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COMBINED SORPTION OF COBALT(II) AND NONIONIC SURFACTANT BY POLYACRYLIC ACID-FUNCTIONALIZED CATION EXCHANGER

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ABSTRACT

The kinetics of combined sorption of cobalt(II) and nonionic surfactant ALM-10 by hydrogen form of Purolite C 106 polyacrylic acid-functionalized cation exchanger were investigated: kinetic curves were obtained determining the concentration of nonionic surfactant spectrophotometrically, and that of cobalt(II) complexometrically. Reducing the initial solution acidity from pH 5 to 8, the coefficients of intraparticle diffusion (D) for cobalt(II) increase, although they decrease as the cation exchanger saturation increases. The sorption of the surfactant proceeds slower than that of cobalt(II).

Regeneration of the cation exchanger was performed using $0.7\,M$ HCl for the removal of cobalt(II), $0.5\,M$ NaOH for the removal of the surfactant, and $0.7\,M$ HCl for the conversion to hydrogen form. Hydrogen form of Purolite C 106 can be applicable for the simultaneous removal of nonionic surfactant

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and cobalt(II) from plating rinse sewage to recover water for recycling.

Key Words: Heavy metal; Nonionic surfactant; Sorption; Cation exchanger

INTRODUCTION

Cobalt is one of the so-called trace metals, playing an important role in biological processes, favoring them at lower concentrations although hindering at higher ones because of complexing thiol groups—SH in the enzymes leading to the development of cancer (1). Cobalt plating processes followed by rinsing operations generate a large volume of wastewater containing cobalt(II) salts toxic to aquatic organisms. Industrial effluents must be purified to such an extent that the quality of natural water is not changed and the permitted concentration for cobalt(II), 1 ppm, is not exceeded (2). Ion exchange can be considered as a mature technology for wastewater treatment, removing metallic ions, and protecting the environment from contamination (3,4). To minimize the toxic discharges and reduce the consumption of water, the metal finishing industry recycles rinsewater. Recycling by ion exchange and zero discharge is considered as a solution for the wastewater problem, especially in plating shops with large work load and high water consumption (5,6). The hydrogen-containing form of weak acid cation exchangers is used for the removal of trace objectionable heavy metals from rinsewater prior to recycling (7,8). Chelating resins including aminophosphonate and iminodiacetic ones can reduce the metal content of the wastewater to practically nondetectable levels (9,10), whereas the concentrated metal cations including cobalt(II) are eluted from the resins and recovered (3,4,11). The sorption of cobalt(II) in carboxylic ion exchangers is higher than that in sulphonic or phosphonate ones (12).

Nonionic surfactants including ALM-10 are often used for the formulation of compositions containing cobalt(II). Wetting, emulsifying, degreasing, lubricating, dispersing, stabilizing, and other surface active properties are inherent to ALM-10 because of both the active chain of ethylene oxide ($-C_2H_4O-$ units) and the alkyl radical present in the surfactant. ALM-10 as one of the universal nonionic surfactants is used widely in electroplating and many other technological processes (13). The concentration of nonionic surfactants in sewage effluents is limited to 2 ppm (14). When the nonionic surfactants are not eliminated, foaming of deionized water and fouling of ion exchangers, tankages, and connecting tubes by microorganisms are the most serious problems faced when treating plating rinsewater by ion exchange. It would be less expensive to eliminate both cobalt(II) and the nonionic surfactant using the same sorbent. The hydrogen-containing form of Purolite C 106 polyacrylic acid-functionalized cation exchanger is under investigation for the cosorption of both the heavy metal (II) and the nonionic surfactant. The affinity of this cation exchanger towards the nonionic surfactant arises from the steric accordance between the structure of the cation exchanger and the structure of the surfactant. The steric accordance of the adjacent carboxyl groups in the cation exchanger (15) with the chain of $-C_2H_4O-$ units in the surfactant favors the sorption (16) proceeding on the basis of hydrogen bond (17,18).

When integrating the sorber filled with cation exchanger into the sewage purification system, the data concerning sorption kinetics would be required. The data concerning mass transfer are required for modeling of ion exchange systems from laboratory to large scale equipment and for calculating the necessary amount of sorbent and the time for the sorption processes (19). Mass transfer as a diffusion process for reaching the sorption equilibrium is related to the solution concentration as well as to the reactions such as acid dissociation and complex formation. These reactions can affect the sorption kinetics as profoundly as they affect the equilibria. The conservation of electroneutrality demands that the exchange between a bead of cation exchanger and liquid be stoichiometric: for each equivalent of counter-ions entering the bead one equivalent of counter-ions must leave. The result is that the total concentration (in equivalents) remains constant in both the bead and the liquid.

Two steps appear to qualify for the control of sorption rate of both metal cations (20) and the organic substances (21): the mass transfer in either the bead or the liquid, whichever is slower. At the concentration $<0.003\,\mathrm{mol\,dm^{-3}}$ the sorption is controlled by diffusion through immobile film, enveloping a bead (film diffusion), whereas, at the concentration $>0.1\,\mathrm{mol\,dm^{-3}}$—by the diffusion within a bead (intraparticle diffusion). At the concentration $0.003-0.1\,\mathrm{mol\,dm^{-3}}$ (the range covered when treating sewage water by ion exchange) the sorption is controlled by the mixed diffusion (both film diffusion and intraparticle one at the same time).

In our previous paper, the kinetics of copper(II) sorption by Purolite C 106 cation exchanger in the presence of oxyethylated alcohols (OS-20) was presented (22). It was determined that the sorption of copper(II) and OS-20 was controlled by the intraparticle diffusion; on increasing the acidity, both the rate of copper(II) intraparticle diffusion and the equilibrium sorption decreased although the corresponding parameters for OS-20 increased; the presence of copper(II) resulted in an increase in OS-20 diffusion rate, however, led to a decrease in the equilibrium sorption (22).

The purpose of this study was to determine the mutual effect of cobalt(II) and the nonionic surfactant ALM-10 onto the rate of their combined diffusion within Purolite C 106 polyacrylic acid-functionalized cation exchanger, considering the application for water recovery from the cobalt plating rinse sewage.

EXPERIMENTAL

The kinetic curves were obtained by stirrer-reactor technique (15) at a stirring intensity of 400 rpm at which the effect of the film diffusion was negligible. The samples of the cation exchanger (0.5 g dry mass) were equilibrated with 100 mL solution at 20°C during the predetermined time. The used concentrations of ALM-10 and $CoCl_2$ in pure solutions and in the mixtures (0.006 mol dm^{-3}) are in the range of concentrations inherent to plating rinsewater treated by ion exchange.

The coefficients of the intraparticle diffusion (D, $m^2\,sec^{-1}$) were calculated (20):

$$D = \frac{B_t r^2}{\pi^2 t}, \tag{1}$$

where r is the radius of bead (cm); t the duration of interaction (sec); B_t the kinetic coefficient calculated on the ground of the degree (F) of the cation exchanger saturation: $F = a/a_{max}$, here a is the comparative sorption determined at the equilibration during the predetermined time (mmol g^{-1}); a_{max} the maximum sorption at the equilibrium (mmol g^{-1}).

Taking into account that the initial solution concentration (0.006 mol dm^{-3}) was within the limits where the sorption was controlled by mixed diffusion, the effective kinetic coefficient of the external mass transfer (β, sec^{-1}) as a supplemental parameter indicating the effect of film diffusion onto the cobalt(II) and ALM-10 sorption rate was also determined. The determination of β was carried out by a fixed-bed technique: the solution containing 0.006 mol dm^{-3} of solute was passed through a glass column (3 cm in diameter) filled with 10 g (dry mass) of the cation exchanger (bead size 0.2–0.5 mm) at a flow rate of 0.0414 cm sec^{-1}. β was calculated using the fundamental equation describing the relationship between C/C_0 and t in a flowing system (23):

$$\ln\left(\frac{C}{C_0}\right) = \frac{\beta C_0 t}{N_0} - \frac{\beta Z}{u}, \tag{2}$$

where C is the effluent equilibrium concentration (mol dm^{-3}); C_0 the influent concentration (mol dm^{-3}); t the duration of the solution passing through the columns (sec); Z the bed height (cm); u the linear flow rate (cm sec^{-1}) defined as the ratio of the flow rate V ($cm^3\,sec^{-1}$) to the cross-sectional area S_c (cm^2), of the bed; N_0 the total sorption capacity of the column at equilibrium (mmol dm^{-3}) of the cation exchanger. N_0 was calculated (19,23):

$$N_0 = \frac{1000 a_{max} m_c}{V_c}, \tag{3}$$

here m_c is the mass of the cation exchanger in the column (g); V_c the volume of the cation exchanger in the column (cm^3). β values decrease with increase in the influent concentration although they increase with increase in the flow rate. β values are also dependent on the mass of the cation exchanger in the column, the bead size, the loading, and other factors (19,23). Consequently, β values calculated according to the method above characterize the sorption of cobalt(II) and ALM-10 only at the conditions above. Cobalt(II) concentration in the solution was determined complexometrically with $0.05\,M$ solution of EDTA disodium salt and murexide; the concentration of nonionic surfactant spectrophotometrically using chromazurol (24); the concentration of carboxyl groups $-$COOH (sorptive capacity of the cation exchanger) with $0.1\,N$ NaOH (15).

Purolite C 106 polyacrylic acid-functionalized cation exchanger (25) was received from Purolite International Ltd (Pontyclun, Mid. Glam., Wales, UK). The cation exchanger was treated with $0.5\,M$ NaOH, washed with distilled water, converted to the hydrogen form with $0.7\,M$ HCl, washed to neutrality, and sieved with 0.2 and 0.5 mm sieves. The fraction containing the granules from 0.2 to 0.5 mm in diameter was dried in air. The concentration of carboxyl groups was 7.3 meq g^{-1}.

The composition of nonionic surfactant ALM-10 produced on an industrial scale (Russia) corresponds to the formula $C_nH_{2n+1}O(C_2H_4O)_mH$, where n is from 12 to 14; $m \sim 10$; mean molecular mass ~643 (13).

All the reagents used were of analytical grade. The solutions were prepared with fresh distilled water.

RESULTS AND DISCUSSION

Effect of pH and ALM-10 on the Rate of Cobalt(II) Diffusion

The dependence of the kinetic coefficient (B_t) on the duration of interaction (t) between the cation exchanger and the solution tends to the straight line for the sorption of both cobalt(II) (Fig. 1) and ALM-10 from the pure solutions as well as from the mixture (Fig. 2). Taking into account that the straight-line dependence refers to the sorption controlled by the intraparticle diffusion (20), the coefficient of intraparticle diffusion (D) may be considered as the main parameter of the sorption rate for both cobalt(II) and ALM-10. Reducing the solution acidity from pH 5 to 8, the D values for cobalt(II) sorption increase (Fig. 3). This is because the equilibrium of carboxylic group ionization in the cation exchanger displaces to the right (here R is the matrix of the cation exchanger consisting of saponified copolymer of methylester of acrylic acid with divinylbenzene).

$$2RCOOH + Co^{2+} \leftrightharpoons (RCOO)_2Co + 2H^+ \qquad (4)$$

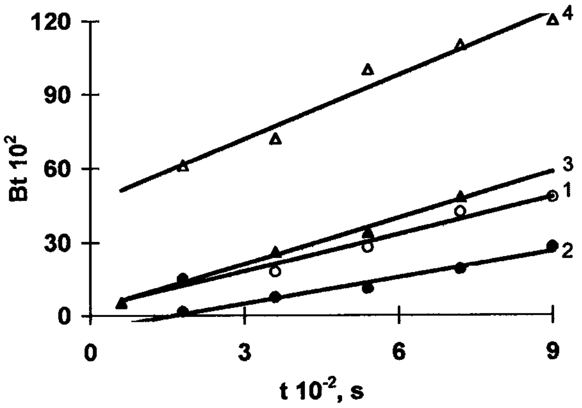

Figure 1. Dependence of kinetic coefficient (B_t) for the sorption of cobalt(II) on the duration of interaction (t) and the solution acidity: 1—without ALM-10 at pH 5; 2—in the presence of ALM-10 at pH 5; 3—without ALM-10 at pH 8; and 4—in the presence of ALM-10 at pH 8.

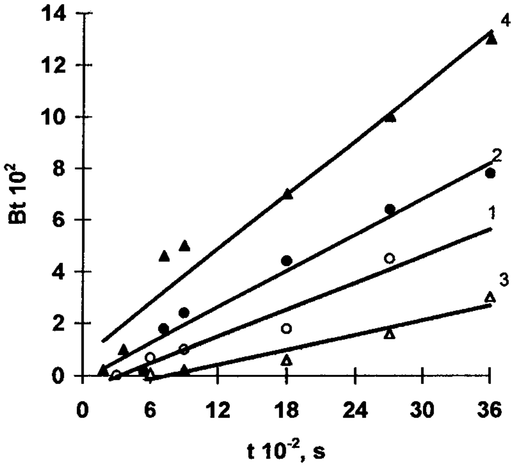

Figure 2. Dependence of kinetic coefficient (B_t) for the sorption of ALM-10 on the duration of interaction (t) and the solution acidity: 1—without cobalt(II) at pH 5; 2—in the presence of cobalt(II) at pH 5; 3—without cobalt(II) at pH 8; and 4—in the presence of cobalt(II) at pH 8.

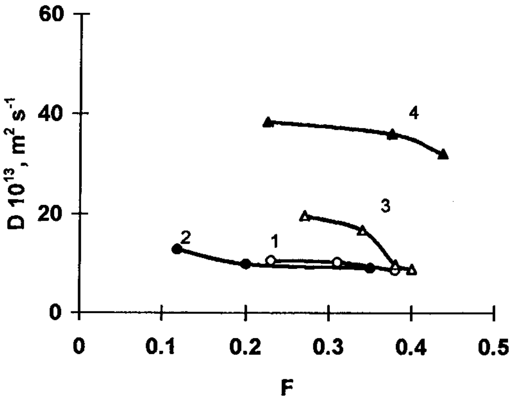

Figure 3. Dependence of the coefficient of intraparticle diffusion (*D*) for cobalt(II) on the degree of the cation exchanger saturation (*F*): 1—without ALM-10 at pH 5; 2—in the presence of ALM-10 at pH 5; 3—without ALM-10 at pH 8; and 4—in the presence of ALM-10 at pH 8.

The displacement is followed by an increase in both the concentration of ionized carboxyl groups and the swelling of the cation exchanger. The swelling favors the intraparticle diffusion (Table 1) resulting in an increase in the comparative sorption of cobalt(II) (Fig. 4). On increasing the degree of saturation of the cation exchanger (*F*), a decrease in *D* values for cobalt(II) is observed at both the pH values investigated. Transition metal(II) (d-element) complexing by the deprotonated form of carboxylic cation exchanger follows the transfer of the electrons from the oxygen atom in hydroxyl to the oxygen atom in carbonyl (15). The transfer leads to equalization of the electronic envelopes surrounding the two

Table 1. Dependence of the Mean Values of Maximum Sorption ($a_{\max}$) and the Coefficients for the Intraparticle Diffusion (*D*) of Cobalt(II) and ALM-10 on the Solution Composition and pH. Initial Concentration of Cobalt(II) and ALM-10 in Solution 6 mmol dm^{-3}; Bed Height 15 cm; Flow Rate 0.0414 cm sec^{-1}

Composition	pH = 5		pH = 8	
	$a_{\max}$ (mmol g^{-1})	$D \times 10^{13}$ (m^{2}sec^{-1})	$a_{\max}$ (mmol g^{-1})	$D \times 10^{13}$ (m^2 sec^{-1})
Co(II) + H_2O	0.073	9.29	0.14	18.23
(ALM-10) + H_2O	0.63	0.30	0.55	0.14
Co(II) + (ALM-10) + H_2O	0.095	15.40	0.089	33.70
(ALM-10) + Co(II) + H_2O	1.12	1.17	0.54	1.90

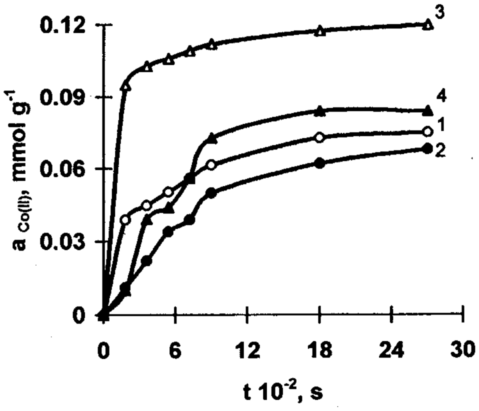

Figure 4. Kinetic curves of cobalt(II) sorption: 1—without ALM-10 at pH 5; 2—in the presence of ALM-10 at pH 5; 3—without ALM-10 at pH 8; and 4—in the presence of ALM-10 at pH 8.

oxygen atoms present in the carboxyl group. The undivided electron pairs pass from the oxygen atoms contained in carboxyl group into the vacant orbitals of the transition metal cations leading to the formation of both the stable cyclic compound on the ground of complex (ionic + coordinate) bond and the coordinate compound on the ground of the single coordinate bond (15).

The effect of nonionic surfactant on the cobalt(II) diffusion rate depends on the acidity of the solution. On decreasing the acidity from pH 5 to 8, the action of ALM-10 leads to the increase in *D* value for cobalt(II) (Fig. 3). This results presumably from the cation exchanger swelling followed by a decrease in the interference of the cation exchanger matrix with cobalt(II) intraparticle diffusion in alkaline media, on the one hand, and an increase in the sorption of cobalt(II) bonded to the surfactant molecules, on the other. The sorption of metal (II) cations bonded to nonionic surfactant (26) can take place through both cobalt(II) on the ground of complex (ionic+coordinate) or single coordinate bond and the surfactant on the ground of hydrogen bond. The simultaneous and subsequently competitive taking up of both cobalt(II) and the surfactant leads to the decrease in the comparative sorption (*a*) of cobalt(II) at the two pH values investigated (Fig. 4). Not only is the sorption of cobalt(II) affected by the surfactants, but also the vice versa holds good.

Effect of pH and Cobalt(II) onto the Rate of ALM-10 Diffusion

On increasing the solution acidity from pH 8 to 5, an increase in *D* values (Fig. 5), comparative sorption (*a*) (Fig. 6), and $a_{\max}$ for ALM-10 is observed

(Table 1). This is because, unlike cobalt(II), the sorption of nonionic surfactant proceeds on the ground of hydrogen bonding; consequently, it is favored by the increase in the concentration of protonated carboxyl groups following the displacement of equilibrium in Eq. (4) to the left. Whereas, in the presence of cobalt(II) the diffusion rate (Fig. 5) and the comparative sorption of ALM-10 (Fig. 6) are higher at pH 8 when compared with the corresponding parameters at pH 5. In alkaline media, the action of cobalt(II) is related to the increase in the concentration of deprotonated carboxyl groups promoting the diffusion and the sorption of the cobalt(II) free cations and the cations bonded to ALM-10, consequently taking up both cobalt(II) and ALM-10. The above effect of cobalt(II) on the rate of ALM-10 sorption prevails over the effect of the decrease in the solution acidity. In the absence of metal cations the diminution of the acidity usually hinders the sorption of nonionic surfactant.

According to D values, the sorption of ALM-10 from both the pure solution and the solution containing cobalt(II) proceeds slower than the sorption of cobalt(II) at the two pH values investigated. This may occur because the hydrogen bond leading to the ALM-10 sorption is weaker than the ionic or coordinate bond leading to the cobalt(II) sorption. The difference in the size of the hydrated species should also be taken into account in this case: ALM-10 molecules are bigger than the cobalt cations; consequently, the matrix of the cation exchanger interferes more strongly with ALM-10 diffusion than with cobalt(II).

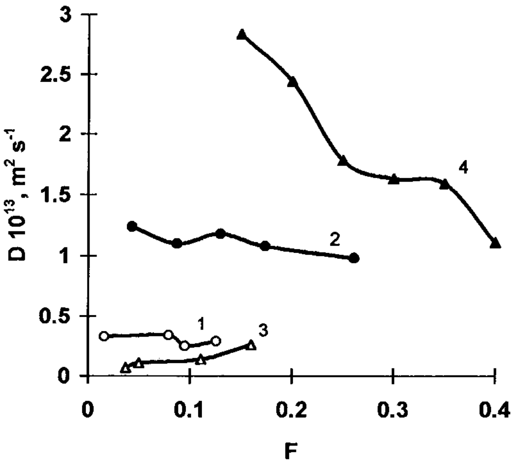

Figure 5. Dependence of the coefficient of intraparticle diffusion (D) for ALM-10 on the degree of the cation exchanger saturation (F): 1—without cobalt(II) at pH 5; 2—in the presence of cobalt(II) at pH 5; 3—without cobalt(II) at pH 8; and 4—in the presence of cobalt(II) at pH 8.

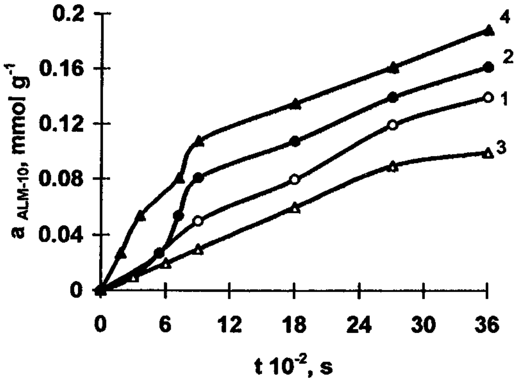

Figure 6. Kinetic curves of ALM-10 sorption: 1—without cobalt(II) at pH 5; 2—in the presence of cobalt(II) at pH 5; 3—without cobalt(II) at pH 8; and 4—in the presence of cobalt(II) at pH 8.

The kinetic coefficient of the external mass transfer (β), calculated on the basis of the plot $\ln(C/C_0)$ vs. time (Figs. 7 and 8) is higher for ALM-10 than for cobalt(II) (Table 2). The difference in β values for the two species may arise from the action of coions having negative charge, subsequently hindering the cobalt(II) film diffusion. The action of ALM-10 promotes the mass transfer of cobalt(II), whereas the action of cobalt(II) hinders the mass transfer of ALM-10.

It is noticeable that both D values (Table 1) and β values (Table 2) for the diffusion of both ALM-10 and cobalt(II) within the hydrogen form of Purolite C

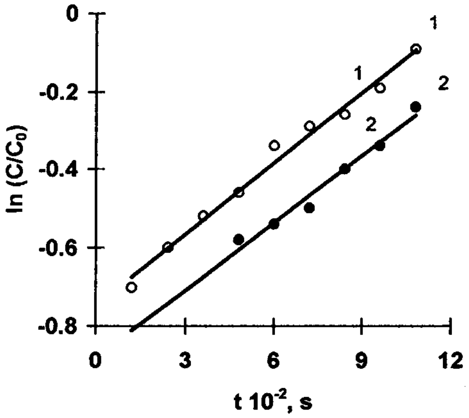

Figure 7. Plots of $\ln(C/C_0)$ vs. time of interaction (t) for cobalt(II) sorption: 1—without ALM-10; and 2—in the presence of ALM-10; initial pH 5.

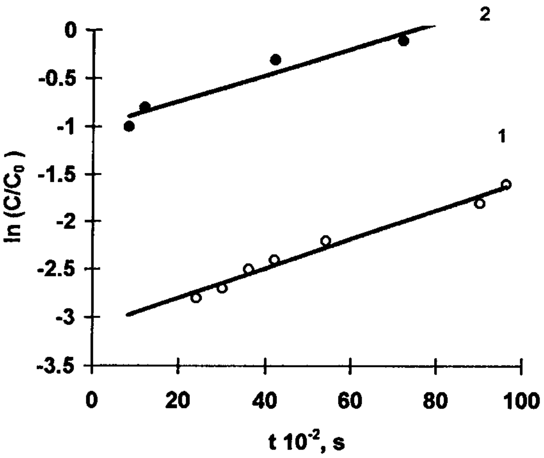

Figure 8. Plots of $\ln(C/C_0)$ vs. time of interaction (t) for ALM-10 sorption: 1—without Co(II); and 2—in the presence of cobalt(II); initial pH 5.

106 cation exchanger are comparable with the corresponding values for the diffusion of other contaminants in the cation exchangers usually used in sewage purification systems (20).

The loading behavior of cobalt(II) and ALM-10 to be removed from the solution in a fixed-bed ion exchanger is usually expressed in terms of C/C_0 as a function of time for a given bed height, giving the so-called breakthrough curves (Fig. 9). The performance of Purolite C 106 packed column with respect to cobalt(II) is better than the performance with respect to ALM-10. The elimination of ALM-10 is promoted by the action of cobalt(II) (Fig. 9, curve 4). The negligible concentrations of cobalt(II) and ALM-10 in the effluent at the breakthrough and sufficiently high equilibrium sorption refer to the possibility of

Table 2. Kinetic Coefficients of the External Mass Transfer (β), Sorption Capacities (N_0) and Correlation Coefficients (R^2) for the Sorption of Cobalt(II) and ALM-10 from Pure Solutions and from the Mixture. Initial Concentration 6 mmol dm^{-3}; Initial pH 5; Bed Height 15 cm; Flow Rate 0.0414 cm sec^{-1}

Composition	$\beta \times 10^2$ (sec^{-1})	N_0 (mmol dm^{-3})	R^2
Co(II) + H_2O	0.206	25.16	0.9870
(ALM-10) + H_2O	0.856	217.14	0.9785
Co(II) + (ALM-10) + H_2O	0.242	32.74	0.9734
(ALM-10) + Co(II) + H_2O	0.276	386.03	0.9300

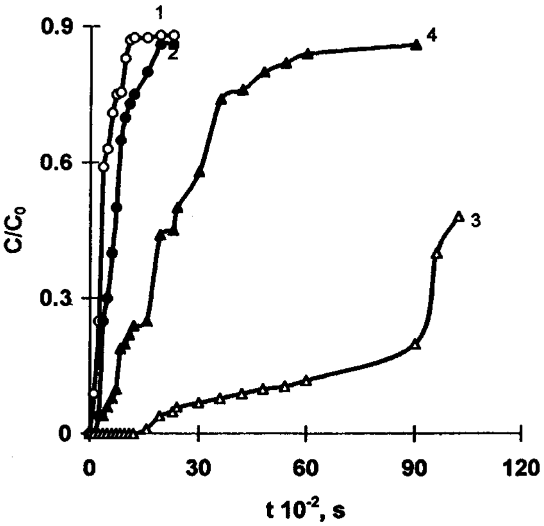

Figure 9. Breakthrough curves for the sorption of: 1—cobalt(II) without ALM-10; 2—cobalt(II) in the presence of ALM-10; 3—ALM-10 without cobalt(II); and 4—ALM-10 in the presence of cobalt(II); initial pH 5.

Purolite C 106 ion exchanger in practical use. Column model can be designed on the grounds of the sorption capacities and the kinetic curves predicting the service time at breakthrough by varying the volume of the ion exchanger and the flow rate according to the volume and the initial concentration of rinsewater treated.

The Regeneration of Purolite C 106 Cation Exchanger Saturated with Cobalt(II) and ALM-10

The action of acid solution on the cation exchanger saturated with both cobalt(II) and ALM-10 leads to the rupture of the bond between carboxyl group and cobalt(II) because the affinity of hydrogen cation to the cation exchanger is higher than that of cobalt(II). The desorption of cobalt(II), and the consequent regeneration of the cation exchanger from cobalt(II) can be achieved using 0.7 *M* HCl (Fig. 10). The simultaneous desorption of ALM-10 and cobalt(II) is also observed to some extent (Fig. 10, curve 3). Whereas, the action of alkaline solution on the cation exchanger saturated with both cobalt(II) and the surfactant, results in the rupture of the hydrogen bond between the protonated carboxyl groups and $-C_2H_4O-$ units in the surfactant. This is because the displacement of equilibrium in Eq. (4) to the right leads to the decrease in the concentration of the

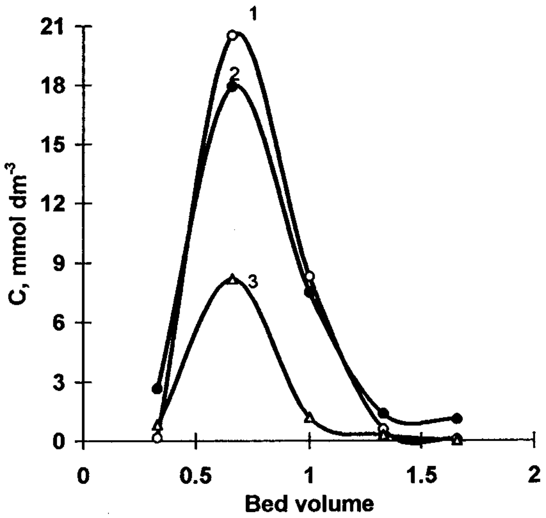

Figure 10. Elution of cobalt(II) (1,2) and ALM-10 (3) from Purolite C 106 packed column saturated with: 1—cobalt(II) without ALM-10; 2—cobalt(II) in the presence of ALM-10; and 3—cobalt(II) in the presence of ALM-10. Eluent 0.7 mol dm^{-3}; concentration in eluate (C) refers: to cobalt(II) (1,2); to ALM-10 (3).

protonated carboxyl groups, which results in the desorption of the surfactant (Fig. 11). The regeneration of the cation exchanger using 0.7 M HCl for the removal of cobalt(II), 0.5 M NaOH for the removal of the surfactant, and 0.7 M HCl for the conversion to hydrogen form enables to recover the sorptive capacity.

CONCLUSIONS

The combined sorption of cobalt(II) and the nonionic surfactant (ALM-10) by hydrogen form of Purolite C 106 polyacrylic acid-functionalized cation exchanger is controlled by the intraparticle diffusion. On reducing the solution acidity from pH 5 to 8, the coefficients of the intraparticle diffusion (D) for cobalt(II) increase. Cobalt(II) sorption is accelerated significantly by the action of ALM-10 at pH 8, although it is almost unaffected at pH 5. With an increase in the cation exchanger saturation (F), the cobalt(II) sorption rate decreases.

The sorption of ALM-10 proceeds slower than that of cobalt(II). Unlike cobalt(II), the rate of ALM-10 sorption decreases with the decrease in the solution acidity. The effect of cobalt(II) on the rate of ALM-10 sorption prevails over the effect of the solution acidity: in the presence of cobalt(II) the ALM-10 sorption rate is higher at pH 8 than at pH 5; whereas, without cobalt(II) the

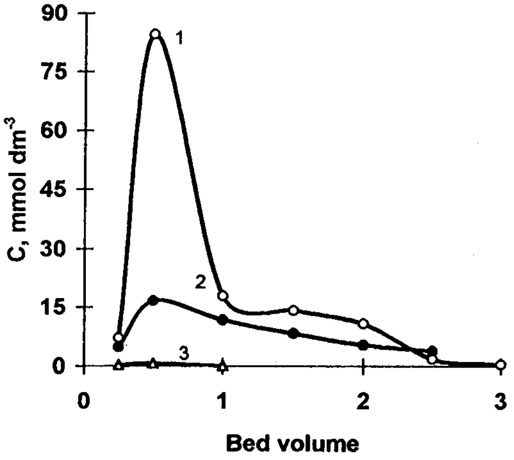

Figure 11. Elution of ALM-10 (1,2) and cobalt(II) (3) from Purolite C 106 packed column saturated with: 1—ALM-10 without cobalt(II); 2—ALM-10 in the presence of cobalt(II); and 3—ALM-10 in the presence of cobalt(II). Eluent 0.5 mol dm^{-3}; concentration in eluate (C) refers to: ALM-10 (1,2); cobalt(II) (3).

sorption rate of the nonionic surfactant is higher in acidic medium than in alkaline one.

D values as well as the kinetic coefficients of the external mass transfer (β) for the sorption of both cobalt(II) and the surfactant by hydrogen form of Purolite C 106 cation exchanger are comparable to the corresponding parameters for other contaminants and other cation exchangers used in the systems of sewage purification.

The regeneration of the cation exchanger enables to recover the sorptive capacity using 0.7 M HCl for the removal of cobalt(II); 0.5 M NaOH for the removal of the surfactant; 0.7 M HCl for the conversion to hydrogen form.

Hydrogen form of Purolite C 106 could be applied for the combined removal of nonionic surfactant and cobalt(II) from cobalt plating rinse sewage in order to recover water for recycling and for preventing the environmental contamination.

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