Evaluation of ammonium citrate tribasic as a leaching reagent for lead-bearing ore

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Abstract: In this study, the leaching process of a lead-bearing ore, consisting mainly of cerussite, in an ammonium citrate tribasic medium was investigated. The parameters including temperature, reagent concentration, particle size, and stirring speed were examined. During leaching process, the lead conversion rate increased with an increase in reagent concentration, reaction temperature, and stirring speed, and a decrease in particle size. The results show that about 95% of lead content was extracted from the samples with particle size range of +75-96 µm after 21 min leaching in 1.25 mol/L ammonium citrate tribasic solution at 800 r/min and 40°C. It was found that the leaching reaction followed the shrinking core model. The results indicated that ammonium citrate tribasic could be used as an effective leaching reagent for extracting lead from lead oxide ore.

Keywords: lead, cerussite, ammonium citrate tribasic, leaching, kinetics

1.   Introduction

Lead, as an important nonferrous metal, is widely applied in the chemical and energy industries, owing to its attractive physical and chemical properties (Nowak et al. 2007, Schröder-Wolthoorn et al. 2008, Badanoiu et al. 2013). Lead is mainly derived from lead-bearing minerals such as galena, cerussite, and anglesite, and from discarded materials containing lead, such as lead-acid batteries and smelting slags (Nikolić et al. 1996, Mikhlin et al. 2004, Yuan et al. 2012). Lead oxide ore originates from the oxidization zone of sulfide ore deposits. Elemental sulfide in sulfide ore deposits is oxidized to sulfuric acid when the sulfide ore weathers. Subsequently, soluble sulfate is formed when sulfuric acid combines with metal cations. The original sulfide ore is decomposed and destroyed gradually to lead carbonate and sulfate minerals. Cerussite ($PbCO_3$) is insoluble and stable as anglesite in the oxidation zone. Anglesite ($PbSO_4$) is a mineral of secondary origin, and is formed by the oxidation of galena in the upper parts of mineral lodes where these have been affected by the weathering processes. Anglesite can be replaced directly by cerussite, especially in a carbonate environment (Sheng, 2013). After a long period of time, various ions interact to form a variety of lead-bearing minerals and gangue minerals. Dolomite, calcite, and silica are included in these ores, which makes it difficult to concentrate and extract the valuable metals through common methods of mineral processing (Wu et al. 2014, Mao et al. 2016, Wu et al. 2016, Deng et al. 2017).

Various reagents have been used in the hydrometallurgical processes of lead, including inorganic and organic acids, sodium hydroxide, and chloride solutions (Wu et al. 2014, Wu et al. 2015, Wu et al. 2016, Han et al. 2017). For acidic leaching, the dissolved lead is associated with other metals such as Fe, Ca, Mg etc., which results in high acid consumption (Qin et al. 2007, Liu et al. 2012, Han et al. 2015). In

the acid leaching processing, the silica gel will be formed due to the existence of the silicon oxide, the formation of the silica gel usually causes the filtration difficultly (Li et al. 2018). Nagib et al. (2000) compared acid leaching with alkaline leaching, and found that alkaline leaching has advantages when recovering lead from fly ash generated from municipal incineration plants. In alkaline leaching, impurities, such as Si, remain in the solid residue, which facilitates the purification process. Zhao et al. (2000) found that over 85% of zinc and lead could be extracted using a cost-effective alkaline leaching process through the addition of sodium sulfide, which produced less than 10% Al impurities. Similarly, Liu et al. (2011) developed an alkaline leaching process for the lead concentrates. Feng et al. (2015) studied the dissolution of the cerussite mineral in sodium hydroxide solutions. Recently, most investigations find that lead can be separated from other coexisting impurities such as the Fe, Ca and the Mg, because of their complexation ability with ammonia in the ammonia leaching system (Sun et al. 2015). Therefore, ammonia leaching process has considerable attention in recent years owing to the advantages of these approaches (Bingöl et al. 2005, Wang et al. 2008, Moradi et al. 2011).

Ammonium citrate tribasic $(NH_4)_3C_6H_5O_7$ is a white crystalline powder, which is soluble in water and acid, but difficult to dissolve in ethanol, ether, and acetone. The molecular weight of Ammonium citrate tribasic is 243.22 g/mol. This reagent has a spherical shape owing to the arrangement of the ammonium groups, as shown below:
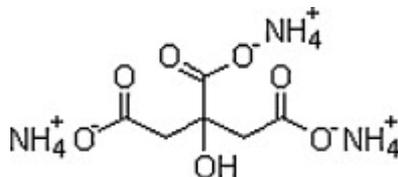
Some studies have indicated that ammonia leaching is a favorable method for extracting lead and zinc from various lead-zinc oxide ore (Meng et al. 1996, Takaoka et al. 2002, Hiroyoshi et al. 2004, Okada et al. 2007). Okada et al. (2007) compared the effectiveness of three kinds of chemical leaching methods (1) acid leaching, (2) alkaline leaching, and (3) ammonia/chloride leaching for leaching Pb and Zn from Melting Furnace Fly Ash (MFA), and noted that alkaline and ammonia/chloride leaching methods were more effective than acid leaching. Takaoka et al. (2002) studied chemical leaching for the compound form of Pb and Zn using CH_3COONH_4. Hiroyoshi et al. (2004) proposed ammonia/chloride leaching, which can leach both metals at the same time by the complex forming reaction of ammonia and chloride. However, no information on ammonia leaching of cerussite using ammonium citrate tribasic has been reported to date. Therefore, in the current study we aimed to investigate the recovery of lead-bearing ore, consisting mainly of cerussite, using ammonium citrate tribasic as a leaching reagent. The effects of several parameters, including temperature, ammonium citrate concentration, particle size, and stirring speed, on lead extraction were investigated and the leaching conditions were optimized.

2. Materials and methods

2.1. Materials

Raw materials were taken from the lead oxide ore belt of Yunnan Chihong Zn&Ge Co., Ltd. The ore with a relatively high grade of lead was selected and then was crushed and sieved. The chemical analysis and phase analysis of lead minerals were performed. The results are shown in Figure 1 and Table 1 respectively.

The results showed that both the Pb content and the SiO_2 content in the sample exceeded 25%. The main lead-bearing mineral in this sample is cerussite, and other lead-bearing minerals such as anglesite and lead sulphide minerals are less. On one hand, cerussite is easily dissolved in the acid solution or the solvent solution containing amine groups; on the other hand, anglesite and lead sulphide are not easily dissolved in these solutions. Therefore, the hydrometallurgy is a suitable method for such lead-bearing ore.

SEM-EDS are performed in a Philip XL30 scanning electron microscope. The solvent used in the experiment was twice distilled water, the ammonium citrate tribasic was analytically pure (China Tianjin Zhiyuan chemical reagent co. Ltd).
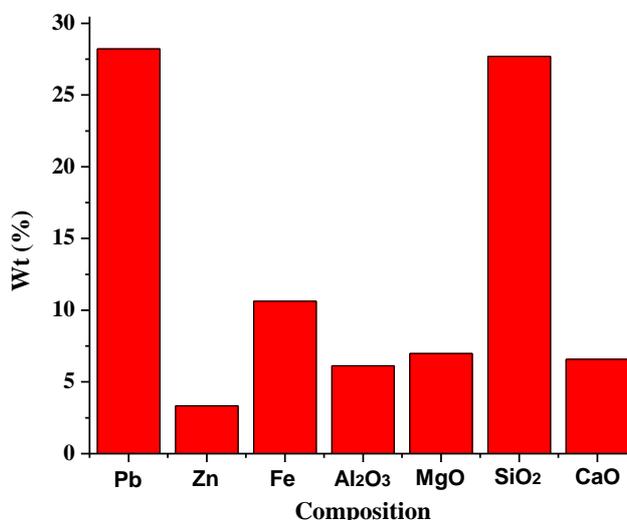
Fig. 1. Chemical contents of main compositions in cerussite-based lead oxide ore

Table 1. Phase analysis of lead minerals

Minerals	Pb grade in various phases/wt%	Proportion of lead in each phase to total lead/%
Lead carbonate	25.13	89.11
Lead sulfide	0.69	2.45
Lead sulfate and others	2.38	8.44
Total Pb	28.20	100.00

2.2. Procedure

A three-necked flask was placed in a thermostatic water bath and a Graham condenser was fitted to one of the necks to condense steam formed during the experiment. A mechanical stirrer was fitted to the second neck to stir the solution, the third neck was used to pipette the solution for analysis, and sealed with a ground glass stopper when not in use. The water bath temperature and the stirring speed could be adjusted. After adjusting the temperature of the solution in the water bath, 5 g samples and 1000 mL of deionized water were added to the three neck-flask for the leaching experiments. The experiments examined the effects of temperature, reagent concentration, stirring speed, and the particle size on conversion rate. During the experiment, 5 mL portions of solution were taken and loaded into sealed medicine bottles. Inductively coupled plasma atomic emission spectroscopy (ICP-AES, ICP-1000 II Japan), was used to test the concentration of lead in the solution and to calculate the conversion rate x. The fraction of dissolved lead (x) was calculated as follows: x= (amount of lead passing to the solution) / (amount of lead in the raw ore).

The effect of parameters on the dissolution process was investigated using the values given in Table 2 for each parameter. In the experiments, while the effect of one parameter was studied, the values of the other parameters shown with asterisks in Table 2 were kept constant. The setting of the parameters are based on parameter values and leaching conversion rates involved in previous studies on the dissolution kinetics of cerusite (Feng, 2015).

Table 2. Parameters and their range used on the experiments

Parameters	Values
Stirring speed (r/min)	200, 400, 600, 800*, 1000
Reaction temperature (°C)	20, 30, 40*, 50, 60
Initial reagent concentration (mol/L)	0.50, 0.75, 1.00, 1.25*, 1.50
Initial particle diameter (µm)	(-380+250), (-250+150), (-150+96), (-96+75)*, -75

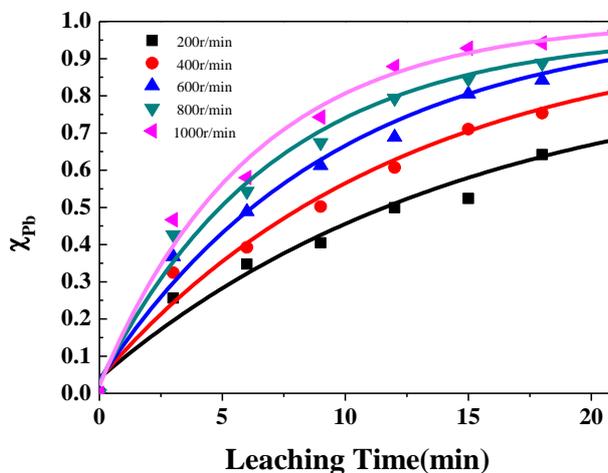*The constant values used when the effect of other parameters was investigated.

Fig. 2. Effect of stirring speed on the Pb extraction

3.   Results and discussion

3.1. Effect of stirring speed

First we examined the effect of the stirring speed on the dissolution of the lead-bearing minerals. We used speeds of 200, 400, 600, 800, and 1000 r/min. In the experiments, the concentration of ammonium citrate tribasic, the size of ore particles, and the reaction temperature were maintained at 1.25 mol/L, +75–96 µm, and 40°C, respectively. The results are shown in Figure 2. The Pb extraction rate increased as the stirring speed was increased.

3.2. Effect of temperature

To determine the effect of reaction temperature on the leaching of the lead-bearing minerals, the experiments were performed at temperatures of 20, 30, 40, 50, and 55 °C. In these tests, the reagent concentration, particle size, and stirring speed were kept constant at 1.25 mol/L, +75–96 µm, and 800 r/min, respectively. The results plotted in Figure 3 show that the conversion rate increased considerably with increasing reaction temperature. At a reaction temperature of 20 °C, about 20% of lead was leached after 6 min of reaction, whereas at a temperature of 60 °C, about 83% of lead was extracted after the same period of time. The results obtained from the temperature tests indicate that the reaction temperature has a significant effect on the extraction of lead from the lead-bearing ore.

3.3. Effect of reagent concentration

When the ore size was +75–96 µm, the temperature was 40°C and the stirring speed was 800 r/min, the leaching time was 21 min. The conversion rates of lead with different reagent concentrations are shown in Fig 4. These experimental results show that the Pb extraction rate increased as the amount of ammonium citrate was increased. When the ammonium citrate was 1.50 mol/L, more than 90% of the lead was leached within 21 min. Therefore, the concentration of ammonium citrate had a considerable effect on the conversion rate.

3.4. Effect of particle size

The effect of particle size on the leaching of lead from lead-bearing ore was examined by using fractions of the sample with particle size of (-380+250), (-250+150), (-150+96), (-96+75) and -75 µm. In these experiments, the values of other parameters were kept constant at 1.25 mol/L, 40 °C, and 800 r/min. Figure 5 shows the effect of particle size on the leaching rate of lead from lead-bearing ore. It is understood that the dissolution rate increases with decreasing particle size. After 21 min of leaching, it was found that the lead extraction from ore increased from 78% to 98% when particle size of ore reduced from (-380+250) to 75 µm. It is clear from these results that the amount of lead extracted from ore increase with decreasing particle size.
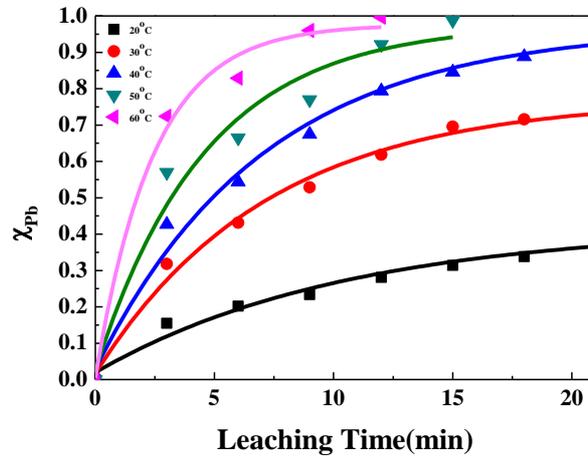
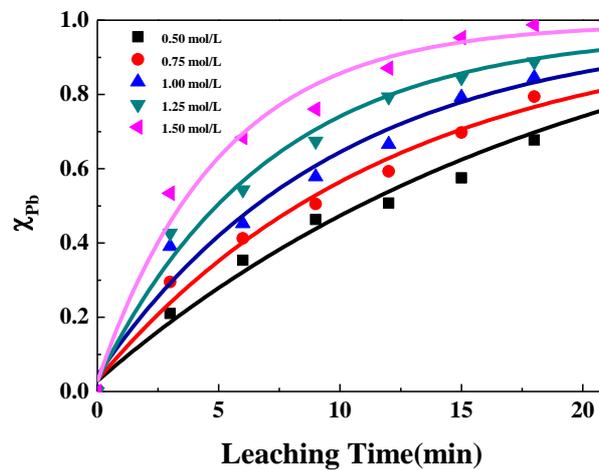Fig. 3. Effect of temperature on the Pb extraction

Fig. 4. Effect of ammonium citrate concentration on the Pb extraction

3.5. Kinetic analysis

During the leaching process, a stable complex may be formed by complexation reaction between ammonium citrate tribasic and lead ions, thereby ammonium citrate tribasic can be as an alternative leaching reagent for lead leaching. There may exist many reaction equations in the aqueous solution, which need to be further confirmed and deeply researched in future. The total reaction equation of
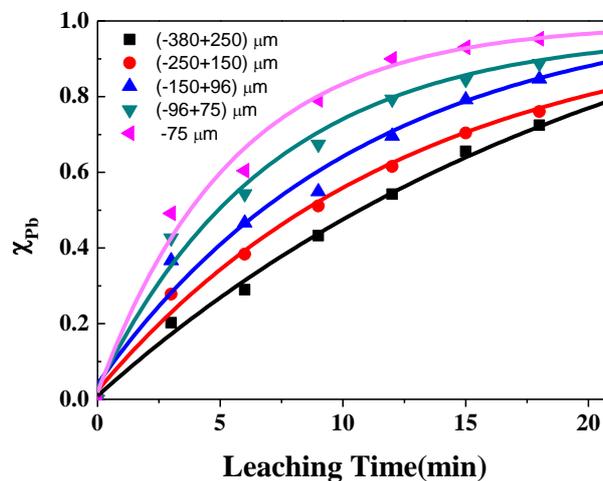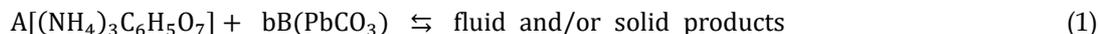

Fig. 5. Effect of ore particle size on the Pb extraction

leaching process can be represented by:

$$A[(NH_4)_3C_6H_5O_7] + bB(PbCO_3) \leftrightarrows \text{fluid and/or solid products} \tag{1}$$

where A, B, and b represent the reagent, the solid undergoing leaching, and stoichiometric coefficient, respectively (Künkül, et al. 2013). The dissolution of minerals in reagent solution is a liquid-solid heterogeneous reaction (Hua 2004, Deng et al. 2017). In generally, the kinetic analysis of reactions is performed by non-catalytic heterogeneous reaction models and the kinetics of leaching reactions is often described by the shrinking core model. According to the shrinking core model, it is thought that the reactions between solid and fluid reactants take place on the outer surface of solid particle. The solid reactant is initially surrounded by a fluid film through in which mass transfer occurs between the solid and the bulk fluid (Künkül, et al. 2013). As the reaction proceeds, the unreacted core of the solid shrinks towards the center of the solid, and a porous product layer forms around the unreacted core (Künkül, et al. 2013).

In this model, the rate of reaction between the solid particle and leaching reagent may be controlled by one of the following steps: diffusion through the fluid film, diffusion through the product layer, or the chemical reaction at the surface of unreacted (Levenspiel, 1972, Künkül, et al. 2013). The related rate equations of these steps can be written as follows:

$$x = k_l t \tag{2}$$

$$1 - 3(1-x)^{2/3} + 2(1-x) = k_d\ t \tag{3}$$

$$1 - (1-x)^{1/3} = k_r\ t \tag{4}$$

where, x is the conversion rate of solid particle, k_l is the apparent rate constant for diffusion through the fluid film, k_d is the apparent rate constant for diffusion through the product layer, k_r is the apparent rate constant for the surface chemical reaction, and t is the reaction time (Künkül, et al. 2013). That is to say equations (2), (3) and (4) refer to the film diffusion control, the product layer diffusion control and the chemical reaction control respectively.

The lead-bearing ore in this study is mainly composed of cerussite, lead sulfate and quartz etc. After leached by ammonium citrate tribasic, the minerals dissolve into the solution. Quartz and other insoluble substances form the leaching residues. Figure 6 shows the SEM-EDS analysis results of samples before and after leaching. The surface of the ore particles before leaching is smooth with distinct edges and corners; after leaching, the surface is rough with edges and corners eroded away, forming cavity. The EDS results before and after leaching show the overall changes of sample during leaching process. In addition, the dissolution rate increased with increasing stirring speed (see Figure 2). If the dissolution were controlled by the chemical reaction, the stirring speed would not have much effect on the dissolution rate.

Therefore, the dissolution process in this work is thought to be a product layer diffusion control process. Hence, we attempted to use equation (3) to describe the leaching behavior of this experiment.

According to equation (3) the relationship curve between $1 - 3(1-x)^{2/3} + 2(1-x)$ and time t at different temperatures is shown in Figure 7(a). The correlation coefficient (R^2) between the fitting curve and the results was greater than 0.90. This the relation $1 - 3(1-x)^{2/3} + 2(1-x)$ shows a linear relationship with time t. Further analysis taking into account the reactions between lead-bearing ore and ammonium citrate tribasic showed that the process is expressed with product layer (or ash layer) diffusion controlled. Therefore, we constructed a semiempirical model as follows:

$$1 - 3(1-x)^{2/3} + 2(1-x) = [k_0 \cdot (C_{Ammonium\ citrate\ tribasic})^\alpha \cdot (P)^\beta \cdot (r)^\gamma\ \ \exp\ (-E/RT)]\ t. \tag{5}$$

In this equation, k_0 is a pre-exponential factor, $C, P, r, E, R,$ and T represent the reagent concentration, particle size, stirring speed, activation energy, universal gas constant, and thermodynamic temperature, respectively. The constants α, β and γ are the reaction order for relating parameters.

On changing the concentration of ammonium citrate tribasic, equation (5) can be given as:

$$1 - 3(1-x)^{2/3} + 2(1-x) = k_0 \cdot (C_{Ammonium\ citrate\ tribasic})^\alpha\ t \tag{6}$$

$$d[1 - 3(1-x)^{2/3} + 2(1-x)]/dt = k_0 \cdot (C_{Ammonium\ citrate\ tribasic})^\alpha. \tag{7}$$

According to the equation (6) and (7), the relationships between $1 - 3(1-x)^{2/3} + 2(1-x)$ and time t

for different reagent concentrations, are shown in Figure 7(b). The slopes of these plots give the value $d[1 - 3(1 - x)^{2/3} + 2(1 - x)]/dt$ under different reagent concentrations. From the relationship $\ln\{d[1 - 3(1 - x)^{2/3} + 2(1 - x)]/dt\}$ vs $\ln C$, as shown in Figure 8(a), the slope was calculated to be is 0.92. Similarly, we can also obtain $\beta = 0.42$ and $\gamma = 0.73$.

The reaction kinetic equation is given as:

$$1 - 3(1 - x)^{\frac{2}{3}} + 2(1 - x) = \left[k_0 \ (C)^{0.92} \ (P)^{0.42}(r)^{0.73}\exp\left(-\frac{E}{RT}\right)\right] t. \tag{8}$$
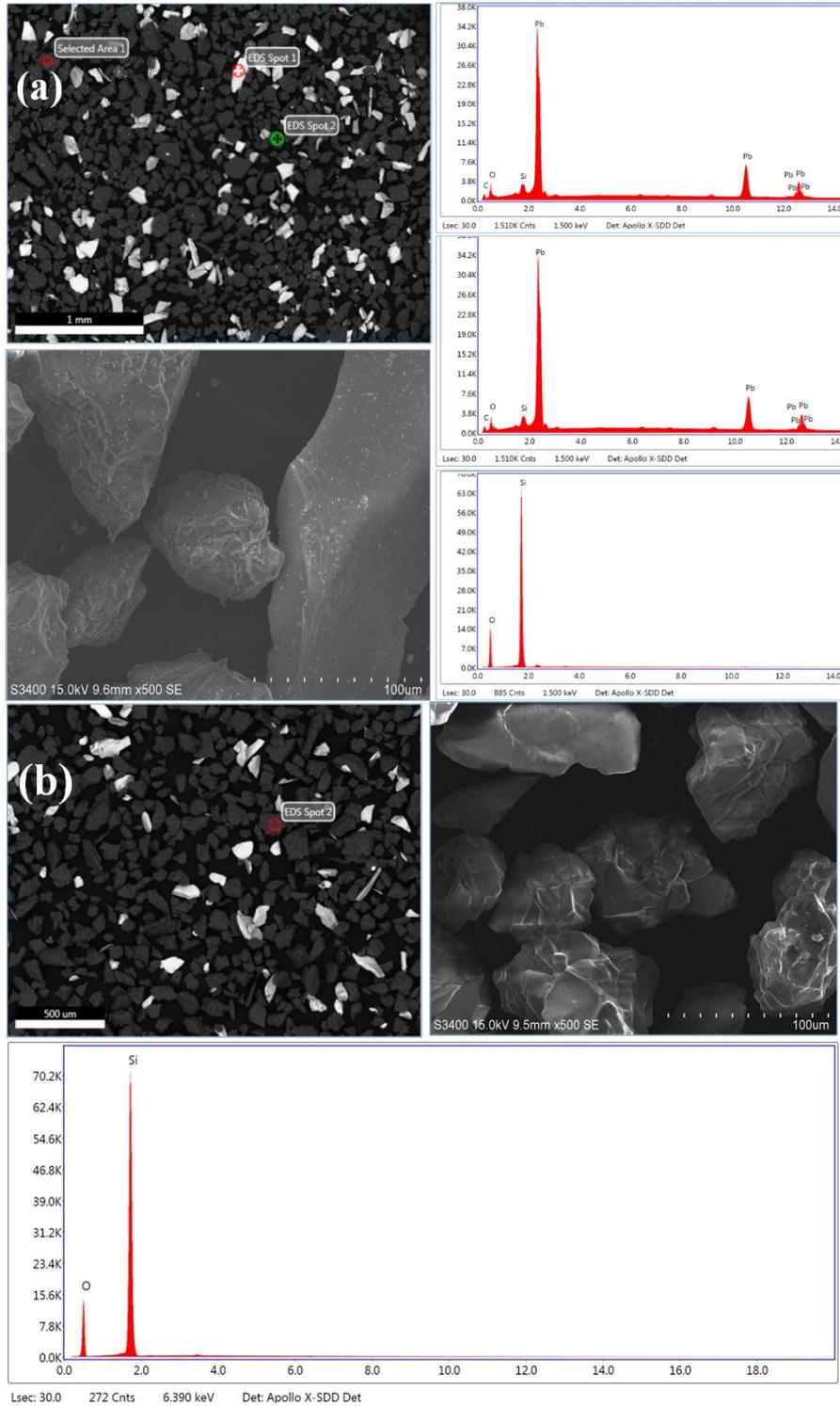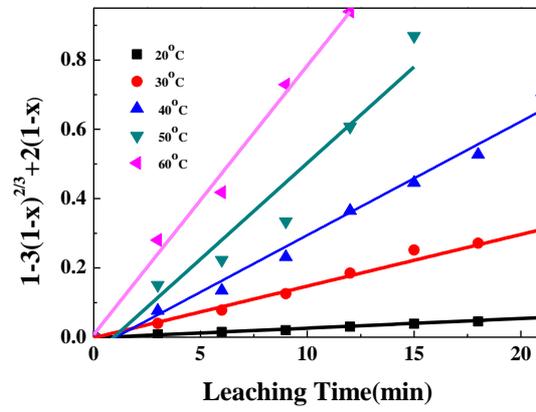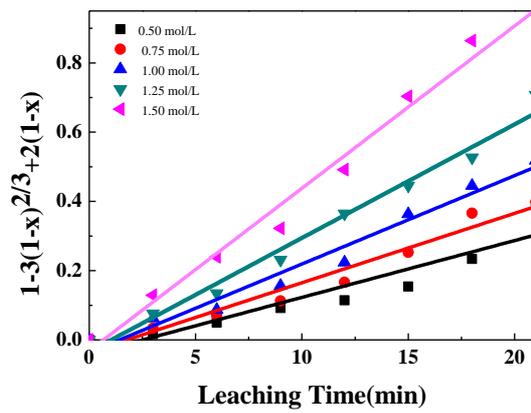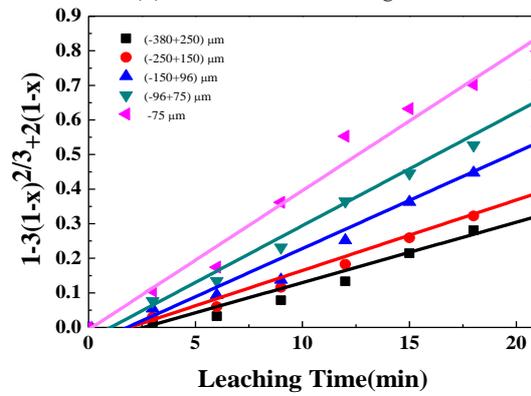

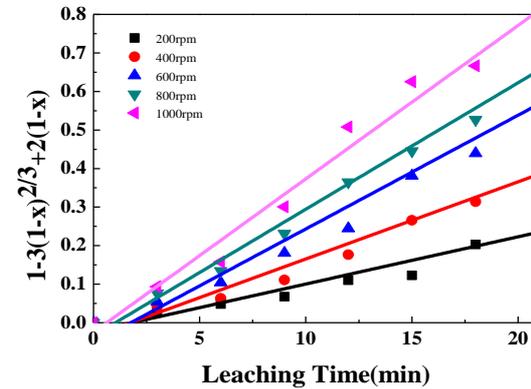Fig. 6. SEM-EDS images of (a) original sample and (b) leached sample

(a) temperature

(b) concentration of reagent

(c) particle size

(d) stirring speed

Fig. 7. Relationship between $1 - 3(1 - x)^{2/3} + 2(1 - x)$ and time t under different conditions:(a) temperature; (b) concentration; (c) particle size; (d) stirring speed

The relationship between $1 - 3(1 - x)^{2/3} + 2(1 - x)$ and $[\ k_0\ (C)^{0.92}\ (P)^{0.42}(r)^{0.73}\exp(-E/RT)]\ t$ is expressed in Figure 9. Although the points in the plot show some scatter, a straight line can be fitted to the data with a correlation coefficient (R^2) greater than 0.90. In terms of Arrhenius equation，we can obtain the apparent activation energy (kJ/mol), E=65.43 kJ/mol. Based on the activation energy, the reaction order and the k_0 value, the kinetic equation for lead-oxide ore leaching in ammonium citrate tribasic solution can be expressed as

$$1 - 3(1 - x)^{\frac{2}{3}} + 2(1 - x)\ =\ [\ 12.44 \times 10^{-3}\ (C)^{0.92}\ (P)^{0.42}(r)^{0.73}\exp(-65430/RT)]\ t\ . \tag{9}$$

(a) reagent concentration
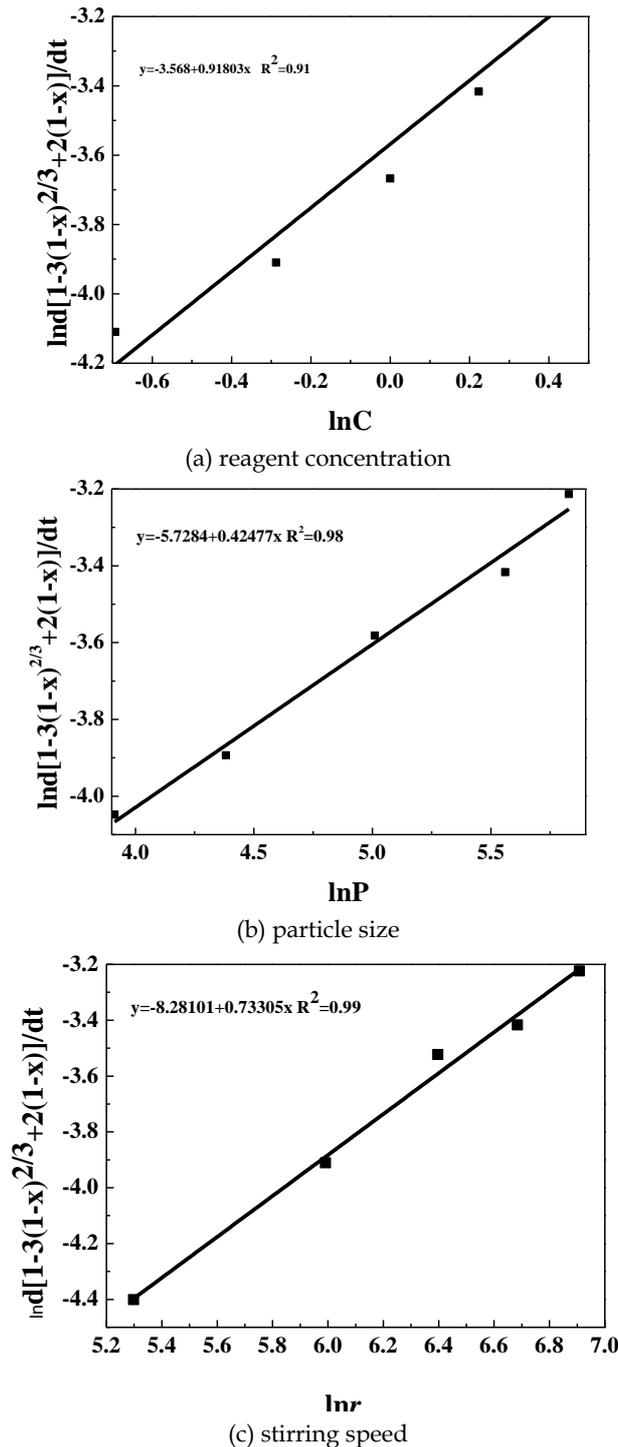
(b) particle size

(c) stirring speed

Fig. 8. Relationship between $\ln d[1 - 3(1 - x)^{2/3} + 2(1 - x)]/dt$ and $\ln C$, $\ln P$, $\ln$ r: (a) ammonium citrate concentration; (b) particle size; (c) stirring speed
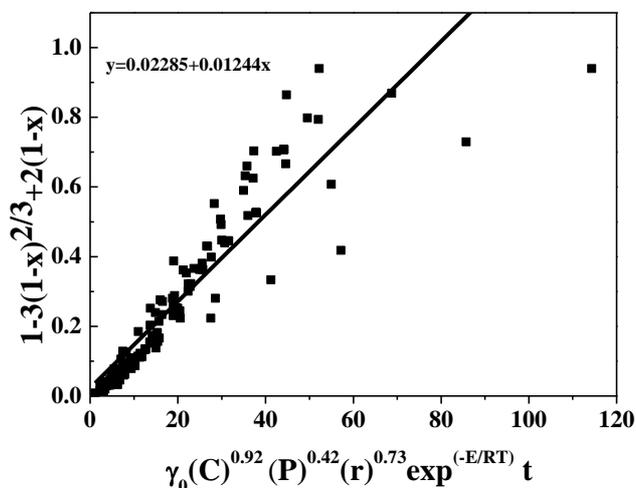
Fig. 9. Relationship between $1 - 3(1 - x)^{2/3} + 2(1 - x)$ and $k_0(C)^{0.92} \ (P)^{0.42}(r)^{0.73}\exp^{(-E/RT)} \ t$

4. Conclusions

In this study, the dissolution of lead-bearing ore in ammonium citrate tribasic solutions was investigated in a batch reactor. The effects of the reaction temperature, particle size, stirring speed, and reagent concentration on Pb conversion rate were proposed. Cerussite was readily dissolved by ammonium citrate tribasic solution giving high recoveries of lead. Approximately 95% of lead content was extracted from the samples with particle size range of +75-96 μm after 21 min leaching in 1.25 mol/L reagent solution at 800 r/min and 40°C. During leaching process, the lead conversion rate increased with an increase in reagent concentration, reaction temperature, and stirring speed, and a decrease in particle size. As a result of kinetic analysis, it was found that the leaching reaction followed the shrinking core model. A mathematical model containing experimental parameters was developed to represent the leaching kinetics:

$$1 - 3(1 - x)^{\frac{2}{3}} + 2(1 - x) \ = \ [\ 12.44 \times 10^{-3} \ (C)^{0.92} \ (P)^{0.42}(r)^{0.73}\exp(-65430/RT)] \ t,$$

where ($C, P, r, \ R$, and T represent the reagent concentration, particle size, stirring speed, universal gas constant, and thermodynamic temperature, respectively). The most important parameters affecting the leaching were time, the temperature of medium, reagent concentration and stirring speed. These results indicated that ammonium citrate tribasic could be used as an effective leaching reagent for extracting lead from lead-bearing ore.

Acknowledgment

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