



Selective reduction process of zinc ferrite and its application in treatment of zinc leaching residues

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Abstract: The traditional zinc hydro-metallurgy generates a large amount of zinc ferrite residue rich in valuable metals. The separation of iron is crucial for resource recycling of valuable metals in zinc ferrite residue. A novel selective reduction roasting–leaching process was proposed to separate zinc and iron from zinc leaching residue which contains zinc ferrite. The thermodynamic analysis was employed to determine the predominant range of Fe_3O_4 and ZnO during reduction roasting process of zinc ferrite. Based on the result of thermodynamic calculation, we found that $V(\text{CO})/V(\text{CO}+\text{CO}_2)$ ratio is a key factor determining the phase composition in the reduction roasting product of zinc ferrite. In the range of $V(\text{CO})/V(\text{CO}+\text{CO}_2)$ ratio between 2.68% and 36.18%, zinc ferrite is preferentially decomposed into Fe_3O_4 and ZnO . Based on thermogravimetric (TG) analysis, the optimal conditions for reduction roasting of zinc ferrite are determined as follows: temperature 700–750 °C, volume fraction of CO 6% and $V(\text{CO})/V(\text{CO}+\text{CO}_2)$ ratio 30%. Based on the above results, zinc leaching residue rich in zinc ferrite was roasted and the roasted product was leached by acid solution. It is found that zinc extraction rate in zinc leaching residue reaches up to 70% and iron extraction rate is only 18.4%. The result indicates that zinc and iron can be effectively separated from zinc leaching residue.

Key words: zinc ferrite; zinc leaching residue; reduction roasting; metal separation

1 Introduction

Zinc ferrite is a by-product of traditional hydro-metallurgical zinc smelting process which is insoluble in low acid conditions for its stable spinel structure. Consequently, zinc and iron in zinc calcines are partially leached into the residues as zinc ferrite, causing the losses of zinc and iron, as well as the zinc leaching residues classified as hazardous waste [1–3].

Increasing number of studies including pyro-metallurgical, hydro-metallurgical and pyro-hydro-metallurgical processes are conducted on the recovery of zinc from zinc ferrite residues [4]. In the most typical pyro-metallurgical processes, the Waelz process for example, coal was used as a reducer and zinc ferrite was reduced into metal zinc and iron at 1100–1400 °C to volatilize and recover zinc as zinc oxide fume. Subsequently, metallic iron-containing residues were

discharged and stockpiled [5,6]. After high zinc and lead recovery, the iron and other valuable metals were wasted and huge fuel was consumed [7]. In hydro-metallurgical processes, mainly acid leaching processes [8–12], zinc ferrite was dissolved under intensive leaching conditions. Iron was dissolved extensively in these conditions and precipitated from solution as jarosite [13,14], goethite [14,15] or hematite [14] before electrowinning. Massive hazardous iron-precipitated residues containing heavy metals Pb, Cd, Mn and Cu are still critical concerns [16]. The large-scale application of alkaline leaching processes was hindered by the low leaching efficiency of zinc ferrite. In pyro-hydro-metallurgical processes including sulphate roasting–leaching [17] and chloridizing roasting–leaching process [18,19], the roasting temperature was much lower than that in the traditional roasting processes, resulting in higher efficiency in energy. However, the high solubility of iron undermined their advantages over traditional hot acid leaching.

The present work focuses on the separation of zinc from zinc leaching residues (ZLR) by a reduction roasting–leaching process. The zinc ferrite in ZLR is selectively reduced into zinc oxide which is dissolved in acid leaching, and magnetite which is remained in secondary leaching residues. To achieve this aim, firstly, the reduction process of pure zinc ferrite is characterized by TG analysis to optimize roasting method. Secondly, the reduction method is applied in the treatment of ZLR and the roasting product is leached to separate zinc and iron.

2 Experimental

2.1 Materials

For pure zinc ferrite preparation, zinc oxide and hematite were mixed according to stoichiometric ratio of zinc ferrite. The synthesis of zinc ferrite included three steps: roasting, acid washing and second roasting. Among these three steps, the duration of first roasting and second roasting were 4 h and 1 h, respectively. The natural annealing was applied in the cooling of roasting product after roasting steps. After first roasting, the hydrochloric acid of 1 mol/L was used to wash the roasting product for 1 h at 40 °C to remove the unreacted oxides. After second roasting, the product was sieved to pass through a 75 μm sieve and the sieved sample was dried to a constant mass for further analysis. Figure 1 shows the SEM image and EDS spectrum of synthesized zinc ferrite, showing that the product was mainly composed of zinc, iron and oxygen, with well-distributed particle size.

ZLR used in this work was obtained from a zinc smelter of Hunan Province, China. The XRD pattern of ZLR (Fig. 2) presented that the ZLR consisted of ZnS, Fe₃O₄, ZnFe₂O₄, PbS, Zn₂SiO₄, PbSO₄, FeS and CaSO₄. The ZLR sample was sieved and dried followed the protocol for zinc ferrite mentioned above.

2.2 Reduction roasting

Reduction roasting of the synthesized zinc ferrite was conducted in TG analysis. The heating rate was 10 °C/min and the reductive gas mixture was introduced when the desired temperature was achieved. After a certain roasting time, the reductive gas mixture was replaced by Ar and the roasting product was cooled in Ar. The ferrous rate $n(\text{Fe}^{2+})/n(\text{total iron})$ and soluble zinc rate $n(\text{soluble zinc})/n(\text{total zinc})$ of roasted product were characterized. The reduction roasting of ZLR was conducted in a tube furnace, and the reduction process followed the protocol for zinc ferrite mentioned above.

2.3 Characterization

Potassium dichromate titration for Fe and EDTA

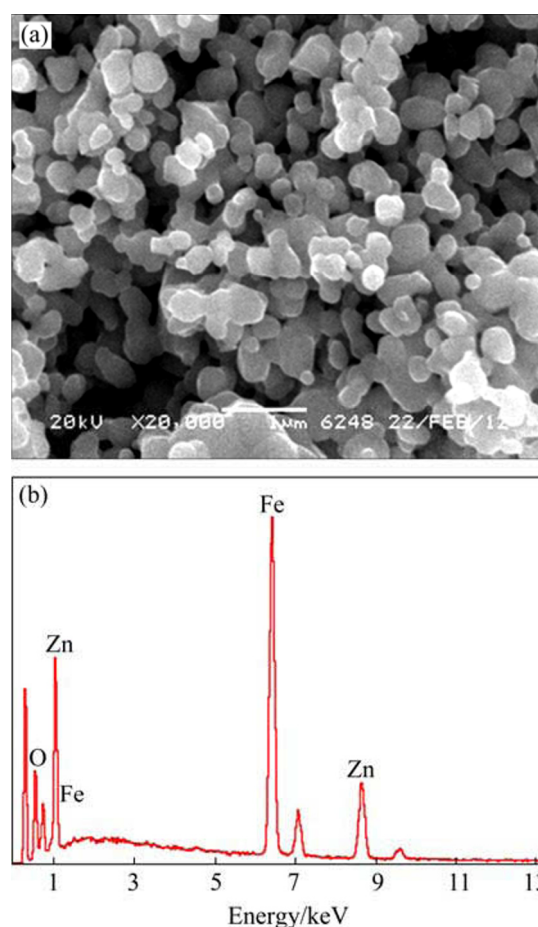


Fig. 1 SEM image (a) and EDS spectrum (b) of synthetic zinc ferrite

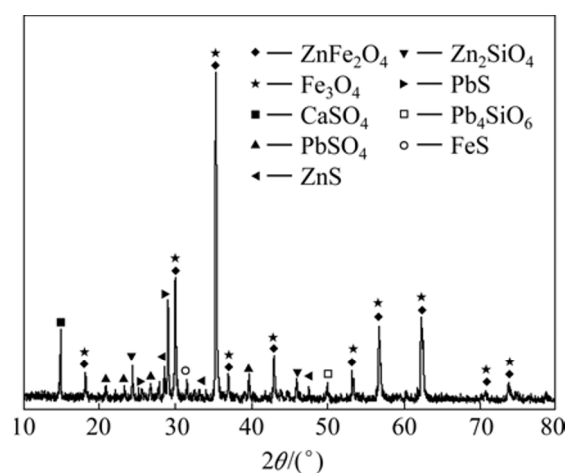


Fig. 2 X-ray diffraction pattern of zinc leaching residue

titration for Zn were performed for the chemical analysis of samples. XRD analysis with Cu K_α radiation (Rigaku, TTR-III) was employed to investigate the phase transform in the products of reduction roasting and leaching. Elements contents in the products of reduction roasting and leaching were determined by ICP-AES analysis (Baird, PS-6).

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