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DUAL-TEMPERATURE ION-EXCHANGE SEPARATION OF COPPER AND ZINC BY DIFFERENT TECHNIQUES.

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ABSTRACT

Separation of copper from zinc-containing acidic solutions has been advanced by different techniques of dual-temperature ion-exchange fractionation on iminodiacetic resin Amberlite IRC 718. Cycling-zone adsorption and parametric pumping (using single-column as well as double-column set-ups) experiments have been carried out in fixed-bed ion-exchange column. Measurement of equilibrium characteristics of the resin-solution system has shown that separation results from the variation of the ions sorbability with temperature. It has been found that temperature variation leads to reverse of selectivity between copper and hydrogen ions. Cycling-zone adsorption technique has allowed the copper content to be decreased up to 4 times in the resin phase and increased up to 1.7 times in the effluent when carrying out the process at 80 and 20 ^{0}C, respectively. The copper concentration has been decreased 4.4 fold after 9 treatment cycles of solution aliquot (15 BV) by single-column mode of the parametric pumping method. Two steps of the fractionation process (loading and elution) have been performed by continuous mode of operation in two counter-current columns. Continuous separation is preferred over fixed-bed column techniques. The steady-state sorption fronts have been formed on both stages of the process due to the reverse of selectivity mentioned above.

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INTRODUCTION

The problem of the separation of copper and zinc ions is frequently encountered in hydrometallurgical recovery of zinc from pyretic ores. Conventionally, zinc is produced by Roast-Leach-Electrowing processes, e.g., [1]. The acidic wastes containing zinc and copper arise after both leaching and electrolytic stages of the processes. Large quantities of acidic effluents containing zinc and copper are also produced from pyretic ores mining. A similar problem of copper and zinc ions separation appears when purifying zinc liquors before the electrolysis stage. The purification circuits used in many of the world's electrolytic zinc plants involve the treatment of solutions with zinc powder in three stages [2]. Extraction and ion-exchange methods for fractionation of copper and zinc ions have also been described in the literature [3,4]. The main drawback of these methods is the expenditure of auxiliary reagents needed for regeneration of treated extractants and resins. This is known to be the main source of waste generation in ion-exchange (and extraction) technologies.

These drawbacks can be overcome by using dual-temperature or some other dual-parametric fractionation techniques. These methods are based on the strong dependence of the bi-phasic equilibrium upon some intensive thermodynamic parameters (e.g., temperature, pressure, ionic strength, pH). The development of dual-temperature separations has been started in early works by Andreev et al. [5] and Wilhelm et al. [6]. The theoretical aspects of such processes have been considered by Pigford et al. [7], Aris [8] , Chen et al. [9], and Wankat [10]. The principles of dual-temperature separations have been reviewed in several monographs, e.g. [11-14].

Parametric separation can be carried out by applying several techniques using both fixed-bed and counter-current column modes of operation. Principles of these techniques have been described in several publications, e.g., [11-14]. The fixed-bed columns are used for performing both cycling-zone adsorption and parametric pumping methods. The basic disadvantage of these techniques results from their periodicity. Connection of several units in a cascade allows one to perform the processes in quasi-continuous mode [7]. The connections can be made in parallel [7] or in series [7,15]. Usually, comparison between one- and multi-column set-ups is carried out when the volumes of solutions being treated are equal. It is more interesting to compare specific throughput (in bed volumes, BV) of single- and multi-columns techniques, especially when connected in series because connecting the columns in parallel has the same specific throughput as one-column set-up.

The preferred mode of operation of dual-parametric separation in counter-current columns is in a continuous process. Theory and practical applications of counter-current columns in separation and purification processes have been reported in a number of monographs [16-18] and periodicals, e.g., [19, 20]. This technique allows the separation to be carried out continuously and to scale down the size of ion-exchange columns and auxiliary equipment needed. The resin and solution from the columns have a constant composition; it decreases the amount of wastes produced; and in certain cases it may completely eliminate waste generation. The theory of dual-temperature separation in counter-current columns has been developed since the 1970s [21], but practical application of this method is still very limited [22]. Furthermore, the only known large-scale process based upon dual-parametric method is the "Sirotherm" process used for water demineralisation that has been described by Bolto et al. [23].

The problem that needs more intensive investigation is the parametric separation of multi-component mixtures. The theory of these processes has been developed mainly for linear isotherms [11, 14] and assuming that sorption of species proceeds independently [9]. Butts et al. have reported that a component with medium sorbability (sodium) is accumulated in the resin phase during the separation by parametric pumping of solution containing K^+, Na^+ and H^+ ions on Dowex 50×8 [24]. Grevillot with co-workers studied dual-temperature separation of Ag^+-Cu^{2+}-Pd^{2+} mixture on Duolite C265 resin [25]. They found that palladium ions are concentrated together with most sorbable Ag^+ ion. The separation of several amino acids in presence of 1.0 M HCl on Dowex 50×8 resin by parametric pumping method has been carried out by Simon et al. [26]. They have shown that all amino acids are concentrated in the top reservoir and purified in the bottom one.

Some chelating resins, e.g. iminodiacetic ion-exchange resins demonstrate high capacity towards copper over zinc ions in acidic solutions [27]. Recently, equilibrium properties of iminodiacetic resins Lewatit TP 207 [28] and Amberlite IRC 718 [29] have been studied at different temperatures in the range from 15 to 80 ^{0}C. It has been shown that sorption of copper increases with temperature for these resins, and they both can be used for dual-temperature separation of copper and zinc ions [28,29].

The aim of the present investigation is to study recovery of copper ions from acidic zinc-containing solutions (modelling industrial liquors and wastes of hydrometallurgical processing of zinc-containing ores) by different techniques based on dual-temperature separation.

EXPERIMENTAL PART

This work was performed with acidic solutions containing 0.024-0.025 N $CuSO_4$ and 0.22-0.24 N $ZnSO_4$ at pH=1.8. The solutions were prepared from commercial $CuSO_4$ $\cdot 5H_2O$ and $ZnSO_4 \cdot 7H_2O$ (KEBO, Sweden), and H_2SO_4 (Merck, Germany) of p.a. quality using de-ionised water. Concentrations of Cu^{2+} and Zn^{2+} were determined by Atomic Absorption Spectrophotometry using AAS 603 (Perkin Elmer). Concentrations of H^+ ions were measured by potentiometric titration using pH-meter pH-537 WTW (Germany) supplied with combined glass electrode. The uncertainty of metal ions determination was less than 1.5%. Iminodiacetic ion-exchange resin Amberlite IRC 718 (kindly supplied by Rohm and Haas) was conditioned by sequential treatment with 0.1 N NaOH and 0.1 N HCl solutions prior to the dual-temperature experiments.

Both dual-temperature separation experiments and determination of equilibrium properties of the resin when contacting with initial solution were performed at 20 and 80 ^{0}C. Experiments were carried out in jacketed glass columns (of inside diameter 1.2 cm) charged with 5 cm^3 (for equilibrium studies) or 10 cm^3 (for separation experiments) of resin suspension. The heights of the resin beds were 5 and 10 cm, respectively. The construction of the column provided for heating/cooling of both resin and solution phases as shown in Fig.1. Desired temperature was maintained by an external thermostat. The feed solution was passed through the column at 2 cm^3/min of flow rate using a peristaltic pump.

For the determination of equilibrium properties [30], the initial solution was percolated through the resin bed until the concentration of the effluent equalled that of influent. Then the excess solution phase was evacuated from the column by a water pump. Sorbed ions were eluted from the resin with 1.0 N H_2SO_4 and analysed. The total capacity of the resin was determined independently by using 0.25 N $CuSO_4$ and the same technique.

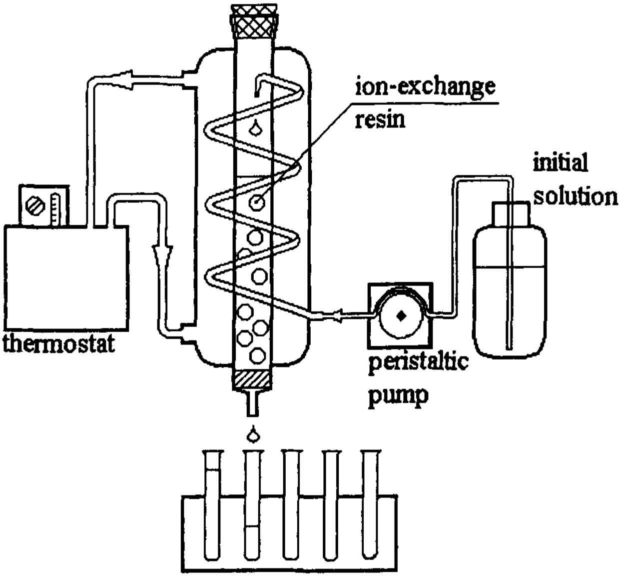

FIGURE 1. Scheme of the laboratory fixed-bed thermostatic unit.

In all separation experiments in the fixed-bed columns, the resin was pre-equilibrated with the feed solution at 20 ^{0}C. In applying cycling-zone adsorption technique, the feed solution was passed through the resin bed in down-flow direction. After passing of each portion of solution (300-400 cm^3), the peristaltic pump was stopped, and the temperature was switched from 80 ^{0}C to 20 ^{0}C (or vice versa). After 0.5-hour break (this time was shown to be sufficient for achieving temperature equilibrium in the column), the pumps were turned on for pumping the solutions through the column. The effluent was collected in portions at given time intervals and analysed.

Parametric pumping experiments were carried out by different techniques using single-column and double-column set-ups. The special porous spacer was placed on top of the resin bed to avoid stirring of the ion-exchanger beads, and a capillary tube was fitted into top side of the column to collect effluent directly over the resin bed. In the single-column mode (see Fig.2a) the column was heated to 80 ^{0}C, and an aliquot of feed solution (150 cm^3) was percolated upward through the resin bed. Effluent was collected

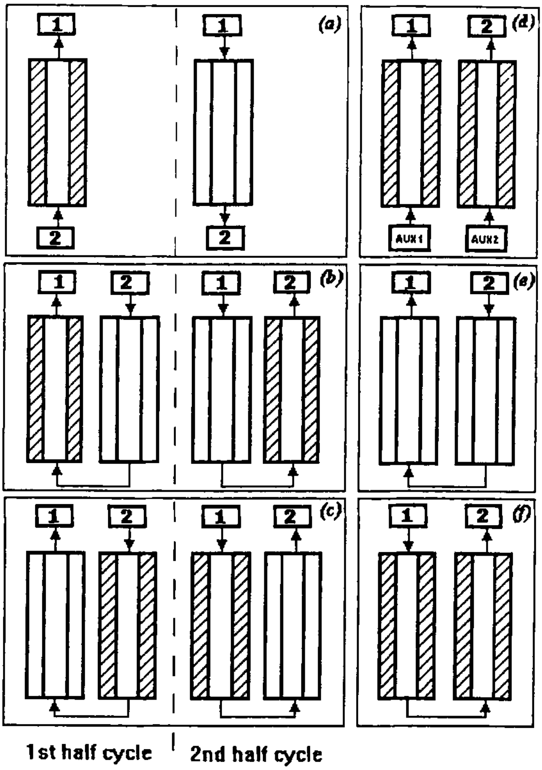

FIGURE 2. Schemes of: (a)- single-column- and (b)-(f) -double-column parametric pumping experiments; (b)- purification- and (c)- enrichment working modes; (d)-(f)- preparation steps: (d)- symmetric for purification mode-, (f)- symmetric for enrichment mode-, and (e)- asymmetric. Hot columns are shaded

in the top reservoir (1), stirred, and analysed. After that, the column was cooled back to 20 ^{0}C. The solution from the reservoir 1 was passed through resin bed in a down-flow direction. The effluent was collected in the bottom reservoir (2) and analysed. Thereafter, the column was heated again and all operations were repeated in a cyclic mode.

In the double-column parametric pumping experiments, the bottom ends of both columns were connected by a short pipe, and different temperatures were maintained in these columns, so effluent outgoing from the first column (cold or hot) was directly fed into the second one (hot or cold). Solution leaving the second column was collected in the reservoir and analysed. After that, the hot column was cooled and the cold one was heated, the columns were exposed at "new" temperatures during 0.5 hour, and all operations were repeated again. Depending on the temperature of the column from which solution was exited in the reservoir, we can arbitrarily separate double-column experiments on two groups: the purification mode, if solution is withdrawn from the hot column (Fig.2b), and the enrichment (concentrating) mode, if the effluent is removed from the cold one (Fig.2c).

The preparation steps were also different for double-column set-up. Special attention is paid to correctly compare results of single- and double-column experiments. In the symmetric mode, (Fig.2d) the first step involved pumping the feed solution (two portions on 150 cm^3 of each) into the bottom ends of two columns from auxiliary reservoirs (AUX 1 and AUX 2). The resin beds were at 80 ^{0}C, and the effluent solutions were collected in the reservoir 1 and 2, respectively. After that, the bottom ends of the columns were connected by pipe, and operations were performed as in the other two-column process (purification mode). One more preparation step was needed for the concentrating mode with symmetric input of feed solution: the columns were cooled and solution from reservoir 2 was passed through both columns to reservoir 1 at 20 ^{0}C (Fig.2e).

Aliquot (300 cm^3) of the feed solution from reservoir 1 entered the top end of one column, and was collected in reservoir 2 from the top end of the other column when carrying out the asymmetric preparation step for both purification and enrichment modes (Fig.2f). Then, cyclic runs were performed as above, the solution was always percolated through the hot column in upward direction as well as through the cold column in down-flow direction. Experimental conditions for parametric pumping runs are summarised in Table 1.

Fractionation experiments in the continuous mode were performed by using laboratory counter-current column schematically plotted in Fig. 3 (inside diameter 1.5 cm; area 1.8 cm^2; height of working section 200 cm). Feed solution was percolated through packed bed of the resin in upward direction with volumetric velocities 8-25 cm^3/min; the resin moved in opposite direction when passing of solution was interrupted for a short time. Design and operations of the column were analogous to

TABLE 1. EXPERIMENTAL CONDITIONS OF PARAMETRIC PUMPING RUNS

	Number of columns	Volume of solution, cm^3	Preparation step	Working mode	Schemes, figures	Results, figures
Run 1	1	150			2a	5-7
Run 2	2	150	symmetric	purification	2b,2d	6
Run 3	2	300	symmetric	purification	2b,2d	6
Run 4	2	300	symmetric	enrichment	2c,2d,2e	7
Run 5	2	300	asymmetric	purification	2b,2f	6
Run 6	2	300	asymmetric	enrichment	2c,2f	7

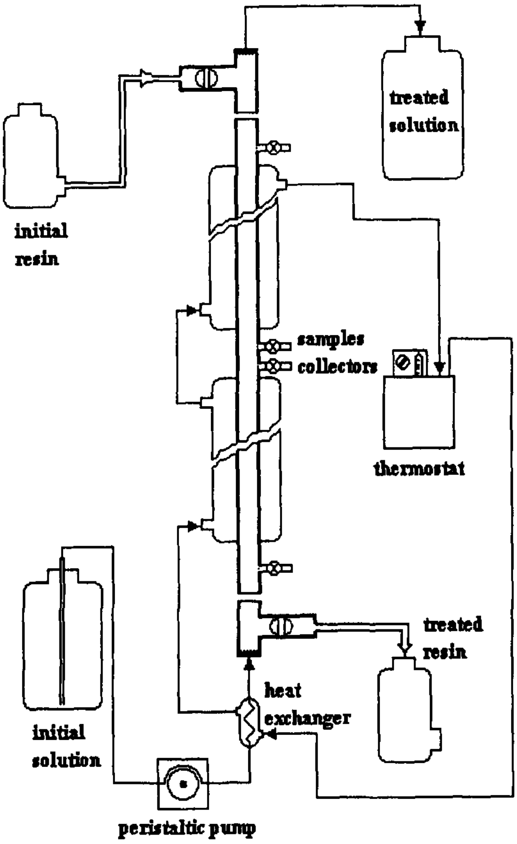

FIGURE 3. Scheme of laboratory counter-current column.

those described early [18, 19]. It should be noted that the column has a thermostatic jacket to keep the resin bed at the desired temperature (by an outside thermostat) and the feed solution was preheated (pre-cooled) up to a given temperature in the jacket prior to its introduction into the column. Two steps of the separation process (loading and elution of copper) were investigated in the counter-current column. The resin withdrawn from the column after one stage (virtually in equilibrium with the initial solution at given temperature) was used as feed in the next process step studies. The loading stage was investigated when treating the feed resin by the initial solution at 80 ^{0}C, while the elution step was studied by the same technique at 20 ^{0}C. Samples of solution (1-2 cm^3) were collected through special capillary collectors (positioned along column) and analysed.

RESULTS AND DISCUSSIONS

Equilibrium Properties

It was found that the total loading capacity depends on temperature and consists 1.52±0.01 meq/ cm^3 of resin suspension. Experimental results of equilibrium properties studies of IRC 718 resin when contacting with feed solution (0.024 N $CuSO_4$ + 0.22 N $ZnSO_4$, pH= 1.8) are given in Table 2. The selectivity coefficients K^i_n for multi-component ion-exchange equilibria were calculated relative to least sorbable ion (zinc), as described [16,31]:

$$K^i_{Zn} = \left(\frac{y_i}{x_i}\right)^{1/z_i} \left(\frac{x_{Zn}}{y_{Zn}}\right)^{1/2}$$

where y and x - equivalent fractions of the ions in equilibrium in the resin and solution phases, respectively; z_i - charge of the ith ion; i - copper or hydrogen ions.

It is seen from Table 2 that fractions of copper and zinc ions in the resin phase increase with temperature while fraction of hydrogen ion $(y_H = 1 - y_{Cu} - y_{Zn})$ decreases, so that K^{Cu}_{Zn} increases slightly while K^H_{Zn} decreases remarkably with the increase of temperature. These changes of ions sorbability at different temperatures lead to reversing the selectivity between hydrogen and copper ions (hydrogen ion is most sorbable at 20 ^{0}C whereas copper ion is most sorbable at 80 ^{0}C). The phenomenon of the

TABLE 2. EQUILIBRIUM PROPERTIES OF AMBERLITE IRC 718 RESIN. TOTAL CAPACITY 1.52 meq/cm^3; TOTAL CONCENTRATION IN SOLUTION 0.258 N.

Temp., ^{0}C	x_{Cu}	x_{Zn}	y_{Cu}	y_{Zn}	K_{Zn}^{Cu}	K_{Zn}^{H}
20	0.087	0.839	0.463	0.086	7.2±0.2	19.2±0.7
80	0.087	0.837	0.785	0.130	7.6±0.2	2.8±0.2

reverse of selectivity has not been observed in previous investigations for analogous systems [28,29]. The possibility of dual-temperature fractionation of ions from solutions under study derives from the remarkable change of copper ion sorbability with temperature. The reversal of selectivity is the reason that the separation can be achieved at conditions when the steady-state sorption fronts (for favourable isotherms) will be formed in the column on both stages of fractionation operations.

Results of copper separation by cycling-zone adsorption method (concentrations of Cu^{2+} and H^+ ions versus volume of passed solution) are plotted in Fig.4. From the Fig. it is seen that the concentration of copper in the effluent decreases up to 4 times and increases up to 1.7 times when the solution is percolated at 80 and 20 ^{0}C, respectively. The concentration of H^+ ions increases by 2.2 times and decreases by 3.2 times at the same conditions. The purified solution throughput estimated from these data is about 10 BV.

Separation by cycling-zone methods was performed on a short resin bed (10 cm), and this does not allow discussion of steady-state sorption fronts. This question will be thoroughly considered below when separating of the ions in the counter-current column. Nevertheless, the volume of the solution needed to pass through the column to attain equilibrium is similar for both stages (hot and cold) of the operation. This fact indirectly verifies possibility of the steady-state fronts formation.

Parametric Pumping Technique

Fractionation of copper and hydrogen ions in single-column units are shown in Fig. 5, where it is seen that copper concentration decreases in the top reservoir (after

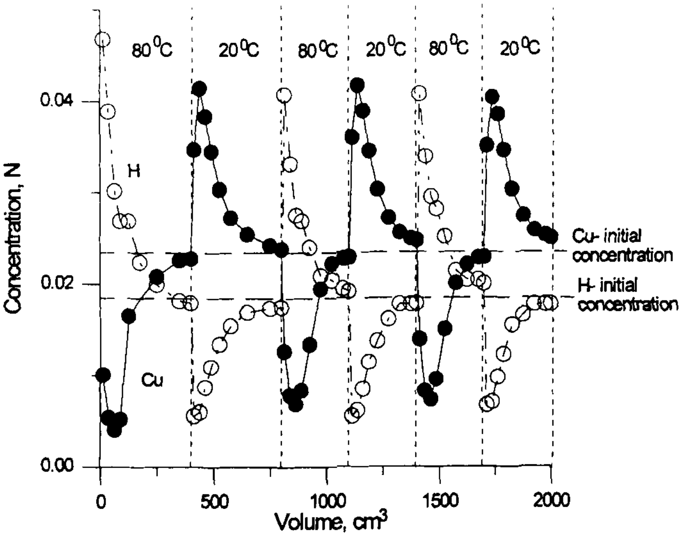

FIGURE 4. Effluent concentration profile when fractionation by cycling-zone adsorption method. Dotted lines correspond to breaks (0.5 h) in solution passing under change of temperature.

percolation through the hot column) and increases in the bottom vessel (after filtration through the cold column). The concentration of hydrogen ion changes in the opposite directions under the same conditions. Concentration of copper ion can be decreased up to 4.4 times or increased up to 1.4 times after 9 cycles of parametric pumping, whereas concentration of hydrogen ion, respectively, increases 1.7 times and decreases 1.9 times. According to Chen model [9] this separation occurs in region 2, when penetration distance of one sorption front (at hot temperature) is less than both penetration distance of the other sorption front (at cold temperature) and length of the resin bed. It means that complete purification of such volume of the feed solution from copper can not be possible. For this goal the volume of solution under treatment should be decreased.

The results of copper purification in two-column units are plotted in Fig.6. The advantage of this set-up over the single column set-up can be obtained only in the first cycle of the runs. Its occurrence is due to the increase of separation effect with the increase of the resin bed length. Usually, multi-column set-ups are operated when

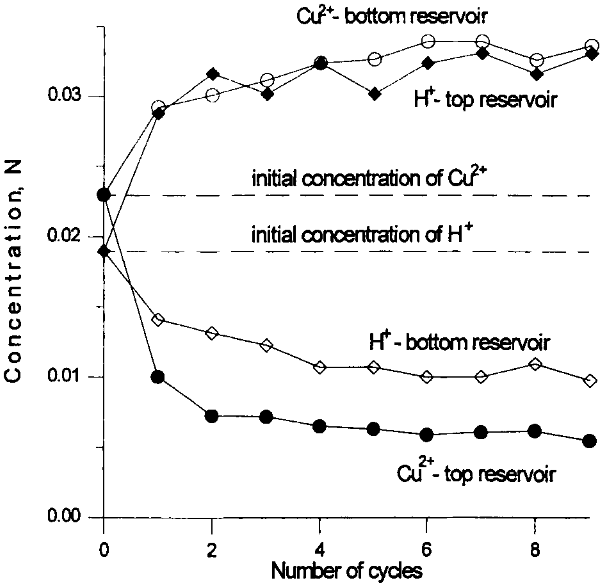

FIGURE 5. Variation of concentrations in the top (1) and bottom (2) reservoirs for single-column parametric pumping (Run 1).

purified solution is partly collected after this step. Unfortunately, the main preference of the parametric pumping method over cycling-zone adsorption technique- accumulation of separation degree upon cycles can not be used in this mode. Under similar conditions, the one-column unit specific throughputs (15 BV), separation by double-column mode occurs (as classified in [9]) in the third region (where penetration distances of sorption front for both cold and hot half cycle are more than length of resin bed). It leads to the increase of copper concentration in the reservoir in the following steps.

Results of ions fractionation in double-column set-up for the enrichment modes are plotted in Fig.7. It is seen that problems that appear for separation by these techniques are the same as for the purification modes. Moreover, the degree of concentration after the first step is less than what is obtained for one-column set-up. When the passing of solution are repeated (at number of cycles more than 3 for run 4, and more than 8 for run 6) copper concentration decreases below the initial one. This

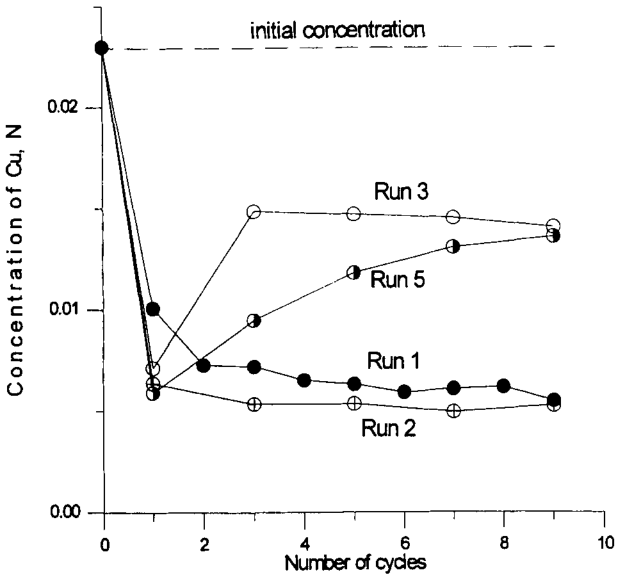

FIGURE 6. Comparison between single-column (Run 1) and purification modes of double-column (Runs 2, 3, and 5) parametric pumping.

behaviour can not be described by Chen model [9]. It should be pointed out that this model has been developed for linear isotherms when assuming that components are sorbed independently from one another. The separation process characteristics for system being studied show that isotherms greatly are not linear and the exchange of ions between the phases occurs coherently. The experiments carried out by using the double-column technique shows that more sophisticated models are required to describe such separation processes.

Counter-Current Column Technique

Steady-state distributions of the ions along the counter-current column when loading stage investigations are plotted in Fig.8a. It is seen that copper ions concentration decreases by 6 times whereas zinc and hydrogen concentrations increase along the column. The length of sorption front is less than 30 cm (at volumetric velocity

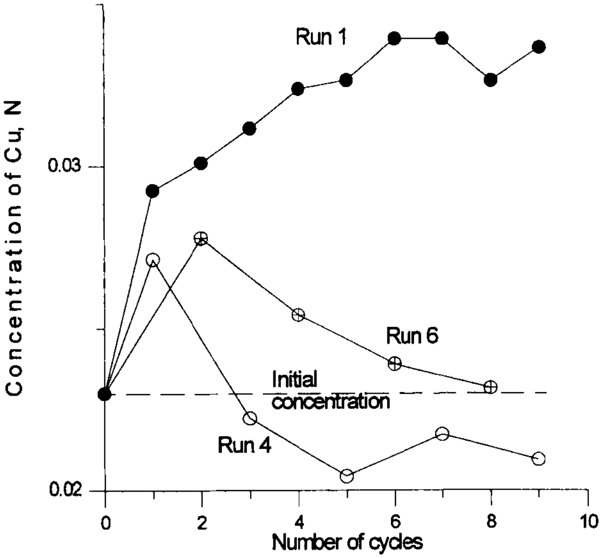

FIGURE 7. Comparison between single-column (Run 1) and enrichment mode of double-column (Runs 4 and 6) parametric pumping.

12 cm^3/min) and, was found, to slightly increases with velocity. The purification of the initial solution from copper in the counter-current column is better than in the fixed-bed column (compare with Fig.4) probably due to the increase of the resin bed length (up to infinity). Results of elution experiments are plotted in Fig. 8b. It is seen that the copper concentration in the solution outgoing from the column is 1.7 times higher than the initial one. Zinc concentration also increases along the column whereas hydrogen ion concentration decreases. The length of the sorption front here is much more than for previous step (80-100 cm at the same volumetric velocity). Experimental results allow one to conclude that these sorption fronts for both stages are steady-state, because their length and shape are kept constant during time, see Figs. 8a and 8b.

Hence, dual-temperature separation in the counter-current columns is preferred to fixed bed fractionation including common advantages of continuous modes reported above.

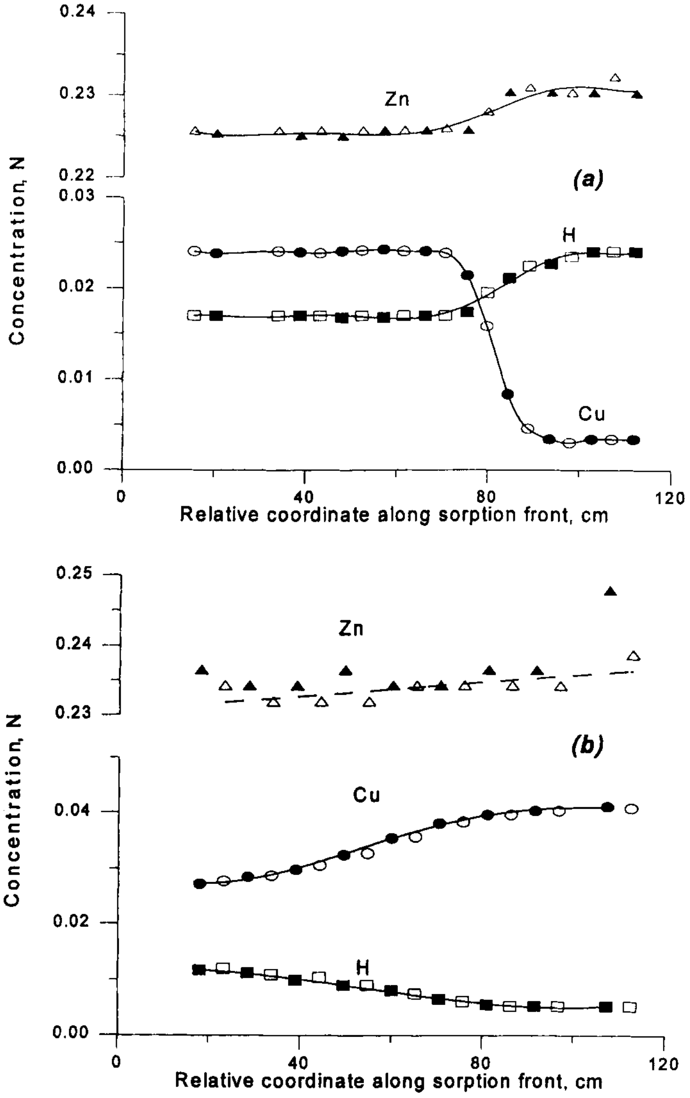

FIGURE 8. Distribution of species concentration along counter-current column when dual-temperature separation with flow rate of solution 12 cm^3/min; a)- loading stage (dark points- after 13.5 hours of operation, white points- after 15.2 hours); b)- elution stage (dark points after 15 hours of operation, white points- after 17 hours).

CONCLUSIONS

Dual-temperature ion-exchange fractionation of copper from zinc-containing acidic solutions on iminodiacetic resin IRC 718 occurs due to change of sorbability of separated ions with temperature. This results in the reverse of selectivity between copper and hydrogen ions that allows to carry out the fractionation process with high efficiency. When separating of the initial solution by the cycling-zone adsorption method, copper concentration in effluent decreases up to 4 times and increases up to 1.7 times after hot (80 ^{0}C) and cold (20 ^{0}C) stages of the process.

Applying single-column mode of parametric pumping technique, the separation effect increases upon cycling. This results in a 4.4-fold purification of the feed solution (15 BV) from copper after 9 cycles. Double-column mode of parametric pumping offers advantage over single-column set-up in the first cycle only due to increase of resin bed length.

The use of counter-current columns for dual-temperature fractionation allows one to carry out separation process continuously, to diminish size and number of both basic and auxiliary equipment. Both stages of the process (loading and elution) occur with formation of steady-state fronts in the counter-current columns due to reverse of the selectivity between copper and hydrogen ions.

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