

Recovery of copper, nickel and cobalt from acidic pressure leaching solutions of low-grade sulfide flotation concentrates

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Abstract

Recovery of copper, nickel and cobalt from the acidic pressure leaching solutions of Jinbaoshan (YN Province, PRC) low-grade sulfide flotation concentrates was investigated. The proposed technique includes four major steps: (1) the acidity adjustment of the acidic pressure leaching solutions; (2) solvent extraction (SX) separation of copper by organic reagent XD5640, and then stripped from the loaded organic phase by H₂SO₄ solution for copper recovery; then (3) iron in raffinates after copper extracting is selectively removed by high-temperature hydrolysis precipitation in an autoclave; and lastly (4) nickel and cobalt are selectively precipitated by Na₂S from the final solutions after removing iron. The experimental results for treating 1 L acidic leaching solutions per batch by this new technique were reported, and some evaluation and further comparisons with previous investigations were also carried out. It was reported that the total percent recovery of Cu could reach 95% or more, and that of Ni and Co were all more than 99%. In the processing, the percent removal of impurities, such as Fe, Mg and Ca, were all also near to 99%.

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1. Introduction

Jinbaoshan proterozoic platinum mine (Yunnan Province, PRC) is the second largest platinum mine in China. It is estimated that the mine has a potential reserve of about 100 tons of Pt + Pd with an average composition of (Pt + Pd) 1.45 g/t, Cu 0.08%, Ni 0.17%, S 0.61% and Fe 9.88% (Zhu, 1999). After flotation processing, the content of (Pt + Pd) is increased from 1.45 g/t (crude) to 86 g/t (flotation sulfide concentrates), and the content of (Cu + Ni) is increased to 6–7% (Liu and Li, 2002). The traditional pyro-metallurgical process is not suitable for treating the as-is flotation concentrates due to the technical difficulties and economic concern over its low content of CuNiS and precious metals. Matte-smelting process has

also serious environmental impacts and the lengthy overall flowsheet results in unavoidable losses of platinum group metals (PGMs). When the mattes are leached by acid to separate base metals, the content of PGMs in the final concentrated residues is only 6–10%, leading to difficulties in the latter PGM refining process. To avoid these limitations a patented new technique involving a two-stage pressure leaching process for the extraction of PGMs from the sulfide flotation concentrates was developed (Chen et al., 2002, 2004). The sulfides were firstly pre-oxidized by pressure acid-leaching to remove the base metals CuNiCo and enrich PGMs; the residues were then subjected to pressure cyanidation to selectively recover the PGMs. Cementation of the final cyanide solution with zinc powder resulted in a PGMs concentrate containing about 70–90% of precious metals. The PGMs enriched cementation residues can be sold as PGM concentrates to refineries for further refining. In the acidic pressure leaching of Jinbaoshan flotation sulfide concentrates, almost all (>99%) of Cu, Ni and Co

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sulfides are oxidized to sulfates and leached into the solution, while most (>94%) of PGMs are leaved and enriched into the leach residues, which results in a separation between platinum metals and Cu, Ni and Co. Although most (82–90%) of the iron is precipitated as Fe_2O_3 and $\text{FeO}(\text{OH})$ and stays in the pressure acidic leach residues, it has no influence on latter cyanide selectively leaching of PGMs. The previous studies (Huang et al., 2006a,b) were more focused on enrichment and extraction of PGMs from that mentioned Jinbaoshan sulphide flotation concentrates, but no further reports on how to recover Cu, Ni and Co from the acidic pressure leaching solutions were carried out. It is found that the released acid from the pressure leaching of sulfides is neutralized by Ca and Mg oxides in concentrates, which is beneficial for obtaining a solution with appropriate acidity to recover Cu, Ni and Co from it by possible solvent extraction (SX) separation techniques.

This report is focusing on the processing of the acidic pressure leaching solution from Jinbaoshan flotation sulfide concentrates, for the purpose of recovering of Cu, Ni and Co from that solution. The evaluation of mass balance of metals (CuNiCo) in the process is carried out also. The processing of the acidic pressure-leaching residue for further enrichment and extraction of PGMs and the studies on the behaviors of Cu, Ni and Co in acidic pressure leach process was discussed in other reports (Chen and Huang, 2006; Huang et al., 2006a,b).

The chemical component analysis of the acidic pressure leaching solutions indicated that the concentration of Cu, Ni and Co were about 5–10 g/L, 3–5 g/L and 0.1–0.5 g/L, respectively, however the impurity content of Fe, Ca and Mg etc. in the solutions were also 2–5 g/L. Obviously, not only low were the concentrations of the objective metals (CuNiCo), but they were also almost the same as that of co-existed impurity elements. The reported extractant systems were not suitable for that acidic pressure leaching solution due to its lower content of Cu, Ni, Co, and higher influence and percent co-extraction of impurity metal ions. This brought further difficulties for separation and recovery of Cu, Ni and Co from that leaching solution.

As for the SX processes of Cu, Ni and/or Co from their hydrometallurgical leaching solutions, the reported candidate solutions all had higher content of main objective metals of Cu, Ni and/or Co, while the impurity content of Fe, Ca and Mg etc. were low enough even unnecessary to be considered their influences on the recovery of Cu, Ni and/or Co (Yang and Qiu, 1998). As practiced in Hajavarta refinery of Outokumpu Co. (South Africa), the chemical analysis of candidate solutions were averagely Ni 130 g/L, Co 0.8–1 g/L, Cu 1 g/L, Mg 8 g/L, Ca 4.6 g/L and Fe 0.1 g/L; in Jilin Nickel Co. (Jilin Province, PRC) were Ni 120–130 g/L, Co 1–2 g/L, Cu 0.003 g/L, MgO 0.042 g/L, CaO 0.056 g/L and Fe 0.004 g/L; in Jinchuan Co. (Gansu Province, PRC) were Ni 65 g/L, Co 7–8 g/L, Cu 3–5 g/L, Fe <0.1 g/L; and in Xinjiang Fukan Co.

(Xinjiang Province, PRC) were Ni 80.3 g/L, Co 4.07 g/L, Cu 0.136 g/L, Fe 0.183 g/L, CaO 0.2 g/L and MgO 1.62 g/L. None of these plants' solutions has the same chemical compositions as mentioned in present paper. Due to these reasons, an appropriate technique for recovery of Cu, Ni and Co from that acidic pressure leaching solutions of Jinbaoshan flotation concentrates should be further investigated.

Since 2004, the following two processing options have been proposed:

- (1) Copper was firstly solvent extracted and recovered by organic reagent Lix 973 or Bk 992; then iron could be further removed by the ferro-vanadium salt hydrolysis method at room temperature; nickel and cobalt were precipitated finally by NaOH from the solutions after removing iron (Liu, 2003).
- (2) Copper was firstly solvent extracted and recovered by organic reagent XD5640; iron and calcium were further separated by P204 solvent extraction; the remnant calcium and most of magnesium were then precipitated by NaF; and nickel and cobalt were precipitated by NaOH from the final solutions (Li et al., 2005).

Preliminary extraction investigations show that:

- (1) For option 1, percent recovery of copper could reach higher than 97%, however that of nickel and cobalt were low (only 70–85%) because of the co-precipitation of them during the processing of removing iron by the ferro-vanadium salt hydrolysis at room temperature. In addition, calcium and magnesium still contained in the solutions after removal of Fe, which resulted in the final obtained NiCo residues still containing many of Ca, Mg, etc. impurities.
- (2) For option 2, although calcium and magnesium impurities could be effectively removed before NiCo precipitation, poisonous fluorides were introduced and difficulties still existed in future practical industrial application because of the lengthy processing flowsheet and high reagent consumption, which will result in higher economic cost.

In present paper, another possible new improved technique is suggested for recovery of CuNiCo from that acidic pressure leaching solutions. Namely, copper is firstly extracted by organic reagent XD5640; iron in the extraction raffinates is then selectively removed by high-temperature hydrolysis precipitation; and lastly nickel and cobalt are selectively precipitated by Na_2S from the final solutions. By this new technique, not only the objective metals (CuNiCo) are extracted at high percent recovery, but also those co-existed impurities, such as Fe, Ca and Mg, can be effectively removed off. The final obtained solutions/concentrates contained a higher content of Cu, Ni and Co. The experimental results for treating 1 L acidic leaching solutions per batch by this new technique are reported,

and some evaluation and further comparisons with previous reports are discussed.

2. Experimental

2.1. Samples and chemical reagents

Table 1 shows the typical chemical composition of acidic pressure leaching solutions from Jinbaoshan flotation concentrates. The solutions are mainly sulfate media from pressure H_2SO_4 leaching processing (temp. 200 °C, 50 g/L H_2SO_4 , time 4–6 h, pressure 1.8 MPa and L:S ratio of 4:1 (see details in Huang et al., 2006a)), and have the average acidity of pH 0.5–1, and the concentration of SO_4^{2-} 0.37 mol/L.

XD5640, produced by Tianjin chemical factory, and C_{12} alkane, produced by Shanghai chemical factory, are industrial grade reagents; HCl, H_2SO_4 , NaClO_3 , Na_2S and NaOH are all analytical pure grade reagents.

2.2. Testing procedure

The experimental procedure includes the following four major steps: (1) The adjustment of the acidity of the acidic pressure leaching solutions. Because the acidity of pressure acid leaching solutions were pH 0.5–1, and the effective pH for selectively extracting of copper by the extractant XD5640 is pH 2 (Chen et al., 1998), then before the SX separation of copper, the solution acidity was adjusted to fit the latter processes. (2) SX separation of copper by organic reagent XD5640, and then stripped from the loaded organic phase by H_2SO_4 solution for copper recovery. (3) Iron in raffinates after copper extracting is then removed by high-temperature hydrolysis precipitation in an autoclave. Finally (4) nickel and cobalt are precipitated by Na_2S from the final solutions after iron removal. For steps (3) and (4), carefully controlled the reaction conditions are needed for selective removal of Fe and precipitation of Ni and Co.

The 1 L batch tests for recovery of Cu, Ni and Co from the acidic pressure leaching solutions were carried out at the following conditions of:

The pH adjustment of the acidic pressure leaching solution: temp. 25 °C, 5% (V/V) NaOH solution added gradually. The pH of the solution was adjusted equaling to 2.

SX and stripping of copper: the extraction organic phase was composed of XD5640 and C_{12} alkane (XD5640 as the extractant, with the concentration of 40% (V/V), and C_{12} alkane as the diluent reagent). The organic phase was contacted with the acidic pressure leaching solutions after pH

adjustment for extraction and separation of copper, and the loaded organic phase was then contacted with 3 mol/L H_2SO_4 solution to strip and recover copper. The phase ratio of the organic to aqueous solution for the extraction and stripping step were 1:1. The reaction temperature of these processes were 25 °C, and the mixing and settling time were of 1–3 min.

Iron removal by high-temperature hydrolysis precipitation: temp. 200 °C, O_2 pressure of 2.0 MPa and time 30 min.

Nickel and cobalt selectively precipitated by Na_2S : temp. 60 °C, 25 g/L Na_2S solution added gradually. The final pH of the solution was controlled equaling to 8.

2.3. Analysis methods and calculation of metal recovery

Metal content in the solution samples were analyzed by either atomic absorption spectroscopy (AAS) or inductively coupled plasma (ICP), dependent on the content level. The extraction recovery of metals is calculated by back-calculated from solution analysis results.

The reported data in experiments, if not specially indicated, represented an average of multiple analyses. For different batch tests (multiple trials), the data variation was indicated. The measurement error for higher concentration data (>1 g/L) was <5%, and for lower ones <10%.

3. Results and discussion

3.1. The acidity adjustment of the acidic pressure leaching solutions

The chemical analysis results of the acidic pressure leaching solutions after the pH adjustment are as shown in Table 2. Here it should be indicated that before the acidity adjustment of the acidic pressure leaching solutions, the original solutions were firstly pre-treated by activated copper powder cementation to recover some dissolved platinum metals after pressure leaching of flotation concentrates due to the existence of chloride in water (see Huang et al., 2006a). Therefore, the average content of copper in the candidate solutions before acidity adjustment (listed in Table 2) is slightly higher than that in original solutions (listed in Table 1). Likewise, the concentrations of other metals, such as Ni, Co, Fe, Ca and Mg are also slightly higher because the copper powder cementation process was carried out at a high temperature (60 °C) and the solutions were condensed.

From the data listed in Table 2, it is shown that the content of copper, nickel and cobalt in solutions have almost no loss in the acidity adjustment process.

3.2. SX separation of copper and its stripping from the pregnant organic phase

One litre of pressure acidic leaching solution after pH adjustment was contacted with the mentioned organic

Table 1
Chemical composition analysis of the acidic pressure leaching solution from Jinbaoshan flotation concentrates

| Element | Cu | Ni | Co | Fe | Ca | Mg |
|------------------------------|------|------|------|------|-------|------|
| Content (g L ⁻¹) | 9.04 | 3.36 | 0.24 | 2.12 | 0.134 | 5.88 |

Table 2

Chemical analysis results of the acidic pressure leaching solutions after acidity adjustment

| Samples (g L ⁻¹) | Batch no. | Volume/L | Cu | Ni | Co | Fe | Ca | Mg | pH |
|------------------------------|-----------|----------|-------|------|------|------|------|------|-------|
| Solution before adjustment | | 1.00 | 12.08 | 5.68 | 0.35 | 3.64 | 0.21 | 7.53 | 0.5–1 |
| Solution after adjustment | No. 1 | 1.21 | 9.97 | 4.63 | 0.32 | 3.00 | 0.17 | 6.14 | 2 |
| | No. 2 | 1.17 | 10.74 | 4.74 | 0.32 | 3.06 | 0.19 | 6.58 | 2 |
| | No. 3 | 1.18 | 10.02 | 4.59 | 0.28 | 3.00 | 0.20 | 6.30 | 2 |

phase for extraction and separation of copper. After four steps of continuous extractions, (Note: That extracting numbers depends on the concentration of copper in the original solution and the loaded capacity of XD 5640.) the loaded organic phase was then contacted with 3 mol/L H₂SO₄ solution to strip and recover copper. The testing results are as shown in Table 3.

Based on the data listed in Table 3, it was calculated that the percent copper extraction by XD 5640 reached 99.9%, and its percent stripping recovery reached 95.5%. The total recovery of copper in the process reaches 95% or more. It is also indicated that the co-extraction rate of nickel and cobalt was near to 0%, with no losses in the process. The extractant-XD5640 has indeed good extraction selectivity and percent recovery for copper. From the stripping solution, copper could be recovered as metallic copper by electrodeposition, or as crystallize CuSO₄ compounds after further removal of other impurities.

In previous report, Li et al. (2005) compared the extraction ability of Lix 973 and Bk 992 with that of XD 5640 for copper recovery. It was found that XD 5640 had a higher percent extraction and better extraction selectivity for other co-existed metal impurities than that of Lix 973 and Bk 992. More importantly, XD 5640 is cheaper than the other two, therefore it is more suitable for future industrial application.

3.3. Selectively removing iron in raffinates after copper extracting by high-temperature hydrolysis precipitation

The solutions after copper SX separation were introduced into an autoclave, and reacted at 200 °C and O₂ pressure of 2.0 MPa for 30 min, the results for removing iron are shown in Table 4.

Calculated based on the data listed in Table 4, the iron percent precipitation is higher than 97.0%, and the percent loss of nickel and cobalt are all near to 0%.

Previous investigation (Liu, 2001, 2003; Zhang et al., 2000) suggested removing iron by the ferro-vanadium salt

hydrolysis method at atmospheric pressure. Because the iron existed in the above raffinate solutions after copper extracting as both Fe²⁺ and Fe³⁺, before the precipitation, some amount of NaClO₃ was added in advance into the solution and heating for about 30 min to oxidized Fe²⁺ totally as Fe³⁺, and then added the lime milk to adjust the solution acidity of pH equal to 4–4.5. By this method, most (about 99%) of the iron could be precipitated, as the data shown in Table 5.

However, in that process the loss of nickel reached 25%, and that of cobalt was 14%. At the same time, the content of calcium in the solutions also increased, which brings further difficulties for latter recovery of nickel and cobalt.

Separation of iron by P204 solvent extraction method had also been employed in the previous researches (Yang and Liu, 1988; Li et al., 2005). Although some calcium were removed in the processing of iron solvent extraction by P204, it was difficult to control the co-extracting process of nickel and cobalt. The losses of them were still unavoidable and to strip iron from the loaded P204 required a high acidity (6 N) HCl solution.

3.4. Selectively precipitation of nickel and cobalt by sodium sulfide from the final solutions

For the treatment of the solution after removing iron, there are two possible ways to recover nickel and cobalt. Those are: (1) further removing calcium and magnesium impurity elements to obtain a relatively pure solution, then separating and recovering of nickel and cobalt from that solution; and (2) directly and selectively recovering of nickel and cobalt from that solution.

It was reported that P507 can extract calcium and magnesium effectively, however it was found that in the processing, nickel and cobalt could also be co-extracted at the same pH conditions (Liu et al., 2002). There were also investigations (Tsakiridis and Agatzini, 2004) on selectively solvent extracting of nickel and cobalt by Cyanex301, without co-extraction of manganese and calcium. However, the

Table 3

Extraction and stripping of copper

| Batch no. | Samples (g L ⁻¹) | Volume/L | Cu | Ni | Co | Fe | Ca | Mg |
|-----------|------------------------------|----------|-------|-------|--------|-------|-------|------|
| No. 1 | Solution before extraction | 1.00 | 10.74 | 4.74 | 0.32 | 3.06 | 0.19 | 6.58 |
| | Solution after extraction | 1.00 | 0.01 | 4.74 | 0.32 | 2.96 | 0.19 | 6.12 |
| | Solution of stripping | 1.00 | 10.25 | 0.071 | 0.0028 | 0.074 | 0.036 | 0.12 |
| No. 2 | Solution before extraction | 1.00 | 10.02 | 4.59 | 0.28 | 3.00 | 0.20 | 6.30 |
| | Solution after extraction | 1.00 | 0.005 | 4.55 | 0.30 | 3.00 | 0.19 | 6.30 |
| | Solution of stripping | 1.00 | 9.97 | 0.015 | 0.0006 | 0.012 | 0.008 | 0.01 |

Table 4
Removing iron by high-temperature hydrolysis precipitation

| Batch no. | Samples (g L ⁻¹) | Volume/L | Mg | Ca | Ni | Co | Fe |
|-----------|-------------------------------|----------|------|------|------|------|------|
| No. 1 | Solution before precipitation | 1.00 | 6.12 | 0.19 | 4.74 | 0.32 | 2.96 |
| | Solution after precipitation | 1.25 | 5.10 | 0.16 | 3.78 | 0.26 | 0.07 |
| No. 2 | Solution before precipitation | 1.00 | 6.30 | 0.19 | 4.55 | 0.30 | 3.00 |
| | Solution after precipitation | 1.18 | 5.42 | 0.17 | 3.91 | 0.26 | 0.02 |

Table 5
Removing iron by ferro-vanadium salt hydrolysis method at atmospheric pressure

| | Volume/L | Mg | Ca | Ni | Co | Fe |
|--|----------|------|------|------|------|-------|
| Solution before precipitation (g L ⁻¹) | 1.00 | 6.12 | 0.19 | 4.74 | 0.32 | 2.96 |
| Solution after precipitation (g L ⁻¹) | 1.31 | 4.65 | 0.56 | 2.71 | 0.21 | 0.004 |

price of Cyanex301 was high, and it was difficult to strip nickel and cobalt from the loaded organic phase. Chemical precipitation of calcium and magnesium in advance by NaF salts to obtain a relatively pure solution for the latter recovery of nickel and cobalt was another method (Lin, 2003), but because the poisonous fluorides were introduced in the processing, the difficulties still existed in practical industrial application. That technique has been used to refine solutions containing high concentration of nickel or cobalt. Because the solution presented in current paper have manganese and calcium concentrations almost the same as those of nickel and cobalt, the economic cost for that technique would be high.

In present paper, another promising method was examined. By using Na₂S and carefully controlling the precipitation conditions, nickel and cobalt could be selectively precipitated and recovered from above solutions after removing iron, while calcium and manganese were not precipitated.

The authors had examined the precipitation behaviors of Ni²⁺, Fe³⁺ and Mg²⁺ as a function of pH by the addition NaOH and Na₂S. The 50 g/L of NaOH or 25 g/L of Na₂S solution was respectively used for deposition experi-

ments, adding gradually into the solutions containing only Ni, Fe or Mg. The experiments were carried at temperature 60 °C, and the results are as shown in Figs. 1–3.

The results presented in Figs. 1 and 2 show that Na₂S can deposit nickel and cobalt efficiently, but it could also

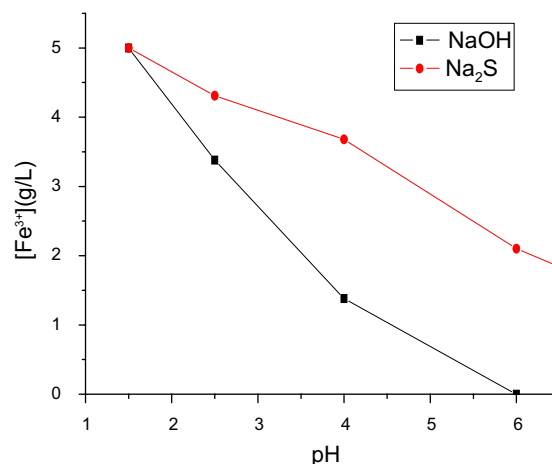


Fig. 2. Deposition of Fe³⁺ by NaOH and Na₂S.

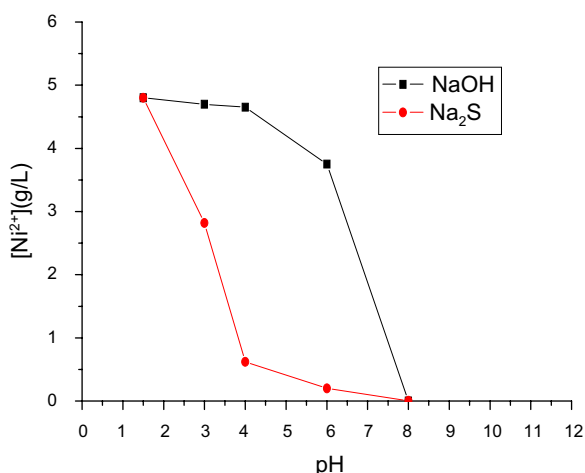


Fig. 1. Deposition of Ni²⁺ by NaOH and Na₂S.

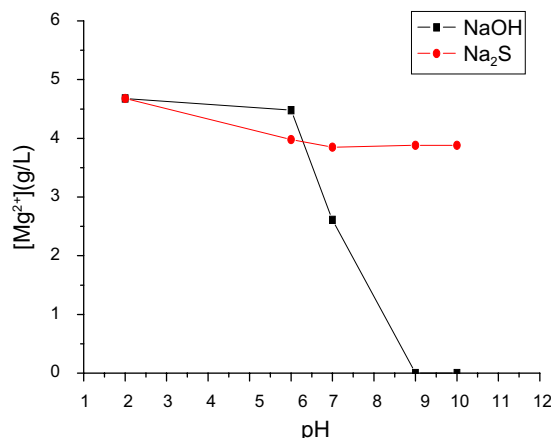


Fig. 3. Deposition of Mg²⁺ by NaOH and Na₂S.

Table 6
Selective precipitation of nickel and cobalt by Na₂S

| Batch no. | Samples (g L ⁻¹) | Volume/L | Ca | Mg | Co | Ni |
|-----------|---|----------|------|------|--------|--------|
| No. 1 | Solution before depositing Ni, Co | 1.00 | 0.56 | 4.65 | 0.21 | 2.71 |
| | Solution after depositing Ni, Co | 2.28 | 0.23 | 2.03 | 0.0005 | 0.001 |
| No. 2 | Solution before depositing Ni, Co | 1.00 | 0.16 | 5.10 | 0.26 | 3.78 |
| | Solution after depositing Ni, Co ^a | 1.82 | 0.09 | 2.86 | 0.0005 | 0.0008 |

^a Note: pH 8, 25 g/L Na₂S.

deposit Fe at the same time. Therefore by using the Na₂S precipitation method, it was needed to remove Fe in advance from the solution before depositing Ni and Co. As can be seen in Fig. 3, when Ni was deposited, Mg remained in the solution, which means Ni and Co can be effectively separated with Mg if controlled appropriate precipitation conditions.

The experimental results of selective precipitation of nickel and cobalt by Na₂S from the experimental solution are shown in Table 6.

Calculated based on the data listed in Table 6, it is indicated that the percent nickel and cobalt precipitation are all more than 99%, while no manganese and calcium are precipitated. Ni and Co can indeed be effectively recovered by Na₂S, and separated from Mg and Ca. It is important to carefully control the solution pH values in the processing. If the pH was high, Mg and Ca would be co-deposited with Ni and Co, while if pH was too low, the precipitation of Ni and Co was incomplete.

After precipitation of Ni and Co by Na₂S, the deposition concentrates can be dissolved into the H₂SO₄ solution for further separation and recovery of Ni and Co.

4. Conclusions

It is concluded that:

- (1) The developed new technique in present work can effectively recover copper, nickel and cobalt from the acidic pressure leaching solutions of Jinbaoshan low-grade sulfide flotation concentrates.
- (2) That technique is also suitable for the processing of such solutions with low content of Cu, Ni and/or Co, but correspondingly almost same concentration of co-existed impurity metal ions of Fe, Ca and/or Mg, for the purpose of recovering objective metals (CuNiCo).
- (3) The total percent recovery of Cu can reach 95% or more, and that of Ni and Co are all more than 99%. In the processing, the percent removal of impurities, such as Fe, Mg and Ca, are all also near to 99%.
- (4) It should be addressed that for the suggested technique in present paper, after the SX and separation process of Cu, to further recover Ni and Co in solutions by using the Na₂S precipitation method, the co-existed Fe ion should be removed in advance and the

final solution pH values in the processing should be carefully controlled for fully precipitation of Ni and Co.

- (5) The whole process for recovery of Cu, Ni and Co is effective and compact, and has almost no environmental impact; therefore it is suitable for the future industrial application.

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