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Adsorption of Ni(II) from aqueous solution by *Bacillus subtilis*

Yasemin Bulut*, Ayten Gül, Zübeyde Baysal, Hüseyin Alkan

Department of Chemistry, Faculty of Science, Dicle University, Diyarbakır 21280, Turkey
Tel. +90 412 2488550/3181; Fax: +90 412 2488039; email: ybulut@dicle.edu.tr

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ABSTRACT

This work reports the application of *Bacillus subtilis* as adsorbent for the removal of Ni(II) from aqueous solution. Batch experiments were conducted to study the effects of several parameters such as, contact time, initial concentration of adsorbate (25–200 mg L^{-1}), temperature (298–318 K), and adsorbent dose (0.05–0.4 g) on Ni(II) adsorption. Equilibrium adsorption isotherms and kinetics were also investigated. The equilibrium experimental data were analyzed by Freundlich and Langmuir models. The kinetic data obtained with different initial concentration and temperature were analyzed using a pseudo-first-order, pseudo-second-order, and intraparticle diffusion equations. The results showed that this novel adsorbent had a high adsorption capacity, making it suitable for use in the treatment of Ni(II)-enriched wastewater.

Keywords: Nickel; Adsorption; *Bacilllus subtilis*; Low-cost adsorbent

1. Introduction

Nowadays heavy metals are among the most important pollutants in surface and ground water. They are extremely toxic elements, which can seriously affect plants and animals and have been involved in causing a large number of afflictions [1]. Therefore, the elimination of these metals from water and wastewaters is important to protect public health. For this reason, development of a new, flexible and environmentally friendly process for the treatment of water and industrial effluents is a major challenge [2]. The treatment methods, such as chemical precipitation, membrane filtration, reverse osmosis, ion exchange, and adsorption have been applied for the removal of heavy metals [3]. However, these methods are not economical and do not exhibit high treatment efficiency, especially at metal concentrations in the range of 0.01–0.1 g/L [4,5]. The permissible limit of Ni (II) is 0.01 mg L^{-1} or mg kg^{-1} in water or soil [5].

Among several chemical and physical methods, adsorption has been found to be superior compared to other techniques for wastewater treatment due to its capability for efficiently adsorbing a broad range of adsorbates and its simplicity of design [6–8]. Many studies have been carried out on the possibility of adsorbents using natural materials such as clays [9–11] and zeolite [12], bioadsorbents such as fungal biomass [13] and macro algae [14] and waste materials such as sawdust [15,16].

Biosorption could be such an alternative method of treatment. It employs a wide variety of biomasses, such as algae, fungi, and bacteria, for removal of metal ions [17–19]. Non-living biomass appears to present specific advantages in comparison with the use of living microorganisms. For instance, cells may be stored or used for extended periods at room temperature; they are not subject to metal toxicity, and nutrient supply is not necessary. Moreover, pretreatment and killing of biomass either by physical or chemical treatment or crosslinking are known to improve the

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*Corresponding author.

biosorption capacity of biomass [20]. It has also been reported that cell wall soluble proteins, which make complexes with metal ions, can be fixed by some denaturation processes such as heat and organic solvents, especially ethanol treatments. Deactivated yeast cells do not release protein and exhibit higher metal ion removal capacity than live yeast [21].

The aim of this study is to investigate the equilibrium adsorption of Ni(II) on *Bacilllus subtilis* (*B. subtilis*). The metal loading capacity of *B. subtilis* biomass was determined as a function of the initial metal ion concentration, contact time, temperature, biosorbent dose, and pH. The biosorption data were analyzed by Freundlich and Langmuir isotherm models. The adsorption capacity of the *B. subtilis* suggests that the material could be used as a low-cost alternative in wastewater treatment for the removal of Ni(II) ions.

2. Experimental

2.1. Preparation of the adsorbent

B. subtilis, a gram positive saprophytic microorganism was incubated in a 5-L nutrient broth growth medium on a rotary shaker at 120 rpm at 37°C for 24 h. The cells were collected by centrifugation at 5.000 rpm for 10 min and then dried at 80°C for 48 h. The powdered biomass was used as biosorbent after sieved to select particle size of less than 200 μm.

2.2. Adsorption experiments

Kinetic and equilibrium experiments related to the effect of initial concentration and temperature on the adsorption of Ni(II) from aqueous solution by *B. subtilis* were performed at natural pHs of solutions. Adsorption studies were performed in 100 mL glass vessels. The suspensions containing known doses of *B. subtilis* and Ni(II) solutions were stirred at 298, 308, and 318 K, with an agitation speed of 150 rpm (Julabo SW23). The concentration of Ni(II) in the aqueous phases at certain times (5–250 min) was measured by a AAS (Unicam model 929). The amount of adsorbed Ni(II) was calculated using the following equation [12,13]:

$$q = (C_o - C_e)V/m \qquad (1)$$

where q is the amount of Ni(II) adsorbed onto unit amount of the biomass (mg g^{-1}); C_o and C_e are the concentrations of Ni(II) in the initial solution and in

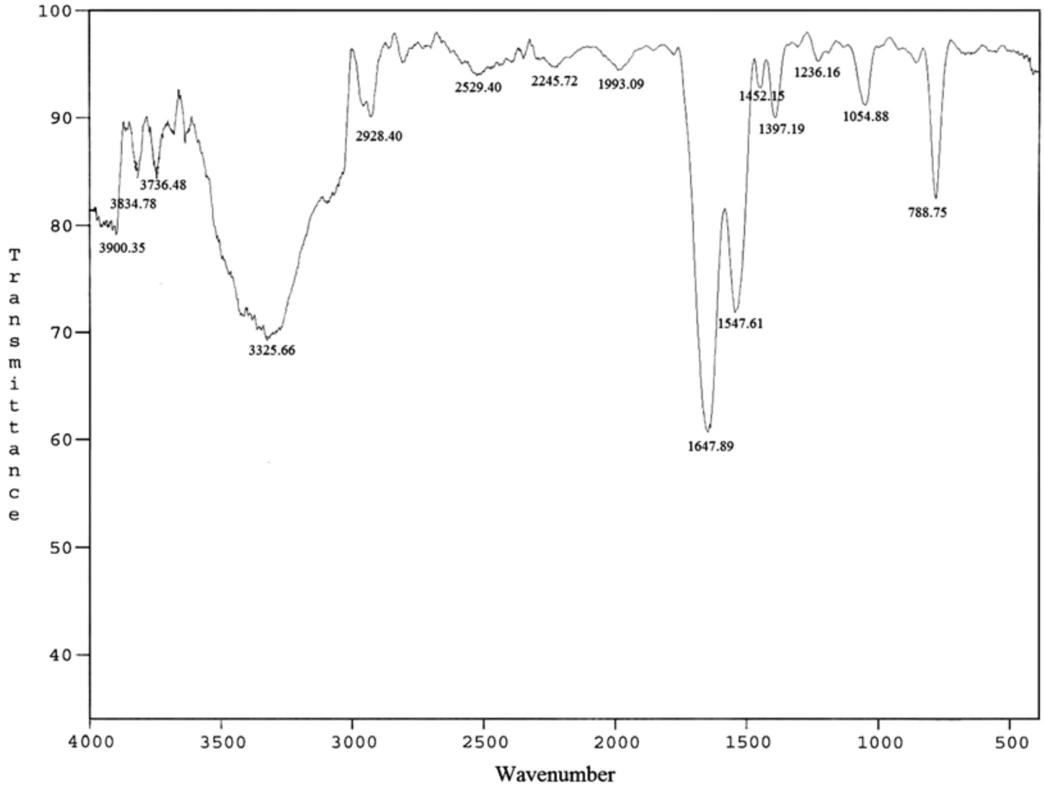

Fig. 1. IR spectra of *B. subtilis*.

the aqueous phase after adsorption, respectively (mg mL^{-1}); V is the volume of the aqueous phase (mL); and m is the weight of the *B. subtilis* (g). The pH was adjusted by adding a small amount of dilute HCl or NaOH solution using a pH meter (Mettler Toledo).

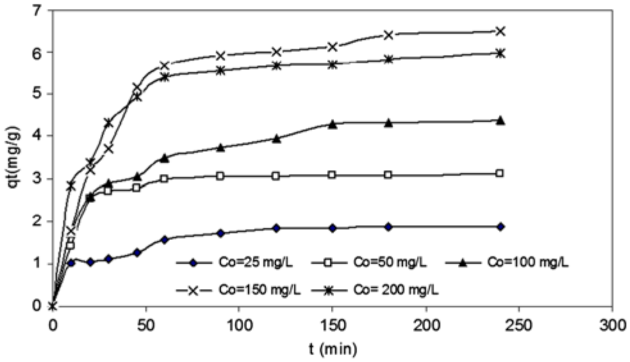

Fig. 2. Effect of contact time on the adsorption of Ni(II) on *B. subtilis* at different initial concentrations. ($T = 298$ K, $V = 100$ mL, $m = 0.8$ g, agitation speed = 150 rpm).

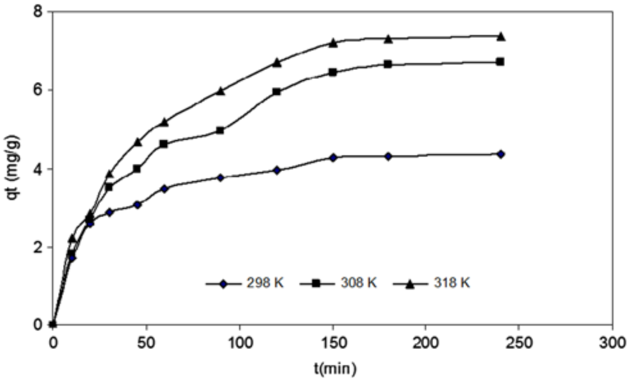

Fig. 3. Effect of temperature on the adsorption of Ni(II) on *B. subtilis* (initial concentration = 100 mg L^{-1}, $V = 100$ mL, $m = 0.8$ g, agitation speed = 150 rpm).

3. Result and discussion

3.1. FT-IR spectral analysis

The IR spectral analysis is important to identify the characteristic functional groups which are responsible for adsorption. The FTIR spectrum of *B. subtilis* biomass in the range 4,000–400 cm^{-1} was measured to determine which functional groups are responsible for the biosorption and the results are shown in Fig. 1. FTIR spectrum of *B. subtilis* biomass shows two distinct peaks at 1,647 and 1,547 cm^{-1} and a broad band at 3,325 cm^{-1}. The peak at 3,325 cm^{-1} may be due to overlapping of –OH and –NH stretching. The sharp peaks at 1,647 and 1,547 cm^{-1} can be assigned to a $-C=O$ stretching in carboxyl or amide groups.

3.2. Sorption results

3.2.1. Effect of initial concentrations and contact time

Figs. 2 and 3 show a plot of the amount of Ni(II) adsorbed (mg g^{-1}) vs. contact time for different initial concentrations and temperatures. Adsorption conditions are given in the figure legend. The contact time necessary to reach equilibrium depends on the initial Ni(II) concentration. It has been shown that the adsorption capacity increases with this concentration [22,23]. As seen from Figs. 2 and 3, high adsorption rates were observed at the beginning and then plateau values (i.e. adsorption equilibrium) gradually reached within 90–150 min for Ni(II). It was observed that the amount of Ni(II) uptake, q_t (mg g^{-1}), increased with the contact time, and the initial concentration at all temperatures. While this equilibrium is achieved in 60 min for the 25–50 mg L^{-1} range, longer times (120 min) are required for higher initial concentrations (100–200 mg L^{-1}). The phenomenon of Ni(II) ions adsorption is a sequential process. After reaching the boundary layer, the Ni(II) ions, diffuse into the surface of the adsorbent and then into its porous structure. At this point the diffusion process takes on special significance due to the fact that, the lower the

Table 1
Adsorption kinetic parameters of Ni(II) on *B. subtilis* different initial concentrations

C_o	Pseudo-first model			Pseudo-second model		
(mg/L)	k_{pf} (1/min)	q_e (mg/g)	R^2	k_{ps} (g/mgmin)	q_e (mg/g)	R^2
25	0.0283	1.067	0.9770	0.026	2.05	0.9959
50	0.0074	1.017	0.5930	0.0447	3.21	0.9992
100	0.0166	1.038	0.9651	0.0104	4.74	0.9982
150	0.0207	1.049	0.9404	0.0121	6.28	0.9993
200	0.0182	1.043	0.9000	0.0059	7.19	0.9964

Table 2
Adsorption kinetic parameters of Ni(II) on *B. subtilis* different initial temperatures

T	Pseudo-first model			Pseudo-second model		
(K)	k_{pf} (1/min)	q_{e} (mg/g)	R^2	k_{ps} (g/mgmin)	q_{e} (mg/g)	R^2
298	0.0221	3.55	0.9619	0.0104	4.74	0.9982
308	0.0230	7.41	0.9364	0.0032	7.9	0.9934
318	0.0270	7.76	0.9751	0.0031	8.63	0.9966

initial concentration, the less competition there is among the Ni(II) molecules to be adsorbed, as a result of which adsorption equilibrium is attained earlier. For this reason, Ni(II) solutions with higher initial concentrations require relatively longer contact time to attain equilibrium, i.e. amount of Ni(II) ions competing. A similar state was observed for the adsorption of Ni(II) from an aqueous solution on the deactivated protonated yeast where the amount of Ni(II) ion sorbed per unit mass of sorbent increased sharply up to 90 min and increased, thereafter, slowly reaching equilibrium [24].

There are several parameters determining the adsorption rate in adsorption process: such as agitation rate in aqueous phase, structural properties of the sorbent (e.g. size, porosity, surface area), amount of the sorbent, properties of the metal ion (e.g. hydrated ionic radius), initial concentration of metal ions, pH, temperature. All individual experimental studies published in the literature have been performed under different conditions, and so, it is not possible to make a reasonable comparison of the adsorption rates reported. But, considering above results it can be said that, we have relatively short equilibrium time.

Kinetic data for heavy metal ions were then represented by the pseudo-first-order (3) and pseudo-second-order (4) equations [5,12,13];

$$\mathrm{Log}(q_{\mathrm{e}} - q_t) = \log q_{\mathrm{e}} - k_{\mathrm{pf}}/\ 2.303\ t \qquad (2)$$

$$t/q_t = 1/k_{\mathrm{ps}}\, q_{\mathrm{e}^2} + 1/q_{\mathrm{e}} t \qquad (3)$$

where q_{e} and q_t are the amounts sorbed in equilibrium and at time t, respectively, k_{pf} and k_{ps} *are* the equilibrium rate constant of pseudo first-order adsorption (min^{-1}) and that of pseudo second-order adsorption ($\mathrm{gmol}^{-1}\,\mathrm{min}^{-1}$), respectively. According to Eq. (2) and (3) the plot of ln $(q_{\mathrm{e}} - q_t)$ vs. *t and that of* t/q_t against t should each give a straight line for the respective model to be applicable. The calculated q_{e}, k_{pf}, k_{ps}, and the corresponding linear regression correlation coefficient R^2 values are summarized in Tables 1 and 2. The correlation coefficient values were lower than those obtained from the pseudo-second-order kinetic model, which is the most commonly used to describe the sorption of Ni(II) ions and basically include all steps of adsorption such as external film diffusion, adsorption, and internal particle diffusion [25].

3.2.2. Effect of temperature

Fig. 3 illustrates the effect of temperature on the adsorption of Ni(II), initially at 100 mg L^{-1}, on *B. subtilis* as a function of time. The equilibrium adsorption capacity was clearly affected by temperature, with the amount of adsorbed Ni(II) increasing from 4.3 to 7.3 mg g^{-1} when the temperature was raised from 298 to 308 K.

The temperature has two major effects on the adsorption process. Increasing the temperature is known to increase the rate of diffusion of the adsorbate molecules across the external boundary layer and in the internal pores of the adsorbent particles as a result of the reduced viscosity of the solution. In addition, the mobility of molecules increases generally with a rise in temperature, thereby facilitating the formation of surface monolayers [23].

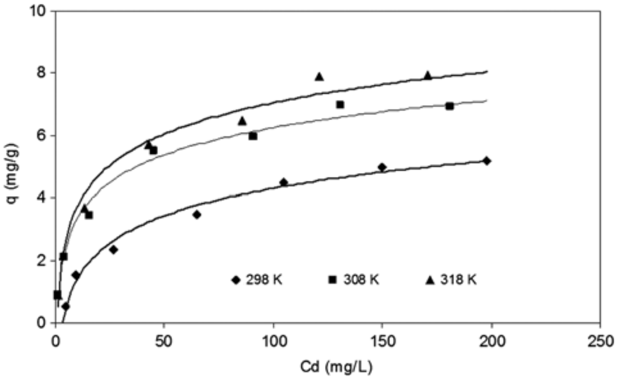

Fig. 4. Adsorption isotherms of Ni(II) on *B. subtilis* at different temperatures ($V = 10$ mL, $m = 0.1$ g, agitation speed = 150 rpm, $t = 150$ min).

Table 3
Adsorption constants for the sorption of Ni(II) on *B. subtilis*

T	Freundlich constants			Langmuir constants		
(K)	k	n	R^2	Q_m (mg/g)	b (L/mg)	R^2
298	0.28	1.72	0.8237	6.40	0.022	0.9874
308	0.915	2.72	0.8655	7.38	0.048	0.9946
318	0.711	2.03	0.9526	8.50	0.066	0.9895

3.2.3. Activation parameters

Considering the three of pseudo-second-order rate constants, k_{ps}, each at a different temperature, for initial conceration $100\,\mathrm{mg\,L^{-1}}$ and using the Arrhenius equation (Eq. (6)), it is possible to gain some insight into the type of adsorption.

$$\ln k_{ps} = \ln\ A - E_a/RT \tag{4}$$

where E_a is the activation energy ($\mathrm{J\,mol^{-1}}$), k_{ps} *is* the pseudo second-order rate constant for adsorption ($\mathrm{g\,mol^{-1}\,s^{-1}}$), A is the temperature-independent Arrhenius factor ($\mathrm{g\,mol^{-1}\,s^{-1}}$), R the gas constant ($8.314\,\mathrm{J\,K^{-1}\,mol^{-1}}$), and T is the solution temperature (K). The slope of the plot of ln k_{ps} vs. $1/T$ can then be used to evaluate E_a (not shown figure). Low activation energies are characteristic of physical adsorption, while higher ones suggest chemisorption [23]. The present results give $E_a = 3.807\,\mathrm{kJmol^{-1}}$ for the adsorption of Ni(II) on *B. subtilis*, indicating that adsorption has a low potential barrier and corresponds therefore to physisorption.

3.2.4. Adsorption isotherms

The equilibrium adsorption isotherm is of great importance in the design of adsorption systems [26]. It is expressed by relating the amount of adsorbate taken up per gram of adsorbent, q_e ($\mathrm{mg\,g^{-1}}$), to the equilibrium solution concentration, C_e ($\mathrm{mg\,L^{-1}}$), at a fixed temperature (Fig. 4). Several isotherm equations are available, and two important isotherms were selected for this study: Langmuir and Freundlich isotherms. Langmuir adsorption isotherm assumes that adsorption takes place at specific homogeneous sites within the adsorbent, and it has been used successfully for many adsorption processes of monolayer adsorption. The linearized Langmuir equation is represented as follows [5,26]:

$$C_e/q_e = 1/b\,Q_m + C_e/Q_m \tag{5}$$

where b is the equilibrium constant or Langmuir constant related to the affinity of binding sites ($\mathrm{Lmg^{-1}}$) or ($\mathrm{Lmol^{-1}}$) and Q_m represents monolayer adsorption capacity when the surface is fully covered with Ni(II) ions (mg/g). Q_m and b were calculated from the slope and intercept of the straight lines of the plot C_e/q_e vs. C_e. Freundlich isotherm is an empirical equation used to describe heterogeneous systems, and it is given in logarithmic form as [24,26]:

$$\mathrm{Log}\ q_e = \log k + (1/n)\log C_e \tag{6}$$

where k is roughly an indication of the adsorption and $(1/n)$ is of the adsorption intensity, k and $(1/n)$

Table 4
Comparison of maximum monolayer capacity for Ni(II) on the other different adsorbents [24]

Organism	Metal uptake capacity (mg/g)
Candida cells (adapted)	30.8
Carbon (granular activated)	1.5
Activated sludge	23.9
Iron hydroxide	7.0
Rice hull	5.8
Sugar beet pulp	10.7
Pseudomonas Syringae	6.0
Deactivated protonated yeast	9.0
Present study	6.4–8.5

Table 5
Values of thermodynamic parameters (kJmol^{-1}) for the adsorption of Ni(II) on *B. subtilis*

T (K)	ln *b*	1/*T*	ΔG (kJ/mol)	ΔH (kJ/mol)	ΔS (KJ/mol)
298	7.164	0.00336	−17.748	43.460	205.971
308	7.944	0.00325	−20.341		
318	8.262	0.00314	−21.844		

can be determined from the linear plot of log q_e vs. log C_e. Adsorption isotherms were obtained in terms of Eqs. (5) and (6) by using experimental adsorption results in these equations. The values for Q_m, *b*, *k*, and *n* are summarized in Table 3.

The isotherm data were calculated from the least square method and the related correlation coefficients (R^2 values) are given in the same table. As seen from Table 3, Langmuir equation represents the adsorption process very well; the R^2 values were all higher than 0.987, indicating a very good mathematical fit. The maximum adsorption capacities (Q_m) of the *B. subtilis* in the studied range are 6.40, 7.38, and 8.50 mg g^{-1} for Ni(II) at 298, 308 and 318 K, respectively. A brief comparison of Ni(II) sorption capacity of the *B. subtilis* with recently reported some composites are given in Table 4 [24].

k is a Freundlich constant showing the adsorption capacity of an adsorbent, and is a constant which shows the strength of the relationship between adsorbate and adsorbent. It is generally stated that the values of *n* in the range 1–10 represent good adsorption [26]. In the present work, the coefficient was $1 < n < 10$, indicating favorable adsorption.

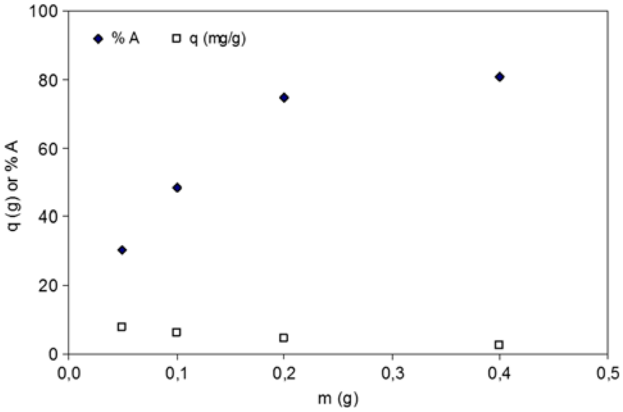

Fig. 5. Effect of adsorbent dosage of Ni(II) (initial concentration = 100 mg L^{-1}, $V = 10$ mL, agitation speed = 150 rpm, $T = 298$ K, $t = 150$ min).

3.2.5. Thermodynamic parameters

The thermodynamic parameters that must be considered to determine the process are free energy change (ΔG°), enthalpy of adsorption (ΔH°), and entropy change (ΔS°) due to transfer of unit mole of solute from solution on the solid–liquid interface [16]. The values of free energy change (ΔG°) for the sorption process were calculated using the following equation [22,26]:

$$\Delta G^\circ = -RT \ \ln \ b \tag{7}$$

where *R* is the universal gas constant (8.314 J/mol K) and *T* is the absolute temperature. Also enthalpy (ΔH°) and entropy (ΔS°) changes can be estimated by the following equation:

$$\ln \ b = \Delta S^\circ/R - \Delta H^\circ/R \ T \tag{8}$$

Thus, a plot of ln *b* vs. 1/*T* should be a straight line. ΔH° and ΔS° values were obtained from the slope and intercept of this plot, respectively. ΔG°, ΔH°, and ΔS° obtained from Eqs. (7) and (8) are given in Table 5. The negative value of ΔG°, indicating that the adsorption process leads to a decrease in Gibbs free energy, confirms the feasibility of the process and the spontaneous nature of the adsorption with a high preference of Ni(II) on *B. subtilis*. However, this value decreases with an increase in temperature, indicating that the spontaneous nature of adsorption is inversely proportional to the temperature. The positive values of ΔS° show the increased randomness at the solid/solution interface during the adsorption process. The adsorbed water molecules, which are displaced by the adsorbate species, gain more translational energy than the energy lost by the adsorbate ions, thus allowing the prevalence of randomness in the system. The positive value of ΔH° (43.46 kJ mol^{-1}) indicates the endothermic nature of the process as it was demonstrated when the effect of temperature on adsorption capacities was studied.

3.2.6. Effect of adsorbent dosage

Adsorbent dosage is an important parameter because this factor determines the capacity of an adsorbent for a given initial concentration of the adsorbate. The effect of adsorbent dosage (adsorbent prepared in different batch) was studied on Ni(II) removal by keeping all other experimental conditions constant (Fig. 5). Adsorption conditions are given in the figure legend. The percentage of adsorption %*A* of Ni(II) adsorbed increasing from 30.34 to 80.57, but the amount of Ni(II) adsorbed decreasing from 7.59 to $2.52\,\mathrm{mg\,g^{-1}}$ when the adsorbent dosage was raised from 0.05 to 0.4 g. The decrease in unit adsorption with increasing dose of adsorbent is basically due to adsorption sites remaining unsaturated during the adsorption reaction [26].

4. Conclusions

The adsorption equilibrium of Ni(II) on *B. subtilis* was attained within times of less than 150 min. The amounts of adsorbed Ni(II) increased when temperature increased. The equilibrium data were described better by Langmuir isotherm model than by Freundlich isotherm. Adsorption of Ni(II) followed pseudo-second-order kinetics. Considering above results, it can be said that the study explained here is an inexpensive method for the removal of Ni(II).

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