The removal of toxic metals from liquid effluents by ion exchange resins. Part XVIII: Vanadium(V)/H⁺/Amberlite 958

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ABSTRACT: The ion exchange resin Amberlite 958 was used to investigate its behaviour on the removal of hazardous vanadium(V) from aqueous media. This investigation was carried out under various hydrodynamic conditions and chemical conditions, such as variation of the stirring speed, variation of the pH of the aqueous solution, resin dosage, initial vanadium(V) in the aqueous solution, and temperature. Vanadium(V) uptake onto the resin was highly dependent on the pH of the solution, and thus, to the vanadium(V) speciation in this phase; the ion exchange process had an endothermic character. The experimental data, under different experimental conditions, were fitted to various models: kinetics (stirring speed), rate law (vanadium concentration) and model isotherms (resin dosage). Multiwalled carbon nanotubes were also investigated on vanadium(V) removal from the solution. Vanadium(V) loaded onto the resin can be eluted under acidic conditions.

KEYWORDS: Amberlite 958; Liquid effluents; Multiwalled carbon nanotubes; Removal; Vanadium(V)

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RESUMEN: La eliminación de metales tóxicos presentes en efluentes líquidos mediante resinas de cambio iónico. Parte XVIII: Vanadium(V)/H⁺/Amberlite 958. La resina de cambio iónico Amberlite 958 se ha empleado en la eliminación de vanadio(V) de disoluciones acuosas. La experimentación se ha llevado a cabo bajo distintas condiciones hidrodinámicas y químicas: variación de la velocidad de agitación, pH del medio acuoso, dosificación de la resina, concentración del metal y temperatura. La carga del metal en la resina depende del pH de la disolución acuosa, y por lo tanto, de las especies de vanadio(V) presentes en este medio, el proceso de intercambio iónico tiene un carácter endotérmico. La carga del metal en la resina se ha modelado empleando distintos modelos y condiciones experimentales: cinética de carga (velocidad de agitación), mecanismo de carga (concentración de vanadio) e isotermas de carga (dosificación de la resina). Asimismo, se ha investigado acerca del uso de nanotubos de carbono de pared múltiple en la eliminación de vanadio(V) del medio acuoso. El metal cargado en la resina puede ser eluido empleando disoluciones ácidas.

PALABRAS CLAVE: Amberlite 958; Efluentes líquidos; Eliminación; Nanotubos de carbono de pared múltiple; Vanadio(V)

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1. INTRODUCTION

Nowadays vanadium is used in many industries (steel-making, non-ferrous alloys, chemicals, aerospacial, catalysis); together the above, mining, smelting, and processing of ores containing this element are the main sources of vanadium in the environment. Also this metal is gaining more practical importance due to its use in vanadium redox flow batteries (Lee et al., 2021). However, the presence of this element in the environment, especially in its V oxidation state, is hazardous for life, producing several diseases when dietary intake exceeds certain values (Barceloux and Barceloux, 1999; Ma and Fu, 2009), and also being suspicious to be carcinogenic (Luz at al., 2018). Moreover, inorganic vanadium is considered more toxic than vanadium-bearing organic compounds (Ghosh et al., 2015).

The above makes of a necessity its removal from aqueous effluents, being, for this purpose, various hydrometallurgical technologies recently reviewed (Le and Lee, 2021) Most recent contributions aimed to the removal of this hazardous element from solutions included the use of ion exchange resins (Abhilasha et al., 2021; Morales et al., 2021) adsorbents (Aregay et al., 2021; Peng et al., 2021), ion exchange resin-activated carbon composites (Bao et al., 2021) and liquid-liquid extraction operation (Ju et al., 2021; Ying et al., 2021). Others suggested technologies included chemical precipitation, lectrokinetic remediation, photocatalysis reduction, coagulation, microbiological treatment, and membrane filtration. The advantages or disadvantages of each one of these operations are summarized elsewhere (Peng et al., 2022)

The present manuscript followed the series of investigations on the use of ion exchange resins in the treatment of solutions containing hazardous elements (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; Alguacil 2021a; Alguacil 2021b; Alguacil and Escudero, 2021). In the present investigation, Amberlite 958 resin was used to study its performance on the removal of vanadium(V), under various experimental conditions and compared some of the results with that obtained on the use of multiwalled carbon nanotubes as adsorbent for this element. The results derived from the ion exchange experiments were fitted to various models to explain the various aspects of the vanadium(V) uptake onto the resin. The elution of vanadium (V) loaded onto the resin under various acidic conditions was also investigated.

2. EXPERIMENTAL

Amberlite 958 (Fluka) had chloride ion as exchange anion, it was formed by a macroreticular acrylic copolymer of 0.67-0.87 mm particle size. Being an anion exchanger, this resin removes metal-anionic complexes from aqueous solutions accordingly to the general equilibrium, Eq. (1):

$$\mathbf{R} - \mathbf{Cl}_{\mathrm{r}}^{-} + \mathbf{X}_{\mathrm{aq}}^{-} \Leftrightarrow \mathbf{R} - \mathbf{X}_{\mathrm{r}}^{-} + \mathbf{Cl}_{\mathrm{aq}}^{-} \tag{1}$$

where R represented to the non-reactive part of the resin, X^{-} to the anionic metal complex, and the subscripts r and aq to the resin and aqueous phases, respectively.

Other chemicals used in the experimentation were of AR grade. Vanadium solutions were prepared from ammonium vanadate, whereas the multiwalled carbon nanotubes (MWCNTs) had the characteristics given elsewhere (Alguacil *et al.*, 2016).

Vanadium(V) loading batch experiments were carried out in a glass reactor vessel (250 mL), containing the corresponding metal aqueous solutions to which resin or MWCNTs dosages were added. Stirring was provided *via* a four blades glass impeller and pH values were adjusted with the appropriate HCl or NaOH solutions, and being measured by a Crison 506-pHmeter. Elution step was investigated on the same basis as above.

Vanadium was analysed in the various aqueous solutions by Inductively Coupled Plasma-Optical Emission Spectroscopy (ICP-OES), within 2% associated error, whereas metal loaded onto the resin or MWCNTs was calculated by the mass balance.

3. RESULTS AND DISCUSSION

3.1. Vanadium(V) uptake onto Amberlite 958

3.1.1. Influence of stirring speed

In order to achieve effective uptake of vanadium(V) on the resin, it was convenient to investigate the influence of the stirring speed on metal uptake. Diffusional resistances encountered during the metal uptake can had an influence on the metal load onto the resin, and also on the time to reach metal distribution equilibrium between the resin and the aqueous phase. In the present investigation, stirring of the aqueous phase-resin system was carried out from 600 to 1000 min⁻¹ (Fig. 1). The factorial approach to the equilibrium (F) was calculated as (Eq. 2):

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



FIGURE 1. Variation of F versus time at various stirring speeds. Aqueous phase: $0.01 \text{ g} \cdot \text{L}^{-1} \text{ V}(\text{V})$ at pH 4; Resin dosage: $1.25 \text{ g} \cdot \text{L}^{-1}$; Temperature: 20 °C.

indicate not appreciable variation in vanadium(V) uptake with the various stirring speeds. This indicated that the aqueous boundary layer was a minimum in this range of stirring speeds, and the resistance due to it was minimized. In the above Eq. (2), $[V]_{r,t}$ and $[V]_{r,e}$ represented the vanadium(V) concentrations in the resin at an elapsed time and at the equilibrium, respectively.

The kinetics order associated to vanadium(V) uptake onto the resin was modelled using different kinetics models: first order, second order, pseudo-first order and pseudo-second order, from this modelling process, it was found that the experimental data best fit (r^2 = 0.9963) to the pseudo-first order kinetic equation (Hemavathy *et al.*, 2019), Eq. (3):

$$\ln([V]_{r,e} - [V]_{r,t}) = \ln[V]_{r,e} - k_{psl}t \quad (3)$$

with k_{ps1} (constant rate) estimated as 0.026 min⁻¹, and $[V]_{r,e}$ of 7.2 mgV·g⁻¹ resin, value with matched well with the experimental value of 7.6 mgV·g⁻¹ resin.

3.1.2. Influence of temperature

The variation of temperature on vanadium(V) uptake was also investigated. In the present investigation, the resin dosage was of $0.16 \text{ g}\cdot\text{L}^{-1}$, whereas the aqueous solution had the same composition than above. The results from the investigation was resumed in Table 1, it can be observed

TABLE 1. Variation of vanadium(V) uptake with temperature

Т, °С	% V uptake	log D	
20	55	1.2	
40	62	1.0	
60	70	0.91	

Time: 3 h. Stirring speed: 800 min-1

that metal uptake increased with the increase of the temperature, being the thermodynamic values of ΔH° , ΔS° and ΔG° estimated from a plot of log D versus 1/T and the corresponding equations (Alguacil and Cobo, 1998; Alguacil *et al.*, 2002). From this plot, it was shown that the ion exchange process presented and endothermic character (ΔH° = 13 kJ·mol⁻¹). Other thermodynamics parameters derived from the experimental data and the convenient mathematical relationships indicated that ΔG° = -5 kJ·mol⁻¹, indicative of a spontaneous process, and ΔS° = 61 J/mol K, this positive value should be explained by an increase in the system randomness. In Table 1, D represented the vanadium distribution coefficient, calculated as:

$$\mathbf{D} = \frac{\left[\mathbf{V}\right]_{\mathrm{r,e}}}{\left[\mathbf{V}\right]_{\mathrm{aq,e}}} \tag{4}$$

where $[V]_{r,e}$ and $[V]_{aq,e}$ were the vanadium(V) concentrations, at the equilibrium, in the resin and in the aqueous solution, respectively.

3.1.3. Influence of the resin dosage and pH variation

The influence of these two variables on vanadium(V) removal from the aqueous solution was investigated using resin dosages varying from 0.15 g·L⁻¹ to 1.25 g·L⁻¹, and solutions containing 0.01 g·L⁻¹ V(V) at pH 1, 4, 7, and 11. The results from this set of experiments were resumed in Table 2. These results showed that in the 4-11 pH range the increase of the resin dosage resulted in a decrease of the vanadium(V) remaining in the solution; results showed in this same Table also demonstrated that vanadium(V) uptake onto the resin was also pH-dependent, though protons did not appear in the overall ion exchange mechanism showed in Eq. (1). This dependence should be attributable to the various vanadium(V) species presented in the aqueous solutions at the various pH values (Fig. 2). At pH 1, and being VO_2^+ the predominant species in the solution, it was logical that vanadium(V) was not loaded onto the resin due to the anion exchanger character of Amberlite 958 resin, and all the vanadium(V) remained in the equilibrated solution. From pH 4 to 11, the percentage of vanadium(V) in the equilibrated solution progressively increased. The electrostatic attractions between

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Resin dosage, g·L ⁻¹	pH 1	pH 4	pH 7	pH 11
0.15	100	45	48	76
0.30	100	23	30	49
0.65	100	14	22	38
1.25	100	5	12	27

 TABLE 2. Percentage of vanadium(V) remaining in the solution at various pH values and resin dosages

	Femperature:	20 °C;	Time: 3	3 h;	Stirring	speed:	800 mir	1 ⁻¹
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FIGURE 2. Vanadium(V) speciation in the aqueous phase (Puigdomenech, 2021).

the positively charged active groups of the resin and the anionic vanadium species favour vanadium loading onto the resin (Li *et al.*, 2020), also, it seemed that the uptake of polyvanadate species was favoured against the presence, at higher pH values, of mononuclear vanadium species in the solution. Within all the resin dosages tested, maximum vanadium(V) uptake was yielded at pH 4, being this result in accordance which it was described in the literature (Wołowicz and Hubicki, 2022).

Considering the above, vanadium uptake onto the resin can be expressed by the next equilibria, at pH 4:

$$\frac{5(R^{+}Cl^{-})_{r} + V_{10}O_{27}OH_{aq}^{5-} \Leftrightarrow}{R_{5}^{+}V_{10}O_{27}OH_{r}^{5-} + 5Cl_{aq}^{-}} (5)$$

at pH 7:

 $R^{+}Cl_{r}^{-} + VO_{2}(OH)_{2_{aq}}^{-} \Leftrightarrow R^{+}VO_{2}(OH)_{2_{r}}^{-} + Cl_{aq}^{-}$ (6)

and at pH 11:

$$2(R^{+}Cl^{-})_{r} + VO_{3}OH_{aq}^{2-} \Leftrightarrow R_{2}^{+}VO_{3}OH_{r}^{2-} + 2Cl_{aq}^{-}$$
(7)

At this pH 11, the decrease in vanadium uptake can be attributable to the lesser exchangeability of the VO₃OH²⁻ species, and competition of OH⁻, presented in the aqueous solution, to be loaded onto the resin (Zhang and Leiviskä, 2020):

$$R^+Cl_r^- + OH_{aq}^- \Leftrightarrow R^+OH_r^- + Cl_{aq}^-(8)$$

In eqs (5-8), R represented the non-exchangeable moiety of the resin, whereas subscripts r and aq represented to the equilibrated resin and aqueous phases.

At pH 4, the equilibrium data were presented in Table 3, these values were used to estimate the loading isotherm. The results of the various fittings indicated that at this pH value, vanadium(V) uptake was best represented (r^2 = 0.9679) by the linear form of the Freundlich isotherm (Elbadawy, 2019):

$$\ln[V]_{r,e} = \ln K_F + \frac{1}{n} \ln[V]_{aq,e} (9)$$

TABLE 3. Equilibrium values at pH 4

Resin dosage, g·L ⁻¹	$[V]_{aq,e}, mg \cdot L^{-1}$	[V] _{r,e} , mg V/g resin
1.25	0.5	7.6
0.65	1.4	13
0.30	2.3	26
0.15	4.5	37

Temperature: 20 °C; Time: 3 h; Stirring speed: 800 min⁻¹

being the values of $\ln K_F$ and 1/n of 2.49 and 0.74, respectively. K_F and n represented relative indicators of uptake capacity and the energy associated to this uptake or the uptake intensity of a given system, respectively. If 1/n relationship was less than 1, as it was in the present system, it was favoured the load of the solute onto the resin.

3.1.4. Influence of vanadium(V) concentration in the aqueous solution

In this case, solutions containing 0.01 or $0.1 \text{ g} \cdot \text{L}^{-1}$ vanadium(V) at pH 4 were put into contact with a resin dosage of $1.25 \text{ g} \cdot \text{L}^{-1}$. The results obtained from this set of experiments were shown in Fig. 3, where F (factorial approach to the equilibrium) values were calculated as in Eq. (2). It can be seen, that the increase of vanadium(V) concentration in the solution had an influence on the time to approach the equilibrium, i.e. at 0.01 g·L⁻¹ V(V) in the solution 80% (F= 0.8) of the equilibrium was reached within one hour of contact between the solution and the resin, whereas using the more concentrated metal solution, this value (80%) was reached within 1.5 hours of reaction. The experimental results were used to estimate the rate law associated to the metal uptake onto Amberlite 958 resin. The results from this fit indicated that, using both metal solutions, diffusion through the aque-



FIGURE 3. Variation of F versus time at two vanadium(V) concentrations in the aqueous solution. Temperature: 20 °C; Stirring speed: 800 min⁻¹.

ous layer dominated vanadium(V) load onto the resin (Lopez Diaz-Pavon *et al.*, 2014):

$$\ln(1-F) = -kt_{(10)}$$

with k (rate constant) values of 0.028 min⁻¹ ($r^2= 0.9931$) and 0.017 min⁻¹ ($r^2= 0.9699$) for the initial metal concentrations of 0.01 g·L⁻¹ and 0.1 g·L⁻¹, respectively.

3.1.5. Vanadium(V) uptake onto multiwalled carbon nanotubes: a comparison

The use of the resin in the removal of vanadium(V) from the solution was also compared with the use of multiwalled carbon nanotubes as adsorbent for the metal. In these experiments, the aqueous solution was of 0.01 g·L⁻¹ at pH 4, whereas the resin or the multiwalled carbon nanotubes dosages were of $0.3 \text{ g}\cdot\text{L}^{-1}$. Other experimental variables were 20 °C temperature, 800 min⁻¹ stirring speed, and 3 hours of equilibration time. The results indicated that when the adsorbent was used, metal load was of 12 mg V/g carbon nanotubes, whereas in the case of the resin this uptake was of near 26 mg V/g resin. Thus, at a first approach, it was apparent that, under the present experimental conditions, the resin was more effective than the carbon nanotubes on the metal uptake, and thus, in the removal of this hazardous metal from the solution.

3.2. Vanadium(V) elution

Metal elution from vanadium(V)-loaded resin was investigated using HCl solutions. This medium was chosen for two reasons: i) to promote the formation of a cationic vanadium(V) species which, as it was shown above, was not loaded onto the resin, and ii) to regenerate the resin to its initial chloride form. These elution experiments were carried out with resin loaded with 6.4 mg V/g resin, and the results were summarized in Table 4. These results showed that metal elution was more effective as the HCl concentration increased, and also when the volume of solution *versus* resin weight relationship increased, however, the use of lower relationships produced more concentrated vanadium solutions, up to 27 mg V/g resin under the present experimental conditions.

TABLE 4. Elution results at various experimental conditions

HCl, M	solution volume/resin weight, mL·g ⁻¹	% V elution
0.01	800	15
1	800	82
2	800	92
2	400	81
2	200	83

Resin: 0.25 g loaded with 1.6 mg V; Temperature: 20 °C; Time: 1 h

4. CONCLUSIONS

- Amberlite 958 resin was used to remove hazardous vanadium(V) from aqueous solutions, being this removal favoured at pH 4, and decreasing when the pH value raised from 4 to 11. At pH 1, and since this metal is present in the aqueous solution as a cation, the resin is unable to remove it from this medium.
- At pH 4, there is little variation in metal uptake onto the resin with the variation of the stirring speed in the 600-1000 min-1 range, thus the system reached a minimum in the thickness of the aqueous layer, and the system obeys the pseudo-first order equation model.
- The increase of temperature in the 20-60 °C range increased vanadium(V) uptake in the resin, having this ion exchange system an endothermic character.
- At pH 4, metal loading is best represented by the linear Freundlich isotherm, and being the 1/n value less than 1, the anion exchange process is considered as favourable.
- A ten fold increment of the initial vanadium(V) concentration in the aqueous phase produced, under a given time, a decrease in the percentage of metal uptake, however, the rate law governing the metal uptake is not dependent on this initial vanadium concentration in the solution, since the various vanadium-bearing solutions best fit to the aqueous diffusion model.
- Vanadium(V) can be eluted from the loading resin by the use of HCl solutions, regenerating the resin, to its original Cl- cycle, in the same process.

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