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The removal of toxic metals from liquid effluents by ion exchange resins. Part XVII: Arsenic(V)/H$^+$/Dowex 1x8

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ABSTRACT: The performance of anionic exchange resin Dowex 1x8 in the removal of arsenic(V) from aqueous solutions was investigated. Batch experimentation was carried out under different variables, including, the stirring speed applied on the system, the pH of the aqueous solution, resin dosage and temperature. Due to the characteristic speciation of arsenic(V) in aqueous phases, the removal of this element from the solution is negligible at highly acidic or alkaline pH values, but it is possible at the aqueous pH range of 4-9, thus, both $HAsO_4^{2-}$ and $H_2AsO_4^-$ species are loaded onto the resin. At the above pH range, arsenic(V) uptake is exothermic. Different models are fitted to the experimental values in order to gain knowledge about this ion exchange system: rate law, kinetics and solute loading onto the resin. This loading is compared against the yielded using non-functionalized multiwalled carbon nanotubes. The elution step is investigated using acidic solutions (HCl medium) as eluent, from the eluted solutions, arsenic(V) can be efficiently stabilized as ferric or calcium arsenates.

KEYWORDS: Arsenic(V); Dowex 1x8; 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 XVII: Arsénico(V)/H$^+$/Dowex 1x8.* Se ha investigado la eliminación de arsénico(V) de disoluciones acuosas mediate la resina de intercambio aniónico Dowex 1x8. Los experimentos se han llevado a cabo bajo diferentes condiciones experimentales como, velocidad de agotación, pH de la disolución acuosa, concentración de la resina y temperatura. Debido a la especiación del arsénico(V) en medio acuoso, la eliminación de este no es posible en medios de bajo o alto valor del pH, pero es posible a valores de pH comprendidos entre 4 y 9, por lo tanto, las especies $HAsO_4^{2-}$ y $H_2AsO_4^-$ son las responsables de la carga del elemento en la resina. En este margen, 4-9, de valores de pH, la eliminación del arsénico del medio acuoso tiene carácter exotérmico. Los datos experimentales se han adaptado a distintos modelos en relación con el mecanismo de la eliminación del arsénico de la disolución, cinética, y carga del elemento en la resina; asimismo, se ha comparado la carga del arsénico en la resina con la que se obtiene con el empleo de nanotubos de carbono de pared múltiple. La etapa de elución se ha investigado empleando disoluciones de ácido clorhídrico, de estos eluidos, el arsénico(V) se puede estabilizar precipitándolo como los arseniatos de hierro(III) o calcio.

PALABRAS CLAVE: Arsenico(V); Dowex 1x8; Efluentes líquidos; Eliminación; Nanotubos de carbono de pared múltiple

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1. INTRODUCTION

Among the elements considered as toxic for humans, arsenic, both in its (V) and (III) oxidation states and both forming part of inorganic or organic compounds, has a pre-eminent position. The toxicity of this element is due to its carcinogenic character, promoting skin, liver, lung and kidney various cancers (Monteiro De Oliveira *et al.*, 2021). Though there are a number of sites around the World affected by the presence of this hazardous element, its removal from landfills, waters, etc., is far to be completely finished, and still is of the upmost necessity in social, environmental and profitable terms.

The removal of arsenic(V) from waters, wastewaters, or generally speaking, aqueous solutions, is a topic of the utmost importance for the scientific community though there are few technologies proposed for this task (Alka *et al.*, 2021; Weerasundara and Bundschuh, 2021). Some recent investigations describe the use of micro-structured carbon-based (Islam *et al.*, 2021) and modified incinerated sewage sludge ash (Gao *et al.*, 2021) adsorbents; ion exchange using various resins (Dadakhanov *et al.*, 2021), resin-supported nano-hydrated zirconium oxide (Deng *et al.*, 2021), and an electrochemical-ion exchange hybrid method (Rathi *et al.*, 2021); various membrane processes (Moreira *et al.*, 2021; Sherugar *et al.*, 2021; Rojas-Challa *et al.*, 2021; Jarma *et al.*, 2021; Lopez *et al.*, 2021); and solvent extraction using a Triton X nonionic surfactant-choline chloride ionic liquid-water mixture (Silva *et al.*, 2021).

In this manuscript, and following the series of investigations proposed by the authors on the use of ion exchange resins in the treatment of solutions containing hazardous elements (Alguacil, 2003; Alguacil *et al.*, 2002a; Alguacil, 2002b; 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), Dowex 1x8 resin is used to investigate its performance in the removal of arsenic(V), from solutions of different pH values, and under various experimental conditions, with some of the results compared with that obtained multiwalled carbon nanotubes as adsorbent for the element. The ion exchange experimental data are fitted to various models to explain the arsenic(V) uptake onto the resin. From the As(V)-loaded resin, the elution step under various acidic conditions is investigated, and from the eluted solution the removal of arsenic(V), as the correspondent iron(III) or calcium(II) arsenates, is proposed.

2. EXPERIMENTAL

Dowex1x8 (Fluka) is a strong basic resin having the trimethylammonium cation, as active group, and being chloride the counteranion. It contained a matrix of styrene-divinylbenzene in the form of spherical beads, with particle size in the 50-100 mesh (150-300 µm) range. Other chemicals used in the experimental work are of AR grade, whereas the multiwalled carbon nanotubes (MWCNTs) have the characteristics given in a previous work (Alguacil *et al.*, 2016).

The experiments performed in batch form, both in arsenic uptake and elution operations, were carried out in a glass reactor vessel (250 mL), containing the arsenic(V) aqueous solution to which the corresponding resin/MWCNTs dosages were added. Stirring was provided *via* a four blades glass impeller at 800 min^{-1} and 20 ºC, except when these variables were investigated. pH values were adjusted with the appropriate HCl or NaOH solutions, and being measured by a Crison 506-pHmeter. In the case of the elution step and due to the lower solution volume used, the stirring speed was maintained at 300 min^{-1}, in order to avoid undesirable ejections from the reaction vessel.

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

3. RESULTS AND DISCUSSION

3.1. Arsenic(V) loading onto Dowex 1x8 resin

The variation of stirring speed was examined in order to investigate its influence on arsenic(V) uptake onto the resin. Experiments were carried out using aqueous solutions of 0.01 $g \cdot L^{-1}$ As(V) at pH values of 1, 4, 9, and 13, whereas the resin dosage was of 1 $g \cdot L^{-1}$. The results derived from these experiments were summarized in Table 1. It can be seen that the removal of As(V) from the solutions of pH 1 or 13 was non-existent. It is also observed

Table 1. Table 1. Influence of the stirring speed on As(V) uptake onto the resin

Stirring speed, min^{-1}	pH 1	pH 4	pH 9	pH 13
600	nil	30	90	nil
700	nil	30	94	nil
800	nil	30	95	nil
900	nil	30	93	nil
1000	nil	30	84	nil

Temperature: 20 °C, Time: 4 h, Values in % uptake

that, at pH 4, the variation of the stirring speed had not influence on the arsenic(V) uptake onto the resin, but reached a maximum in the 700-900 min^{-1} range when the experiments were carried out with solutions at pH 9. Moreover, after increasing the stirring speed at 1000 min^{-1} an evident decrease in arsenic(V) uptake was found; this result was consistent with the formation of local equilibria between the resin bead and the surrounded solution which decreased the arsenic(V) load onto the resin. These results indicated that, at pH 4 and in the whole range of the investigated stirring speeds, the system had reached a minimum in the thickness of the aqueous boundary layer and, thus, arsenic(V) maximized. The same behaviour, using the solution of pH 9 and in the 700-900 min^{-1} range, was found. From Table 1, it was noticed that the percentage of arsenic(V) onto the resin was by far greater at pH 9 than in the solution of pH 4.

Using the corresponding experimental data, the rate law governing the loading of As(V) onto the resin was determined by plotting (Fig. 1) the value of F versus time, being F defined as:

$$F = \frac{[As]_{r,t}}{[As]_{r,e}} \qquad (1)$$

In the above equation, $[As]_{r,t}$ and $[As]_{r,e}$ were the arsenic concentrations in the resin at an elapsed time and at the equilibrium, respectively. The results represented in Fig. 1 showed the apparent difference in the resin performance during arsenic(V) uptake at the various pH values. At pH 4, 80% (F= 0.8) of the corresponding maximum arsenic uptake was achieved in about 10 min, whereas at pH 9, this exchange rate was reached in about 50 min. Further, the fit of the experimental data to the various models indicated that, for both solutions at pH 4 or 9, the best fitting was to the aqueous diffusion model (Lopez Diaz-Pavon *et al.*, 2014):

$$\ln(1-F) = -kt \qquad (2)$$

with rate constants k of 0.21 min^{-1} (r^2= 0.9206) and 0.03 min^{-1} (r^2= 0.9833) in the case of the solution at pH 4 and pH 9, respectively.

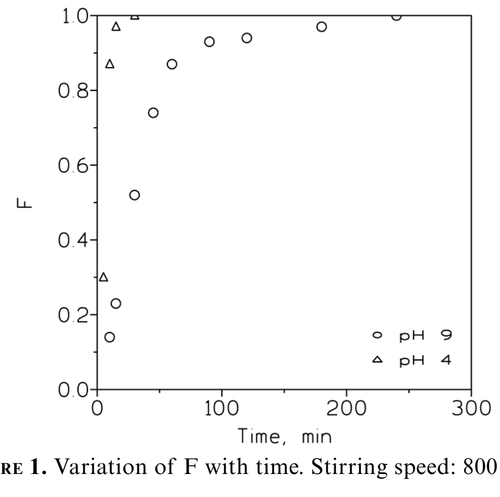

Figure 1. Variation of F with time. Stirring speed: 800 min^{-1}.

The influence of the temperature on arsenic(V) uptake onto the resin was also investigated; in this case, the experiments were performed on the solutions of pH 4 and 9 and the same resin dosage than above. The results derived from these series of experiments were shown in Table 2. It can be seen that, for the two solutions, the increase of the temperature produced a decrease in the arsenic(V) uptake onto the resin. Thermodynamic values were estimated from a plot of log D_{As} versus temperature and the slope (ΔH°) and intercept (ΔS°) of the expression:

$$\log D_{As} = \frac{\Delta S^{o}}{2.3R} - \frac{\Delta H^{o}}{2.3R}\frac{1}{T} \qquad (3)$$

where D_{As} represented to the arsenic distribution coefficient and R is the gas constant.

Table 2. Influence of temperature on As(V) uptake onto the resin

Temperature, °C	pH 4	pH 9
20	30^{a}	95^{a}
40	21^{a}	90^{a}
60	15^{a}	83^{a}
ΔH°, kJ·mol^{-1}	-18	-28
ΔS°, kJ·mol^{-1} K^{-1}	-6x10^{-2}	-6x10^{-2}
ΔG°, kJ·mol^{-1}	0.4	-9

Stirring speed: 800 min^{-1}, Time: 4 h, ^{a}Values in % uptake

The arsenic distribution coefficient is calculated as:

$$D_{As} = \frac{[As]_{r,e}}{[As]_{aq,e}} \qquad (4)$$

where $[As]_{r,e}$ and $[As]_{aq,e}$ are the arsenic concentrations in the equilibrium in the resin and in the aqueous solution, respectively.

The value of ΔG° was estimated by the next expression:

$$\Delta G^{o} = \Delta H^{o} - T\Delta S^{o} \qquad (5)$$

Thus, using both solutions the exchange reaction was associated to an exothermic process (negative change of enthalpy) and a non-spontaneous (positive ΔG°) or spontaneous (negative ΔG°) ion exchange process at pH 9 and pH 4, respectively. The negative value of ΔS° indicated a process which decreased its randomness.

As it was described from the data presented in Table 1, the removal of arsenic(V) from solutions at pH values of 1 or 13 was negligible, thus it can be ascertain that arsenic(V) uptake onto the resin was greatly dependent on the pH of the aqueous solution. Moreover, two defined zones of arsenic uptake can be described. One at pH 4 with a percentage of arsenic uptake onto the resin of 30%. The other at pH 9, in which the loading of this element reached about 95% and, thus, the arsenic(V) uptake onto Dowex 1x8 resin was associated to the exchange of chloride ions from the resin with the corresponding predominant arsenic(V) species within the above pH values. Then, at pH 4, the exchange reaction was:

$$R - Cl_r + H_2AsO^{-}_{4aq} \Leftrightarrow R - H_2AsO_{4r} + Cl^{-}_{aq} \qquad (6)$$

whereas at pH 9, the equilibrium associated to the anion exchange process was:

$$2(R - Cl)_r + HAsO^{2-}_{4aq} \Leftrightarrow R_2 - HAsO_{4r} + 2Cl^{-}_{aq} \qquad (7)$$

It was also apparent the preference of the resin towards the divalent anion (greater uptake) and against the monovalent arsenic species.

Studying the influence of the variation in resin dosage on arsenic(V) solutions, it was found that both, in the solutions of pH 4 and 9, the increase in the resin dosage decreased the arsenic concentration in the resin, but decreased the equilibrium arsenic concentration in the solution (Table 3). Experimental data were fitted to various isotherms models, and the results of these fitting indicated that at pH 4, the linear form of the Freundlich isotherm (Elbadawy, 2019) best represented the loading data:

$$\ln[As]_{r,e} = \ln K_F + \frac{1}{n}\ln[As]_{aq,e} \qquad (8)$$

This isotherm is an empirical model which assumed that the uptake surface sites are heterogeneous presenting a non-uniform distribution of heat of uptake over the resin surface.

Using the solution of pH 9, the best fit corresponded to the linear form of the Langmuir Type-1 isotherm (Ayawei *et al.*, 2017):

$$\frac{[As]_{aq,e}}{[As]_{r,e}} = \frac{1}{K_L[As]_{r,m}} + \frac{1}{[As]_{r,m}}[As]_{aq,e} \qquad (9)$$

where $[As]_{r,m}$ represented to the maximum concentration of arsenic loaded onto the resin. The Langmuir model or monolayer model is based on assumption that the exchange sites are homogenously distributed over the resin surface, there is not interaction between loaded molecules, and all the active sites have the same affinity for exchange of a single molecular layer, the values of the parameters corresponding to the two fittings were shown in Table 4. Furthermore, and taking into account the next relationship (Jamali and Akbari, 2021):

$$R_L = \frac{1}{1 + K_L[As]_{aq,0}} \qquad (10)$$

where $[As]_{aq,0}$ is the initial arsenic concentration in the aqueous solution, the value of R_L= 0.024 (separation factor) was derived, and thus, the ion exchange process was favourable.

Table 3. Influence of the resin dosage on As(V) equilibrium data

Resin dosage, g·L^{-1}	pH	$[As]_{r,e}$ mg·g^{-1}	$[As]_{aq,e}$, mg·L^{-1}
0.5	4	6.0	7.0
0.25	4	3.7	5.4
2.5	4	2.3	4.2
5	4	1.2	3.8
0.25	9	25.6	3.6
0.38	9	24.5	0.8
0.5	9	19.0	0.5
0.65	9	15.0	0.25
1.25	9	7.9	0.15

Aqueous phase: 0.01 g·L^{-1} As(V) at pH 4 or pH 9, Temperature: 20 °C, Time: 4 h, Stirring speed: 800 min^{-1}

Table 4. Parameters of the isotherms fitting

pH	Isotherm	r^2	K_F, mg·g^{-1}	1/n	K_L, L·mg^{-1}	$[As]_{r,m}$, mg·g^{-1}
4	Freundlich	0.9335	0.056	2.4		
9	Langmuir	0.9957			4	27

It was examined the variation in the arsenic(V) uptake onto the resin at various elapsed times, when resin dosage was of 0.5 g·L^{-1} or 1.25 g·L^{-1}. The results of these experiments were shown in Fig. 2, and they were used to estimate the fit of the data to several kinetics models. The results of the corresponding fittings were shown in Table 5, as it was indicated in this Table and using both arsenic-bearing solutions, the best fit corresponded to the first-order kinetic model (Wust *et al.*, 1999):

$$\ln[As]_{aq,t} = \ln[As]_{aq,0} - kt \qquad (11)$$

in the above equation, $[As]_{aq,0}$ and $[As]_{aq,t}$ represented to the arsenic concentrations, in the aqueous solution, at time zero and at elapsed time, respectively.

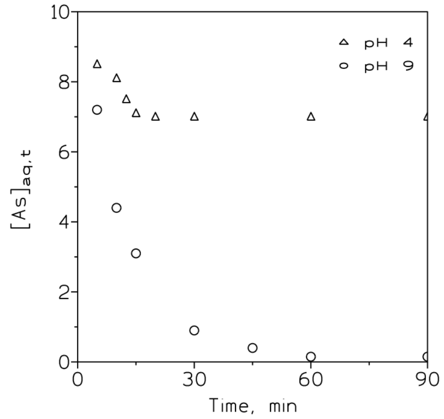

FIGURE 2. Variation of the arsenic concentration in the aqueous solution with the reaction time. Aqueous phase: 0.01 g·L-1 As(V) at pH 4 or pH 9; Resin dosage: 0.5 g·L^{-1} (pH 4) or 1.25 g·L^{-1} (pH 9); Temperature: 20° C; Stirring speed: 800 min^{-1}.

TABLE 5. Results of the fit to kinetic models

pH	model	r^2	K, min^{-1}	$[As]_{aq,0}$, mg·L^{-1}
4	first order	0.9162	0.0146	9.1
9	first order	0.9942	0.0695	8.9

The performance of Dowex 1x8 resin was compared to that of multiwalled carbon nanotubes. In these series of experiments aqueous solutions contained 0.01 g·L^{-1} As(V) at pH 1, 4, 9, or 13, whereas the resin or the MWCNTs dosages were of 1.25 g·L^{-1}, other experimental variables were temperature of 20° C, time of 4 hours and stirring speed of 800 min^{-1}. The results from these experiments showed that both and MWCNTs were ineffective in the removal of arsenic from solutions of pH 1 and 13, however, the same performance was found for the carbon nanotubes in the treatment of the solutions of pH 4 or 9, with the arsenic concentration in the respective aqueous solutions maintained fixed at 0.01 g·L^{-1}. As it was shown above, in these conditions of pH 4 and 9, the arsenic removal rate, using 1.25 g·L^{-1} resin dosage, was of 46% or 98.5%, respectively.

3.2. Arsenic(V) elution

Considering the resin performance with the pH values of the aqueous solutions, it was showed that arsenic(V) was not loaded onto the resin either at highly acidic or alkaline pH values, thus, the elution step was considered using solutions with these two extreme pH values. Table 6 summarized the results of these investigations; firstly, it can be seen that in alkaline medium the percentage of arsenic elution is very low, thus, this medium was not adequate for the recovery of arsenic(V) from the arsenic-loaded resin, against these results, acidic solutions in HCl medium were adequate for the removal of the element from the loaded resin. Using $HAsO_4^{2-}$ as the species loaded onto the resin (Eq. (7)), the elution process can be represented by the next equilibrium:

$$R_2 - HAsO_{4_r} + 2HCl_{aq} \Leftrightarrow 2R - Cl_r + H_3AsO_{4_{aq}} \quad (12)$$

At the same time that arsenic was recovered and concentrated in the eluate, the resin was regenerated.

3.3. Post-treatment of the As-bearing eluate

After the elution stage, the arsenic(V) from the concentrated eluate can be recovered for safe dumping by precipitation with iron(III) or calcium(II) salts, to form the corresponding iron(III) or calcium(II) arsenates but in amorphous form. The various conditions for such precipitations were sufficiently described in the literature (Navarro *et al.*, 2004; De Klerk *et al.*, 2015; Doerfelt *et al.*, 2016), and basically consisted in the addition of the iron/calcium salt to the arsenic(V) solution, raising the pH value to the adequate pH value for the precipitation of the arsenate salt, filtration, drying and storage. Depending of the overall conditions, sometimes seemed adequate to use temperature and pressure

TABLE 6. Elution results

Eluent	Aqueous/resin ratio, mL·g^{-1}	% Elution	[As] in the eluate, mg·L^{-1}
HCl 0.1 M	2000^a	99	8.9
HCl 0.1 M	1000^a	99	17.7
HCl 0.1 M	500^a	99	35.4
HCl 0.01 M	500^a	85	30.4
NaOH 0.1 M	800^b	14	1.2
NaOH 0.1 M	400^b	8	1.3
NaOH 0.1 M	200^b	7	2.3

^{a}Resin loaded with 17.9 mg·g^{-1}; ^{b}Resin loaded with 16.7 mg·g^{-1}; Temperature: 20° C; Stirring speed: 300 min^{-1}. Time: 30 min in the precipitation stage (Monhemius and Swash, 1999), using this procedure, crystalline escorodite (iron(III) arsenate) was formed, being this salt much stable than the corresponding amorphous scorodite.

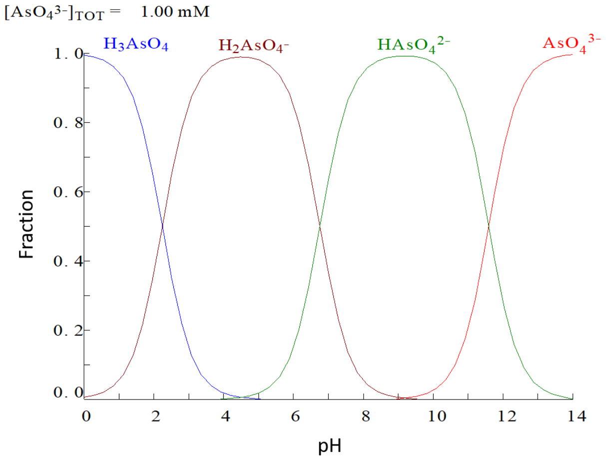

FIGURE 3. Arsenic(V) species distribution versus pH. Diagram generated by MEDUSA® software package and Hydra database.

4. CONCLUSIONS

- Under a wide range of pH values, from 4 to 9, Dowex 1x8 resin seemed adequate to remove toxic arsenic(V) from aqueous solutions. This pH-dependence is consequence to the form in which arsenic(V) is present in the solutions (Fig. 3), thus, $H_2AsO_4^-$ and $HAsO_4^{2-}$ being the arsenic species which are loaded onto the resin, moreover, the divalent species are loaded onto the resin at higher percentages than the monovalent one. Thus, two defined pH values are used in the investigation, pH 4, being $H_2AsO_4^-$ the predominant species, and pH 9 with the $HAsO_4^{2-}$ predominance.
- At pH 4, the variation of the stirring speed (600-1000 min^{-1}) has negligible influence in the arsenic uptake onto the resin, being the rate law dominated by aqueous diffusion; the anion exchange process is exothermic, and the arsenic loading onto the resin responded to the Freundlich isotherm model and the first-order kinetic model.
- At pH 9, maximum arsenic uptake onto the resin is obtained in the 700-900 min^{-1} range, and the process best fits to the aqueous diffusion law; the anion exchange process is also exothermic, and the arsenic uptake onto the resin followed the Langmuir Type-1 isotherm model.
- In the 4-9 range of pH values, and against the resin performance, multiwalled carbon nanotubes does not remove arsenic(V) from the solution at any extent.
- The elution of the arsenic loaded onto the resin is best performed using HCl solutions, and at the same time that arsenic is removed from the resin, this is regenerated. From the eluate, arsenic can be precipitated in the form of amorphous iron(III) or calcium(II) arsenates.

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