental data, so Ni²⁺ and Cr⁶⁺ adsorption is monolayered.

The equilibrium factor (R_L), derived from the Langmuir isotherm, is calculated according to equation 3:^[26,27]

$$1) R_L = \frac{1}{1 + (K_L C_0)}$$

R_L can be interpreted according to Table 4.

The computed R_L are 0.63, 0.38 and 0.18 for Ni²⁺ adsorption and 0.65, 0.42 and 0.21 for Cr⁶⁺ adsorption onto 5, 10 and 20 (g/L) DAS, respectively.

According to Table 4, results showed that adsorption of Ni²⁺ and Cr⁶⁺ onto DAS is favorable. Other researches have also confirmed that heavy metal adsorption onto a biosorbent is favorable.^[14]

Kinetics study

Kinetic models are very useful to determine mechanisms such as mass transfer and chemical reaction. Some models have been used in batch reactors to explain the transport of species inside the adsorbents. Among them are the homogeneous surface diffusion, pore diffusion, and heterogeneous diffusion models.^[28,29] Tables 5 and 6 show the kinetic equation and results of the parameter calculation, respectively.

Table 2: Characteristics of studied isotherms

Isotherms	Equation	Liner form	Reference
Freundlich	$q_e = K_f C_e^{1/n}$	$\log q_e = \log K_f + \left(\frac{1}{n}\right) \log C_e$	[23]
Langmuir	$q_e = \frac{Q_m K_L C_e}{1 + K_L C_e}$	$\frac{C_e}{q_e} = \left(\frac{1}{K_L Q_m}\right) + \left(\frac{1}{Q_m}\right) C_e$	[24]
BET	$q_e = \frac{K_B C_e Q_m}{(C_s - C_e) \left[1 + (K_B - 1) \left(\frac{C_e}{C_s}\right)\right]}$	$\frac{C_e}{(C_s - C_e) q_e} = \left(\frac{1}{K_B Q_m}\right) + \left(\frac{K_B - 1}{K_B Q_m}\right) \frac{C_e}{C_s}$	[25]

Table 3: Results of isotherms calculations

Isotherm type	Isotherm parameter	DAS dose (g/L) for Ni ²⁺			DAS dose (g/L) for Cr ⁶⁺		
		5	10	20	5	10	20
Freundlich	n	1.78	1.97	1.03	1.64	1.77	1.81
	K _F	0.51	0.71	0.95	0.58	0.76	0.98
	R ²	0.88	0.89	0.88	0.96	0.96	0.97
Langmuir	Q _m	1.67	0.94	0.49	2.33	1.17	0.68
	K _L ^m	0.24	0.28	0.34	0.11	0.14	0.15
	R ²	0.94	0.97	0.97	0.99	0.99	0.99
	BET	Q _m	1.64	1.01	0.53	1.89	1.01
BET	K _B ^m	10.69	117.61	134.49	44.32	57.79	56.39
	R ²	0.94	0.96	0.96	0.98	0.98	0.98

Table 6 shows that the intraparticle diffusion kinetic model is the most appropriate model for data expression. The maximum rate constants for Ni²⁺ and Cr⁶⁺ adsorption onto DAS are 0.044 and 0.042 (mg/g min^{0.5}), respectively.

The k₂ and K_{diff} are rate constants in the second order and intraparticle diffusion kinetic models, respectively. According to Table 5, the k₂ and K_{diff} for Ni²⁺ adsorption are higher than those for Cr⁶⁺. The calculated q_{cal} for Cr⁶⁺ adsorption is more than for Ni²⁺, and it is a confirmed isotherm calculation. The α and h parameters in the second order and the Elovich

kinetic models define the initial absorption rate. These values for Cr⁶⁺ adsorption are more than for Ni²⁺. The β in the Elovich model is the desorption constant, which is greater for Ni²⁺ adsorption.

Therefore, according to the results presented in Table 7, activated sludge can be considered as a low-cost adsorbent and is relatively efficient for Ni²⁺ and Cr⁶⁺ removal from aqueous solutions.

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Table 4: Equilibrium factor for the shape of isotherms

R _L value	Type of isotherm
R _L > 1	Unfavorable
R _L = 1	Linear
R _L = 0	Irreversible
0 < R _L < 1	Favorable

Table 5: Kinetics and their linear forms

Kinetic	Equation	Liner form	References
Pseudo first order	$\frac{dq_t}{dt} = k_1(q_e - q_t)$	$\log(q_e - q_t) = \log(q_e) - \frac{k_1}{2.303}t$	[15]
Pseudo second order	$\frac{dq_t}{dt} = k_2(q_e - q_t)^2$	$\frac{t}{q_t} = \left(\frac{1}{k_2 q_e^2}\right) + \left(\frac{1}{q_e}\right)t$	[30]
Elovich	$\frac{dq_t}{dt} = \alpha \exp(-\beta q_t)$	$q_e = \left(\frac{1}{\beta}\right) \ln(\alpha\beta) + \left(\frac{1}{\beta}\right) \ln t$	[15]
Intraparticle diffusion	-	$q_t = K_{diff}t^{0.5} + C$	

Table 6 Parameters obtained from kinetic models with different Ni²⁺ and Cr⁶⁺ initial concentrations

Kinetic	Kinetic parameter	Ni ²⁺ concentration (mg/L)			Cr ⁶⁺ concentration (mg/L)		
		5	10	20	5	10	20
Pseudo first order	k ₁	0.02	0.02	0.19	0.02	0.02	0.02
	q _e cal	0.26	0.49	0.87	0.26	0.42	0.95
	R ²	0.87	0.88	0.78	0.86	0.84	0.73
Pseudo second order	k ₂	0.026	0.007	0.001	0.019	0.005	0.001
	q _e cal	0.27	0.59	1.59	0.29	0.63	2.17
	h × 1000	1.89	2.44	2.53	1.59	1.98	4.71
	R ²	0.90	0.89	0.39	0.81	0.80	0.23
Elovich	α	0.61	1.31	2.69	0.66	1.31	2.75
	β	19.16	10.69	6.15	19.31	11.24	6.35
	R ²	0.93	0.92	0.85	0.91	0.92	0.84
Intraparticle diffusion	K _{diff}	0.013	0.024	0.044	0.013	0.023	0.042
	C	0.26	0.066	0.15	0.031	0.067	0.153
	R ²	0.98	0.98	0.94	0.97	0.98	0.97

Table 7: Comparison between various adsorbents used for Ni²⁺ and Cr⁶⁺ on the basis of adsorption capacity

Biosorbent	Nickel (II)		Chromium (VI)	
	Q _m (mg/g)	References	Q _m (mg/g)	References
<i>Aspergillus niger</i>	14.1	[16]	<i>Aspergillus niger</i>	7.2 [16]
<i>Chlorella vulgaris</i>	0.36	[20]	Activated alumina	1.6 [31]
<i>A. Baumannii</i>	8.8	[32]	Ecklonia mass	107.1 [33]
Ecklonia mass	23.4	[33]	Bacillus cereus M ¹ ₁₆	70.25 [34]
<i>P. aeruginosa</i>	5.7	[32]	Wheat brad	0.94 [17]
<i>Calotropis procera</i>	0.94	[35]	Pomegranate husk	35.2 [15]
DAS	2.17	Present study	DAS	2.23 Present study

Nomenclature

C	Thickness of the boundary layer(mg/g)
C_0	Initial concentration (mg/L)
C_e	Equilibrium concentration in solution (mg/L)
C_s	Saturation concentration in solution(mmol/L)
C_t	Equilibrium concentration in solution at time t (mg/L)
h	Initial sorption rate (mg/g minute)
k_1	Pseudo first-order rate constant (1/minute)
k_2	Pseudo second-order rate constant (mg/g minute)
K_B	BET constant
K_{dif}	Intraparticle diffusion rate constant (mg/g minute ^{0.5})
K_f	Freundlich isotherm constants (L/g)
K_L	Langmuir isotherm constants (L/mg)
n	Adsorption intensity
q_e	Equilibrium adsorbent concentration on adsorbent (mg/g)
$q_{e\text{ cal}}$	Calculated values of q_e (mg/g)
Q_m	Maximum monolayer capacity (mg/g)
q_t	Adsorbed metal concentration at time t (mg/g)
R^2	Correlation coefficients
R_L	Dimensional separation factor
α	Initial adsorption rate (mg/g min)
β	Desorption constant (g/mg)

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