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Review

Application of Cyanex® extractant in Cobalt/Nickel separation process by solvent extraction

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This study presents a detailed review on the advanced hydrometallurgical treatment involving the extraction and separation of Cobalt (Co) and Nickel (Ni) by Cyanex® extractants. The structures, properties and applications of various Cyanex® extractants such as Cyanex®272, Cyanex®301, Cyanex®921, Cyanex®923 and Cyanex®421X were discussed and compared. Cyanex®272 thus proved to be the most appropriate solvent extractant for the separation of Co and Nickel from sulphate and chloride media due to its stability to common oxidant, better physicochemical properties and its ability to avoid gypsum crystallization in stripping-electrowinning circuit. Finally, various solvent extraction (SX) techniques for the extraction and separation of Co and Ni using Cyanex® extractants were discussed as well as newer processes of extraction and separation.

Key words: Hydrometallurgy, Cobalt, Nickel, solvent extraction, Cyanex® extractants, Cyanex®272, Cyanex®301, Cyanex®921, Cyanex®923, Cyanex®421X.

INTRODUCTION

The separation of Cobalt (Co) from Nickel (Ni) in aqueous solution has always been a problem for hydrometallurgists. Their adjacent positions in the transition metal series in the periodic table result in aqueous chemical behaviour that is too similar for the development of easy separation routes (Adekola et al., 2010). Co and Ni were separated traditionally by processes based on the selective oxidation and/or precipitation of Co from either sulphate or chloride solution and such processes are still in use today. While alkyl amines are the extractant of choice for the separation of Co from Ni from chloride liquors (Flett, 2004), for the weakly acidic sulphate liquors the alkyl phosphorus acids have found significant commercial application at various location around the world (Hofirek and Nofal, 1995). Because of the high Ni to Co ratio encountered in liquors produced in sulphate-based high Ni matte leach processes or those produced in the acid pressure leaching of Ni laterites, very high separation factor (>1000) are required. Cyanex®272 has become the reagent of choice for such purposes (Rickelton and Nucciarone, 1997).

The separation of Co and Ni by precipitation processes has been and is still carried out commercially by a number of processes. Thus, sulphide precipitation can be used to completely remove Co from Ni process leached liquor. It can also be used to precipitate Ni from Co rich liquors. Co and Ni can also be separated by oxidative precipitation; strong oxidants such as Chlorine, Ammonium persulphate, caro's acid or ozone is needed in practice (Nishimura and Umetsu, 1992; Wyborn and McDonagh, 1996). The use of air under pressure has also been reported. This formed the basis for cobaltic amine process for Co-Ni separation (Nyman et al., 1992).

Ni powder is also precipitated selectively by reduction of aqueous solution containing Ni and Co ammines in concentrated ammonium sulphate solution at around 240°C with hydrogen gas at a total pressure of up to 3103 kPa. When the concentration of Ni in the solution is lowered to around that of Co, the reaction is stopped and the solution discharged from the autoclave leaving Ni powder inside (Burkin, 1987).

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Ion exchange separation of Co and Ni is most readily accomplished from chloride solution where advantage can be taken of the tendency for Co to form complex chloride anions, that is, $[CoCl_3]^-$, $[CoCl_4]^{2-}$, which Ni does not. These complexes are quite weak, however, relatively high concentrations of chloride ion are needed to produce the $[CoCl_4]^{2-}$ species. Although no great degree of selectivity between Ni(II) and Co(II) is achievable by ordinary cation exchange resins, chelating ion exchangers such as XFS4195, XFS4196 and XFS43084 can offer separation opportunities (Grinstead and Tsang, 1983).

Separation of Ni and Co is possible by direct hydrogen reduction of Ni and Co loaded di-ethylhexyl phosphoric acid (DEHPA) solution. Ni can be selectively reduced in the presence of Co from a metal-loaded DEHPA phase in an autoclave at 140°C and an initial pressure of 120 atm (Monhemius, 1994). It is also possible to recover Ni selectively from aqueous solutions by direct hydrogen reduction in the presence of Co.

SOLVENT EXTRACTION (SX)

Solvent extraction (SX) is one of the most useful techniques that are used for the selective removal and recovery of metal ions from aqueous solution and it is largely applied in the purification process in chemical and metallurgical industries (Thurman and Mills, 1992; Dean, 1998). SX makes use of an organic compound capable of extracting the metal ion of interest, or a complex of it, from the aqueous phase into an immiscible organic solution. Conventional SX techniques however needs large amount of organic solvents and often creates environmental problems (Rudberg et al., 1992). The advantages of SX are simplicity and rapidity, the solvents are easily recoverable, and solvents are stable, transparent to ultraviolet (UV), not emulsifying during extraction and as selective as possible. In these processes, metal ion containing solution is contacted with a selective solvent. After extraction, stripping follows the process. SX is very difficult for quantitative separation of metal ions because of low driving force, and then a large amount of solvent is required. These make the extraction and stripping of desired species very expensive (Alfassi and Wai, 1992). More environmental friendly technology is needed nowadays, and there have been increasing attentions to extract metal ions by supercritical fluid extractants, solid phase extraction and bioleaching.

Principles of solvent extraction

SX procedures utilize non-uniform distribution of substances between two immiscible liquid phases. Enrichment of the substance in one of the phases is dependent on many factors, such as pH, metal concentration and ionic strength in aqueous phase, salt concentration, reagent concentration in organic phase, contact time and temperature. Under suitable conditions, a substance of interest can be transferred to one of the phase, while unwanted substances are retained in the other. The transfer of the solute from one liquid phase to another involves extraction reactions, which permit the establishment of liquid-liquid equilibrium. The distribution of solute M (equilibrate between an aqueous phase and an organic solvent) can be described by an equilibrium equation (Rydberg, 1992):

$$[M]_{aq} <=> [M]_{org} \qquad (1)$$

Thus, when this distribution reaches equilibrium, the distribution ratio (D) of the solute concentrations between the two phases is:

$$D = [M]_{org} / [M]_{aq} \qquad (2)$$

Where $[M]_{org}$ is the concentration of a solute in the organic phase and $[M]_{aq}$ is concentration in the aqueous phase.

If the aqueous: organic (A:O) ratio is equal to 1, then the percentage of metal extracted (%E) would be:

$$\%E = 100D / 1 + D \qquad (3)$$

An important extraction is characterized by a high value of D>>1, whilst a very small value of D<<1 characterizes a very feeble extraction.

SOLVENT EXTRACTION OF COBALT AND NICKEL

Presently, most of the commercial Co/Ni SX plants operate using dialkyl phosphinic acid extractant, Cyanex®272 (Olivier, 2011). In contrast to the resin ion exchange (development) and precipitation process briefly discussed above, SX with this reagent does offer the opportunity for better Co/Ni separation with high yields and purity of the separated metals. Depending on the leach liquors composition, different reagents can be used for Co/Ni SX; the anion exchangers (extractants) from chloride solution and cation (acidic chelating) extractants from sulphate solutions. For Co-Ni separation by anion exchange the same situation exists as for resin anion exchangers with the most important ligand in the aqueous phase being chloride. The extracted anion species has been shown to be $[CoCl_4]^{2\ -}$. For cation exchangers, only the alkyl phosphoric, phosphonic and phosphinic acids show selectivity for Co over Ni, all the rest that is, carboxylic acids, β-ketone, 8-hydroxylquinolines (8-HQs) and hydroxyoximes, show marginal selectivity for Ni(II) over Co(II). The separation factor of Co from Ni in weakly acidic sulphate solutions

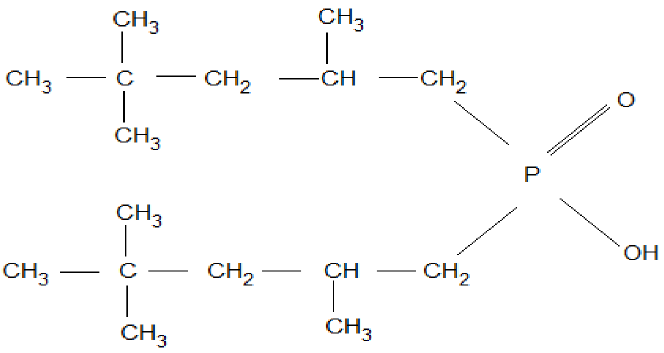

Figure 1. Bis(2,4,4-trimethylpentyl) phosphinic acid.

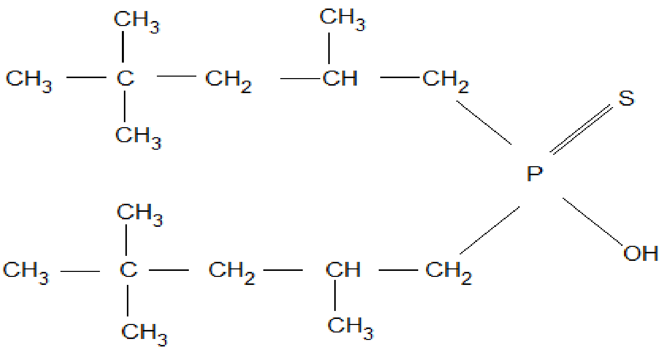

Figure 3. Bis(2,4,4-trimethylpentyl) monothiophosphinic acid.

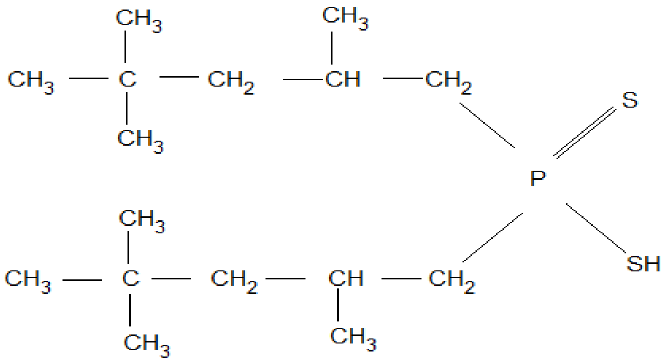

Figure 2. Bis(2,4,4-trimethylpentyl) dithiophosphinic acid.

increases in the following series: phosphoric < phosphonic < phosphinic acid (Luo et al., 2006).

CYANEX® EXTRACTANTS

Cyanex® extractants are phosphorus-based products that act either as a chelating extractants or as a solvating extractants (Cyanex Industries Inc., 2007). Chelating extractants include Cyanex®272 and Cyanex®301. Cyanex®272 solvent extractant reagent was developed specifically for the separation of Co from Ni by SX. It is estimated that 40% of Co in the western hemisphere is produced using Cyanex®272 SX reagent, at plants in America, Canada, Africa, China and Australia. Cyanex®272 can also be used to separate the rare earth elements from one another. The acid concentration required for metal stripping is lower than when phosphoric or phosphonic acids are used as extractant. Cyanex®301 SX reagent is an analogue of Cyanex®272. Dithiophosphinic acid (Cyanex®301) also exhibit interesting extraction characteristics for the recovery of Co and Ni. A potential advantage of Cyanex®301 is the ability to extract both Co and Ni under very acidic conditions, thus, avoiding the need of adding alkali for pH adjustment of the acidic leach liquors. However, the stability, better physicochemical properties and ability to avoid gypsum crystallization in stripping-electrowinning circuit makes Cyanex®272 the extractant of choice.

Solvating extractants include Cyanex®921, Cyanex®923 and Cyanex®471X. Cyanex®921 and Cyanex®923 have the potential in a wide range of applications. Specific applications are the recovery of organic solutes and/or inorganic acids from waste effluents, and metal extraction processes. Unlike its phosphine oxide analogues, Cyanex®471X is a Lewis base. It will only complex readily with metals that exhibit the characteristics of soft Lewis acids. Examples of metals falling upon those criteria are Pd(II), Pt(II), Ag(I), Cd(II), Hg(I), Hg(II) and Au(III) (Cyanex Industries Inc., 2007).

Cyanex®272 extractant

The active component of the Cyanex®272 extractant is a bis(2,4,4-trimethylpentyl) phosphinic acid (Figure 1). Metal ions are extracted through a cation exchange mechanism. Although Cyanex®272 is selective for Co in the presence of Ni, a variety of other cations can also be extracted depending on pH of the solution (Saragi et al., 2009; Zhang et al., 2001).

Cyanex®301 and Cyanex®302 extractants

Cyanex®302 and Cyanex®301 are the monothio- and dithio- derivative of Cyanex® 272 with chemical formula $R_2PS(OH)$ and $R_2PS(SH)$, respectively (Gotfryd, 2005). The active component of Cyanex®301 extractant is bis(2,4,4-trimethylpentyl) dithiophosphinic acid (Figure 2), while the active component of Cyanex®302 extractant is bis(2,4,4-trimethylpentyl) monothiophosphinic acid (Figure 3). These sulphur-containing compounds are much stronger acids than their analogous oxy-acid, Cyanex®272. As such, they are capable of extracting

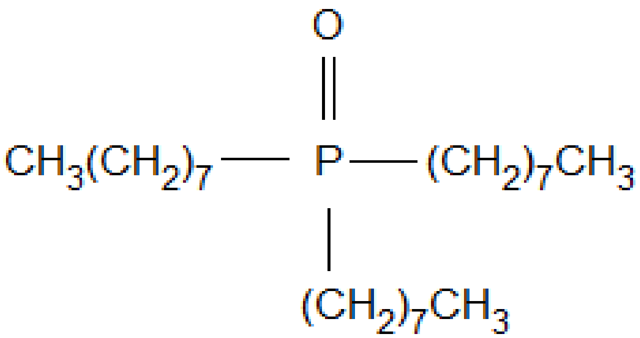

Figure 4. Trioctylphosphine oxide (TOPO).

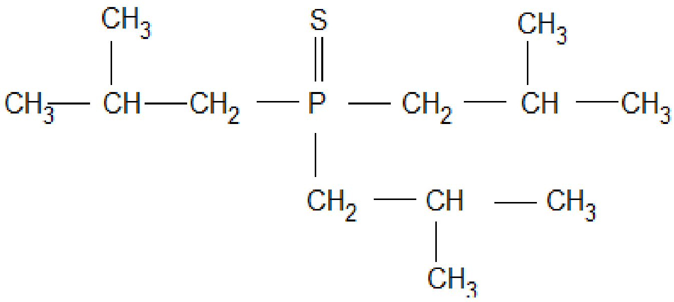

Figure 5. Tributyl phosphinic sulphide (TIBPS).

many metals at lower pH < 2. They do not discriminate between heavy metals at this pH range, however, a high degree of selectivity of extraction of heavy metals versus the alkaline earths is observed. Cyanex®301 was originally developed for the selective extraction of Zinc (Zn) from effluent streams also containing Calcium, such as those generated in the manufacturing of rayon by the viscose (Zhang et al., 2001).

Cyanex®921, Cyanex®923 and Cyanex®471X extractants

Cyanex®921 extractants (Figure 4) better known as trioctylphosphine oxide (TOPO) has been used commercially for many years to recover Uranium from wet process phosphoric acid.

Cyanex®923 is a liquid phosphinic oxide which has potential applications in the SX recovery of both organic and inorganic solutes from aqueous solution for example, carbonxylic acid and arsenic. It is a mixture of four trialkyl phosphine oxide.

$$R_3P(O)\ R_2R^IP(O)\ R^I_3P(O)$$

Where R is the normal octyl, and R^I is normal hexyl Cyanex®471X is a soft Lewis base and will only complex readily with metals that exhibit the characteristics of soft Lewis acids. It is useful for the selective recovery of silver and in the separation of palladium from platinum. The active component of Cyanex®471X is tributyl phosphinic sulphide (TIBPS) (Figure 5).

Cyanex®921, Cyanex®923 and Cyanex®471X are not suitable extractants for the extraction and separation of Co and Ni (Cyanex Industries Inc., 2007). The physicochemical properties of Cyanex® extractants are presented in Table 1.

Comparison of Cobalt and Nickel separation coefficient for various extractants

The most important extractant is Cyanex®272. Cyanex®301 and Cyanex®302 are not very stable in contact with Cu^{2+}, Cd^{2+} and common oxidants (Sole and Cole, 2001). Table 2 illustrates great advantages of Cyanex®272 over earlier extractants (Gotfryd, 2005).

Additionally, Cyanex®272 almost does not extract Calcium at optimal for Co^{2+} extraction condition (pH 5.0 to 5.5). This allows avoiding gypsium crystallization in stripping-electrowinning circuit. Alkyl phosphonic and alkyl phosphinic acids as Co^{2+} extractants have one common and important disadvantage. If saturated to level of 12 to 20 g/dm^3 Co^{2+}, they highly increase their viscosity to over 150 cst (even to 400 cst). At such conditions, mixing and pumping can be almost impossible. Maximum concentrations of extractants, and consequently of Co^{2+} extracted, should be limited to medium levels and/or elevated temperature should be applied to avoid problems of too high viscosity. The elevated temperature is moreover advantageous because the selectivity increases with increasing temperature.

SOLVENT EXTRACTION OF COBALT/NICKEL BY CYANEX® EXTRACTANTS

Cyanex®272 has been adopted as the reagent of choice for various laterite acid pressure leach project in Australia. Thus, the Murrin project uses SX with Cyanex®272 for Co/Ni separation from mixed sulphide pressure leach liquor. Figure 6 shows the Murrin purification flowsheet.

In the Pilot plant SX of Co and Ni for Alvin's Nkomati Project, Co was recovered from calcium-saturated solution using Cyanex®272. Ni was subsequently extracted using versatic acid. The loaded organic was stripped using spent electrolyte, producing advance electrolyte for the recovery of Ni by electrowinning. Co was then recovered with >99.5% extraction efficiency, reducing Co from 1.8 g/L to 10 mg/L. The Co/Ni ratio in the Co product solution was >1500. Ni SX was optimized to recover 99% reducing the Ni concentration from 32 to 0.3 g/L, while minimizing Calcium deportment to the Ni

Table 1. Physicochemical properties of Cyanex® extractants.

Property	**Cyanex®272**	**Cyanex®301**	**Cyanex®302**
Appearance	Colourless to light amber liquid	Green mobile liquid	Pale yellow
Specific gravity	0.92 at 24°C	0.95 at 24°C	0.95 at 24°C
Viscosity	14.2 cP at 25°C; 37 cP at 50°C	78 cP at 24°C	195 cP at 24^{o}C
Solubility in H_2O	16 µ/ml at pH 2.6; 38 µ/ml at pH 3.7	7 mg/L*	3 mg/L at 50^{o}C
pKa	6.37	2.61	5.63
Boiling point	>300°C	Decomposes at 220°C	205°C
Pour point	-32°C	-34°C	Approx. -20°C
Flash point (closed cup)	-	165°F (74°C)	>205°F (>96°C)
Ignition temperature	108°C	74°C	96°C
Specific heat	0.48 cal/gm/°C at 52°C	-	-
Thermal conductivity	2.7 × 10-4 cal/cm/°C	-	-

	Cyanex®921	**Cyanex®923**	**Cyanex®471X**
Appearance	Off white, waxy solid	Colourless mobile liquid	Off white crystalline solid
Specific gravity	0.88 at 25°C; 0.84 at 61°C	0.88 at 25°C	0.91 at 22°C
Viscosity	15.0 cP at 55°C	40 cp at 25°C; 13.7 cP at 50°C	-
Solubility in H_2O	-	>10 mg/L	43 µg/ml at 24°C
Melting point	47 - 52°C	-	58 - 59°C
Boiling point	-	310°C at 50 mm Hg	-
Flash point (closed up setaflash)	-	182°C	-
Auto ignition temperature	-	281°C	-
Vapour pressure	-	0.09 mm Hg at 31°C	-
Thermal conductivity	-	0.00302 cal/cm/s/°C at 25°C 0.00288cal/cm/s/°C at 120°C	-

*Solubility will be lower in aqueous solutions containing dissolved salts; $pK_a = - \log K_a$ (K_a – equilibrium constant of acidic dissociation).

Table 2. Comparison of Co(II) / Ni(II) separation coefficients for various extractants.

Extractant	$ß^{Co}_{Ni}$	**pH of optimal Co extraction**	$\Delta pH_{50\%}^{Ni\text{-}Co}$	
			20°C	**50°C**
DEHPA	14	3.6 - 3.8	0.35	0.70
PC-88A	280	5.0	1.21	1.48
Cyanex®272	7000	5.3 - 5.5	1.58	1.94

$ß^{Co}_{Ni}$ = Coefficient of Co(II) / Ni(II) separation = D(Co) / D(Ni); D(M) = coefficient of metal ion (M) distribution; $\Delta pH_{50\%}^{Ni\text{-}Co} = pH_{50\%}^{Ni} - pH_{50\%}^{Co}$; $pH_{50\%}^{M}$, so called “pH of half extraction of ion M”; that means for D(M) =1.

electrowinning circuit (Sole et al., 2002). The overall process flowsheet is as shown in Figure 7.

The Bulong project used SX directly on the leach liquor after purification. Thus, any Iron, Aluminum and Chromium present in the leach liquor were removed hydrolytically in the two step precipitation to yield liquor at pH 4.2 to 4.5. Co together with Manganese (Mn) and Zn present in the liquor was extracted with Cyanex®272. The Ni in the raffinate was then extracted and separated from Magnesium with carboxylic acid, Versatic 10 (Scole and Cole, 2001). The result of continuous miniplants showed that extraction with Cyanex®272 can achieve 97.5% Co recovery and >99% removal of Mn and Zn with good separation of Co and Ni with Co:Ni ratios in the strip of >1000:1. Figure 8 shows the Bulong Ni/Co purification flowsheet.

Cawse on the other hand uses mixed hydroxide precipitation followed by ammonia releach, which allowed for use of the Ni SX step. A simplified overall Cawse process flowsheet is as shown in Figure 9.

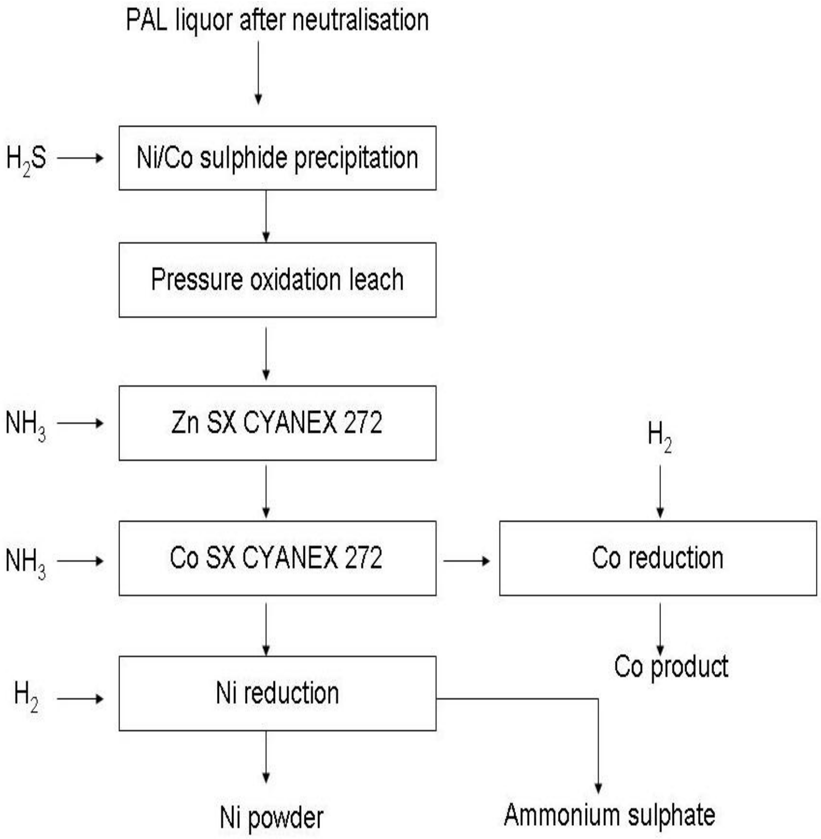

Figure 6. Murrin Murrin purification flowsheet (Motteram et al., 1996).

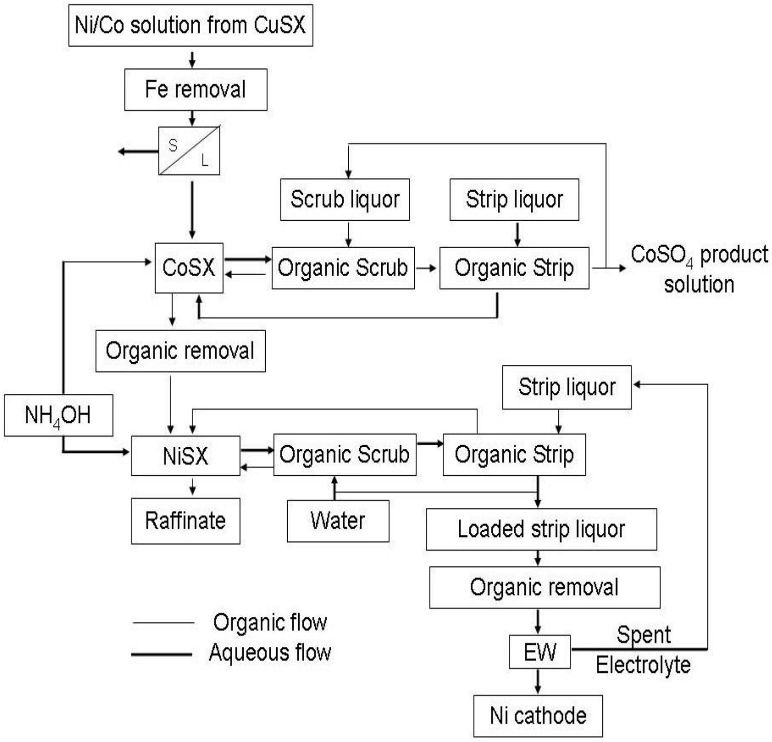

Figure 7. Process flowsheet for treatment of Nkomati Copper SX.

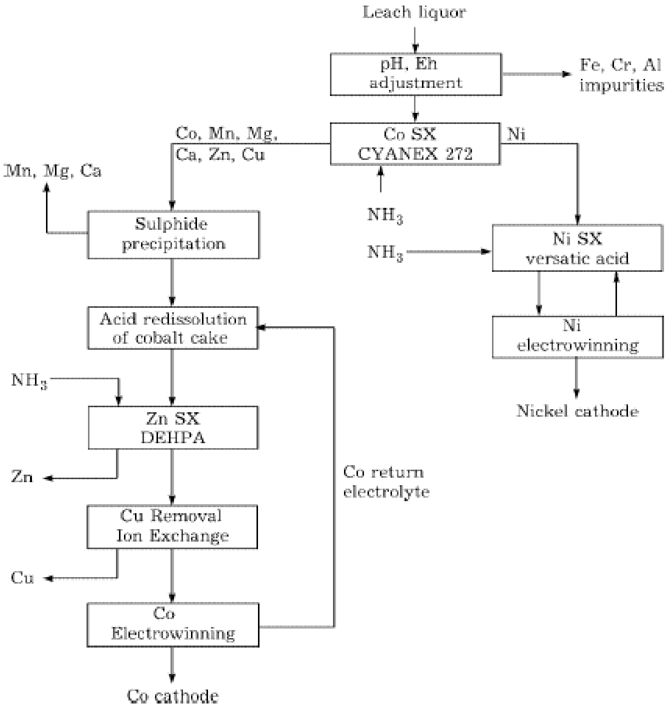

Figure 8. Bulong Ni/Co purification flowsheet (Flett, 2004).

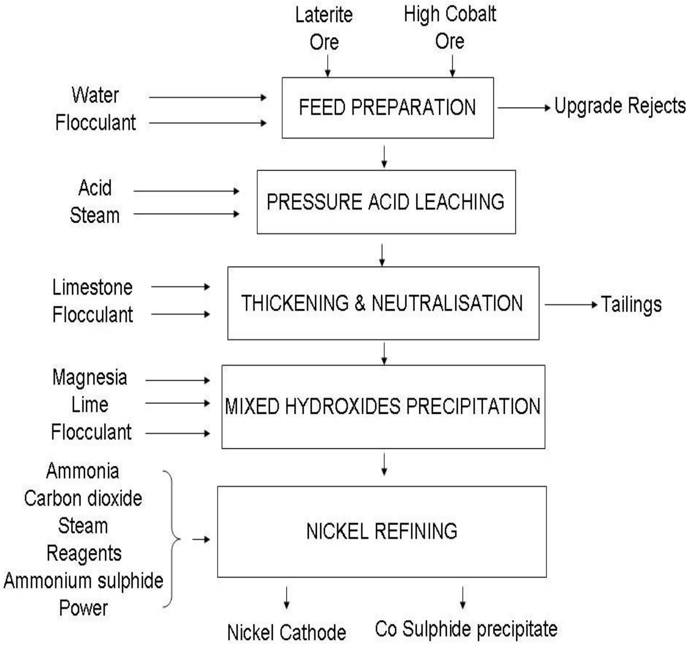

Figure 9. Cawse purification flowsheet (Taylor and Jansen, 2000).

Lenhard (2008) investigated the extraction and separation of Co(II) and Ni(II) from sulphate solutions with different initial volume fractions of Cyanex®302, Cyanex®272 and their mixture, in kerosene as diluent. Under the investigated range of conditions, Lenhard reported that Cyanex®302 outperformed Cyanex®272 in Co-Ni separation. In the extraction of Co and Ni with different mixtures of Cyanex®302 and Cyanex®272, no evidence for any synergistic effects was found.

Ahmed et al. (1992) in their preliminary investigations

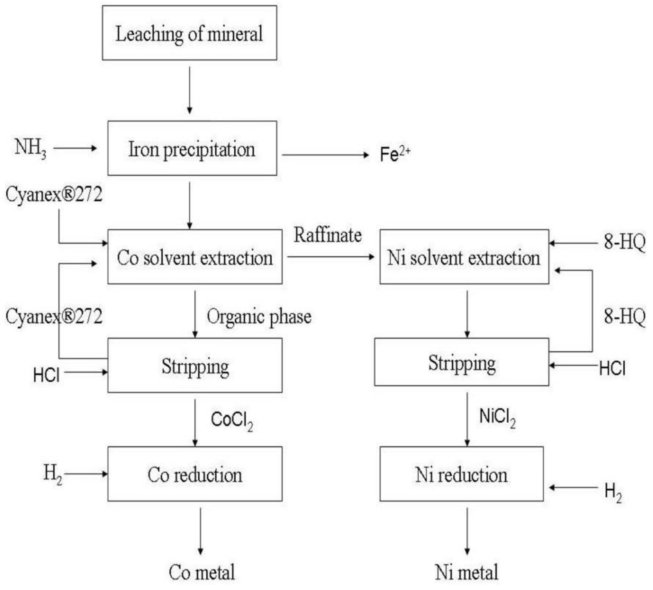

Figure 10. Proposed hydrometallurgical flowsheet for the extraction and separation of Co and Ni by Cyanex®272 from lateritic soil (Ayanda, 2009).

reported that only Cd(II) was extracted with Cyanex®923, while Co(II) and Ni(II) were not extracted. Different parameters affecting the extraction of Cd(II) with Cyanex®923 such as hydrochloric acid, hydrogen ion, extractant and metal concentrations and temperature were also investigated. They reported that Co(II) was found to be extracted with Cyanex®272 at pH 5.8 thereby leaving Ni(II) in the solution.

Gandhi et al. (1993) proposed the extraction of Co(II) at pH 8.0 with 5 × 10^{-3} M Cyanex®272 in chloroform. Co(II) was stripped with 0.5 M nitric acid and separated from Vanadium, Chromium, Ni, Mn, Iron and Zn.

Tait (1993) investigated the use of Cyanex®301, Cyanex®302 and Cyanex®272 for the extraction of Co(II) and Ni(II) from a sulphate medium. He reported that all the reagents extracted Co selectively with Cyanex®302 exhibiting better separation characteristics than Cyanex®272, which in turn showed a higher selectivity than Cyanex®301. The separation ($pH_{0.5}^{Ni}$- $pH_{0.5}^{Co}$) found for Cyanex®302 was 2.6 pH units, compared to 1.7 pH units for Cyanex®272 and 1.1 pH units for Cyanex®301.

Ribeiro et al. (2004) studied the extraction of Co and Ni using Cyanex®302. They studied a high Ni/Co initial concentration ratio of 63.3:1 and found that the feed phase pH has a profound effect on the emulsion liquid membrane (ELM) process due to its straight relation with the extraction chemistry.

Kyung-Ho and Debasish (2006) reported the use of Cyanex®272 for the extraction of Co from a solution containing Co and Ni in a sulphate medium followed by Copper (Cu) extraction. Impurities such as Cu and Iron were removed from the leach liquor by precipitation method before Co extraction. They reported that an increase in the concentration of Cyanex®272 increased the extraction percentage of Co due to the increase of equilibrium pH. Co extraction efficiency of > 99.9% was achieved with 0.20 M Cyanex®272 in two counter-current stages at an aqueous: organic (A:O) phase ratio of 1.5:1. Complete stripping of Co from the loaded organic containing 2.73 g/L Co was carried out at pH 1.4 by a synthetic Co spent electrolyte in two stages at an A:O ratio of 1:2. The enrichment of Co during extraction and stripping operations was reported to be carried out at about 3.5 times.

The extraction of Co from Cu-Zn-free solution was carried out using Cyanex®272 in kerosene followed by the extraction of Ni from the Co-free solution with Na Cyanex®272 in kerosene by Parhi et al. (2008). They reported that the extraction of Co and NiI increased with increasing equilibrium pH and extractant concentration. They also reported that the highest separation factor for Co and Ni was obtained with 0.1 M Cyanex®272 at pH 5.46.

Adekola et al. (2010) reported the hydrometallurgical treatment involving the SX and recovery of Co and Ni from hydrochloric acid leached solution of laterite using Cyanex®272 and 8-HQ, both diluted in kerosene. Experimental results showed that Cyanex®272 was selective for Co in 4 M HCl, while 8-HQ was found to be selective for Ni. The average percentage of Co extracted by Cyanex®272 was 94.71%, while 99.98% of Ni was extracted by 8-HQ. 1.0 M HCl was also found to be effective for the stripping of Co and Ni from both Cyanex®272 and 8-HQ, respectively. The hydrometallurgical flowsheet for the extraction and separation of Co and Ni from lateritic soil as proposed is shown in Figure 10.

CONCLUSION

Commercial operations for separation of Co from Ni have been successfully carried out using precipitation, ion exchange resin, pressure hydrogen reduction and SX. SX does offer the opportunity of complete separation with high yields and purity of the separated metals. Under comparable conditions of the solvent extractants, the Co-Ni separation increases in the order: phosphoric < phosphonic < phosphinic acids. Among the various Cyanex® extractants, Cyanex®921, Cyanex®923 and Cyanex®471X are not suitable for the extraction and separation of Co and Ni. Cyanex®301 was developed for the selective extraction of Zn from effluent streams containing Calcium. Tait (1993) and Lenhard (2008) found Cyanex®302 to be better Co and Ni extractant than Cyanex®272 in their experimental results, Ahmed et al. (1992) also reported that Cyanex®923 is not suitable for the extraction of Co(II) and Ni(II), while Cyanex®471X will only complex readily with metals that exhibit the characteristics of soft Lewis acids. The mono and thio analogues of Cyanex®272 enable the extraction to be carried out at a much lower pH as a result of the replacement of oxygen by sulphur. However, Cyanex®272 is considered to be the most preferable Cyanex® extractant for the extraction and separation of Co and Ni from the stripping conditions point of view (Sole and Hiskey, 1992), because of its stability to Cu^{2+}, Cd^{2+} and common oxidant (Gotfryd, 2005). Cyanex®272 is also primarily designed for the separation of Co and Ni from both sulphate and chloride media. Finally, Cyanex® extractants can be used to extract and separate vast majority of metal cations, this can be achieved by varying the experimental conditions.

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