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

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Abstract

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 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, magnesium, calcium, versatic 10 acid and LIX®84-IC.

1. 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 U.S.A. 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 a need to produce more pure and marketable grades for Ni through the use efficient purification routes such as solvent extraction.
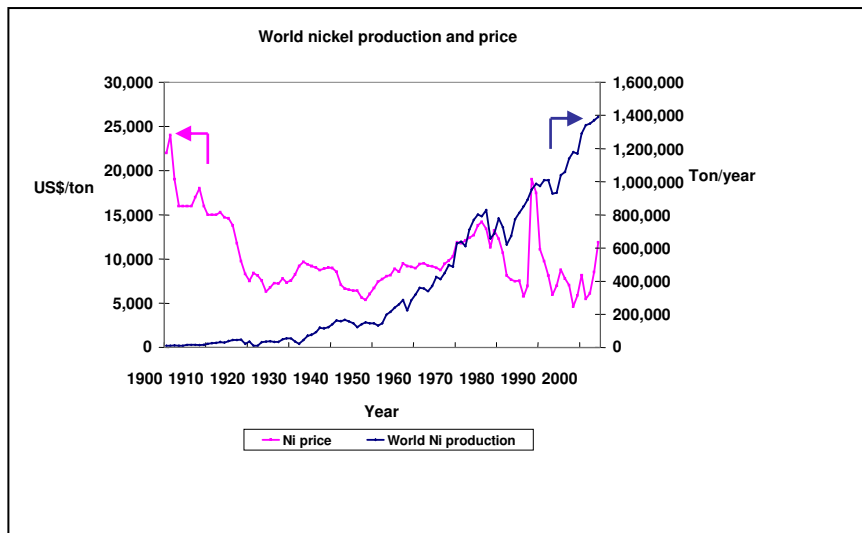
Figure 1 World nickel production and prices since 1900 (*Goonan T.G. et al, 2006*)

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 which 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 which 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 the hydrometallurgical Ni processing. SX is a method used for the removal, separation and concentration of metallic species in an aqueous 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 found 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 stand alone reagent. Further more, V10 has a disadvantage of high water solubility at elevated pH levels of 6-7 required for Ni recovery; 150ppm 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 into developing mixtures of solvents with improved extraction power and enhanced mass transfer and kinetics through a phenomenon known as 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 to the current investigation. On the contrary to the paper by Preston (1983), an investigation in 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 sort out extensive use in copper SX circuits (Sole, 2007), its use in HPAL Ni laterite treatment is equally significant in process 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 nonylacetophenone 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 of for Ni in the presence Co is poor. However improvements can be done 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 use of Cyanex 272 (Tsakiridis, 2003 and 2004). The current work adopted a system where Co has been removed and the only impurities present being Mg and Ca. From this criterion, the Bulong process fits into this description as it also processed sulphate based solutions.

# 2. Experimental Plan

2.1 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:
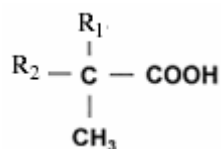
where R_1 and R_2 are alkyl groups, containing 9 carbon atoms together.

The molecular weight is 172 and the density (24 °C) is 0.91 g/cm^3. 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 (S.A.). 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:
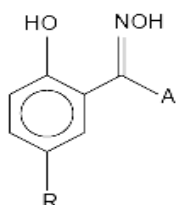
where A is CH_3 and R is C_9H_{19} or $C_{12}H_{25}$

The molecular weight of LIX$^®$84-IC is 263 and has a density (25 °C) of around 0.94 g/cm^3.

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

2.2 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 Ni^{2+}, 15.0 g/l Mg^{2+} and 0.5 g/l Ca^{2+} was prepared by dissolving AR grade $NiSO_4.6H_2O$, AR grade $MgSO_4.7H_2O$ and AR $CaSO_4.2H_2O$ respectively in distilled water. These chemical were supplied by Merck Chemical S.A. No initial pH adjustments were made to the solution except on equilibrium.

2.3 Shake-out tests

All batch tests were conducted at 40 °C at an O/A ratio of 1/1. A 200ml reactor was used for mixing the organic and the aqueous solutions using a magnetic stirrer using a 20mm 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 min, a pre determined time; the mixture was allowed to settle for 3 min to separate. A 20 ml sample of the aqueous phase was taken for pH measurement and pH adjusted by either addition of H_2SO_4 or NaOH. After mixing for another 15 min, 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 setup is shown in Figure 2.
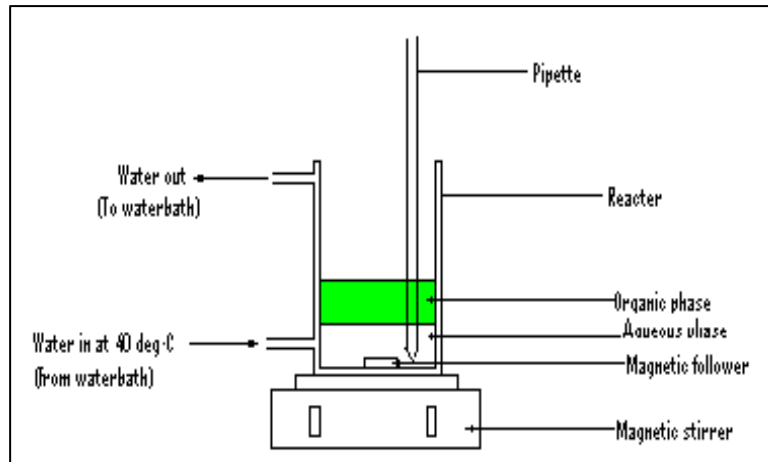
Figure 2: Experimental setup

2.4 Chemical analysis

All the chemical analysis was 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 gotten from aqueous solution analysis. The % Extraction was calculated by the following formula:

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

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)

3. Results and discussion

3.1 pH-Metal extraction isotherms

The pH-extraction isotherms for Ni, Mg and Ca were determined by shakeout 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 0.5M V10 (Figure 3), 0.5M LIX®84-IC (Figure 4) as well as a mixture of 0.5M V10 and 0.5M 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 and 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.
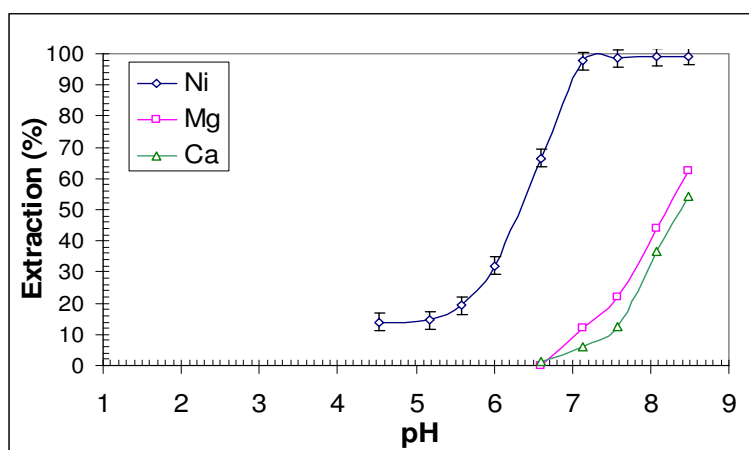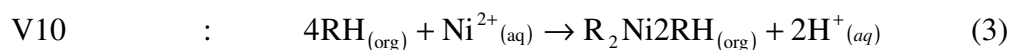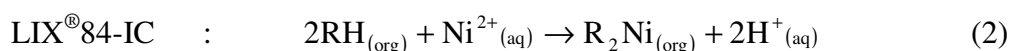
$$\text{LIX}^{\circledR}\text{84-IC} \quad : \quad 2RH_{(org)} + Ni^{2+}{}_{(aq)} \rightarrow R_2Ni_{(org)} + 2H^+{}_{(aq)} \qquad (2)$$

$$\text{V10} \quad : \quad 4RH_{(org)} + Ni^{2+}{}_{(aq)} \rightarrow R_2Ni2RH_{(org)} + 2H^+{}_{(aq)} \qquad (3)$$
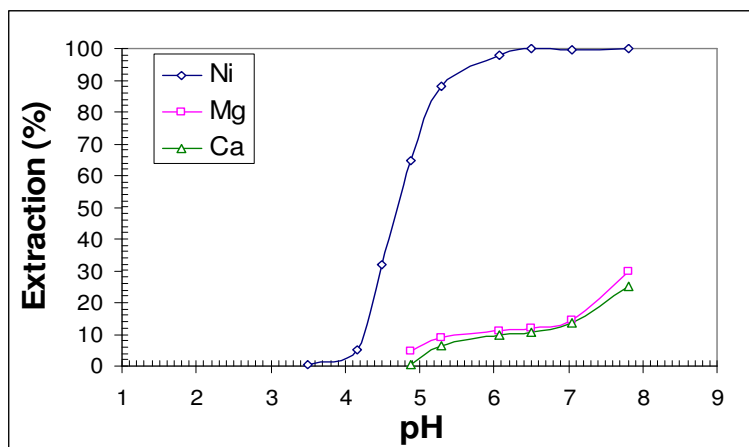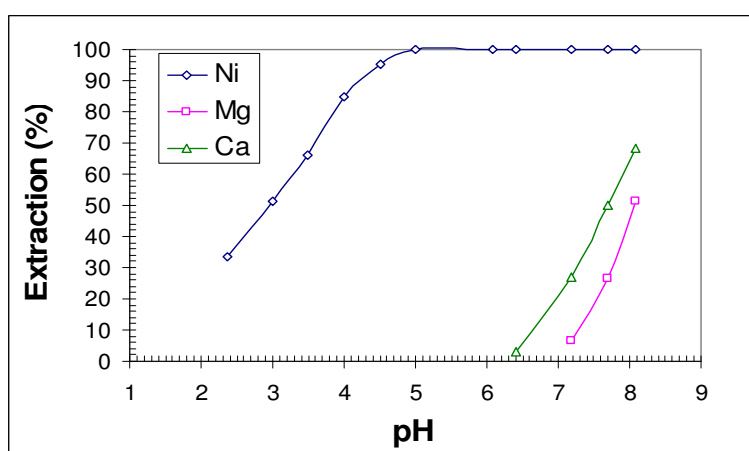

**Figure 3:** pH dependence of 0.5M V10 in xylene with Ni, Mg and Ca at 40 °C with O/A = 1/1

Figure 4: pH dependence of 0.5M 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 0.5M V10 and 0.5M LIX®84-IC in xylene at 40 °C with O/A = 1/1

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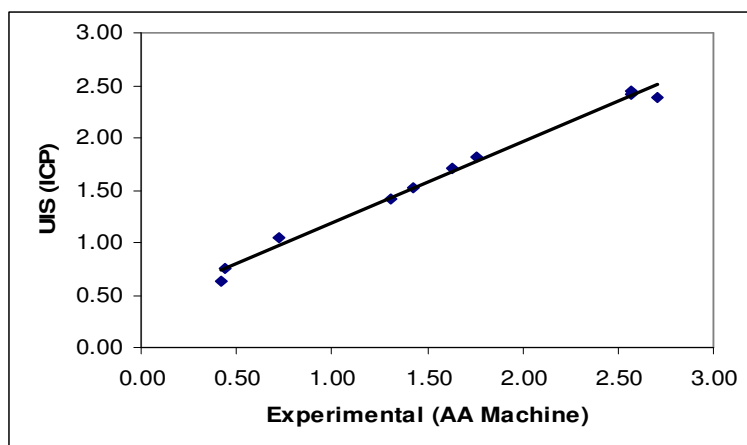
Figure 6: Validation of results obtained from experiment (AA machine) with those from an external laboratory UIS (ICP)

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 to a system which 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 0.5M 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.
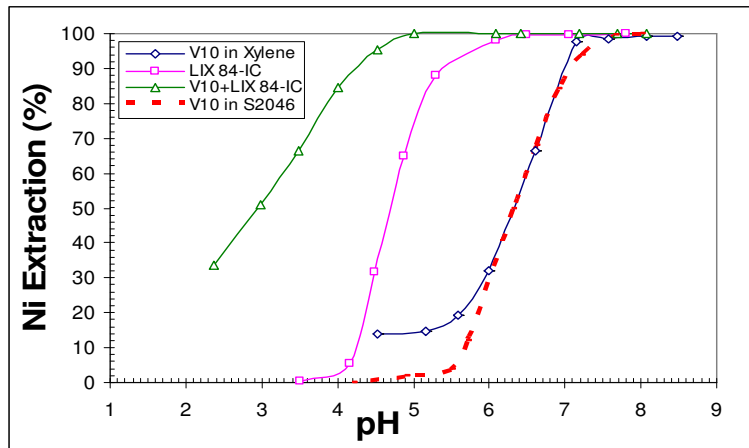
Figure 7: pH dependence of 0.5M V10 in xylene, 0.5M LIX®84-IC in xylene, 0.5M V10 and 0.5M LIX®84-IC in xylene and 0.5V10 in Shellsol 2046 for Ni at 40 ˚C with O/A=1/1
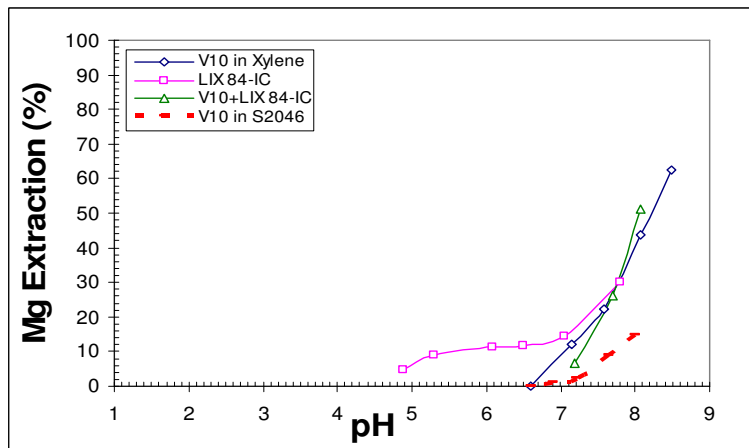
Figure 8: pH dependence of 0.5M V10 in xylene, 0.5M LIX®84-IC in xylene, 0.5M V10 and 0.5M LIX®84-IC in xylene and 0.5V10 in Shellsol 2046 for Mg at 40 ˚C with O/A=1/1
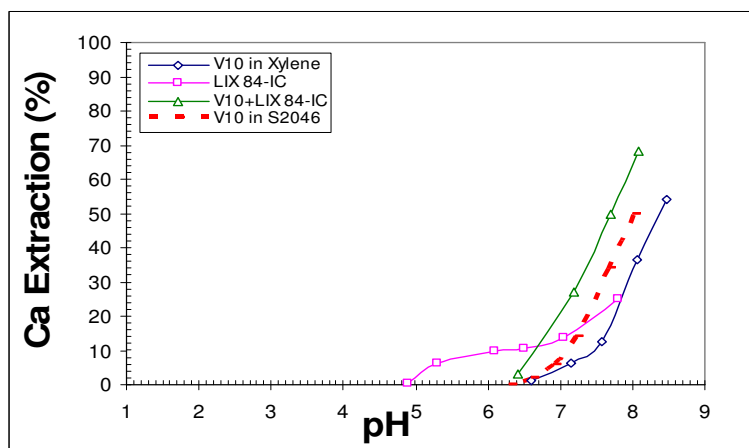
Figure 9: pH dependence of 0.5M V10 in Xylene, 0.5M LIX®84-IC in Xylene, 0.5M V10 and 0.5M LIX®84-IC in xylene and 0.5V10 in Shellsol 2046 for Ca at 40 ˚C with O/A=1/1

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 1 with all values positive. This was a confirmation of the synergistic shifts observed to all the elements under consideration.

Table 1: Experiment results for the 0.5M V10-0.5LIX®84-IC system at 40˚C with O/A=1/1

0.5M 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

The overall separation factors between Ni and the impurities considered were determined by the $\Delta pH_{50}^{(metal-Ni)}$ (the difference between the pH values for 50% extraction of the impurity and Ni before and after LIX[®]84-IC addition) values. Table 2 is a summary of the results. The difference in $\Delta pH_{50}^{(Metal-Ni)}$ values show that the Ni-Mg separation factor is relatively larger than that of Ni-Ca.

Table 2: Results at 40 °C for the different treatment codes to investigate the effects of variable levels

|	0.5V10		Difference (Synergism - No synergism)
	No Synergism	0.5M LIX[®]84-IC	
$\Delta pH_{50}^{(Mg-Ni)}$	1.95	5.15	3.20
$\Delta pH_{50}^{(Ca-Ni)}$	2.1	4.75	2.65

3.2 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 0.5M 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.

3.2.1 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 0.6M.
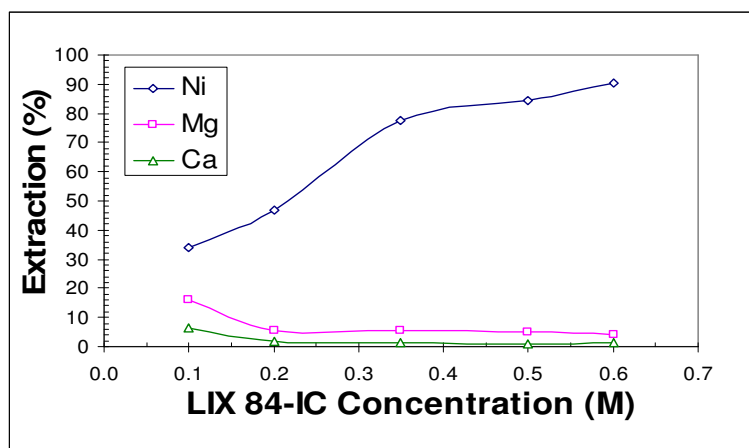
Figure 10: Effect of LIX®84-IC concentration on Ni, Mg and Ca extraction at pH 4 and T= 40 °C

3.2.2 Various combinations of variables

A two level statistical design was performed to obtain the optimum conditions. Table 3 shows the factors, variables and the levels investigated for various combinations for a fixed temperature of 40 °C. The fixed factors which were considered were;

1. 0.5M V10
2. 0.5M LIX®84-IC
3. pH 5.00
4. O/A ratio of 1/1

Table 3: 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

The results for the unique combinations for different treatment codes are shown in Table 4. The 'bc' treatment code with 0.5M V10, 0.45M 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 0.45M at an O/A ratio of 1:2.

Table 4: Results for the V10 alone (no synergism) and the V10-LIX®84-IC system at 40˚C with O/A=1/ 1

Treatment Code	[V 10]	[LIX 84-IC]	pH	O/A	Temp (℃)	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

3.2.3 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 relative increase in organic losses
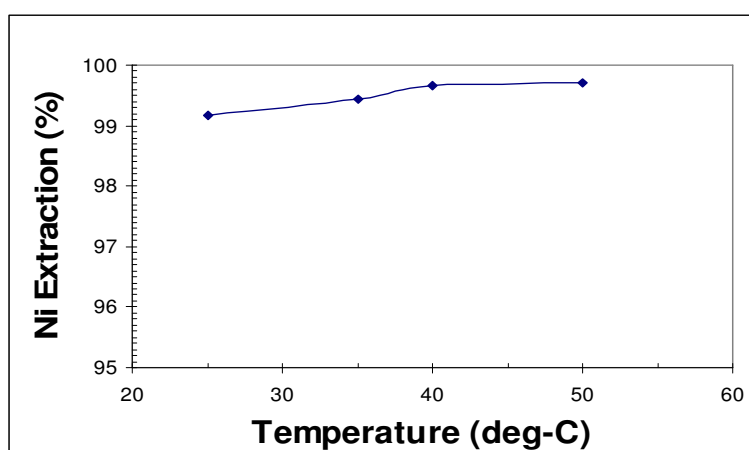

Figure 11: Effect of temperature on Ni extraction by the 0.5M V10-0.45M LIX®84-IC in xylene at pH 5.00 with O/A=1:2

Overall 0.5M V10, 0.45M 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.

3.3 Kinetics

3.3.1 Loading

Loading experiments were conducted at 0.5M V10, 0.45M 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 a 1 min as compared to approximately 60% for V10-LIX®63 system.
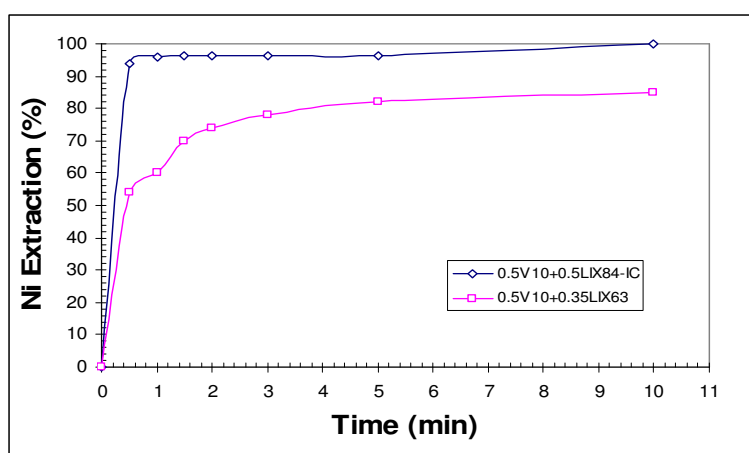
Figure 12: Nickel loading kinetics for V10-LIX®63 at O/A ratio 1:1 and V10- LIX®84-IC systems at 40 °C with O/A = 1:2 at pH 5.00

3.3.2 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 30-40ml, 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 5minutes 95% of Ni was stripped as compared to the 32% in V10-LIX®63 system.
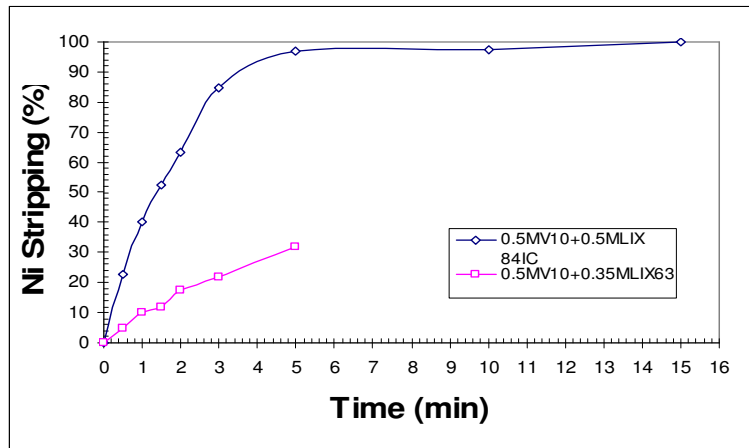
Figure 13: Nickel stripping kinetics for V10-LIX®63 and V10-LIX®84-IC systems at 40 ˚C with A/O=1/1 at pH 5.00

4. 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.

5. Acknowledgements

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