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Adsorption of cadmium (II) and nickel (II) on dolomite powder

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ABSTRACT

In this work, the adsorption behavior of cadmium and nickel was studied using natural dolomite powder. The adsorption experiments were carried out to investigate adsorption parameters including metal ions concentration, solution pH, contact time, and temperature using a batch technique. The equilibrium adsorption isotherm data of the metal ions adsorption were best described by Freundlich, Langmuir, and Dubinin–Radushkevich isotherm models. The adsorption capacity was obtained 1.46 and 1.70 mg/g for cadmium and nickel, respectively. Kinetic studies revealed that the initial uptake was rapid and equilibrium was established in, 120 and 105 min for Cd(II) and Ni(II), respectively. The results showed that the data followed the pseudo-second-order reaction. Thermodynamic analysis showed that the adsorption of the metal ions on dolomite is feasible and exothermic. The mean free energy values obtained using the Dubinin–Radushkevich model for the ions showed that the adsorption of nickel and cadmium ions onto dolomite occurs via a physical process for all the temperatures.

Keywords: Adsorption; Kinetic and thermodynamic; Cadmium (Cd); Nickel (Ni); Dolomite

1. Introduction

Toxic heavy metal contamination in the aquatic environment has been of great concern. Heavy metals are widely distributed in the rivers and waterways and elsewhere [1]. The most important problem is the accumulation of toxic metals in food structures poisoning human and animals. Cadmium and nickel were selected as representative heavy metals commonly found in metal rich wastewater. Cadmium and nickel are toxic and frequently encountered together in industrial wastewaters such as plating and cadmium–nickel battery, phosphate fertilizers, mining, pigments, and stabilizers. They can be introduced into surface waters in significant amounts by the industrial effluents [2–6].

Presence of these metals in waste stream and ground water is a very serious environmental concern since these metal ions are toxic to various life forms. They have cumulative effect and tend to accumulate in the living organisms causing various diseases. Therefore, reduction in the pollutant to an acceptable level is necessary [7,8].

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A number of techniques are available for removal of heavy metal ions from aqueous solutions. These are ion exchange, solvent extraction, reverse osmosis, electrochemical reduction precipitation, and adsorption [9–12]. Whereas, some methods such as ion exchange and reverse osmosis are costly, others such as precipitation techniques have problems for disposal of metal-containing sludge. Sorption methods are considered flexible, easy to operate, with much less sludge disposal problems [13,14].

Various researchers have described the adsorption of Cd(II) and Ni(II) ions from aqueous solutions onto various adsorbents. Several natural and synthetic adsorbents have been reported in the literature. In recent years, special attention has been focused on the use of natural adsorbents as an alternative to replace the conventional adsorbents [15–18].

However, new adsorbents with local availability, high adsorption capacity as well as economic suitability are still needed. This has led many researchers to investigate for cheaper substitutes such as coal, fly ash, silica gel, chitosan, agricultural wastes, wood wastes, bentonite, dolomite, perlite, and clay materials. Recently, dolomite has been found as a low-cost adsorbent for removal of metal ions [19–23].

In the present research, the adsorption behavior and potential of dolomite in the removal of Ni(II) and Cd(II) ions from aqueous solutions has been studied. The adsorbent was analyzed using FTIR, XRD, and BET analysis to obtain its characteristics. The adsorption of Ni(II) and Cd(II) ions from aqueous solution was experimentally investigated as a function of metal ions concentration, solution pH, contact time, and temperature. The experimental data were evaluated using isotherm, kinetic, and thermodynamic models.

2. Experimental materials and procedures

Experimental investigations of the adsorption process were carried out using batch technique due to its simplicity and reliability. Cadmium and nickel solutions and standards were prepared by using analytical grade cadmium chloride ($CdCl_2$) and nickel chloride ($NiCl_2 \cdot 6H_2O$) (from Merck Company) with distilled water. The solutions of Cd(II) and Ni(II) were prepared from stock solutions containing 1,000 mg/L of Cd(II) and Ni(II), respectively.

Adsorption experiments were performed at different conditions using the necessary adsorbent in a 250 mL conical flasks containing 100 mL of the metal ion solutions. The initial concentrations of the metal ions were selected to be either 10, 20, 30, 40, or 50 mg/L for adsorption isotherm experiments. Samples were collected at 5, 10, 15, 30, 45, 60, 90, 120, 240, and 360 min to determine optimal contact time. In order to study the dependence of removal efficiency on pH, some experiments were conducted in the pH range of 2–7.5. To determine the effect of temperature and thermodynamic parameters, the experiments were performed at three different temperatures (293.15, 308.15, and 323.15 K). During the adsorption process, the flasks were agitated on a mechanical shaker at 200 rpm. The aqueous samples were analyzed using an inductively coupled plasma-atomic emission spectrometer. The adsorption capacity of adsorbent was calculated through the following equation:

$$q_e = \frac{(C_i - C_e) \times V}{m} \tag{1}$$

The average absolute value of relative error (AARE), is used to compare the predicted results with the experimental data. This is defined as follows:

$$\mathrm{AARE} = \frac{1}{N} \times \sum_{i=1}^{N} \left| \frac{\text{Predicted value} - \text{Experimental value}}{\text{Experimental value}} \right| \tag{2}$$

3. Results and discussion

3.1. Adsorbent characterization

The dolomite sample used in this work was supplied from Tabriz (Iran). The dolomite was ground and sieved on a series of test sieves, and then, it was directly used as adsorbent without any chemical and thermal treatment. The structure of the dolomite group is taken from the calcite group structure. The calcite group structure is layered with alternating carbonate layers and metal ion layers. Dolomite is a carbonate mineral composed of calcium magnesium carbonate $CaMg(CO_3)_2$. The chemical composition of the dolomite used in these experiments was determined by X-ray diffraction and it is summarized in Table 1. The specific surface area of dolomite is 1.85 m^2/g. It was measured by BET nitrogen adsorption. The structure of dolomite was detected by XRD scanning in 2θ range of 0–120, and the obtained pattern is presented in Fig. 1.

The dolomite powder was sieved to obtain a particle size lower than 90 μm. The particle size distribution of the dolomite powder is given in Table 2.

FTIR spectra analyses for the dolomite before and after adsorption were undertaken, and the results are

Table 1
Chemical composition of dolomite

Compound	Percentage (%)
CaO	74.26
MgO	21.42
Na_2O	1.26
Al_2O_3	1.09
SO_3	0.85
Sc_2O_3	0.38
Fe_2O_3	0.22
Loss on ignition	0.52

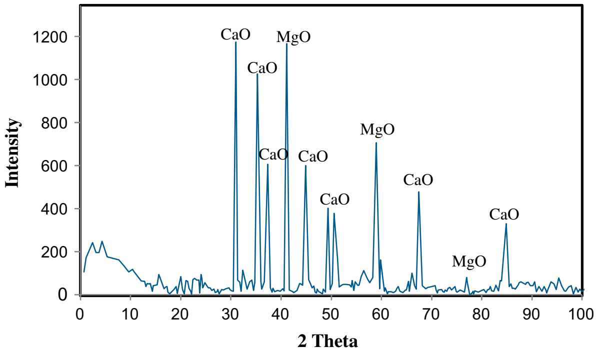

Fig. 1. XRD pattern of dolomite.

Table 2
Particle size distribution of dolomite powder

Size of particles	%
$d < 10\,\mu m$	12.3
$10\,\mu m < d < 25\,\mu m$	16.6
$25\,\mu m < d < 50\,\mu m$	20.7
$50\,\mu m < d < 75\,\mu m$	24.8
$75\,\mu m < d < 90\,\mu m$	25.6

illustrated in Figs. 2 and 3. Dolomite FTIR analysis shows main absorption bands of dolomite at 3,466, 1,437, 871, and 723 cm^{-1}. The results are in good agreement with the absorption frequencies, demonstrated by previous researchers [24,25]. Bands around 3,440 cm^{-1} are related to the presence of bonded hydroxyl group (OH). The band at 1,437 cm^{-1} may be assigned to HCO_3^- group. The FTIR spectra in Fig. 3 confirm that there is a shift of some functional group bands. Differences in the spectra would indicate bonding between the metal with active sites on the dolomite due to adsorption. Dolomite is a double salt, in contact with water, the phenomena of dissolution and hydration will occur. The charge of the surface is therefore the consequence of the formation of the ionic species to the solid–liquid interface, which is a function of pH [26].

Below the point of zero charge, the surface of dolomite is positively charged species. Charges occurring on the dolomite after adsorption of the metal ions are reflected in the board band present at 3,466 cm^{-1}, which is perhaps assigned to the electrostatic attraction between the metal ions and the protonated OH groups at PH 5.5. The metal ions are possibly adsorbed on the (Ca, Mg)OH_2^+ sites in the following form:

$$-(Ca,Mg)OH \leftrightarrow -(Ca,Mg)O^- + H^+ \quad (3)$$

$$-(Ca,Mg)O^- + K^{2+} \leftrightarrow -(Ca,Mg)O - K^+ \quad (4)$$

where K^{2+} is Cd(II) or Ni(II). After contact with Ni(II) and Cd(II) solutions, the dolomite exhibited FTIR spectra with a clear decrease in peak intensities. This could be attributed to an interaction between (Ni(II) and Cd(II)) species and dolomite function group. The stretching intensity is much weaker than that of fresh dolomite, which indicates that the hydrophilic property of the metal ions loaded dolomite is noticeably weakened.

3.2. Effect of pH

The pH of the adsorbate solutions has been identified as the most important parameter governing sorption of metal ions on different adsorbents. The effect of pH on the adsorption of Cd(II) and Ni(II) onto dolomite was studied at pH range of 2.0–7.5, metal concentration 10 mg/L, and adsorbent concentration 1.0 g/L. The adsorption capacity of metal ions versus pH is shown in Fig. 4. It shows that the adsorption of Cd(II) and Ni(II) ions was highly dependent on the initial pH of heavy metal ions solution. The maximum Cd(II) and Ni(II) adsorption was observed at the almost pH 5.5.

At low pH values, the low adsorption observation can be explained due to increase in positive charge density on the surface sites, and thus, electrostatic repulsion occurs between the metal ions and the edge group with positive charge (Ca,Mg–OH_2^+) on the surface:

$$-(Ca,Mg)OH + H - OH \overset{H+}{\rightarrow} (Ca,Mg)OH_2^+ + OH^- \quad (5)$$

Therefore, the surface charge developed at low pH is not suitable for adsorption of the metal ions. When

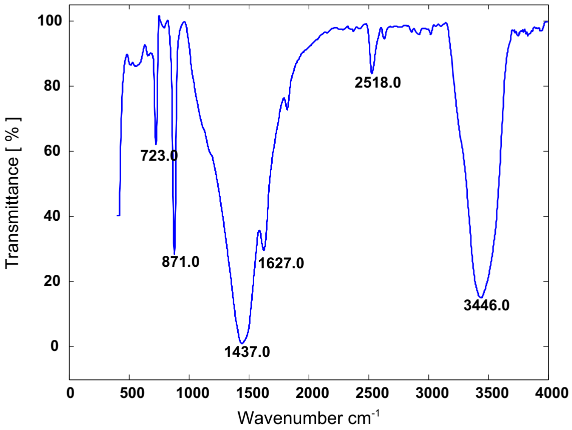

Fig. 2. FTIR spectra of dolomite before adsorption.

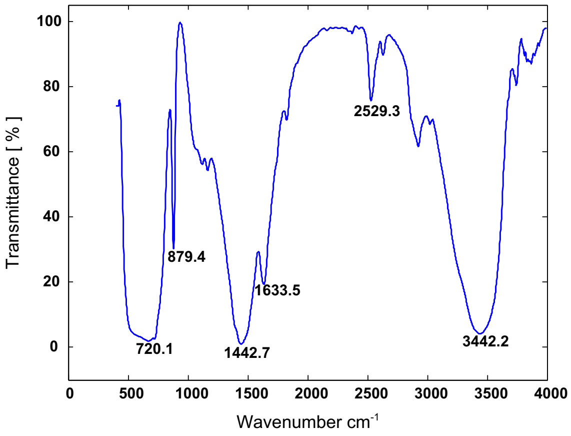

Fig. 3. FTIR spectra of dolomite after adsorption.

the pH was increased, the competing effect of H^+ ions decreased and the positively charged Cd^{2+} and Ni^{2+} (and also $Cd(OH)^+$ $Ni(OH)^+$) ions hook up the free binding sites. Hence, the metal uptake was increased on the surface of the adsorbent with the increase in pH.

In the experiments, which were conducted at the pH higher than 6, metal precipitation appeared. It is obvious that in the alkaline range precipitation plays main role in removing the Cd(II) and N(II) ions attributed to the formation of precipitate of $Cd(OH)_{2(s)}$ and $Ni(OH)_{2(s)}$, respectively. Therefore, the pH of 5.5 was selected to be the optimum pH for further experimental studies.

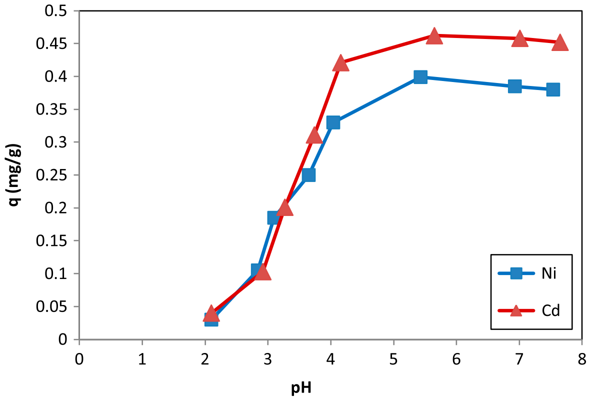

Fig. 4. Effect of pH on the adsorption of Cd(II) and Ni(II) onto dolomite at 293.15 K.

3.3. Adsorption isotherm

Adsorption is usually described through isotherms, that is, the amount of adsorbate adsorbed on the adsorbent as a function of its concentration at constant temperature. The quantity adsorbed is nearly always normalized by the mass of the adsorbent to allow comparison of different materials. The Langmuir adsorption model is the most common model used to quantify the amount of adsorbate adsorbed on an adsorbent as a function of concentration at a given temperature [27]. The saturated monolayer curve can be represented by the expression:

$$q_e = \frac{q_m \times K_L \times C_e}{1 + K_L \times C_e} \tag{6}$$

The values of Langmuir constants q_m and K_L with the correlation coefficients and AARE values for Cd(II) and Ni(II) ions are presented in Table 3. It is clear that the isotherm data of Cd(II) were well described by Langmuir isotherm model. Fig. 5 indicate the nonlinear relationship between the amount (mg) of Cd(II) and Ni(II) ions adsorbed per unit mass (g) of dolomite against the concentration of Cd(II) and Ni(II) remaining in the solution (mg/L).

The Freundlich isotherm model is considered to be appropriate for describing both multilayer sorption and sorption on heterogeneous surfaces [28]. The Freundlich equation may be written as:

$$q_e = k_f \times C_e^n \tag{7}$$

where q_e is the amount of solute adsorbed per unit weight of adsorbent; C_e equilibrium concentration of solute in the bulk solution. The values of Freundlich constants together with the correlation coefficients and AARE values at different temperatures are presented in Table 3. The n values are between 0 and 1 indicating that the adsorption of Cd(II) and Ni(II) ions onto dolomite is favorable at studied conditions. Fig. 6

Table 3
Langmuir, Freundlich, and Dubinin–Radushkevich constants for adsorption of Cd(II) and Ni(II) on dolomite at different temperature

Isotherm equations	293.15 K		308.15 K		323.15 K	
	Cd(II)	Ni(II)	Cd(II)	Ni(II)	Cd(II)	Ni(II)
Langmuir						
K_L	0.0825	0.0113	0.0613	0.0197	0.0605	0.0512
q_m	1.622	5.411	1.190	2.463	0.788	0.697
R^2	0.9674	0.933	0.962	0.940	0.974	0.916
Freundlich						
K_f	0.238	0.086	0.141	0.095	0.102	0.0932
n	0.469	0.811	0.497	0.656	0.466	0.434
R^2	0.895	0.946	0.918	0.905	0.972	0.919
D–R						
β	1.5E–8	2.2E–8	3.1E–8	1.8E–8	1.8E–8	1.5E–8
E	5.8	4.7	4.1	5.2	5.3	5.7
q_m	1.49	1.70	1.83	1.02	0.64	0.65
R^2	0.850	0.970	0.965	0.991	0.920	0.965

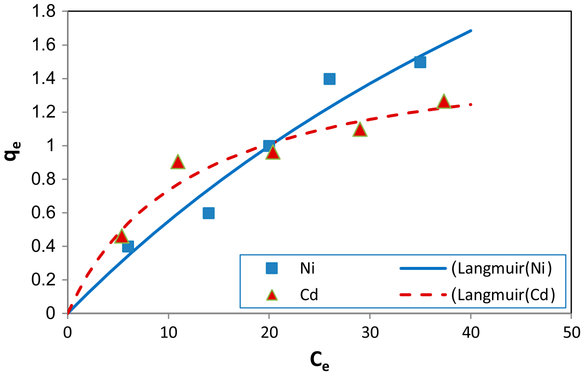

Fig. 5. Langmuir isotherm plots for the adsorption of Cd(II) and Ni(II) onto dolomite at 293.15 K.

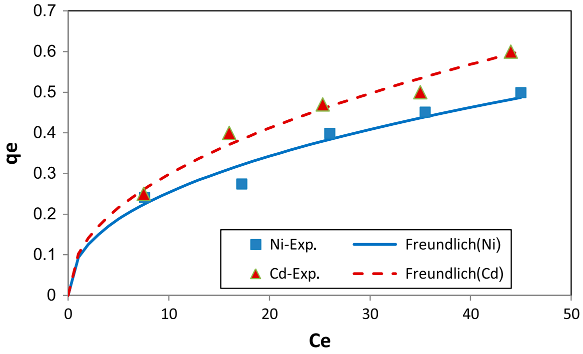

Fig. 6. Freundlich isotherm plots for the adsorption of Cd(II) and Ni(II) onto dolomite at 293.15 K.

shows good agreement between experimental data and Freundlich model results for Ni(II) adsorption.

Dubinin–Radushkevich isotherm model (D–R) predicts the mechanism of the adsorbate sorption onto the adsorbent, and it is used to calculate the mean free energy of adsorption. The nonlinear D–R isotherm is expressed as:

$$q_e = q_m \exp(-\beta\varepsilon^2) \tag{8}$$

The linearized form of the equation is given as:

$$\ln q_e = \ln q_m - \beta\varepsilon^2 \tag{9}$$

where q_e is the amount of solute adsorbed per mass of adsorbent (mg/g), q_m is the maximum adsorption capacity (mg/g), β is the D–R constant $(\mathrm{mol}^2/\mathrm{J}^2)$ *m* and ε is the Polanyi potential (J/mol), which can be calculated as:

$$\varepsilon = RT(1 + 1/C_e) \tag{10}$$

where R is the gas constant (J/mol K), T is the absolute temperature (K), and C_e is the equilibrium concentration of the adsorbate in aqueous solution (mg/L). The values of q_m and β are presented in Table 3. The mean free energy of adsorption (E) was calculated from the β values using the following equation:

$$E = \frac{1}{\sqrt{2\beta}} \tag{11}$$

The E value is used to ascertain the type of adsorption process under consideration. If this value is between 8 and 16 kJ/mol, the adsorption process can be assumed to involve chemical sorption. On the other hand, values lower than 8 kJ/mol indicate that the adsorption process is of a physical nature. In this study, the E values obtained using the D–R constant in Table 3, indicating that the adsorption of Ni(II) and Cd(II) ions onto dolomite occurs via a physical process for all the temperatures.

The shape of the isotherm can also be considered when predicting whether an adsorption system is favorable or unfavorable. The essential characteristic of a Langmuir isotherm can be expressed in terms of a dimensionless separation factor or equilibrium parameter (R_e), which is defined by the following equation [29]:

$$R_e = \frac{1}{1 + K_L \times C_e} \tag{12}$$

According to the value of R_e, for $0 < R < 1$ adsorption is favorable. For the adsorption experiments, the isotherm shapes are between 0.2 and 0.8. Therefore, the adsorption of Cd(II) ion and Ni(II) ions onto dolomite is favorable.

3.4. Kinetic modeling

In order to establish the equilibrium time for maximum uptake and to know the kinetics of adsorption process, Cd(II) and Ni(II) adsorption on dolomite was investigated as a function of contact time. The effect of shaking time on adsorption of Cd(II) and Ni (II) is illustrated in Fig. 7. This figure indicates that the metal concentration in aqueous solution decreases rapidly during first 50 min and remains nearly constant after 120 min for Cd(II) and 105 min for Ni(II).

Most of the metals removals occur in the 50 min. The slower step lasted for the next 70 and 55 min for Cd(II) and Ni(II), respectively. The two steps sorption, the first rapid and the second slower, has been extensively reported in literature [1,2]. The rapid step is probably due to the abundant availability of active sites on the material, and with the gradual occupancy of these sites, the sorption becomes less efficient in the slower step. Based on these results, a shaking time of 120 min for Cd(II) and 105 min for Ni(II) was assumed to be suitable for subsequent sorption experiments.

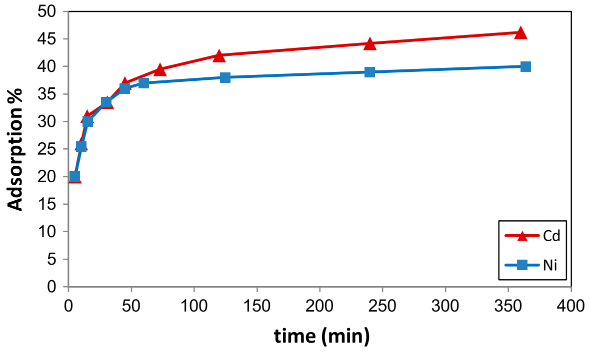

Fig. 7. Effect of contact time on the adsorption of Cd(II) and Ni(II) ions by dolomite at 293.15 k.

Adsorption of Cd(II) and Ni(II) from an aqueous phase by dolomite can be explained by using kinetic models and examining the rate-controlling mechanism of the adsorption process. The study of adsorption kinetics is desirable as it provides information about the dynamics of the adsorption process in terms of the order and the rate constant which are of significance in designing and modeling an efficient adsorption operation. First-order and pseudo-second-order models are the most common kinetic models used to determine the concentration of adsorbate as a function of time. The first-order rate equation is as follows:

$$\ln(q_e - q_t) = \ln q_e - K_1 t \text{ or } q_t = q_e(1 - \exp(-K_1 t)) \tag{13}$$

First-order parameters are presented in Table 4. The pseudo-second-order equation is also based on the sorption capacity of the solid phase. The differential equation of this model is given as:

$$\frac{dq_t}{dt} = K_2(q_e - q_t)^2 \tag{14}$$

For the same boundary conditions, the integrated form becomes:

$$\frac{t}{q_t} = \frac{1}{K_2 \times q_e^2} + \frac{1}{q_e} \times t \text{ or } q_t = \frac{K_2 q_e^2 t}{1 + K_2 q_e t} \tag{15}$$

Figs. 8 and 9 show the nonlinear form of first order and pseudo-second order for the adsorption of Cd(II) and Ni(II). The kinetic parameters for the adsorption of Cd(II) and Ni(II) onto dolomite are summarized in Table 4. The results in Table 4 indicate that the kinetics data of Cd(II) and Ni(II) adsorption can be fitted more accurately by the pseudo-second-order model.

3.5. Thermodynamic modeling

The temperature dependence of adsorption parameters was studied in the temperature range of

Table 4
Kinetic parameters for the adsorption of Ni(II) and Cd(II) ions onto dolomite based on the first-order and pseudo-second-order kinetic equations

Kinetic equation	Cd(II)	Ni(II)
$q_{e(exp.)}$ (mg/g)	0.462	0.400
Lagergren first order		
k_1 (1/min)	0.096	0.120
$q_{e(cal.)}$ (mg/g)	0.411	0.370
R^2	0.842	0.920
AARE%	7.84	5.31
Pseudo-second order		
k_2 (g/mg min)	0.300	0.463
$q_{e(cal.)}$ (mg/g)	0.447	0.401
R^2	0.968	0.995
AARE%	3.62	1.36

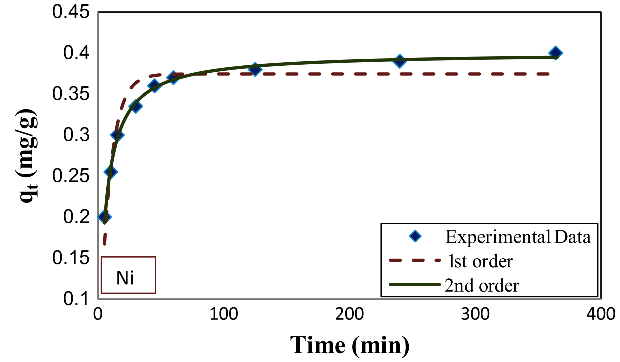

Fig. 8. Effect of contact time on adsorption rate for first order and second order (pH: 5.5; adsorbent dose: 1 g/L; temperature: 293.15 K).

293.15–333.15 K. Thermodynamic parameters were determined using the following equation:

$$\Delta G^o = -R \times T \times \ln K_D \tag{16}$$

The enthalpy change (ΔH°) and entropy change (ΔS°) of adsorption are obtained from the following equation:

$$\ln K_D = \frac{\Delta S^o}{R} - \frac{\Delta H^o}{R \times T} \tag{17}$$

According to thermodynamics, the Gibb's free energy change is also related to the enthalpy change (ΔH°) and entropy change (ΔS°) at constant temperature by the following equation:

$$\Delta G^o = \Delta H^o - T \times \Delta S^o \tag{18}$$

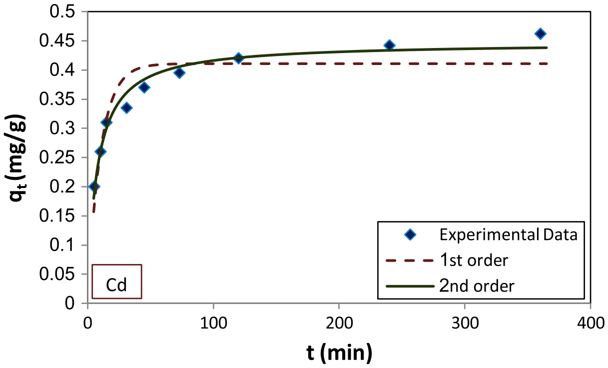

Fig. 9. Effect of contact time on adsorption rate for first order and second order (pH: 5.5; adsorbent dose: 1 g/L; temperature: 293.15 K).

The values of ΔS° and ΔH° can be determined from the intercept and the slope of the plot of ln K_D vs. $1/T$ (Fig. 10). The distribution coefficient (K_D) values decreased with temperature indicating the exothermic nature of the adsorption process. The values of ΔH°, ΔS°, and ΔG° are given in Table 5. The Gibb's free energy value is negative, and decrease in the value of ΔG° with increase in temperature shows that the process occur spontaneously and it is easier at high temperature. Metal ions in aqueous media are hydrated. When the ions get adsorbed on the adsorbent surface, water molecules previously first-order rate equation constants and pseudo-second-order rate equation constants for adsorption of Cd(II) and Ni(II) onto dolomite bonded to the metal ion get released and dispersed in the solution; this results in an increase in the entropy.

The adsorption capacity (q_m) of different adsorbents to adsorb Cd(II) and Ni(II) ions as reported in the literature is compared in Table 6. It can be seen that the q_m value differs widely for different adsorbents. Comparison of q_m values shows that dolomite

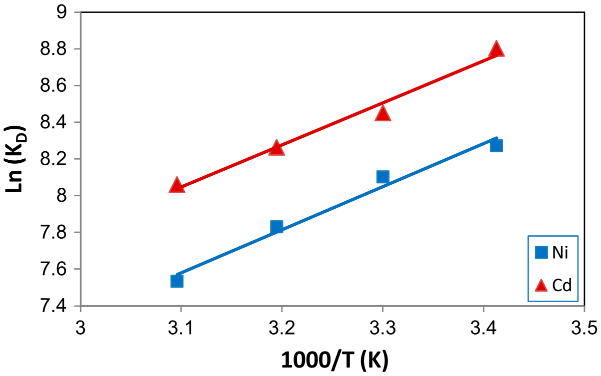

Fig. 10. Plot of ln K_D vs. $1/T$ for the adsorption of Ni(II) and Cd(II) on dolomite.

Table 5
Thermodynamic parameters of Ni(II) and Cd(II) adsorption onto dolomite

Metal ion	ΔH° (kJ/mol)	ΔS° (kJ/mol K)	ΔG° (kJ/mol)			
			293.15 K	303.15 K	313.15 K	323.15 K
Ni(II)	−19.49	2.57e–3	−20.24	−20.27	−20.30	−20.32
Cd(II)	−19.01	7.85e–3	−21.31	−21.39	−21.47	−21.55

Table 6
Adsorption capacity of various adsorbents for the adsorption of Cd(II) and Ni(II)

Natural adsorbent	q_m (mg/g)		References
	Cd(II)	Ni(II)	
Seyitomer fly ash	0.19	1.17	[30,31]
Expanded perlite	1.79	2.24	[3]
Bagasse fly ash	5.22	6.52	[16]
Rice husk ash	2.56	4.87	[7]
Afsin-Elbistan fly ash	0.28	0.99	[30,31]
Amberlite IR-120 resin	85.4	48.07	[1]
Clinoptilolite	4.22	13.03	[5]
Formaldehyde treated *Cystoseira indica*	19.56	16.17	[32]
Live *Aspergillus niger*	1.31	1.75	[33]
NaOH pretreated *Aspergillus niger*	3.43	0.96	[33]
Brewer's yeast	10.17	5.34	[4]
Olive stone waste	6.54	2.13	[34]
Dolomite	1.46	1.70	(This work)

exhibits a reasonable capacity for Cd(II) and Ni(II) adsorption from aqueous solutions. The cost, abundant and locally available of the adsorbent are also important issues that must be considered when selecting an adsorbent.

4. Conclusion

The adsorption of Cd(II) and Ni(II) from aqueous solutions was investigated using dolomite powder. Adsorption experiments were carried out at different conditions to determined optimum values for the adsorption parameters. The experiments showed that the favorable pH for maximum Cd(II) and Ni(II) adsorption was 5.5. Langmuir, Freundlich, and Dubinin–Radushkevich isotherm models were used for analyzing the isotherm data. The isotherm data of Ni(II) and Cd(II) adsorption were well described by Freundlich and Langmuir isotherm model, respectively. The maximum sorption capacity was determined to be 1.46 mg/g for Cd(II) and 1.70 mg/g for Ni(II). The kinetic results showed that the adsorption process followed well the pseudo-second-order model. The values of thermodynamic parameters showed that the adsorption process is feasible and exothermic. Comparison of maximum adsorption capacity of different adsorbents shows that dolomite exhibits a reasonable capacity for Cd(II) and Ni(II) adsorption from aqueous solutions.

Acknowledgment

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Symbols

C_i — initial metal ion concentrations, mg/L
C_e — equilibrium metal ion concentration in solution, mg/L
m — mass of adsorbent, g
N — number values of data points
K_L — Langmuir equilibrium constant
k_f — constant indicative of the relative adsorption capacity
K_1 — adsorption rate constant
K_2 — rate constant of pseudo-second order
K_D — equilibrium constant
n — constant of adsorption intensity
q_e — adsorption capacity at equilibrium, mg/g
q_m — maximum adsorption capacity, mg/L
q_t — adsorption capacity at time t, mg/g
R_e — isotherm shape
R — universal gas constant, 8.314 J/mol K
t — time, min
T — temperature, K
V — volume of solution, L

References

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