



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

Gang YU¹, Ning PENG², Lan ZHOU³, Yan-jie LIANG², Xiao-yuan ZHOU¹,
Bing PENG², Li-yuan CHAI², Zhi-hui YANG²

1. Changsha Engineering & Research Institute Ltd. of Nonferrous Metallurgy, Changsha 410011, China;
2. School of Metallurgy and Environment, Central South University, Changsha 410083, China;
3. School of Business, Hunan University, Changsha 410082, China

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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.

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Corresponding author: Lan ZHOU; Tel:+86-731-88822899; E-mail: zhouz10520@vip.sina.com

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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 , Fe_3O_4 , ZnFe_2O_4 , PbS , Zn_2SiO_4 , PbSO_4 , FeS and CaSO_4 . 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

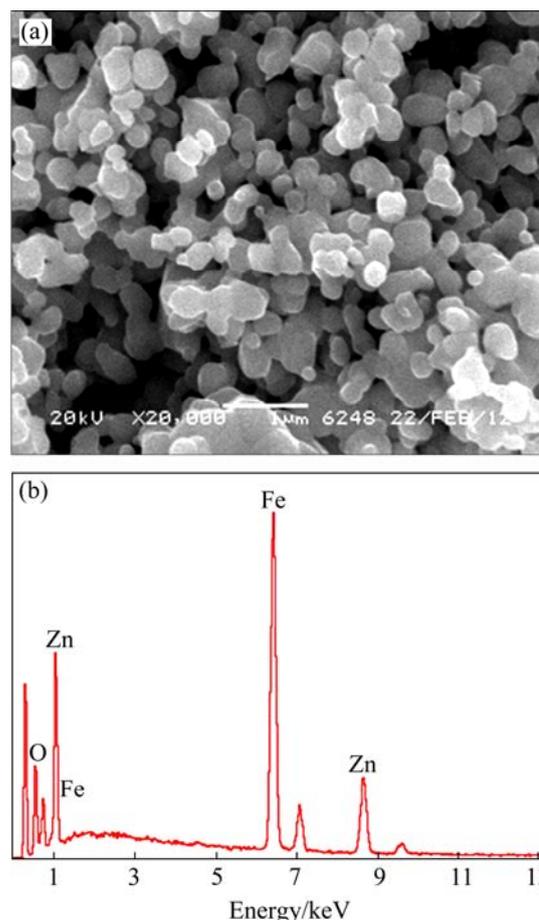


Fig. 1 SEM image (a) and EDS spectrum (b) of synthetic zinc ferrite

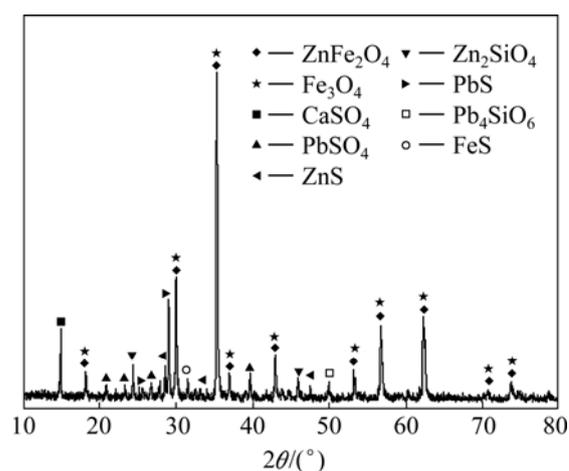


Fig. 2 X-ray diffraction pattern of zinc leaching residue

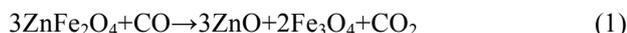
titration for Zn were performed for the chemical analysis of samples. XRD analysis with $\text{Cu K}\alpha$ radiation (Rigaku, TTR-III) was employed to investigate the phase transform in the products of reduction roasting and leaching. Elements contents in the products of reduction roasting and leaching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

3 Results and discussion

3.1 Thermodynamic analysis

The phase equilibrium diagram of Fe–Zn–O system under 800 °C obtained by software Fact Sage 6.2 is shown in Fig. 3. The route of oxygen loss process of zinc ferrite is presented by dotted line in Fig. 3. It can be seen that the reduction of zinc ferrite can be divided into four stages. The phase composition at the first stage consists of magnetite (spinel) and zinc oxide (zincite), which are the target products of magnetic roasting. At the second stage, the magnetite is further reduced to ferrous oxide (amonoxyde), thus the phase composition consists of magnetite (spinel), ferrous oxide (amonoxyde) and zinc oxide (zincite). At the third stage, metal iron is generated, and the phase composition consists of metal iron (BCC_A2) and zinc oxide (zincite). At the final stage, the phase composition consists of metal iron and zinc. Therefore, the selective reduction roasting of zinc ferrite is actually to prevent the second stage of the oxygen loss process.

In order to control the pathway of oxygen loss process through the modification of roasting conditions, the predominance-area diagram of Zn–Fe–C–O at 800 °C (Fig. 4) is gained using software FactSage 6.2. The possible reactions in the reduction process of zinc ferrite are as follows:



The selective reduction of zinc ferrite is to enhance Reaction (1) and to prevent Reaction (2) and Reaction (3), indicating that the generation of ferrous oxide or metal iron should be prevented. It can be seen from Fig. 4 that the ratio of $V(\text{CO})/V(\text{CO}+\text{CO}_2)$ is the key factor affecting the pathway of oxygen loss process. The area between Line (1) and Line (2) is the predominance-area for phase composition consisting of Fe_3O_4 and ZnO, and the relationship between $\lg p_{\text{CO}}$ and $\lg p_{\text{CO}_2}$ for the Line (1) and Line (2) are as follows:

$$\text{Line (1)} \quad \lg p_{\text{CO}} = \lg p_{\text{CO}_2} - 1.6266$$

$$\text{Line (2)} \quad \lg p_{\text{CO}} = \lg p_{\text{CO}_2} - 0.28585$$

It is known that through the calculation, the ratio of $V(\text{CO})/V(\text{CO}+\text{CO}_2)$ should be kept from 2.31% to 34.11% for the selective reduction of zinc ferrite at 800 °C.

3.2 Selective reduction of zinc ferrite

Based on thermochemical analysis, thermogravimetric (TG) analysis was used to characterize the reduction process of zinc ferrite under different reduction conditions, and the soluble zinc rate and ferrous rate of the roasted products were analyzed at the same time. Here, the soluble zinc rate represents the proportion of soluble zinc to total zinc, while ferrous rate represents the proportion of ferrous iron to total iron. Taking magnetite for example, its ferrous rate is 33.3%. Therefore, the targets of selective reduction roasting of zinc ferrite are to obtain the highest soluble zinc rate and to keep ferrous rate near 33.3%.

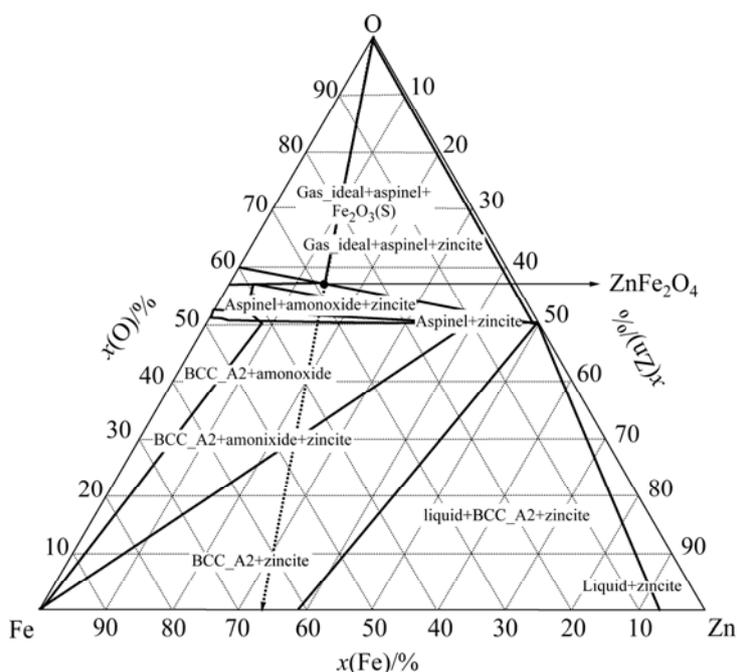


Fig. 3 Phase equilibrium diagram of Fe–Zn–O system under 800 °C

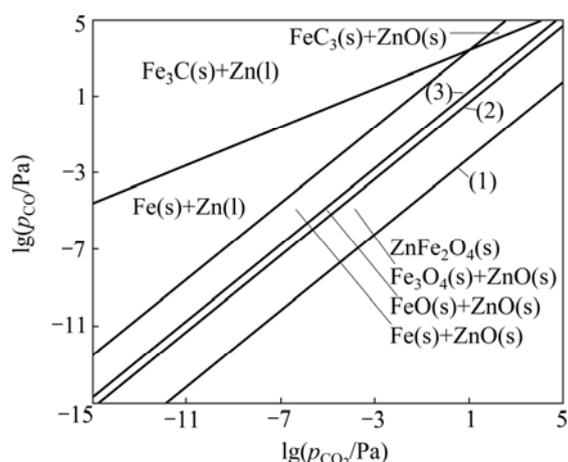


Fig. 4 Predominance-area diagram of Zn-Fe-C-O at 800 °C

3.2.1 Effect of temperature on selective reduction of zinc ferrite

The effect of temperature on the reduction process of zinc ferrite was examined when CO content, $V(\text{CO})/V(\text{CO}+\text{CO}_2)$ and roasting time were kept at 8%, 20% and 75 min, respectively. Firstly, the mass losses during reduction roasting of zinc ferrite under 600, 650, 700, 750 and 800 °C were investigated and the results are shown in Fig. 5. It can be seen that TG curves of zinc ferrite under different temperatures are similar. The mass loss of zinc ferrite increases with the rise of temperature. The mass loss rates are 1.34% at 600 °C, 1.67% at 650 °C, 1.76% at 700 °C, 1.83% at 750 °C and 1.92% at 800 °C, respectively. The TG curve at 600 °C has a gentle inclination, and then maintains the trend to decline when the reaction time reaches up to 75 min, indicating that zinc ferrite is decomposed slowly under low temperature and reaction does not reach equilibrium at 75 min. When the temperature ranges from 650 to 750 °C, there is a platform appearing after 60 min in TG curves. However, the platform disappears when the temperature increases over 800 °C. The continual mass loss is caused by over-reduction of magnetite.

Chemical analysis results indicate that the soluble zinc rate increases obviously along with the raising temperature (Fig. 5(b)). The soluble zinc rate increases substantially from 53.69% to 68.26% as the temperature increases from 600 to 650 °C. Thereafter, soluble zinc rate maintains an increase at a relative low rate with the rise of temperature. For instance, the soluble zinc rate is 70.90% at 700 °C, 71.07% at 750 °C and 73.38% at 800 °C. Meanwhile, the ferrous rate has a similar trend to soluble zinc rate with the rise of temperature. The ferrous rate is 19.56% at 600 °C, 23.63% at 650 °C, 24.66% at 700 °C, 25.34% at 750 °C, and 26.76% at 800 °C. High temperature could further accelerate the reduction of

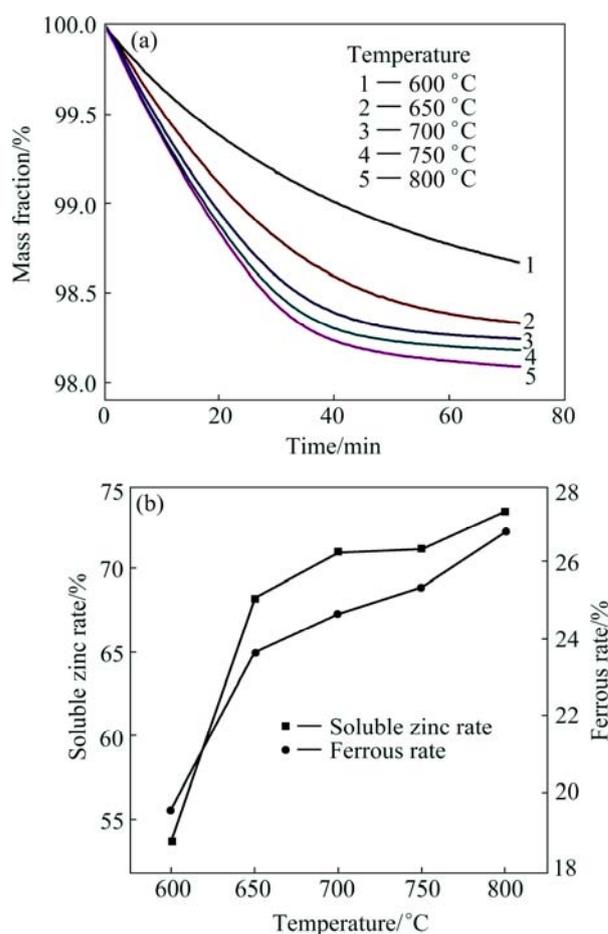


Fig. 5 Effect of temperature on TG curves (a) and soluble zinc rate and ferrous rate (b) for roasting products from selective reduction

magnetite to ferrous oxide, which could cause some difficulties in the separation of iron from zinc for zinc leaching residue. Thus, the reduction temperature should be maintained in the range of 700–750 °C.

3.2.2 Effect of CO content on zinc ferrite selective reduction

Low CO content can cause low reduction rate in magnetic roasting of zinc ferrite, while high CO content will bring high risk of CO because CO is a toxic gas. The effect of CO content on zinc ferrite selective reduction was studied under temperature of 750 °C with 75 min. As shown in Fig. 6, the mass loss rate of zinc ferrite increases with the rising of CO content. The mass loss rates are 0.54%, 1.51%, 1.80%, 1.82% and 1.84% at CO contents of 2%, 4%, 6%, 8% and 10%, respectively. When the CO content is 10%, the platform of TG curve appears at about 40 min.

The soluble zinc rate increases significantly with the increasing CO content under weaker reducing atmosphere ($\varphi(\text{CO}) < 6\%$). For instance, the soluble zinc rate increases from 22.22% to 64.60% with increasing

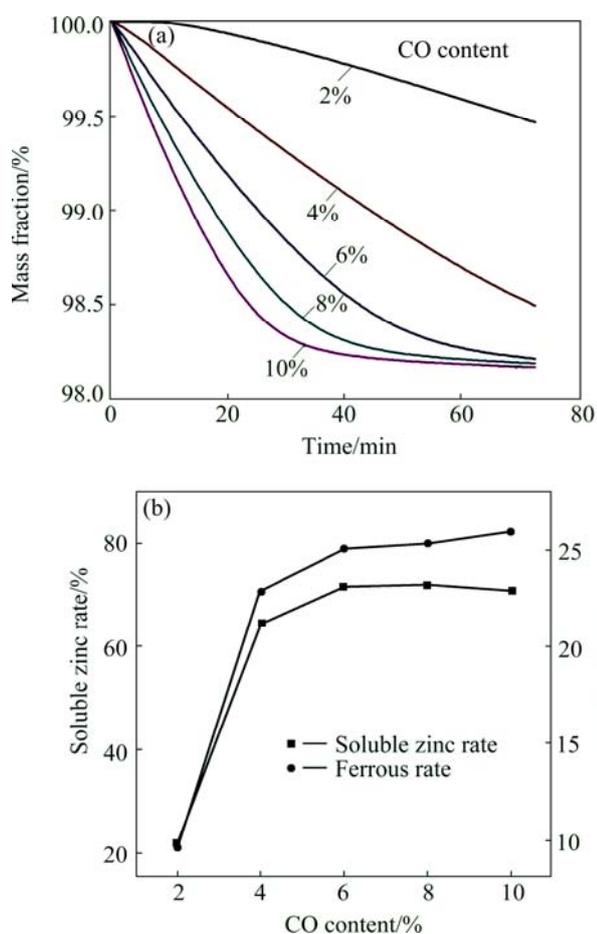


Fig. 6 Effect of CO content on TG curves (a) and soluble zinc rate and ferrous rate (b) for roasting products from selective reduction

CO content from 2% to 4%. However, when CO content is above 6%, soluble zinc rate has no significant change. The ferrous rate varies in a similar trend to soluble zinc rate. The results show that zinc ferrite is decomposed faster with higher CO content, and reaction achieves an equilibrium within 60 min with CO content of above 6%.

3.2.3 Effect of $V(\text{CO})/V(\text{CO}+\text{CO}_2)$ ratio on zinc ferrite selective reduction

The ratio of $V(\text{CO})/V(\text{CO}+\text{CO}_2)$ is the most significant factor for selective reduction of zinc ferrite according to thermal calculation. The mass loss of zinc ferrite in the reduction process under different $V(\text{CO})/V(\text{CO}+\text{CO}_2)$ ratios was investigated at temperature of 750 °C with 75 min of roasting time. As shown in Fig. 7(a), mass loss significantly depends on the $V(\text{CO})/V(\text{CO}+\text{CO}_2)$ ratios. The mass loss rates are 1.71%, 1.83%, 1.94%, 2.40% and 2.71% with $V(\text{CO})/V(\text{CO}+\text{CO}_2)$ ratios at 15%, 20%, 30%, 40% and 50%, respectively. It can also be observed that all TG curves have a slope before 30 min. And the mass loss rates in all the TG curves differ after 30 min. A platform in the TG

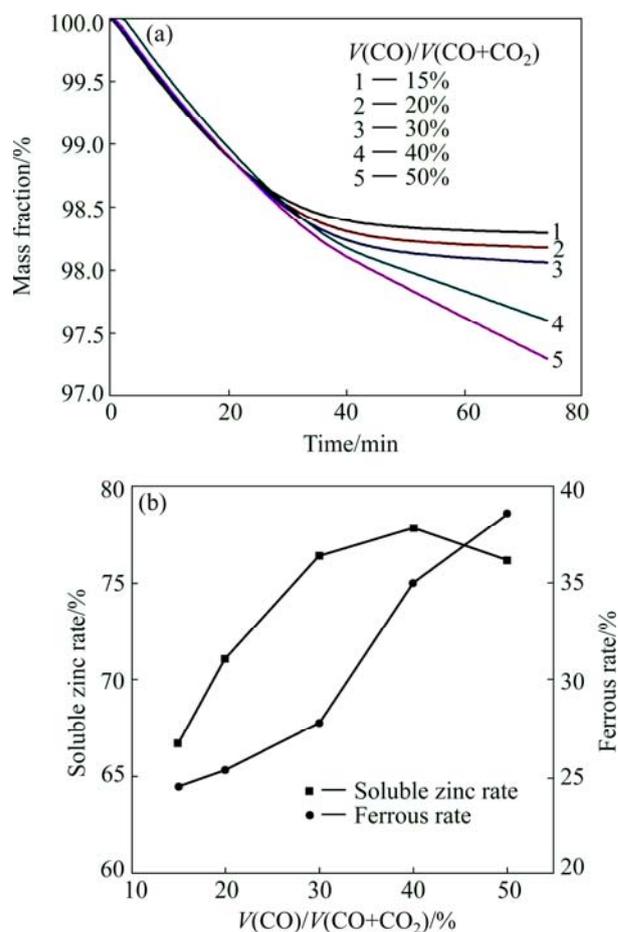


Fig. 7 Effect of ratio of $V(\text{CO})/V(\text{CO}+\text{CO}_2)$ on TG curves (a) and soluble zinc rate and ferrous rate (b) for roasting products from selective reduction

curves with lower $V(\text{CO})/V(\text{CO}+\text{CO}_2)$ ratio (less than or equal to 30%) is obtained after 60 min. The theoretic mass loss of zinc ferrite in the selective reduction is 2.21% when zinc ferrite is entirely decomposed into zinc oxide and magnetite, but the mass loss of zinc ferrite in the present experiment exceeds the theoretical value when CO ratio is over 30%, indicating that zinc ferrite is over reduced. Thus, the optimal $V(\text{CO})/V(\text{CO}+\text{CO}_2)$ ratio for the selective reduction of zinc ferrite is 30%.

Figure 7(b) shows the effect of $V(\text{CO})/V(\text{CO}+\text{CO}_2)$ ratio on the soluble zinc rate and ferrous rate of roasting product. The soluble zinc rate of the roasted sample has its maximum value at $V(\text{CO})/V(\text{CO}+\text{CO}_2)$ ratio of 40%. When $V(\text{CO})/V(\text{CO}+\text{CO}_2)$ ratio increases to 50%, the soluble zinc rate decreases due to the over reduction of magnetite (the ferrous rate exceeds the theoretic value of magnetite). Ferrous oxide and zinc ion can enter into the crystal lattice of ferrous oxide and then form a zinc iron solid solution. Thus, the $V(\text{CO})/V(\text{CO}+\text{CO}_2)$ ratio should be kept at a lower level (less than or equal to 30%) for the selective reduction of zinc ferrite.

3.3 Separation of Fe and Zn from roasting product of ZLR

The selective reduction roasting process was applied in the treatment of zinc leaching residues (ZLR) under the optimum roasting conditions established in Section 3.2. The SEM-EDS patterns of ZLR before and after roasting are shown in Fig. 8 and Fig. 9, respectively. It can be seen that ZLR particle is loose and has complex surface composition which contains multiple chemical elements including Ca, Al and S. The average particle size of samples increases because of self-sintering after magnetic roasting. In addition, Zn is released from ZLR in the form of zinc oxide and coated on the particle surface, leading to difficult detection of Ca and Al. Although the increase of particle size inhibits the following acid leaching, the coating of zinc oxide on the particle surface can promote the dissolving of zinc.

After roasting, ZLR was further used for zinc and iron leaching. The effects of leaching acidity, liquid-to-solid ratio (L/S), temperature and time on zinc and iron leaching rate were investigated. Figure 10(a) shows the effect of acidity on zinc and iron leaching rate with L/S ratio of 10:1 after leaching for 10 min at 45 °C. Iron leaching rate has a monotonic increase with

acidity, while the zinc leaching rate increases initially when the acidity is lower than 90 g/L, thereafter it decreases. The extraction rates of zinc and iron has a similar trend of increasing before L/S ratio of 10:1 and then the corresponding values decrease. The zinc extraction rate has its maximum value at a L/S ratio of 10:1 (Fig. 10(b)). As shown in Fig. 10(c), iron extraction rate increases rapidly with the increase of temperature, while the extraction rate of zinc increases slowly when the temperature is lower than 45 °C, and then it raises rapidly over 45 °C. In order to obtain a balance between high zinc extraction and low iron extraction, the optimal leaching temperature is selected as 55 °C. As shown in Fig. 10(d), the zinc extraction rate steadily increases before 20 min, and then it maintains constant. However, iron extraction increases with increasing time until leaching for 90 min. To improve production efficiency and obtain a high zinc extraction rate and low iron extraction rate, the optimum leaching time is chosen as 20 min.

Table 1 shows the contents of Zn, Fe, S and Ag in different samples. It can be seen that the contents of the above elements in the roasted and leached samples change as compared with the raw sample. The contents

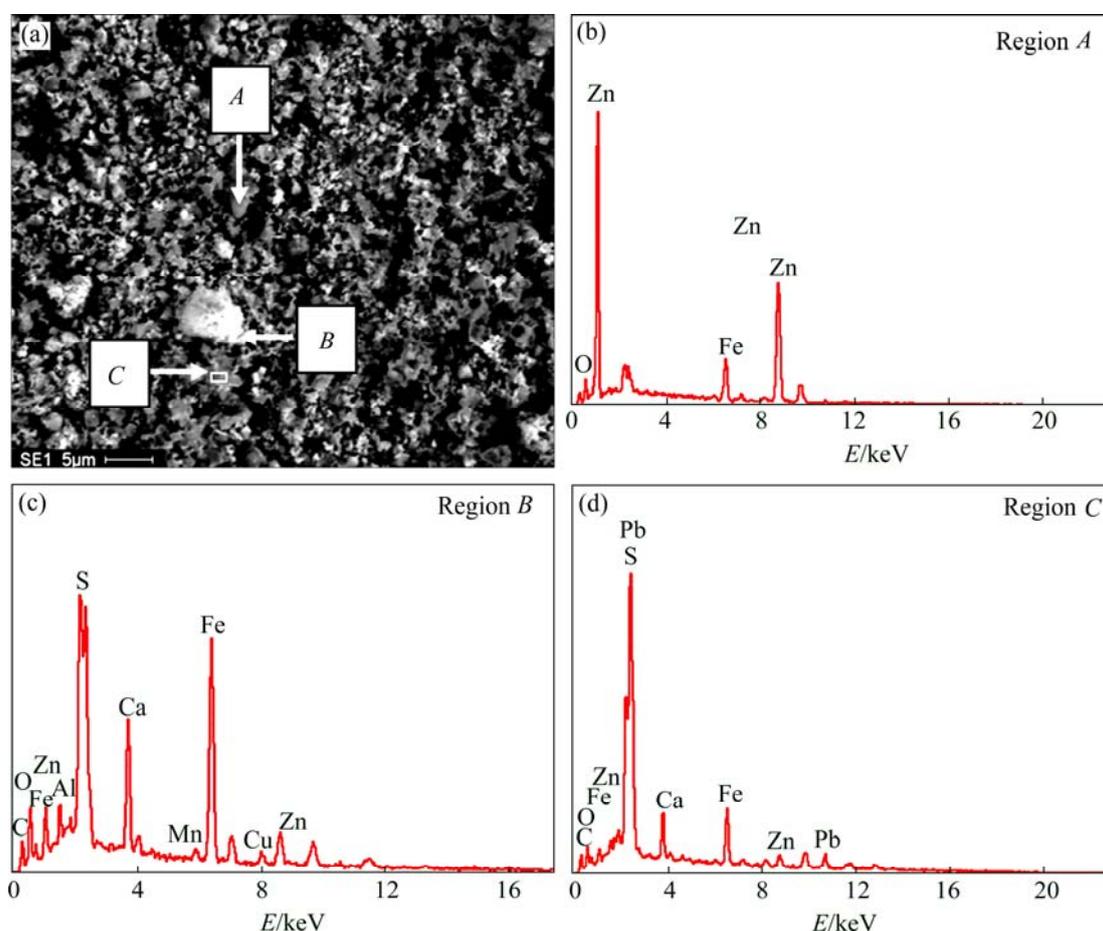


Fig. 8 SEM image (a) and EDS spectra (b, c, d) of zinc leaching residue before reduction roasting

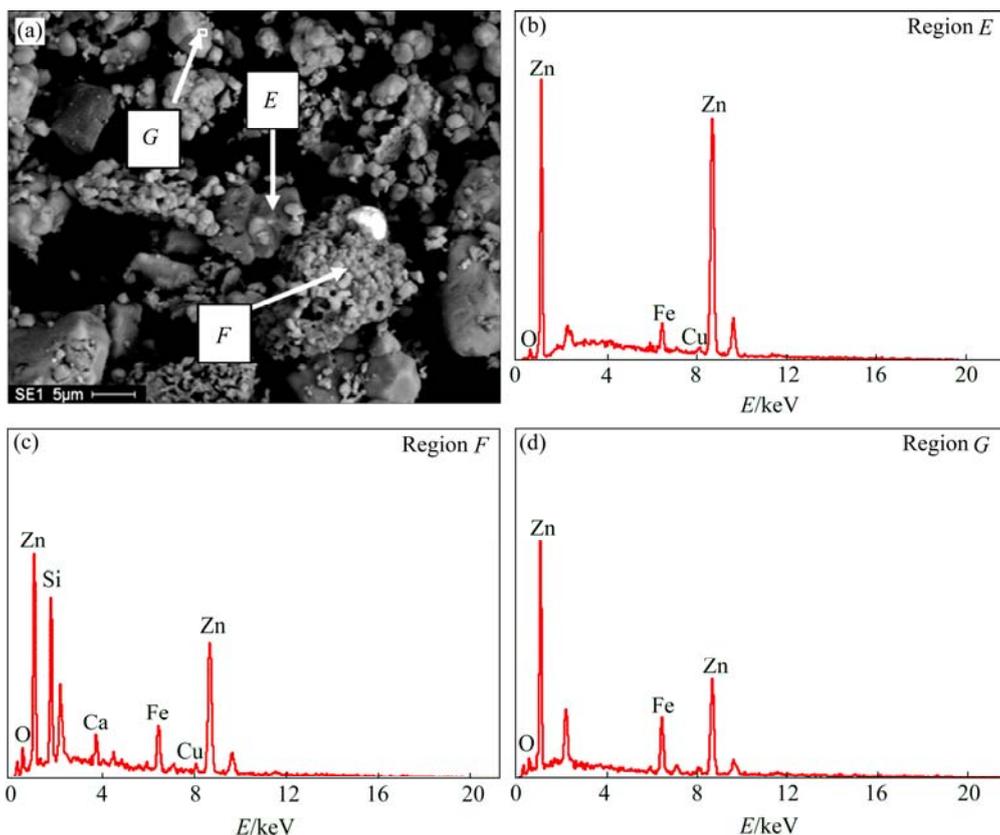


Fig. 9 SEM image (a) and EDS spectra (b, c, d) of zinc leaching residue after reduction roasting

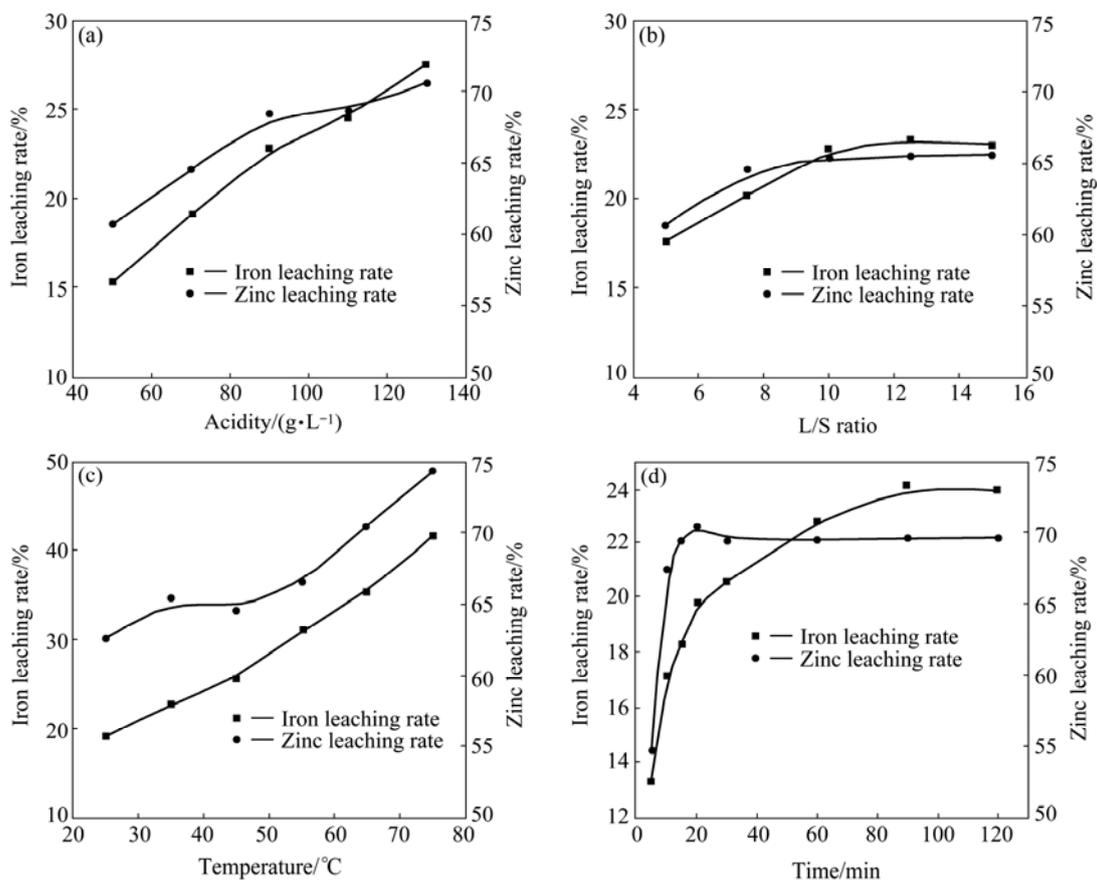


Fig. 10 Extraction of zinc and iron under different leaching conditions: (a) Acidity; (b) Liquid-to-solid ratio; (c) Temperature; (d) Time

Table 1 Contents of Zn, Fe, S and Ag of different samples (mass fraction, %)

Sample	Zn	Fe	S	Ag
Raw material	19.74	23.91	3.02	0.037
Roasted product	19.57	24.02	3.14	0.037
Leaching residues	11.56	38.09	6.04	0.081

of Fe and Ag increase from 24.02% and 0.037% in the roasted sample to 38.09% and 0.081% in the leached sample, respectively. Whilst, it is noted that the contents of S and Zn are 6.04% and 11.56%, respectively, which indicates that the main zinc phase in the leached residue is zinc sulfide. The large amount of zinc sulphide causes the high zinc grade in the leached residues of this process and limits zinc recovery. It is suggested that enhanced oxidation roasting such as over-roasting, should be applied in the initial desulphurization of zinc blend to remove zinc sulfide as possible.

4 Conclusions

1) Thermodynamic analysis of zinc ferrite shows that in the range of $V(\text{CO})/V(\text{CO}+\text{CO}_2)$ ratio of 2.68%–36.18%, zinc ferrite is preferentially decomposed into Fe_3O_4 and ZnO .

2) The optimal conditions for reduction roasting of zinc ferrite are as follows: temperature 700–750 °C, CO content 6% and $V(\text{CO})/V(\text{CO}+\text{CO}_2)$ ratio 30%. This result is consistent with the thermodynamic analysis.

3) When the optimum reduction roasting conditions of zinc ferrite are applied in the roasting of zinc leaching residue followed by acid leaching, zinc extraction rate in zinc leaching residue reaches up to 70% and iron extraction rate is only 18.4%. This result is a improvement for the treatment of ZLR, while it still needs further enhancing by adopting modified roasting process, for example, multi-stage reduction roasting–leaching process.

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铁酸锌选择性还原及其在锌浸出渣处理中的应用

余刚¹, 彭宁², 周兰³, 梁彦杰², 周晓源¹, 彭兵², 柴立元², 杨志辉²

1. 长沙有色冶金设计研究院有限公司, 长沙 410011;

2. 中南大学 冶金与环境学院, 长沙 410083;

3. 湖南大学 工商管理学院, 长沙 410082

摘要: 传统湿法炼锌过程产生大量富含有价金属资源的铁酸锌废渣, 铁分离是实现铁酸锌废渣中有价金属资源回收的关键。提出含大量铁酸锌的锌浸出渣选择性还原焙烧-浸出分离铁和锌的新方法。通过热力学分析确定铁酸锌分解过程中 Fe_3O_4 和 ZnO 产物的优势区域, 并发现 $V(\text{CO})/V(\text{CO}+\text{CO}_2)$ 比是控制铁酸锌还原焙烧产物物相的关键因素, 在 $V(\text{CO})/V(\text{CO}+\text{CO}_2)$ 比在 2.68%–36.18% 范围内, 铁酸锌优先分解生成在 Fe_3O_4 和 ZnO 。通过 TG 分析, 确定铁酸锌还原焙烧的最佳条件为焙烧温度 700–750 °C, CO 体积分数 6%, $V(\text{CO})/V(\text{CO}+\text{CO}_2)$ 30%。基于上述研究结果, 对富含铁酸锌的锌浸渣进行还原焙烧处理, 焙烧产物经酸浸后, 锌的浸出率达 70%, 铁的浸出率仅为 18.4%, 实现锌浸渣中锌和铁的有效分离。

关键词: 铁酸锌; 锌浸出渣; 还原焙烧; 金属分离

(Edited by Yun-bin HE)