PYRITE TAILINGS AS A SECONDARY RESOURCE FOR BASE METALS RECOVERY

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ABSTRACT

Pyrite tailings from operating industrial facilities, beyond their primary component, frequently contain valuable base metal minerals (e.g., Co, Ni, Cu) that are in constant demand and are critical for 'green energy' technologies. Despite the comparatively low concentration of Ni and Co in these tailings, their overall reserves are potentially significant.

The objective of this study was to investigate the recovery of nickel, cobalt, and copper from leach sulfuric solutions derived from nickel-cobalt-bearing pyrite concentrate using solvent extraction.

It was established that selective metal recovery necessitates the preliminary precipitation of iron as jarosite at a controlled pH. From the resultant iron-free solution, selective copper extraction was achieved using Acorga 5640 in kerosine at a pH range of 2.8–3.0. This was followed by the co-extraction of Ni and Co using Cyanex 272 combined with TBP at a pH of 5.0.

To obtain high-purity cobalt and nickel sulfate, the optimal solvent extraction scheme was determined to be 2E+1Sc+1S (2 Extraction stages + 1 scrabbing stage + 1 Stripping stage).

As a result of this research, pure iron hydroxide, along with marketable nickel, cobalt, and copper sulfates, were successfully recovered. Further concentration of the final cobalt sulfate solution requires a subsequent sorption process.

Keywords: pyrite tailings, nickel, cobalt, copper, iron, solvent extraction, chemical precipitation.

INTRODUCTION

Pyrite tailings from operating mining enterprises, in addition to the dominant sulphide component, often contain minerals of several technologically important non-ferrous metals (Co, Ni, Cu, etc.). These metals remain in consistently high demand, particularly due to their widespread use in «green energy» technologies. They commonly occur in the form of pentlandite ($(Fe,Ni)_9S_8$), chalcopyrite ($CuFeS_2$), and other sulphides [1–2].

The pyrite concentrate generated by the Sokolov–Sarbai Mining and Processing Industrial Association (SSGPO JSC) represents a valuable anthropogenic resource. Although the concentrations of Ni and Co in these tailings are relatively low, their total

reserves are potentially substantial. Processing such materials not only reduces the volume of accumulated waste and releases land areas, but also mitigates environmental risks by preventing soil and water contamination with heavy metals (including acid mine drainage, AMD) [3]. In addition, it enables the recovery of valuable products such as iron, nickel, cobalt, copper, and sulfur (as sulfur dioxide for sulphuric acid production).

Given the sharp increase in global demand for nickel and cobalt, pyrite tailings can be regarded as an important secondary resource for their recovery. The methods used to extract Ni and Co from pyrite tailings can generally be grouped into several categories: pyrometallurgical, hydrometallurgical, and combined approaches. Pyrometallurgical processes (e.g., roasting followed by smelting) can be applied; however, they are less commonly used due to the low concentrations of nickel and cobalt and the high contents of sulfur and iron, which result in significant energy consumption and the generation of large volumes of off-gases.

Nevertheless, roasting may be employed as a preparatory step for subsequent hydrometallurgical treatment-for example, to oxidize sulphide minerals and enhance their solubility. Hydrometallurgical processing of pyrite tailings is considered the most promising approach due to its operational flexibility and lower environmental impact. Leaching of the tailings represents the key stage of hydrometallurgical processing.

To transfer valuable components into solution, various leaching approaches are employed, including acid leaching with sulfuric acid (H_2SO_4) [4], hydrochloric acid (HCl) [5], or nitric acid (HNO_3) [6], as well as mixed-reagent leaching [7], pressure (autoclave) leaching with different oxidizing agents [8], and bioleaching of nickel and cobalt using microorganisms (Acidithiobacillus ferrooxidans) for sulphide oxidation.

However, direct leaching is not always effective, and for the efficient recovery of valuable metals from Ni–Co-bearing pyritic materials, a combined processing route is considered optimal. This approach typically involves roasting of sulphide materials followed by leaching [9]. Depending on the tailings' mineralogical composition, sulfidizing roasting (with limited oxygen supply), chloridizing roasting, or oxidizing roasting (with air introduction) may be used. Under oxygen-limited conditions, roasting not only promotes the formation of soluble metal sulphides but also generates sulfur-containing gases produced during sulfur oxidation, which represent a valuable feedstock for sulfuric acid production.

For the efficient separation of valuable metals—copper, nickel, and cobalt—from pregnant leach solutions (PLS) that contain significant amounts of impurities, primarily iron formed during the leaching of roasted pyrite concentrate, various hydrometallurgical methods are employed. The most widely used approaches include mixed sulphide precipitation (MSP), mixed hydroxide precipitation (MHP), and direct solvent extraction (DSX) [10, 11].

Among these, solvent extraction (SX) stands out as one of the most effective and technologically advanced methods for processing such solutions due to its cost-efficiency, operational simplicity, and environmental acceptability [12]. However, successful SX performance requires thorough pre-removal of iron, particularly when its concentration exceeds 2–3 g/L, as elevated Fe levels adversely affect phase separation, extractant efficiency, and subsequent refining stages.

The extractants used for the selective recovery and concentration of copper from multicomponent pregnant leach solutions are oxime-based reagents, including both aldoximes and ketoximes. For the separation of nickel and cobalt, the most widely applied extractants are organophosphorus reagents such as D2EHPA (di-2-ethylhexylphosphoric acid), PC-88A (mono-2-ethylhexyl ester of 2-ethylhexylphosphonic acid), and Cyanex 272 (2,4,4-trimethylpentylphosphinic acid) [13, 14]. In addition, carboxylic acids-including Versatic 10, a mixture of isomeric synthetic C10-carboxylic acids whose principal component is 2-methyl-2-ethylheptanoic (neodecanoic) acid-are also employed [10], as well as high-molecular-weight amines. Among the extractant classes mentioned, organophosphorus acids and chelating oxime compounds are the most widely used and commercially available due to their high selectivity, resistance to contamination, and suitability for large-scale industrial application.

In recent years, considerable research effort has been directed toward the use of deep eutectic solvents (DESs) for the selective separation of cobalt and nickel. However, despite substantial scientific progress, these systems have not yet achieved broad industrial implementation due to limitations in stability, regeneration capacity, and process integration [15].

The processing approach proposed for pregnant leach solutions derived from pyrite tailings enables the simultaneous recovery of iron and non-ferrous metals-copper, nickel, and cobalt-which are critically important for «green energy» technologies. This contributes to the development of a closed-loop raw material processing scheme and reduces the overall environmental impact.

The aim of the present study was to investigate the extraction of nickel, cobalt, and iron from pregnant leach solutions obtained by leaching roasted pyrite tailings. Particular attention was given to the preliminary removal of iron, as well as to the selective solvent-extraction separation of non-ferrous metals at subsequent stages.

MATERIALS AND METHODS

The solvent extraction (SX) experiments were carried out using an iron-rich pregnant leach solution obtained by leaching the roasted product of pyrite tailings. The chemical composition of the calcine is presented in Table 1.

Table 1 Chemical composition of sinder obtained at 650 °C

Contents, %												
$Fe_{общ}$	Cu	Co	Ni	$S_{общ}$	Pb	Zn	SiO_2	Al_2O_3	CaO	MgO	TiO_2	As
15,78	0,20	0,018	0,20	3,7	0,0010	0,034	34,63	12,97	11,32	3,55	0,64	0,005

As a result of sulfuric acid leaching of the pyrite concentrate calcine (≈90–100 °C, 60 min, S:L = 1:2), a PLS was obtained with the following composition (g/L): Fe 131.05; Ni 1.61; Co 1.49; Cu 1.

Iron removal from the pregnant solutions was carried out by precipitation in an acidic sulfate medium through the addition of Na^+ ions (Na_2SO_4) to form jarosite. The

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precipitation was performed at 90 °C and pH ≈ 1.5 for 3 h under vigorous stirring. Under these conditions, Fe^{3+} converted into a stable jarosite phase, ensuring an iron removal efficiency of up to 99 %, while the accompanying non-ferrous metals remained predominantly in solution.

The resulting precipitate was filtered, washed with slightly acidic water, and analyzed by X-ray diffraction (XRD) to confirm the formation of the jarosite phase.

In the solvent extraction (SX) stage, Acorga 5640 was used for copper extraction, while Cyanex 272 with TBP was employed for the selective separation of cobalt and nickel. Escaid 110 was used as the diluent for all extractant systems.

To prepare the stripping solutions-analogues of spent electrolytes-sulfuric acid and sulfate salts of nickel and cobalt were used. All reagents for the aqueous phases were of analytical grade.

To determine the metal content in the samples, a Shimadzu AA-7000 atomic absorption spectrometer from Japan was used. The analysis followed standard atomic absorption spectroscopy methods using an acetylene/air flame. The instrument was calibrated with standard solutions of the corresponding elements.

RESULTS

Iron precipitation

For the selective recovery of copper, nickel, and cobalt, it is first necessary to selectively remove iron and obtain a well-filterable precipitate with minimal losses of non-ferrous metals.

During iron precipitation in the form of jarosite, copper, nickel, and cobalt are practically not involved in co-precipitation and remain in the pregnant solution predominantly as soluble sulfate complexes, which ensures their retention for the subsequent stages of selective extraction.

The mechanism governing selective precipitation is associated with the fact that the formation of the jarosite structure, $Na_3(SO_4)_2(OH)_6$, occurs exclusively through the hydrolysis and crystallization of trivalent iron in the presence of sulfate ions and Na^+ cations. In contrast, Cu^{2+}, Ni^{2+}, and Co^{2+} ions do not undergo hydrolysis under conditions of pH ~1.5. In such an acidic medium, copper remains in solution predominantly as the aquo-sulfate species $[Cu(H_2O)_6]^{2+} + SO_4^{2-}$, nickel as $[Ni(H_2O)_6]^{2+} + SO_4^{2-}$ as the counterion, and cobalt as $[Co(H_2O)_6]^{2+} + SO_4^{2-}$. At high sulfate concentrations, stable Me–SO_4 complexes may form, which do not produce stable hydroxysulfate phases. Their hydrolysis constants are significantly higher, and the equilibrium is shifted toward the formation of soluble sulfate complexes. This prevents the incorporation of Cu, Ni, and Co into the jarosite lattice or their adsorption onto particle surfaces, ensuring that they remain in solution in stable dissolved forms. Such thermodynamic and kinetic selectivity provides nearly complete retention of Ni, Co, and Cu in the pregnant leach solution.

The calculated yield of the solid phase (Na-jarosite) is approximately 0.77 mol/L, which corresponds to about 370–380 g/L of filter cake. The iron precipitate formed at 95 °C in the presence of seed material is characterized as dense and highly filterable.

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A critical factor in the process is the control of non-ferrous metal losses. When maintaining the pH at no higher than 1.5–1.8 and ensuring effective stirring and washing, the contents of copper, nickel, and cobalt in the jarosite cake (total losses) do not exceed 0.5–1.0%. The solution obtained after iron precipitation-with the following composition (g/L): 1.502 Ni, 1.401 Co, and 1.0 Cu-was subsequently directed to the solvent-extraction separation of the metals.

Solvent Extraction of Cobalt and Nickel

The selective recovery of copper, nickel, and cobalt from the solutions remaining after iron precipitation is one of the most critical stages in the integrated pyro-hydrometallurgical processing of pyrite concentrates. Solvent extraction is currently widely applied for the recovery of copper, cobalt, and many other non-ferrous metals. Acorga 5640 is an efficient reagent for the selective extraction of copper, while Cyanex 272 is commonly used for cobalt extraction [20].

During the solvent extraction of copper, the following reaction takes place:

$$HR(l) + CuSO_4\,(aq) \rightarrow CuR_2 + H_2SO_4(aq)$$

where HR refers to the oxime-based extractant Acorga 5640.

H_2R_2 denotes the phosphinic acid–based extractant Cyanex 272.

Copper extraction proceeds via the formation of a chelate complex with Acorga 5640.

Solvent Extraction Experiments

Copper extraction was carried out at room temperature within the pH range of 1 to 4 using the Acorga 5640 extractant. The results of copper separation from the leach solutions are presented in Table 2 and illustrated in Figure 1.

Table 2 Results of Copper Extraction from the Leach Solutions

pH	Cu	Co	Ni	D_{Cu}	D_{Co}	D_{ni}	$\beta_{Cu/Co}$	$\beta_{Cu/Ni}$
	E, %							
1	35,56			0,55				
1,5	54,68			1,21				
2	69,77			2,31				
2,5	91,42	0,05		10,66	0,001		21299,37	
3	93,41	0,1		14,17	0,001		14160,33	
3,5	93,74	0,5	0,1	14,97	0,005	0,0008	2979,914	19545,64
4	93,88	2,4	0,2	15,34	0,025	0,0015	623,8214	10000,57

As demonstrated by the copper extraction experiments, the optimal pH value for its selective recovery is 3.0. At pH 3 and above, the degree of co-extraction of cobalt and nickel increases significantly (Figure 1). At pH 3.0 (the optimal value), the separation factors for the metals are as follows: $\beta_{Cu/Co}$=14160.33. Nickel is practically not extracted under these conditions. The separation factor $\beta_{Cu/Ni}$ at pH 3 is 19545.64, which confirms the high selectivity of the Acorga M5640 extractant for removing copper from the pregnant solution.

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Copper stripping was performed using sulfuric acid at a concentration of 100–200 g/L, with an O/A ratio of 1:1 at room temperature. The mixing and phase-settling time was 7–10 minutes. Metal concentrations were determined by titrimetric and atomic absorption analyses. The stripping results are presented in Figure 2.

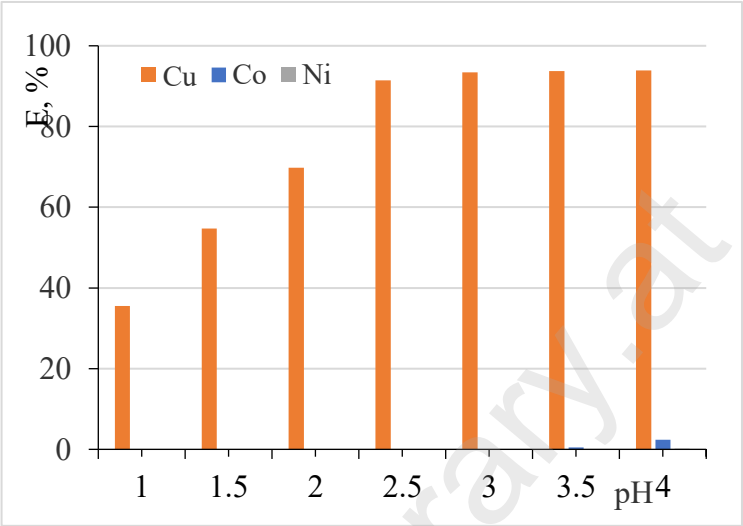


Figure 1 Dependence of metal extraction on solution pH, E(Me), % vs. pH

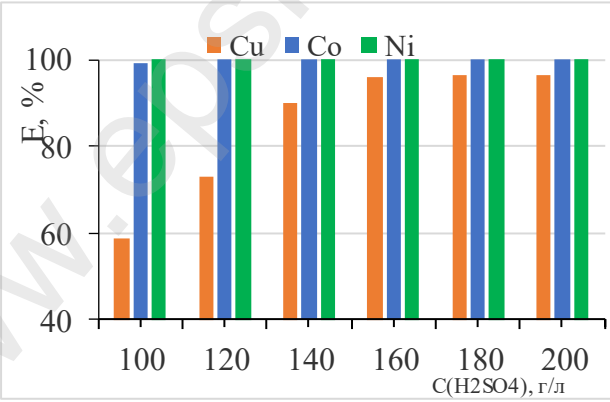


Figure 2 Dependence of metal stripping efficiency on acid concentration, E(Me), % vs. acid concentration

The results of metal stripping with sulfuric acid (at concentrations between 100 and 200 g/L) show that a high recovery of nickel (100%) and cobalt (99% and above) is achieved already at an acid concentration of 100 g/L. The high stripping efficiency for nickel and cobalt is attributed to the fact that they do not form strong complexes with the oxime extractant Acorga 5640 used in the process.

In contrast to these metals, copper stripping requires a substantially higher concentration of sulfuric acid-no less than 160 g/L. Under these conditions, the recovery reaches 96.6%. Further increasing the acid concentration is not justified, as it does not lead to any significant improvement in the stripping performance.

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For the refining and concentration of copper, a solvent extraction circuit configuration of 3E+1S (three extraction and one stripping stages) was selected. McCabe-Thiele analysis indicated that 3 extraction stages are sufficient to achieve a recovery of >90% at an organic-to-aqueous (O:A) phase ratio of 1:2, ensuring adequate loading of the organic phase (16 vol.% Acorga M5640). A single stripping stage utilizing a spent electrolyte containing 35 g/L of copper and 170 g/L H_2SO_4 was found sufficient to produce an enriched electrolyte containing 46.12 g/L copper, suitable for subsequent electrowinning.

Separation of Nickel and Cobalt

The extraction of divalent metal cations (M^{2+}) using the commercial reagent Cyanex 272, as well as related phosphoric, phosphonic, and phosphinic acids, typically results in the formation of complexes with a 4:1 ligand-to-metal stoichiometry, generally represented by the formula $[M(L)_2(LH)_2]$.

$$2H_2R_2(l) + CoSO_4\,(aq) \rightarrow CoR_2 \cdot H_2R_2\,(l) + H_2SO_4(aq) \qquad (2)$$

Cobalt stripping proceeds according to the following reaction:

$$CoR_2 \cdot H_2R_2\,(l) + H_2SO_4(aq) \;\rightarrow\; 2H_2R_2(l) + CoSO_4\,(aq) \qquad (3)$$

The formation of cobalt complexes with the Cyanex 272 extractant exhibits markedly higher selectivity compared with nickel, as Co(II) ions form a tetrahedral polymeric complex of the general formula CoR_2, whereas nickel ions predominantly form an octahedral hydrated complex. This behavior arises from differences in their electronic structures: Co^{2+} has the configuration $1s^22s^22p^63s^23p^63d^7$, while Ni^{2+} has $1s^22s^22p^63s^23p^63d^84s^2$.

The Co^{2+} cation, containing seven electrons in its 3d shell, exhibits a stronger tendency toward complex formation. However, the difference in the number of d-electrons determines the preferred coordination geometry and the stability of the resulting complexes. This structural distinction governs the differences in the distribution coefficients and thus accounts for the high selectivity of Cyanex 272 toward cobalt.

The experiments on cobalt–nickel separation were carried out using a solution containing 1.50 g/L of nickel and 1.399 g/L of cobalt. Extraction was performed at pH 3–6 and a temperature of 55 °C using a 10% solution of the extractant Cyanex 272 in kerosene with the addition of 5% tributyl phosphate (TBP) (Figure 3).

Stripping Results

The results of metal stripping (Figure 4) showed that a sulfuric acid concentration of 60 g/L is sufficient for the complete transfer of nickel into the aqueous phase, while cobalt requires an acid concentration of 100 g/L. These findings confirm that nickel forms a weaker complex with the extractant compared with cobalt.

Analyzing the results of extraction and re-extraction experiments, the following conclusions can be drawn: selective cobalt extraction is possible using 10% Cyanex 272 with TBF additive at pH 5 and a temperature of 50 °C. The overall cobalt extraction rate is 94.23%. Nickel is completely transferred to the raffinate.

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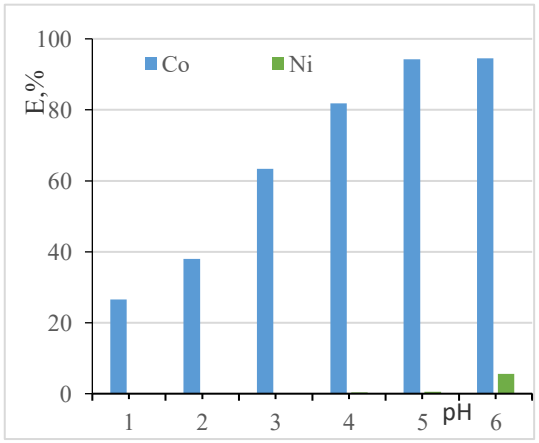


Figure 3 Dependence of metal extraction on solution pH

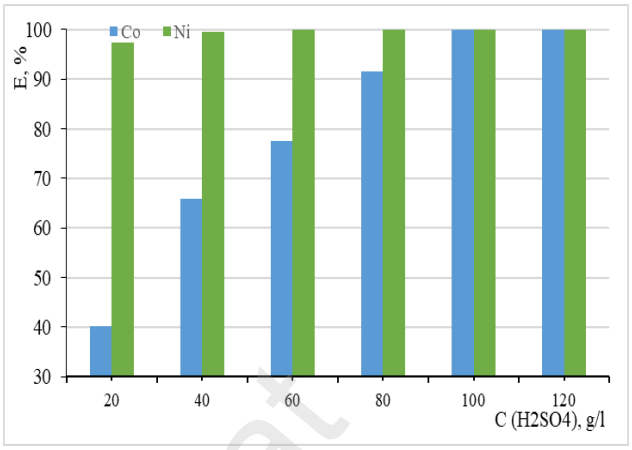


Figure 4 Dependence of metal re-extraction on sulfuric acid concentration

Calculation of isotherms (McCabe-Thiele method)

The most convenient graphical tool for analyzing and calculating the technological characteristics of the liquid extraction process of a system are the extraction and re-extraction isotherms (McCabe-Thiele diagrams), obtained experimentally. The characteristics of isotherms change when the system parameters change and directly depend on the conditions of their construction.

Based on the results of our research, SYENQO performed calculations of extraction schemes. The solutions after removal of iron and copper contained: nickel from 1.5 g/l (g/dm³) and cobalt from 1.4 g/l (g/dm³). Based on these calculations, an extraction isotherm was constructed (Figure 5) and the process of selective separation of nickel and cobalt was modeled according to the 2E+1Sc-1S scheme (two extraction stages + one scrabbing stage and one stripping stage) for PLS.

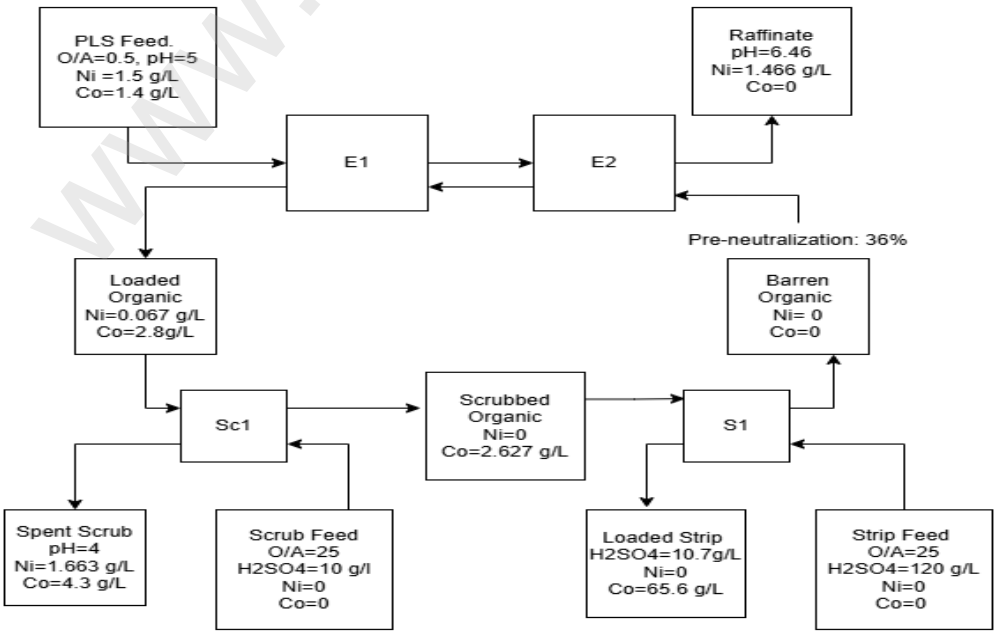


Figure 5 Simulation of the liquid extraction process for selective separation of cobalt and nickel from PLS by liquid extraction

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Scrabbing Stage (1Sc)

The presence of nickel, resulting from its accumulation during the circulation of the organic phase, is undesirable for producing cobalt of high purity or cobalt sulfate. To remove nickel from the cycle, a scrabbing stage (Sc 1) is introduced after the two extraction stages. The organic phase is washed with a sulfuric acid solution (10.00 g/L) at a high phase ratio of O:A = 25:1 (aqueous to organic).

According to the calculations, after this scrabbing step the cobalt concentration in the organic phase decreases by 0.093 g/dm³ (corresponding to 11.54% Co losses), while the nickel concentration decreases from 0.056 g/dm³ to 0 g/dm³ (100% Ni removal).

Stripping Stage (1S)

The cobalt-loaded organic phase is subsequently subjected to stripping using sulfuric acid with a concentration of 30 g/dm³. After a single stripping stage (conditions: pH 1.194; T = 50 °C; O:A = 20:1), a nickel-free cobalt sulfate electrolyte with a concentration of 14.257 g/dm³ is obtained.

Based on the process flowsheet comprising two extraction stages, one scrabbing stage, and one stripping stage (2E + 1W + 1S), a purified solution containing 1.61 g/L nickel sulfate was produced. This solution is suitable for further processing to obtain nickel sulfate by precipitation at pH 9 followed by dissolution in sulfuric acid, or alternatively through sorption concentration.

Implementing the solvent extraction process following the 2E + 1Sc + 1S scheme under laboratory conditions enabled selective separation of cobalt and nickel, yielding a sulfate stripping solution containing 64.55 g/L cobalt and 1.4 g/L nickel. The resulting sulfate solutions (nickel raffinate and cobalt strip liquor) are directed to subsequent processing, including the production of high-purity precipitates in the form of sulfates or metallic cobalt and nickel powders by electrolysis.

CONCLUSION

Pyrite tailings generated by mining and metallurgical operations represent a potentially valuable secondary resource rich in metals critical for the development of “green energy” technologies. To integrate this anthropogenic material into the metallurgical cycle, an integrated processing approach was adopted, comprising oxidative roasting under controlled oxygen-deficient conditions, followed by acid leaching and downstream hydrometallurgical treatment.

The processing of the pregnant leach solution (PLS)-containing 131.05 g/L Fe, 1.61 g/L Ni, 1.49 g/L Co, and 1.0 g/L Cu-was carried out using a sequential metal-separation strategy. Initially, deep purification was achieved by precipitating iron as Na-jarosite. This step was followed by the selective extraction of copper using 16% Acorga M5640 in a 3E + 1S configuration, with the organic phase loaded to 46 g/L of Cu in the electrolyte. Subsequently, cobalt and nickel were recovered through solvent extraction using an organic phase consisting of 10% Cyanex 272 with 5% TBP as a phase modifier. The extraction circuit operated under a 2E + 1Scr + 1S arrangement, enabling the production of cobalt sulfate containing 65.6 g/L Co.

This processing route ensures the production of iron powders and the recovery of high-purity non-ferrous metals (Cu, Ni, Co), which are essential precursors for the

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manufacture of batteries, electrochemical systems, and other key components of sustainable energy technologies.

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