



Selective leaching of zinc and lead from electric arc furnace dust using citrate and H₂SO₄ solutions. A kinetic perspective

Lixiviación selectiva de zinc y plomo del polvo de un horno de arco eléctrico utilizando soluciones de citrato y H₂SO₄. Una perspectiva cinética

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Abstract

The electric Arc Furnace Dust (EAFD) sample leaching using two different organic carboxylic anions has been previously studied, as a separate article. The aim of the present research work (Part II) is the study on the leaching kinetics of EAFD, comparing the efficiency of sodium citrate with that of sulfuric acid solutions. The effect of the solid / liquid ratio, temperature and reagent concentration in the leaching solutions on the metallic dissolution was analyzed. In both cases, the more stable phase of franklinite (ZnFe₂O₄) experienced minimal decomposition at room temperature, although almost complete extraction of zinc was possible with sulfuric acid at higher temperatures. The kinetics of franklinite decomposition conformed to the reaction-controlled Shrinking Core Model. Using the Arrhenius expression, the apparent activation energies for franklinite and lead dissolutions in H₂SO₄ were evaluated. On the other hand, citrate showed promise due to its selectivity in leaching non-ferrous metals oxides (ZnO and PbO).

Keywords: Kinetics; leaching; EAFD; zinc; lead; Sodium-citrate; sulfuric-acid.

Resumen

La lixiviación de muestras de polvo de horno de arco eléctrico (EAFD) utilizando dos aniones carboxílicos orgánicos diferentes se estudió previamente como un artículo separado. El objetivo del presente trabajo de investigación (Parte II) es el estudio de la cinética de lixiviación del EAFD, comparando la eficiencia del citrato de sodio con las soluciones de ácido sulfúrico. Se analizó el efecto de la relación sólido / líquido, la temperatura y la concentración de reactivo en las soluciones de lixiviación sobre la disolución metálica. En ambos casos, la fase más estable de franklinita (ZnFe₂O₄) experimentó una descomposición mínima a temperatura ambiente, aunque fue posible una extracción casi completa de zinc con ácido sulfúrico a temperaturas más altas. La cinética de la descomposición de franklinita se ajustó al modelo de núcleo retráctil controlado por reacción. Usando la expresión de Arrhenius, se evaluaron las energías de activación aparentes para las disoluciones de franklinita y plomo en H₂SO₄. Por otro lado, el citrato se mostró prometedor debido a su selectividad en la lixiviación de óxidos de metales no ferrosos (ZnO y PbO).

Palabras clave: Cinética; lixiviación; EAFD; zinc; plomo; Citrato-de-sodio; ácido-sulfúrico.

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1 Introduction

The disposal and treatment of the waste produced by smelting and refining processes are the main problems facing the steel industry around the world. The electric arc furnace, for example, generates exhaust gases that carry numerous fine particles upon exiting the furnace (Madfias, 2009). These particles are retained in the filters of the gas purification system and are known as EAFD (Electric Arc Furnace Dust). It is estimated that each year the steel industry produces between 5 and 7 million tons of EAFD worldwide (Machado *et al.*, 2006). The concern regarding their generation is that they have been classified as hazardous waste, due to their content of heavy and leachable metals such as lead and cadmium. (Bruckard *et al.*, 2005; Castells, 2009; Hagni *et al.*, 1991). However, EAFD also arouse great interest in the development of techniques for their treatment, due to the added value of their high zinc content. Therefore, several authors have proposed processes to treat this type of waste, which simultaneously mitigate their environmental impact and recover some of the valuable metal contents.

The traditional route to recover non-ferrous metals is through pyrometallurgical processes, where zinc and lead are volatilized and collected as oxides while obtaining a slag rich in iron. For these processes, temperatures between 900 to 1000 ° C are required (Gamboa, 2017). This allows the charge to be progressively heated while chemical reactions are occurring. Despite their important industrial impact, these processes require large investments and elevated energy consumption. They also generate a significant quantity of toxic gases and metals, such as copper and nickel, are lost to the slag. (De Buzin *et al.*, 2017). There are other techniques for recycling EAFD by making self-reducing briquettes from a mixture of the residue and secondary fly ash with additions of coke as a reducing agent. Through an agglomeration process with a sulfide liquor, the mixture is ground to obtain the uniform particle size for the production of briquettes; however, this grinding process tends to make the recycling method of EAFD more expensive. (Madfias, 2009).

EAFD have been used as an alternative material in the production of asphalt mixtures to repair cracks in the pavement (Sayadi & Hesami, 2017), as a secondary raw material in the production of self-compacting mortars (Lozano-Lunar *et al.*, 2019a; Lozano-Lunar *et al.*, 2019b), as a raw material in the

production of glazes for ceramic tiles (Silva *et al.*, 2019), among others. However, the authors report the need for constant vigilance both in the process and in products manufactured with EAFD additions, due to the presence of elements such as lead, cadmium, and zinc and chlorine compounds that can interfere with both processing and the quality of the products (De Buzin *et al.*, 2017).

On the other hand, hydrometallurgical metal recovery processes have been proposed, based on conventional inorganic acids and other alternatives. For zinc extraction from EAFD for example, recoveries above 60% are achieved with H₂SO₄ solutions (Oustadakis *et al.*, 2010), as well as with HCl (Baik & Fray, 2000); however, their lack of selectivity constitutes the main drawback. Since they originate from steel production, the iron content in the EAFD is high. For this reason, the leaching process should be modified to avoid difficulties during the downstream stages of electro-recovery (Torres & Lapidus, 2017). Previous studies have reported the selective capacity of some organic agents for specific metals (Gabos *et al.*, 2009; Lin *et al.*, 2006; Torres & Lapidus, 2016; Yang *et al.*, 2015), as well as the possibility of their reutilization in several leaching/electrowinning cycles, without considerable damage to their metal dissolution capacity (Torres & Lapidus, 2017).

In the first part of this research (Borda & Torres, 2021) two different organic carboxylic anions (sodium citrate and oxalic acid) compared in the selective dissolution of zinc contained in EAFD. Under ambient conditions, treatment of EAFD with sodium citrate solutions yielded better results compared to its counterpart. However, in an attempt to increase extractions, in this second part the effect of citrate leaching against a conventional inorganic agent (H₂SO₄) is studied. Likewise, this research studies the phenomena involved in the dissolution kinetics of zinc as well as lead in an attempt to achieve the selective recovery of non-ferrous metals that permit a moderate recycle capacity of this waste.

2 Experimental

2.1 Material

The previous study (Borda & Torres, 2021) revealed that the grain sizes (diameters) of the EAFD particles have a homogeneous trend. Therefore, it was not necessary to work with a specific size of material.

In addition, the mineralogical characterization showed that the presence of ferrites with a spinel structure, such as franklinite (ZnFe_2O_4) and magnetite (Fe_3O_4) were the predominant iron phases. Oxides such as zincite (ZnO), hematite (Fe_2O_3), litharge (PbO), copper oxide (Cu_2O), and silica (SiO_2) are also present in the sample (Borda & Torres, 2021).

2.2 Leaching methods and reagents

All the experiments of this research were carried out in the Hydrometallurgy Laboratory of the Universidad Autónoma Metropolitana-Iztapalapa. Sodium citrate and sulfuric acid were used for the leaching tests and their effect on metal extraction were studied. The influence of the solid:liquid (S:L) ratio on the process kinetics was analyzed. Leaching experiments were carried out in Pyrex reactors, using BDC 2002 digital electric stirrers with paddle-type propellers, speeds of 500 rpm, and ambient pressure, for 3 hours. It was found that at 500 rpm the effect of stirring speed is eliminated, ruling out possible mass transfer effects (data not shown here). The kinetic analysis was performed based on experimental data for the dissolution of zinc and lead at different temperatures; for temperature control a jacketed reactor was employed, with recirculation of ethylene glycol solution (40% v/v).

Borda & Torres (2021), compared different pH values (3, 6, and 9) for the selective leaching of zinc contained in EAFD. Based on the results, the pH of the citrate was adjusted to 3 with a dilute solution of HNO_3 . It can be deduced that the solubility of Zn in this type of waste, as in electronic waste, depends on the acidity of the extractants (García-Arreola *et al.*, 2015). The chemical reagents used in this study were analytical grade (Reactivos Química Meyer®).

Samples were taken every hour of the leach to determine metal extractions; the metallic content in the leaching liquor was quantified by microwave plasma atomic emission spectrometry (MP-AES) and by atomic absorption spectrometry (AAS, Varian SpectraAA 220fs), using lamps, standards and procedures recommended by the manufacturer. To achieve iron precipitations in the H_2SO_4 liquor, its pH was varied with 10% NaOH.

Table 1. Maximum extraction of metals (3 hours) with different proportions of S:L. Conditions: 0.5 M sodium citrate, pH 3; 500 rpm.

Extraction (%)	S:L ratio (g/L)		
	12.5	25.0	50.0
Zn	57	59	50
Pb	82	84	72
Fe	2.9	2.5	2.1

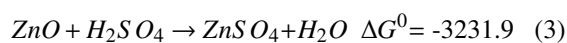
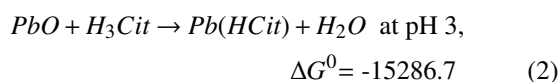
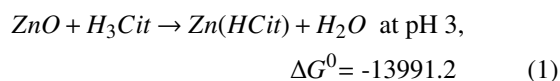
3 Results and discussion

3.1 S:L ratio effect

Metal extractions can be improved by choosing the most suitable S:L ratio for EAFD leaching. Experiments were carried out using 12, 25 and 50 g/L of citrate solution under ambient conditions. The results in table 1 show that the non-ferrous metals dissolution improved as the S:L ratio was decreased. This behavior may be expected since the fine granulometry of EAFD could produce agglomerations, reducing the contact area during leaching, in addition to the depletion of reagents. Non-ferrous metal extractions do not show significant variation when using of 12 or 25 g/L ratios. However, since it is not efficient to operate at low S /L ratios in industrial operations, 25 g/L will be used in the remaining experiments.

3.2 Leaching at Room Temperature (RT)

The extraction of zinc and lead from EAFD was studied under ambient temperature and a 0.5 M concentration for each reagent. The H_2SO_4 solution achieved better results than citrate in the extraction of zinc by approximately 16% (figure 1). However, the inorganic agent does not leach lead, due to the formation of a solid species with the ion SO_4^{2-} ; on the other hand, citrate showed its high capacity to form soluble species with this heavy metal.



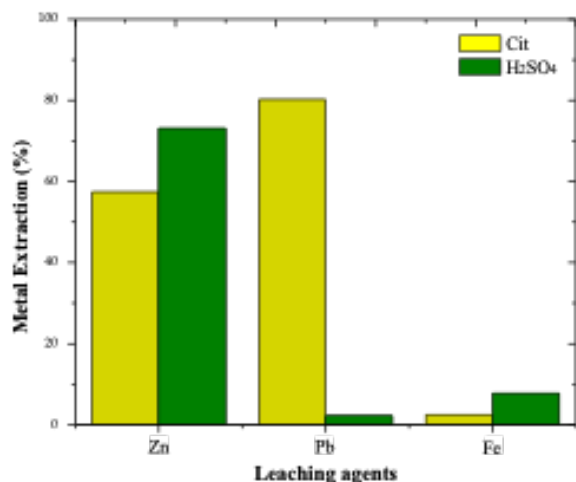
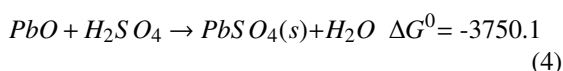


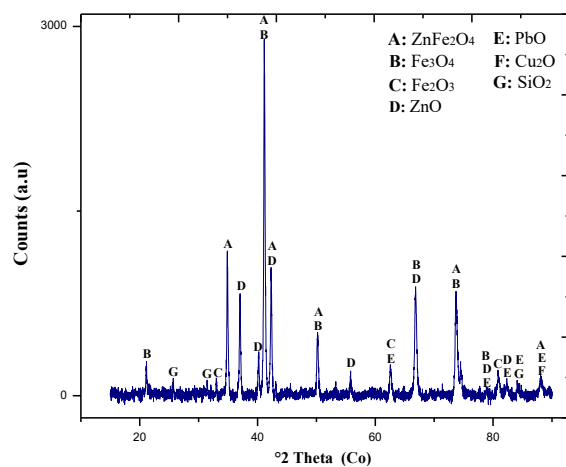
Figure 1. Leaching with Cit, H₂SO₄; Zn, Pb and Fe. Conditions: 500 rpm, 0.5 M, 25g / L, 3h; pressure and ambient temperature.



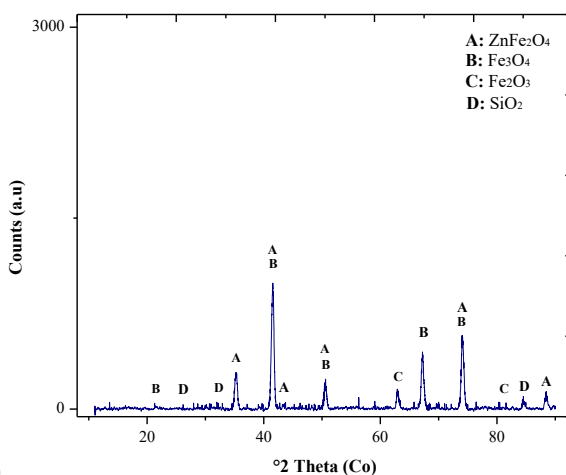
The dissolved iron content was slightly higher in the H₂SO₄ solution, although an unfavorable result for selective leaching of the non-ferrous metals.

Both systems exhibit rapid Zn leaching kinetics; the initial dissolution of ZnO results in good extractions in a short time. The presence of franklinite inhibits the total extraction of zinc from the waste. The diffraction pattern after citrate leaching (Figure 2) shows that the main difference with the pattern of the raw sample is the disappearance of oxides such as zincite and litharge, while the franklinite and magnetite ferrites remain in the solid. It is difficult to distinguish the reactivity of franklinite from that of magnetite since their XRD patterns are similar. The stability of these ferrites requires reducing conditions for their decomposition (Zhang *et al.*, 2011), although this also leads to a greater dissolution of iron. On the other hand, silica like ferrites is difficult to dissolve under these mild working conditions.

Unlike citrate, H₂SO₄ achieves a minimal decomposition of ferrites at room temperature; this is reflected in the increased extractions of iron and zinc. Franklinite dissolution is attractive because it improves zinc recovery; however, the H₂SO₄ solution has the disadvantage of not removing the lead contained in the EAFD, leaving a toxic residue after leaching. On the other hand, despite the dissolution of 80% of the lead content, the mild citrate conditions alone do not have the capacity to decompose the



a)



b)

Figure 2. Diffractogram of EAFD. (A) Sample as received, (B) sample after citrate leaching at room temperature.

spinel, losing approximately 40% of zinc when only the ZnO is leached (Figure 3)

3.3 Effect of reagents concentration

The effect of citrate or sulfuric acid concentration was studied: 0.15, 0.25, and 0.5 M at ambient pressure and temperature and 500 rpm. Although as the molar concentration increases, a slight rise in metal extraction may be noted (Figure 3), this is not directly proportional to the variation in the concentration of the agents.

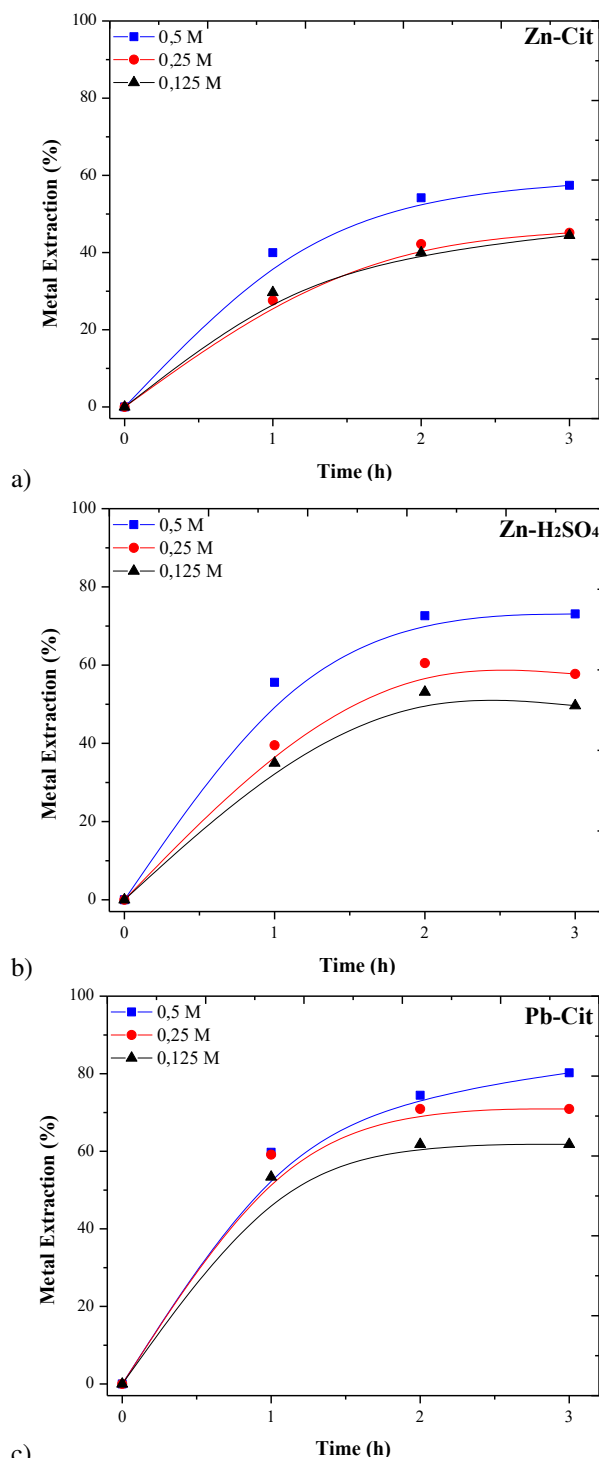


Figure 3. Effect of the citrate and H₂SO₄ concentrations on metal leaching. Conditions: 500 rpm, 25g / L, ambient temperature and pressure.

Low zinc recoveries can be attributed to insufficient H₃Cit and acid in the solution (reagent

depletion); once they are consumed after the second hour, the reaction basically stops. This explains the behavior observed using concentrations of 0.125 and 0.25 M; the maximum metal extraction is 45% for citrate and 45-50% for H₂SO₄ approximately. A similar case occurs with lead, probably influenced by the pH changes during leaching due to the consumption of citrate and H⁺ ions, extraction at the lowest concentrations tends to stop after the second hour. However, the H₃Cit and acid amount in the solution should be investigated to better assert and understand the effect of molar concentration on the process

3.4 Effect of temperature on metal extraction

The influence of temperature was studied to clarify the leaching kinetics in each case. The reaction rate varies widely with temperature (Avery, 2002; Levenspiel, 1972). The temperature range used was chosen based on the citrate efficiency proven in previous studies (Torres *et al.*, 2018), where it was found that at temperatures above 60 °C, final extractions were lower than those observed at room temperature. The results show that the variation in temperature causes different behavior for each reagent tested. With respect to citrate leaching, zinc and lead dissolution improve up to 30°C; at higher temperatures, the extraction stabilizes or even declines; some authors have reported that citrate degrades easily and is not an efficient complexing agent at temperatures above 30°C (Blanco *et al.*, 2012) and more so, in the presence of oxidizing agents (Torres *et al.*, 2018) since these are also unstable when they are heated (Pinna *et al.*, 2019). On the other hand, for sulfuric acid (Figure 4A), all metal extractions increase with temperature, although lead dissolution is still negligible.

The large fraction of zinc recovered at 50°C (96%) is attractive, due to decomposition suffered by franklinite at high temperatures (Figure 5). The main disadvantage is the collateral dissolution of iron (almost 40%), due to the substitution process for high acid activity and temperature of the spinels in each case, especially that of franklinite. Approximately 60% of iron remains in the residue in the form of magnetite.

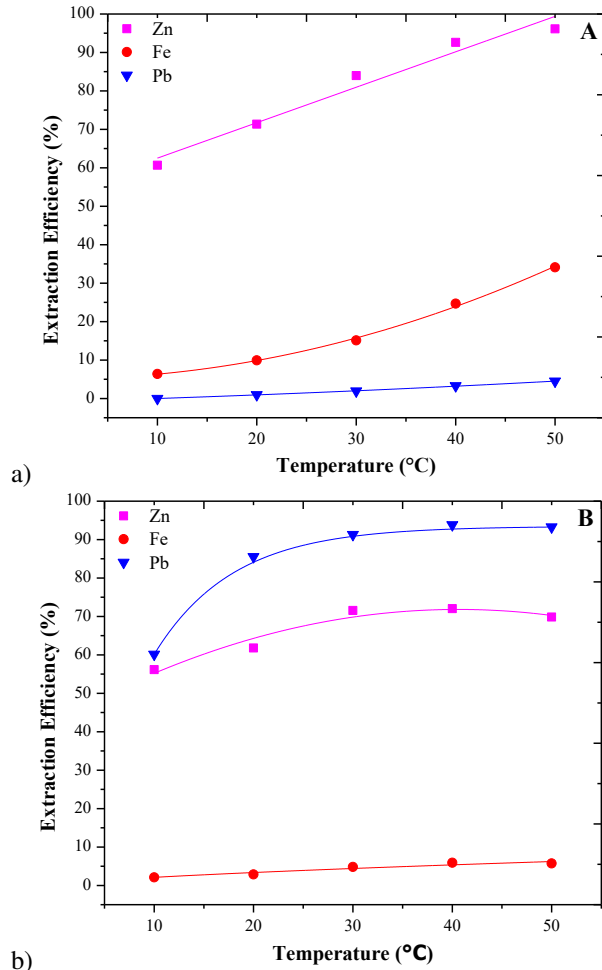


Figure 4. Effect of temperature on metal extraction efficiency. (Conditions H₂SO₄ (A) and citrate (B) 0.5 M; 500 rpm; 25g / L; 3 hours.

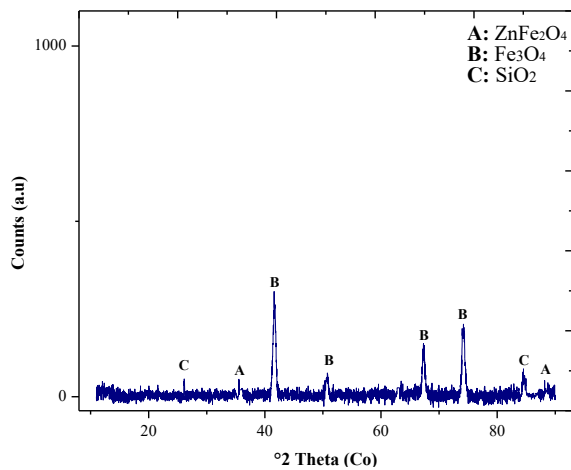


Figure 5. Diffractogram of EAFD after H₂SO₄ leaching at 50°C.

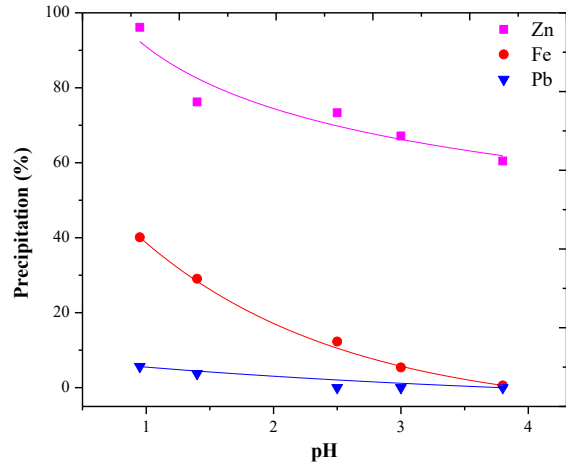


Figure 6. Leaching liquor precipitation by pH modification. (Conditions: 0.5 M H₂SO₄ liquor, with additions of a 10%wt. NaOH solution).

The elevated extraction of iron obliges a subsequent treatment of the leaching liquor. However, the aim to achieve a zinc-rich liquor through a pH adjustment was not successful since when the iron is completely removed (pH 3.8), 30% of the zinc coprecipitates (figure 6).

With the increase in the leaching temperature, the energy available for atomic and molecular collisions is greater, thus improving the rate of a chemical reaction; however, at the same time, the problem of low selectivity of sulfuric acid is more problematic. The treatment of the liquor results inefficient. Lead remains as a precipitate, and the amount of zinc in the solution is the same as that obtained at room temperature. The experiments for the iron removal were also carried out by testing its oxidation with ozone (to convert Fe²⁺ to Fe³⁺); however, the results were not favorable (data not included).

3.5 Extraction kinetic analysis

The experimental results were adjusted to the reaction-controlled SCM kinetic model (Shrinking Core Model). This model considers that the reaction occurs from the surface to the inner particle forming a core of unreacted material (spinel), leaving in its place a porous layer of inert solids (Levenspiel, 1999; Sohn & Wadsworth, 1979).

The test results are applicable to this model due to the slow decomposition of the franklinite in sulfuric acid. For this, the expression was followed:

$$\frac{t}{\tau} = 1 - r_c/R_p = 1 - (1 - X_B)1/3 \quad (5)$$

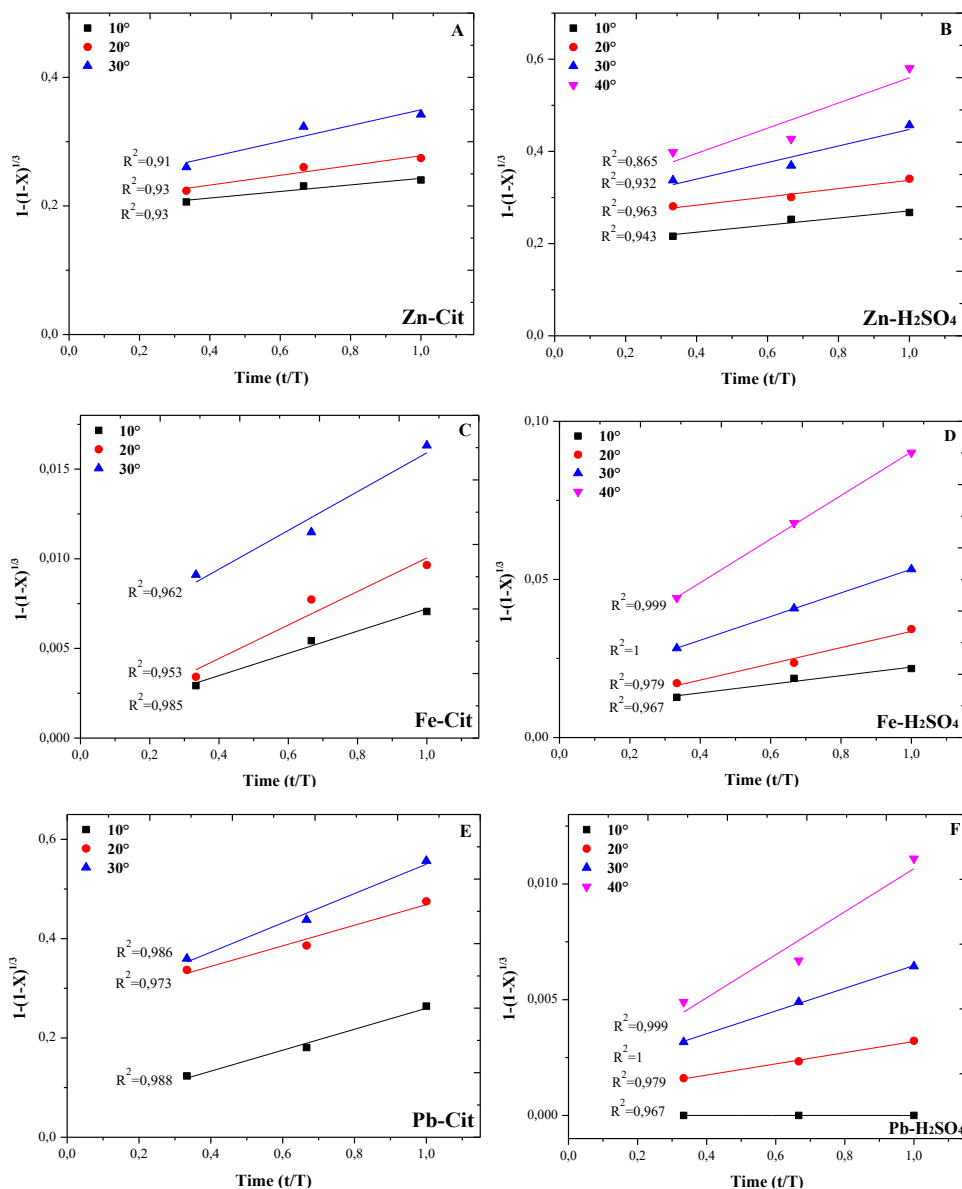


Figure 7. Data adjusted to the SCM. A (Zn-Cit), B (Zn-H₂SO₄), C (Fe-Cit), D (Fe- H₂SO₄), E (Pb-Cit), F (Pb-H₂SO₄). Conditions: Citrate and H₂SO₄ 0.5 M; 500 rpm; 25g EAFLD/L.

where,

- t = Time
- τ = Total test time
- R_p = Original particle radius
- r_c = Radius of unreacted core (reaction surface) at any time, t
- X = Fractional conversion of phase, in this case franklinite

Figure 7 shows the experimental data plotted in terms of equation (5): $1 - (1 - X)^{1/3}$ versus

dimensionless leaching time (t/τ) for different temperatures; as may be observed, the kinetic behavior of the leaching for both reagents is different. The linear adjustment of the results to the chosen model (SCM) do not pass through the origin as predicted; this is because zinc extraction combines the dissolution from two phases: the first corresponding to the spontaneous leaching of ZnO (approximately 60% of zinc), while the second represents the slow decomposition of franklinite, affecting the overall kinetics of the process.

The spinel decomposition is also reflected in the behavior of iron leaching, since only this phase of iron is leached under these conditions. Regarding lead, its extraction is nearly negligible due to its low solubility in sulfuric acid solutions.

As has been shown, citrate extracts zinc from the ZnO phase, which is almost spontaneous, however, franklinite decomposition only occurs slowly; this is confirmed in the iron plot. Furthermore, the kinetic model only applies to temperatures up to 30°C, because of the citrate destruction at higher temperatures. Lead is present as PbO, which is easily dissolved in the citrate.

To finalize the analysis of the leaching kinetics regarding the temperature effect, the apparent activation energy of each M-Cit and M-H₂SO₄ system was calculated, using the k value obtained in the adjustment based on the equation from Arrhenius:

$$k(T) = A \exp\left(\frac{E_a}{RT}\right) \quad (6)$$

Where, $k(T)$ represents the temperature dependent kinetic coefficient, A_e is the collision frequency factor, E_a indicates the apparent activation energy of the leaching reaction, R is the universal gas constant, and T is the leaching temperature in K. The logarithm of the kinetic rate constant ($\ln k$) versus $1/T$ shows a linear relationship (figure 8). The apparent activation energies, calculated from the slope were found to be 34, 25, and 20 kJ / mol for zinc, lead, and iron, respectively, in citrate solution and 33, 51, and 39 kJ / mol in sulfuric acid. In the sulfuric acid case, the similarity between the iron and zinc extraction slopes are consistent with the slow decomposition of the spinel. The activation energies are consistent with a reaction-controlled process.

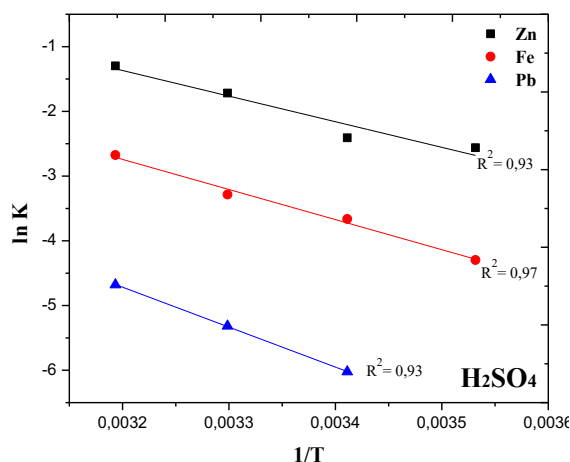
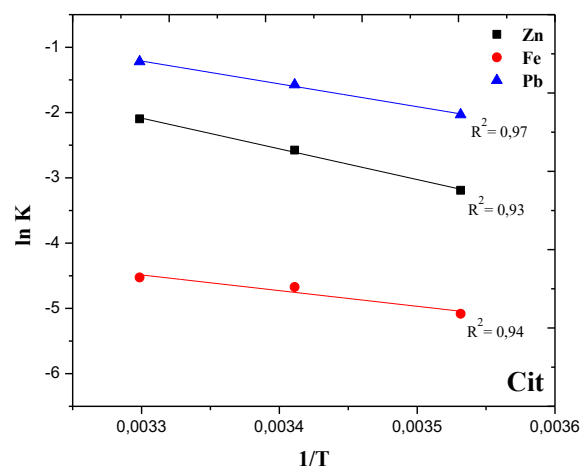


Figure 8. Arrhenius diagram for Zn and Pb leaching. Conditions: Citrate 0.5 M; 500 rpm; 25g EAFD/L.

Conclusions

In this study, it was proven that the dissolution of the zincite phase (ZnO) is rapid with both the citrate and the sulfuric acid solutions, even at ambient temperatures. Citrate manages to remove the lead contained in the EAFD, to yield a residual product of the process with non-toxic characteristics. However, since the franklinite phase (ZnFe₂O₄) does not decompose under these mild working conditions, approximately 40% of the zinc remains in this residue after the zincite dissolution. Nonetheless, because of this property, the pregnant leach solution is not plagued by the presence of iron.

On the other hand, sulfuric acid promotes quantitative zinc extraction by achieving the decomposition of franklinite, which consequently leads to a greater dissolution of iron. The experimental data comply with the chemical reaction controlled shrinking core model for the decomposition of franklinite; additionally, this mechanism is confirmed by the calculated activation energies for both zinc and iron.

For improved EAFD management, it is convenient to extract zinc from both zincite and franklinite phases using sulfuric acid solutions at moderate temperatures (>40°C), to separate out the iron from the pregnant leach solution and, subsequently, detoxify the residue by leaching the lead using citrate solutions. Subsequent studies will be necessary to achieve iron precipitation without affecting the dissolved zinc content.

Acknowledgments

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