Studies on solvent extraction of copper and cyanide from waste cyanide solution

Feng Xie*, David Dreisinger

Department of Materials Engineering, University of British Columbia, 309-6350 Stores Road, Vancouver, BC, Canada V6T 1Z4, Canada

ABSTRACT

The recovery of copper and cyanide from waste cyanide solution with the guanidine extractant (LIX 7950) and the modified amine extractant (LIX 7820) has been investigated. Copper can be effectively extracted from alkaline cyanide solutions by both extractants. The free cyanide remains in the aqueous phase due to the preferential extraction of $Cu(CN)_3^{2-}$ over $Cu(CN)_4^{3-}$ and CN^- by the extractants. The selectivity of the metals with the extractants under different cyanide levels has been examined. High cyanide levels tend to depress extraction of copper and silver cyanides, but exhibit insignificant effect on extraction of gold, zinc, nickel and iron cyanides. A possible solution to the separation of copper cyanides and free cyanide in cyanide effluents has been suggested, by which copper can be concentrated into a small volume of solution and the barren cyanide solution recycled to the cyanidation process.

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1. Introduction

The use of highly toxic cyanide in the gold mining industry has attracted great public concern especially after several cyanide spill disasters happened over the last couple years [1]. Environmental constraints controlling the discharge of cyanide from gold plants are being tightened by the local governments worldwide and the concentration of free cyanide and the weak-acid-dissociable (WAD) cyanide species discharged into the tailing should be controlled below a strict limit, at many sites 50 mg/L to tails and 0.1 mg/L or less of any discharge to a receiving waterway [2]. On the other hand, the common occurrence of copper minerals in gold ores is also a matter of concern to the cyanidation process. Most copper minerals are easily and readily soluble in the cyanide solution used in gold leaching, which usually results in high cyanide consumption [3]. They also cause serious problems during cyanide effluent treatment since the equilibriums among copper cyanide complexes can buffer the free cyanide content within the tailing waters and act as a free cyanide sink. More cyanide can be liberated due to the equilibrium shift from $Cu(CN)_4^{3-}$ and $Cu(CN)_3^{2-}$ to $Cu(CN)_2^-$ and finally to the CuCN precipitate when the concentration of free cyanide decreases with time. This results in a high level of free cyanide in the tailing ponds. On the contrary, when excess cyanide exists in the tailings, copper cyanide may uptake the cyanide ion to form stable complexes with a higher CN ligand number [4,5]. Detoxifying cyanide by traditional destruction technologies, such as by

using INCO's SO_2/air method, hydrogen peroxide or Caro's acid, to levels that meet stringent environmental regulations significantly increase the operational cost and may result in a significant economical penalty in loss of valuable copper in cyanide effluent [6,7]. There has been therefore growing interest in the recovery of valuable metals (principally copper) and cyanide from cyanide effluent arising from the gold mining industry.

Acidification, volatilization and recovery (AVR) and some modifications, including sulphidization–acidification–recycle–thickening (SART) process thereof have been developed and practiced in some gold operations, but the high consumption and cost of reagents has significantly limited application [6,8]. The indirect recovery of metals and cyanide with pre-concentration by activated carbon has been proposed [9,10]. The low adsorption capability has severely hampered wide application in practice. Carbon is more suitable for use as a polishing process to remove cyanide to low levels when the initial cyanide concentration is already below about 1–5 mg/L [2]. The recovery of valuable metals and cyanide from cyanide effluent by ion-exchange resins has been extensively studied [11–14]. In the Augment process, CuCN-precipitated resin is used as the adsorbent for the adsorption of soluble copper cyanide from the leachate of copper-containing ore. The loaded copper cyanide species are eluted from the resin using a concentrated copper cyanide solution having a molar ratio of cyanide to copper (CN/Cu) of approximately four. The resin can be regenerated via conversion to the CuCN form with sulfuric acid and the eluate is submitted to electrowinning (EW) to produce copper cathodes [15]. Another example is the Vitrokele process, which uses strong-base resins based on a highly cross-linked polystyrene structure (Vitrokele[TM] 911 and 912, which probably have a quater-

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* Corresponding author. Tel.: +1 604 822 1357; fax: +1 604 822 3619.
  E-mail address: xiefeng@interchange.ubc.ca (F. Xie).

nary amine functionality). The loaded resins are eluted with strong cyanide eluant to recover copper cyanide species. Precious metals and other strongly bound metal cyanide complexes are "crowded" from the resins with tetracyanozincate ($Zn(CN)_4{}^{2-}$). Sulphuric acid is used in the last elution cycle to destroy most of the cyanide complexes and thus to regenerate the resins [16]. One potential disadvantage of the processes is that the precipitated CuCN may block the resin pores, decreasing the opportunity for additional metal cyanide complexes to be adsorbed into the resin. If cobalt is present in the effluent, the possible polymerization of adsorbed cobalt cyanide complexes under strongly acidic conditions will poison the resins [3,11,13].

Solvent extraction (SX) technology offers an alternative method for metal and cyanide recovery from waste cyanide solution. Since the extraction kinetics of the SX systems are usually fast and the process can operate through continuous stages, relative small organic inventory will be required. Ideally, the SX of metal species from waste cyanide solution should be accomplished at weak to moderately strong alkaline conditions since the cyanidation process is usually carried out at pH 10–11. The use of LIX 7820 (a solvent mixture of Aliquat 336 and nonylphenol) to recover copper from cyanide solution has been proposed [17,18]. The extraction and stripping of metal cyanide complexes is believed to occur via an ion-exchange mechanism:

$$(Q^+X^-)_{org} + (HP)_{org} + OH^- \leftrightarrow (Q^+P^-)_{org} + X^- + H_2O \qquad (1)$$
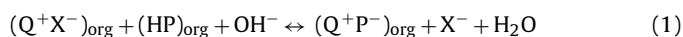
where Q^+ is the quaternary ammonium cation, HP is the protonated form of the nonylphenol, and X^- is the extracted anion [19]. Under low pH conditions, nonylphenol is protonated and the quaternary ammonium compounds extract an anion from the aqueous phase. Under more highly alkaline conditions, nonylphenol starts to be significantly converted to the highly hydrocarbon-soluble phenoxide anion (P^-) and forms an ion pair with the quaternary ammonium cation (Q^+P^-). Consequently, the extracted anion will be gradually expelled to the aqueous phase with increasing equilibrium pH. The recovery of copper and cyanide by LIX 7950 (based on formulation of a trialkylguanidine but with a higher concentration than LIX 79) has also been investigated [20,21]. The guanidine extractant exhibits a pK_a of approximately 12. It is capable of being protonated to form an ion pair with the metal cyanide complexes at pH below 11 and is converted to the neutral guanidine functionality resulting in metal stripping. The standard chemical reaction for loading and alkali stripping can be represented as below:

$$nRG_{org} + nH_2O + A^{n-} = (RGH^+)_nA_{org}{}^{n-} + nOH^- \qquad (2)$$
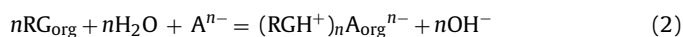
where RG represents the extractant and RGH^+ is the protonated form of the extractant. A^{n-} represents the anion, which has n negative charge [21,22]. These investigations proved that copper can be effectively extracted from alkaline cyanide solutions by the proposed extraction systems and the cyanide levels and the presence of other anions may potentially affect the extraction of copper and cyanide.

In this work, continuous laboratory tests on the use of the reagents LIX 7950 and LIX 7820 for copper and cyanide recovery from waste cyanide solution have been conducted which focus on the extraction and stripping behaviors of copper cyanides under different cyanide to copper molar ratios. The effect of cyanide levels on extraction of metal cyanide complex anions commonly occurring in cyanide tailings with the extractants was also examined. The potential application of the two extraction systems for recovery of copper and cyanide from waste cyanide solution is discussed.

Table 1
The major component in the mixture solution.

Component	Concentration (mg/L)
Ag (I)	49.5
Au (I)	53.5
Cu (I)	46.5
Zn (II)	55.7
Ni (II)	47.2
Fe (II)	52.1

2. Experimental

2.1. Materials

The extractants LIX 7950 and LIX 7820 were used as supplied by the manufacturer. LIX 7950 is a trialkylguanidine extractant. The solvent mixture LIX 7820 comprises the quaternary amine, Aliquat 336, combined with 4-nonylphenol in a 1:2 molar ratio [17]. N-dodecane and n-octane were used as the diluents for the two extractants, respectively, and 1-dodecanol was used as the modifier for LIX 7950. Synthetic copper cyanide solutions were made up from CuCN and NaCN. A mixed solution of metal cyanides with varied cyanide content was prepared from the corresponding metal cyanides (AuCN, AgCN, $Zn(CN)_2$, $Ni(CN)_2$ $4H_2O$, $K_4Fe(CN)_6$ $3H_2O$, respectively) and the content of metal species is shown in Table 1. All the chemicals were reagent grade.

2.2. Procedure

The extraction and stripping tests were carried out in a sealed beaker and mixing was provided by a mechanical agitator with glass impellers. Equilibrium pH was adjusted by direct addition of concentrated H_2SO_4 solution (50% v/v) or NaOH solution (2 mol/L). All the extraction and stripping tests were conducted at indoor temperature. Initial investigations showed that the equilibrium between the two phases could be established rapidly for both extractants. Both solution pH and copper extraction were constant after 2–3 min of mixing. In subsequent experiments, 10 min contact time was arbitrarily chosen for equilibrium establishment. When equilibrium was reached, phase separation was conducted in a separatory funnel. Samples of the aqueous solution were filtered to remove any entrained organic before analysis. The organic samples for stripping were filtered with 1 PS Phase Separation paper. The copper content in the aqueous sample was analyzed by atomic absorption spectrophotometry (AAS) and copper in the organic phase was calculated by mass balance. Total cyanide content in the aqueous solution was determined by a standard distillation method and the cyanide content in the organic phase was calculated by mass balance. Copper and total cyanide content in some selected organic samples were also analyzed. Sodium hydroxide solution (1 mol/L) was used to thoroughly strip the loaded copper and cyanide (by three times contact at a phase ratio of unity) and then the same procedure as above was used to determine copper and total cyanide in the resulting strip solution. It was confirmed that good agreement between analytical and calculated results was obtained.

3. Results and discussion

3.1. Extraction of copper cyanide solution

The extraction of copper and cyanide by the extractants from synthetic copper cyanide solutions with different copper and cyanide contents was examined. The extraction tests were carried out with a phase ratio of unity and the equilibrium pH was controlled at 10 ± 0.05. The extraction results for LIX 7950 and LIX 7820 are shown in Tables 2 and 3, respectively. For both extractants, the
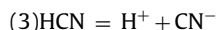
Table 2
The extraction of Cu and CN with LIX 7950 under different CN/Cu ratios (org., 10% v/v LIX 7950 and 50 g/L 1-dodecanol in n-dodecane; A/O = 1, pH 10.50 ± 0.05).

| Initial aqueous		Extraction (%)		CN/Cu ratios	
Cu (mmol/L)	CN/Cu	Cu	CN	CN/Cu in aq.	CN/Cu in org.
1.57	5	99.8	61.0	975.0	3.06
4.72	5	88.1	52.6	19.9	2.99
7.87	5	64.0	38.3	8.6	2.99
1.57	10	99.1	29.9	778.9	3.02
4.72	10	79.7	24.5	37.3	3.07
7.87	10	54.4	16.0	18.4	2.94

extraction of copper and cyanide decreases with an increase of copper content. For the solutions with same copper content, the higher the cyanide to copper molar ratio (CN/Cu), the lower the extraction of copper and cyanide. The mass balance of copper and total cyanide shows that the CN/Cu ratios in the aqueous phase after extraction were much higher than the initial CN/Cu values, but the molar ratio of loaded cyanide to loaded copper in organic phase were all close to three even when the initial CN/Cu ratio was as high as 10. The analysis of the stripping solutions of the loaded organic samples further confirmed this result. The speciation calculation of copper and cyanide indicates that under the experimental conditions, copper mainly occurs as $Cu(CN)_3{}^{2-}$ and $Cu(CN)_4{}^{3-}$ and cyanide as free cyanide (CN^-) and complexed cyanide in the aqueous phase [21]. According to the extraction results (Tables 1 and 2), it can be then deduced that the extractants preferentially extract $Cu(CN)_3{}^{2-}$ over $Cu(CN)_4{}^{3-}$ and CN^- and only $Cu(CN)_3{}^{2-}$ has been extracted by the extractants. The effect of phase ratio on extraction of copper and cyanide with the LIX 7950 further confirmed the speculation. The extraction isotherms of copper and cyanide with the extractant were established by varying the volume ratio of the aqueous phase to the organic phase (A/O) from 2:1 to 1:8. The equilibrium pH was controlled at 10.50 ± 0.05. The results are shown in Figs. 1 and 2. According to Fig. 1, two stages of extraction with the designated extractant solution are sufficient for above 93% of copper extraction efficiency (CN/Cu = 5, under equilibrium pH 10.50 ± 0.05, A/O = 1.2).

The extraction isotherms of cyanide are different from those of copper, when a high A/O ratio (1/8) is used, the copper content in the aqueous phase tends to zero, but the cyanide content in the aqueous phase is still significant. Accordingly, the extraction efficiency of total cyanide is only about 50% and the rest remains in the raffinate. The speciation calculation shows that most of the free cyanide remains in the aqueous solution due to the preferential extraction of $Cu(CN)_3{}^{2-}$ over $Cu(CN)_4{}^{3-}$ and CN^- by the extractant. Under the experimental conditions, the extraction process of copper and cyanide with the extractants is believed to be controlled by the following equilibriums (the formations of Cu^+, $CuCN$, $Cu(CN)_2{}^-$ and Cu_2O are negligible [21]):

$$(3)\ HCN = H^+ + CN^-$$

$$(4)\ Cu(CN)_3{}^{2-} + CN^- = Cu(CN)_4{}^{3-}$$

Table 3
The extraction of Cu and CN with LIX 7820 under different CN/Cu ratios (org., 2% v/v LIX 7820 in n-octane; A/O = 1, pH 10.50 ± 0.05).

Initial aqueous		Extraction (%)		CN/Cu ratios	
Cu (mmol/L)	CN/Cu	Cu	CN	CN/Cu in aq	CN/Cu in org
1.57	5	96.9	58.5	74.7	3.01
3.94	5	63.2	38.6	8.3	3.05
7.87	5	35.2	21.3	6.1	3.02
1.57	10	93.4	28.6	107.9	3.06
3.94	10	57.4	17.3	19.4	3.02
7.87	10	32.3	9.7	13.3	2.99
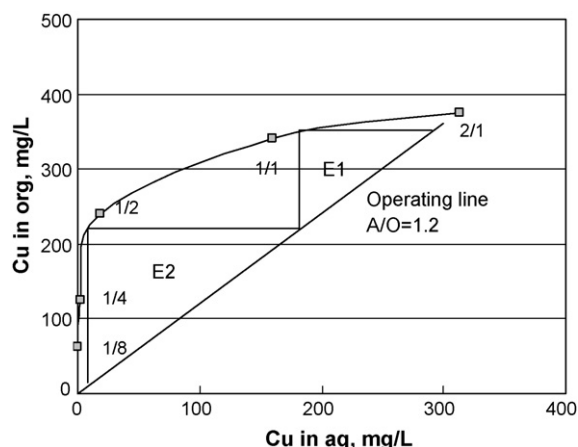
Fig. 1. The distribution isotherms of copper extraction with LIX 7950 (org., 10% v/v LIX 7950 and 50 g/L 1-dodecanol in n-dodecane; aq., CN/Cu = 5, pH$_{eq}$ 10.50 ± 0.05).

$$(5)\ 2RG_{org} + 2H_2O + Cu(CN)_3{}^{2-} = 2(RGH)Cu(CN)_{3\,org} + 2OH^-$$
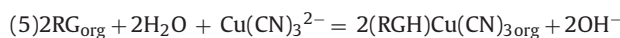
where RG_{org} represents the extractant.

3.2. Extraction of the mixture solution

A host of metal cyanide anions could be present in cyanide effluents. SX of mixtures of metal cyanides (Table 1) by the extractants was examined. The extraction tests were conducted at phase ratio of unity and pH controlled at 10.50 ± 0.05. The distribution constants of the metals (D, defined as the ratio of the extracted metal concentration over the metal concentration in the aqueous phase when equilibrium pH is established) with the two extractants under different cyanide levels are shown in Figs. 3 and 4, respectively. As expected, copper extraction decreases significantly with increasing cyanide levels. Silver extraction also decreases evidently with an increase of the cyanide content. Comparatively, the effect of cyanide level on extraction of gold, zinc, nickel and iron is negligible. According to the distribution constants of the six metals, a selectivity order is observed as follows: Au > Ag > Zn > Ni > Cu > Fe.

The result is in accordance with those reported in the literatures [19,21–25]. Obviously, the extractants have exhibited different affinities for the metal cyanide species in the
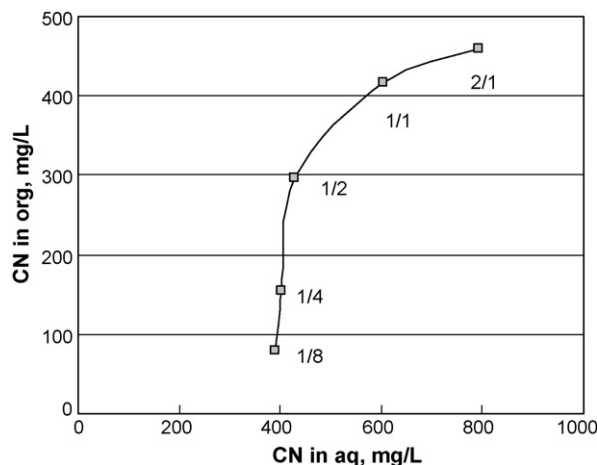
Fig. 2. The distribution isotherms of cyanide extraction with LIX 7950 (org., 10% v/v LIX 7950 and 50 g/L 1-dodecanol in n-dodecane; aq., CN/Cu = 5; pH$_{eq}$ 10.50 ± 0.05).
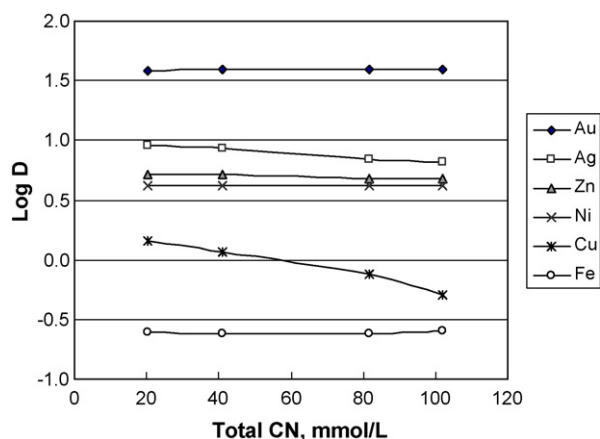
Fig. 3. The distribution constants of the metals with LIX 7950 under different cyanide levels (org., 10% v/v LIX 7950 and 50 g/L 1-dodecanol in n-dodecane; aqueous solution as Table 1; A/O = 1, pH_{eq} 10.50 ± 0.05).
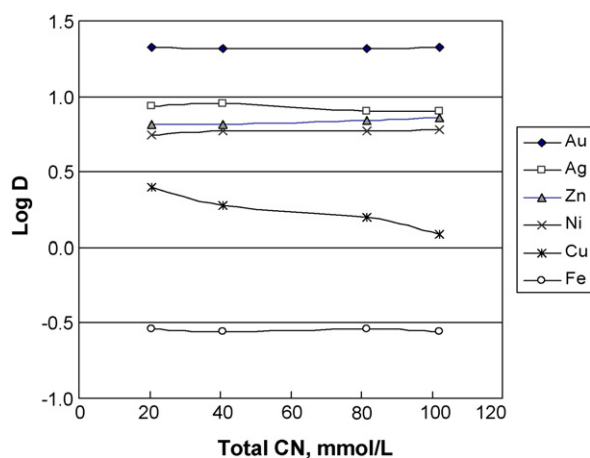
Fig. 4. The distribution constants of the metals with LIX 7820 under different cyanide levels (org., 2% v/v LIX 7820 in n-octane; aqueous solution as Table 1; A/O = 1, pH_{eq} 10.50 ± 0.05).

solution. The mass balance calculation of the metals and cyanide indicates the extracted cyanide mainly occurred as complexed cyanide (supposed the extracted complexes are $Au(CN)_2^-$, $Ag(CN)_2^-$, $Cu(CN)_3^{2-}$, $Zn(CN)_4^{2-}$, $Ni(CN)_4^{2-}$, $Fe(CN)_6^{4-}$) and most of the free cyanide remains in the aqueous phase after extraction.

The phenomenon of preferential extraction and the selectivity order can be explained by charge density effect and geometrical factors. It is believed that the ion that has a lower charge density (lower charge and/or larger size) will be preferentially extracted over the ions with higher charge density by the amine extractants [23,24]. In general, those complexes with lower CN^- coordination numbers will be extracted preferentially by the amine extractants

over those with higher numbers and the lower charged metal cyanide complexes will be extracted preferentially over higher charged complexes. For example, $Cu(CN)_3^{2-}$ ion will be preferentially extracted over $Cu(CN)_4^{3-}$ since $Cu(CN)_3^{2-}$ has a lower charge than $Cu(CN)_4^{3-}$ and exhibits a lower overall charge density. Since a higher CN/Cu ratio favors the formation of $Cu(CN)_4^{3-}$, about 83% of copper occurs as $Cu(CN)_3^{2-}$ and the 17% of copper as $Cu(CN)_4^{3-}$ when CN/Cu ratio is 5 and when CN/Cu ratio increases up to 10, about 47% of copper occurs as $Cu(CN)_3^{2-}$ and 53% of copper as $Cu(CN)_4^{3-}$, a high level of cyanide tends to depress the extraction of copper by the extractants [21]. Cyanide levels may exhibit similar effect on silver extraction with the extractants. The equilibrium reactions between silver and cyanide can be simply expressed as following (supposed the formation of Ag_2O and AgCN is negligible) [26]:

$$Ag^+ + 2CN^- = Ag(CN)_2^- \qquad (6)$$

$$Ag(CN)_2^- + CN^- = Ag(CN)_3^{2-} \qquad (7)$$

$$Ag(CN)_3^{2-} + CN^- = Ag(CN)_4^{3-} \qquad (8)$$

Since $Ag(CN)_2^-$ ion has a lower charge than $Ag(CN)_3^{2-}$ and $Ag(CN)_4^{3-}$ ions, it will exhibit a lower overall charge density and will be preferentially extracted by the extractants. A high cyanide level favors the formation of $Ag(CN)_3^{2-}$ and $Ag(CN)_4^{3-}$ and thus depresses silver extraction. Although CN^- ion has only one charge, its small size makes it tend to form a tight salvation shell in water and leads to its poor extraction by the extractants, which is a relative large molecule [27,29]. It was believed that in the mixture solution, gold coordination is relative simple and only complex $Au(CN)_2^-$ ions occur and zinc, nickel and iron tend to form the stable cyanide complexes, $Zn(CN)_4^{2-}$, $Ni(CN)_4^{2-}$ and $Fe(CN)_6^{4-}$, respectively. Their affinities with the extractant molecules are much higher than that of free cyanide and subsequently the extraction of these metals by the extractants is not sensitive to cyanide levels. The singly charged $Au(CN)_2^-$ and $Ag(CN)_2^-$ ions exhibited stronger affinity with the extractants than the other metal cyanide complexes. The bigger molecular size of $Ag(CN)_2^-$ than $Ag(CN)_2^-$ favors its greater extractability by the extractants. Though all with the same charge, the charge density for $Zn(CN)_4^{2-}$ and $Ni(CN)_4^{2-}$ are lower than for $Cu(CN)_3^{2-}$ as they have four coordinated CN^- ligands and $Cu(CN)_3^{2-}$ has three. The formation of the highest charged $Fe(CN)_6^{4-}$ in cyanide solution results in the least extraction of iron by the extractants.

Geometrical factors could be another cause for selective extraction of cyanide complexes [24,28,29]. $Cu(CN)_4^{3-}$ has a tetrahedral shape compared to $Cu(CN)_3^{2-}$ and $Cu(CN)_2^-$ which are triangular planar and linear, respectively and the extraction of $Cu(CN)_4^{3-}$ would require three extractant molecules per copper while $Cu(CN)_3^{2-}$ would only require two. As a result, the former was poorly extracted compared to the other two. Similarly, $Ag(CN)_3^{2-}$ ion has a trigonal shape and $Ag(CN)_4^{3-}$ ion is tetrahedral compared to $Ag(CN)_2^-$ ion, which is linear and the extraction of $Ag(CN)_2^-$ ion would only require one extractant molecule per Ag while $Ag(CN)_3^{2-}$

Table 4
Stripping of loaded copper and cyanide by NaOH and NaOH + NaCN solutions.

Stripping solution	LIX 7950[a]		LIX 7820[b]	
	Cu stripping (%)	CN stripping (%)	Cu stripping (%)	CN stripping (%)
0.5 M NaOH	91.0	90.8	89.5	89.6
1 M NaOH	93.1	93.4	91.9	92.3
0.5 M NaOH + 0.002 M NaCN	91.9	92.3	90.0	90.4
0.5 M NaOH + 0.01 M NaCN	92.7	92.5	91.2	91.3

[a] 10% (v/v) LIX 7950 in dodecane, 50 g/L 1-dodecanol, initial loaded Cu and total CN are 3.62×10^{-3} M and 1.10×10^{-2} M, respectively, unity O/A ratio.
[b] 2% (v/v) LIX 7820 in n-octane, initial loaded Cu and total CN are 2.16×10^{-3} M and 6.52×10^{-2} M, respectively, unity O/A ratio.

and $Ag(CN)_4{}^{3-}$ would require two and three, respectively. The preferential extraction of $Zn(CN)_4{}^{2-}$ over $Ni(CN)_4{}^{2-}$ is probably due to the fact that $Zn(CN)_4{}^{2-}$ has a tetrahedral shape while $Ni(CN)_4{}^{2-}$ is square planar [30]. The distribution of charge over $Zn(CN)_4{}^{2-}$ is more uniform than $Ni(CN)_4{}^{2-}$ and favors the combination with the extractant molecules.

3.3. Stripping of loaded copper and cyanide

It is desirable to recover the loaded metals using a simple technique so that the solvent can be reused. Varied solutions of NaOH and NaCN have been tested for stripping of the loaded copper and cyanide and the results are summarized in Table 4. For both extractants, the loaded copper and cyanide can be effectively stripped by moderate strong NaOH solutions (0.5–1 mol/L). For LIX 7950, at phase ratio of unity, about 93% of copper and cyanide can be stripped off by 1 mol/L NaOH solution. For LIX 7820, above 92% stripping efficiency of copper and cyanide can be obtained with the same stripping solution. The presence of small amount of NaCN in the stripping solution favors the stripping of copper and cyanide. This is probably due to the formation of the less extractable $Cu(CN)_4{}^{3-}$ when extra cyanide was added.

3.4. Potential application

According to the research results, the extractants can be used in an incorporated SX circuit for separation of copper cyanide complexes from those waste cyanide solutions arising from the gold plants in mining industry. Through the SX circuit, copper and partial cyanide can be concentrated into a small volume of strip solution, which can be further treated by EW, AVR or similar processes to recover copper products and cyanide. Due to the preferential extraction, most of the free cyanide will remain in the raffinate, which allows for the potential recycling of the barren solution to the cyanidation process. The need for clarified feed solution for SX will not be a limitation while targeting heap-leaching solutions, overflow stream or dam return water from tailings. For operations using carbon-in-pulp (CIP) for the recovery of gold, it will be necessary to thicken and wash the solids in order to produce a clarified feed solution for SX circuit. However, the cost of solid/liquid separation may be substantial and the economic factors must be taken into consideration. Gold, silver and zinc may be also extracted into the organic phase if they are present in the effluents. Most of the iron cyanides will accumulate in the barren solution and their potential effect on the cyanidation process has to be considered [30].

4. Conclusions

The recovery of copper and cyanide from cyanide solution by a guanidine extractant (LIX 7950) and the modified amine (LIX 7820) has been investigated. Copper can be effectively extracted from alkaline cyanide solutions by both extractants. The preferential extraction of $Cu(CN)_3{}^{2-}$ over $Cu(CN)_4{}^{3-}$ and free cyanide (CN^-) has been confirmed. High cyanide levels tend to depress copper and silver cyanides loading, but exhibit insignificant effect on extraction of gold, zinc, nickel and iron. A selectivity order of Au > Ag > Zn > Ni > Cu > Fe for both extractants has been observed.

The effective stripping of loaded copper and cyanide can be accomplished by using moderate strong sodium hydroxide solutions (0.5–1 mol/L). The presence of small amount of NaCN in the stripping solution favors the stripping of the loaded copper and cyanide.

A possible solution to the separation of copper cyanide species and free cyanide in cyanide effluents is suggested by which copper can be extracted and concentrated into a small volume of solution for further recovery and the barren cyanide solution recycled to the cyanidation process.

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