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Reductive Leaching of Cobalt Using Sulfuric Acid Plant Effluents: A Pathway to Circularity in the Minerals and Metals Value Chain

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Abstract

This study investigates the potential use of effluents from sulfuric acid production as a reducing agent for cobalt leaching. The aim is to reduce production costs by substituting sodium metabisulfite (SMBS) with effluents rich in sodium bisulfite. Leaching experiments were conducted to determine the most effective reducing-agent dose, and its addition point. The findings show that the reducing agent dose has a significant impact on the cobalt extraction, with optimal yields approaching 90% for both SMBS and effluents at 5 kg of SO_2 per ton of ore, with the reducing agent added at t = 1 h. Changes the reducing agent addition point from t = 1 h to t = 0 h had a beneficial effect on copper extraction by avoiding the precipitation of Chevreul's salt, but they had little effect on cobalt recovery. The results confirm that effluents can be used as a cost-effective alternative to SMBS, as long as they are processed to raise the concentration of bisulfite, even though using raw effluents results in a slightly higher acid consumption.

Keywords

Reducing Agent, Cobalt, Effluents, Sodium Metabisulfite

1. Introduction

The growing adoption of electric vehicles and green energy technologies has driven a substantial and sustained increase in global cobalt demand, with EVs now

representing the largest and fastest growing end-use segment [1]-[3]. This demand is reinforced by cobalt's essential role in high-energy-density cathode chemistries (NCM/NCA) and by its exceptional physicochemical and mechanical properties ferromagnetism, ductility, and high-temperature and corrosion resistance which underpin its wide-ranging use in modern engineering, from superalloys to electronics and aerospace [3] [4]. Owing to its low crustal abundance and its characteristic association with sulfide mineralization, cobalt is only rarely mined as a primary commodity and is instead predominantly recovered as a by-product of copper and nickel production [5] [6]. A dominant share of global cobalt output originates from the African Copperbelt, and more specifically from the Democratic Republic of Congo (DRC), which alone supplies the majority of the world's primary cobalt [3] [6]. Within this district, cobalt is chiefly hosted in heterogenite (CoOOH), a supergene oxide phase in which cobalt occurs predominantly in its trivalent (Co^{3+}) state [5] [7]-[9].

The presence of Co^{3+} in heterogenite strongly limits its dissolution during conventional sulfuric acid leaching: Co(III) is poorly soluble under acidic conditions, whereas its reduction to Co(II) yields the readily soluble Co^{2+} species, making the establishment of an appropriate reducing environment a key requirement for efficient cobalt extraction [9]-[12]. The effectiveness of reductive leaching is therefore governed not only by the intrinsic reducibility of the cobalt-bearing phases but also by the nature, dosage, and addition strategy of the reducing agent. Several reductants have accordingly been investigated for oxidized Cu-Co ores, including ferrous ions, sulfur dioxide (SO_2), sodium metabisulfite (SMBS), and metallic copper, each entailing distinct trade-offs in reduction efficiency, reagent cost, and environmental burden: SO_2 requires stringent gas-handling infrastructure, ferrous sulfate introduces substantial iron loads into the leachate, and sulfite-based reagents add sodium ions that increase the downstream salt burden [4] [11]-[13]. Among these, SMBS remains the reductant of choice at both laboratory and industrial scale, owing to its favorable cost-efficiency ratio: it reacts readily with sulfuric acid to generate SO_2 in situ, which acts as the active reducing species and promotes the conversion of Co(III) to soluble Co(II) in Copperbelt oxide ores [4] [9] [14]. However, reductant addition can also induce secondary reactions involving previously dissolved metals, particularly copper, potentially leading to the precipitation of copper sulfite phases and thereby affecting overall metal recovery. Consequently, optimizing both reducing-agent dosage and addition point is essential to maximize cobalt dissolution while limiting reductant consumption and undesirable side reactions [11]. These considerations are particularly relevant for industrial leaching circuits, where excessive SO_2 consumption and incomplete utilization of the reducing agent may increase reagent and acid consumption and raise operational and environmental concerns [9].

At Tenke Fungurume Mining (TFM), approximately 185 km northwest of Lubumbashi, SMBS is routinely used to promote cobalt reduction during leaching [15]. However, the associated sulfuric acid plant generates effluents containing

sodium bisulfite, formed through the neutralization of SO_2 with sodium hydroxide (Equation (1)). Since bisulfite constitutes the active reducing species generated upon SMBS dissolution, these effluents represent a potential alternative reducing, and currently underexploited, source reducing power for cobalt leaching circuits [11]. The corresponding reactions are summarized in Equations (2)-(5):

$$NaOH + SO_2 \rightarrow NaHSO_3 \quad (1)$$

$$Na_2S_2O_5 + H_2O \rightarrow 2NaHSO_3 \quad (2)$$

$$Na_2S_2O_5 + H_2SO_4 \rightarrow Na_2SO_4 + 2SO_2 + H_2O \quad (3)$$

$$2NaHSO_3 + H_2SO_4 \rightarrow Na_2SO_4 + 2SO_2 + 2H_2O \quad (4)$$

$$2CoO(OH) + H_2SO_4 + SO_2 \rightarrow 2CoSO_4 + 2H_2O \quad (5)$$

This study evaluates sulfuric acid plant effluents as an alternative reducing agent for cobalt leaching, with the objective of reducing SMBS consumption and operating costs. Ten leaching tests, in addition to a blank test without reducing agent, were conducted to optimize the reducing-agent dosage, and addition point. Since cobalt is recovered as a by-product of copper leaching at TFM, pH, particle size, pulp density, and sulfuric acid concentration were maintained at the operating conditions used for copper leaching.

2. Materials and Methods

2.1. Materials

Ore and effluent samples collected consisting primarily of heterogenite and malachite, sampled from the ROM pad, while the effluents and HG raffinate were collected from the sulfuric acid plant and SX plant respectively. Both materials were prepared and characterized prior to leaching experiments conducted in the TFM laboratory.

2.2. Experimental Setup

Leaching experiments were performed in a 1-L beaker immersed in a Precisdig thermostatic water bath. A Faithful mechanical stirrer provided constant agitation and ensured adequate suspension and mixing of the pulp. The pH was monitored throughout each experiment using a Mettler Toledo pH meter. A schematic representation of the experimental setup is presented in **Figure 1**.

2.3. Experimental Procedure

A grindability test was first performed to determine the grinding time required to obtain a particle size of 75 µm (**Table 1**). Eleven subsequent leaching tests were conducted using the conventional univariate approach, including five using sodium metabisulfite (SMBS), five using acid plant effluents, and one blank test without reducing agent. For each reducing agent, four tests were performed at different dosages and one varying the reducing addition point (**Table 2**).

For each experiment, 201.00 g of ore was mixed with HG raffinate to obtain a pulp density of 30%. The slurry was agitated at 350 rpm and maintained at 41˚C,

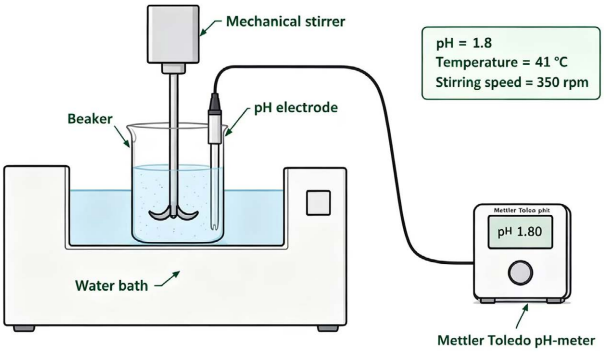


Figure 1. Experimental montage.

Table 1. Grindability test conditions.

Grinding (min)	Feed (g)
4	600
6	600
8	600
10	600

Table 2. Experimental conditions.

N°	SMBS				Effluents			
	SO_2 dosage (kg/t)	Volume (ml)	Time (h)	Addition point	SO_2 dosage (kg/t)	Volume (ml)	Time (h)	Addition point
1	4	12	6	t = 1 h	4	31	6	t = 1 h
2	5	15	6	t = 1 h	5	39	6	t = 1 h
3	6	18	6	t = 1 h	6	46	6	t = 1 h
4	7	21	6	t = 1 h	7	54	6	t = 1 h
5	5	15	6	t = 0 h	5	39	6	t = 0 h

with a sulfuric acid concentration of 10 N and an initial pH of 1.8. The pH was continuously monitored and adjusted, when necessary, by adding sulfuric acid. In the dosage-variation tests, the reducing agent was introduced 1h after the onset of leaching. All leaching experiments were carried out for a fixed duration of 6h. At the end of each experiment, the slurry was filtered to separate the leachate from the solid residue. The feed and residues were dried and analyzed by X-ray fluorescence (XRF), while the leachates were analyzed by inductively coupled plasma optical emission spectrometry (ICP-OES). Below, t = 0 h is the start of the reaction and t = 1 h is one hour into the reaction. To assess leaching performances, extraction recovery was calculated from the feed and tailings grades as shown in Equation (6), where F and G are respectively the feed and tail masses, g and f, feed and tail grades.

$$\text{Recovery (\%)} = \frac{Ff - Tt}{Ff} \times 100 \quad (6)$$

The 10 N sulfuric acid solution was prepared by diluting concentrated solution (Equation (7)), with the acid mass derived from volume using the normality formula (Equation (8)). Bisulfite/SMBS dosage for a target SO_2 quantity was calculated from the stoichiometry of Equations (2) and (3).

$$N_1V_1 = N_2V_2 \tag{7}$$

where 1 and 2 indicate the concentrated and diluted acid solution respectively, while N and V represent the normality (N) and the volume (L) respectively.

$$N = \frac{m \times x}{V \times Mm} \tag{8}$$

where m the mass (g), x the equivalents per mole (eq/mol), and Mm the molar mass (g/mol).

3. Results and Discussion

The chemical compositions of the ore and sulfuric acid plant effluents are presented in Table 3 and Table 4, respectively. The ore contained 1.121% Cu, of which 94% was acid-soluble, and 0.443% Co, with 82% solubility. The concentrations of Fe, Mn, Ca, Mg, and Al were 1.740%, 0.041%, 0.27%, 3.54%, and 2.86%, respectively. The effluents contained 0.003 g/L Mn, 0.209 g/L Ca, 0.810 g/L Mg, 2.120 g/L Al, 9.339 g/L Na, and 13.077 g/L S. Based on their chemical composition, the sodium bisulfite concentration was estimated at approximately 42.5 g/L, while SMBS was prepared at 100 g/L. However, the HG raffinate contains 0.86 g/L Cu, 1.526 g/L Co, and notable I; purities (Fe, Mn, Ca, Mg, Al).

The ore was subsequently ground to the target particle size for the leaching tests. The grindability data (Figure 2) were fitted using the regression equation given in Equation (8), where x represents grinding time and y the percentage passing 75 µm.

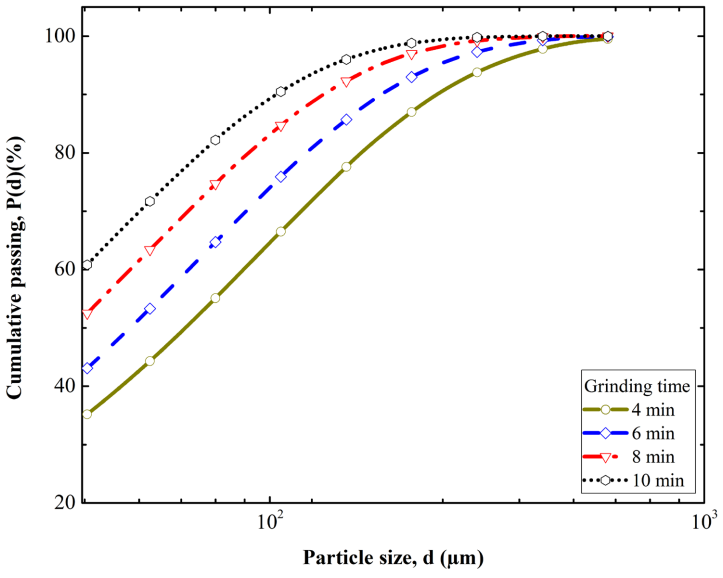


Figure 2. Grindability curve.

Table 3. Chemical composition of the ore sample.

Analysis	T-Cu %	T-Co %	As-Cu %	As-Co %	Fe %	Mn %	Ca %	Mg %	Al %
Sample	1.121	0.443	1.054	0.364	1.740	0.041	0.27	3.54	2.86

Table 4. Effluents and HG raffinate composition.

Analysis	Cu g/L	Co g/L	Fe g/L	Mn g/L	Ca g/L	Mg g/L	Al g/L	Na g/L	S g/L
Effluents	0.000	0.000	0.000	0.003	0.209	0.810	2.120	9.399	13.077
HGraff	0.868	1.526	1.464	0.889	11.50	0.315	0.351	-	-

$$y = 4.565x + 37.22 \quad (9)$$

With $R^2 = 0.996$, Equation (9) estimated 9.37 min to achieve 75 µm.

3.1. Effect of Reducing-Agent Dosage

The blank test was conducted to quantify cobalt dissolution in the absence of a reducing agent. A cobalt extraction of 80.79% was achieved, indicating that most of the cobalt was readily leachable under the prevailing conditions.

At a dosage of 4 kg SO_2/t, cobalt extraction reached 83.67% with SMBS and 86.37% with acid plant effluents (Figure 3). Increasing the dosage to 5 kg SO_2/t increased extraction to 87.85% and 89.46%, respectively. Further increases in dosage did not improve cobalt recovery: at 6 kg SO_2/t, recoveries decreased to 82.14% for SMBS and 85.79% for effluents, while at 7 kg SO_2/t they reached 83.07% and 87.62%, respectively.

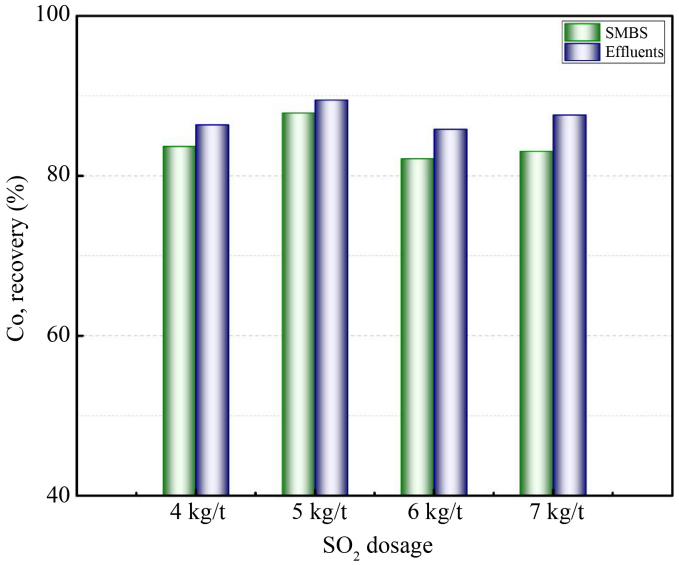


Figure 3. Cobalt extraction when the reducer dose is varied.

These results confirm that cobalt extraction is dosage-dependent, with 5 kg SO_2/t identified as the highest performing dosage for both reducing agents. At this

dosage, the effluents achieved a reductive performance comparable to that of SMBS, while their performance is consistent with the operating conditions currently applied at TFM.

Notably, the difference between the blank test (80.79%) and the maximum extraction obtained with the reducing agents (<10 percentage points) indicates that the contribution of reductive leaching to overall cobalt dissolution was relatively limited.

This may further suggest that the reduction of trivalent to bivalent cobalt is also governed by additional operating parameters that were not varied in this study, as their values were held constant to meet the requirements of copper leaching.

3.2. Effect of Reducing-Agent Addition Point

The effect of reducing-agent addition point was investigated by shifting the addition point from t = 1 h to t = 0 h with 5 kg of SO_2/t. As shown in **Figure 4**, cobalt recoveries were 88.39% with SMBS and 88.10% with acid plant effluents. Thus, advancing the reducing-agent addition from t = 1 h to t = 0 h had no significant effect on cobalt recovery, irrespective of the reducing agent used.

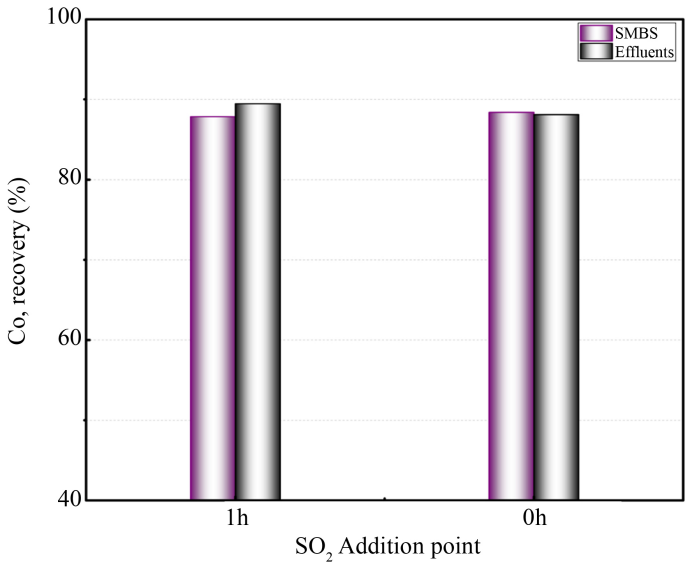


Figure 4. Cobalt extraction as a function of leaching time and reducing-agent addition point.

In contrast, earlier addition significantly improved copper recovery. With SMBS, Cu extraction increased from 89.92% to 92.05%, while for effluents extraction increased from 89.05% to 93.97%. This improvement can be attributed to the effect of the reducing-agent addition point on Chevreul's salt precipitation ($Cu_2SO_3{\cdot}CuSO_3{\cdot}2H_2O$). As reported by [9] and [5], the timing of reducing-agent addition can influence copper recovery by promoting or suppressing the formation of this copper sulfite phase. In the present study, addition at t = 0 h limited Chevreul's salt precipitation, thereby increasing copper recovery (**Figure 5**). A

similar effect was reported by [11] when the addition point was shifted from t = 3 h to t = 0 h.

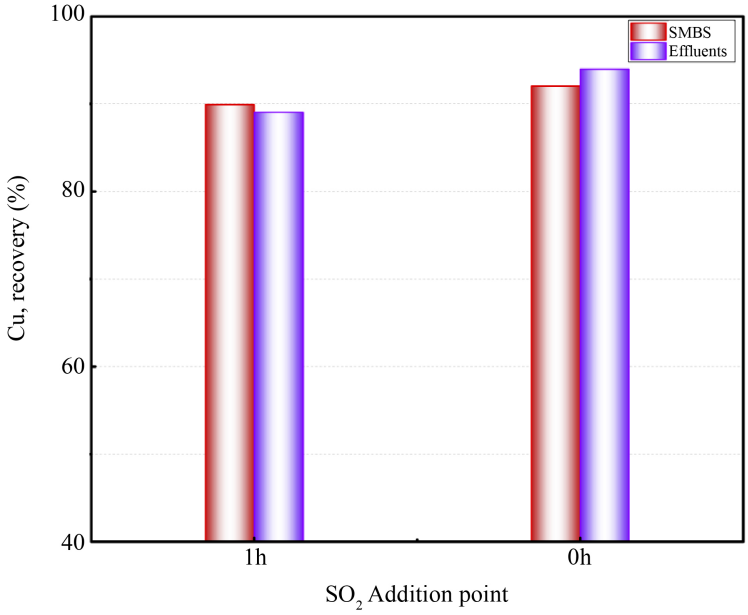


Figure 5. Copper extraction as a function of reducing-agent addition point.

3.3. Effect of Effluents on Impurity Leaching and Acid Consumption

Impurity dissolution and acid consumption at 5 kg of SO_2/t are presented in Figure 6 and Figure 7, respectively. Compared with SMBS, the use of effluents had little effect on Fe and Mn dissolution but substantially decreased Al leaching. Fe, Mn, and Al extractions reached 24.13%, 86.88%, and 26.77%, respectively, with SMBS, compared with 24.45%, 89.46%, and 16.03% with effluents.

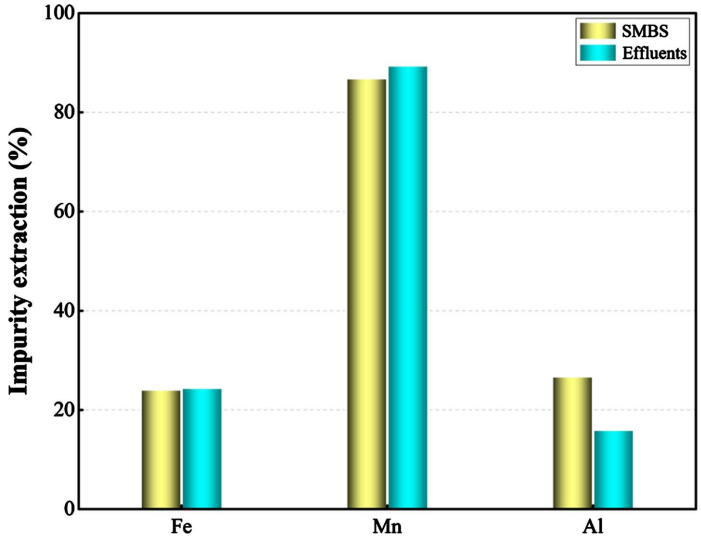


Figure 6. Extraction of impurities during leaching.

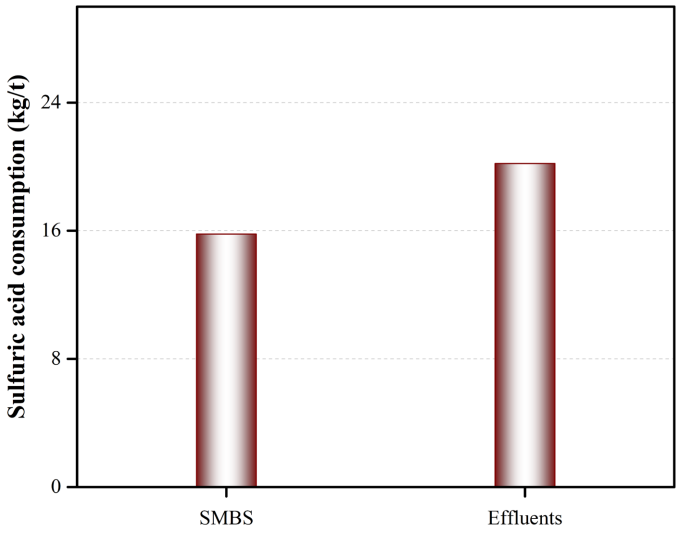


Figure 7. Leaching-agent consumption.

Table 5 shows that effluent use also increased H_2SO_4 consumption by approximately 4.4 kg/t at an SO_2 dosage of 5 kg/t. This additional acid demand is likely associated with the lower bisulfite concentration of the effluents (42.5 g/L) compared with the SMBS solution (100 g/L), requires a larger volume of reducing solution to achieve the target SO_2 dosage and, consequently, the resizing of some existing equipment. However, the cost analysis presented in **Table 6**, based on unit prices of USD 850/t for sulfuric acid and USD 960/t for SMBS shows that replacing SMBS with raw effluents could save TFM up to USD 3.3/t of ore, which represents an economically attractive option. These savings could increase further, as sulfuric acid is produced on site.

Table 5. Sulfuric acid consumption.

N°	SO_2 dosage (kg/t)	Sulfuric acid consumption (kg/t)	
		SMBS	Effluents
1	4	15.1	19.5
2	5	15.8	20.2
3	6	15.1	23.2
4	7	15.8	23.9
5	5	15.7	20.3

Table 6. Cost analysis between excess acid consumption and use of SMBS.

Item	Consumption (kg/t ore)	Unit price (USD/t reagent)	Cost (USD/t ore)
Acid overconsumption	4.4	850	3.74
SMBS (for 5 kg SO_2/t ore)	7.42	960	7.12
Recoverable amount			3.38

Increasing the bisulfite concentration through effluent pretreatment could therefore reduce acid consumption and improve both the effectiveness and practical applicability of the effluents as an alternative reducing agent under TFM plant conditions.

4. Conclusions

Leaching experiments conducted under TFM operating conditions demonstrated that cobalt extraction is strongly dependent on reducing-agent dosage, with recoveries approaching 90% at 5 kg SO_2/t for both SMBS and acid plant effluents. Increasing the dosage beyond this level did not improve cobalt recovery. Advancing the reducing-agent addition from t = 1 h to t = 0 h had little effect on cobalt extraction but enhanced copper recovery by limiting Chevreul's salt precipitation.

Overall, acid plant effluents exhibited a reductive performance comparable to that of SMBS, demonstrating their potential as an alternative reducing agent for cobalt leaching. However, the use of untreated effluents increased H_2SO_4 consumption by approximately 4.4 kg/t ore, mainly due to their lower bisulfite concentration, although the additional acid cost remains economically offset by the SMBS savings. Increasing the bisulfite concentration through effluent pretreatment, or partially substituting SMBS with effluents, could therefore provide a practical approach to reducing SMBS consumption while maintaining leaching performance.

Author Contributions

Conceptualization and methodology, Emmanuel Ciza Murhabazi, Kaniki Tumba, Emmanuel Kalimwera Lungwe and Erick wa Mudiata Koshi; formal analysis and investigation, Emmanuel Ciza Murhabazi, Emmanuel Alexandre Kasororo, and Emmanuel Kalimwera Lungwe; writing original draft preparation, Emmanuel Ciza Murhabazi, Kaniki Tumba and Emmanuel Alexandre Kasororo; writing review and editing, Emmanuel Ciza Murhabazi, Emmanuel Alexandre Kasororo, and Kaniki Tumba; Supervision, Kaniki Tumba, Emmanuel Kalimwera Lungwe and Erick wa Mudiata Koshi. All authors have read and agreed to the published version of the manuscript.

Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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