The removal of toxic metals from liquid effluents by ion exchange resins. Part XV: Iron(II)/H⁺/Lewatit TP208

Francisco José Alguacil

Centro Nacional de Investigaciones Metalurgicas (CENIM, CSIC), Avda. Gregorio del Amo 8, 28040 Madrid, Spain (Corresponding author: fjalgua@cenim.csic.es)

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ABSTRACT: Iron(II) was removed from aqueous solutions by the use of the cationic ion exchange resin Lewatit TP208 (Na⁺ form). The influence of different experimental variables was investigated on the ion exchange process: stirring speed, temperature, pH of the solution and resin dosage. At stirring speeds included in the 300-1200 min⁻¹, iron(II) uptake onto the resin fitted well to the film-diffusion model, whereas the increase of the temperature, from 20 to 60 °C, was accompanied by an increase in the metal loaded onto the resin, thus, resulting in an endothermic ion exchange process. This ion exchange process was influenced by the variation of the pH of the solution: there was a decrease in metal uptake when the pH of the solution was shifted towards more acidic values. Also the ion exchange process was sensitive towards the variation of Lewatit TP208 resin dosage, being the process associated to the Freundlich isotherm model. This variation in the resin dosage had also an influence in the fitting of the experimental data, at various resin dosages, with the kinetics model associated to them. At the highest resin dosage (1 g·L⁻¹) the exchange process fitted well (r²= 0.999) to the pseudo-second order model, however, at the lowest resin dosage (0.13 g·L⁻¹), the process fitted (r²= 0.997) the first-order model. Metal uptake was compared with that yielded with other cation-exchange resins and also against the use of multiwalled carbon nanotubes. Iron(II) loaded onto the resin was eluted using acidic solutions.

KEYWORDS: Iron(II); Lewatit TP208; Liquid effluents; Multiwalled carbon nanotubes; Removal

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RESUMEN: La eliminación de metales tóxicos presentes en efluentes líquidos mediante resinas de cambio iónico. Parte XV: Iron(II)/H⁺/Lewatit TP208. Se ha investigado el uso de la resina de intercambio catiónico Lewatit TP208 en la eliminación de hierro(II) de disoluciones acuosas. En esta eliminación, se han considerado varias variables experimentales, incluyendo, la velocidad de agitación aplicada al sistema, la temperatura, el pH de la disolución acuosa y la dosificación de la resina. Para velocidades de agitación incluidas en el intervalo 300-1200 min⁻¹, el proceso de intercambio catiónico responde al modelo de difusión en la disolución acuosa. El aumento de la temperatura, entre 20 y 60 °C, va asociado a un aumento de la concentración de hierro(II) cargado en la resina, por lo que el sistema tiene un carácter endotérmico. La variación del pH del medio acuoso hacia pH ácidos (desde 5 a 1) da lugar a una disminución en la carga del metal en la resina. Asimismo, la dosificación de la resina Lewatit TP208 afecta a la eliminación del hierro(II) de la disolución acuosa, los resultados experimentales se ajustan a la isoterma de Freundlich. Los modelos cinéticos asociados a este sistema dependen también de la dosificación de la resina: usando la concentración de resina más alta (1 g·L⁻¹) los datos experimentales se ajustan ($r^2= 0,999$) al modelo de pseudo-segundo orden, mientras que con la concentración de resina más baja (0,13 g·L⁻¹) el modelo cinético es el primer-orden ($r^2=0,997$). Los resultados experimentales obtenidos con la resina Lewatit TP208 se han comparado con los obtenidos con otras resinas de intercambio catiónico y con el uso de nanotubos de carbono de pared múltiple. El hierro(II), cargado en la resina, puede ser eluido mediante el uso de disoluciones ácidas.

PALABRAS CLAVE: Efluentes líquidos; Eliminación; Hierro(II); Lewatit TP208; Nanotubos de carbono de pared múltiple

ORCID ID: Francisco José Alguacil (https://orcid.org/0000-0002-0247-3384)

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1. INTRODUCTION

The removal of toxic-hazardous metallic species presents in wastewaters, liquid effluents and, in general, aqueous environments is of the upmost importance due to its social impact and its implications on human health and the environment. Iron is one of these hazardous metals. In aqueous streams, iron can be present as iron(II), iron(III) or a mixture of both, and while both are essential for life, an excess in their ingesta (hyperferremia) is often accompanied with the increase of the formation of reactive oxygen species in cells, increasing, as a consequence of the above, the risk of cancer, heart disease, haemochromatosis; besides, iron accumulation in the central nervous system is connected with other serious illnesses.

The excess of the presence of iron(II) and (III) ions in the body comes from their use in agriculture and industry, that eventually ends into contaminated rivers, ponds, lakes, etc.

The presence of iron species in waters also increased the risk of undesirable bacterial growth within waterworks and water distribution system, resulting in the deposition of a slimy coating on the pippin system. The presence of iron in drinking-water is normally under 0.3 mg·L⁻¹, but may be higher in communities where different iron salts are used as coagulating agents in water-treatment plants and where iron-bearing materials are used in the civil engineering associated with water distribution. Despite all the above, no health-based guideline value for iron is proposed (WHO, 2020).

The removal of iron(II) from aqueous solutions had been also of interest for various research groups, and particularly, several ion exchange resins and/or adsorbents were used recently in this role, i.e. 732-type strong acid cation exchange resin, in an ascorbic acid and EDTA medium, effectively loaded Fe(II) at acidic pH values (Zhou *et al.*, 2018), multiwalled carbon nanotubes modified with EDTA, and also containing amino and car-

boxyl groups removed Fe(II) from aqueous solutions (Desouky, 2018), whereas nanosized calcium deficient hydroxyapatite was other material used in the removal of this element from solutions (Van Dat et al., 2019). MTS9100, C107E, MTS9570 and MTS9501, TP214, MTS9301 ion exchange resins were used in the removal of iron(II) (and other metals) from solutions (Bezzina et al., 2019; Bezzina et al., 2020), in the case of these resins, the acid present in the aqueous solution had a definitive role in the removal (or not) of the various metal ions investigated. δ -MnO₃/zeolite nanocomposites were used to simultaneously remove Fe²⁺, Mn²⁺, and NH₄⁺-N from groundwater (Ma et al., 2019). The commercially available Purolite S957 resin was other cationic exchange resin used in the removal of Fe(II) from solutions (Moghimi et al., 2020), in this case, the investigation was oriented to the treatment of copper electrolytes. The effect of the presence of Fe(II) or Fe(III) on the removal of Cu(II) by a chelating resins was also investigated (Botelho Junior et al., 2020), in this investigation, both batch and column operational modes were used.

Next on the series (Alguacil *et al.*, 2002; Alguacil, 2002; Alguacil, 2003; Alguacil, 2017a; Alguacil, 2017b; Alguacil, 2018a; Alguacil, 2018b; Alguacil and Escudero, 2018; Alguacil, 2019a; Alguacil, 2019b; Alguacil, 2019c; Alguacil and Escudero, 2020; Alguacil, 2020a; Alguacil, 2020b), in the present work, the ion exchange process of Fe^{2+} from aqueous solutions of various pH using Lewatit TP208 cationic exchange resin is investigated. Experimental variables influencing Fe^{2+} uptake onto the resin are investigated, and the resin performance is evaluated against other ion exchange resins and multiwalled carbon nanotubes. Elution of the metal, from Fe^{2+} -loaded resin, by HCl solutions is also presented.

2. EXPERIMENTAL

Lewatit TP208 resin (Fluka) has a crosslinked styrene-DVB matrix, containing sulfonic groups in

Na⁺-form, and having the form of spherical beds of 410 μ m mean size. Other cationic exchange resins used in the present work were purchased from the Fluka catalogue. The characteristics of the multi-walled carbon nanotubes (Sigma-Aldrich) used in the work were given elsewhere (Alguacil *et al.*, 2016; Alguacil *et al.*, 2017). All the other chemicals used in this investigation were of AR grade.

Iron(II) uptake and elution experiments were carried out by the standard procedure mentioned in other works of this series, whereas iron(II) concentration in the aqueous solutions was analyzed by AAS, and the metal uptake onto the resin was calculated by the mass balance.

3. RESULTS AND DISCUSSION

3.1. Iron(II) uptake onto the resin

Figure 1 showed the speciation of iron(II) in aqueous solutions against the pH values of this phase. It can be seen, that in solution, iron(II) existed as the predominant species, from pH 0 to pH vales near 8, in the form of the Fe^{2+} cation. The same speciation characteristics were obtained when the counter-anions were chloride or nitrate.



FIGURE 1. Iron(II) speciation in the solution as a function of the pH value (Puigdomenech, 2020).

Thus, in the 0-8 pH range, iron(II) was loaded onto the resin by the next equilibrium:

$$2(R - SO_{3}Na)_{r} + Fe_{aq}^{2+} \Leftrightarrow (R - SO_{3}^{-})_{2}Fe_{r}^{2+} + 2Na_{aq}^{+}$$
(1)

where R was the non-exchangeable part of the resin, and the subscript aq and r represented the aqueous and resin phases, respectively. Accordingly, with the above, Fe^{2+} uptake onto the resin responded to a cationic exchange reaction.

3.1.1. Influence of the stirring speed

To achieve effective loading of Fe^{2+} onto the resin, it is convenient to investigate the effect of the stirring speed applied on the system and its influence on the metal uptake. In the present investigation, stirring of the system was carried out from 300 to 1200 min⁻¹, and results derived from this experimentation showed that maximum metal uptake at the equilibrium (32 mg·g⁻¹) was not influence by the stirring speed. These results indicated that the aqueous boundary layer become constant and a minimum in the above range of stirring speeds. However, the stirring speed influenced the time in which the system reached the equilibrium (Fig. 2), in this figure the y-axe is the fractional achievement of the equilibrium, defined as:

$$F = \frac{\left[Fe\right]_{r,t}}{\left[Fe\right]_{r,e}}$$
(2)



FIGURE 2. Variation of F with time: Aqueous phase: 0.01 g·L⁻¹ Fe(II) at pH 5; Resin dosage: 0.25 g·L⁻¹; Temperature: 20 °C.

where $[Fe]_{r,t}$ and $[Fe]_{aq,t}$ are the iron concentration in the resin at an elapsed time and at the equilibrium, respectively. From this figure it can be observed that from 600 to 1200 min⁻¹ the equilibrium was reached after approximately 180 min, whereas in the case of 300 min⁻¹, this state was reached after 300 min.

From the above results, it was derived that the rate law associated with the load of iron(II) onto the resin best fitted to the film-diffusion controlled model (Table 1). The expression of such model was described elsewhere (Lopez Diaz-Pavon, 2014):

$$\ln(1-F) = -kt \tag{3}$$

in the expression, F was the fractional achievement of the equilibrium (Eq. (2)) and k was the rate constant.

Stirring speed, min-1	r ²	k, min ⁻¹
300	0.966	0.01
600-1200	0.994	0.03

 TABLE 1. Fitting of the film-diffusion controlled model to the experimental results

3.1.2. Influence of the temperature

Using aqueous solutions of 0.01 g·L⁻¹ Fe²⁺ at pH 3, and resin dosages of 0.13 g·L⁻¹, the influence of the temperature (20-60 °C) on Fe²⁺ uptake onto the resin was investigated. In these series of experiments, the reaction time was of 5 h, and the stirring speed of 1000 min⁻¹. Experimental results indicated that the increase of the temperature increased the metal loading onto the resin from 9.6 mg·g⁻¹ at 20 °C to 30 mg·g⁻¹ at 60 °C. If the metal distribution coefficient between the resin and the aqueous phases was estimated as:

$$D = \frac{[Fe]_{r,e}}{[Fe]_{aq,e}}$$
(4)

in this range of temperature, iron(II) uptake onto the resin was endothermic with ΔH° = 31 kJ·mol⁻¹. The values of other thermodynamic values estimated for the exchange process were ΔG° = 1 kJ·mol⁻¹ and ΔS° = 103 J·mol⁻¹ K⁻¹; thus, the Fe²⁺ uptake was non-spontaneous and with increasing randomness. In Eq. (4), [Fe]_{r,e} had the same significance that in Eq. (2), and [Fe]_{aque} was the corresponding metal concentration in the aqueous solution at the equilibrium.

3.1.3. Influence of the aqueous pH value

To assess the role of the aqueous solution pH on Fe^{2+} uptake onto the resin, the variation of this variable in the range of 1 to 5 was carried out using aqueous phases of 0.01 g·L⁻¹ Fe(II) at these various pH values and resin doses in the 0.13-1 g·L⁻¹. It is clear from Fig. 3 that the metal uptake decreases with a decrease in pH from 5 to 1. The decrease of the metal uptake onto the resin with the decrease of the pH value can be attributed to the interaction of H⁺ and Fe²⁺ with the active sites of the resin, and their respective affinity to be exchanged with the Na⁺ ions of the active groups of the resin

3.1.4. Influence of resin dosage

To investigate the effect of the resin dosage on Fe^{2+} uptake onto the cationic exchanger, several experiments were performed at pH 5. The results (Table 2) show that the percentage of Fe^{2+} removal from the solution increases when increasing the resin dosage. These results can be attributed to that increasing the cationic exchanger dosage large number of exchange sites for metal uptake is provided, resulting the above in an increase in the percentage

of iron(II) uptake onto the resin.

TABLE 2. Influence of the resin dosage on the Fe^{2+} uptake

Resin dosage, g·L ⁻¹	% Fe ²⁺ removal	[Fe] _{r,e} , mg·g ⁻¹	[Fe] _{aq,e} , mg∙L ⁻¹
0.13	65	52	3.5
0.25	80	32	2
0.5	85	17	1.5
1	90	9	1

Aqueous phase: 0.01 g·L⁻¹ Fe²⁺ at pH 5; Temperature: 20 °C; Stirring speed 1000 rpm; Time: 5 h

Experimental data best fitted ($r^2 = 0.964$) to the Freundlich isotherm model (Frohlich *et al.*, 2019):

$$\ln[Fe]_{r,e} = \ln K + \frac{1}{n} \ln[Fe]_{aq,e}$$
(5)

with ln K= 2.3 and 1/n of 1.4. The Freundlich model may be indicative that the exchange process is performed on heterogeneous surfaces, and since n<1, the exchange process is undesirable.

The kinetics models associated to the present system were evaluated using the two extreme resin dosages, 0.13 and 1 g·L⁻¹, used in this investigation. The results from this fit were shown in Table 3. At the lower resin dosage, the kinetics best responded to the first-order kinetic model (Wust *el al.*, 1999):

$$\ln[Fe]_{aq,t} = \ln[Fe]_{aq,0} - kt$$
(6)

TABLE 3. Fit of the kinetic models to the experimental values

Resin dosage, g·L ⁻¹	Model	r ²	k
0.13	first-order	0.997	5.6x10 ⁻³ min ⁻¹
1	pseudo-second order	0.999	7x10 ⁻³ g·mg ⁻¹ min ⁻¹

being $[Fe]_{aq,0}$ and $[Fe]_{aq,t}$ the iron concentrations in the solution at the initial and at elapsed time, respectively. From the model, it is obtained that $[Fe]_{aq,0}$ is 9.97 mg·L⁻¹, value which fits notably well with the experimental of 10 mg·L⁻¹.

At the highest resin dosage (1 g·L⁻¹), the experimental data fitted to the pseudo-second order kinetic model (Hao *el al.*, 2017):

$$\frac{t}{[Fe]_{r,t}} = \frac{1}{k[Fe]_{r,e}^2} + \frac{1}{[Fe]_{r,e}}t$$
(7)

being $[Fe]_{r,e}$ and $[Fe]_{r,t}$, defined as in eq. (2). In this case, the value of $[Fe]_{r,e}$ was 10 mg·g⁻¹ which was practically the same that the experimental value found as 9 mg·g⁻¹.

3.2. Iron(II) uptake onto different resins and multiwalled carbon nanotubes

The performance of Lewatit TP-208 resin on Fe(II) uptake was compared against other cationic exchange resins and also when multiwalled carbon nanotubes are used to remove Fe²⁺ from the aqueous solution. Table 4 resumed the results derived from the investigation. It can be seen, that higher Fe²⁺ uptakes were obtained with resins containing sulfonic acid groups (H⁺ or Na⁺ form) as active exchange sites. In the case of multiwalled carbon nanotubes, iron(II) uptake onto the adsorbent is 4.5 times lower than the metal loaded onto TP-208 resin.

 TABLE 4.
 Fe(II) uptake onto various resins and multiwalled carbon nanotubes (MWCNT)

Exchanger/ adsorbent	Functional group	[Fe] _{r or MWCNT, e} ? mg·g ⁻¹
Lewatit K2621	sulfonic group, H ⁺	33
Amberlite 200	sulfonic group, H ⁺	38
Lewatit SP112	sulfonic group, Na ⁺	33
Lewatit OC1026	DEHPA	6
Lewatit TP260	phosphonic acid, Na ⁺	8
Lewatit TP208	iminodiacetic acid, Na+	4
Lewatit TP 208	iminodiacetic acid, Na+	9ª
MWCNT	none	2ª

Aqueous phase: 0.01 g·L⁻¹ Fe(II) at pH 2 except ^aat pH 5; Resin dosage: 0.25 g·L⁻¹; ^aLewatit TP208 and MWCNT dosage: 1 g·L⁻¹; Temperature: 20 °C; Stirring speed: 1000 min⁻¹; Time: 5 h

3.3. Fe²⁺ elution

Since as one can be seen from Fig. 3, the percentage of iron(II) loaded onto the resin decreased as the value of the pH of the aqueous solution decreased, the elution of iron(II) loaded onto the resin was investigated using acidic solutions. Thus, a 1 M HCl solution was used as eluent on the resin loaded with 16 $mg \cdot g^{-1}$ Fe(II), HCl was chosen as acid medium due to the possibility of



FIGURE 3. Influence of the pH on the percentage of Fe^{2+} removal from the solution. Temperature: 20 °C; Stirring speed: 1000 rpm; Time: 5 h.

the reuse of the solutions to yield an end-product of pharmaceutical interest.

The results from this investigation were summarized in Table 5. From them, it can be seen that the variation in the volume of solution (V_s) /resin weight relationship had little influence on the percentage of Fe(II) elution (averaging 75%), however, the iron(II) concentration in the resulting solution can be increased more than ten times with respect to the initial feed solution. The resin can be regenerated to its Na⁺ form by washing it with NaOH solutions.

TABLE 5. Fe(II) elution from loaded resin

V _s /resin weight, mL·g ⁻¹	% Fe(II) elution	[Fe] _{ac} , mg·L ⁻¹
400	74	29
200	72	58
100	79	126
	51	

Temperature: 20 °C; Time: 0.5 h

4. CONCLUSIONS

Lewatit TP208 resin removes Fe²⁺ from aqueous solutions by a cationic exchange reaction releasing Na⁺ ions to the liquid waste; however, metal uptake onto the resin is pH-dependent. The stirring speed applied on the system has not any influence on the iron(II) concentration load, in the resin, at the equilibrium, but influences the time to achieve it. In the 300-1200 min⁻¹ range, the rate law associated to the exchange process is best represented by the film-diffusion controlled model. The increase of the temperature increases the percentage of iron(II) loaded onto the resin, resulting in an endothermic process (ΔH° = 31 kJ·mol⁻¹). The increase in the resin dosage results in a decrease of the metal concentration load onto the resin, but the equilibrium iron(II) concentration, in the corresponding solution, decreases as the resin dosage increases. The kinetic models associated to the exchange process are dependent of the resin dosage:

i) at the lowest resin dosage of $0.13 \text{ g}\cdot\text{L}^{-1}$, the process responded to the first-order kinetic model,

ii) at the highest resin dosage of 1 $g \cdot L^{-1}$, the process responded to the pseudo-second kinetic model.

- The exchange process is best represented by the Freundlich isotherm model, being the kinetics. At acidic conditions of pH 2, resins containing sulfonic groups present better iron(II) removal results than TP208 resin, though at pH 5 this resin presents best results with respect to the famed multiwalled carbon nanotubes.
- Iron(II) elution from loaded resin is accomplished using hydrochloric acid.

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