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Simultaneous solvent extraction of cobalt and magnesium in the presence of nickel from sulfate solutions by Ionquest 801

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Abstract: The simultaneous extraction of Co(II) and Mg(II) from nickel sulfate solutions has been carried out using the organophosphonic extractant Ionquest 801 diluted in Exxsol D-80. Statistical design and analysis of experiments were used in order to determine the main effects and interactions of the solvent extraction parameters, which were the extraction pH at equilibrium, the temperature, the extractant concentration and the organic/aqueous phase ratio. A statistically designed experiment was also carried out to study the stripping of the Ionquest 801 organic phase loaded with cobalt and magnesium by sulfuric acid solution. The number of stages required for both extraction and stripping processes of cobalt and magnesium was evaluated. The results of continuous counter-current mini-plant tests demonstrated the simultaneous recovery of cobalt and magnesium from nickel sulfate solution.

Keywords: Ionquest 801; nickel; cobalt; magnesium; separation

INTRODUCTION

The Laboratory of Metallurgy of the National Technical University of Athens has developed and patented a novel integrated hydrometallurgical method suitable for treating low-grade nickel oxide ores efficiently and economically. It involves heap leaching of the ore by dilute sulfuric acid at ambient temperature, purification of the leach liquors and recovery of nickel and cobalt.[1–4]

A typical composition of the pregnant solution produced from heap leaching of Greek low-grade nickeliferous laterites with sulfuric acid is $Ni^{2+} = 5\,g\,dm^{-3}$, $Co^{2+} = 0.6\,g\,dm^{-3}$, $Fe^{3+} = 22\,g\,dm^{-3}$, $Al^{3+} = 6\,g\,dm^{-3}$, $Cr^{3+} = 1\,g\,dm^{-3}$ and $Mg^{2+} = 5\,g\,dm^{-3}$.[1] After the removal of iron, chromium and aluminium by chemical precipitation,[5] the leach liquor containing nickel, cobalt and magnesium must also be separated and recovered so that the barren solution can be recycled to heap leaching. In the present paper a solvent extraction process for the separation of cobalt and magnesium from nickel sulfate solutions was studied using the organophosphonic extractant Ionquest 801.

The extraction and separation of cobalt and nickel from sulfate, chloride and ammoniacal solutions have been of interest to hydrometallurgists for a long time. The separation of both metals has been achieved in materials such as copper converter slag, chromite overburden, nickel laterite, manganese sea nodules, etc, although the separation of these two metals is particularly difficult from weak sulfate solutions.[6–8] This is due to the very similar chemical behaviour exhibited by these metals in aqueous solutions.

A lot of work has also been performed in the field of solvent extraction to develop extractants for nickel and cobalt separation. Although some of these reagents have been developed for the selective extraction of nickel(II) over cobalt(II), the more usual route has been to extract cobalt(II), leaving nickel(II) in the aqueous phase. It is the latter route that has found application in nickel refineries of INCO, Outokumpu and Matthey Rustenburg. Cyanex 272, the most widespread extractant in the hydrometallurgical recovery of nickel, has been used by Rickelton *et al*,[9] who have reported the superiority of Cyanex 272 for cobalt/nickel selectivity, compared with the analogous phosphoric and phosphonic acids, to obtain highly pure cobalt metal in a mini-plant-scale continuous counter-current extraction. Ritcey *et al*[10] have developed a complete process, for the Resolute Bulong project, for the separation of cobalt and nickel in the presence of Mn and Mg.[10] Their study proposed the use of Cyanex 272 first to extract cobalt and manganese at pH 4.5, leaving nickel and magnesium

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in the solution. Subsequently, their separation was carried out using Versatic 10 in the pH range 6.8–7.0. Ritcey and Lucas[11] have also used 0.5 mol dm^{-3} Kelex 100 with 10% isodecanol in Solvesso 150 to determine the preferential extraction of Cu, Fe(III), Ni, Zn, Co, Fe(II), Mn, Ca and Mg from acidic solutions in the pH range 0.5–6.0. Sole and Hiskey[12] have reported a comparison of the solvent extraction behaviour of the first-row transition elements from acid sulfate solutions, with distribution coefficients shifting to lower pH with increasing sulfur substitution and decreasing pK_a value of the extractant. The effect was greater with respect to nickel. Sumitomo Metal Mining Company used Versatic 10 for the extraction of cobalt and nickel from sulfate leach liquors arising from the pressure oxidisation leaching of mixed sulfides.[13] Subsequently, the Co/Ni separation was carried out by solvent extraction with tri-n-octyl amine. Stripping was accomplished with dilute HCl to produce cobalt and nickel chloride solutions. Mihaylov *et al*[14] have developed a novel hydrometallurgical process for nickel and cobalt recovery from Goro laterite ore. Their study proposed the simultaneous extraction of 0.6 g dm^{-3} Co and 6 g dm^{-3} Ni from 3 g dm^{-3} Mn and 10 g dm^{-3} Mg sulfate solutions by 15% Cyanex 301 at an aqueous/organic phase ratio of 2.5. It was reported that cobalt and nickel could be totally extracted at a pH below 2.0, while the other metals remained in the aqueous solution.

The present research work was aimed at the solvent extraction of cobalt and magnesium in the presence of nickel from synthetic sulfate solutions by Ionquest 801. Cobalt and magnesium stripping from the loaded organic phase was also studied using dilute sulfuric acid. Nickel, which remained in the aqueous phase, can be concentrated by Cyanex 272 and then stripped by synthetic nickel spent electrolyte to produce a solution suitable for nickel electrowinning. Subsequently, cobalt and magnesium separation can be achieved by the carboxylic acid Versatic 10.[13]

Factorial design of experiments and statistical analysis of the data were used to determine the main effects and interactions of the chosen factors and select the optimum conditions.[15] The factors studied were equilibrium pH, temperature, extractant concentration and aqueous/organic (A/O) phase ratio. Cobalt and magnesium isotherms were constructed for both the extraction and stripping processes, and the number of stages required was predicted under the optimum conditions established by the statistical analysis. Extraction and stripping processes were further studied in a continuous counter-current laboratory solvent extraction unit.

EXPERIMENTAL

Reagents

The extractant Ionquest 801 was kindly supplied by Albright and Wilson Americas (now Rhodia; Richmond, VA, USA). The reactive component $C_{16}H_{34}PO_3H$ is the mono(2-ethylhexyl) ester of 2-ethylhexylphosphonic acid.[16] The molecular weight is 306 and the density (24 °C) 0.94 g cm^{-3}. The extractant was used without further purification. The organic diluent Exxsol D-80 was obtained from Exxon Chemicals Europe (Machelen, Belgium). In all cases the organic phases were modified with 5% tri-n-butylphosphate (TBP).

Procedure

The initial aqueous phase was a synthetic solution with concentrations of Co^{2+} (0.63 g dm^{-3}), Ni^{2+} (3.8 g dm^{-3}) and Mg^{2+} (5.75 g dm^{-3}) similar to those of a real heap leach liquor. All the experiments were conducted in a 500 cm^3 five-necked, round-bottomed split reactor fitted with a glass stirrer, reflux condenser, thermometer and pH electrode. The two phases were contacted by rapid stirring and the pH was controlled by small additions of 5 mol dm^{-3} NaOH using an automatic titration device (Metrohm Dosimat/Impulsomat, Herisau, Switzerland). Heating was provided by an electric mantle and the temperature of the liquid was controlled by a Fisons Scientific Apparatus monitor (Loughborough, UK). Stripping of the loaded Ionquest 801 was carried out using dilute sulfuric acid. The initial strip liquor was a 3 or 4 mol dm^{-3} sulfuric acid solution. Continuous tests were carried out in a laboratory solvent extraction unit (Bell Engineering Model 282, Tucson, AZ, USA).

Nickel, cobalt and magnesium in the aqueous phases (aqueous feed and extraction raffinate) were analysed using a Perkin-Elmer Model 4100 atomic absorption spectrometer (Boston, MA, USA).

RESULTS AND DISCUSSION

Effect of pH on extraction

In order to determine the ability of Ionquest 801 to selectively extract cobalt and magnesium in the presence of nickel from a sulfate solution, extraction tests were carried out at various equilibrium pH values under certain other conditions.

The results, presented in Fig 1, show that Ionquest 801 can efficiently extract both cobalt and magnesium at pH 5.0, whereas nickel co-extraction at this pH reached 7%.

Statistical study of extraction process

Factorial design and analysis of experiments were used in order to determine the main effects and interactions of the solvent extraction factors. The factors studied and their levels are shown in Table 1. Parameters which were kept constant during experimentation were stirring speed = 1200 rpm, initial cobalt concentration = 0.63 g dm^{-3}, initial nickel concentration = 3.8 g dm^{-3} and initial magnesium concentration = 5.75 g dm^{-3}.

The main responses under investigation were the percentages of cobalt and magnesium extraction in the organic phase. The concentrations of cobalt

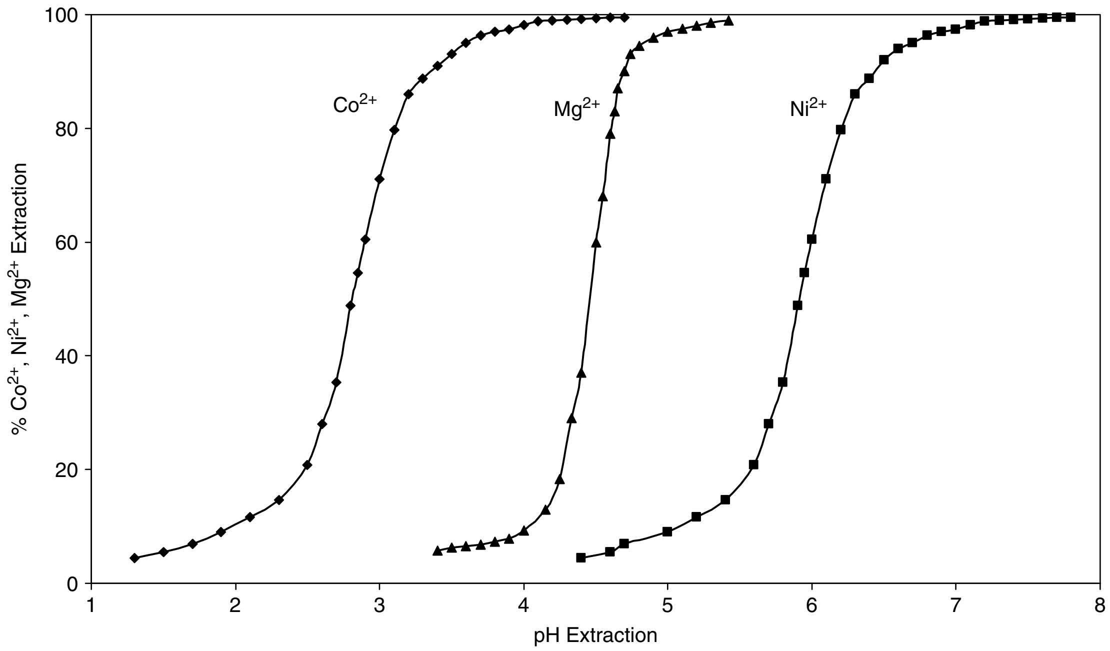

Figure 1. Cobalt and magnesium selectivity over nickel sulfate solutions with Ionquest 801. Organic phase: 20% Ionquest 801 in Exxsol D-80 with 5% TBP; $T = 40\,^\circ\mathrm{C}$; aqueous phase: $0.63\,\mathrm{g\,dm^{-3}}\ \mathrm{Co^{2+}}$, $3.8\,\mathrm{g\,dm^{-3}}\ \mathrm{Ni^{2+}}$, $5.75\,\mathrm{g\,dm^{-3}}\ \mathrm{Mg^{2+}}$.

Table 1. Cobalt and magnesium solvent extraction by Ionquest 801 — minimum and maximum levels of variables

Factor	Variable	Low level	High level	Units measured
A	Extraction pH (pH)	4.5	5.0	pH units
B	Temperature (*T*)	25	40	°C
C	Extractant concentration (*C*)	5	20	% v/v
D	Organic/aqueous (O/A) phase ratio (*R*)	1:2	1:1	—

and magnesium before and after solvent extraction experiments and the extraction percentages are given in Table 2.

As can be seen in Table 2, in all runs of the factorial design, high percentages of cobalt and magnesium extraction in the organic phase were obtained, which proves the applicability of Ionquest 801 for the simultaneous extraction of $\mathrm{Co^{2+}}$ and $\mathrm{Mg^{2+}}$ from sulfate solution in the presence of $\mathrm{Ni^{2+}}$. Nickel co-extraction did not exceed 6.5% under the current experimental conditions. This nickel could be easily scrubbed, as shown in the next subsection.

To study the main effects and interactions of the factors on cobalt and magnesium extraction, a Yates analysis and an analysis of variance were carried out on the percentages of $\mathrm{Co^{2+}}$ and $\mathrm{Mg^{2+}}$ extraction in the organic phase. The results are shown in Tables 3 and 4 respectively.

Regarding magnesium extraction, the main effects A, C and D and the two-factor interactions AB and CD were found to be statistically significant at $\alpha = 0.01$ and positive (Table 4). In the range of variables studied, the extractant concentration (C) and the O/A phase ratio (D) had the largest effects, with a wide gap separating them from the remaining contrasts. The interaction CD seemed plausible as well, being non-additive of a pair of influential factors.

It should be stressed, however, that because of statistically significant interactions the main effects should not be interpreted individually, but the interacting variables should be considered jointly. Thus the extraction pH (A) interacted with temperature (B) even though the latter's main effect was not significant. The interaction occurs because at the high pH value the increase in temperature causes higher magnesium extraction.

The existence of statistically significant interactions meant that a model based on the main effects only would not be suitable and that the response surface was curved in the particular region of the present design.

Based on the above, the suggested 'best'-fitting model for magnesium extraction in the ranges of variables studied and under the present conditions of experimentation is

$$\begin{aligned} Y = 90.3 &+ 0.25X_1 + 1.1X_3 + 0.5X_4 \\ &+ 0.15X_1X_2 + 0.2X_3X_4 \end{aligned} \tag{1}$$

where Y is the predicted value of magnesium extraction and X_1, X_2, X_3 and X_4 are coded variables related to the natural variables by the equations

$$X_1 = \frac{\mathrm{pH} - 4.75}{0.25}, \qquad X_2 = \frac{T - 32.5}{7.5}$$

$$X_3 = \frac{C - 12.5}{7.5}, \qquad X_4 = \frac{R - 0.75}{0.25}$$

Regarding cobalt extraction with Ionquest 801 (Table 3), only the main effects A and C were

Table 2. Results of cobalt and magnesium solvent extraction from nickel sulfate solutions by Ionquest 801

	Cobalt concentration (mg dm^{-3})					Magnesium concentration (mg dm^{-3})				
		After extraction					After extraction			
Treatment code	Before extraction	I	II	Average	% Co^{2+} extraction	Before extraction	I	II	Average	% Mg^{2+} extraction
(1)	650	55.6	51.3	53.5	91.78	5750	653	629	641.0	88.85
a	650	54.9	49.7	52.3	91.95	5750	648	624	636.0	88.94
b	650	57.8	52.7	55.3	91.50	5750	633	653	643.0	88.82
ab	650	54.1	47.5	50.8	92.18	5750	618	642	630.0	89.04
c	650	41.8	39.2	40.5	93.77	5750	531	552	541.5	90.58
ac	650	40.2	35.2	37.7	94.20	5750	518	542	530.0	90.78
bc	650	42.8	36.7	39.8	93.88	5750	537	561	549.0	90.45
abc	650	39.1	36.1	37.6	94.22	5750	507	537	522.0	90.92
d	650	56.0	52.9	54.5	91.62	5750	624	602	613.0	89.34
ad	650	54.9	46.9	50.9	92.17	5750	611	586	598.5	89.59
bd	650	56.7	55.7	56.2	91.35	5750	611	629	620.0	89.22
abd	650	54.4	50.5	52.5	91.93	5750	593	572	582.5	89.87
cd	650	44.1	35.7	39.9	93.86	5750	491	467	479.0	91.67
acd	650	40.4	35.5	38.0	94.16	5750	483	460	471.5	91.80
bcd	650	47.4	42.0	44.7	93.12	5750	499	479	489.0	91.50
abcd	650	37.2	32.0	34.6	94.68	5750	384	364	374.0	93.50

Table 3. Yates analysis and analysis of variance — response: % Co^{2+} extraction by Ionquest 801

		Yates analysis							Analysis of variance		
Treatment code	Response Co^{2+} extraction (%)	1	2	3	4	Divisor	Effect	Indentification	$t = \text{eff}/s_e$	$t_{0.01}$ $\Phi = 16$	Significance at $\alpha = 0.01$
(1)	91.78 ± 0.40	183.73	367.41	743.48	1486.37	16	92.9 ± 0.10	Average			
a	91.95 ± 0.40	183.68	376.07	742.89	4.80	8	0.60 ± 0.20	A	3.00	2.92	S
b	91.50 ± 0.40	187.97	367.07	1.62	−0.65	8	−0.08 ± 0.20	B	−0.41	2.92	NS
ab	92.18 ± 0.40	188.10	375.82	2.99	1.71	8	0.21 ± 0.20	AB	1.07	2.92	NS
c	93.77 ± 0.40	183.79	0.85	0.08	17.41	8	2.18 ± 0.20	C	10.88	2.92	S
ac	94.20 ± 0.40	183.28	0.77	−0.73	0.65	8	0.08 ± 0.20	AC	0.41	2.92	NS
bc	93.88 ± 0.40	188.02	1.13	0.42	0.47	8	0.06 ± 0.20	BC	0.29	2.92	NS
abc	94.22 ± 0.40	187.80	1.86	1.29	0.63	8	0.08 ± 0.20	ABC	0.39	2.92	NS
d	91.62 ± 0.40	0.17	−0.05	8.66	−0.59	8	−0.07 ± 0.20	D	−0.37	2.92	NS
ad	92.17 ± 0.40	0.68	0.13	8.75	1.37	8	0.17 ± 0.20	AD	0.86	2.92	NS
bd	91.35 ± 0.40	0.43	−0.51	−0.08	−0.81	8	−0.10 ± 0.20	BD	−0.51	2.92	NS
abd	91.93 ± 0.40	0.34	−0.22	0.73	0.87	8	0.11 ± 0.20	ABD	0.54	2.92	NS
cd	93.86 ± 0.40	0.55	0.51	0.18	0.09	8	0.01 ± 0.20	CD	0.06	2.92	NS
acd	94.16 ± 0.40	0.58	−0.09	0.29	0.81	8	0.10 ± 0.20	ACD	0.51	2.92	NS
bcd	93.12 ± 0.40	0.30	0.03	−0.60	0.11	8	0.01 ± 0.20	BCD	0.07	2.92	NS
abcd	94.68 ± 0.40	1.56	1.26	1.23	1.83	8	0.23 ± 0.20	ABCD	1.14	2.92	NS

statistically significant at $\alpha = 0.01$ and positive, meaning that these variables operate independently.

The corresponding suggested ‘best’-fitting model for cobalt extraction is

$$Y = 92.9 + 0.29X_1 + 1.09X_3 \tag{2}$$

where

$$X_1 = \frac{\text{pH} - 4.75}{0.25}, \qquad X_3 = \frac{C - 12.5}{7.5}$$

Based on the statistical analysis of the results, the optimum conditions determined for simultaneous cobalt and magnesium extraction from nickel sulfate solution by Ionquest 801 are pH = 5.0, $T = 40\,^{\circ}\text{C}$, extractant concentration = 20% and O/A phase ratio = 1:1.

Scrubbing of loaded organic phases

Ionquest 801 loaded with cobalt and magnesium from the extraction tests was subjected to scrubbing with dilute sulfuric acid at pH = 5.0, O/A = 4:1, $T = 40\,^{\circ}\text{C}$ and agitation = 1200 rpm for 5 min. It was found that the nickel co-extracted with cobalt and magnesium in the organic phase could be effectively scrubbed from the loaded organic, with final co-extraction not exceeding 0.5%. Under these conditions, cobalt transferred to the scrub solution from the organic phase reached 1%.

Table 4. Yates analysis and analysis of variance — response: % Mg^{2+} extraction by Ionquest 801

	Response	Yates analysis							Analysis of variance		
Treatment code	Mg^{2+} extraction (%)	1	2	3	4	Divisor	Effect	Indentification	$t = \text{eff}/s_e$	$t_{0.01}$ $\Phi = 16$	Significance at $\alpha = 0.01$
(1)	88.85 ± 0.21	177.8	355.7	718.5	1445.0	16	90.3 ± 0.06	Average			
a	88.95 ± 0.21	177.9	362.8	726.6	4.1	8	0.5 ± 0.11	A	4.55	2.92	S
b	88.80 ± 0.21	181.4	358.1	1.1	1.8	8	0.2 ± 0.11	B	2.02	2.92	NS
ab	89.05 ± 0.21	181.4	368.5	3.1	2.7	8	0.3 ± 0.11	AB	3.01	2.92	S
c	90.60 ± 0.21	178.9	0.4	0.0	17.6	8	2.2 ± 0.11	C	19.92	2.92	S
ac	90.80 ± 0.21	179.2	0.7	1.8	1.5	8	0.2 ± 0.11	AC	1.81	2.92	NS
bc	90.45 ± 0.21	183.5	1.0	0.4	1.2	8	0.1 ± 0.11	BC	1.51	2.92	NS
abc	90.95 ± 0.21	185.0	2.1	2.3	1.7	8	0.2 ± 0.11	ABC	1.83	2.92	NS
d	89.30 ± 0.21	0.1	0.0	7.1	8.1	8	1.0 ± 0.11	D	9.20	2.92	S
ad	89.60 ± 0.21	0.3	0.0	10.4	2.0	8	0.3 ± 0.11	AD	2.35	2.92	NS
bd	89.25 ± 0.21	0.2	0.3	0.3	1.7	8	0.2 ± 0.11	BD	1.82	2.92	NS
abd	89.91 ± 0.21	0.5	1.5	1.1	1.8	8	0.2 ± 0.11	ABD	2.14	2.92	NS
cd	91.70 ± 0.21	0.3	0.1	0.0	3.3	8	0.4 ± 0.11	CD	3.84	2.92	S
acd	91.80 ± 0.21	0.7	0.3	1.2	0.8	8	0.1 ± 0.11	ACD	0.98	2.92	NS
bcd	91.50 ± 0.21	0.1	0.4	0.2	1.3	8	0.2 ± 0.11	BCD	1.64	2.92	NS
abcd	93.50 ± 0.21	2.0	1.9	1.5	1.4	8	0.2 ± 0.11	ABCD	1.51	2.92	NS

Statistical study of stripping process

On the basis of conclusions drawn from preliminary experimentation, the following variables and levels were chosen in order to study the stripping of cobalt and magnesium from the organic phase by dilute sulfuric acid: H_2SO_4concentration = 3 and 4 mol dm^{-3}; organic/aqueous phase ratio = 1:1 and 2:1.

Parameters with constant values throughout the design were as follows: Ionquest 801 organic phase, $[Co^{2+}]_{org} = 0.6\,g\,dm^{-3}$ and $[Mg^{2+}]_{org} = 5.6\,g\,dm^{-3}$; temperature = 40 °C; stirring speed = 1200 rpm.

The responses under investigation were the percentages of cobalt and magnesium stripped from the loaded Ionquest 801. The concentrations of cobalt and magnesium in the organic phase before and after stripping and their recoveries are given in Table 5.

As the table shows, high percentage stripping of Co^{2+} and Mg^{2+} from the organic phase was obtained in one stage, indicating the easy stripping of Ionquest 801 by dilute sulfuric acid. The Yates analysis and the analysis of variance carried out on the data showed (Tables 6 and 7) that none of the factors was statistically significant. This means that the results differ only because of experimental error and that stripping could be conducted under any combination of the factor values within the ranges studied.

Thus the optimum stripping conditions were determined as H_2SO_4 concentration = 3 mol dm^{-3} and O/A phase ratio = 2:1 at 40 °C.

Table 5. Results of cobalt and magnesium stripping from loaded Ionquest 801

	$[Co^{2+}]_{org}$ (mg dm^{-3})					$[Mg^{2+}]_{org}$ (mg dm^{-3})				
		After stripping					After stripping			
Treatment code	Before stripping	I	II	Average	% Co^{2+} recovery	Before stripping	I	II	Average	% Mg^{2+} recovery
(1)	600	11.0	11.4	11.2	98.13	5600	221	223	222	96.04
a	600	6.9	7.7	7.3	98.78	5600	214	218	216	96.14
b	600	6.2	6.6	6.4	98.93	5600	210	204	207	96.30
ab	600	4.0	5.0	4.5	99.25	5600	185	189	187	96.64

Table 6. Yates analysis and analysis of variance — response: % Co^{2+} recovery from loaded Ionquest 801

	Response	Yates analysis					Analysis of variance		
Treatment code	Co^{2+} recovery (%)	1	2	Divisor	Effect	Identification	$t = \text{eff}/s_e$	$t_{0.01}$ $\Phi = 4$	Significance at $\alpha = 0.01$
(1)	98.13 ± 0.18	196.91	395.09	4	98.78 ± 0.09	Average			
a	98.78 ± 0.18	198.18	0.97	2	0.49 ± 0.18	A	2.43	4.6	NS
b	98.93 ± 0.18	0.65	1.27	2	0.64 ± 0.18	B	3.18	4.6	NS
ab	99.25 ± 0.18	0.32	−0.33	2	−0.17 ± 0.18	AB	−0.83	4.6	NS

Table 7. Yates analysis and analysis of variance — response: % Mg^{2+} recovery from loaded Ionquest 801

		Yates analysis					Analysis of variance		
Treatment code	Response Mg^{2+} recovery (%)	1	2	Divisor	Effect	Identification	$t = \text{eff}/s_e$	$t_{0.01}$ $\Phi = 4$	Significance at $\alpha = 0.01$
(1)	96.04 ± 1.06	192.18	385.12	4	96.28 ± 0.53	Average			
a	96.14 ± 1.06	192.94	0.44	2	0.22 ± 1.06	A	0.21	4.6	NS
b	96.3 ± 1.06	0.10	0.76	2	0.38 ± 1.06	B	0.36	4.6	NS
ab	96.64 ± 1.06	0.34	0.24	2	0.12 ± 1.06	AB	0.11	4.6	NS

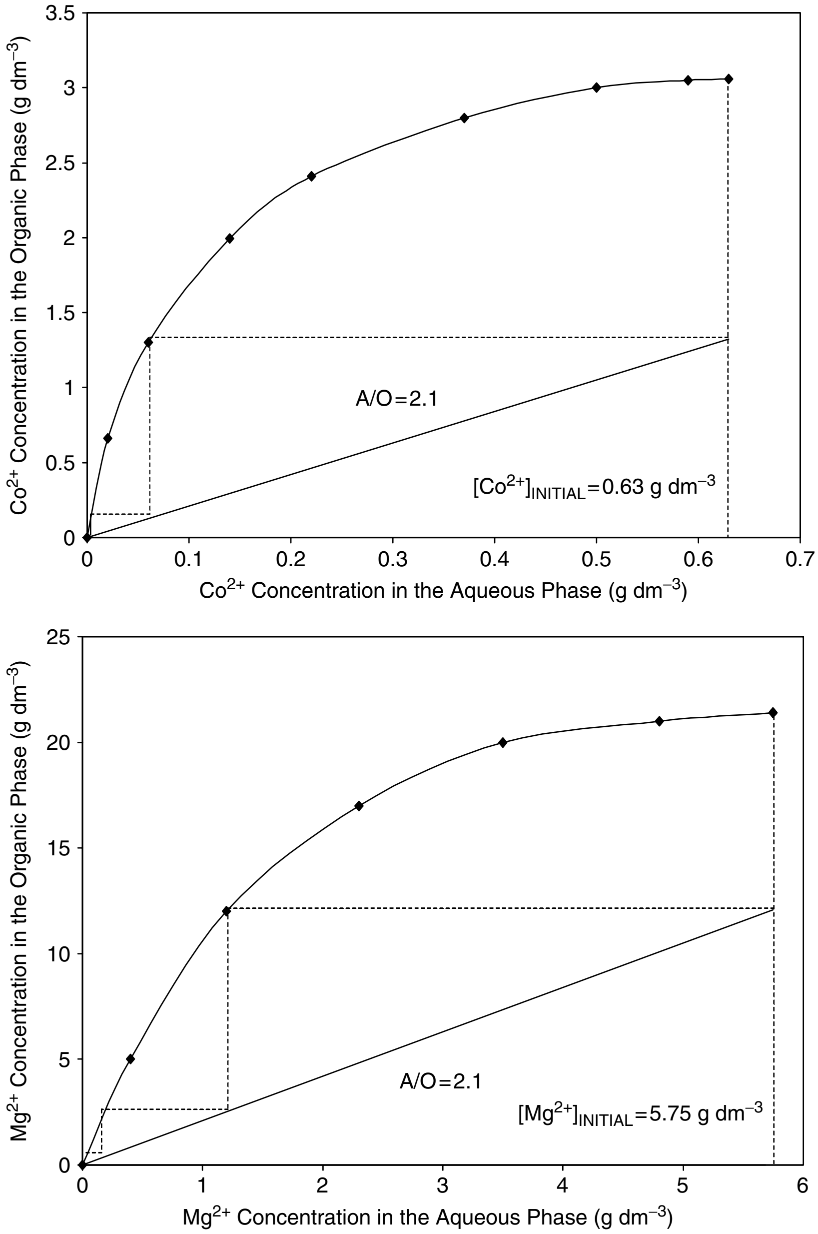

Figure 2. McCabe–Thiele diagrams for Co^{2+} and Mg^{2+} extraction by Ionquest 801.

Construction of Co^{2+} isotherms (extraction–stripping) using Ionquest 801

As Ionquest 801 proved to be an efficient extractant for the simultaneous extraction of cobalt and magnesium from sulfate solutions in the presence of nickel, a McCabe–Thiele diagram was constructed for Co^{2+} and Mg^{2+} with the above reagent, and the number of counter-current stages required was predicted. Cobalt and magnesium extraction isotherms were determined under the optimum conditions indicated by the preceding statistical analysis: Ionquest 801 concentration = 20% (v/v); diluent = Exxsol D-80; extraction pH = 5.0; $T = 40\,°C$. The McCabe–Thiele diagrams indicated two

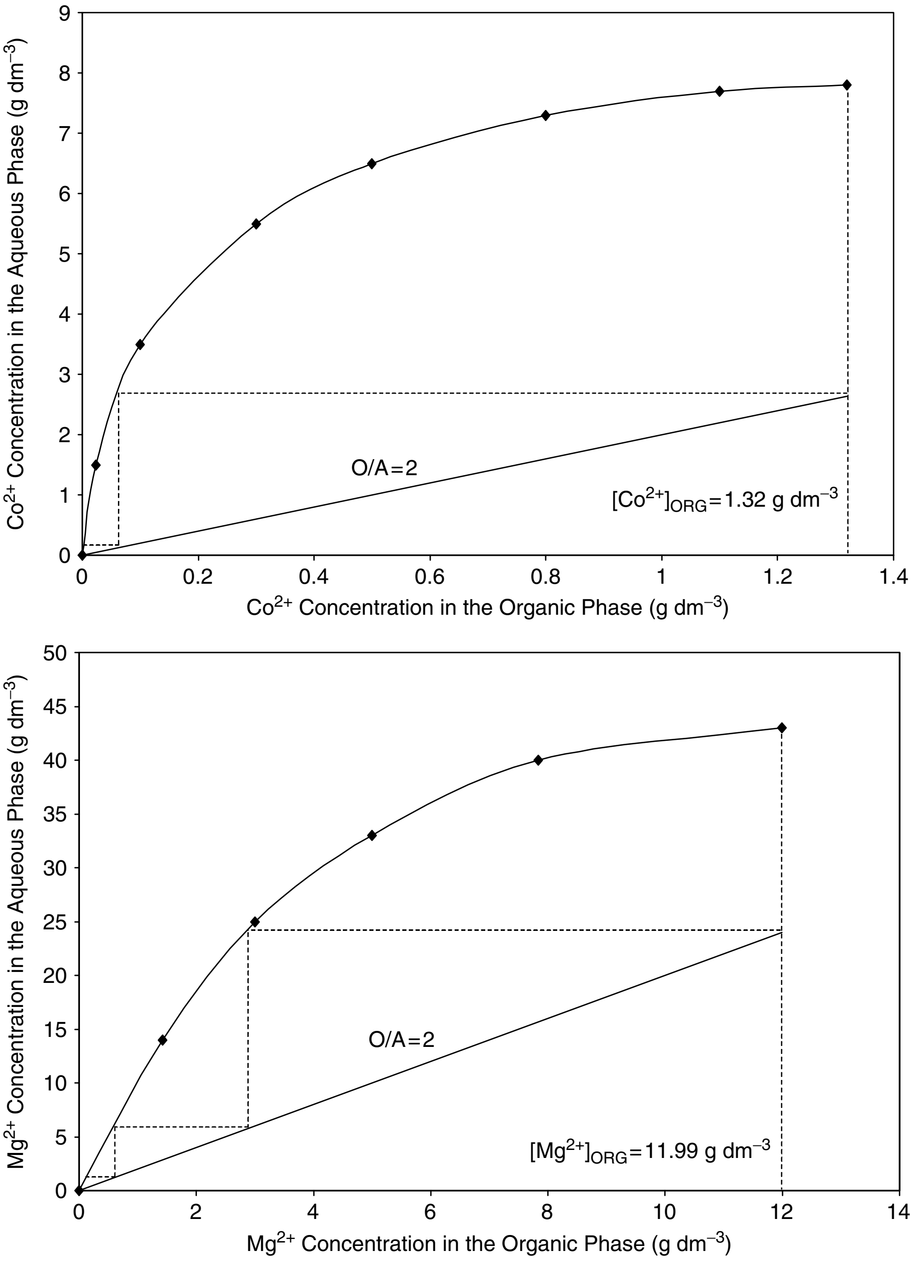

Figure 3. McCabe–Thiele diagrams for Co^{2+} and Mg^{2+} stripping from loaded Ionquest 801 organic phase.

stages of extraction for cobalt and three stages for magnesium at A/O phase ratio = 2.1 (Fig 2). The McCabe–Thiele interpretation for cobalt and magnesium stripping from the loaded organic phase indicated two stages for cobalt recovery and three for magnesium (Fig 3) at $H_2SO_4 = 3\,\text{mol}\,\text{dm}^{-3}$, temperature = 40 °C and O/A phase ratio = 2.

Continuous counter-current test

Continuous tests were carried out in a laboratory horizontal mixer–settler solvent extraction unit (Bell Engineering Model 282). The effective volume was 185 cm^3 for each mixer and 364 cm^3 for the settler. The mixer–settler units and inter-stage piping were constructed of high-density polyethylene (HDPE). Inter-stage solution transfer was by pumping impellers. Solutions were pumped from HDPE holding tanks into the plant using dosing pumps with appropriate tubing. The stirring speed was 850 rpm. The operating conditions for the extraction, scrubbing and stripping of cobalt and magnesium from a nickel sulfate solution, which were carried out in sequence, were:

- solvent: 20% (v/v) Ionquest 801 in Exxsol D-80 with 5% TBP;
- aqueous feed liquor: 0.63 g dm^{-3} Co, 3.8 g dm^{-3} Ni, 5.75 g dm^{-3} Mg as sulfates, pH = 5.0;
- extraction stages: 3;
- scrubbing stages: 1;
- stripping stages: 3;
- temperature: 40 °C;
- running time in each stage: 3 min;
- scrub feed: dilute sulfuric acid (pH 5.0);
- strip feed: 3 mol dm^{-3} H_2SO_4 solution;

- flow rates ($cm^3\ min^{-1}$): aqueous feed 20.57, solvent feed 9.8, scrub feed 9.8, strip feed 4.9.

Under these conditions, cobalt and magnesium extraction by Ionquest 801 in the presence of Ni^{2+} reached 99.7 and 99.5% respectively. The final nickel co-extraction after scrubbing did not exceed 0.5%. Cobalt and magnesium stripping by $3\,mol\,dm^{-3}\,H_2SO_4$ reached 99.7 and 99.3% respectively.

CONCLUSIONS

The results obtained in the present work showed that the extractant Ionquest 801 can be used as a reagent to extract cobalt and magnesium from nickel sulfate solutions.

More specifically, Ionquest 801 diluted in Exxsol D-80 with 5% TBP could extract 95% cobalt and 94% magnesium in one stage at $pH = 5.0$, $T = 40\,°C$, extractant concentration = 20% and A/O phase ratio = 1:1.

After scrubbing with dilute sulfuric acid at $pH = 5.0$, nickel co-extraction in the loaded Ionquest 801 was reduced to 0.5%.

Cobalt and magnesium stripping from the loaded organic phase by $3\,mol\,dm^{-3}\,H_2SO_4$ reached 99 and 97% respectively in one stage at $T = 40\,°C$ and O/A phase ratio = 2:1.

The number of stages required for the extraction of cobalt and magnesium was three at A/O phase ratio = 2.1. In a continuous counter-current operation under the optimum extraction conditions, cobalt and magnesium extraction reached 99.7 and 99.5% respectively. Finally, cobalt and magnesium could be totally stripped in three stages at O/A phase ratio = 2.

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