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Recovery of Cu-Ce from copper slag by using flotation and chemical methods

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ABSTRACT

In our days, slags are considered secondary resources due to the metals they contain. Copper slags having economical operating grades in terms of Zn, Cu, Ce, Ni, Co, Mo, and V, have complex mineralogy due to their thermal processing based formations. Methods of recovery for copper and other precious metals from slags, have been studied, and for which, flotation plants in industrial scale are operated. In cases where physical or physico-chemical mineral processing methods are not sufficient, hydrometallurgical methods are applied. In this study, the flotation method was applied to a copper slag with 0.9% Cu grade. Copper recovery and the concentration grade were determined to be approximately 40 and 22.7% Cu, respectively. Metal extractions were examined through direct leaching tests. Using H_2SO_4 in the leaching, resulted in gel formation, leading to an investigation of the leach parameters using HCl. Cu and Ce extractions of around 70-80% were attained as a result of a test performed for 2 hours with the addition of 25 g/L H_2O_2 at a rate of 20-25% solids and 300 g/L HCl concentration. An examination of the metal relations, indicated that the dissolution behavior of Cu and Ce was quite similar to the Al dissolution (R^2=0.9).

1. Introduction

Production of copper in the present day is performed based on methods such as oxidation in high temperatures, namely the pyrometallurgical method and dissolution through acidic or alkaline solutions, known as the hydrometallurgical method. 80% of the copper produced today are obtained through the pyrometallurgical method with the remaining 20% being obtained through the hydrometallurgical processes (Schlesinger et al., 2011).

Copper minerals, 80% of which contain sulphide, are most prominently found as chalcopyrite ($CuFeS_2$), bornite (Cu_5FeS_4) and chalcosine (Cu_2S). The pyrometallurgical method is preferred in production of pure copper from sulphide minerals, as copper demonstrates a low level of dissolution in aqueous mediums. In order; ore preparation, processing, smelting/converting and purification are the steps followed in the pyrometallurgical method. Hydrometallurgical methods on the other hand are applied in cases of oxidized copper ores, low grade sulphide containing copper ores, scrap metal or metal recovery from waste. In order; leaching, purification and electrolytic recovery are the steps to be followed in hydrometallurgical methods (Beşe, 2017).

In pyrometallurgical methods, a large quantity of slag is produced as a byproduct of copper production. For each ton of copper produced, approximately 2.5 tons of copper slag is obtained. Copper production on a country basis amounts to 4 mt/y for the USA, 2 mt/y for Japan and 360.000, 244.000 and 60.000 t/p

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for Iran, Brazil and Oman, respectively (Najimi et al., 2011).

Copper slags similar to run-of-mine copper ores in terms of their chemical composition, can be used as secondary copper resources. Slags are usually stored in bulks near the smelter. They generally contain 30-40% iron, 35-40% silicate, less than 10% alumina and oxidized calcite and approximately 1% copper; which makes them suitable in delivering the needs in their usage areas (Sanchez and Sudbury, 2013). According to the studies conducted on utilization of these slags, new areas of use such as road metaling material, casting raw material, cement additive and roofing materials were discovered (Chen et al., 2011).

Due to economic reasons, slag waste is now a necessity to be recovered with additional methods as it produces 3% grade Cu when obtained with pyrometallurgical methods and approximately %50 Cu in its flash matte (Uzkut and Tunçer, 1981).

In addition to flotation, hydrometallurgical methods are also preferred in utilization of copper slags. For the process of flotation, cooled and solidified copper slag is prepared after the size reduction processes. A faster cooling process leads to larger copper crystals whereas a slower cooling process results in finer sizes, in which even when fine grinding is process applied, sometimes liberation may never be observed. Copper can be found in oxidized, sulfide containing and metallic forms inside the slag. Thus, as the collector material, various combinations may be used. Collector reagents can be exemplified as xanthates, thiocarbamates and dithiophosphates. Information on certain plants performing copper slag flotation are given in Table 1 (Yılmaz, 2018).

The core principle in hydrometallurgical methods is the dissolution of valuable parts of the rock using acidic or alkaline solution environments. Sulfuric acid, nitric acid, hydrochloric and hydrofluoric acids can be named as the most commonly used acidic solubilizers. High consumptions of acidic solubilizers are not sought after, therefore in cases where the usage is high, alkaline solubilizers are preferred. Alkaline solubilizers, although perform a more selective dissolution, have relatively lower rates of efficiency in metal dissolution. Most commonly used solubilizers can be exemplified as sodium hydroxide, calcium hydroxide, ammonium hydroxide, sodium carbonate, ammonium carbonate, and sodium sulfide (Çakır et al., 2016). Additionally, following sulfidation roasting, water leaching helps move metallic valuables to the solution. Parameters that can be considered advantageous for the hydrometallurgical methods of metal recovery from slags can be counted as follows: 1) prompt and basic process; 2) small amount of initial investment and low operational costs; 3) obtaining products with high added value; 4) flexible capacity scaling (Yao et al., 2018)

Simultaneous dissolution of iron and silica can occur in leaching copper slags with sulphuric acid. Moreover, the dissolution of silica causes gelling in the pulp. Iron-silicate bearing mineral phase inside the

Table 1- Information about the copper slag flotation plants.

Plant	Saganoseki, Japan	Toyo, Japan	Pasar, The Philippines
Capacity, tpd	450	450	370
Feed Grade, % Cu	8.33	6.5	12.5
Concentrate Grade % Cu	21.80	28.00	29.0-33.0
Tailing Grade % Cu	0.65	0.4	0.5-0.6
Recovery, %	95.0	95.0	97.0-98.0
Crushing Circuit	Jaw and 2 Cone Crusher	Roll And 2 Cone Crusher	Jaw and Cone Crusher
Grinding Circuit	Ball Mill x2	Ball Mill	Ball Mill x2
Flotation Particle Size, µm	%50 - 44 µm	%90 - 44 µm	%75 - 45 µm
Flotation Time, min	30	30	30
Collector	SIPX, Thiocarbamate	KAX, Thiocarbamate	KAX, Dithiocarbamate
Frother	Pine Oil, MF560	Pine Oil	Pine Oil, NF183

slag, reacts to sulphiric acid resulting in silicic acid. The gel structure formed in this process not only affects the metal dissolution efficiency but complicates the solid-liquid separation following the leaching process (Banza et al., 2002).

In studies conducted by Basir and Rabah (1999), the dissolubility of metals Cu, Zn, and Pb using copper slags in various acidic and alkaline environments were investigated. In experiments performed using NH_4OH, 81% Cu and 73% Cu dissolubility efficiencies were obtained albeit a very high NH_4OH concentration such as 13 M was used to achieve such rates of efficiency. This projects a very high level of consumption. Although using ammonium hydroxide results in high metal dissolubility efficiencies, reactive consumption levels are also incremental.

Different size fraction flotation attributes of slag wastes belonging to ETİ Copper Smelting Plant were examined by Yılmaz (2018). In accordance with the working specifications of the plant, 45 and 150 micrometers were selected as fine and large particle sizes for the flotation process, respectively. In the mentioned study, a mixture obtained using slag waste from two different furnace types, flash and converter were used in the ratio of 4:1, in order. For the selection of primary collector, kinetic tests with xanthate type KAX and SIPX reagents in two sizes were performed. For the larger particle size, using SIPX, a concentration of 10.98% Cu was obtained with 89.3% recovery efficiency whereas, with KAX, 8.33% Cu concentration with 97.5% recovery efficiency was observed. In support, flotation tests using Mercaptobenzothioazole, R-Isobutyl Dithiophosphate, and Modified Thiocarbamate reagents. In flotation tests, for the larger particle size, with a reagent combination of SIPX+DTP, 11.46% Cu-containing concentrate was produced with 92.87% Cu recovery efficiency. For the finer particle size, on the other hand, 8.19% Cu-containing concentrate was produced with 97.30% Cu recovery efficiency, marking the best results.

The study conducted by Çakır et al. (2016) on copper recovery from copper reffination slag flotation wastes, was performed in two stages namely the leaching process post-calcination of copper slag and direct leaching. The copper slag was put through a mechanical activation process in different duration and rpm values. With direct leaching post-mechanical activation process, low dissolution efficiencies of 10-12% were obtained. Subsequently in the study, with the leaching process following the calcination pre-treatment, relatively higher copper dissolution values were obtained. Calcination processes were performed at 600°C with a duration of 2 hours. During the process, it was aimed to form sulfide-containing structures by adding Na_2SO_4 in different amounts. The copper slag which was calcinated with 80% Na_2SO_4 by weight, was leached at 100 °C with a duration of 1 hour, resulting in %60 Cu dissolution efficiency.

In this study, the aim was to selectively dissolve metals of Cu, Ce and Fe contained inside the selected copper slag waste, targeting to obtain a leach solution rich in Cu and Ce metals. At the outset, flotation was considered and possibilities of obtaining a concentration with sellable quality and efficiency were researched. Subsequently, a dissolution attempt was made with H_2SO_4 leach resulting in gelling which consequently led to HCl being used for the rest of the study. In the dissolution process, the effects of parameters such as reaction times, pulp temperature, acid concentration, H_2O_2 concentration, and solid ratio on metal dissolution were examined, the relation of dissolution behaviors of the metals was researched.

2. Material and Method

In this chapter, information regarding the results of chemical analyses of the sample, mineral composition, methods such as leaching and flotation that are used in the experiments are given.

2.1. Material

Within the scope of experimental studies on the copper slag from the Balıkesir region; following the characterization examination, flotation and leaching experiments were conducted. Mineralogical analyses were performed with XRD (X-ray Diffractometer) and chemical analyses were with ICP (Inductively Coupled Plasma).

The XRD analysis was performed to determine the mineral phases of the slag, peak values of delafossite, a copper-iron oxide mineral, and olivine, known as a slag regulator were observed (Figure 1).

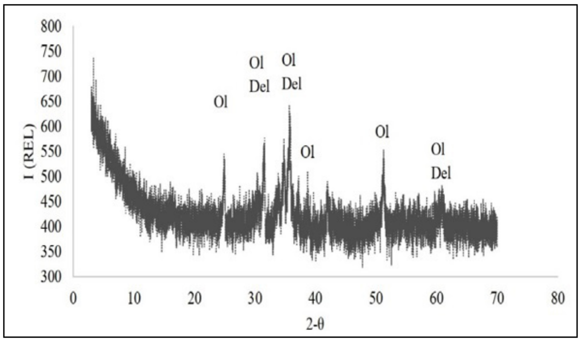

Figure 1- XRD results for the copper slag (O1: Olivine, Del: Delafossite).

Table 2- Chemical analysis results.

Element	%	Element	ppm
Al_2O_3	4.29	Ce	78.64
CaO	7.00	Co	100.64
Fe_2O_3	57.29	Ga	45.22
CuO	1.13	Se	11.90
K_2O	0.55	V	120.68
MgO	0.60	Mo	108.56
MnO	0.25	Au	0.10
Na_2O	0.19		
PbO	0.35		
SiO_2	27.86		
Fire Ignition	0.10		

According to the chemical analysis results (ICP), the copper slag contains approximately 40% Fe, 2.2% Al, 5% Ca, 0.90% Cu and up to 78 ppm Ce. The results of the analysis are presented in Table 2. An integrative evaluation of both XRD and ICP results in points to the dominance of metal-containing phases and the remaining content being silicate minerals.

Sieving tests results and metal distributions of various particle size are given in Table 3. According to the cooling conditions of the slags, minerals can collect on different size groups. Nevertheless, according to the fractional analysis results no such event is observed for Cu and SiO_2. Irregular crystal structure formed in rapid cooling can may be the reason.

2.2. Flotation Experiments

In the flotation experiments within the context of processing tests, standard type bond mill was used for grinding. All flotation tests were performed in -38 µm size fraction group using a Denver D-12 flotation machine. Each experiment was conducted with 30% solid ratio, using 2 kg of sample. In the flotation experiments, KAX (potassium amyl xanthate) and SIPX (sodium isopropyl xanthate), which are commonly used amyl xanthate type collectors and combinations of reagents A208, A238, A407 and A412 were used as supplementary materials. KAX and SIPX being >96% pure, is of Chinese origin. Other reagents, considered as supplementary materials, were procured from Solvay.

2.3. Leaching Experiments

In the leaching experiments within the context of chemical processing, tungsten disc mills from the brand Retch were used for grinding. The material was mixed in 500 ml glass beakers using magnetic mixers with temperature control from the brand IKA. In the leaching experiments, chemicals H_2SO_4 (Merck, 97%) and HCl (Merck, 37%) were used as solvent medium. Dissolution efficiencies were followed up with analysis performed on the solids as well.

Table 3- Sieving test and chemical analysis of run of mine copper slag.

Particle Size, mm	Amount %	Cu, %		Fe, %		Ce, ppm		SiO_2, %	
		Grade	Distribution	Grade	Distribution	Grade	Distribution	Grade	Distribution
+4	3.9	0.75	3.3	39.16	4.0	78.92	3.9	26.70	3.7
-4+3	14.9	0.79	13.1	39.72	15.6	68.92	13.0	26.30	14.0
-3+2	17.0	0.93	17.6	39.09	17.6	80.86	17.5	26.60	16.2
-2+1	22.3	0.86	21.5	38.67	22.8	84.93	24.1	27.10	21.7
-1+0.5	13.9	0.81	12.7	38.11	14.0	56.99	10.1	27.90	14.0
-0.5	28.0	1.02	31.9	35.32	26.1	88.54	31.5	30.20	30.3
Total	100.0	0.90	100.0	37.90	100.0	78.74	100.0	27.86	100.0

3. Mineral Processing

Detailed results of flotation and leaching experiments are presented below.

3.1. Flotation Experiment Results

In flotation experiments, obtaining a concentrate with high copper content was aimed. Xanthate type collectors and supplementary materials were used tandem in the process. The reason for choosing Aerofloat 208 and Aerofloat 238 reagents based on dithiophosphate, A407 and A412 based on mercaptobenzothiazole and dithiophosphate as supplementary materials can be explained as these reagents having common usage in flotation of oxidized ores.

Grade and recovery values of the concentrate are given in Table 4. According to the flotation test results, the highest copper grade of 22.71% was obtained with SIPX, A208 and A238 reagent combination whereas the highest copper recovery of 43.7% was achieved with KAX, SIPX and A407 reagents. Although in concentrations Cu grades of 17-22% were reached, lower than acceptable copper recovery efficiencies were observed. Upon these results, due to the flotation method being not suitable for metal recovery from slag, rest of the study was continued with chemical processing experiments.

3.2. Leaching Experiments

3.2.1. Effects of Duration and Temperature

In order to determine the dissolution behavior of the metals such as Cu, Ce and Fe, a series of experiments with variable temperature and duration values were conducted. In the experiment the solid ratio of 10% and pH value of 2.5 were kept as constants to examine the effects of temperature and duration. (Figure 2)

In the experiments examining the correlation of duration and temperature on the dissolution, temperature was observed to have no effect on copper dissolution, whereas the highest Ce dissolution was achieved at 25 °C. In terms of the duration, a decrease in the pace of dissolution was observed by the end of a 2-hour period for Cu and Ce elements, which indicated 2 hours was the optimal reaction duration. (Figure 2)

3.2.2. Effects of Particle Size

Solvent-mineral contact is one of the most important parameters. Thus, upon achieving adequate particle liberation by applying various size reduction processes, solvent-mineral contact was aimed to be kept at maximum. The cooling conditions of the slag is an important parameter in determining the liberation particle size of the valuable metals contained within. A slow cooling of the slag results in larger crystals whereas with rapid cooling, mineral crystals are not able to grow to sufficient proportions, occasionally being trapped in other mineral matrixes, as inclusions. Even with micronized grinding applied, particle liberation is not achieved sufficiently.

In experiments examining the effects of particle size on dissolution, the solid ratio, pH, duration and temperature values were kept constant. In all experiments, the effect of particle size on metal dissolution efficiency was examined by keeping 25 ºC, pH: 2.5, 10% solids ratio and 120 minutes leaching time as constants (Figure 3).

These results clearly indicate the direct correlation of decreasing particle size with increasing dissolution efficiency. Decreasing the particle size to 38 micrometers does not lead to a considerable Cu dissolution efficiency whereas to an increase from 35.8% to 46.4% for Ce, and from 17.5% to 33% for Al. Following the fine grinding, due to its effects on the

Table 4- Copper slag flotation test results.

Collector Type	Concentrate Amount, %	Cu		Fe		SiO_2			Ce	
		Grade %	Efficiency %	Grade %	Efficiency %	Grade %	Efficiency %	Grade %	Efficiency %	Verim, %
A407, SIPX, KAX	2.5	15.95	43.7	30.15	2.1	18.80	1.7	0.02	2.3	2.3
A208, A238, SIPX	1.6	22.71	40.4	20.99	0.9	18.30	1.0	0.03	1.8	1.8
SIPX, A412, A238	1.9	16.90	36.5	33.59	1.8	18.80	1.3	56.40	1.6	1.6
Feed	100.0	0.90		40.01		25.90		78.00		

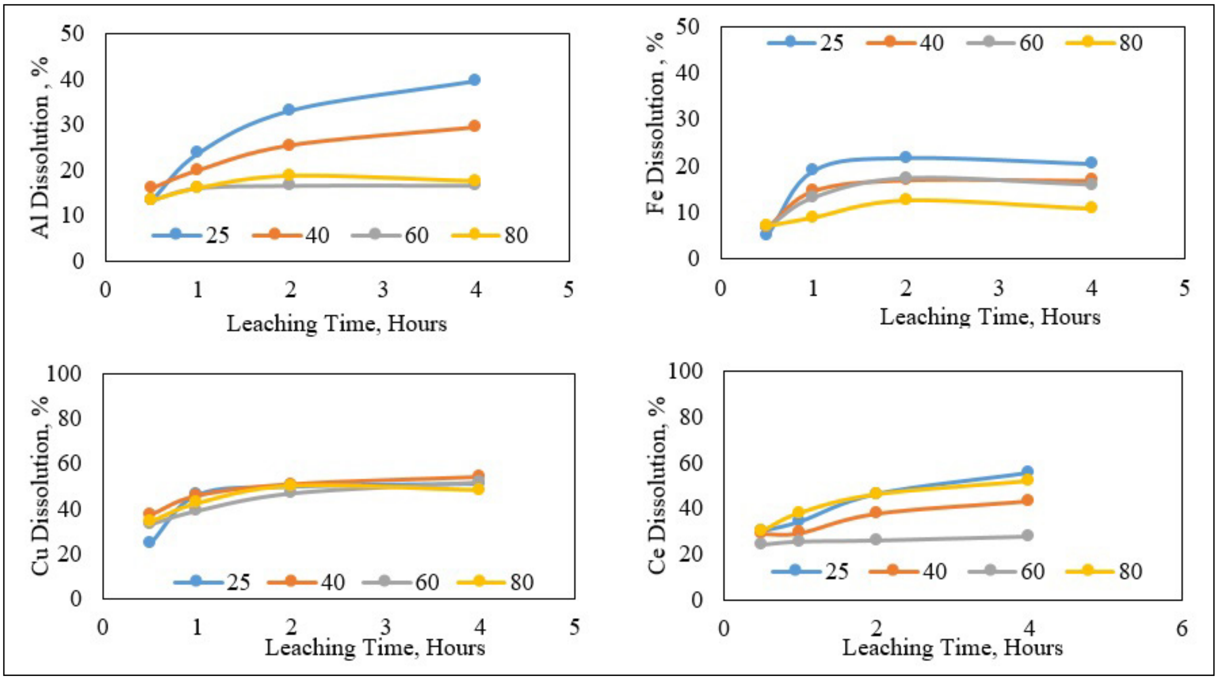

Figure 2- Correlation of duration and temperature on the dissolution efficiencies of Cu-Ce-Fe-Al, with constant solid ratio of 10% and pH value of 2.5.

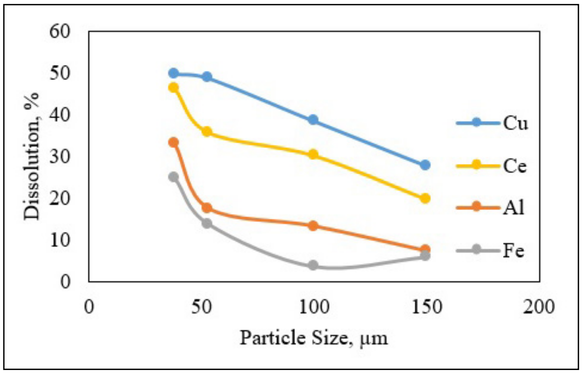

Figure 3- Effects of the particle size on dissolution rate.

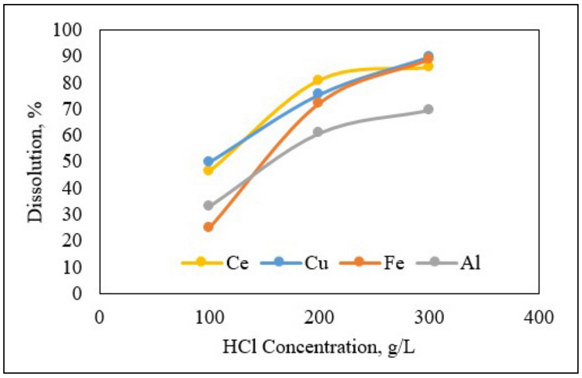

Figure 4- Effects of acid concentration on dissolution rate.

Ce dissolution efficiency, the subsequent experiments were conducted at 38 micrometers particle size as constant.

3.2.3. Effects of Acid Concentration

In order to examine the effects of the acid concentration, a series of experiments were conducted using various acid concentrations with a duration of 2 hours at 25 °C. Results of these experiments are given in Table 4.

Increasing the acid concentration from 100 g/L to 200 g/L, an overall increase in metal dissolution efficiencies was observed. Moreover, an increase to 300 g/L resulted in a considerable increase in Cu and Fe dissolution efficiencies while only causing a mild increase for Al and Ce. Although the dissolution efficiency for Al reached 69.8%, it was observed to stay relatively lower in comparison to the other metals. Dissolution efficiencies of Cu, Fe and Ce metals for 300 g/L were determined to be 89.5%, 88.7% and 85.6%, respectively (Figure 4).

Additionally, effects of high acid concentrations such as 400 g/L and 500 g/L on metal dissolution were examined. Gelling of the pulp post-leaching was observed and solid-liquid separation was not possible. Therefore, although the curves were not straightened, the most suitable acid concentration was selected as 300 g/L HCl.

3.2.4. Effects of H_2O_2 Concentration

In experiments conducted to recover valuable metals from copper slag, sufficient dissolution efficiencies for Cu and Ce metals at high acid concentrations were obtained. In addition, it was aimed to reach even higher efficiencies using a highly oxidizing agent H_2O_2. Therefore, experiments were performed using various H_2O_2 concentrations. With the results given in Figure 5, observations were made that showed an additional 25 g/L, increased Cu dissolution efficiency from 50% to 75%, Ce dissolution efficiency from 46% to 70%, Fe dissolution efficiency from 25% to 67% and Al dissolution efficiency from 33% to 60%. However, it must be noted that increasing H_2O_2 concentration to 50, 75 and 100 g/L results in no considerable increase in the metal dissolution efficiency as per Figure 2.

Dimitrijevic et al. (2017) demonstrated three separate experimental setup strategies in recovery of valuable metals from copper slags using hydrochloric acid. By only adding hydrochloric acid, the first experimental setup had only achieved 36% Cu dissolution efficiency by the end of 2 hours. As per their report, the second setup, a powerful oxidizer 3M H_2O_2 was added inside the pulp in the beginning of the experiment and 40% Cu dissolution rate was achieved. In the third and the last experiment, H_2O_2 was continuously added throughout the duration of the experiment and determined a dissolution rate of 73% was achieved by the end of the 2-hour period. In the meantime, they reported that continuous addition of H_2O_2 prevented the dissolution of iron, dissolution efficiency of which stood around 11%.

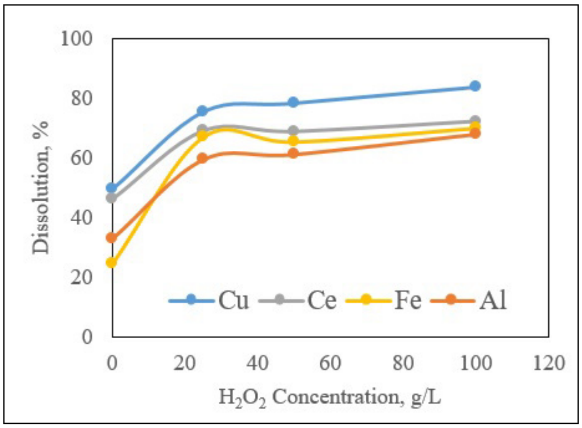

Figure 5- Effects of H_2O_2 concentration on dissolution rate.

3.2.5. Effect of Solid Ratio

Experiments were conducted in order to examine the effect of solid ratio on metal dissolution. For which, solid ratios of 10, 20, 30, 40% were used along with 300 g/L HCl and constant 25 g/L H_2O_2 concentration, at room temperature. Results obtained are given in Figure 6.

As it is seen in the Figure 7, increasing the solid concentrations has negatively impacted the dissolution of metals Fe, Cu and Ce. Fe was observed to be the most affected. Although 80-90% Cu and Ce dissolution efficiencies were reached, taking into consideration the industrial conditions, achieving 70-80% rate of dissolution seems feasible with 20-25% solid ratios. The increase in the solid ratio resulted in an observable decrease in Fe dissolution efficiency. Additionally, an observation was made that a more selective dissolution process occurred at 40% solid ratio than 10%.

Another topic that distinguishes this research from the rest of the works in literature is the amount and type of solids used. In previous studies, following sulfidation and chlorination leaching; dissolution with water (Altundoğan et al., 1997; Arslan and Arslan, 2002; Altındoğan ve Turan, 2012) or direct H_2SO_4 leaching with low solid ratios were used. (Herreros et al., 1998; Altundoğan et al., 2004;

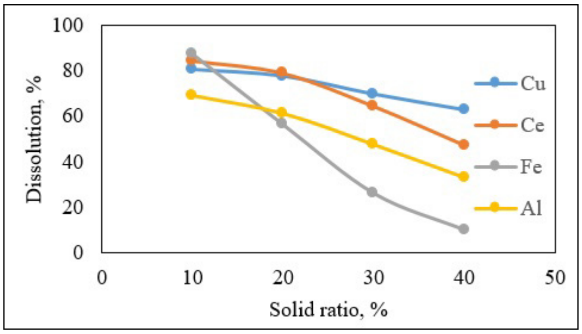

Figure 6- Effects of solid ratio on dissolution rate.

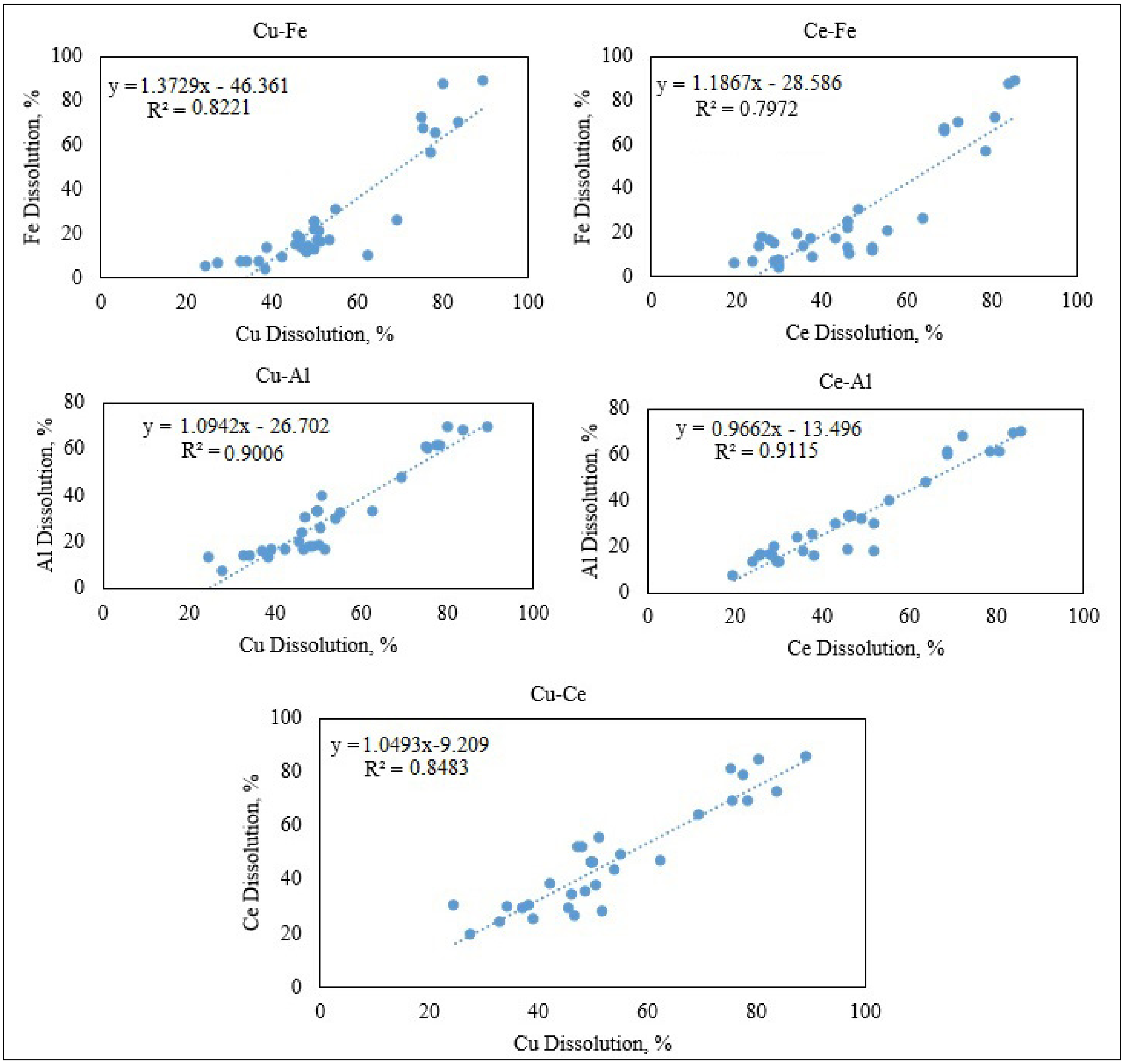

Figure 7- Correlation of Cu-Ce-Fe-Al dissolution rates.

Beşe, 2007) However in this study, due to H_2SO_4 dissolution process being physically impossible, HCl is alternatively used. Although Cu and Ce dissolution rates reach their highest values with low solid ratios, this method allows for acceptable rates of dissolution (70-80%) of metals with 20-25% solid ratios.

4. Results

In this study, mineral processing possibilities of a 0.90% Cu and 78.74 ppm Ce containing copper slag was examined with flotation and hydrometallurgical methods. Based on the results obtained, the following observations were made;

- Although a concentration at a threshold Cu value can be obtained using different origin flotation reagents as a combination, flotation rates were determined to stay below acceptable levels,

- Temperature had no effect on the dissolution efficiency and the value for Cu dissolution dropped following the increase in temperature, 2 hours being sufficient amount of time for the reaction,

- A considerable amount of increase is seen for the Ce dissolution rate upon fine grinding process albeit no considerable change in the Cu dissolution rate,

- Increasing acid concentrations led to a substantial increase in dissolution rates with 300 g/L acid addition being determined as sufficient for Ce and Al metals dissolution,

- Above 300 g/L HCl acid concentration led to gelling and obstructs solid-liquid separation.

- H_2O_2 amounts more than 25 g/L had no positive effects on metal dissolution,

- Increasing solid ratios generally led to a decrease in metal dissolution efficiencies, Fe being affected the most,

- Examination among metal dissolution rates led to the observation that Cu and Ce dissolution rates had a strong correlation (R^2= 0.91) with Al, therefore these metals were co-operating.

In conclusion, the highest Cu and Ce dissolution rates (80.5% Cu and 84.1% Ce) were achieved with leaching under the conditions of -38 µm particle size group, 300 g/L HCl concentration, 25 °C temperature, 10% solid ratio, 25 g/L H_2O_2 addition and 2 hour duration. However, it seems possible to achieve 70-80 percent dissolution rates with 20-25% solid ratios.

Additionally, although no detection was made in the XRD analysis, an assumption can be made as to Cu-Ce and Al being inside a similar mineral phase. To support this assumption, detailed mineralogical analyses must be made on run-of-mine and leach cake.

Moreover, with the flotation method, a concentrate of 23% Cu can be obtained with approximately 40% efficiency. Although the concentrate obtained is of sellable quality, low efficiency is the biggest disadvantage. Alternatively, leaching on flotation waste can be considered, which requires a detailed investigation of leaching conditions on run-of-mine samples.

Selective dissolution of metals, in comparison to other methods, is one of the most important advantages of chemical processing. At the same time, with the dissolution method the relation of metals with each other can be examined, which gives insight into their mineralogical context. This is beneficial for following the characterization on an elemental basis since mineral detection in materials such as slag waste is not possible due to the complex structures formed by the thermal processing.

All results obtained from the leaching experiments were shown as a figure. Upon examining the dissolution rates of select metals, a strong correlation is understood between the dissolution rates of Cu, Ce and Al metals. The correlation value of Cu-Al is determined to be 0.90 whereas Ce-Al is 0.91. Cu-Fe and Ce-Fe correlation values are 0.82 and 0.79 respectively which brings an assumption that Cu and Ce elements are probably with a Al containing structure rather than Fe.

In addition, high iron concentration leads to the suppression of peaks belonging to other structures in the XRD analysis. Although no Al mineral was detected during the XRD analysis, a statement about copper and cerium elements being related to an Al-containing mineral phase can be made due to the high degrees of metal dissolution correlations.

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