



Study on separating of zinc and iron from zinc leaching residues by roasting with ammonium sulphate



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ABSTRACT

The hydrometallurgical processes for zinc recycling from zinc leaching residues (ZLR) generate a huge volume of iron residues. Iron residues lead to serious losses of metal value and secondary pollution. Efficient separation of zinc and iron is an ideal idea for avoiding iron residues by control zinc and iron phase from ZLR. In this study, a novel process for separating zinc and iron present in ZLR is developed consisting of sulfates roasting and selective leaching. The extraction of zinc increased to 92.63% while iron decreased to 2.04%. Zinc was leached efficiently from ZLR for comprehensive recovery with iron concentration of zinc enriched leachate below 0.38 g/L, so that the effective separation of zinc and iron was achieved. The iron content from this new process was increased higher than 45% for a potential use as an iron-making material after the removing of lead, zinc and cadmium from the leached residue. The iron removal process and iron residues could be avoided in the hydrometallurgical process.

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1. Introduction

Zinc leaching residue (ZLR) is a solid waste produced from traditional zinc hydrometallurgy process. It brings the loss of metal values (Boyanova et al., 2014) and secondary pollution (Alex et al., 2013; Coruh et al., 2013; Coruh and Ergun, 2010; Guo et al., 2010; Hu et al., 2014). Many pyrometallurgical (Holloway et al., 2007a; Jiang et al., 2006) and hydrometallurgical (Li et al., 2013; Wang et al., 2013, 2014; Wu et al., 2012) processes were developed to treat this residue. High temperature and high concentration of acid solution are needed to leach zinc from ZLR in hydrometallurgical process. However, a large amount of iron is also leached simultaneously and needs to be removed from leachate. It produces considerable amount of iron-containing residues (Ismael and Carvalho, 2003). There is no way to treat iron residues efficiently at present. The iron residues not only occupy precious land, but also threaten the environment (Han et al., 2014; Ju et al., 2011). Hence, the iron residues are a big trouble in recycling zinc from ZLR by hydrometallurgical process.

Zinc and iron present in ZLR are mainly in the form of zinc ferrite. The key point to avoid iron residues is to effectively separate zinc and iron in leaching process after decomposing zinc ferrite. In respect of decomposing zinc ferrite and separating zinc and iron, many researchers have conducted varying studies such as ZLR is roasted with sodium carbonate and then leached with acid solution. In this process,

manganese carbonate is added to inhibit the extraction of iron, but there is more than 40% of iron in leachate (Holloway et al., 2007b). The separation of zinc and iron could be achieved by caustic roasting-caustic leaching due to the different solubility of sodium zincate (Na_2ZnO_2) and ferric oxide (Fe_2O_3) in leaching with sodium hydroxide solution (Xia and Pickles, 1999). Similar treatment of zinc ferrite is also effective by roasting with $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ (Leclerc et al., 2003). Our research group has developed a new process to deal with zinc ferrite by using pyrometallurgical method. In this process, zinc ferrite is decomposed to zinc oxide (ZnO) and ferroferric oxide (Fe_3O_4) by roasting with carbon or carbon monoxide. Zinc and iron are separated due to the different solubility of zinc oxide and ferroferric oxide by controlling acidity of leaching solution (Li et al., 2012). However, in above methods, there are some shortcomings such as inducing a large amount of sodium and chloride ion into leachate (Leclerc et al., 2003; Xia and Pickles, 1999), the long roasting time (Leclerc et al., 2003), and hard controlling of roasting atmosphere (Wu et al., 2014; Yan et al., 2014). Therefore, it is still a challenge to treat iron residues which are produced from ZLR.

This study aims at separating zinc and iron by roasting ZLR with ammonium sulfate followed by water leaching. No impurities into leachate, a higher metal extraction and a better operating condition are achieved by roasting with ammonium sulfate (Abd-Elzaher, 1999; Liu et al., 2012; Saleh and Hassan, 2004). Furthermore, there are more advantages such as the zinc sulfate leachate would be directly returned to oxidative roasting–acid leaching–electrowinning process, the roasted residues could be leached with water instead of acid or alkali. Thermodynamic modeling based on the HSC program was conducted to predict different reactions during roasting. The formation of gases during the

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decomposition of ammonium sulfate was studied to understand the function of gases for decomposing zinc ferrite. The effect of roasting parameters on the phases of zinc and iron, the decomposition rate of zinc ferrite, and the extractions of zinc and iron were also investigated in this study.

2. Experimental

2.1. Materials

Representative ZLR used in this study was from a zinc hydrometallurgical plant in Hunan province, China, in which zinc sulfide concentrate was roasted, then leached to produce ZLR. The elemental composition of ZLR was analyzed with inductively coupled plasma (ICP) (Table 1), indicating that zinc and iron occupied 18.40% and 22.90% of total mass quantity respectively. Zinc ferrite (ZnFe_2O_4) is the major phase (60.92%) in ZLR, and zinc sulfate hydrate ($\text{ZnSO}_4 \cdot 6\text{H}_2\text{O}$), zinc sulfide (ZnS), willemite (Zn_2SiO_4), anglesite (PbSO_4) and segnitite ($\text{PbFe}_3(\text{AsO}_4)(\text{AsO}_3\text{OH})(\text{OH})_6$) are the minor phases presented in ZLR (Fig. 1 and Table 2). Ammonium sulfate used in this study is analytical grade reagent (content of ammonium >99.0%) supplied by Tianjin Chemical Reagent plant (China).

2.2. Analytical procedures

The content of zinc and iron in ZLR/leachate were determined by ICP (iCAP6300). The phases of initial, roasted and leached residues were detected by X-ray diffraction analysis (XRD, Rigaku, D/Max 2500; Cu/ $\text{K}\alpha$ radiation, 40 kV/250 mA). Thermogravimetric analyzer was coupled to the FTIR spectrophotometer (TG-FTIR, STA449F3A-TENSOR27, argon atmosphere 100 mL/min, heating rate of 10 °C/min, the spectral range of 4000–400 cm^{-1}) to investigate the pyrolysis of ammonium sulfate. ZLR was subjected to a series of zinc extraction tests to determine the quantities of zinc in various forms of oxide, sulfide, sulfate, silicate and ferrite according to chemical methods suggested by Zhang, 1992 (Liang et al., 2012). The quantities of zinc ferrite from roasted residues were determined by separating sulfate, oxide, silicate, and sulfide according to chemical methods.

2.3. Experimental equipment and procedure

2.3.1. Experimental equipment

Thermal concept HTK40-18/S; chamber size of 345 mm × 345 mm × 360 mm; heating element of silicon molybdenum rod; rated power of 110 kw; maximal temperature of 1800 °C; temperature control accuracy of ±1 °C.

2.3.2. Experimental procedure

(1) Zinc ferrite sulfates roasting

All samples were dried, ground and sieved to about 90% of particle size below 74 μm prior to roasting experiments. Roasting reaction was conducted in the furnace. ZLR of 20 g were blended in a desired ammonium sulfate/zinc ferrite mass ratio for 20 min by using a mortar and pestle, and then put into a porcelain crucible of 300 mL. The porcelain

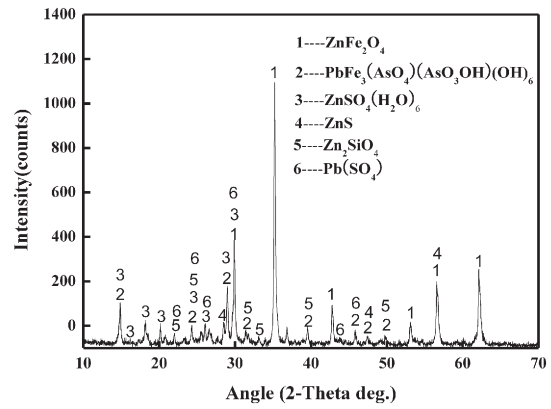


Fig. 1. X-ray diffraction pattern of ZLR.

crucible which loaded with mixture was put into muffle furnace after heating up to desired temperature. The roasted samples were taken out and cooled to room temperature, then weighed, ground and subjected to determine the quantities of zinc ferrite and extract zinc and iron by leaching. Offgas from roasting process was absorbed with water. The pH of absorption solution was adjusted to 7 to precipitate ammonium sulfate by sulfuric acid or ammonia. The decomposition rate of zinc ferrite (η) is calculated according to the following equation:

$$\eta = \frac{W_0 - W_r}{W_0} \times 100 \quad (1)$$

where W_0 is the zinc content of zinc ferrite from initial residues (g), W_r is the zinc content of zinc ferrite in roasted residues (g).

Roasted residues of 10 g were leached to extract zinc and iron with water at 80 °C for 20 min under liquid/solid ratio of 10/1 mL/g. Volume of filtrate was measured after filter. The extractions of Zn and Fe (ε) are calculated according to the following equation:

$$\varepsilon = \frac{c \times v}{m} \times 100\% \quad (2)$$

where c is the content of zinc and iron from filtrate (g/L), v is the volume of filtrate (L), and m is the quantities of zinc and iron in roasted residues (g).

(2) Sulfates selectively roasting

The mixture of ammonium sulfate and ZLR was roasted in the temperature range from 500 to 700 °C for the required time according to experimental procedures (1). The final roasted residues were taken out and cooled down to room temperature, then leached to extract zinc and iron with water. Offgas from roasting process was absorbed with absorbent on experimental procedures (1). The decomposition rate of zinc ferrite and the extractions of Zn and Fe were calculated with Eqs. (1) and (2) respectively.

Table 1
Chemical composition of ZLR by ICP.

Elements	Content (wt.%)	Elements	Content (wt.%)	Elements	Content (wt.%)
Zn	18.40	In	0.052	Ca	1.6
Fe	22.9	Mn	1.5	Cu	0.75
Pb	4.4	Al	1.1	Ag	0.024
Cd	0.26	S	6.4	As	0.52

Table 2
Phases composition of zinc in ZLR obtained by chemical analysis.

Phase	w(Zn)%	Phase occupation ratio/%
ZnSO ₄	4.45	23.93
ZnO	1.29	6.93
Zn ₂ SiO ₄	0.24	1.29
ZnS	1.29	6.93
ZnFe ₂ O ₄	11.33	60.92
Total	18.60	100

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