

# Extraction of Valuable Metals from High-Iron Zinc Sulfide Concentrate by Reductive Leaching

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**Abstract:** This study was conducted as part of the development of a new process to recover valuable metals of zinc, copper and indium from high-iron zinc sulfide concentrate (HIZSC) by the reductive leaching of high-iron zinc neutral leaching residue (HIZNLR) with zinc concentrate precipitation of copper with iron powder, neutralization of free sulfuric acid with zinc calcine, precipitation of indium with zinc powder and hematite precipitation to reject iron. Among these stages, reductive leaching is the crucial step, which makes that valuable metals in the materials can be effectively leached and ferric iron in the solution is reduced to ferrous iron. The reductive leachate enables copper, indium and iron separation from the solution in the subsequent process. The results show that by increasing the sulfuric acid concentration, HIZSC addition and prolonging the reaction time, the leaching efficiency of zinc, copper, indium and iron in the sample materials was significantly increased. The maximum metal-leaching efficiency was obtained; 84.3% of the iron was in its soluble ferrous state, and zinc ferrite in the HIZNLR was almost entirely dissolved under the experimental conditions of an H<sub>2</sub>SO<sub>4</sub>-to-HIZNLR mass ratio of 1.6, a liquid-to-solid ratio of 11.34 mL/g, a temperature of 90°C, an HIZSC addition of 1.05 times of the stoichiometric amount and a reaction time of 5 h.

**Keywords:** High-Iron Zinc Ore, Neutral Leaching Residue, Reductive Leaching, Leaching Efficiency

## 1. Introduction

Because ideal zinc concentrates become increasingly scarce, the local zinc industry focuses significant attention on the high-iron zinc sulfide ore with totals nearly seven million tons in the Yunnan Province of China [1]. The high-iron zinc sulfide concentrate (HIZSC) has an average zinc content of 45.0% and an iron content of 17%, which is not suitable for treatment using the conventional zinc hydrometallurgy process; thus, researchers have been studying the feasibility of processing this type of ore [2-4]. The first company to perform oxygen pressure acid leaching of HIZSC was operated in the Yunnan Province of China in 2005 with a zinc designing production capacity of 100,000 t/y; iron was removed using a

combination of jarosite and goethite, and the zinc production was only 10,000 t in 2005 [5]. Iron control is one of the key issues in HIZSC treatment.

Based on the previous study [6], a new process is presented to efficiently recover the valuable metals of zinc, copper and indium in HIZSC and reject iron as hematite. The process consists of five stages: reductive leaching of high-iron zinc neutral leaching residue (HIZNLR) with HIZSC, precipitation of copper with metallic iron, neutralization of free sulfuric acid with zinc calcine, precipitation of indium with zinc powder and hematite precipitation to reject iron. Figure 1 shows the schematic diagram of the new HIZSC treatment process.

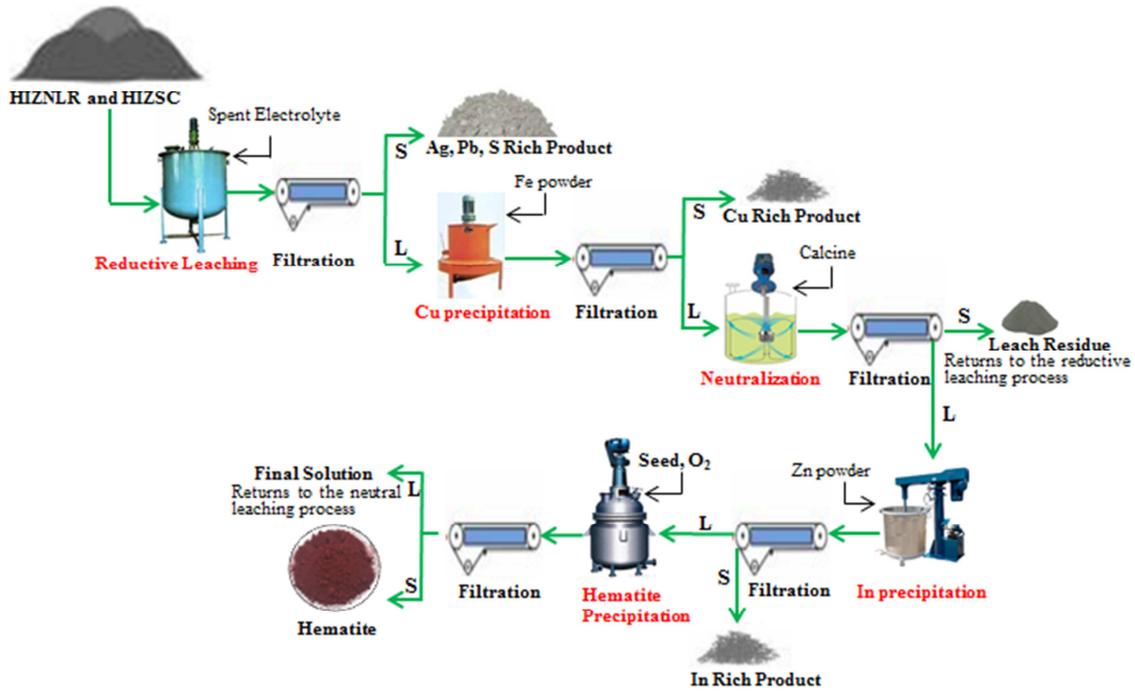


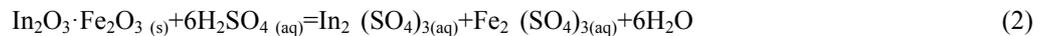
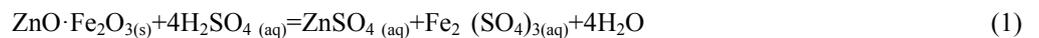
Figure 1. Schematic diagram of the new HIZSC treatment process.

In this process, HIZNLR is leached by spent electrolyte using HIZSC as the reducing agent to increase the valuable-metal leaching efficiency of zinc, copper and indium, and the ferric iron leached from HIZNLR is simultaneously reduced to ferrous iron in an acidic environment by the sulfide sulfur in the HIZSC. The slurry produced from the reductive leaching stage is filtered to separate the lead-, silver- and sulfur-containing residue. The solution obtained from filtration is treated in three steps to recovery copper and indium. In the first step, metallic iron powder is added to the solution to precipitate copper, and 99% of the copper in the solution is collected as metallic copper. In the second step, the copper-free solution is neutralized to pH 4.0 with zinc calcine, and the slurry is filtered to separate the leaching residue, which contains undissolved zinc, indium and copper; then, the

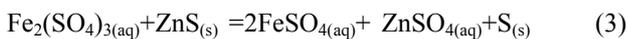
residue returns to the reductive leaching process. In the third step, indium in the solution from the neutralization step is recovered by adding zinc powder. Finally, the solution is fed into the hematite precipitation autoclave, where the ferrous ion is oxidized and precipitated as hematite at an elevated temperature under oxygen pressure. The product liquor, which contains zinc and sulfuric acid generated by the hydrolysis of iron, is recycled to the neutral leaching process.

A significant portion of zinc, iron and indium in the HIZNLR occurs in the forms of zinc ferrite ( $ZnO \cdot Fe_2O_3$ ) and indium ferrite ( $In_2O_3 \cdot Fe_2O_3$ ), and the reductive leaching process involves:

(i) Dissolution of zinc ferrite or indium ferrite and the release of ferric ions:



(ii) Reduction of ferric ions produced from the HIZNLR to ferrous ions by sulfide sulfur in the HIZSC:



By dissolving the HIZNLR in reactions (1) and (2), ferric ions are released. However, the high concentration of ferric ions decelerates the dissolution rate of zinc ferrite [7-11]. Furthermore, it is difficult to separate and recover zinc and indium from the leaching solution when most iron ions in the leaching solution are ferric ions. Studies have shown that the reducing conditions in the leaching process can improve the dissolution rates of zinc from zinc ferrite [12-13].

Markus et al.[14-15] examined the reduction kinetics of ferric iron to ferrous iron using a sphalerite concentrate and

found that the reaction rate increased with an increasing ferric iron concentration and the amount of the reducing agent in proportion to the amount of ferric iron. Wu et al. [16] investigated the reductive leaching of zinc residue to recover gallium using  $SO_2$  gas as a reductant. They demonstrated that more than 95% of the iron is reduced by sulfur dioxide into the solution. Zhang et al. [17] observed that the addition of hydrazine sulfate as a reductant for the sulfuric acid leaching of cadmium from a zinc neutral leaching residue. Under optimal conditions, 90.81% of Cd, 95.83% of Zn and 94.19% of Fe were extracted. In Akita Zinc Co., Ltd., zinc neutral leaching residue is leached with spent electrolyte and  $SO_2$  gas as the reductant [18].

In previous investigations, the reductive leaching of zinc

residuals has mainly focused on relatively low-iron (under 10% in concentrate) zinc materials and used sulfur dioxide as the reducing agent. Information relating to the reductive leaching of the high-iron zinc leaching residue (iron content above 15% in the concentrate) with HIZSC as the reducing agent is not available in the literature. In the present work, as part of the new process above, HIZNLR, which is produced from an HIZSC by roasting and neutral leaching under conditions accordant with the industrial practice, has been used for reductive leaching studies in the presence of HIZSC at atmospheric pressure. The sample materials were characterized by mineralogical analysis, and leaching studies have been conducted to understand the effects of various leaching parameters (sulfuric acid concentration, liquid-to-solid ratio, reaction time and HIZSC addition) on the zinc, copper, indium and iron extractions and the reduction of

ferric to ferrous iron.

## 2. Experiments

### 2.1. Materials Feed and Mineralogical Analysis

The HIZSC in the present study was from the Yunnan Province of China. It was the product of the flotation operations of zinc sulfide minerals, which was further ball-milled. The HIZNLR was prepared from the HIZSC under conditions accordant with the industrial practice. Table 1 shows the particle size distribution of the HIZSC and HIZNLR in this study. The chemical compositions of these materials are shown in Table 2. The mineralogical analysis of the samples was performed using XRD, and the results are shown in Figures. 2 and 3.

Table 1. Particle size distribution of the HIZSC and HIZNLR.

HIZSC		HIZNLR	
Size/ $\mu\text{m}$	Cumulative percentage passed	Size/ $\mu\text{m}$	Cumulative percentage passed/%
74	100	147	100
61	86.2	104	92.3
53	60.9	74	71.5
43	33.5	61	40.6

Table 2. Chemical composition of the experimental materials.

Component	Zn	Fe	In, g/t	S	Cu	Pb	Sn	Ag, g/t	SiO <sub>2</sub>
HIZSC%	45.37	16.82	386.7	32.31	0.96	0.081	0.069	43.68	2.43
HIZNLR%	25.45	34.73	853.6	3.52	1.47	0.172	0.113	73.14	5.44

As shown in Table 2, the HIZSC contained 45.37 wt.% zinc, 16.82 wt.% iron, 32.31 wt.% sulfur, 0.96 wt.% copper, 386.7 g/t indium and 2.43 wt.% silicon dioxide. Minor amounts of other metals were also present, but there was less than 0.1 wt. % of each. The iron content in the HIZSC was higher than the conventional zinc sulfate concentrate in the present zinc refinery.

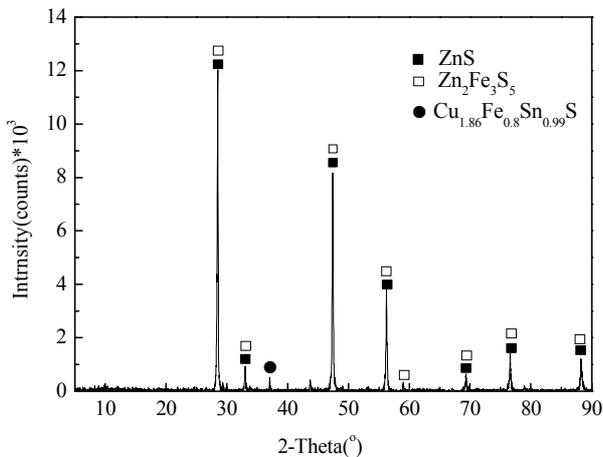


Figure 2. XRD pattern of the HIZSC.

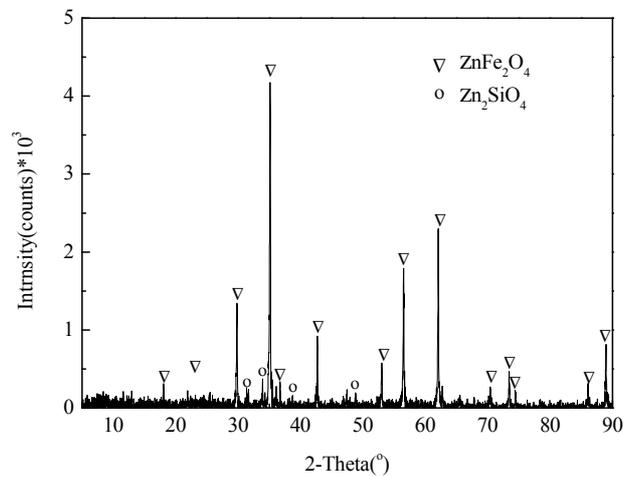


Figure 3. XRD pattern of the HIZNLR.

Figure 2 shows that the main mineralogical phases in the HIZSC are sphalerite and marmatite with a small quantity of polymetallic sulfide. Pyrite and pyrrhotite, which are common minerals in zinc sulfide concentrate, are not detected in the sample materials. As shown in Figure 3, undissolved zinc ferrite and a small amount of zinc silicate are detected in the HIZNLR. The mineralogical compositions of zinc and iron in the sample materials were further identified, and the results are shown in Tables 3 and 4, where the Zn content in the mass fraction of zinc in the HIZSC or HIZNLR, and the Zn

percentage is the mass fraction of different compounds in the sample materials. The same notations is also for Fe. Tables 3 and 4 show that 95.33% Zn and 94.35% Fe occur in the form

of sulfide in HIZSC, and zinc ferrite and zinc silicate account for a large percentage of the HIZNLR, which is consistent with the XRD results in Figures. 2 and 3.

**Table 3.** Mineralogical composition of zinc in both HIZSC and HIZNLR.

Constituent/pct	HIZSC		HIZNLR	
	Zn content	Zn percent	Zn content	Zn percent
Sulfide	43.25	95.33	0.87	3.42
Carbonate	0.56	1.23	1.32	5.19
Silicate	0.82	1.81	3.76	14.77
Ferrite	0.32	0.71	17.68	69.47
Others	0.42	0.93	1.82	7.15
Total	45.37	100	25.45	100

**Table 4.** Mineralogical composition of iron in both HIZSC and HIZNLR.

Constituent/pct	HIZSC		HIZNLR	
	Fe content	Fe percent	Fe content	Fe percent
Sulfide	15.87	94.35	0.15	0.43
Magnetite	0.46	2.73	2.38	6.85
Silicate	0.13	0.77	1.27	3.66
Hematite	0.24	1.43	28.47	81.98
Others	0.12	0.71	2.46	7.08
Total	16.82	100	34.73	100

The spent electrolyte in the experiments contained 185.6 g/L H<sub>2</sub>SO<sub>4</sub> and 52.33 g/L Zn<sup>2+</sup>.

## 2.2. Equipment and Leaching Procedure

Leaching experiments were performed in a three-necked 2-L-capacity glass reactor with a continuously stirring mechanical stirrer, a sample collection and a condenser tube to prevent evaporation loss. The glass reactor was heated in a water bath and maintained at 90°C for all tests. The required amount of spent electrolyte was transferred to the reactor and placed in the water bath. After the test temperature was attained, 80g of HIZNLR and a calculated amount of HIZSC with the particle size of 74 μm was added to the reactor. Then, the solution was agitated at 300 rpm for all tests. During each experiment, the samples were collected at regular times and immediately filtered on a Buchner vacuum filter to separate the liquor from the slurry. The solids were thoroughly washed and subsequently dried at 80°C before analysis. The liquids were analyzed to obtain the zinc, iron, indium and copper concentrations. The leaching efficiency of the metals is calculated using formulas (4) and (5).

$$\eta'(\%) = \frac{(C \times V - 52.33 \times V') \times 100}{C_1 \times \omega_1 + C_2 \times \omega_2} \quad (4)$$

$$\eta(\%) = \frac{C \times V \times 100}{C_1 \times \omega_1 + C_2 \times \omega_2} \quad (5)$$

where  $\eta'$  is the leaching efficiency of zinc;  $\eta$  is the leaching efficiency of indium, copper or iron; C is the metal concentration in the leachate, g/L; V is the leachate volume, L; V' is the volume of spent electrolyte, L; C<sub>1</sub> is the wt.% of metals in the HIZNLR;  $\omega_1$  is the amount of HIZNLR; C<sub>2</sub> is the wt.% of metals in the HIZSC;  $\omega_2$  is the amount of HIZSC.

Zinc was analyzed by complex titration with EDTA. The

ferrous ion concentration was determined by titration against potassium dichromate with sodium diphenylamine-4-sulfonate as an indicator. The total iron concentration was similarly determined, but the ferric ion was first reduced with stannous chloride before titration against potassium dichromate. Indium and copper were determined by ICP with mass spectrometric detection. Some solids were analyzed using XRD to determine their composition.

## 3. Results and Discussion

The main variables are the H<sub>2</sub>SO<sub>4</sub>-to-HIZNLR mass ratio, liquid-to-solid ratio (mL:g), reaction time and HIZSC addition. The results are summarized as below.

### 3.1. Effect of the Sulfuric Acid Concentration

The initial sulfuric acid concentration was determined by the dissolution reaction of zinc ferrite, indium ferrite and copper oxide in the HIZNLR with sulfuric acid.

The effect of the sulfuric acid concentration on the leaching efficiency of zinc, copper and indium and the concentrations of total iron and ferrous ions in the leachate was studied for the H<sub>2</sub>SO<sub>4</sub>-to-HIZNLR mass ratios of 1, 1.2, 1.4, 1.6 and 2.0. The liquid-to-solid ratio was 11.34, the reaction time was 5 h, the temperature was 90°C, and the HIZSC addition was 1.05 times the stoichiometric amount. The results in Figure 4 indicate that an increase in an H<sub>2</sub>SO<sub>4</sub>-to-HIZNLR mass ratio from 1 to 2.0 produced a significant increase in the leaching efficiency of zinc, copper, indium and iron. The amount of leached metals was 68.91% Zn, 48.9% Cu, 74.79% In and 55.43% Fe, whereas 95.65% Zn, 97.66% Cu, 97.41% In and 92.46% Fe were leached when the H<sub>2</sub>SO<sub>4</sub>-to-HIZNLR mass ratio increased from 1.0 to 2.0.

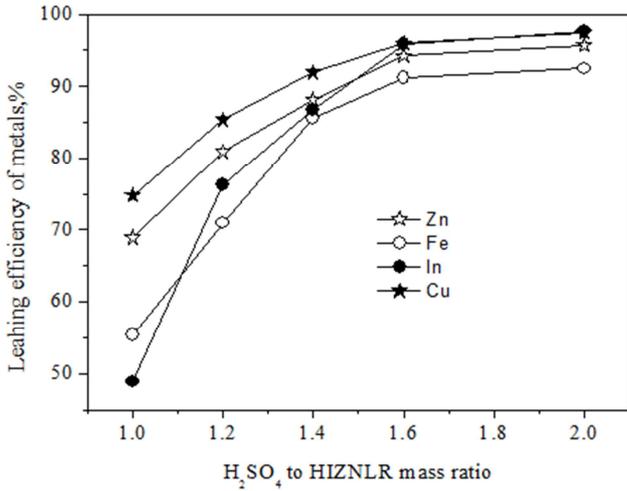


Figure 4. Effect of the H<sub>2</sub>SO<sub>4</sub>-to-HIZNLR mass ratio on the metal extraction.

Figure 5 shows that the concentrations of total iron and ferrous ions in the leachate increased when the H<sub>2</sub>SO<sub>4</sub>-to-HIZNLR mass ratio increased to approximately 1.4, and a further increase in the H<sub>2</sub>SO<sub>4</sub>-to-HIZNLR mass ratio to 2.0 had no significant effect. The H<sub>2</sub>SO<sub>4</sub>-to-HIZNLR mass ratio of 1.6 was selected in later tests to increase the extraction rates of zinc, copper and indium.

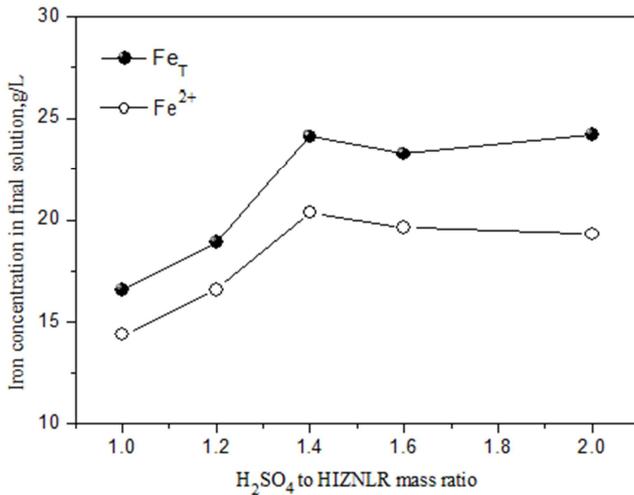


Figure 5. Effect of the H<sub>2</sub>SO<sub>4</sub>-to-HIZNLR mass ratio on the Fe<sub>T</sub> and Fe(II) concentrations in the leachate.

### 3.2. Effect of the Liquid-to-Solid Ratio

Figure 6 shows the amounts of extracted zinc, copper, indium and iron as a function of the liquid-to-solid ratio of 6.44-16.24 mL/g of solid materials. These data were obtained using a H<sub>2</sub>SO<sub>4</sub>-to-HIZNLR mass ratio of 1.6, a reaction time of 5 h, a temperature of 90 °C and a HIZSC addition of 1.05 times the stoichiometric amount. Figure 6 shows that an increase in a liquid-to-solid ratio from 6.44 to 13.79 slightly increased the zinc leaching efficiency from 92.79% to 94.56% and decreased the copper, indium and iron leaching efficiency, but a further increase of the liquid-to-solid ratio to 16.24 had no significant effect. The liquid-to-solid ratio significantly affects the concentrations of total iron (Fe<sub>T</sub>) and the ferrous

ion (Fe (II)) in the leachate.

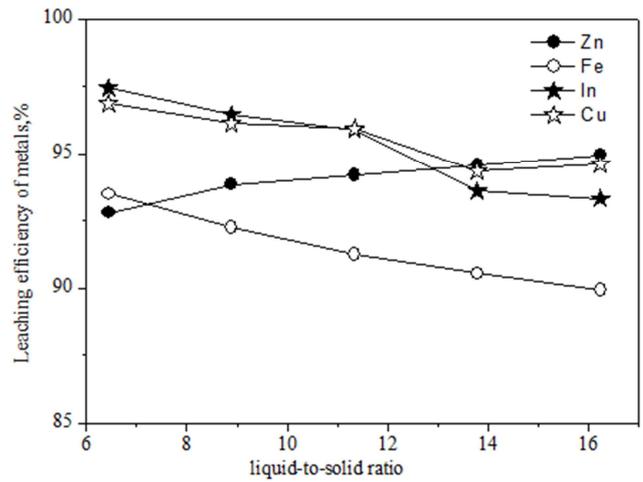


Figure 6. Effect of the liquid-to-solid ratio on the metal extraction.

Figure 7 shows that the metal concentrations in the experimentally obtained leachate with a liquid-to-solid ratio of 6.44 were 36.9 g/L Fe<sub>T</sub> and 30.1 g/L Fe (II), whereas they were 18.45 g/L Fe<sub>T</sub> and 16.37 g/L Fe (II) with a liquid-to-solid ratio of 16.24. This resulted from the dilution of the leachate by increasing the liquid-to-solid ratio.

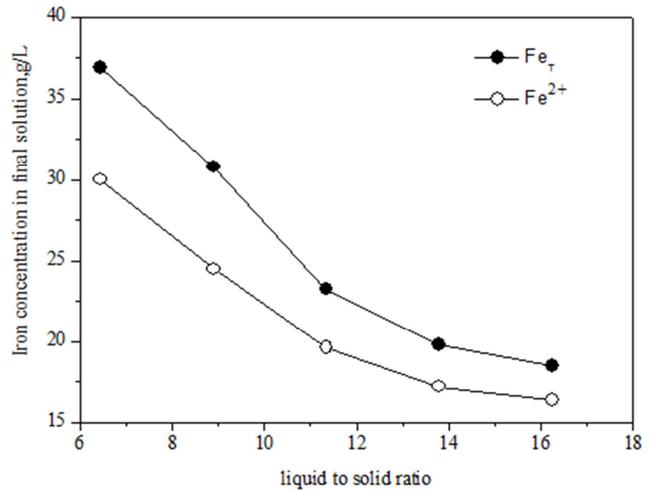


Figure 7. Effect of the liquid-to-solid ratio on the Fe<sub>T</sub> and Fe(II) concentrations.

### 3.3. Effect of the Reaction Time

Figures 8 and 9 show the effect of the leaching time on the dissolution efficiency of the studied ores under the condition of an H<sub>2</sub>SO<sub>4</sub>-to-HIZNLR mass ratio of 1.6, a liquid-to-solid ratio of 11.34 mL/g, a temperature of 90 °C and an HIZSC addition of 1.05 times the stoichiometric amount. As shown in Figure 8, the extraction rates of zinc, copper, indium and iron significantly increased in the reaction time range. The overall zinc, copper, indium and iron extractions after 5 h of leaching were 94.21%, 95.93%, 95.89% and 91.24%, respectively. Figure 9 shows that after 5 h of leaching, the Fe<sub>T</sub> and Fe (II) concentrations increased from 16.75 to 23.25 g/L and 11.95 to

19.6 g/L, respectively. The differential concentrations of  $Fe_T$  and  $Fe(II)$  are almost identical in the range of 1-5 h of leaching time.

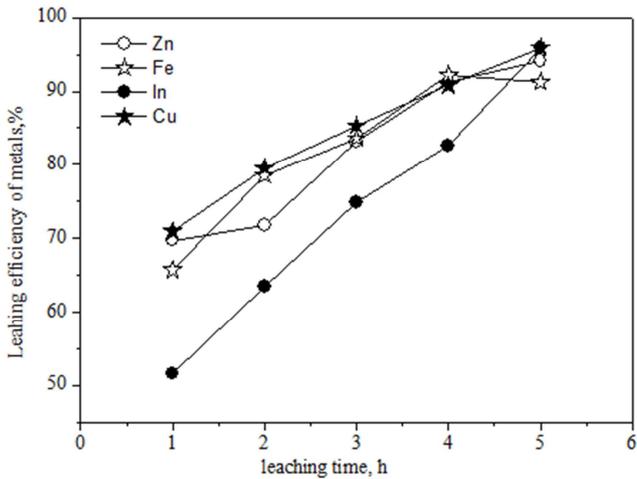


Figure 8. Effect of the leaching time on the metal extraction.

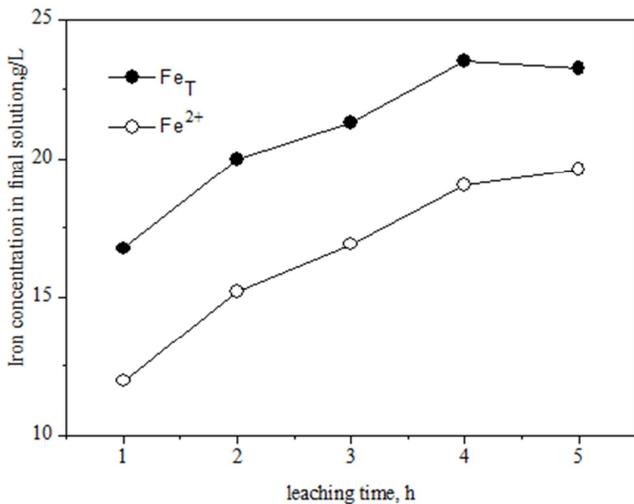


Figure 9. Effect of the leaching time on the  $Fe_T$  and  $Fe(II)$  concentrations.

### 3.4. Effect of the HIZSC Addition

The HIZSC addition was calculated using reaction (3), and experiments were performed for different multipliers times of the stoichiometric amounts of HIZSC (0-1.3) with an  $H_2SO_4$ -to-HIZNLR mass ratio of 1.6, a liquid-to-solid ratio of 11.34 mL/g, and a temperature of 90°C for 5 h.

The results in Figures. 10 and 11 clearly indicate that the effect of the HIZSC reducing agent is significant in the atmospheric acid leaching of the HIZNLR. Figure 10 shows that without using HIZSC as the reducing agent, the leaching efficiency of zinc, copper, indium and iron were only 88.75%, 90.83%, 81.43% and 78.63%, respectively, which implies that the HIZNLR was only partially leached. This result may be attributed to the high  $Fe(III)$  concentration in the leaching solution, which decelerated the dissolution of zinc ferrite (Ramachandra Sarma et al., 1976). Figure 10 also shows that the leaching efficiency of metals significantly increased when the HIZSC addition

increased to approximately 0.8 times the stoichiometric amount; thereafter, it decreased when the HIZSC addition increased from 0.8 to 1.3. Figure 11 shows that the  $Fe(II)$  concentration is approximately zero, and the total iron in the leachate is almost  $Fe(III)$  in the absence of HIZSC as the reducing agent. To effectively recover copper and indium from the leachate and minimize the formation of jarosite or basic iron sulfate, which would contaminate the hematite product with sulfate, the iron in the leachate should be in the ferrous state. By adding a certain amount of HIZSC as the reducing agent, ferric iron is simultaneously reduced to ferrous iron.

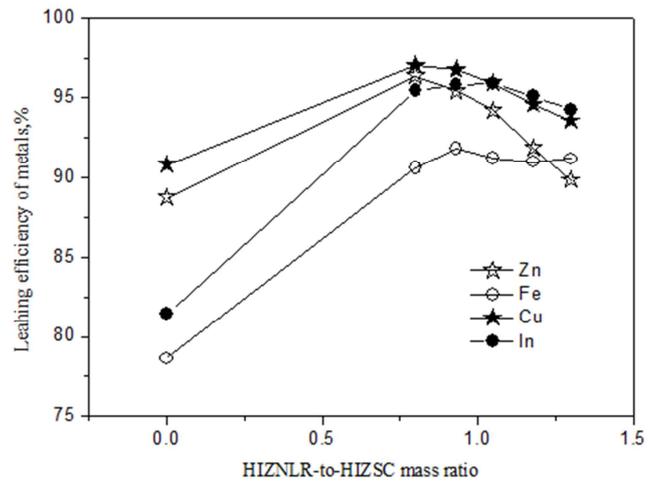


Figure 10. Effect of the HIZNLR-to-HIZSC mass ratio on the metal extraction.

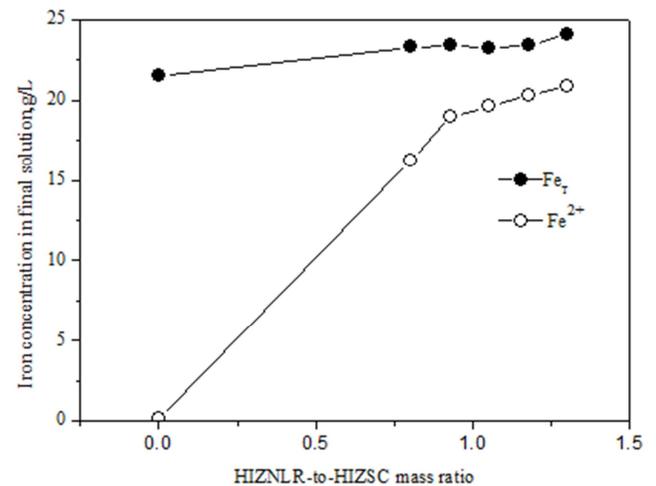


Figure 11. Effect of the HIZNLR-to-HIZSC mass ratio on the  $Fe_T$  and  $Fe(II)$  concentrations.

The leaching residues obtained without an HIZSC reducing agent and with 1.05 times the stoichiometric amount of HIZSC were detected using XRD, and the results are shown in Figure 12. Figure 12 shows that the main compound in the leaching residue produced without addition of HIZSC is undissolved zinc ferrite. With 1.05 times the stoichiometric amount of HIZSC, the dissolution of zinc ferrite was not retarded, and zinc ferrite was almost entirely dissolved.

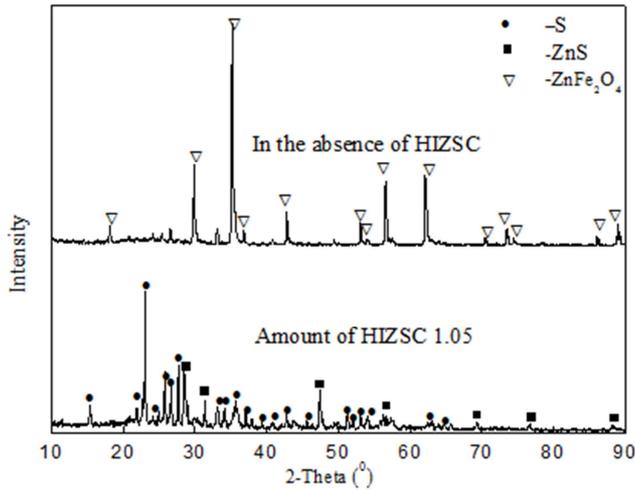


Figure 12. XRD pattern of leaching residues.

Table 5. Chemical composition of the reductive leaching residue.

components	Zn	Fe	S	Cu	In, g/t	Pb, g/t	Ag	SiO <sub>2</sub>
Content, %	11.25	13.41	36.57	0.39	211.84	0.81	634.7	14.34

Table 6. Concentration of the main component in the leachate.

components	Zn <sup>2+</sup>	Fe <sub>T</sub>	Fe <sup>2+</sup>	Cu <sup>2+</sup>	In <sup>3+</sup>	H <sub>2</sub> SO <sub>4</sub>	Sn <sup>2+</sup>	SiO <sub>2</sub>	As <sup>3+</sup>	F <sup>-</sup>	Cl <sup>-</sup>
Concentration, g/L	101.5	23.25	19.6	1.424	0.092	38.34	0.025	1.108	0.24	0.152	0.262

The proposed flowsheet for the HIZSC treatment comprises five main steps, among which the reductive leaching of HIZNLR with HIZSC is the key stage. During the reductive leaching process, HIZSC can successfully be used as a reducing agent and simultaneously leached. The reduction of ferric iron in the HIZNLR to ferrous iron and the subsequent oxidation and precipitation of ferrous iron as hematite will solve the problems in both the jarosite and goethite processes. Valuable metals such as copper, indium and silver, which are rich in HIZSC, can also be collected since the solution pH can be maintained at a relatively high level without ferrous iron precipitating as hydroxide. The proposed flowsheet is simple, fittable, and environmentally friendly and has a high recovery level of valuable metals.

## 4. Conclusions

The HIZSC has a higher iron content than the conventional zinc sulfate concentrate used in zinc refineries. The major fractions of zinc are associated with phalerite and marmatite. Reductive leaching is required to leach maximum zinc, copper and indium from the mixture of HIZSC and HIZNLR.

A new process including reductive leaching, copper sedimentation, neutralization, indium precipitation and hematite precipitation was presented to efficiently recover the valuable metals of zinc, copper and indium in HIZSC and reject iron as hematite.

The reductive leaching process is required to leach maximum zinc, copper and indium from sample materials, and ferric iron can be effectively reduced to ferrous iron, which is advantageous for the copper, indium and iron separation from the leachate in subsequent processes.

## 3.5. Characterization of the Leaching Residue and Leachate

The chemical compositions of the leaching residue and leachate obtained under the experimental conditions (H<sub>2</sub>SO<sub>4</sub>-to-HIZNLR mass ratio of 1.6; liquid-to-solid ratio of 11.34 mL/g; temperature of 90 °C; HIZSC addition at 1.05 times the stoichiometric amount; reaction time of 5 h) were analyzed, and the results are shown in Tables 5 and 6. Table 5 shows that the leaching residue is a silver-, sulfur- and zinc-rich byproduct, which can be further treated to recover these elements. Table 6 shows that 84.3% of the iron in the leachate was in the ferrous state, which enables copper precipitation from the leachate by metallic iron and precipitation of indium with zinc powder; iron can also be precipitated as hematite by the oxydrolisis of ferrous ions.

The zinc, copper, indium and iron leaching efficiency were 94.21%, 95.93%, 95.89% and 91.24%, respectively, 84.3% of the iron was in its ferrous state, and zinc ferrite was almost entirely dissolved under the experimental conditions of an H<sub>2</sub>SO<sub>4</sub>-to-HIZNLR mass ratio of 1.6, a liquid-to-solid ratio of 11.34 mL/g, a temperature of 90 °C, an HIZSC addition of 1.05 times the stoichiometric amount and a reaction time of 5 h.

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