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Innovative methodology for comprehensive utilization of high iron bearing zinc calcine



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ABSTRACT

An innovative method for comprehensive utilization of high iron bearing zinc calcine was proposed, and the recovery of zinc and iron was studied. The thermodynamic analysis and roasting experiments were firstly carried out to study the decomposition of $ZnFe_2O_4$ which could be selectively converted to ZnO_4 and Fe_3O_4 under optimized roasting conditions. The roasted zinc calcine was then subjected to low acid leaching followed by magnetic separation. The effects of operating conditions on the recovery of zinc and iron and the mineralogical changes during the process were investigated. The results showed that about 91% of zinc was extracted and most of iron was separated into the leach residue as magnetite at 40 °C for 10 min with 90 g/L H_2SO_4 and liquid/solid ratio at 10 mg/L, while about 91% of iron was reported to the concentrate with iron grade of 45.5% after a magnetic separation at 35 kA/m. Besides, lead impurity was transformed, separated and concentrated into the tailing in the form of PbSO₄ and Pb.

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1. Introduction

Zinc is an important metal required for various applications in metallurgical, chemical and textile industries, and is mainly recovered from zinc sulfide concentrates through the conventional hydrometallurgical process, including oxidative roasting, acid leaching, purification and electrowinning steps, in the world [1–3]. In the roasting step, zinc ferrite (ZnFe₂O₄) is generated as an inevitable byproduct of the oxidative roasting [4,5]. Furthermore, the higher iron is contained in zinc sulfide concentrates, the more amount of zinc ferrite will be generated. Since it is very stable and hardly dissolved in the conventional acid leaching process, leading to considerable amounts of leach residues are produced [6,7].

Concerns over the difficulty of zinc recovery and the potential environmental risks have been raised because of the undesired product, and consequently, various pyrometallurgical and hydrometallurgical routes are employed to dispose it for further recovering zinc. Reported as one of the most common methods, Waelz process is actually recycling zinc in the form oxide that is convenient for leaching process [8,9]. Although most of zinc can be recovered after reduction roasting above 1000 °C, a fatal drawback of these pyrometallurgical methods is high energy

consumption and strict requirement on dust collection system. By contrast, hydrometallurgical processes are regarded as more low-carbon and eco-friendly to treat those materials containing zinc ferrite [10–15]. However, a high-temperature acid leaching is required to destroy its stable structure. Meanwhile, the zinc sulfate solution obtained always contains a high content of iron, which must be removed from the solution prior to other impurities such as Ag, Cu, Pb, Ni, Co and Cd, are precipitated to enable the production of high quality zinc [16,17]. The jarosite and goethite process are the two common precipitation methods, which generate considerable quantities of residues containing toxic impurities, such as Pb, Ag, Cd and As [18,19]. These precipitation residues have stable chemical structure and contain low grade of iron and precious metals, resulting in the difficult recycle of Fe, Pb, and Ag, occupation of precious land and secondary pollution [20].

Considering the drawbacks of pyrometallurgy and hydrometallurgy, some researchers recently paid their attentions to a novel approach that is the combination of pyrometallurgical and hydrometallurgical process, which is, mainly using the method of roasting, such as transformation roasting [21,22], sulphation roasting [14] and reduction roasting [23–25], to convert zinc ferrite into soluble zinc phase for facilitating zinc extraction by neutral or low acid leaching process. These new methods for reducing energy consumption and environment protection have a tremendous potential, but they are still in experimental stage and their industrial application cannot be achieved until a lot of problems now presented will have been solved in the future.

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To resolve the problem of large amounts of leach residues, most methods reported have been conducted for treating the secondary resources, which are based on the perspective of waste governance. Little ideas, however, are based on the perspective of prevention to impede the generation of hazardous waste in the conventional process of zinc production. This cannot meet the requirement of mass zinc production from high iron bearing zinc sulfide concentrates which is nowadays being increasingly used, because low iron bearing zinc ores are gradually exhausted in the world. Consequently, a novel method for comprehensive utilization of high iron bearing zinc calcine was proposed by our group based on the idea of prevention. The technology developed is to add a reduction roasting process performed at lower temperature after the oxidative roasting above 900 °C for taking full advantage of the heat generated in the previous process to selectively decompose zinc ferrite produced into zinc oxide and magnetite [26-28]. The zinc oxide generated can be extracted selectively by low acidic solution, and meantime, the magnetite obtained is separated into the leach residue and can be further recovered by magnetic separation. Besides, other valuable metals, such as Pb, Ag and In, are thus concentrated in the iron tailing, which is conducive to subsequent recovery. The application of this method will not only simplify the process of zinc production and fulfill the comprehensive utilization of iron bearing zinc calcine, but also reduce the anticorrosion requirement of leaching equipment and lower the quantity of leach residue.

The present paper focused on discussing the recovery of zinc and iron from high iron bearing zinc calcine by the proposed process. The effects of reduction roasting, acid leaching and magnetic separation parameters on the transformation of zinc ferrite, the selective extraction of zinc and the recovery of iron were investigated, respectively, and the optimum experiments conditions were therefore established. The mineralogical changes during the process were also studied by XRD, SEM and LPSA.

2. Experimental

2.1. Materials

The high iron bearing zinc calcine used in this study was collected from a zinc hydrometallurgical plant in Inner Mongolia, China. It was the oxidative roasting product of high iron bearing zinc sulfide concentrates. The zinc calcine was analyzed by X-ray fluorescence spectroscopy (XRF, Rigaku, model ZSX Primus II), results of which are shown in Table 1. The phase compositions of zinc and iron shown in Tables 2 and 3, respectively, were analyzed by chemical methods. It can be seen that zinc is mainly in the form of zinc oxide (83.3%) while iron mostly exists as zinc ferrite (82.5%). Mineralogical structure of the zinc calcine was characterized by X-ray diffraction analysis (XRD, Rigaku, TTR-III), whose results indicate that the zinc calcine is mainly composed of zinc oxide, zinc ferrite (ZnFe₂O₄), magnetite (Fe₃O₄), sphalerite (ZnS) and zinc silicate (ZnSi₂O₄) (see Fig. 1). All the peaks of ZnFe₂O₄ and Fe₃O₄ are almost overlapped, which was also observed by other researchers [23,29,30]. This will increase the difficulty to distinguish the two phases in the experiment.

Carbon monoxide (CO) was used as a reducing agent in the roasting process to avoid the over-reduction of zinc and iron phases, which usually occurred in traditional roasting process

Table 1Chemical composition of high iron-bearing zinc calcine (mass, %).

Zn	Fe	S	Pb	Cu	Si	Ca	Mn	Cd
57.37	12.1	2.44	1.27	0.915	0.89	0.569	0.532	0.154

Table 2Phase composition of zinc in high iron-bearing zinc calcine.

Phase composition	ZnO	ZnFe ₂ O ₄	ZnSO ₄	ZnS	Zn ₂ SiO ₄
Zn distribution (%)	83.30	12.84	2.01	1.38	0.47

Table 3Phase composition of iron in high iron-bearing zinc calcine.

Phase composition	ZnFe ₂ O ₄	Fe ₃ O ₄	FeS ₂	Fe ₂ CO ₃	Others
Fe distribution (%)	82.53	10.38	2.91	0.45	3.73

when carbon was used. Carbon dioxide (CO_2) was chosen to control the reducibility of roasting atmosphere for selective transformation of zinc ferrite. Nitrogen (N_2) was applied as a supplementary and protective gas. Sulfuric acid (H_2SO_4) and sodium hexametaphosphate [($NaPO_3$)6] of chemical grade were used in the leaching and magnetic separation experiments, respectively.

2.2. Experimental method

In the roasting step, for each test, 150 g of the zinc calcine was taken into the rotary kiln with a corundum reaction tank (cylinder size: L = 300 mm, d = 150 mm) designed by the authors, whose schematic diagram had been given in another paper [27], and heated under N₂ atmosphere to a desired reduction temperature. A mixture gas of CO, CO₂ and N₂ was then introduced into the reduction system for reduction roasting. After the roasting, the roasted zinc calcine was cooled to room temperature under the protection of N2, and then it was removed from the rotary kiln, weighed and reserved in closed vessel for related analysis and subsequent experiment. Besides, the rotation rate of rotary kiln was constant at 5 rpm in the whole roasting process. In the leaching step, 100 g of the roasted zinc calcine obtained under optimized conditions was put into a three-neck flask containing sulfuric acid solution, which was heated by a thermostatic mantle and provided with a mechanical stirrer. After the leaching was finished, the leachate was separated from the residue by vacuum filtration and analyzed for zinc and iron by chemical method (potassium dichromate titration for Fe and EDTA titration for Zn). The leach residue was dried, weighed, and preserved for related analysis and subsequent magnetic separation. Control experiments using high iron bearing zinc calcine were conducted simultaneously in order to investigate the effect of reduction roasting on the leaching of zinc and iron. After the leaching process, the leach residue was subjected to magnetic separation by low intensity magnetic separator (model: XCGS- Φ 50) in an attempt to recover iron. The effects of magnetic intensity and (NaPO₃)₆ addition on recovery of iron were investigated.

In addition, other two characterization techniques were also used in this study. One is Laser Particle Size Analyzer (LPSA, MICRO-PLUS), which was used to investigate the particle size distributions of the roasted zinc calcine and the leach residue. The other is Scanning Electron Microscopy (SEM, JEOL, JSM-6490LV) applied to the morphology study.

3. Results and discussion

3.1. Decomposition of ZnFe₂O₄ by reduction roasting

Gibbs free energy change (ΔG) is the principal criterion for judging whether a chemical reaction can occur, and a reaction is likely to happen when its ΔG is negative. The ΔG of chemical reaction at a temperature is calculated by its isothermal equation

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