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KINETICS OF Pb AND Zn LEACHING FROM ZINC PLANT RESIDUE BY SODIUM HYDROXIDE

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Abstract

In the hydrometallurgical zinc production processes, important amount of hazardous solid extraction residue containing unextractable Zn and Pb is generated. Due to increasing demand of metals and the depletion of high grade natural resources, these types of wastes are gaining great importance in the metallurgical industries. In this study, selective leaching and leaching kinetics of Pb and Zn from zinc extraction residue were investigated. For this purpose; the effects of NaOH concentration, contact time, stirring speed and temperature on the Pb and Zn recovery from the residue were studied. The shrinking core model was applied to the results of the experiments. Leaching results showed that 85.55% Pb and 21.3 % Zn could be leached under the optimized conditions. The leaching of Pb and Zn were found to fit well to shrinking core model with ash layer diffusion control. Activation energy values for Pb and Zn leaching were calculated to be 13.645 and 22.59 kJ/mol, respectively.

Keywords: Zinc extraction residue; Recovery; Leaching; Shrinking core model.

1. Introduction

Zinc is one of the most widely used metals. It is mainly produced from sulphides, carbonates and partly from various secondaries and wastes containing zinc such as electric arc furnace dusts, zinc ash, zinc dross, scraps, slags by hydrometallurgical, pyrometallurgical or their combination processes [1].

The combined systems consist of roasting-leaching-electrowinning processes are employed in many countries. In these processes, ZnO-rich calcine is first produced from the concentrates and then zinc in the calcine is leached with hot sulphuric acid solution. A pregnant solution and a solid leach residue are obtained after liquid-solid separation by rotary filter. Pregnant zinc solution is purified and zinc is won by electrolysis. The leach residue is classified as a hazardous waste due to the presence of significant amounts of leachable heavy metals [2].

Zinc leach residues contain significant amounts of precious metals such as lead, silver, cadmium and unextractable zinc. Therefore, they are generally stockpiled in many plants to recover these precious metals in the future [3]. Due to increasing demand of metals and the depletion of high grade natural resources, these types of secondaries and wastes are gaining great importance in the metallurgical industries. In order to recover metallic values from the secondaries/wastes, a lot of researches have been made [3-19]. Depending on the related researches, it can be stated that the hydrometallurgical processes are the most convenient recovery techniques for the metal recovery. In the hydrometallurgical processes, different lixiviants such as sulphuric acid [5,12,16-24], hydrochloric acid [5,18,24], nitric acid [25,26], caustic soda [21,27-32], brine solution [3,33-36], ammonia, ammonium carbonate, ammonium chloride, some carboxylic acids [1] are generally used. Acidic leach solutions, caustic soda and brine solution have been found to be very effective lixiviant for the leaching of lead and zinc. Although high leaching yields can be obtained in the acidic leach solutions and brine solutions, they are not applicable due to releasing of the important amount of impurities and chloride ions, which is a disadvantage for electrowinning of zinc, into the leach solution. Caustic soda is the most suitable lixiviant in this respect for the amphoteric lead and zinc. In the caustic soda leach process, lead and zinc are selectively dissolved in sodium hydroxide solution rejecting nonamphoterics in the residue. The process has been attempted for the dissolution of zinc from different sources such as iron and steel making dusts [37,38], electric arc furnace dust [27-29], smithsonite Zn–Pb ores [30], fly ash generated from municipal incineration plants [21]. But, a detailed study on the lead and zinc recovery from the zinc extraction residue has not been made and its kinetics has not

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been investigated up to now. In this study, selective leaching, leaching kinetics of lead and zinc from zinc extraction residue with NaOH and separation both metals from the leach solution were investigated.

2. Materials and methods

2.1. Materials

The zinc extraction residue used in the study was obtained from Çinkur Plant located in Kayseri, Turkey. The chemical and mineralogical compositions of the residue were determined by Philips PW-2404 XRF and Shimadzu XRD-6000, respectively. It was dried at room temperature for ten days and then sieved to obtain particles smaller than 75 micron (-200 mesh) prior to use.

Chemical analysis shows that the zinc extraction residue contains 15.14 % Pb, 12.25 % S, 7.98 % Zn, 6.19 % Ca, 6.74 % Si, 5.44 % Fe and 1.85 % Al as major elements.

Insoluble lead sulphate generates during the leaching of ores with sulphuric acid in zinc production and remains in the extraction residue. Mineralogical analysis was verified this situation. The major mineralogical phases in the residue were determined to be gypsum [$CaSO_4.2H_2O$], anglesite [$PbSO_4$], massicot [PbO], quartz [SiO_2], maghemite [Fe_2O_3], hercynite [Al_2FeO_4] and franklinite [$ZnFe_2O_4$].

2.2. Experimental procedure

Leaching experiments were conducted in 250-ml glass flasks that were magnetically stirred. A mass of 20 g of the solid slag fraction was placed into the reactor and 100 ml of leaching solution were added to maintain the liquid/solid (L/S) ratio of 5. The temperatures of the leach solutions were thermostatically kept controlled at 25-85°C with ±0.5°C sensitivity. Stirring speed, NaOH concentration and contact time in the leaching experiments were changed in the range of 100-500 rpm, 5-30 % (w/v) and 15-360 min, respectively. Independent batch reactors were used for every time sampling.

After each leaching, the leachates were filtered, the leach residues were washed with distilled water and then the wash solutions were added to main pregnant leach solution. The solutions were chemically analyzed by Perkin Elmer AAnalyst 800 model AAS for lead and zinc. Recovery percentages of both metals were calculated according to lead and zinc contents of the leachates.

In order to separate Pb from Zn in leach solutions, chemical precipitation with sodium sulphide was applied. For this purpose, different weight ratios of sodium sulphide to the Pb used were tested to precipitate whole lead ions in the leach solution. Mineralogical compositions of the solid materials were determined by X-Ray diffractometer.

Each experiment was performed in duplicate and arithmetic averages calculated from the values varied within ±2% were taken into account.

3. Results and discussion

3.1. Effect of NaOH concentration

Zinc extraction residue contains 15.14 % Pb, 7.98 % Zn, 6.74 % Si and 1.85 % Al which can dissolve in the NaOH solution. Stoichiometric amount of NaOH to be required to dissolve these components were determined to be 0.309 g-NaOH/g-residue by taking to account the following reactions.

$$PbSO_{4(s)} + 3NaOH_{(aq)} \rightarrow [Pb(OH)_3]^-_{(aq)} + SO_4^{2-}{}_{(aq)} + 3Na^+_{(aq)} \tag{1}$$

$$ZnFe_2O_{4(s)} + 2NaOH_{(aq)} \rightarrow Na_2ZnO_{2\,(aq)} + H_2O_{(l)} + Fe_2O_{3(s)} \tag{2}$$

$$SiO_{2(s)} + 2NaOH_{(aq)} \rightarrow Na_2SiO_{3(aq)} + H_2O_{(l)} \tag{3}$$

$$Al_2O_{3(s)} + 2NaOH_{(aq)} \rightarrow 2NaAlO_{2(aq)} + H_2O_{(l)} \tag{4}$$

Some preliminary leaching experiments were performed on the basis of the a stoichiometric amount of NaOH under the conditions of liquid/solid ratio of 5, contact time of 60 min and temperature of 25°C. In these experiments, about 16 % Pb and 1 % Zn could be leached from the residue. These leaching percentages show that a stoichiometric amount of the NaOH is insufficient to extract the whole Pb and Zn in the residue. In order to obtain effective Pb and Zn leaching yields, higher stoichiometric amounts of NaOH than a stoichiometric amount (6.18 % (w/v) NaOH) were tested.

The effect of NaOH concentration on Pb and Zn leaching from the zinc extraction residue is presented in Fig. 1. It shows that the leaching yields of Pb and Zn are strongly dependent on NaOH concentration. The maximum leaching of Pb (85.55 %) is obtained when 15 % (w/v) NaOH is used. Leaching yield of Pb increases with the increasing NaOH concentration up to 15 % (w/v). But, it sharply decreases above this concentration value as high NaOH concentration increased the viscosity of the solution and reduced the diffusion rate of the ions. In the experiments carried out with the solutions having higher NaOH concentration, solid-liquid separation was difficult due to the high viscosity of the solution. Similar results have been found by Xia and Pickles (1999a) [27], Dutra et al. (2006) [38], Zhao and Stanforth (2000) [39] and Orhan, (2005) [40].

Under the investigated conditions, it has been observed that the leaching yields of Zn (maximum 21.47 %) are very poor (Fig. 1). This situation can be attributed that the Zn in the residue founds in the form of franklinite (zinc ferrite). Franklinite, which is major form of Zn in some wastes such as steel mill electric arc furnace dusts, leaching residues of roasted zinc sulphide concentrates, are refractory oxide and has low solubility [30, 41]. In some research related zinc recovery from the zinc ferrites, it has been reported that the zinc ferrites are very stable and low solubility in most alkaline solutions [21, 27, 30, 39, 42,43]. The findings are consistent with results of these related researches.

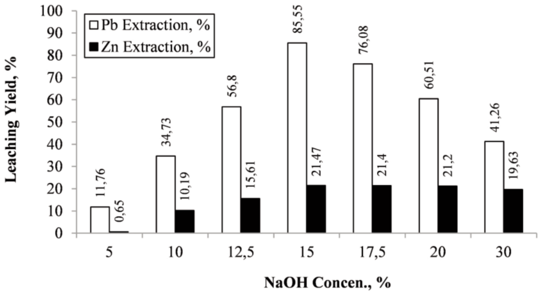

Figure 1. *Effect of NaOH concentration on the leaching of Pb and Zn from zinc extraction residue [liquid/solid ratio: 5, contact time:120 min, stirring speed: 300 rpm, leaching temperature: 25°C].*

3.2. Effect of contact time

In order to investigate the effect of contact time on Pb and Zn leaching from the zinc extraction residue, a series of leaching experiments were performed under the experimental conditions of NaOH concentration of 15 %, stirring speed of 300 rpm and temperature of 25°C. The results are presented in Fig. 2. The leaching yields increased with the increasing contact time during the first 120 min and almost remain constant thereafter. Therefore, it can be stated that the contact time of 120 min is suitable for leaching. Under these operating conditions, 85.55 % Pb and 21.47 % Zn could be leached.

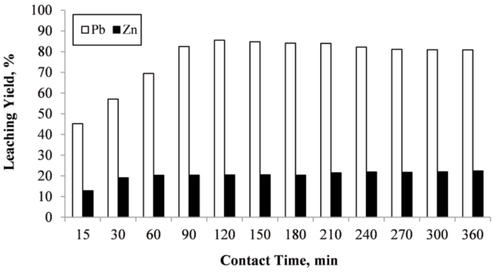

Figure 2. *Effect of contact time on the leaching of Pb and Zn from zinc extraction residue [liquid/solid ratio: 5, NaOH concen.: 15%, stirring speed: 300 rpm, leaching temperature: 25°C]*

3.3. Effect of stirring speed

The effect of stirring speed on the Pb and Zn dissolution from the zinc extraction residue was investigated at stirring speeds of 100; 200; 300; 400; 500 rpm at 25°C in the leach solution containing 15 NaOH % (w/v) for 120 min. The leaching yields of Pb and Zn increase with the increasing stirring speeds up to 300 rpm and they almost remain constant over the 300 rpm (Fig. 3). When the stirring speed was increased from 300 rpm to 500 rpm, the increases in the leaching percentages of Pb and Zn were calculated to be 1.78 % and 1.61 %, respectively. In spite of 66.6 % percentage increasing in the stirring speed, due to the low increases in the leaching yields, it can be stated that the 300 rpm is most appropriate stirring speed. Consequently, all subsequent runs were made using 300 rpm.

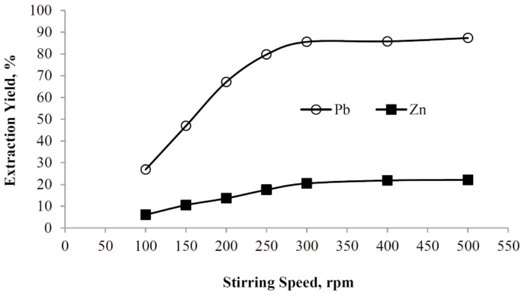

Figure 3. *Effect of stirring speed on the leaching of Pb and Zn from zinc extraction residue [liquid/solid ratio: 5, NaOH concen.: 15%, contact time: 120 min, leaching temperature: 25°C]*

3.4. Effect of temperature

Fig. 4 shows the results of experiments carried out at different temperatures in the range of 25 to 85°C. The leaching yields of Pb markedly decreased with the increasing temperature; however, the leaching yields of Zn were almost constant under the same conditions. The leaching yields of Pb decreased from 85.55 % at 25°C to 67.04 % at 85°C due to precipitation of lead. XRD analysis of the precipitates formed at high temperatures showed that the lead in the leach solution partially precipitated in the form of lead oxide hydroxide [$Pb_5O_3(OH)_4$] and wickenburgite [$CaPb_3Al_2Si_{10}O_{24}(OH)_6$]. Chen et al. have also found the leaching recovery of Pb and Cd decreased with the increase of temperature in their study [43]. In another study that it has been investigated pressure leaching of high silica Pb–Zn oxide ore in sulfuric acid medium, it has been reported that the recovery yield of Zn increased with increasing temperature, however, the recovery yields of both Fe and Pb decreased under the same condition [45]. In spite of the increase in temperature, Zn leaching yields were still low. This result reflects that alkaline leaching does not has ability to solve the zinc from the zinc ferrite. Havlik et al. have found similar result and they have reported that the alkaline leaching processes are limited, however, by their inability to recover zinc from zinc ferrite unless a reducing roast is carried out first [43].

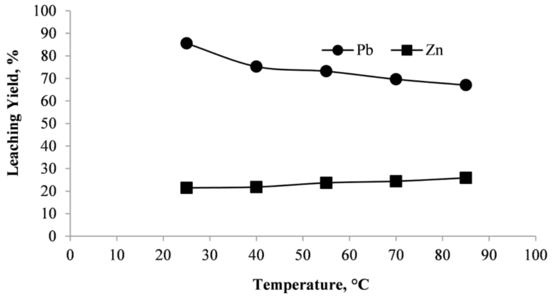

Figure 4. *Effect of temperature on the leaching of Pb and Zn from zinc extraction residue [liquid/solid ratio: 5, NaOH concentration: 15 %(w/v), contact time: 120 min, stirring speed: 300 rpm].*

3.5. Kinetic analysis

The leaching of Pb and Zn from the zinc extraction residue in the NaOH solutions includes heterogeneous reactions represented by Eqs. 1 and 2. The shrinking core model can be used to describe the leaching kinetics of the fluid-solid heterogeneous reaction system. The model considers that the leaching process is controlled either by the diffusion of reactant through the solution boundary layer, or through a solid product layer (generally called as ash layer) or by rate of the surface chemical reaction by assuming the solid particle retains its initial spherical shape. Eqs. 5-7 of the shrinking core model given in following can be used to describe the leaching kinetics when the diffusion of NaOH through the solution boundary layer, or the solid ash layer, or the surface chemical reaction are the rate-controlling step, respectively [46].

$$\alpha = 3.b.k_d.[NaOH]^n/\rho.r_o = K_d.t \tag{5}$$

$$1\text{-}3(1\text{-}\alpha)^{2/3} + 2(1\text{-}\ \alpha) = 6.b.D_{eff}.[NaOH] / \rho.r_o^2 = K_D.t \tag{6}$$

$$1\text{-}(1\text{-}\alpha)^{1/3} = b.k_s.\ [NaOH]^n/\rho.r_o = K_R.t \tag{7}$$

Where; α is the fractional conversion, b is stoichiometric coefficient, k_s is the chemical reaction rate constant, [NaOH] is the sodium hydroxide concentration, n is the reaction order with respect to NaOH, ρ is the molar density of zinc extraction residue, r_o is the initial radius of the particle, D_{eff} is the effective diffusion coefficient, K_d and K_D are the kinetic parameters for diffusion controls through the solution boundary layer and solid ash layer, respectively, K_R is the kinetic parameter for surface reaction control and t is the reaction time.

When the leaching process is controlled by diffusion through the solid ash layer, a plot of $1\text{-}3(1\text{-}\alpha)^{1/3} + 2(1\text{-}\ \alpha)$ versus time is a straight line with a slope of K_D. Similarly, if the surface chemical reaction is the rate-controlling step, a plot of $1\text{-}(1\text{-}\alpha)^{1/3}$ versus time gives a straight line with a slope of K_R [46]. In the case of high stirring speed, the diffusion through the solution boundary layer is neglected. As shown in Fig. 5, there is a good fit between the experimental data and Eqs. 6 and 7, indicating that the leaching kinetics of Pb from the zinc extraction residue indeed can be described by the shrinking core model with the ash layer diffusion control together chemical reaction. Fig. 6 shows that the experimental results obtained for the Zn leaching fit the equation of the ash layer diffusion (Eq. 6).

In order to determine the activation energy, Arrhenius equation was applied to the data obtained from each temperature for Pb and Zn leaching. According to Arrhenius equation, plot of log K versus 1/T for the K values determined from Equations 6 and 7 are straight lines where the slope is ($-E_a$ / R). Depending on the activation energy value, it can be decided that the heterogeneous reaction mechanism is

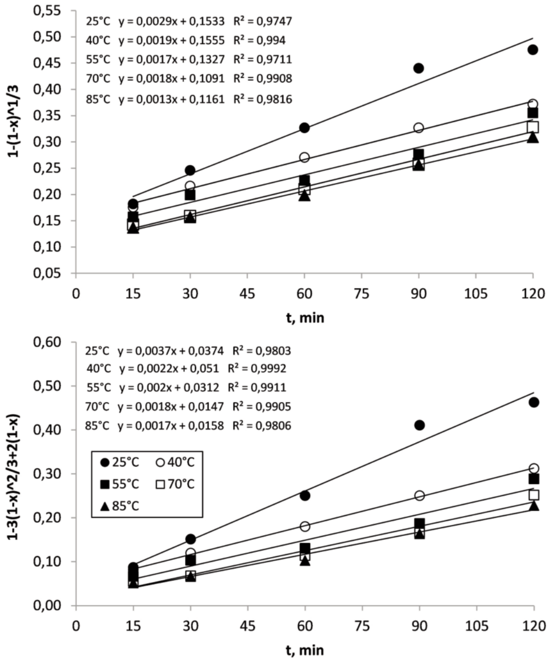

Figure 5. *Shrinking core model for leaching of Pb from zinc extraction residue at different temperatures.*

chemical or diffusion-controlled. The activation energy values higher than 40 kJ/mol usually indicate chemical reaction-controlled processes whereas the lower values than 20 kJ/mol usually indicate the diffusion-controlled processes [46, 47]. Activation energy ($-E_a$) values for Pb and Zn leaching from the zinc extraction residue were calculated to be 13.645 and 22.59 kJ/mol, respectively. Although the leaching data of Pb fit well both of Eqs. 6 and 7 (Fig. 4), depending on the activation energy values, it can be stated that the diffusion-controlled mechanisms control the Pb leaching. Xia and Pickles [27] and Terry and Monhemius [48] have been reported the similar finding.

The activation energy value obtained for Zn leaching, is close to 20 kJ/mol implied diffusion-controlled processes, confirms that the leaching mechanism is controlled by the ash layer diffusion. This finding is also consistent with those recorded by Abdel-Aal [49] and Bodas [50].

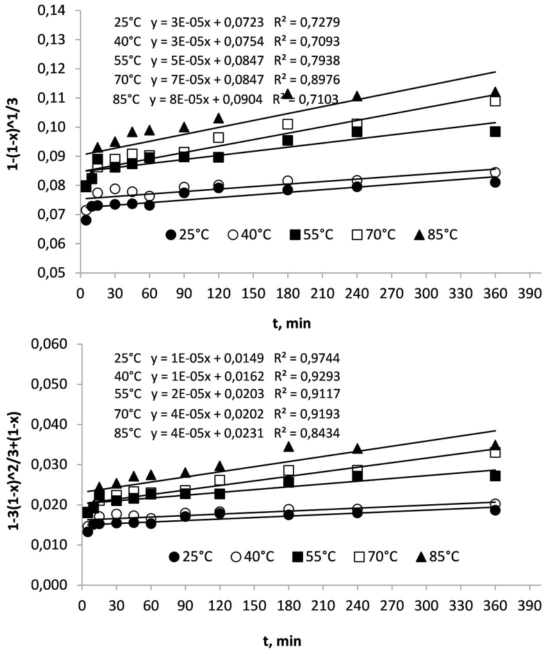

Figure 6. *Shrinking core model for leaching of Zn from zinc extraction residue at different temperatures.*

3.6. Separation of Pb from Zn in alkaline leach solution

The dissolution properties of Pb and Zn are similar due to their amphoteric properties. Therefore, both of Pb and Zn dissolve in alkaline and acidic solutions. In order to separate Pb from Zn in acidic and alkaline solutions, cementation process using Zn powder and sulphide precipitation process with sodium sulphide are usually applied, respectively [39]. In this study, Pb was precipitated selectively by addition of sodium sulphide to the leaching solution. It has been determined that 99.85 % Pb can be precipitated when the mole ratio of sodium sulphide to the Pb is over 1.50. The precipitate was identified mineralogically as mainly of galena [PbS] and minor amount of wurtzite [ZnS] by XRD analysis.

3.7. Characterization of the leach residue

The chemical and mineralogical compositions of the zinc extraction residue before and after leaching were examined. The results of XRD analysis carried out to determine mineralogical compositions are presented in Fig. 7. The XRD analysis shows that lead-containing massicot [PbO] and largely anglesite [$PbSO_4$] minerals disappear while the less soluble zinc-containing franklinite [$ZnFe_2O_4$] minerals predominate after the leaching. The chemical analysis of the NaOH leach residue is given in Table 1. The chemical analysis also confirms the results of XRD analysis. As indicated in Table 2, 15.14 % Pb and 7.98 % Zn content of the zinc extraction residue were changed to 3.1 % and 11.34 %, respectively.

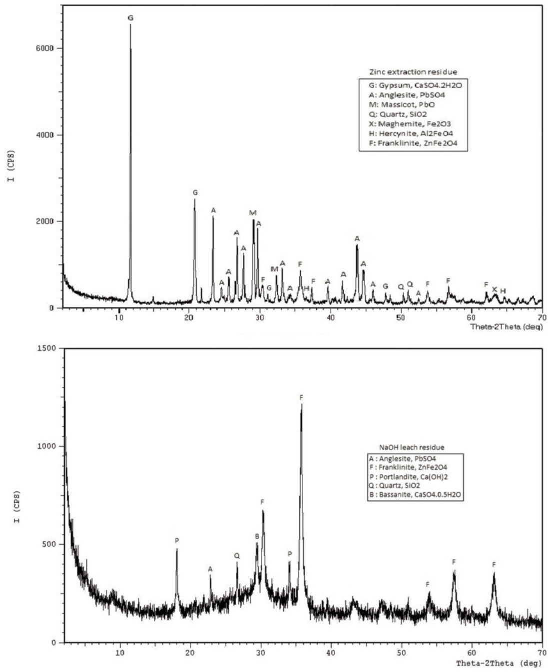

Figure 7. *XRD patterns of the zinc extraction residue before and after the NaOH leaching*

Table 1. *The chemical composition of the zinc extraction residue before and after NaOH leaching*

Sample	Pb %	Zn %	Ca %	Fe %	Al %
Before leaching	15.14	7.98	6.19	5.44	1.85
After leaching	3.1	11.34	11.91	12.43	2.92

4. Conclusions

In the present study, the leaching kinetics of Pb and Zn from the zinc extraction residue in NaOH solution was investigated by taking into consideration the parameters of NaOH concentration, contact time, stirring speed and temperature. It was found that the leaching yields of Pb and Zn were strongly dependent on NaOH concentration and temperature. The optimum leaching conditions were found to be NaOH concentration: 15 %, stirring speed: 300 rpm, leaching time: 120 min and temperature: 25°C. Under the optimized conditions, it has been determined that the satisfactory leaching yield of Pb (85.55 %) can be obtained, but, leaching of Zn is very poor (21.47 %). The leaching recoveries of Pb and Zn were repetitive and reliable.

It was determined that the dissolved concentrations of the impurities such as Fe, Al and Ca were negligible levels due to their mineralogical structure. Therefore, it can be stated that the proposed process consists of a hydrometallurgical treatment of the zinc extraction residue based on selective leaching of Pb and Zn without destroying the impurities that can not dissolve in the alkaline solutions.

The leaching of Pb and Zn were found to fit well to shrinking core model with ash layer diffusion control. Activation energy values for Pb and Zn leaching from the zinc extraction residue were calculated to be 13.645 and 22.59 kJ/mol, respectively.

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