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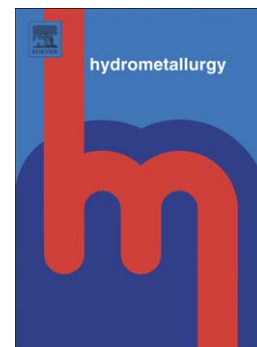
Recovery of cobalt sulfate from spent lithium ion batteries by reductive leaching and solvent extraction with Cyanex-272

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# Recovery of cobalt sulfate from spent lithium ion batteries by reductive leaching and solvent extraction with Cyanex-272

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## ABSTRACT

Cobalt sulfate was recovered from crushed and screened prismatic type spent lithium ion batteries (LIBs) containing 5~20 % Co, 5~7 % Li, 5~10 % Ni, 15% organic chemicals, and 7% plastics together with Cu, Al, Fe, and Mn. Cobalt was reductively leached from the -16 mesh fraction in 1 h by stirring with 2 M H<sub>2</sub>SO<sub>4</sub> and 6 vol% H<sub>2</sub>O<sub>2</sub>, at 60°C and 300 rpm using a solid/liquid ratio of 100 g/L to give a cobalt concentration of 28 g/L, corresponding to a leaching efficiency of >99 %. Metal ion impurities such as copper, iron, and aluminium were precipitated as hydroxides from solution by adjusting pH to 6.5. Cobalt was then selectively extracted from the purified aqueous phase by equilibrating with 50% saponified 0.4 M Cyanex 272 at an equilibrium pH ~6. The McCabe-Thiele plot predicted 99.9% cobalt extraction in a 2 stage counter-current operation with A/O ratio of 1/2. Separation factors for the extraction of Co/Li and Co/Ni at pH 6 were close to 750. The stripping of the

loaded organic phase with 2 M H<sub>2</sub>SO<sub>4</sub> produced a solution of 96 g/L Co from which pure pigment grade cobalt sulfate could be recovered by evaporation/recrystallisation. Overall, 92% cobalt could be recovered from the spent lithium ion batteries.

*Keywords:* Spent Li-ion battery; Reductive acid leaching; Cyanex 272; Cobalt sulfate recovery.

## 1. Introduction

The demand for secondary batteries as a power source of widely used portable electronic equipments such as cellular phones, lap-top computers, and PDAs, has dramatically increased recently. For example, the worldwide production of lithium ion secondary batteries (LIBs) has increased from 250 million in 1998 (10% in Korea) to 2,044 million in 2007 (Takao, 2007). The predicted waste generated from spent LIBs in Korea in 2006 was 300-500 tons (Sohn et al., 2006). LIBs consist of heavy metals, organic chemicals and plastics in the proportion of 5~20% cobalt, 5~10% nickel, 5~7% lithium, 15% organic chemicals, and 7% plastics, depending upon the manufacturing process. Since LIBs are substituting for other secondary batteries due to lightness and good performance, recycling of metals from them becomes increasingly important for the economy and environment.

The current status of the recycling process for LIBs has been reviewed in several studies. The recovery of valuable metals from spent LIBs mainly involves the mechanical processes, thermal treatment, mechano-chemical and dissolution processes. Chemical processes mainly involve hydrometallurgical unit

operations such as acid or alkaline leaching, chemical precipitation, separation and electrochemical recovery (Zhang et al., 2002; Shin et al., 2006; Sohn et al., 2004; Contestabile et al. 2001). However, some operations have inherent problems such as slow kinetics, solid–liquid separation, high cost, and low purity (Swain et al., 2004). Solvent extraction has been widely used for the recovery and separation of metals from leach liquors, effluents, and waste materials. The demand for high purity metals and recent trends towards environmentally friendly technology has focused more attention onto solvent extraction, which appears to meet the requirements for performance and economics.

Recovery of metals from waste LIBs by leaching followed by solid-liquid separation and solvent extraction using PC-88A, D2EHPA, Cyanex 272, or Cyanex 302 has been reported by several groups (Zhang et al., 1998; Wu et al., 2004; Nan et al., 2005; Swain et al., 2006). The reported studies have largely focused on synthetic solutions of compositions comparable to leach liquors of waste LIBs or the cathodic material scrap generated during the material manufacturing process. In particular, previous work has focused mainly on cobalt and lithium separation or cobalt and nickel separation from synthetic solution or cathodic material. In this study, the material flow and recovery of all metal components of spent lithium ion batteries are considered in the hydrometallurgical process, but only cobalt was considered viable as a recoverable metal. It was not considered feasible to solvent extract and recover pure copper, which is the other significant metal present, whilst the recovery of lithium as lithium carbonate and the proper treatment of the final sodium sulfate solution requires further study.

The aim of this study was to focus on the leaching and separation of cobalt from other impurity metals in spent LIBs to recover pure cobalt sulfate for pigment use.

## 2. Materials and methods

### 2.1. Materials

The spent LIBs were subjected to physical treatment which involved discharging, dehydration, drying and crushing steps (Shin et al, 2008). After screening, the crushed LIBs were separated to powders of three different size fractions: +8 mesh, -8/+16 mesh, and -16 mesh. The fraction of -16 mesh was selected for reductive leaching.

### 2.2. Leaching and elimination of impurities

The leaching tests were conducted using 2 M  $\text{H}_2\text{SO}_4$ , 6 vol%  $\text{H}_2\text{O}_2$ , reaction temperature 60 °C, agitation speed 300 rpm, solid/liquid ratio 100 g/L, reaction time 2 h. The metal ion concentration in the solution was analyzed by AAS (Perkin Elmer, AAnalyst 400). Removal of Fe, Cu, and Al from the liquor, as well as fast filtration of the residue was performed by adjusting the pH to 6.5 using 4 M NaOH and 50 mass%  $\text{CaCO}_3$  solution followed by filtration. The residue was washed with distilled water for recovering cobalt ions remaining in the residue and the washed liquor was reused in the leaching step. In addition, the residue can be treated by dilute sulfuric acid washing to remobilize the metals and recover pure gypsum.

### 2.3. Solvent extraction

The commercial extractant, bis (2,4,4-trimethylpentyl) phosphinic acid (Cyanex 272) was supplied by Cytec Inc. and was used in the as-received form without further purification. Commercial grade extra-pure kerosene (bp 180-270 °C) (Junsei Chemicals Co. Ltd., Japan) was used as a diluent for all sets of experiments. Stock solutions of Cyanex 272 were prepared by diluting Cyanex 272 in kerosene at a predetermined mass ratio and were saponified to 50% Na-Cyanex 272 of different composition in the range 25%-75% by adding stoichiometric amounts of concentrated NaOH solution. All preliminary experiments were carried out at ambient temperature ( $25 \pm 1$  °C) to examine the effect of pH, saponification %, and Cyanex 272 concentration in the range 0.03 M – 0.54 M.

The aqueous phase free of impurities such as Fe, Cu, and Al, after precipitation and filtration, was equilibrated with different volumes of 50% saponified 0.5 M Cyanex 272 by shaking in a separating funnel for 30 min in a shaking machine. The initial pH of the aqueous phase was controlled by adding dilute  $\text{H}_2\text{SO}_4$  or NaOH solutions before equilibration. The equilibrium pH and metal concentrations in the aqueous phase were measured after separation of the aqueous and organic phases. All samples were analyzed by AAS (Perkin Elmer, AA400) and pH was measured by a pH meter (Orion, 3 star). The sampled solution was immediately filtered through IPS phase separation paper and the metal ions concentration in solution was analyzed by AAS.

The stripping of cobalt from the loaded organic phase was carried out with 2 M H<sub>2</sub>SO<sub>4</sub> solution.

The distribution ratio, D, was calculated as the concentration ratio of metal present in the organic phase to that in the aqueous phase at equilibrium. From the D values, the percentage extraction and separation factor ( $\beta$ ) were calculated using Eqs. 1 and 2:

$$\text{Percentage extraction} = \frac{100 D}{D + (V_{\text{aq}}/V_{\text{org}})} \quad (1)$$

where V<sub>aq</sub> and V<sub>org</sub> are the volumes of the aqueous and organic phases, respectively, and

$$\text{Separation factor } \beta_{\text{Li}}^{\text{Co}} = \frac{D_{\text{Co}}}{D_{\text{Li}}} = \frac{[\text{Co}]_{\text{org}}/[\text{Co}]_{\text{aq}}}{[\text{Li}]_{\text{org}}/[\text{Li}]_{\text{aq}}} \quad (2)$$

### 3. Results and discussion

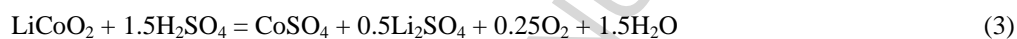
#### 3.1. Physical separation of waste LIBs

Tables 1 list the mass% of different fractions obtained after physical separation of LiBs and the composition of metals in each fraction respectively. The fraction of -16 mesh with a high cobalt content of 11.9% was chosen for reductive leach tests.

#### 3.2. Leaching and precipitation

Table 2 shows a summary of some of the published results on cobalt leaching from LiBs to

demonstrate the effect of acid, peroxide and other factors. The cobalt dissolution after 10 min leaching in 1 M H<sub>2</sub>SO<sub>4</sub> at 75-80°C in the absence of H<sub>2</sub>O<sub>2</sub> is 37.3% which increases to 53.1% in 2 M H<sub>2</sub>SO<sub>4</sub>. This can be related to the acid decomposition reaction given by Eq. 3. In the presence of H<sub>2</sub>O<sub>2</sub>, the cobalt dissolution at 80°C in 2 M H<sub>2</sub>SO<sub>4</sub> after 10 min increased to 75.4% in 15% H<sub>2</sub>O<sub>2</sub> and 94.6% in 20% H<sub>2</sub>O<sub>2</sub>. These results indicate the reductive leaching is caused by H<sub>2</sub>O<sub>2</sub> as described by Eq. 4.



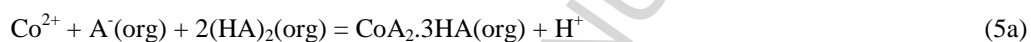
In the present study, cobalt dissolution at a high S/L ratio of 100 g/L at 60°C after 10 min was 98% (Table 2). The enhanced dissolution at a relatively low temperature of 60°C is a result of the higher stability of H<sub>2</sub>O<sub>2</sub>. At temperatures above 60°C H<sub>2</sub>O<sub>2</sub> tends to rapidly decompose to H<sub>2</sub>O and O<sub>2</sub>.

Table 3 lists the concentration of metal ions in the leach liquor. It can be deduced that not all the copper and nickel were leached under the conditions used to optimise cobalt extraction. However, this work did not aim to recover these metals separately. The adjustment of pH to 6.5 removed over 99% Fe, Cu and Al as their hydroxides. In the case of Mn, the removal was only about 15%. The total loss of cobalt was about 7% due to the incorporation with hydroxide solids during precipitation. Overall, in the leaching and neutralization process approximately 1 kg of H<sub>2</sub>SO<sub>4</sub>, 0.25 kg NaOH and 0.40 kg CaCO<sub>3</sub> were consumed per litre of cobalt leach solution.



### 3.3. Solvent extraction with saponified Cyanex 272

The acidic form of Cyanex 272 exists as a dimer (HA)<sub>2</sub> whereas the saponified form is a monomer. The existence of dimer species in the organic phase for a sulfate medium has been reported by other investigators (Tsakiridis et al., 2004). The extraction of cobalt by partially saponified Cyanex 272 can be expressed by Eq. 5a (Ritcey and Ashbrook, 1984):



To select the best extractant concentration and equilibrium pH of the aqueous phase with regard to high percentage extraction of cobalt, the effect of 50% saponified Cyanex 272 concentration in the range 0.03 M – 0.54 M and aqueous phase equilibrium pH in the range from 4.5 to 6.5 was studied at A/O ratio of 1/2. The results demonstrate that the percentage extraction of cobalt increases with increasing equilibrium pH of the aqueous phase and extractant concentration (Fig.1). The optimum equilibrium pH of the aqueous phase appears to be in the range 5.5–6.0 with >95–98 % Co extraction and low co-extraction of Ni (~1%). Separation factors of Co/Li and Co/Ni for extraction using 50% saponified 0.4 M Cyanex 272 at equilibrium pH 6 were close to 750.

The effect of saponification ratio of Cyanex 272 (25 ~ 75 %) on the extraction of metals from clarified leach liquors at an initial pH 6.5 using 0.45 M Cyanex 272 is presented in Fig. 1. The cobalt extraction increases from 55% to 100% with the increase in saponification ratio from 25% to 75%, which

also causes an increase in equilibrium pH from 4.8 to 8.4. The extraction of nickel and lithium also increases at higher saponification ratios and equilibrium pH. At a given equilibrium pH, the 50% saponified Cyanex 272 gave higher cobalt extraction. The co-extraction of Ni and Li increased only at higher pH when Co extraction reached ~100%.

Fig. 2 shows a linear relationship of logarithmic distribution coefficient for cobalt extraction as a function of pH. The linear relationships of lines (a) and (b) of slope ~1 for 50% and 25-50% saponification respectively are consistent with Eq. 5b. However, the results from literature shown in line (c) for 65% saponification indicate that the slope tends to decrease from unity at higher saponifications due to competing extraction.

Fig.3 shows a logarithmic plot of distribution coefficient as a function of the concentration of Cyanex 272. The linear relationship at low concentration of Cyanex at 50% saponification for data points representing an equilibrium pH ~5 is also close to unity. In the present study for selective extraction of cobalt from nickel and lithium, the 50 % saponified ratio was considered for further investigation.

### 3.4. Counter-current extraction and stripping

Previous studies showed that a two stage counter-current extraction using Cyanex 272 and TBP can quantitatively recover cobalt from a synthetic solution of  $\text{CoSO}_4\text{-Li}_2\text{SO}_4\text{-Na}_2\text{SO}_4$  (Swain et al., 2006). Fig. 4 shows the loading isotherm and the McCabe-Thiele plot for cobalt extraction from the clarified leach

liquor using 50% saponified 0.4 M Cyanex 272. It also predicts quantitative extraction of cobalt in a two stage counter-current operation using an A/O ratio of 1/2.

From the McCabe-Thiele plot, it can be concluded the cobalt extraction increased from 65 to 99.9% at an increase of O/A ratio from 2 to 5 and decreased from 44 to 40% with an increase of A/O ratio from 1 to 2. Quantitative extraction of cobalt was achieved in two stages.

The cobalt concentration in the loaded organic is 6.9 g/L (Fig. 4). This prediction was confirmed by low concentration of only 0.5 mg/L Co in the raffinate after two stages, corresponding to >99.9% extraction efficiency. The co-extraction of Li and Ni into the loaded organic was 1.2 mg/L and 0.7 mg/L, respectively. Considering the percent extraction, phase ratio and the minimum stages required for the complete removal of cobalt, an O/A ratio of 2 was selected in order to achieve extraction of a major portion of cobalt in two counter-current states.

As shown in Table 4, the stripping of the loaded organic with 2 M H<sub>2</sub>SO<sub>4</sub> at O/A ratio 11.7/1 produced a cobalt sulfate solution containing 96 g/L Co and impurities under 50 mg/L. This solution can be evaporated to recover cobalt sulfate crystals as a pigment source.

#### 4. Conclusions

The physical treatment of spent lithium ion battery produced, magnetic materials (3.5%) and

three different fractions of non-magnetic material. The fine fraction (36.4% -16 mesh) contains most of the cobalt as  $\text{LiCoO}_2$ . Reductive acid leaching of the fine fraction for 1 h at  $60^\circ\text{C}$  with 2 M  $\text{H}_2\text{SO}_4$  and 6 vol% of  $\text{H}_2\text{O}_2$  resulted high leaching efficiencies of cobalt (98%) and lithium (97%). Adjustment of the leach liquor pH to 6.5 precipitated >99% Fe, Cu and Al as hydroxides along with about 7% Co.

The recommended system for selective extraction of cobalt from nickel and lithium is 50% saponified 0.4 M Cyanex 272 at optimum equilibrium pH 5.5 ~ 6.0 which gave 95-98% Co extraction and ~1% Ni extraction. The slope of  $\log D_{\text{Co}}$  vs. pH plot for this system is unity as expected from the published stoichiometry for extraction. The slope of  $\log D_{\text{Co}}$  vs  $\log [\text{Cyanex 272}]$  plot is also close to unity at low Cyanex concentrations and at equilibrium pH ~5. The McCabe-Thiele plot for extraction of cobalt, predicts 99.9% Co extraction in a 2 stage counter-current operation with A/O ratio of 1/2. The stripping of the loaded organic phase with 2 M  $\text{H}_2\text{SO}_4$  at O/A ratio 11.7/1 produced an aqueous solution of 96 g/L Co with a low impurity content of 50 mg/L, which can be used to recover  $\text{CoSO}_4$  as a pigment material. Overall, 92% of cobalt could be recovered from spent lithium ion batteries. Further work is continuing on recovering high purity  $\text{LiCO}_3$  and the best methods for treating the  $\text{Na}_2\text{SO}_4$  effluent.

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Table 1.

Metal composition of different LIB physical fractions

	Co%	Li%	Cu%	Fe%	Mn%	Ni%	Al%	Mass (kg)	Mass (%)
Magnetic material	0.006	0.0006	0.09	1.6	0.007	0.9	0.1	0.3	3.5
+ 8 mesh	9.3	1.1	7.4	0.08	0.1	0.4	7.8	4.2	49.4
8~16 mesh	2.1	0.3	0.08	0.01	0.002	0.006	0.09	0.65	7.6
- 16 mesh	11.9	1.3	4.6	0.2	0.04	0.1	5.1	3.1	36.5
Total	23.3	2.7	12.2	1.9	0.1	1.4	13.1	8.5 (0.25)*	100 (2.9)*

\* The value inside brackets is weight loss during physical treatment.

Table 2.

Comparison of leaching results of spent LIBs under different conditions

S/L (g/L)	T °C	Lixiviant	Co leached %		
			10 min	60 min	120 min
50 <sup>a</sup>	75	1M H <sub>2</sub> SO <sub>4</sub>	37.3	39.7	41.3
50 <sup>a</sup>	80	2 M H <sub>2</sub> SO <sub>4</sub> , 0% H <sub>2</sub> O <sub>2</sub>	53.1	61.5	-
		2 M H <sub>2</sub> SO <sub>4</sub> , 15% H <sub>2</sub> O <sub>2</sub>	75.4	83.1	-
		2 M H <sub>2</sub> SO <sub>4</sub> , 20% H <sub>2</sub> O <sub>2</sub>	94.6	100	-
100 <sup>b</sup>	60	2 M H <sub>2</sub> SO <sub>4</sub> , 6% H <sub>2</sub> O <sub>2</sub>	-	98	-

a. Shin et al., 2005, 300 rpm; b. this work, 250 rpm, -16 mesh



Table 3.

Concentration of metals in the leach liquor before and after precipitation.

mg/L	Co	Li	Ni	Fe	Mn	Cu	Al	Ca	Na	pH
Leach liquor <sup>a</sup>	24880	3000	37.94	159.5	16.06	782.7	1800	13	37.6	0.17
pH 6.5 solution <sup>b</sup>	13800	2040	14.84	0.429	10.57	0.422	3.4	480	4200	6.58
Washing solution <sup>c</sup>	9425	912	15.74	0.52	3.1	0.247	0.214	430	1570	5.72
Loss (%) <sup>d</sup>	6.7	1.6	19.4	99.4	14.9	99.9	99.8	-	-	-

- 16 mesh powder for 60 min at 100 g L<sup>-1</sup> pulp density, 2 M H<sub>2</sub>SO<sub>4</sub>, 6% H<sub>2</sub>O<sub>2</sub>, 250 rpm and 60 °C.
- After adjusting pH to 6.5 by adding 4 M NaOH solution and 50 mass% CaCO<sub>3</sub> solution.
- Liquor after washing the residue with distilled water.
- Loss from leach liquor to residue based on mass balance.

Table 4.

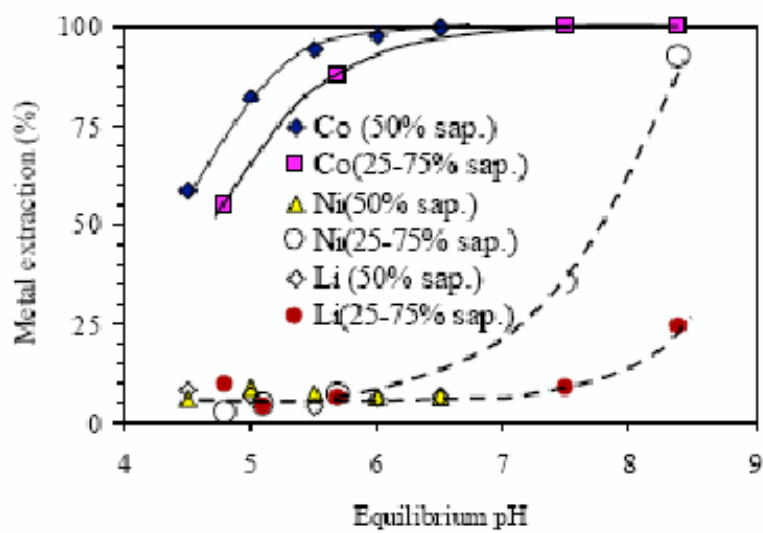
Composition of cobalt sulfate solution recovered after stripping with 2 M H<sub>2</sub>SO<sub>4</sub>

Metal (mg/L)	Co	Mn	Ni	Li	Cu	Na	Fe	Al	Ca	pH
Raffinate	0.5	0.02	12.8	2032	0.4	11650	0.4	0.5	428	5.8
Stripped solution <sup>*</sup>	96000	22	1.6	3.4	1.1	11.7	2.3	4	4.1	0.3

<sup>\*</sup>8% CoSO<sub>4</sub> in loaded liquor, O/A = 11.7/1

Figure captions

Fig. 1 Effect of pH and saponification on metal ion extraction by Cynex 272.



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Fig. 2

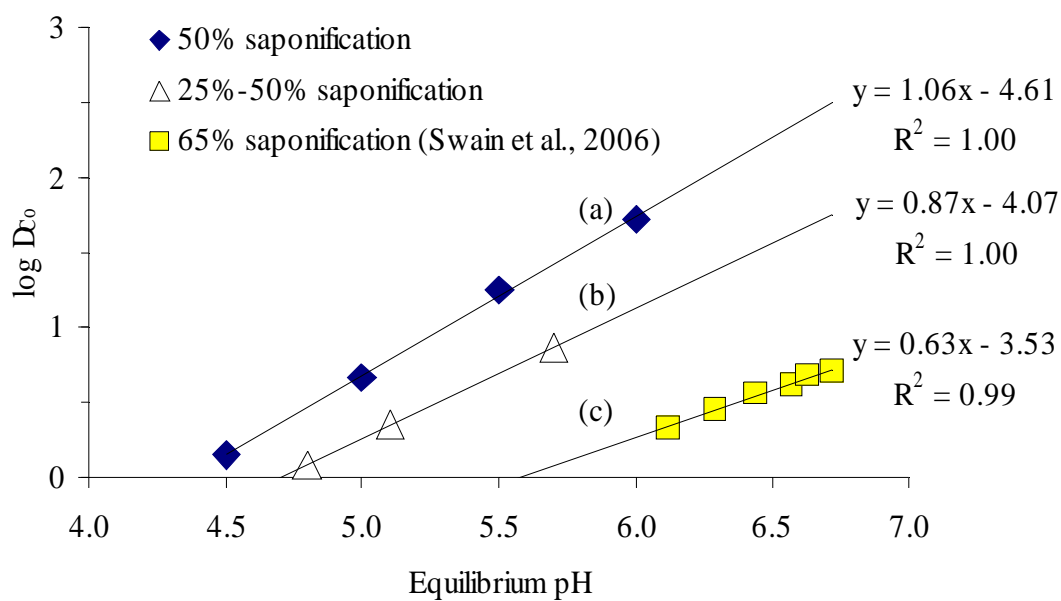


Fig. 2 Effect of pH and saponification of Cyanex 272 on cobalt distribution coefficient (data from our experiment, and Swain et al., 2006).

Fig. 3. Effect of Cyanex 272 concentration at 50% saponification on distribution coefficient of cobalt.

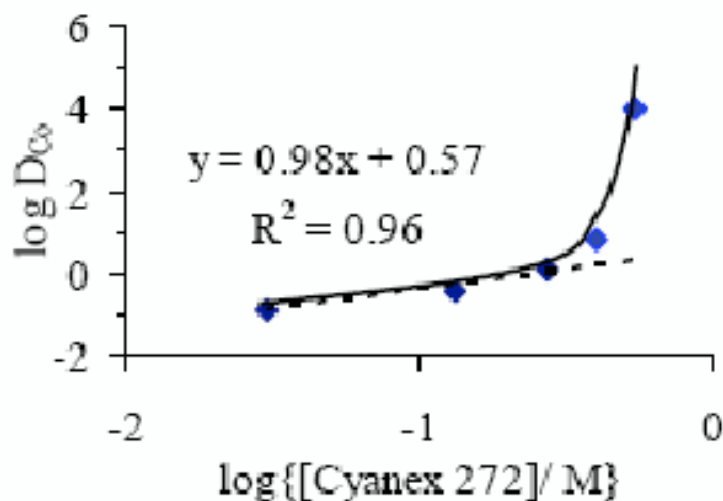


Fig. 4. McCabe-Thiele plot for cobalt extraction using 50% saponified 0.4 M Cyanex 272  
(Feed solution: Co = 13.8 g/L, Li = 2 g/L, Ni = 15 mg/L, pH = 6.6)

