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RESEARCH ARTICLE

MULTI-METAL RECOVERY FROM FLOTATION TAILINGS WITH CITRIC ACID ON THE NACL MEDIA

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ABSTRACT

After the flotation process of oxidized lead-zinc ores, the high amount of metal (especially zinc metal) in its content cannot be recovered and is stored as tailing. Ore and (therefore) tailings are found together with gangue minerals such as calcite, and dolomite, which are oxide/carbonate minerals. Precious minerals are zinc, lead, silver, and iron-containing minerals such as smithsonite, hydrozincite, plumbojarosite, and goethite. The particle size of the sample taken from this tailing was determined as d_{80}= 78.22µm. In order to recover these ore tailings with high metal content, the dissolution of citric acid, which is a weak organic acid, in NaCl medium was investigated. A basic experimental condition determined was applied as 0.5 M citric acid, 200 g/L NaCl, 1 hour time, 60 ℃ temperature, and 10% solids ratio. As a result, zinc, lead, silver and iron dissolved up to 66.85%, 56.53, 40.68, and 27.74%, respectively. According to the results of this experiment, keeping each of these parameters constant, 0.125-1 M citric acid, 50-400 g/L NaCl, 15-120 minutes leaching time, 25-95 ℃ leaching temperature, and 5-40% solids metal. The efficiency of the gain yields was tried to be determined. When the final results are examined, there are 60-80% zinc, 40-70% lead, 0.01-35% iron, and 11-83% silver recovery efficiencies. In light of these results, it is thought that industrial-scale improvements in multi-metal recovery from oxidized ore tailings may improve positive results.

Keywords: *Flotation tailings, Oxidized lead-zinc, Silver, Citric acid, NaCl, Leaching.*

1. INTRODUCTION

Lead-zinc ores are generally found in oxide, sulfide, and mixed forms. Among these mineralizations, the most enriched are sulfide ores, and they can be recovered by the flotation process [1-3]. In oxide ores, recovery for lead can be made by flotation. It is based on the principle of flotation by sulfurizing the ore surface and adding a collector such as a xanthate. One of the deposits in Turkey is located in the Zamantı region [4,5].

Important oxidized zinc minerals such as smithsonite ($ZnCO_3$), willemite (Zn_2SiO_4), hydrozincite ($2ZnCO_3.3Zn(OH)_2$), zincite (ZnO), and hemimorphite ($Zn_2SiO_3.H_2O$). Although these ores are generally found together with lead-containing minerals, they cannot be recovered by flotation like these minerals. High-grade zinc and iron minerals and low-grade lead can be found in the wastes of these flotation concentrates [6].

In general, lead oxide minerals are cerusite ($PbCO_3$), anglesite ($PbSO_4$), and jarosite-containing beudantite $PbFe_3(AsO_4)(SO_4)(OH)_6$, plumbojarosite $PbFe_6(SO_4)_4(OH)_{12}$ minerals [3, 7-10].

The dissolution reaction of citric acid ($C_6H_8O_7$) in water (Eq. (1)-(2)-(3)) and the reactions between this acid and some minerals of zinc (Eq. (4)) [11], lead (Eq. (5)), and iron (Eq. (6)) [12-14] are as follows:

$$C_6H_8O_{7(s)} + H_2O_{(l)} \leftrightarrow H_3O^+_{(aq)} + C_6H_7O_7{}^-_{(aq)} \qquad (1)$$

$$C_6H_7O_7{}^-_{(s)} + H_2O_{(l)} \leftrightarrow H_3O^+_{(aq)} + C_6H_7O_7{}^{2-}_{(aq)} \qquad (2)$$

$$C_6H_7O_7{}^{2-}_{(s)} + H_2O_{(l)} \leftrightarrow H_3O^+_{(aq)} + C_6H_7O_7{}^{3-}_{(aq)} \qquad (3)$$

$$3ZnO_{(s)} + 2C_6H_8O_{7(aq)} \longrightarrow 3\ Zn^{2+} + 2C_6H_7O_7{}^{3-}_{(aq)} + 3H_2O_{(l)} \qquad (4)$$

$$1.5PbO + C_6H_8O_7 \leftrightarrow 1.5Pb^{2+} + C_6H_5O_7{}^{3-} + 1.5H_2O \qquad (5)$$

$$Fe_3O_4 + C_6H_8O_7 + 2e^- \leftrightarrow 3FeO + C_6H_5O_7{}^{3-} + H_2O + H^+ \qquad (6)$$

The reaction of zinc oxide with salt (NaCl) leaching (Eq. (7)) [15,16] occurs as follows:

$$ZnO + iCl^- + 2H^+ \rightarrow ZnCl_i{}^{2-i} + H_2O \qquad (7)$$

i=1, 2, 3, 4

The dissolution of lead oxide minerals by salt leaching (Eq. (8)) [1, 17-21] is as follows:

$$PbSO_4 + 2Cl^- \rightarrow PbCl_2 + SO_4{}^{2-} \qquad (8)$$

The dissolution reaction of silver with salt and/or hydrochloric acid is as follows (Eq. (9)) [22-24]:

$$Ag(s) + 2Cl^- + H^+ + \tfrac{1}{4}\ O_2(g) = AgCl_2{}^- + \tfrac{1}{2}\ H_2O \qquad (9)$$

For the recovery of lead and silver from lead sulfates in zinc smelter wastes, recovery by roasting, sulfuric acid, and salt leaching were investigated. Sulfides were oxidized step by step, followed by neutral leaching and chlorine dissolution, recoveries of 90-95% Pb, 60-70% Ag were achieved [25]; [26].

Pressure leaching was carried out with nitric acid from concentrates containing Pb-Ag sulfide minerals. The best conditions for the study investigated at a nitric acid concentration of 0.13–0.65 M at operating conditions (130-170 °C) higher than the melting temperature of sulfur (119 °C), 90 min leaching time, in 0.65 M nitric acid medium, and leaching time at 130 °C. approximately 90% Ag and 80% Pb recovery was obtained [27].

Recovery of copper, zinc and lead from brass melting slag by hydrometallurgical processes was investigated. Metal extraction increases in acidic environment with an increase in temperature and in terms of the types of these acids, respectively, as sulfuric, hydrochloric and nitric acids [28].

Metal recovery was made from oxidized Zn/Pb ore by performing two-stage alkali leaching process. Firstly, the ore, which was reduced to a certain size (0.2 mm), was dissolved by caustic (NaOH) leaching process at 90℃ alkaline leaching temperature, 120 min leaching time. Then, metal recovery efficiencies of over 80% were obtained by leaching sodium sulfide ($Na_2S·9H_2O$) at the same temperature [29].

Metal recoveries from zinc leaching wastes by sulphate roasting and water leaching processes were investigated. In this study, the ferric sulfate/zinc ferrite ratio was 1.2, and then dissolved with water at a roasting temperature of 640 °C for 1 hour. As a result of the experiments, the recovery efficiencies were 92.4% Zn, 93.3% Mn, 99.3% Cu, 91.4% Cd, and 1.1% Fe [30].

In light of all this research, this study was carried out in order to evaluate the metals in oxide ore flotation wastes with the dissolution efficiency of citric acid in a salt medium.

2. MATERIAL and METHOD

2.1. Material

The samples used in the experiments were taken from the oxide lead-zinc flotation tailings of the Havadan Mining (Kayseri, Yahyalı) Plant. The moisture of the tailings was dried at about 105 °C, blended, and the sample splits separated. Citric acid leaching was applied to these tailings in a NaCl medium. As test parameter values; 0.125-1 M citric acid concentration, 50-300 g/L sodium chloride concentration, 15-120 minutes leaching time, 25-95℃ leaching temperature, and 5-40% solids were investigated. All test results were compared with the experiment performed at 0.5 M citric acid and 200 g/L NaCl concentrations, 1-hour time, 60℃ temperature, and 10% solids.

Experiments were carried out in a fume hood with a magnetic stirrer with heater. After dissolving, the liquid solution was separated, and the solid was washed and precipitated. Then these samples by drying were sent for analysis.

When the XRF analysis and chemical analysis results of the test sample were examined (Table 1); 2.29% Pb, and 6.28% Zn were detected. In addition, it was determined that the densest elements were Fe (19.40%) and Si (4.39%).

Table 1. The chemical analysis of test sample [10].

Element	**%**	**Element**	**%**
Fe	19.40	**Pb**	2.29
Si	4.39	**As**	0.33
Zn	6.28	**Mg**	0.38
Al	0.98	**K**	0.24
Ca	2.31	**Ti**	0.16

In the particle size distribution analysis of the test sample (Figure 1), it was found that d_{80}= 78.22 µm.

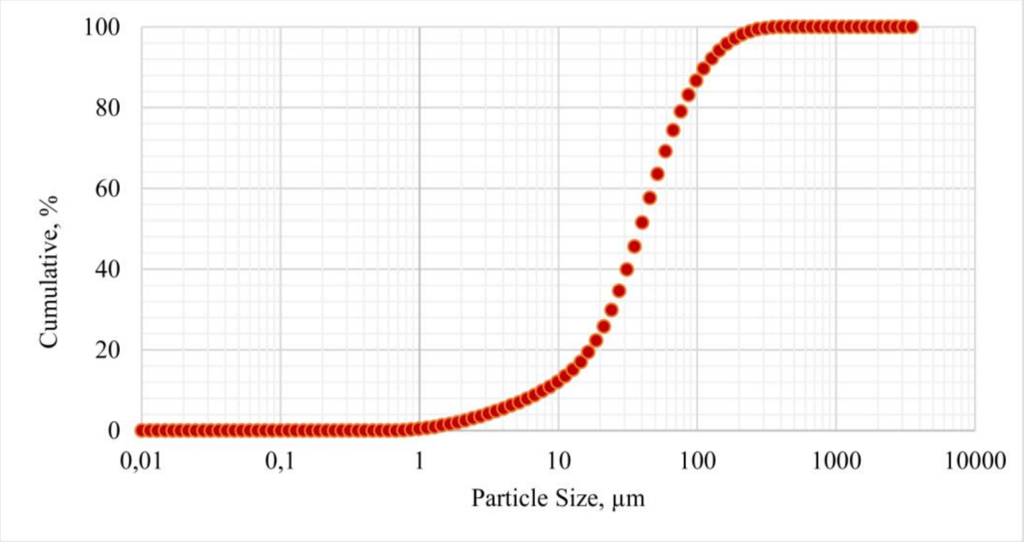

Figure 1. Particle size distribution [10].

In XRD analysis of the test sample (Figure 2), calcite ($CaCO_3$), dolomite ($CaMg(CO_3)_2$), goethite ($FeO(OH)$), hydrozincite ($ZnCO_3.3Zn(OH)_2$), plumbojarosite ($PbFe_6(SO_4)_4(OH)_{12}$), quartz (SiO_2), and smithsonite ($ZnCO_3$) minerals were determined [31].

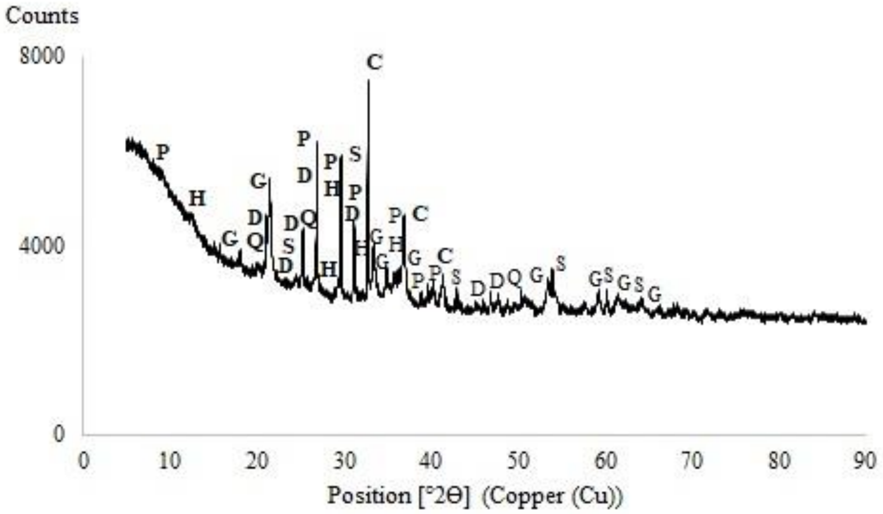

Figure 2. XRD analysis of the tailing sample used in the experiment (C: calcite, D: dolomite, G: goethite, H: hydrozincite, P: plumbojarosite, Q: quartz, S: smithsonite) [31].

3. RESULTS and DISCUSSION

As shown in Figure 3 below, an experiment was conducted under the conditions of 0.5 M citric acid and 200 g/L NaCl concentration, 60℃ leaching temperature, 1 hour leaching time, and 10% solids ratio. As a result of the experiment, the metal recoveries are 40.68%Ag, 56.53%Pb, %66.85Zn, and 27.74%Zn, respectively.

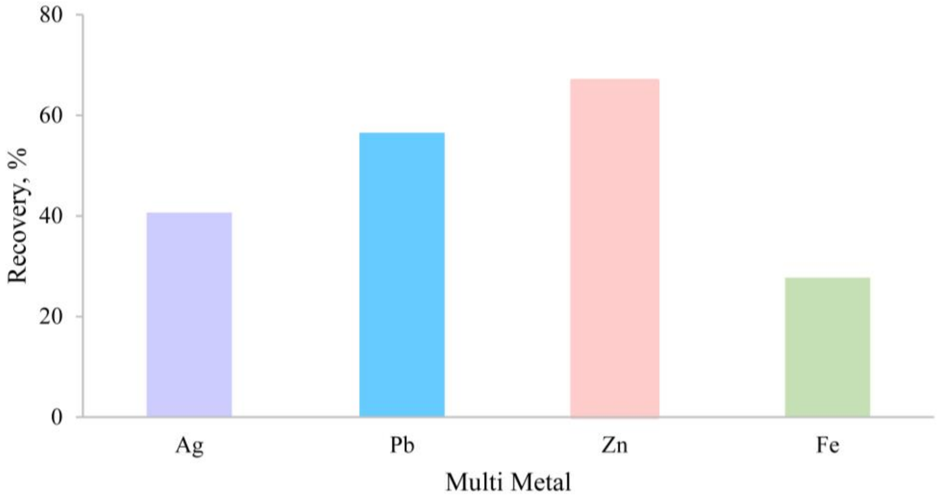

Figure 3. According to stable test, multi-metal recoveries (%) [0.5 M citric acid and 200 g/L NaCl concentration, 60℃ leaching temperature, 1 hour leaching time, 10% solids].

3.1. The Effect of Solid Ratio

In the experiments in which the % solids ratio parameter was examined (Figure 4), 0.5 M citric acid concentration, 200 g/L NaCl concentration, 60℃ leaching temperature and 1 hour leaching time were kept constant. As a result of the experiment, it was observed that the metal recovery efficiency increased as the % solid ratio decreased. Zn recoveries were found to be 17.92-66.85%, Pb recoveries 37.19%-56.53% and Fe 0.12-27.74%. Only Ag recovery efficiencies (26-83%) increased between 20-40%. At the solubility of smithsonite ore in ammonium chloride medium, zinc recovery reached up to 91% in 5 M ammonium chloride, 240 minutes leaching time, 90°C leaching temperature, 84–110 micron grain size and 1/10 solid ratio (g/mL) experiments [32]. In the NaOH dissolution process from refractory hemimorphite zinc oxide ore, 6-12 solid ratio was investigated. Metal recoveries are obtained as 73% Zn, 45% Al, 11% Pb, 5% Cd, and <0.1% Fe at 65-76 μm grain size, 5 M sodium hydroxide, and 10:1 liquid:solid ratio, at 358 K, 2 hours leaching time [33].

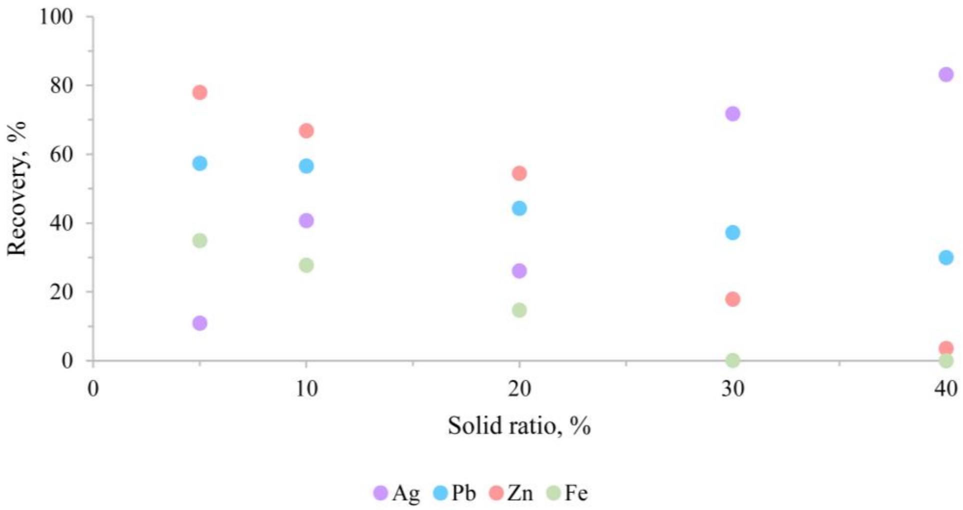

Figure 4. Multi-metal recovery efficiencies obtained according to % solid ratio [0.5 M citric acid concentration, 200 g/L NaCl concentration, 60°C leaching temperature, 1 hour leaching time].

3.2. The Effect of Citric Acid Concentration

In the citric acid concentration difference experiments (Figure 5), 200 g/L NaCl concentration, 60℃ leaching temperature, 1 hour leaching time and 10% solids ratio were kept constant. As a result of the experiment, it was observed that the Zn recovery (16.59-66.85%), Pb recovery (36.16%-56.53%), and Fe recovery (19.39-27.74%) increased as the concentration increased. Recovery of silver decreased by about 20% at 1M citric concentration.

The concentration of citric acid in the range of 0.01-0.5 M in the dissolution of zinc oxide was investigated. It reached up to 70% Zn extraction at a concentration of 0.5 M acid in 60 minutes at 25 °C [12].

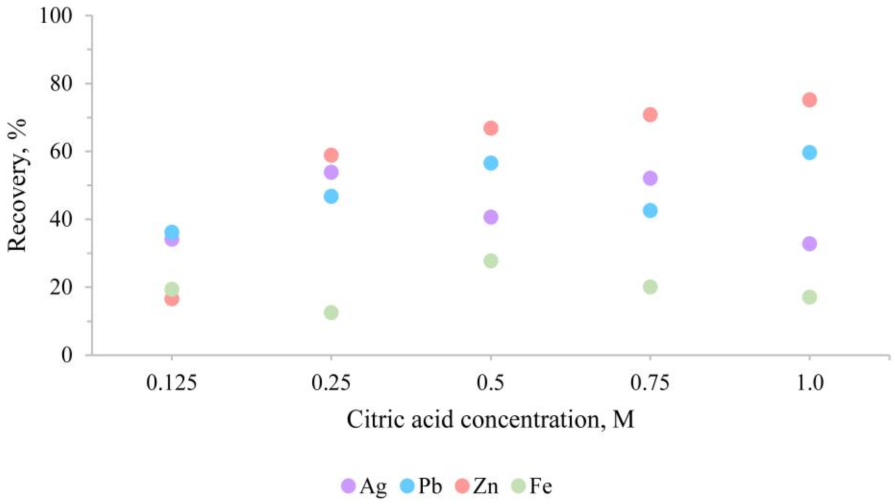

Figure 5. Multi-metal recovery obtained according to citric acid concentration difference [at 200 g/L NaCl concentration, 60°C leaching temperature, 1 hour leaching time, 10% solids].

3.3. The Effect of Leaching Time

In the experiments in which the leaching times were examined (Figure 6), the parameters of 0.5M citric acid concentration, 200 g/L NaCl concentration, 60°C leaching temperature and 10% solids ratio were selected in the leaching time range of 15-120 minutes. The recoveries of metals are 13.70-40.68% for Ag, 42.89-56.53% for Pb, 47.22-66.85% for Zn, and 14.09-27.74% for Fe. It was observed that the leaching time for Ag, Pb, and Fe increased up to 60 minutes with the increase of the time in this parameter. Extraction of zinc and other precious metals from low grade zinc oxide ore with iminodiacetate aqueous solution was investigated. The leaching time was studied between 1-6 hours. Optimum zinc recovery was achieved at 76.6% in 4 hours leaching time, 70 °C, pH 8, L/S 5:1, and 0.9 M iminodiacetate aqueous solution [34]. Zinc recoveries (42-44%) in the leaching time (between 30-150 minutes) do not differ much in the application of 1/10 solids ratio, 25°C leaching temperature and 0.5 M citric acid on low grade ZnO wastes [35].

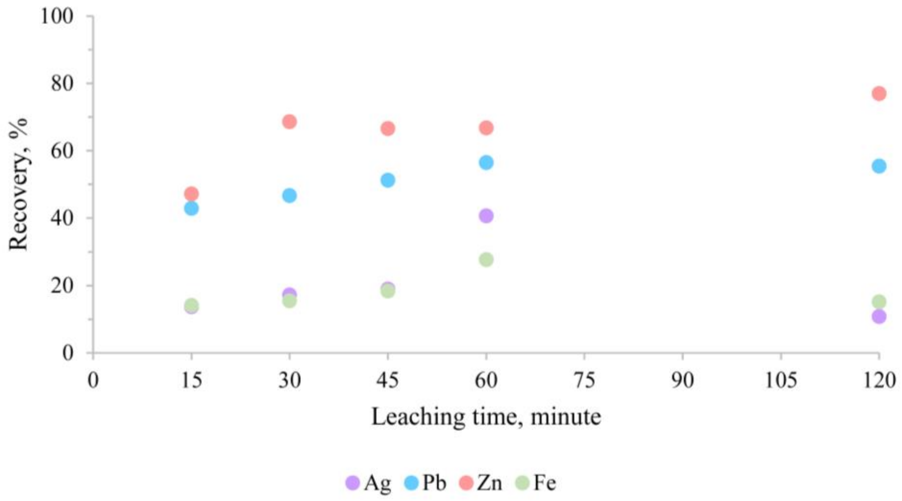

Figure 6. Multi-metal recovery efficiencies obtained according to leaching times [at 0.5M citric acid concentration, 200 g/L NaCl concentration, 60°C leaching temperature, 10% solids].

3.4. The Effect of Leaching Temperature

In the experiments in which leaching temperatures (25-95℃) were investigated (Figure 7), the parameters of 0.5M citric acid concentration, 200 g/L NaCl concentration, 60 minutes leaching time, and 10% solids ratio were selected. In the recovery of metals, it was determined that Ag and Fe recoveries decreased at 95 °C, lead had close values, and zinc increased in direct proportion with the increase (29.15-78.59%) in temperature. In the aforementioned study, where the recovery of ZnO ore with citric acid was mentioned, in the temperature increase examination, the solubility increased towards 60 minutes leaching time at 25-50℃ temperature ranges and was obtained around 90% [12]. When the effect of the leaching temperature parameter of zinc oxide wastes was investigated (30-80℃), the zinc recovery reached up to 65% in 0.5 M concentration of citric acid, 1:10 solid-to-liquid ratio, and 60 min reaction time [35].

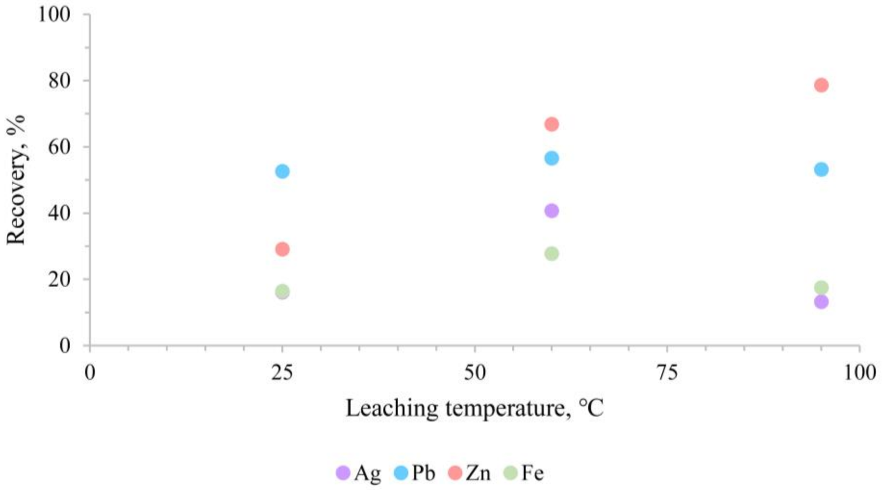

Figure 7. Multi-metal recovery efficiencies according to leaching temperatures [at 0.5M citric acid concentration, 200 g/L NaCl concentration, 1 hour leaching time, 10% solids].

3.5. The Effect of NaCl Concentration

In the experiments where NaCl concentrations were examined (Figure 8), 0.5M citric acid concentration, 60℃ leaching temperature, 1 hour leaching time, and 10% solid ratio were constant. The metal recoveries are 15.02-40-68% for Ag, 42.60-56.53% for Pb, 56.16-78.50% for Zn, and 13.43-27.74% for Fe, respectively. When the citric acid concentration in the chlorine medium (0.05 mol/L) was investigated, it was observed that the zinc solubility reached nearly 100% in leaching times of up to 60 minutes in the temperature range of 25-40 ℃ [12]. Zinc recovery (90%) was obtained after sulphate (H_2SO_4) roasting, and lead (92%) and silver (62%) were recovered in the subsequent NaCl dissolution process. The NaCl concentration range was studied as 60-320 g/L. Ag was obtained in the range of 60-120 g/L with 55-63% and Pb in the range of 60-315 g/L with 17-92% recovery efficiencies [36].

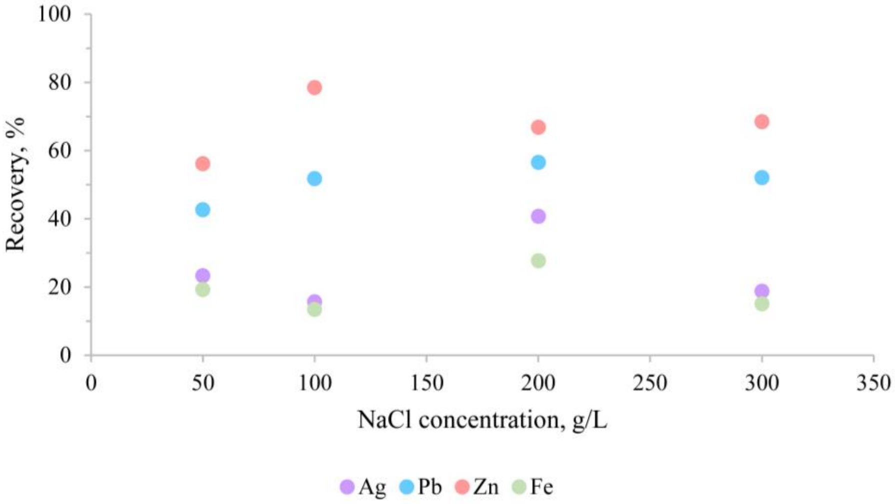

Figure 8. Multi-metal recovery efficiencies according to NaCl concentrations [at 0.5M citric acid concentration, 60°C leaching temperature, 1 hour leaching time, 10% solids].

In the metal recovery of citric acid leaching, it was stated that the metal recovery took place in the following order [13,14] (Eq. (10)):

$$\text{Citric Acid Zn>Cr>Pb>Mn>Fe} \quad (10)$$

Similarly, in these experiments, it is seen that the most intense zinc is recovered, followed by lead and finally iron. In addition, less recovery of iron in the recovery of precious metals such as lead, zinc and silver is a result of the selective behavior of organic acids [10,11, 37,38].

4. CONCLUSIONS

In this study, flotation tailings of oxidised Pb/Zn ores were investigated in terms of solubility of organic acids in citric acid and NaCl medium.

The tailing sample used in the experiments contains 2-3% Pb, 5.5-6.5% Zn, 19-20% Fe, and 20-35g/t Ag, and generally consists of smithsonite, hydrozincite, plumbojarosite, and goethite minerals. The particle size of the test sample was below about 100 microns, and re-grinding was not required.

In the experiment performed at 0.5 M citric acid and 200 g/L NaCl concentration, 60 minutes leaching time, 60℃ leaching temperature, and 10% solids, the recovery efficiencies of the metals were obtained as 66-67% Zn, 56-57%Pb, 27-28% Fe, and 40-41% Ag. These experimental conditions were compared in all other experiments performed.

When the % solid ratio difference was examined, it was determined that as the ratio decreased, metal recovery efficiencies (18-78% Zn, 37-58% Pb, 0-35% Fe, and 11-84% Ag) increased (except for silver only). This increase in silver can be explained by the reasons such as the low amount of availability and therefore the solubility may be faster than other elements.

According to the experiments investigating the citric acid concentration difference (0.125-1M), 17-76% zinc, 36-60% lead, 13-28% iron, and 33-54% silver metal recovery were obtained.

In the 15-120 minute leaching time differences experiments, similar to the other experiments, it was determined that zinc had the highest recovery efficiency and recovery efficiencies of around 57/80%, especially in terms of Pb/Zn.

It was determined that zinc reached 79% efficiency in 25-95 ℃ leach temperature experiments.
In the NaCl concentration differences experiments, zinc was obtained with recovery efficiencies in the range of 56-79%, lead 43-57%, iron 14-28%, and silver 15-41%.

As a result, it has been determined that chlorine leaching of citric acid, a low-cost organic acid, is possible in the recovery of multi-metals from these oxidised ore tailings, especially in terms of Pb/Zn (60 and 80%). In addition, as an important advantage of these acids, it is thought that iron has lower gains, and silver is important in terms of precious metal recovery, although it varies.

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