Calcium and magnesium rejection from sulphate solutions in lateritic nickel solvent extraction using Versatic 10 acid-LIX®84-IC system

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Synopsis

The world mineral industry has over the years experienced growth in the production of nickel (Ni) due to its use not only in the catalytic industry but mainly also to its demand in the stainless steel production industry. Hence there is a need to produce more pure and marketable grades of Ni through the use of efficient purification routes such as solvent extraction. The V10-LIX®84-IC system resulted in nickel (Ni) extraction isotherm shifting to lower pH levels. The pH window of opportunity for the extraction of Ni subsequently increased in solution containing magnesium (Mg) and calcium (Ca) even though the system also resulted in slight shifts to less positive pH values for Mg and Ca curves. Results showed a synergistic effect of LIX®84-IC when added to V10 for all elements, Ni, Mg and Ca. The pH_{50} values for elements Ni, Mg and Ca in the V10-LIX®84-IC system were 2.95, 8.10 and 7.70 respectively. Comparing results obtained when using V10 alone produced ΔpH_{50} for Ni, Mg and Ca being 3.35, 0.15 and 0.70 respectively, which was a confirmation of the synergistic shifts for all the elements. The $\Delta pH_{50}^{(Mg-Ni)}$ value was 1.95 with no synergism and 5.15 with synergism and that for $\Delta pH_{50}^{(Ca-Ni)}$ was 2.10 with no synergism and 4.75 with synergism. This showed that the Ni-Mg separation factor was relatively larger than the Ni-Ca. Improved loading and stripping kinetics were also observed.

Keywords: Nickel solvent extraction separation, magnesium, calcium, versatic 10 acid and LIX®84-IC.

Introduction

The world mineral industry has over the years experienced growth in the production of nickel (Ni) due to its demand in stainless steel production used in consumer goods, chemical processing and construction. More recently, the demand has been invigorated by the growth in China and the industrial growth in Europe, Japan and USA. Another particular use is in the production of hydride electrical storage cells. Figure 1 shows the world nickel production statistics since 1900, which illustrates a general increase in both production and price. Hence there is a need to produce more pure and marketable grades for Ni through the use efficient purification routes such as solvent extraction.

Extraction routes for Ni can be pyrometallurgical and/or hydrometallurgical, with the former having been replaced gradually by the later method due to expected savings on capital and operating costs as well as a way of exercising more environmentally friendly metallurgical works. Various processes and methods have been developed to recover Ni from either the sulphide concentrates or its oxide ores, laterites, with oxide ores accounting for 70% of the world's Ni resource but only contributing 30% of the world's Ni requirements. Laterite ores are regarded as oxidized sulfides that have undergone the weathering process to form oxides because of being closer to the surface. Laterite ore processing now accounts for the growing proportion of Ni production due to the depletion of known sulphide deposits and also the abundance of known, unexploited laterite resource.

Laterites are generally low grade ores containing high amounts of acid consuming gangue. Laterite ores are difficult to concentrate using the normal reduction roast-leaching method but new technologies such as high pressure acid leach (HPAL) have made Ni recoveries from these ores more attractive. Thus, a growing interest in Ni extraction from laterite ores through HPAL and solvent extraction-electrowinning process has since been established.

There are five well-known processes that treat laterite ores by solvent extraction as the main purification step. These include the Cawse project, Bulong, Murrin Murrin, Goro and Queensland Nickel processes. All except the Queensland process, which uses the roast-leach process, use HPAL method although the down-stream processes for the separation of Ni from impurities differ for each one of them (Denegan, 2006; Sole, 2001 and 2007; Tsakiridis, 2003 and 2004).

Solvent extraction (SX) plays a major part in a hydrometallurgical Ni processing. SX is a method used for the removal, separation and concentration of metallic species in an aqueous

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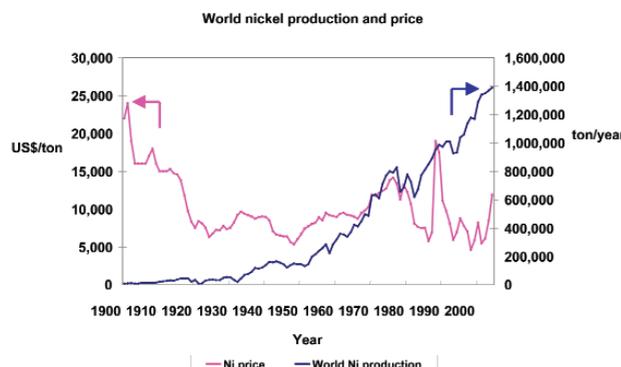
Figure 1—World nickel production and prices since 1900 (Goonan T.G. et al., 2006)

media. Common impurities dissolved in solution include calcium (Ca), zinc (Zn), magnesium (Mg), iron (Fe) and manganese (Mn). For Mg, up to 16g/ℓ in solution has been found to result from leaching of nontronitic clays, a laterite ore, in the Bulong process (Karadakis *et al.*, 2005) prior to Ni solvent extraction. Calcium also occurs in significant amounts and thus the removal of these two elements is essential to avoid build-up in recirculating streams.

Examples of extractants mostly used in the recovery of Ni in solution are Versatic 10 (V10), di-2-ethylhexyl phosphoric acid (D2EHPA) and Cyanex 272 and 301. V10 has been found to give very positive results and thus been extensively used in major Ni SX works. However, V10, a carboxylic acid, displays relatively poor selectivity towards Ni over some common impurities such as Mg, Ca and ferrous Fe (Preston *et al.*, 2000) when used as a standalone reagent. Furthermore, V10 has a disadvantage of high water solubility at elevated pH levels of 6–7 required for Ni recovery; 150 ppm at pH 6.5 (Schekers *et al.*, 2006). Extraction at low pH levels will vastly reduce its losses.

The search for alternative extractants and the general reluctance to develop new reagents by manufacturers due to costs has led to the development of mixtures of solvents with improved extraction power and enhanced mass transfer and kinetics through a phenomenon called synergism. Numerous synergistic systems have been studied, which use V10 as the major extractant due to its relatively improved selectivity, availability and low cost when mixed with solvents such as alkylpyridines (Preston *et al.*, 2000) and hydroxyoximes such as LIX®63 (Cheng, 2006).

Synergism between a ketoxime and V10 has not yet received much attention even though previous reports declined this possibility due to assumed significant interaction between the oxime and V10 via hydrogen-bond formation, which reduces the amount of free extractant availability (Preston, 1983). This observation was the driver of the current investigation. Contrary to the paper by Preston (1983), an investigation into the possibility of using a ketoxime, LIX®84-I, as an extractant in Ni SX proved successful (Mackenzie *et al.*, 2006). In as much as LIX®84-I has extensive use in copper SX circuits (Sole, 2007), its use in HPAL Ni laterite treatment is equally significant in processes such as the Cawse and the Queensland. LIX®84-I based circuits have emerged as strong candidates for many new laterite nickel projects (Mackenzie *et al.*, 2006). For this reason, LIX®84-I, also known as 2-hydroxy-5 nonylace-

tophenone oxime, can be considered to be an effective extractant, which may be enhanced in a synergistic system with an equally powerful reagent such as V10.

Although Co in many Ni extraction circuits is a by-product, it is regarded as an impurity when it comes to Ni solvent extraction. Selectivity for Ni in the presence Co is poor. However, improvements can be made by controlling the oxidation state of Co (Sole, 2007). Cobalt co-loading in Ni SX by LIX®84-I causes organic degradation in the process (Mackenzie *et al.*, 2006). Prior Co removal is therefore essential and this can be done by using Cyanex 272 (Tsakiridis, 2003 and 2004). The current work adopted a system where Co has been removed and the only impurities present are Mg and Ca. From this criterion, the Bulong process fits into this description as it also processed sulphate based solutions.

Experimental plan

Organic solution

Versatic 10 (V10) acid used in this study was supplied by Chem Quest South Africa. V10 is a synthetic C10 carboxylic acid, also known as Neodecanoic acid. It is derived from a C9 olefin and displays specific branching at the alpha position and has a high tertiary acid content, whilst also exhibiting a low acid strength and outstanding solubility in organic diluents, and remaining negligibly soluble in water (Versatic 10 product data sheet, Chem quest).

The structure of V10 may be represented as follows:
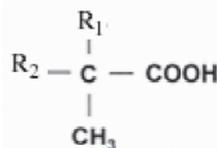
where R_1 and R_2 are alkyl groups, containing 9 carbon atoms together.

The molecular weight is 172 and the density (24°C) is 0.91 g/cm³. The working pH of V10 is generally between 4 and 8. The order of extraction of metal cations by V10 follows the order of hydrolysis constants of metal ions (Tsakiridis *et al.*, 2004).

LIX®84-IC was supplied by the Cognis Corp. South Africa (SA). LIX®84-IC falls under the LIX®84-I series, which includes LIX®84-INS. These reagents are a blend of the ketoxime 2-hydroxy-5-nonylacetophenone oxime in a hydrocarbon diluent. LIX®84-IC is a concentrated version of LIX®84-IC, concentrated by a factor of 1.4 on volume basis.

The structure of LIX®84-IC may be represented as follows:
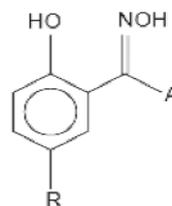
where A is CH₃ and R is C₉H₁₉ or C₁₂H₂₅

The molecular weight of LIX®84-IC is 263 and has a density (25°C) of around 0.94 g/cm³.

Both extractants were used without further purification. The organic solution was prepared by diluting V10 and LIX®84-IC to 0.5 M in xylene obtained from Merck Chemicals SA.

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Aqueous solution

The test was specifically performed to a lateritic HPAL type leach solution prior to electrowinning. An assumption made was that most of the Co would have been removed earlier in the process. A synthetic Bulong process Ni SX feed containing 3.0 g/ℓ Ni²⁺, 15.0 g/ℓ Mg²⁺ and 0.5 g/ℓ Ca²⁺ was prepared by dissolving AR grade NiSO₄.6H₂O, AR grade MgSO₄.7H₂O and AR CaSO₄.2H₂O respectively in distilled water. These chemicals were supplied by Merck Chemical SA. No initial pH adjustments were made to the solution except on equilibrium.

Shake-out tests

All batch tests were conducted at 40°C at an O:A ratio of 1:1. A 200 ml reactor was used for mixing the organic and the aqueous solutions using a magnetic stirrer using a 20 mm magnetic follower to stir at 750 rpm. Analytical grade sulphuric acid (H_2SO_4) and sodium hydroxide (NaOH) were used to adjust the pH of the solution as required. After mixing for 15 minutes, a predetermined time, the mixture was allowed to settle for 3 minutes to separate. A 20 ml sample of the aqueous phase was taken for pH measurement and the pH adjusted by either addition of H_2SO_4 or NaOH. After mixing for another 15 minutes, the mixture was allowed to separate and the pH was measured and adjusted. The test was continued until the equilibrium pH was obtained, after which an aqueous solution sample was taken for chemical analysis. The schematic experiment set-up is shown on Figure 2.

Chemical analysis

All the chemical analyses were conducted on the aqueous phase using a GBC909 atomic adsorption (AA) machine. For results, verification purposes, some samples were submitted to an external laboratory, UIS Analytical Services, which used the ICP type analysis by OES Perkin Elmer Optima 3000 machine. The amount of metal loaded on the organic was calculated as the difference between the initial concentration and that from aqueous solution analysis. The % extraction was calculated by the following formula:

$$\%E = \frac{n_{M,aq,i} - n_{M,aq,f}}{n_{M,aq,i}} \times 100 \qquad [1]$$

where
$\%E$ = Extraction (%)
$n_{M,aq,i}$ = Initial amount of metal in aqueous solution
$n_{M,aq,f}$ = Final amount of metal in aqueous solution (raffinate)

Results and discussion

pH-metal extraction isotherms

pH-extraction isotherms for Ni, Mg and Ca were determined by shake-out tests at different equilibrium pH values with O:A ratio of 1:1. The pH-extraction isotherms at 40°C of the three elements of interest were determined for 0.5 M V10 (Figure 3), 0.5 M LIX®84-IC (Figure 4) as well as a mixture of 0.5 M V10 and 0.5 M LIX®84-IC (Figure 5). The V10-LIX®84-IC system resulted in the shift of the Ni pH-extraction isotherm to lower pH levels, thereby widening the gap between Ni and the impurities Mg and Ca. A pH window of opportunity was observed to be between pH 5 and 6 (Figure 5). The explanation was assumed to be due to a

higher availability of LIX®84-IC to extract Ni than V10 only. Two moles of LIX®84-IC (Sole, 2007) as compared to four moles of V10 are required to take up a single Ni mole. This is illustrated by Equations [2] and [3] for LIX®84-IC loading and V10 respectively.

$$\text{LIX®84-IC} : 2RH_{(org)} + Ni^{2+}_{(aq)} \rightarrow R_2Ni_{(org)} + 2H^+_{(aq)} \qquad [2]$$

$$\text{V10:} 4RH_{(org)} + Ni^{2+}_{(aq)} \rightarrow R_2Ni2RH_{(org)} + 2H^+_{(aq)} \qquad [3]$$
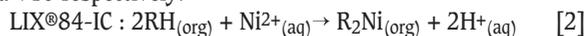
The fact that the LIX®84-IC curves are at relatively higher pH values than the V10-LIX®84-IC mixture is evidence that LIX®84-IC is not the only active reagent to extract Ni but a synergistic effect resulted due to the addition of LIX®84-IC to V10. A shift to lower pH values has an added benefit of reducing V10 losses to the aqueous solution and also reduces the cost of neutralizing reagent additions. Verification of results was done by sending a set of samples to an external
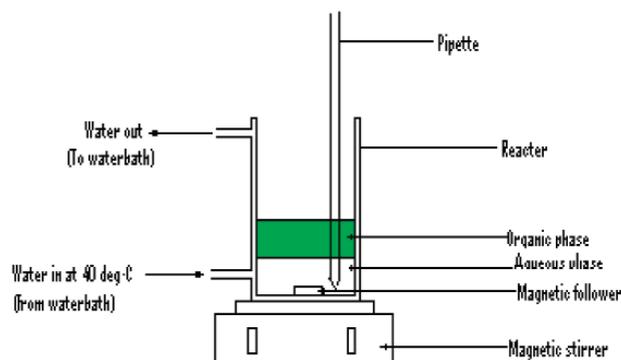

Figure 2—Experimental set-up
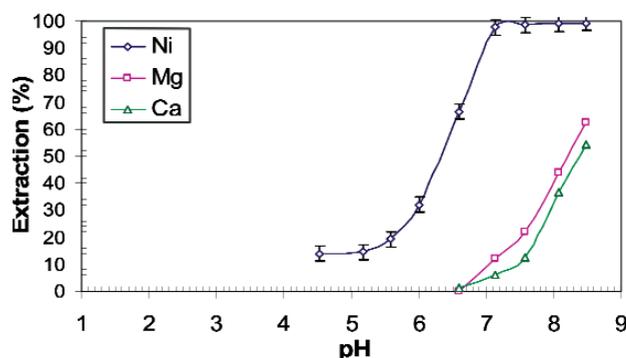
Figure 3—pH dependence of 0.5 M V10 in xylene with Ni, Mg and Ca at 40°C with O:A = 1:1
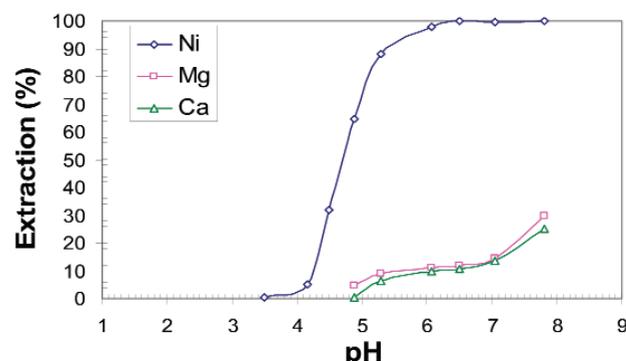

Figure 4—pH dependence of 0.5 M LIX®84-IC in xylene with Ni, Mg and Ca at 40°C with O:A = 1:1
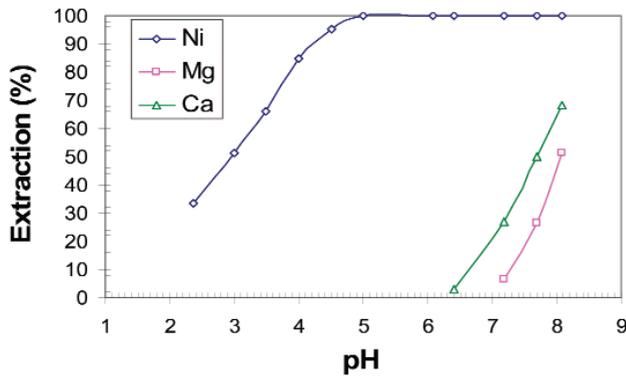
Figure 5—Extraction pH isotherms of Ni, Mg and Ca by a mixture of 0.5 M V10 and 0.5 M LIX®-IC in xylene at 40°C with O:A = 1:1

Figure 6—Validation of results obtained from experiment (AA machine) with those from an external laboratory UIS (ICP)

laboratory, UIS analytical laboratories. A comparison between the results obtained from the AA and those supplied by UIS is shown in Figure 3, in the form of error bars, and Figure 6. It is apparent that the two sets of results are relatively in good agreement.

Further analysis was done by plotting the response for individual elements Ni (Figure 7), Mg (Figure 8) and Ca (Figure 9) to V10, LIX®84-IC as well as the mixture of V10 and LIX®84-IC. These plots were matched to those from previous studies in which V10 diluted in Shellsol 2046 (S2046) was used as an extractant at 40°C with O:A ratio of 1:1 (Denegan, 2006). It is important to note that comparison was done with a system that used a different diluent, S2046 instead of xylene. However, it has been reported by Preston (2000) that the extraction of Ni is not greatly affected by the diluent in the organic phase (Preston, 2000). The plots showed great similarities in isotherms involving extractions by 0.5 M V10 for both the experiment and those by Denegan. The significant shift in Ni extraction isotherm to lower pH levels was clearly illustrated in the V10-LIX®84-IC system as well as slight shifts to less positive pH values for Mg and Ca curves, with Ca having a major shift. This showed a synergistic effect of LIX®84-IC for all elements Ni, Mg and Ca.

The pH_{50} (the pH for 50% extraction) values for Ni, Mg and Ca in the V10-LIX®84-IC system are shown on Table I, with all values being positive. This was a confirmation of the synergistic shifts observed of all the elements under consideration.

The overall separation factors between Ni and the impurities considered were shown by the ΔpH_{50} (metal-Ni) (the difference between the pH values for 50% extraction of the impurity and Ni before and after LIX®84-IC addition) values. Table II is a summary of the results. The difference in ΔpH_{50} (Metal-Ni) values show that the Ni-Mg separation factor is relatively larger than that of Ni-Ca.

Effect of variables

In order to obtain the optimum conditions for extracting Ni and effectively rejecting Ca and Mg, a factorial design was performed to investigate the effects of variables on extraction. Firstly the pH was fixed at pH 4 and the effect of LIX®84-IC concentration on extraction was determined at a constant V10 concentration of 0.5 M and fixed temperature of 40°C. The effect of various combinations on a two-level variable basis was later tested at a fixed temperature of 40°C. For a chosen combination of pH, LIX®84-IC concentration and O:A ratio temperatures of 25, 35, 40 and 50°C were eventually tested.
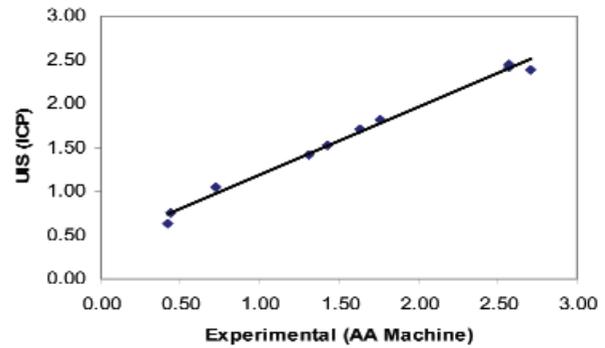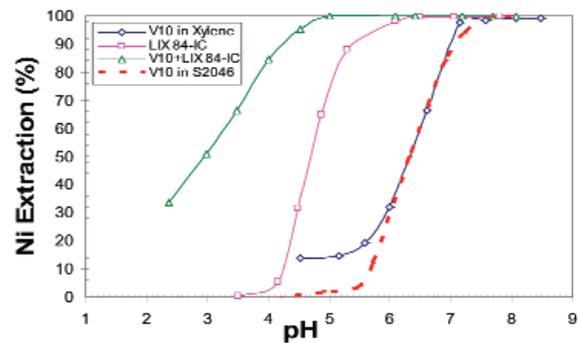

Figure 7—pH dependence of 0.5 M V10 in xylene, 0.5 M LIX®84-IC in xylene, 0.5 M V10 and 0.5 M LIX®84-IC in xylene and 0.5 V10 in Shellsol 2046 for Ni at 40°C with O:A = 1:1
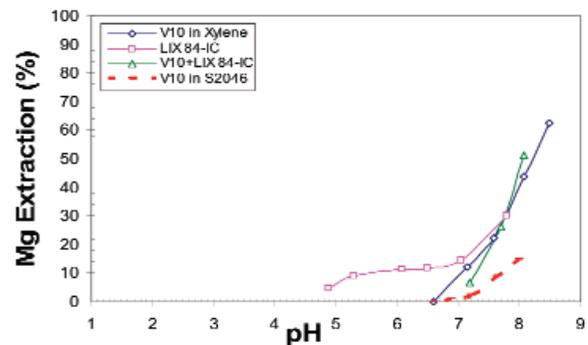

Figure 8—pH dependence of 0.5 M V10 in xylene, 0.5 M LIX®84-IC in xylene, 0.5 M V10 and 0.5 M LIX®84-IC in xylene and 0.5 V10 in Shellsol 2046 for Mg at 40°C with O:A = 1:1
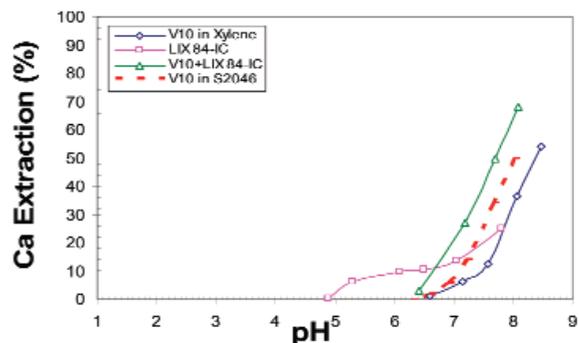

Figure 9—pH dependence of 0.5 M V10 in Xylene, 0.5 M LIX®84-IC in Xylene, 0.5 M V10 and 0.5 M LIX®84-IC in xylene and 0.5 V10 in Shellsol 2046 for Ca at 40°C with O:A = 1:1

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Table I

Experimental results for the 0.5 M V10-0.5LIX®84-IC system at 40°C with O:A = 1:1

0.5 M V10	pH	Experiment		
		Ni	Mg	Ca
No Synergism	pH$_{50}$	6.30	8.25	8.40
0.5M LIX® 84-IC	pH$_{50}$	2.95	8.10	7.70
	ΔpH$_{50}$	3.35	0.15	0.70

LIX®84-IC concentration effects

Figure 10 shows the effect of LIX®84-IC concentration at pH 4. There is a general increase in Ni extraction with LIX®84-IC concentration accompanied by a simultaneous decrease in Mg and Ca extractions. This shows the high selectivity of the system for Ni over Mg and Ca. Furthermore, results showed that there is no need to increase LIX®84-IC concentration beyond 0.45 M, otherwise any benefits in terms of extraction can only be realized at 0.6 M.

Various combinations of variables

A two-level statistical design was performed to obtain the optimum conditions. Table III shows the factors, variables and the levels investigated for various combinations for a fixed temperature of 40°C. The fixed factors that were considered were;

➤ 0.5 M V10
➤ 0.5 M LIX®84-IC
➤ pH 5.00
➤ O:A ratio of 1:1

The results for the unique combinations for different treatment codes are shown in Table IV. The 'bc' treatment code with 0.5 M V10, 0.45 M LIX®84-IC, pH 5.00 and O:A ratio of 1:2 was chosen as the one which gave optimum condition due to low amounts of Mg and Ca being co-extracted with Ni. The other benefits of this combination are the cost savings on organic utilization since the concentration of LIX®84-IC used is a low 0.45 M at an O:A ratio of 1:2.

Effect of temperature

The effect of temperature was then studied for the 'bc' combination. Figure 11 illustrates the plot obtained from this test. There is a general increase in Ni extraction with temperature. The results showed that the optimum reasonable temperature to work at is 40°C bearing in mind that working at 50°C may results in extensive evaporation of the organic phase, resulting in a relative increase in organic losses.

Overall 0.5 M V10, 0.45 M LIX®84-IC, pH 5.00, O:A ratio of 1:2 and temperature of 40°C were considered as the optimum conditions for Ni extraction for this particular study. Loading and stripping kinetics were then investigated under these conditions.

Kinetics

Loading

Loading experiments were conducted at 0.5 M V10, 0.45 M LIX®84-IC, pH 5.00, O:A ratio of 1:2 at a temperature of 40°C. Figure 12 shows the results of this test. In comparison to the V10-LIX®63 system (Cheng, 2006), the V10-LIX®84-IC has improved kinetics with 95% extracted within 1 minute as compared to approximately 60% for the V10-LIX®63 system.

Table II

Results for the V10 alone (no synergism) and the V10-LIX®84-IC system at 40°C with O:A = 1:1

	0.5 V10		Difference (synergism – no synergism)
	No synergism	0.5 M LIX®84-IC	
ΔpH$_{50}$(Mg-Ni)	1.95	5.15	3.20
ΔpH$_{50}$(Ca-Ni)	2.1	4.75	2.65
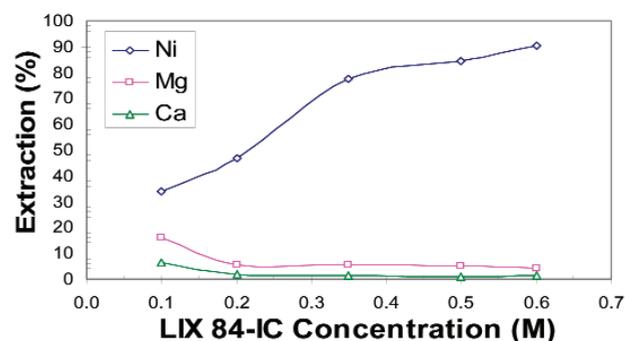
Figure 10—Effect of LIX®84-IC concentration on Ni, Mg and Ca extraction at pH 4 and T = 40°C

Table III

Treatment code, variable and level of investigation

Factor	Variable	Level	
		High	Low
a	pH	5.00	4.50
b	[LIX 84-IC]	0.50	0.45
c	O:A	1.00	0.50

Table IV

Experiment results at 40°C for the different treatment codes to investigate the effects of variable levels

Treatment Code	[V 10]	[LIX 84-IC]	pH	O:A	Temp (°C)	Ni %Extraction	Mg %Extraction	Ca %Extraction
a	0.50	0.50	5.00	1:1	40.00	99.92	14.43	0.00
b	0.50	0.45	5.00	1:1	40.00	99.95	5.37	11.25
ab	0.50	0.45	4.50	1:1	40.00	99.66	3.10	3.13
c	0.50	0.50	5.00	1:2	40.00	99.71	65.96	65.00
ac	0.50	0.50	4.50	1:2	40.00	96.50	8.77	13.13
bc*	0.50	0.45	5.00	1:2	40.00	99.67	3.25	2.50
abc	0.50	0.50	4.50	1:1	40.00	99.79	14.47	12.35

* Chosen combination

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Stripping

Stripping kinetics investigations were conducted on the loaded organic, loaded under the earlier determined optimum conditions. The loaded organic solution was initially scrubbed with a solution containing 15 g/ℓ Ni at an A:O ratio 4:1. No pH adjustments were made. Depending on the availability of the loaded organic solution, between 30–40 ml, stripping kinetic tests were conducted. As compared to the V10-LIX®63 system (Cheng, 2006), improved stripping kinetic on the loaded V10-LIX®84-IC system were obtained at an A:O of 1:1 for the 40°C temperature. Figure 13 shows the results obtained. Within 5 minutes 95% of Ni was stripped as compared to the 32% in the V10-LIX®63 system.
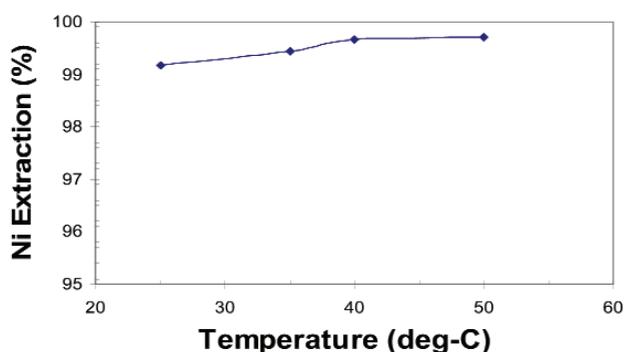

Figure 11—Effect of temperature on Ni extraction by the 0.5 M V10-0.45 M LIX®84-IC in xylene at pH 5.00 with O:A = 1:2
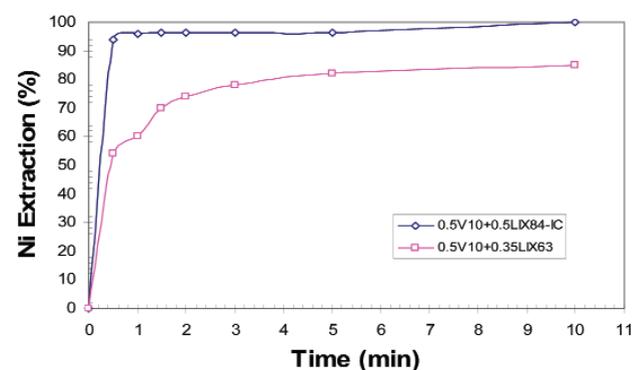

Figure 12—Nickel loading kinetics for V10-LIX®63 at O:A ratio 1:1 and V10- LIX®84-IC systems at 40°C with O:A = 1:2 at pH 5.00
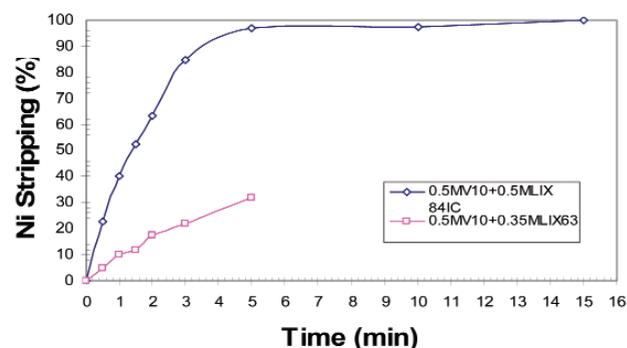

Figure 13—Nickel stripping kinetics for V10-LIX®63 and V10-LIX®84-IC systems at 40°C with A:O = 1:1 at pH 5.00

Conclusion

The conclusions drawn up are as follows:

➤ LIX®84-IC has a synergistic effect when mixed with V10 for Ni, Mg and Ca.

➤ The Ni extraction curve was shifted most significantly while those for Mg and Ca were slightly shifted to lower pH levels when LIX®84-IC was added to V10. This showed a higher selectivity for Ni by the V10-LIX®84-IC system. A pH window of opportunity between pH 5 and 6 results in 100% Ni extractions.

➤ Improved loading and stripping kinetics were observed.

➤ Aspects of costs of using a combination of the two reagents for possible commercial use can also be looked at. The system may offer savings on organic reagent costs since an O:A ratio of 1:2 can give equally good results. Another benefit to this system is the extraction of Ni at low pH values (100% extraction pH 5 to 6), which also offers savings on neutralization reagent additions compared to working at typical Ni SX pH levels of around 6.5 or more.

➤ Further test work is required in order to consolidate these results.

Acknowledgements

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