

# A PYRO-HYDROMETALLURGICAL PROCESS FOR THE RECOVERY OF ZINC FROM JAROSITE WASTE

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#### Abstract

The paper presents a procedure for obtaining Zn from jarosite waste raw material by combining lowtemperature roasting at 530°C and leaching in water with its simultaneous separation from Fe. By lowtemperature roasting, iron was converted from the  $Fe_2(SO_4)_3$  form into  $Fe_2O_3$ , which is insoluble in water and slightly soluble in acid. As zinc sulfate decomposes at 740°C, the zinc remains in the form of ZnSO4, which is easily dissolved in water. In this way, the selectivity of Zn over Fe was achieved. Jarosite is the residue of hot-acid leaching. It is obtained according to the old technology of obtaining zinc. Jarosite with a relatively high content of 4.7% Zn is a technological choice for obtaining products of commercial quality. From the experimental results, by roasting 100g of jarosite for 4 hours at 530°C in electric furnace and leaching the roasted sample in water under the conditions: S:L =1:5, room temperature, time 1h, the leaching degree of 91.97% and 9.60% were obtained for Zn and Fe respectively. By applying the precipitation, using 1MNaOH, on leaching solution, 99.93%Fe is precipitated to the pH=4. Zinc precipitates at a pH > 5.5, so it completely remains in leach solution. Zn in the form of ZnSO4, can be further treated by the electrolysis process in order to obtain pure electrolytic Zn, or by precipitation with Na<sub>2</sub>CO<sub>3</sub> it can be obtained as ZnCO<sub>3</sub> concentrate of commercial quality.

*Keywords*: (jarosite, Zn recovery, low temperature roasting, water leaching, metals selectivity)

### 1. INTRODUCTION

In Serbia, Jarosite + Pb–Ag waste sludge has been disposed of for years, which should be the last solution in the hierarchy of waste management, after prevention of waste generation, reuse of waste, recycling and other types of waste utilization in accordance with the principles of circular economy. Jarosite + Pb-Ag sludge, obtained from central Serbia Elixir Factory "Zorka" in Šabac is waste sludge that was obtained from the hot acid leaching process. The product was obtained using traditional refining technology for obtaining zinc, whereby, in addition to Pb-Ag precipitate, jarosite is also obtained. This waste raw material, in addition to zinc, contains copper, lead, silver, gold and indium in quantities that should not be neglected. The processing proposed in this paper may have commercial benefits compared to the processing methods proposed by many authors. There are many studies in which different procedures for the treatment of jarosite raw materials are presented. Some authors presented the procedure of acid leaching as well as brine leaching for the extraction of Zn and Pb [1-3]. Pyrometallurgical processes for the processing of zinc secondary raw materials and waste deposits from zinc hydrometallurgy are very efficient, but they require expensive industrial plants with a large capacity to be economical [4,5]. This work presents a pyrohydrometallurgical route, for processing jarosite tailing waste with the possibility of separating the present metals and obtaining zinc.

### 2. EXPERIMENTAL

### 2.1 Chemical analysis of jarosite

Chemical analysis of the jarosite sample was performed and presented in Table 1.

л	1 - Element contents (wt/o dry basis) of a jarosite sai							
	Element	Cu	Zn	Fe				
	Element	%	%	%				
	Content	0.7	5.39	30.61				

Table 1 - Element contents (wt% dry basis) of a jarosite sample

### 2.2 Mineralogical Composition of a Jarosite Sample

The Jarosite XRD pattern is shown in Figure 1. It can be seen that beside jarosite, mineral forms of magnetite and anglesite are present.



Figure 1 - Diffractogram of a Jarosite tailings waste sample

## 3. RESULTS AND DISCUSSION

### **3.1 Jarosite roasting and water leaching of calcine**

The experiment of low-temperature roasting of a 100g sample of jarosite was performed in a stationary electric furnace for 4 hours at 530 <sup>o</sup>C. The X-ray powder diffraction of the calcine is shown in Figure 2.



Figure 2 - Diffractogram of calcine obtained by jarosite roasting at "530 °C"

After roasting, the phases present in the calcine were hematite, anglesite and magnetite. After the roasting stage, the calcine is leached in water. Table 2, shows the roasting and leaching conditions for the experiment that showed the best metal recovery.

	Roasting time 4h		Leaching with H <sub>2</sub> O (S:L=1:5), t=1h, pH after leaching	Leaching degree		ree
	ample mass (g)	Temperature roasting (°C)	рН	Zn %	Cu %	Fe %
1.	100	530	2.25	91.97	91.07	9.60

Table 2 - Conditions of jarosite roasting and leaching with degrees of metal leaching

From the Table 2, can be shows that the metals recovery is 91.97%, 91.07%, and 9.60% for Zn, Cu, and Fe respectively. After leaching, under the conditions of S:L = 1:5, leaching time of 1 h, at room temperature, the measured pH of the leach solution was 2.25. It can be noted that in the experiment, there was enough acid to carry out the leaching process, in other words, no acid needed to be added. The reason for the greater degree of leaching of Zn and Cu is that during the roasting process at low temperatures, they remained in the form of sulfate, which is easily leached in water, while iron was transformed into hematite, which is practically insoluble. By measuring the pH after leaching, it was determined that there is no need to leach in acid. For this reason, we decided to use water for the leaching process. Based on past experience gained through many years of research, it was decided that the S:L ratio should be 1:5, which is a reasonable ratio. The selected leaching time of 1h was sufficient because sulfates are easily soluble in water.

## **3.2 Precipitation of Fe**

The leach solution containing Cu, Zn and Fe was treated to separate the present metals. Fe precipitation was done at pH=4 using 1M NaOH. The results show in Table 3 that 100% Zn and 96% Cu remained in the solution after precipitation, while 94% Fe was precipitated from the solution. In this way, Fe was separated in the form of Fe(OH)<sub>3</sub> precipitate, while zinc and copper in the form of sulfates are present in the solution.

Metals	Zn%	Cu%	Fe%
Leaching degree	91.97	91.07	9.6
The percentage content of metal in the solution after precipitation related to the content in the leach solution	100	96.00	6.3
The percentage content of metals in the solution related to the content of the same in the starting raw material	91.97	87.43	0.61

Table 3 - Percent metal content in solution after leaching and precipitation

It can be seen from Table 3, that the percentage metal content of 91.97% zinc and 87.43% copper in the solution in relation to the content of the same in the starting raw material, in fact, represents the utilization of these metals.

# **3.3** Cementation of Cu from solution after Fe precipitation

After the precipitation of iron, in the leach solution, macro components Zn and Cu are present in the form of sulfates as  $ZnSO_4$  and  $CuSO_4$ . In order to achieve selectivity and separation of copper from zinc, copper cementation was carried out at pH=2 with zinc powder, a metal that is more electronegative than copper The necessary amount of zinc for the cementation process was calculated based on the stoichiometrically required amount with 50% excess.

#### 3.4 Precipitation of Zn from solution using Na<sub>2</sub>CO<sub>3</sub>

After copper cementation, the solution was filtered and prepared for obtaining  $ZnCO_3$  from  $ZnSO_4$ . The pH value of the solution was measured, which after Cu cementation was pH=2.84. Precipitation of Zn as  $ZnCO_3$  was carried out up to pH=8 by adding 10% Na<sub>2</sub>CO<sub>3</sub>. The obtained zinc carbonate was washed, dried and submitted to chemical analysis. Zinc carbonate is shown in Figure 3.



Figure 3 - Zinc – carbonate

The characterization of the obtained zinc carbonate was done and showed the following content: Zn=51%, Cu=0.7% i Fe=0.8%.

### 4. CONCLUSION

Zinc from jarosite tailings waste was obtained through the following main stages of treatment: i) jarosite roasting to convert iron sulfate into insoluble hematite, ii) leaching in water in order to leach Zn, iii) precipitation of Fe from leaching solution with 1MNaOH, iv) cementation of Cu with zinc, v) precipitation of Zn from ZnSO<sub>4</sub> solution with Na<sub>2</sub>CO<sub>3</sub>. This paper shows that the combination of roasting and leaching provides a promising process for obtaining ZnCO<sub>3</sub> concentrate of commercial quality. The total recovery of Zn after leaching, Fe precipitation and carbonate precipitation was 91.97%. The quality of the obtained ZnCO<sub>3</sub> was: Zn=51%, Cu=0.7% and Fe=0.84.

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