

The removal of toxic metals from liquid effluents by ion exchange resins. Part V: Nickel(II)/H⁺/Dowex C400

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ABSTRACT: The cationic exchange resin Dowex C400 was used to remove nickel(II) from aqueous solutions of different pH values and under various experimental conditions: stirring speed of the aqueous solution/resin system, temperature, resin dosage and aqueous ionic strength. The selectivity of the resin was investigated against the presence of various metals in the aqueous solution, and the removal of nickel(II) from aqueous solutions was also compared with results obtained using multiwalled carbon nanotubes or functionalized (carboxylic groups) multiwalled carbon nanotubes as adsorbents. According to batch experimental data, best fit of the results is obtained with the Freundlich model, whereas the ion exchange process is best explained by the pseudo-first order model. Experimental data fit well to the moving boundary controlled model. Elution of the nickel(II) loaded onto Dowex C400 resin is fully possible using acidic solutions.

KEYWORDS: Dowex C400; Liquid effluents; Multiwalled carbon nanotubes; Nickel(II); Removal

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RESUMEN: *La eliminación de metales tóxicos presentes en efluentes líquidos mediante resinas de cambio iónico. Parte V: níquel(II)/H⁺/Dowex C400.* Se ha empleado la resina de intercambio catiónico Dowex C400 en la eliminación de níquel(II) de disoluciones acuosas de distintos valores de pH y en varias condiciones experimentales: velocidad de agitación del sistema acuoso/resina, temperatura, dosificación de la resina y disoluciones acuosas de distinta fuerza iónica, investigándose la eliminación del níquel de medios acuosos que contenían varios metales, así como las posibilidades de la resina frente a la utilización de otros potenciales adsorbentes como son los nanotubos de carbono de pared múltiple y los nanotubos de carbono de pared múltiple funcionalizados con grupos carboxílicos. Los resultados experimentales indican que la carga del níquel(II) en la resina responde al modelo de Freundlich, mientras que los modelos cinéticos y de control indican que el proceso de intercambio catiónico responde al modelo de pseudo-primer orden y núcleo recesivo. La elución del níquel(II) se realiza con disoluciones acidas.

PALABRAS CLAVE: Dowex C400; Efluentes líquidos; Eliminación; Níquel(II); Nanotubos de carbono de pared múltiple

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1. INTRODUCTION

Though nickel is considered a hazardous element, there is not a clear evidence that soluble nickel salts alone are responsible for cancer, or whether when they appeared in conjunction with insoluble nickel form. In fact, this element does not appear in the list of harmful elements in primary and secondary drinking water standards (USEPA, 2017).

The overall consideration of nickel toxicology has lead to a recent number of reports about the removal of this metal from aqueous solutions using different technologies (El-Bahy and El-Bahy, 2016; Guan *et al.*, 2016; Jain *et al.*, 2016; Melo *et al.*, 2016; Otremska and Gega, 2016; Taha *et al.*, 2017; Yousef *et al.*, 2016; Zhang and Chen, 2016; Moghbeli *et al.*, 2017). Next in the series of articles published by the author about the use of ion exchange technology in the removal of toxic metals (Alguacil, 2002; Alguacil *et al.*, 2002; Alguacil, 2003; Alguacil, 2017), the present work reports about the use of the cationic exchange resin Dowex C400 on the removal of nickel(II) from aqueous solutions and under various experimental conditions, and different mathematical models were used to fit the various aspects in relation of the cationic exchange process.

2. EXPERIMENTAL

Dowex C400 (Fluka) having the characteristics shown in Table 1 were used to investigate the removal of nickel(II) from solutions. Other chemical used in the investigation were of AR grade, whereas the experimental procedure used here was the same as described in previous works (Alguacil, 2002; Alguacil *et al.*, 2002; Alguacil, 2003; Alguacil 2017). The multiwalled carbon nanotubes presented the characteristics described elsewhere (Alguacil *et al.*, 2016), whereas the carboxylic functionalized multiwalled carbon nanotubes were obtained from the Aldrich catalogue, ref. 755125.

All the loading experiments were carried out using 200 mL of the aqueous solution, 800 rpm and 20 °C, except when these variables were investigated.

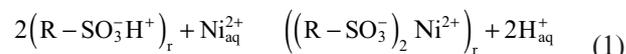
Metals in solution were analysed by AAS spectrometry, whereas metals load in the resin was calculated by the mass balance.

3. RESULTS AND DISCUSSION

3.1. Nickel loading onto the resin

Being Dowex C400 a cationic exchanger, in which the sulfonic group is the active one, the equilibrium

which describes the removal of nickel(II) from the solutions in the 0-5 pH range is represented by (Eq. (1)):



where, R is the non-reactive part of the resin, and r and aq represented the resin and the aqueous solution, respectively.

In batch ion exchange-adsorption experiments, the stirring speed could play a key-role in the performance of the system, since the correct stirring speed allows to gain a maximum in the metal loaded in the corresponding solid resin-adsorbent; however, this variable is often neglected in many scientific reports (AlOmar *et al.*, 2017; Drasinac *et al.*, 2017; Kim and Kwak, 2017; Ogden *et al.*, 2017; Wang and Liu, 2017). In the present work, aqueous solutions containing 0.01 gL⁻¹ Ni(II) at pH 5 was put into contact with 0.5 gL⁻¹ of the resin, and the system was stirred at different stirring speeds ranging 200-1000 rpm. Results obtained from this set of experiments are shown in Fig. 1. The maximum in adsorption loading is reached in the 800-1000 rpm range, and thus, the thickness of the aqueous solution nickel layer reached a minimum and a maximum in nickel(II) uptake is accomplished.

The influence of the temperature on metal load onto the resin was investigated using a resin dose of 0.25 gL⁻¹ and aqueous solution of 0.01 gL⁻¹ Ni(II) at pH 5. The results from Table 2 showed that the values of the nickel distribution coefficient, defined as (Eq. (2)):

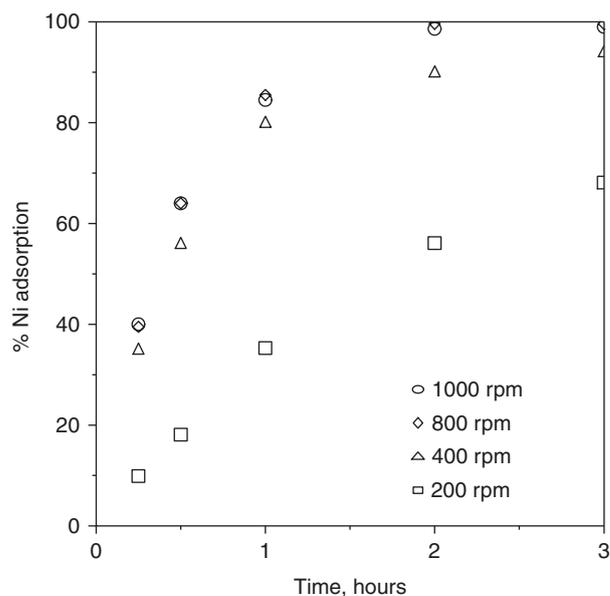


FIGURA 1. Influence of stirring speed on nickel loaded onto the resin.

TABLE 1. Characteristics of Dowex C400 resin

| | |
|-----------------------------|---------------------------------|
| Active group | Sulfonic in H ⁺ form |
| Matrix | Crosslinked styrene-DVB |
| Particle form and mean size | Spheres, 410 μm |

TABLE 2. Influence of the temperature on nickel(II) loading^a

| T (°C) | D (Lg-1) | log D |
|--------|----------|-------|
| 20 | 156 | 2.2 |
| 40 | 794 | 2.9 |
| 60 | 3996 | 3.6 |

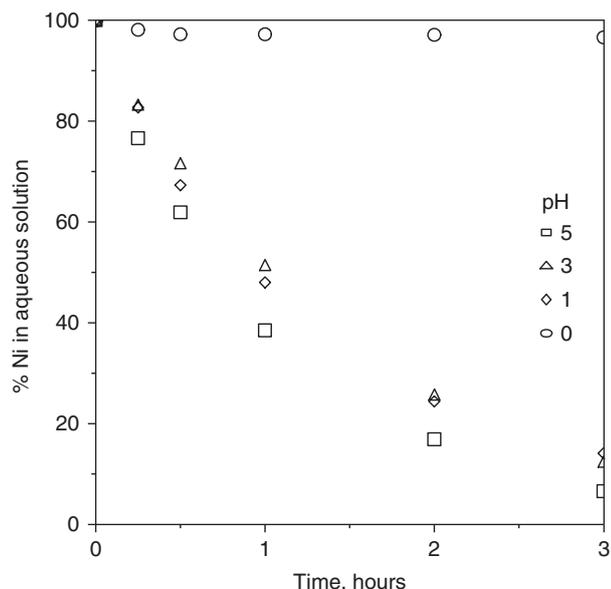
^aTime: 5 h

FIGURA 2. Influence of the aqueous pH on nickel removal from solution.

$$D = \frac{[\text{Ni}]_r}{[\text{Ni}]_{\text{aq}}} \quad (2)$$

where $[\text{Ni}]_r$ and $[\text{Ni}]_{\text{aq}}$ represented the nickel(II) concentrations at equilibrium in the resin and in the aqueous solution, respectively, increases as the operational temperature increases.

Thermodynamic parameters were estimated using conventional relationships, indicating that the reaction is endothermic (ΔH° 65 kJmol⁻¹), and ΔS° and ΔG° are 111 kJmol⁻¹K and 33 kJmol⁻¹, respectively. The positive ΔG° value indicated that the exchange process is not spontaneous, and the also positive ΔS° value is an indication of increasing randomness at the liquid-resin interface during the exchange process.

The influence of the aqueous pH, ranging 0-5, on metal load onto the resin was investigated using 0.25 gL⁻¹ of resin and an aqueous solution containing 0.01 gL⁻¹ Ni(II). As can be seen from Fig. 2 and Table 3, the pH of the aqueous solution has little influence in the metal uptake in the pH range 1-3, though some decrease in the metal load can be observed as the pH shifted to more acidic

TABLE 3. Influence of pH on metal uptake

| pH | Ni(II) (mgL ⁻¹) | Ni(II) (mg.g ⁻¹) |
|----|-----------------------------|------------------------------|
| 5 | 0.25 | 39.0 |
| 3 | 0.4 | 38.4 |
| 1 | 0.5 | 38.0 |
| 0 | 9.6 | 1.6 |

Resin: 0.25 gL⁻¹

TABLE 4. Influence of resin dosage on metal uptake

| Resin (gL ⁻¹) | [Ni(II)] (mgL ⁻¹) | [Ni(II)] (mg.g ⁻¹) |
|---------------------------|-------------------------------|--------------------------------|
| 0.5 | 0.03 | 19.9 |
| 0.25 | 0.25 | 67.2 |
| 0.13 | 1.6 | 67.2 |
| 0.05 | 4.5 | 110.0 |

TABLE 5. Metal uptake at various aqueous ionic strengths

| I (M) | ^a Ni(II) uptake (%) | ^a Ni(II) uptake, (mg.g ⁻¹) |
|-------|--------------------------------|---|
| 1 | - | - |
| 0.5 | 0.9 | 0.37 |
| 0.25 | 15.0 | 6.0 |
| 0.13 | 40.5 | 16.2 |
| 0 | 93.4 | 37.4 |

^aAfter 3 h

values. However, this nickel uptake completely falls off when the pH of the solution reached values near 0, although this behaviour will be useful (see below) when the nickel(II) elution is considered.

The influence of the resin dosage on nickel uptake onto the resin was also investigated, when the resin dosage was changed between 0.5 and 0.05 gL⁻¹, and the results obtained are summarized in Table 4. As it is expected, nickel adsorption increases with the resin dosage until reaching, under the present experimental conditions, a metal uptake in the 110 mg.g⁻¹ range.

The aqueous ionic strength is another experimental variable which could influence the metal loading onto the resins, and its effect on the present system was investigated by the use of different concentrations of lithium chloride in the aqueous solution. After 3 h of contact between 0.25 gL⁻¹ resin and an aqueous solution containing 0.01 gL⁻¹ Ni(II), the results presented in Table 5 showed a continuous decrease in the percentage of nickel uptake, or nickel concentration in the resin, as the aqueous ionic strength is increased, being this a point to consider when *real* solutions need to be treated since they have normally ionic strengths greater than zero.

It is important to remark that when *real* solutions are considered is that very often the targeted metal is accompanied by a miriade of other metals, thus, the selectivity of the present system is next investigated

TABLE 6. Performance of Dowex C400 resin in binary solutions at 1:1 molar metals relationship

| System | Elements pair | D (L g ⁻¹) | $\beta_{Ni/M}$ |
|--------|---------------|------------------------|----------------|
| 1 | Ni(II) | 196 | 0.99 |
| | Mn(II) | 198 | |
| 2 | Ni(II) | 196 | 0.98 |
| | Cu(II) | 199 | |
| 3 | Ni(II) | 196 | 2.6 |
| | Co(II) | 75.9 | |
| 4 | Ni(II) | 196 | 8.7 |
| | Zn(II) | 22.6 | |
| 5 | Ni(II) | 196 | <0.005 |
| | In(III) | >40000 | |

Aqueous solution: 0.17 mmolL⁻¹ each metal at pH 5. Resin dosage: 0.25 gL⁻¹. Temperature: 20 °C. Time: 5 h

using binary solutions containing nickel(II) and another metal in equimolar concentrations. Values of metal distribution coefficients (see Eq. (2)) and separation factors β , calculated as in Eq. (3), are shown in Table 6:

$$\beta = \frac{D_{Ni}}{D_{Metal}} \quad (3)$$

It can be seen from this Table 6, that the separation of nickel(II) from the accompanying metals, $\beta > 1$, is only possible in the case of zinc(II) and cobalt(II), being manganese(II) and copper(II) more or less equally loaded onto the resin than nickel(II) (not separation possible here), whereas indium(III) is by far loaded preferably to nickel ($\beta < 1$). It is worth to mention here that in all the experiments using binary solutions, the loading curve versus time for nickel(II), as represented in Fig. 3, always responded to the same pattern and with practically the same quantitative results for each time, which also corresponded to the data obtained when only nickel(II) is present in the aqueous solution (nickel uptake of 39 mg.g⁻¹ at equilibrium versus 39.2 mg.g⁻¹ for binary solutions) this means that apparently the population or crowding effect showed in other separations technology, i.e. liquid membranes, did not appear here (Alonso *et al.*, 2006).

It is worth to brief mention here, that when the population or crowding effect, in a given system, appeared, it means that the value of the metal uptake of a target element in, i.e. the binary solution, decreased in comparison with the same value obtained when the given element is alone in solution.

The performance of Dowex C400 was compared with results obtained in the removal of nickel(II) by other smart adsorbents like multiwalled carbon nanotubes (MWCN) and carboxylic functionalized

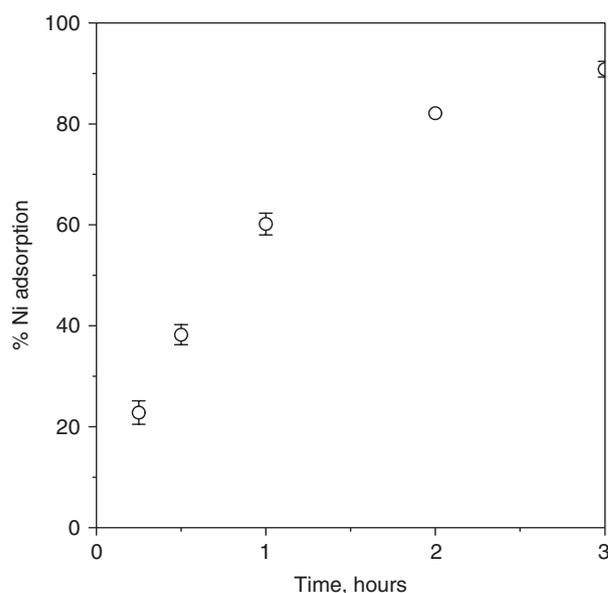


FIGURA 3. Nickel(II) loaded onto the resin from binary solutions.

TABLE 7. Comparison of nickel exchanger-adsorbents

| Exchanger-adsorbent | Ni uptake (mg g ⁻¹) |
|---------------------|---------------------------------|
| Dowex C400 | 19.9 |
| MCN | <1.0 |
| CMCN | 4.4 |

Aqueous solution: 0.01 gL⁻¹ Ni(II) at pH 5. Resin or carbon nanotubes dosage: 0.5 gL⁻¹. Temperature: 20 °C. Time: 5 h

multiwalled carbon nanotubes (C-MWCN) are. Results for these series of experiments are shown in Table 7. At the present experimental conditions, the resin has a better performance, in terms of nickel removal from the aqueous solution, than either type of carbon nanotubes tested in this work.

The adsorption data were used to examine their fit to the Langmuir or Freundlich models (Alguacil *et al.*, 2016). The results of these tests show that the adsorption is best fit to the Freundlich model (r^2 : 0.993):

$$\ln[Ni]_{r,e} = \ln K_f + \frac{1}{n} \ln[Ni]_{aq,e} \quad (4)$$

with $1/n$ 0.33 and $\ln K_f$ 4.1. In the above equation, $[Ni]_{r,e}$ and $[Ni]_{aq,e}$ represented the nickel concentrations at equilibrium in the resin and in the aqueous solution, respectively, whereas K_f is a Freundlich constant.

Adsorption or exchange kinetics was also evaluated. The pseudo-first and pseudo-second order rate equations, Eqs. (5 and 6), respectively:

TABLE 8. Results of the numerical calculations of the adsorption kinetics models.

| Model | r ² | Constant rate |
|---------------------|----------------|--|
| Pseudo-first order | 0.993 | k ₁ = 1.4x10 ⁻² min ⁻¹ |
| Pseudo-second order | 0.991 | k ₂ = 4.9x10 ⁻⁴ g mg ⁻¹ min ⁻¹ |

$$\ln([Ni]_{r,e} - [Ni]_{r,t}) = \ln[Ni]_{r,e} - k_1 t \quad (5)$$

$$\frac{t}{[Ni]_{r,t}} = \frac{1}{k_2 [Ni]_{r,e}^2} + \frac{t}{[Ni]_{r,e}} \quad (6)$$

were used to estimate their fit to the experimental data. In the above equations, $[Ni]_{r,e}$ represented the nickel concentration in the resin at equilibrium, $[Ni]_{r,t}$ represented the metal concentration in the resin at an elapsed time, t is the elapsed time and k_1 and k_2 are the respective rate constant for each model. Numerical results, Table 8, showed that the kinetics is slightly best explained within the pseudo-first order model.

Moreover, the exchange process was evaluated by the use of the models described elsewhere (López Díaz-Pavón *et al.*, 2014). Results of the experimental data fitting to the models, showed that the rate law governing (best fitting) the nickel exchange within the resin is a liquid-solid phase reaction which includes a moving boundary process described by:

$$3 - 3(1 - F)^{2/3} - 2F = kt \quad (7)$$

with rate constant k 3.5x10⁻³ min⁻¹ and r^2 0.988. In the above equation, the fractional attainment to the equilibrium, F , was calculated by the next relationship:

$$F = \frac{[Ni]_{r,t}}{[Ni]_{r,e}} \quad (8)$$

where, $[Ni]_{r,t}$ and $[Ni]_{r,e}$ are the nickel concentrations in the resin at and elapsed time t and at equilibrium, respectively.

3.2. Metal elution from the loaded resin

As it is mentioned above, and since nickel is poorly loaded onto the resin at pH values in the zero range, it seemed logical the use of mineral acids to accomplish both the nickel elution from the resin and the resin regeneration in the

TABLE 9. Results of elution experiments

| Eluant | Resin (mg) | Solution volume (mL) | Ni(II) eluted (%) | Ni(II) (mg L ⁻¹) |
|-----------------------------------|------------|----------------------|-------------------|------------------------------|
| HCl 1M | 0.1 | 25 | 91.3 | 56.4 |
| H ₂ SO ₄ 1M | 0.1 | 25 | 98.5 | 60.9 |
| H ₂ SO ₄ 1M | 0.1 | 12.5 | >99 | 123-124 |

Time: 1 h

same step. Results obtained from these series of experiments are summarized in Table 9. It can be seen, that, under the same experimental conditions, a slightly best percentage of nickel elution is obtained when sulphuric acid solution is used as eluant, whereas the change in the amount of resin/volume of eluant solution also slightly influenced the percentage of nickel recovered from the loaded resin (98.5% versus >99%). These results lead to obtain aqueous solutions with concentrations, at least, one magnitude order greater than the original aqueous feed (0.1 gL⁻¹ in the eluted solution versus 0.01 gL⁻¹ in the original feed). Beside the elution of nickel, the use of these acidic solutions result in the resin regeneration by shifting the equilibrium shown in Eq. (1) to the left.

4. CONCLUSIONS

- The removal of nickel(II) from aqueous solutions, in the 1-5 pH range, can be successfully done using the resin Dowex C400, however, at zero pH this removal falls off, the increase of the aqueous ionic strength also decreases the metal uptake onto the resin. The endothermic exchange process (between H⁺ from the resin and Ni²⁺ from the solution) is best explained by the moving boundary model, being the kinetics best explained by the pseudo-first order kinetic rate.
- From binary solutions, nickel(II) can be separate from cobalt(II) and zinc(II), almost equally uptake onto the resin than manganese(II) and copper(II), whereas indium(III) is best loaded onto the resin than nickel. In any case, the uptake of nickel onto the resin is independent of the accompanying metal in the solution and also almost equal to that when only nickel is present in this solution (at 0.25 gL⁻¹ resin dosage, nickel uptake of 39.2 mg.g⁻¹ from binary solutions versus 39 mg.g⁻¹ for nickel monoelmental solution).
- With respect to nickel removal from solutions, the performance of Dowex C400 resin is better than that of the multiwalled carbon nanotubes or carboxylic functionalized multiwalled carbon nanotubes.

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