



Research Journal of  
**Environmental  
Sciences**

ISSN 1819-3412



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## Biosorption of Nickel from Batch Reactor using the Powder of Waste Activated Sludge

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### ABSTRACT

In the present study, the biosorption of nickel ions from aqueous solutions by powder of waste activated sludge was studied. The effect of contact time, pH and shaking rate were investigated. Pseudo-second-order model described the biosorption kinetics better than the Lagergren model. Kinetic experiments showed that nickel concentrations reached equilibrium within 2 h. We found that the biosorptive capacities were dependent on nickel solution pH, with pH 4 being optimal. The influence of the shaking rate on the biosorption capacity of the biomass shows that an optimum value can be obtained between 150 to 250 rpm. The Langmuir isotherm model was applied to experimental equilibrium data of nickel biosorption. The result showed that maximum nickel adsorption capacity of powder of waste activated sludge ( $q_{max}$ ) was 11.442 mg g<sup>-1</sup> and the Langmuir constant ( $k_d$ ) was 13.9611 mg L<sup>-1</sup>.

**Key words:** Biosorption, nickel, sludge, isotherm, kinetics

### INTRODUCTION

As today's technology progresses and increment in industrial activities, a high volume of wastewater containing heavy metal is released to the natural environment (Pahlavanzadeh *et al.*, 2010). Heavy metal pollutants are extremely pernicious because they are environmentally persistent and toxic. Nickel is one of the metals being widely used in alloys, pigments, electroplating processes, electrical contacts and battery manufacturing (Roskill Information Service, 2000). Nickel belongs to the so-called "essential" metals and is identified as a component in a number of enzymes, participating in important metabolic reactions, such as ureolysis, hydrogen metabolism, methane biogenesis and acidogenesis (Akhtar *et al.*, 2004). But, nickel(II) ion intake over the permissible levels results in different types of disease such as pulmonary fibrosis, renal edema, skin dermatitis and gastrointestinal distress (e.g., nausea, vomiting, diarrhea) (Akhtar *et al.*, 2004). The US Environmental Protection Agency (EPA) requires nickel in drinking water not to exceed 0.5 mg L<sup>-1</sup> (WHO, 2006). It is therefore, essential to remove Ni(II) from wastewater before disposal (Gupta *et al.*, 2010).

The removal of such contaminants up to levels approved by national or international agencies has not totally been solved by conventional chemical precipitation procedures (Hammami *et al.*, 2007). Chemical precipitation, ion exchange, reverse osmosis, solvent extraction and adsorption methods found limited applications in heavy metal removal due to high operational costs together with the production of undesired wastes which require a secondary handling (Veglio *et al.*, 2003). Biosorption is an effective method of removing heavy metals ions from wastewater. Metal ions present in wastewater are biosorbed onto the surfaces of activated sludge bacteria by fast passive adsorption followed by slow metal ion uptake (Aksu, 2005).

Biosorption mainly involves cell surface complexation, ion exchange and microprecipitation (Gupta *et al.*, 2000). Some advantages of biosorption for removal of heavy metals over chemical and physical methods can be summarized as follows:

- Excess sludge from wastewater treatment plants may be used as biosorbent
- Low cost, free availability and possible reuse of the biosorbent
- High biosorption capacity because of large surface area of sludge organisms
- Selective adsorption of metal ions
- Operation over a broad range of environmental conditions (Pamukoglu and Kargi, 2007)

It was well-reported that some single species isolated from activated sludge have high potential to adsorb heavy metals (Hammami *et al.*, 2002; Leung *et al.*, 2001; Gulnaz *et al.*, 2004). The excess activated sludge provides an excellent opportunity for removal of heavy metals by biosorption because of its availability on the plant site and free use (Aksu, 2005). Biological wastewater treatment produces a biological sludge consisting of inert materials and microorganisms. Currently, there are limited reuse/disposal options for biomass due to their high concentrations of heavy metals and the risk of pathogens. Previous research had shown the ability of this biomass to remove metals from the wastewater stream (Norton *et al.*, 2004). Waste activated sludge consists of the nonliving microorganisms which are no longer required in the wastewater-treatment process and are ready for disposal. Dewatered waste activated sludge has a solid content of 13%. To overcome the risk of pathogens as well as the transportation and storage issues associated with live return activated sludge; dewatered nonliving waste activated sludge was chosen for this study.

## **MATERIALS AND METHODS**

**Chemicals:** All chemicals were used as analytical reagent grade and were used without further purification. Nickel solutions were prepared according to the 'Standard Methods' (Eaton *et al.*, 1998) from analytical reagent-grade nickel dust ( $\text{Ni}(\text{NO}_3)_2 + 6 \text{H}_2\text{O}$ , Merck Company). Stock 0.1 M nickel solutions were initially prepared and preserved with 1.5 mL of concentrated  $\text{HNO}_3$  per liter (Eaton *et al.*, 1998) then diluted prior to use. In all cases where samples needed to be stored, they were preserved as detailed in the 'Standard Methods'. The solution pH was adjusted to the required value with  $\text{HNO}_3$  or  $\text{NaOH}$ . Acid washing with a solution of 1  $\text{HNO}_3$  + 1 water followed by a triple rinse with distilled water was conducted to avoid metal uptake onto the glassware (Eaton *et al.*, 1998).

**Preparation of sorbent:** In this study the required sorbent were sourced from the waste sludge produced at the Ekbatan wastewater-treatment plant in Tehran. This treatment plant was chosen because the wastewater treated there is of domestic origin and it has low background concentration

of nickel. Pretreatment of that included dewatering at 103-104°C and grinding to grain size of between 50 to 120 standard meshes. The initial concentration of nickel in this powder was measured by nitric acid digestion (Eaton *et al.*, 1998) and atomic-adsorption analysis. The initial value was 0.00293415 mM g<sup>-1</sup> of dry powder of waste activated sludge.

**Experimental procedure:** This survey was a part of a project (No: ENV-1-83123) of the Iran Water Resources Management Co. (under the Ministry of Energy) from Jan., 2005 to Jan., 2006. The laboratory phase carried out on environment college laboratory of Tehran University. The tests were conducted in laboratory scales in a batch reactor system of 800 mL volume. Nickel solutions of different concentrations were prepared by adequate dilution of the stock solution with deionized distilled water. Batch equilibrium experiments were carried out by adding a measured amount of powder of waste activated sludge to the nickel solutions. The solution was placed on an electric mixer and this solution mixed until equilibrium was reached. The powder of waste activated sludge was removed by filtration through a 0.45-µm membrane filter (Millipore) and the filtrates were analyzed for residual nickel concentration by atomic-adsorption spectrophotometry (Unicam 919). The experiments were conducted in a temperature-controlled room (25±1°C). All experiments were conducted at least in duplicate and the average reported in the results.

In the kinetic studies, nickel solutions at concentrations of 0.25 and 0.75 mM were used in conjunction with 1.0 g (dry wt.) of powder of waste activated sludge at 25±1°C. Reaction times of 5, 10, 20, 30, 60, 120, 180, 300 and 420 min were investigated. A shaking rate of 200 rpm and the initial pH of 4 were used.

When investigating the effect of initial pH value, metal concentrations of 0.25 and 0.75 mM were used in conjunction with 1.0 g (dry wt.) of powder of waste activated sludge at 25±1°C. Initial solution pH was adjusted to 2, 3, 4 or 6. The reaction time was 2 h and a shaking rate of 200 rpm was used.

In the shaking-rate studies, metal concentrations of 0.25 and 0.75 mM were used in conjunction with 1.0 g of powder of waste activated sludge at 25±1°C. Shaking rates of 50, 100, 200 and 300 rpm were investigated. The reaction time was 2 h and an initial pH of 4 was used.

For Sorption equilibrium studies, powder of waste activated sludge masses of 0.5, 1, 2 and 4 g (dry wt.) were used in conjunction with solution concentrations of 0.25 and 0.75 mM at 25±1°C. The initial pH was set to 4, the reaction time was 2 h and a mixing speed of 200 rpm was used. These results were used for adsorption isotherm studying and modeling.

## RESULTS AND DISCUSSION

**Biosorption kinetics of nickel uptake:** Nickel-adsorption kinetic profiles at initial solution concentrations of 0.25 and 0.75 mM are shown in Fig. 1; the nickel-uptake equilibrium time at these concentrations was 2 h. With an increase of the initial concentration of nickel solution, the relative adsorption rate increased in the first 5 min. However, the adsorption rate decreased afterwards.

With increased initial concentration of nickel, the adsorption rate (adsorption to initial concentration ratio) decreased. More specifically, at the concentration of 0.25 mM, the adsorption was 42.1% of the total nickel in the system, however for concentrations of 0.75 mM the adsorption dropped to 23.7%. Nevertheless, an increase in the initial concentration of a solution will generally increase the total metal adsorption. In previous studies on zinc-ion adsorption by biosolids (dry waste sludge), Norton *et al.* (2004) found that at concentrations of 0.076 and 0.3 mM equilibrium was achieved in 5 h at pH 4. In their studies the maximum adsorption was 0.006 and 0.026 mM g<sup>-1</sup> of biosolids, respectively.

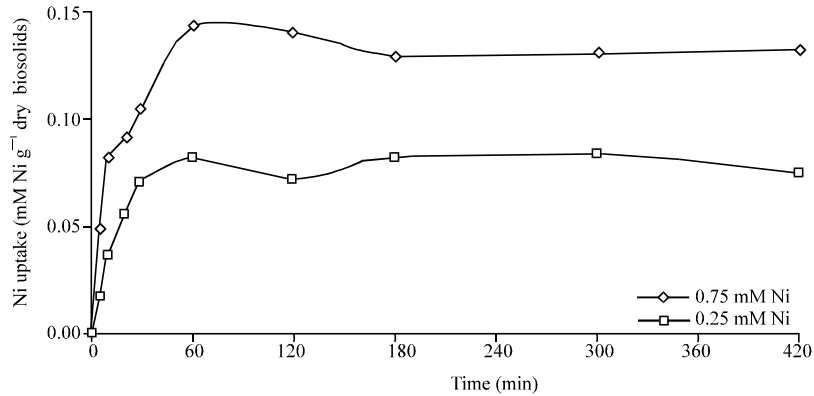


Fig. 1: Nickel-adsorption kinetic profile at 0.25 and 0.75 mM concentrations

Two different kinetic models were used to fit the experimental data for nickel biosorption. The pseudo-first-order Lagergren model is generally expressed as (Hammami *et al.*, 2007):

$$\frac{dq}{dt} = k_{1,ads}(q_e - q) \quad (1)$$

where  $q_e$  ( $mg\ g^{-1}$ ) and  $q$  are the amounts of adsorbed metal ions on the biosorbent at equilibrium and at any time  $t$ , respectively and  $k_{1,ads}$  is the Lagergren rate constant for the first-order biosorption. Integrating Eq. 1 between the limits  $t = 0$  to  $t = t$  and  $q = 0$  to  $q = q_e$  provides:

$$\log(q_e - q) = \log q_e - \frac{k_{1,ads}}{2.303} t \quad (2)$$

Linear plots of  $\log(q_e - q)$  versus  $t$  indicated the applicability of this kinetic model. However, to adjust Eq. 2 to fit the experimental data, the value of  $q_e$  (the equilibrium sorption capacity) must be pre-estimated by extrapolating the experimental data to  $t = \infty$  (Kaewsarn, 2002).

The pseudo-second-order model is based on the assumption that biosorption follows a second order mechanism; thus, the rate of occupation of adsorption sites is proportional to the square of the number of unoccupied sites:

$$\frac{dq}{dt} = k_{2,ads} (q_e - q)^2 \quad (3)$$

where  $k_{2,ads}$  is the rate constant for the second-order biosorption ( $g\ mg^{-1}\ min$ ). Integrating (3) from  $t = 0$  to  $t = t$  and  $q = 0$  to  $q = q_e$  and then linearizing it yields Eq. 4:

$$\frac{dq}{dt} = k_{2,ads} (q_e - q)^2 \quad (4)$$

Here,  $q_e$  and  $k_{2,ads}$  can be calculated from the slope and the intercept, respectively, of a plot of  $t/q$  versus  $t$ . It is important to note that it is not necessary to estimate the experimental value of  $q_e$  for the application of such a model (Kaewsarn, 2002).

The results showed a good fit for nickel adsorption kinetics with the pseudo-second-order model; in fact, the correlation coefficient for the second-order kinetic model was equal to 1 (exactly 0.977). A value of  $q_e$ , 0.164 (mM Ni g<sup>-1</sup> dry powder of waste activated sludge), was obtained which was almost equal to the 0.178 mM g<sup>-1</sup> read from the adsorption diagram. The rate constant of the second-order biosorption,  $k_{2,ads}$ , was calculated as 0.00765.

Pamukoglu and Kargi (2006) showed that adsorption of copper (II) ions by pre-treated powdered waste sludge followed the pseudo-second order kinetic model.

Naddafi *et al.* (2005) showed that Biosorption of Lead (II) and Cadmium (II) by Brown Algae *Sargassum sp.* Biomass followed the pseudo-second order kinetic model. In studies on copper biosorption by brown seaweed, it was shown that copper adsorption by *Sargassum chromophyta* followed a pseudo-first-order Lagergren model (Antunes *et al.*, 2003).

Yan and Viraraghavan (2003) showed that biosorption of heavy metals like lead, nickel, cadmium and zinc by *Mucor rouxii* biomass did not follow the Lagergren equation; consequently, they conformed to the Ho false second-degree model.

**Effect of pH on nickel uptake:** Figure 2 shows that the effect of pH on the adsorption of nickel. As shown here, the effect of pH on adsorption with 0.25 and 0.75 mM nickel was almost the same. The metal uptake at pH 2 was negligible; thus, indicating the possibility of using this pH effect for metal elution and biomass regeneration. In fact, under pH 2, nickel was leached out of the biomass and into solution. This clearly demonstrated that nickel uptake increases with solution pH.

The increase of pH from 2 to 3 slightly increased the adsorption rate; however, from pH 3 to 4 the adsorption rate increased rapidly. At pH greater than 4 the adsorption rate increased insignificantly; therefore, the maximum rate of adsorption was at a pH of slightly more than 4.

Kaewsarn (2002) showed that the biosorption of copper by a *Padina sp.* was affected by pH. At pH below 2 the adsorption was minimal and with an increase of pH up to 4 the adsorption increased exceedingly; again, above that value the increase was insignificant.

According to Esmaeili *et al.* (2007) results, the maximum nickel uptake was obtained at pH 7 by brown algae *sargassum sp.*

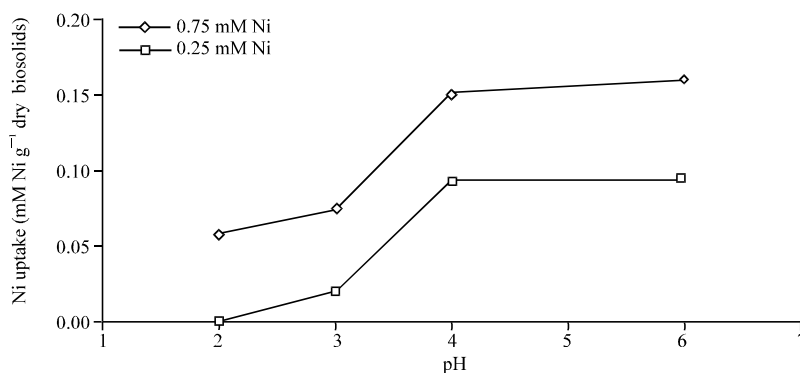


Fig. 2: The effect of pH on nickel adsorption at 0.25 and 0.75 mM concentrations

Norton *et al.* (2004) showed that in zinc biosorption by biosolids, pH 2 was suitable for zinc elution and with the increase of pH from 2 to 3, the adsorption rate increased exceedingly but afterwards (up to a pH of 6) the increase was insignificant.

Mameri *et al.* (1999) suggested that low pH could limit metal adsorption by biomaterial. The ion exchange between metal ions and hydronium ions of some constituent compounds in the biomaterial is believed to be involved in the biosorption mechanism (because at low pH there are more hydrogen ions). Different metals have different pH optima, due to the different solution chemistry of the metals (Macaskie and Dean, 1989). The low biosorption capacity at pH values below 4.0 was attributed to hydrogen ions competing with metal ions on the sorption sites (Huang *et al.*, 1991; Tsezos and Volesky 1981). In other words, at lower pH, due to the protonation of the binding sites resulting from a high concentration of protons, the negative charge intensity on the sites was reduced, resulting in the reduction or inhibition of the binding of metal ions (Kapoor *et al.*, 1999). In fact, most microbial surfaces are negatively charged because of the ionization of functional groups, thus contributing to their metal binding (Huang *et al.*, 1988; Hughes and Poole, 1989).

Biosorbent materials primarily contain weak acidic and basic functional groups. It follows from the theory of acid-based equilibria that, in the pH range of 2.5-5, the binding of heavy-metal cations is determined primarily by the state of dissociation of the weak acidic groups. Carboxyl groups (-COOH) are the most important groups for metal uptake by biological materials (Kratochvil and Volesky, 1988; Puranik and Paknikar, 1997). The ionic states of cell-wall functional groups can be used to explain the pH dependence of biosorption. Low-pH conditions allow hydrogen and hydronium ions to compete with zinc for metal binding sites on the biomass, causing poor zinc uptake. At higher pH values, there are lower numbers of competing hydrogen ions and more ligands exposed with negative charges, resulting in greater zinc sorption (Norton *et al.*, 2004).

**Effect of shaking rate on nickel uptake:** The investigation of the influence of the shaking rate on the biosorption capacity of the biomass showed that an optimum value was obtained at between 150 to 250 rpm (Fig. 3). This moderate shaking rate in gave the best homogeneity to the metal solution-biomass granule mixture.

Figure 3 shows that the effect of shaking was more significant with higher concentrations of nickel in solution. Here, the nickel intake reached a peak at a shaking rate of around 200 rpm for

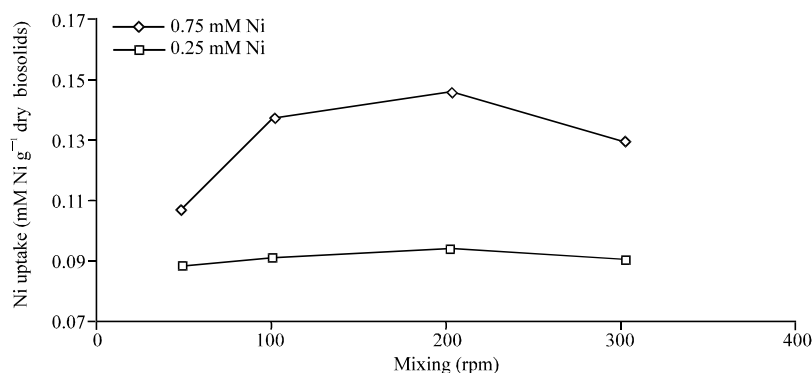


Fig. 3: The effect of shaking rate on nickel adsorption at 0.25 and 0.75 mM concentrations

both 0.25 and 0.75 mM solutions; however, the impact of shaking rate on adsorption in the 0.75 mM was higher, as observed from the slopes of the curves.

In studies on zinc adsorption by inactive *Streptomyces rimosus* bacteria, Mameri *et al.* (1999) showed that 250 rpm was the optimal shaking rate for zinc adsorption by 3 g of *Streptomyces rimosus* biomass at a pH of 6.5 at 20°C and a concentration of 100 mg L<sup>-1</sup>. Norton *et al.* (2004) chose the optimal shaking rate of 200 rpm in their studies on zinc adsorption by biosolids.

In his studies on nickel adsorption by sewage ash, Weng (2002) selected the optimal shaking rate as 170 rpm.

The reason why adsorption decreased at lower shaking rates is the biomass granulates agglomerated and took much more time to reach equilibrium. These results could be attributed to experimental conditions and to a too-short contact time; at higher speeds, the vortex phenomenon was encountered (Mameri *et al.*, 1999).

**Adsorption isotherm modeling studies:** The Langmuir adsorption model was used to correlate the isotherm data obtained at constant solution pH values. The linear form of the Langmuir Eq. is:

$$\frac{C_{eq}}{q_{eq}} = \frac{K_d}{q_m} + \frac{1}{q_m} C_{eq} \quad (5)$$

where,  $C_{eq}$  is the equilibrium metal solution concentration,  $q_{eq}$  is the amount of metal adsorbed onto the powder of waste activated sludge at equilibrium,  $q_{max}$  is the Langmuir constant for the maximum metal uptake and  $K_d$  is also the Langmuir constant related to the energy or net enthalpy of adsorption by the Arrhenius equation (Yan and Viraraghavan, 2003; Chong and Volesky, 1995; AWWA, 1990; Liu *et al.*, 2004).

From this equation, the maximum nickel adsorption capacity ( $q_{max}$ ) was determined to be 0.195 mM g<sup>-1</sup> (11.442 mg g<sup>-1</sup>) with a Langmuir constant of 0.23796 mM L<sup>-1</sup> (13.9611 mg L<sup>-1</sup>). These values compare well with other data reported in the literature.

According to the results of Padmavathy (2008), the maximum capacity of nickel adsorption in the Langmuir model is 0.167 mM g<sup>-1</sup> by Bakers yeasts.

Hammami *et al.* (2007) reported a maximum nickel uptake capacity of 0.15 mM g<sup>-1</sup> (Langmuir model) using the activated sludge.

According to the results of Gupta *et al.* (2010), the maximum capacity of nickel adsorption in the Langmuir model was 0.697 mM g<sup>-1</sup> by *Oedogonium hatei* algae.

Ozer and Ozer, (2003) reported the maximum nickel uptake capacity of adsorption in Langmuir model was 46.33 mg g<sup>-1</sup> by *Sacaromicos servici* fungus.

## CONCLUSION

The following conclusions can be drawn from the laboratory experiments:

- The kinetic experiments showed that the metal-uptake equilibrium took 2 h for nickel at concentrations of 0.25 and 0.75 mM and its adsorption kinetics followed a pseudo-second-order model



- The increase of pH from 2 to 3 increased the adsorption insignificantly, whereas the increase of pH from 3 to 4 enlarged the adsorption exceptionally and also at pH values over 4, the increase in adsorption was not significant, therefore, the maximum nickel adsorption was at a pH slightly over 4 and under pH 2 would be suitable for metal elution
- The optimum shaking rate for nickel adsorption was 150-250 rpm at 0.25 and 0.75 mM concentrations
- The adsorption capacity of the dry powder of activated sludge was  $0.195 \text{ mM g}^{-1}$  ( $11.442 \text{ mg g}^{-1}$ ) under Langmuir adsorption isotherm model. This compares favorably with other biosorbents. It indicates that the potential for further study on the use of powder of waste activated sludge is for the biosorption of nickel and other metals

### ACKNOWLEDGEMENTS

The financial support (project No: ENV-1-83123) of the Iran Water Resources Management Co. (under the Ministry of Energy) is gratefully acknowledged. The authors would also like to thank the laboratory experts of the Environmental faculty of the University of Tehran for their contributions to this study.

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