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

Francisco José Alguacil

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

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ABSTRACT: Lewatit TP208 cationic exchange resin was used to remove iron(III) from aqueous solutions under different experimental variables: stirring speed applied to the system, aqueous pH and resin dosage, temperature and metal concentration in the aqueous solution. Maximum metal uptake was achieved around 900 min⁻¹, and the exchange process was dependent both on the variation of the aqueous pH value and the resin dosage. The increase of the temperature was accompanied by an increase of iron(III) uptake onto the resin, thus, demonstrating the endothermic nature of the ion exchange process. Also the percentage of iron(III) removed from the solution strongly depended on the initial iron(III) concentration in the aqueous feed solution. Iron(III) uptake onto the resin was investigated in the presence of other metal ions: Cu(II), Zn(II), Cr(III) and In(III) in the feed solution, and it was also compared with that of Fe(II). This comparison was extended to that about the performance of this resin against that of multiwalled carbon nanotubes with respect to Fe(III) uptake. This metal ion loaded onto the resin can be eluted using acidic solutions.

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

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RESUMEN: La eliminación de metales tóxicos presentes en efluentes líquidos mediante resinas de cambio iónico. Parte XVI: Iron(III)/H⁺/Lewatit TP208. Se ha empleado la resina de cambio iónico Lewatit TP208 en la eliminación de Fe(III) de disoluciones acuosas bajo distintas condiciones experimentales: velocidad de agitación aplicada al sistema, pH del medio acuoso y dosificación de la resina, temperatura y concentración de hierro(III) en la disolución acuosa. Se obtiene un máximo en la carga de hierro(III) en la resina empleando una velocidad de agitación de 900 min⁻¹, siendo este proceso de carga del metal dependiente del valor del pH dela disolución acuosa y de la dosificación de la resina. El aumento de la temperatura da lugar a una disminución de la carga de hierro(III) en la resina, resultando en un proceso endotérmico. La eliminación de Fe(III) de la disolución acuosa también depende da la concentración inicial de este elemento en la misma. Se ha investigado el proceso de carga de Fe(III) en la resina en presencia (disoluciones binarias) de otros elementos metálicos en la disolución: Cu(II), Zn(II), Cr(III), In(III), y también se ha comparado con la carga de Fe(II) en la resina Lewatit TP208. Asimismo, se compara el uso de la resina con el empleo de nanotubos de carbono de pared múltiple en la eliminación de Fe(III) de la disolución acuosa. El hierro(III) cargado en la resina se puede eluir empleando disoluciones ácidas.

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

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

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1. INTRODUCTION

Iron(III) is another metallic element that usually accompanied to Mankind, and as others elements it is considered both essential and toxic to human life. Iron is an essential micronutrient and of importance for most life forms and is widely used in a variety of different proteins to carry out various functions (Geissler and Singh, 2011). The hazardousness of iron is demonstrated by its role in the development of diseases including cancer, ischemia, lung diseases, ageing as well as various neurodegenerative diseases (Recalcati et al., 2010). Moreover, iron can induce cell death by generating free radicals as it interconverts between Fe²⁺ and Fe³⁺ forms (Nagla et al., 2017). The limited solubility of iron, especially iron(III) is a challenge as well as an advantage since it limits its toxicity.

However, a major iron related environmental health problem to humans, and much more common than iron toxicity, is iron-deficiency because of inadequate amounts of available iron in the diet (USEPA, 2004), leading to anemia due to a decreased production of functional hemoglobin.

Besides all the above issues in relation with iron, the presence of iron(III) in waters is accompanied by some characteristics as odor, taste and colour; its presence also causes corrosion and staining effects. It was established a secondary MCL (maximum contaminant level) of 0.3 mg L⁻¹ (USEPA, 2020), being noticeable effects above this value on the three waters characteristics mentioned previously; as an example, Fig. 1 shows water contaminated by iron(III) in CENIM-CSIC premises.



FIGURE 1. Tap water contaminated with iron at D-building of CENIM-CSIC premises (January 2021).

Together with the above health issues, iron is often found in the processing of other metals from raw materials or secondary wastes (Caravaca *et al.*, 1994), and having little or non monetary value, its removal is also of importance in this field since its presence can be detrimental on the recovery of most valuable metals.

In the case of the presence of iron, as Fe³⁺, in liquid medium, several technologies are being using in the separation or elimination of the element from the aqueous solutions containing it. These technologies included solvent extraction with organic derivatives of phosphoric acid (Wang et al., 2020) or mixtures of alcohols and octadecanamide (Zhu et al., 2021), ion exchange using humic acids isolated from brown coals of Ekibastuz basin (Dauletbay et al., 2020) or ion exchange resins (El-Hamid et al., 2020; Zhang et al., 2020), adsorption onto zeolite and bentonite (Bakalár et al., 2020), natural or synthetic aluminosilicates (Flieger et al., 2020), and hydroxyapatite (Hamad et al., 2020), also solar technology was used to investigate its application on the removal of iron(III) from solutions (Arzate et al., 2020).

Following the series of works devoted to the removal of toxic metals from aqueous solutions (Table 1), the present manuscript investigates about the cationic exchange process between Fe^{3+} and Lewatit TP208 resin. Different variables influencing metal ion uptake onto the resin are investigated, and models describing this uptake are also presented. The resin performance is compared against that of multiwalled carbon nanotubes, and data on the competitive exchange process of Fe^{3+} and Cu^{2+} , Zn^{2+} , Cr^{3+} or In^{3+} are given. Elution of the metal can be accomplished using acidic solutions.

 TABLE 1. The series of investigations on the use of resins in the removal of toxic metals

Element	Resin	Reference
Cr(VI)	Dowex 1x8	Alguacil et al., 2002
Cd(II)	Lewatit TP260	Alguacil, 2002
Cu(II)	Amberlite 200	Alguacil, 2003
Cr(III)	Lewatit SP112	Alguacil, 2017a
Ni(II)	Dowex C400	Alguacil, 2017b
Mn(II)	Lewatit K2621	Alguacil, 2018a
Mn(VII)	Amberlite 958	Alguacil, 2018b
As(III)	Dowex 1x8	Alguacil and Escudero, 2018
Pb(II)	Amberlite 120	Alguacil, 2019a
Sb(III)	Ionac SR7	Alguacil, 2019b
Co(II)	Lewatit TP260	Alguacil, 2019c
Hg(II)	Lewatit SP112	Alguacil and Escudero, 2020
Zn(II)	Lewatit OC-1026	Alguacil, 2020a
In(III)	Dowex-400	Alguacil, 2020b
Fe(II)	Lewatit TP208	Alguacil, 2021

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. The characteristics of the multiwalled carbon nanotubes (MWCNTs, Sigma-Aldrich) used in the work are given elsewhere (Alguacil *et al.*, 2016; Alguacil *et al.*, 2017). All the other chemicals used in this work are of AR grade.

Iron(III), and metals, uptake onto the resin and iron elution experiments were carried out by the same procedure described in other investigations of these series; iron(III) and other metals concentrations in the aqueous solutions were analyzed by AAS, and the metal uptake onto the resin, or the carbon nanotubes, was calculated by the mass balance.

3. RESULTS AND DISCUSSION

3.1. Iron(III) uptake onto the resin

Being Lewatit TP208 a cationic exchange resin it is logical to attribute Fe^{3+} uptake onto the resin to the next equilibrium:

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

In the above equation, R represented the non-reactive part of the resin, and the subscript aq and r represented the aqueous and resin phases, respectively.

3.1.1. Influence of the stirring speed

Experiments performed to investigate the influence of the stirring speed on Fe(III) uptake onto the resin was first conducted using aqueous solutions of 0.01 g·L⁻¹ Fe(III) at pH 4 and resin dosage of 0.25 gL⁻¹, whereas the stirring speed was varied in the 600-1200 min⁻¹ range. It is worth to note here that previous experiments indicated that $0.01 \text{ g}\text{L}^{-1}$ iron(III) solutions at pH 4 were stable in the 4 h period. Results from this experiments were shown in Table 2, it can be seen that metal uptake increased progressively in the 600-900 min⁻¹ range, then becomes practically constant in the 900-1000 min⁻¹ range, and decreased at higher stirring speeds. The above results can be explained in terms that in the 600-900 min⁻¹ range, the increase of the stirring speed progressively decreased the thickness of the aqueous diffusion layer until it reached a minimum at 900-1000 min⁻¹, this minimum was accompanied by a maximum in iron(III) uptake onto the resin. At stirring speeds above 1000 min⁻¹, the decrease in the percentage of metal uptake can be explained by the formation of local equilibria between the resin

beads and the surrounding solution, resulting in a non-efficient mixing between the aqueous and resin phases.

TABLE 2. Influence of the stirring speed on Fe(III) uptake

Stirring speed, min-1	% Fe(III) uptake	
600	23	
800	45	
900	52	
1000	50	
1100	36	
1200	27	

Temperature: 20 °C. Time: 4 h

Processing of the experimental data, at 900 min⁻¹, demonstrated that the rate law governing Fe³⁺ up-take onto the resin is attributed ($r^2= 0.9917$) to the moving boundary process (Lopez Diaz-Pavon *et al.*, 2014):

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

where, F is the fractional approach to the equilibrium, defined as:

$$F = \frac{[Fe]_{r,t}}{[Fe]_{r,e}}$$
(3)

being $[Fe]_{r,t}$ and $[Fe]_{r,e}$ the iron(III) concentration in the resin at an elapsed time and at the equilibrium, respectively. The derived value of the rate constant k is $5x10^{-3}$ min⁻¹.

3.1.2. Influence of the aqueous pH and resin dosage

The variation of the aqueous pH on the iron(I-II) uptake was investigated using aqueous solutions of 0.01 gL⁻¹ Fe(III) at pH values in the 1-4 range and resin dosages in the 0.25-1 gL⁻¹ range. The results from these experiments were shown in Fig. 2 and Table 3. Figure 2 showed the variation in the percentage of Fe(III) uptake onto the resin at various resin dosages. It can be seen that this percentage decreased, for every resin dosage investigated, as the pH of the solution was shifted to more acidic values. Since there were not protons taking part in the exchange process (see Eq. (1)), this decrease was attributable to a competitive reaction between Fe³⁺ and H⁺ to bond or exchange with the active group of the resin.

Table 3 showed the Fe(III) equilibrium loading values resulting from the experiments, accordingly with that which was expected this loading decreased with the decrease of the pH value; however, and for every pH value, there was not appreciable variation in the metal uptake concentration using the



FIGURE 2. Percentage of Fe(III) uptake at various pH values and resin dosages. Temperature: 20 °C. Time: 4 h. Stirring speed: 900 min⁻¹

 TABLE 3. Fe(III) equilibrium loading concentrations (mg×g-1) at various resin dosages

pН	0.25 g·L ⁻¹	0.38 g·L ⁻¹	0.5 g [.] L ⁻¹	1 g·L-1
4	21	19	19	9.9
3	20	19	19	9.9
2	15	17	18	9.6
1	4.8	4.8	5.0	4.7

Experimental conditions as in Fig. 2

0.25-0.5 g·L⁻¹ resin dosage range, this almost near constant Fe(III) uptake concentration was extended, to the 0.25-1 g·L⁻¹ resin dosage range, at pH 1.

Experimental data were best fitted (Table 4) to the Langmuir Type-I model (Al-Ghamdi *et al.*, 2019):

 TABLE 4. Results of the Langmuir Type-I fit to the experimental data

pН	r^2	K, L·mg ⁻¹	$[Fe]_{r,m}$, mg·g-1	R _L
4	0.9982	8.3	21	0.012
3	0.9986	10	20	9.9x10 ⁻³
2	0.9905	345	16	3.0x10 ⁻⁴
1	0.9810	3.6	5	0.027

$$\frac{\left[\mathrm{Fe}\right]_{\mathrm{aq,e}}}{\left[\mathrm{Fe}\right]_{\mathrm{r,e}}} = \frac{1}{\left[\mathrm{Fe}\right]_{\mathrm{r,m}}K} + \frac{1}{\left[\mathrm{Fe}\right]_{\mathrm{r,m}}}\left[\mathrm{Fe}\right]_{\mathrm{aq,e}} \qquad (4)$$

in this equation, $[Fe]_{aq,e}$ was the iron concentration in the aqueous solution at the equilibrium, $[Fe]_{r,m}$ was

the maximum iron(III) concentration in the resin, and K the Langmuir constant.

The separation factor R_L associated to the Lagmuir equation was estimated accordingly to the next relationship:

$$R_{L} = \frac{1}{1 + K[Fe]_{aq,0}}$$
(5)

where $[Fe]_{aq,0}$ is the initial iron concentration in the aqueous solution. The values of R_L estimated for each pH are shown in Table 4, since they are included between zero and 1 values ($0 < R_L < 1$), in all the cases, the cationic exchange process is favourable.

3.1.3. Influence of the temperature on Fe(III) uptake

The variation of the temperature in the 20-60 °C was used to investigate the influence of this variable on the metal uptake onto the resin. In these experiments aqueous feed solutions of 0.01 g·L⁻¹ Fe(III) at pH 2 and resin dosages of 0.25 g·L⁻¹ were used. The contact time was of 4 h and the stirring speed of 900 min⁻¹. Results indicated that there was a continuous increase on the metal load onto the resin from 15 mg·g⁻¹ (38% uptake) at 20 °C to 21 mg·g⁻¹ (52% uptake) at 60 °C. Experimental results were fitted to the usual thermodynamic relationships to estimate the thermodynamic character of this cation exchange process. The iron(III) distribution coefficient between the resin and the aqueous solution was calculated accordingly to:

$$D_{Fe} = \frac{[Fe]_{r,e}}{[Fe]_{aq,e}}$$
(6)

where $[Fe]_{r,e}$ and $[Fe]_{aq,e}$ were the iron concentrations in the resin and in the aqueous solution at the equilibrium, respectively. A plot of log D_{Fe} versus 1/T resulted in a straight line ($r^2=1.0$) to calculate the enthalpy and entropy associated to the exchange process. The positive enthalpy value (12 kJ·mol⁻¹) was associated to an endothermic process, and its low value was consistent with the low energy characteristics of ion exchange processes. The positive entropy value (48 J·mol⁻¹K⁻¹) was an indication that the ion exchange process was associated with an increase of the process dissorded. The negative ΔG° value of -2 kJ·mol⁻¹ was an indication of the spontaneous nature of the system.

3.1.4. Influence of the initial metal concentration in the aqueous feed solution

These experiments were carried out using resin dosages of 0.5 gL^{-1} and aqueous solutions con-

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FIGURE 3. Dimensionless factorial approach to equilibrium (F) versus time at various initial iron(III) concentrations in the aqueous feed solution. Temperature: 20 °C. Stirring speed: 900 min⁻¹.

taining 0.005-0.08 g·L⁻¹ Fe(III) at pH 2. The results derived from the experiments were shown in Fig. 3, plotting the factorial approach to the equilibrium F versus time. These F values were calculated as in Eq. (3). From Fig. 3, it can be seen that there was little variation on the change of F value with time in the 0.01-0.08 g·L⁻¹ Fe(III) concentrations range, whereas with the lowest iron(III) concentration of 0.005 g·L⁻¹, the 50% of metal uptake was reached at shorter elapsed time (about 30 min) than with the higher iron(III) concentrations (near 60 min). This tendency was maintained for every elapsed time.

The kinetic equation associated to the Fe³⁺ uptake onto the resin was estimated using the two extreme metal concentrations, 0.005 and 0.08 g·L⁻¹, used in this investigation. At the lowest metal concentration, the results best responded (r^2 = 0.9977) to the pseudo-second order kinetic model (Mohagheghian *et al.*, 2017):

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

At the highest metal concentration (0.08 g·L⁻¹), the experimental data best fitted (r^2 = 0.9961) to the pseudo-first order kinetic model (Hamza *et al.*, 2019):

$$\ln\left(\left[Fe\right]_{r,e} - \left[Fe\right]_{r,t}\right) = \ln\left[Fe\right]_{r,e} - kt \qquad (8)$$

TABLE 5. Fit of the kinetic models to the experimental values

Metal concen- tration, g·L ⁻¹	Model	^ª [Fe] _{r,e} , mg∙g ⁻¹	k
0.005	pseudo-2nd order	11	2.5x10 ⁻³ g·mg ⁻¹ min ⁻¹
0.08	pseudo-1st order	48	0.011 min ⁻¹

^aExperimental values: 9.9 and 51 mg·g⁻¹

The results derived from both fits were summarized in Table 5. It also can be seen that the equilibrium iron(III) concentration in the resin, calculated from both models, can be compared reasonably well with the experimental data.

3.1.5. Iron(III) uptake using multiwalled carbon nanotubes: a comparison with resin results

The uptake results using the resin were compared with experiments carried out using multiwalled carbon nanotubes. In these series of experiments aqueous solutions of 0.01 g L⁻¹ Fe(III) at pH values of 2 or 4 were used, whereas resin or MWCNTs dosages were of 1 g L⁻¹. The results, summarized in Table 6, showed that iron(III)-loading onto Lewatit TP208 was better, at the two pH values investigated, than that of MWCNTs, and thus, that under the present experimental process, the cation exchange process

TABLE 6. Iron(III) uptake (mg·g·l) onto Lewatit TP208 orMWCNTs

	pH 2	pH 4
Lewatit TP208	9.8	9.9
MWCNTs	nil	4.2

Temperature: 20 °C. Time: 4 h. Stirring speed: 900 min⁻¹

was a more efficient process, in terms of iron(III) removal from aqueous wastes, than the adsorption process.

3.1.6. Iron(III) competitive uptake versus other cations

The performance of Lewatit TP208 resin, in terms of iron(III) uptake, was compared to that when the aqueous feed solution contained an accompanying cation. Thus, binary solutions containing 0.01 g·L⁻¹ Fe³⁺ and 0.01 g·L⁻¹ of Cu²⁺, Zn²⁺, Cr³⁺ or In³⁺ at pH 2 were put into contact with a resin dose of 0.25 g·L⁻¹. The results derived from the set of experiments were given in Table 7. The corresponding values of the

TABLE 7. Fe³⁺ competitive uptake onto the resin

System	[M] _{aq,e} , mmol [.] L ⁻¹	[M] _{r,e} , mmol [.] g ⁻¹	D _{Fe/M}	$m{\beta}_{Fe/M}$
$Fe^{3+}-Zn^{2+}$	0.11-0.12	0.26-0.14	2.4-1.2	2.0
Fe ³⁺ -Cu ²⁺	0.11-0.11	0.27-0.15	2.5-1.4	1.8
$Fe^{3+}-Cr^{3+}$	0.11-0.19	0.28-0.070	2.5-0.37	6.8
Fe ³⁺ -In ³⁺	0.11-0.045	0.29-0.17	2.6-3.8	0.68

Temperature: 20 °C. Time: 4 h. Stirring speed: 900 min⁻¹

distribution coefficients were calculated as in eq. (6), whereas the values of the separation factor, $\beta_{Fe/M}$, were calculated as:

$$\beta_{\rm Fe/M} = \frac{D_{\rm Fe}}{D_{\rm M}} \tag{9}$$

Thus, and from the results present in the above Table, it can be deducted that Fe^{3+} was loaded onto the resin, under the present experimental conditions, preferably to Cu^{2+} , Zn^{2+} and Cr^{3+} , and it can be separate from these elements, since $\beta_{Fe/M}$ values were greater than 1. In the case of In^{3+} , the experimental $\beta_{Fe/In}$ value indicated that Fe^{3+} can not be separate from In^{3+} , again under the present experimental conditions, but In^{3+} can be separate from Fe^{3+} , as the $\beta_{In/Fe}$ value of near 1.5 indicated.

 Fe^{3+} uptake was compared to that of Fe^{2+} uptake onto the resin, but in this case using monoelemental solutions. The aqueous feed phase contained 0.01 g L⁻¹ Fe³⁺ or Fe²⁺ at pH 2, and the resin dosage was of 0.25 g L⁻¹. The results indicated that Fe³⁺ uptake was greater than that of Fe²⁺, 15 mg g⁻¹ versus 4 mg g⁻¹, and thus, Fe³⁺ can be separate from Fe²⁺.

3.2. Iron(III) elution

Accordingly, with the results obtained in subsection 3.1.2., a shift of the aqueous pH value towards more acidic values leaded to a decrease in the percentage of iron(III) uptake onto the resin, thus, it was logical to use acid solutions as eluants for the present systems. The elution experiments were carried out using a resin dosage of $0.5 \text{ g } \text{L}^{-1}$ loaded with 7 mg·g⁻¹ of Fe³⁺, as elution solutions 1M sulphuric acid or hydrochloric acid were used. The results were showed in Table 8, and whereas the reaction

TABLE 8. Metal elution from Fe³⁺-loaded resin

Eluant	V _{aq} /W _r , mL∙mg ⁻¹	time, min	% elution	Fe(III) mg [.] L ^{.1}
1 M H ₂ SO ₄	200	15-60	52	18
1 M HCl	200	15-60	67	23
1 M HCl	400	15-60	82	14

Temperature: 20 °C. Stirring speed: 300 min⁻¹.

time had not effect on the elution yields, it can be seen that the HCl solution performed, in terms of the percentage of Fe^{3+} elution, better than the sulphuric acid one, also the increase of the volume of elution solution/resin weight (V_{aq}/W_r) relationship increased this percentage.

It is expected that these elution results will be improve under continuous implementation, i.e. columns. Once iron(III) is eluted, the resin is returned to its Na^+ form by washing it with the adequate NaOH solution

4. CONCLUSIONS

Iron(III), in the form of Fe³⁺ cation, can be remove from liquid solutions or effluents by the use of Lewatit TP208 resin. Maximum metal loading onto the resin is achieved at a stirring speed of 900 min⁻¹, and a minimum in the thickness of the aqueous boundary layer is reached. Fe³⁺ uptake onto Lewatit TP208 responded to a cation exchange reaction, which released Na⁺ cations to the aqueous phase, however, the metal loading process is pH-dependent, and this dependence is attributable to a competitive reaction between H⁺ and Fe³⁺ of the aqueous medium to link with the active group of the resin. Also, the percentage of metal uptake onto the resin is dependent on the resin dosage. The increase of the temperature leads to an increase of the percentage of Fe³⁺ uptake, thus resulting in an endothermic exchange process. The variation of the initial metal concentration in the aqueous solution also influences the percentage of metal uptake, decreasing this value as the initial iron(III) concentration in the solution increases from 0.005 to 0.08 g L⁻¹. By the use of Lewatit TP208 resin, Fe^{3+} can be separate from Cu^{2+} , Zn^{2+} and Cr^{3+} , but not from In^{3+} , which presented a distribution coefficient value higher than that of Fe^{3+} . Fe^{3+} is loaded onto the resin far better than Fe^{2+} . With respect to multiwalled carbon nanotubes, Fe^{3+} load onto the resin is higher, at pH values of 2-5, than that of the one yield by the carbon nanotubes. Fe³⁺ elution, from the metal-loaded resin, can be accomplished by the use of acidic solutions.

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