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Recovery of metals from the roasted lead-zinc tailings by magnetizing roasting followed by magnetic separation



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ABSTRACT

To comprehensively reuse lead-zinc tailings and avoid environmental pollution, the enrichment of valuable metals and the recovery of iron from the roasted lead-zinc tailings (RM), the cinder after the oxidation roasting of lead-zinc tailings, were performed using magnetizing roasting followed by magnetic separation. The transformation of iron in the RM during the magnetizing roasting was investigated by studying the effects of roasting temperature and ratio of coal to RM on the iron recovery. The results showed that most of iron in the RM was transformed from Fe₂O₃ to Fe₃O₄ at the roasting temperature of 720 °C and the ratio of coal to RM of 7:100. The iron recovery rate reached 82.16%, and the iron content in the magnetic concentrate reached 62.14%. The investigation of the enrichment characteristics of the metals in the magnetizing showed that Ag, Ga, Pb and Mn could be enriched in the magnetic tailing, with enrichment ratios of 1.64, 1.39, 1.35 and 1.24, respectively, under the conditions of a roasting temperature of 720 °C and a ratio of coal to RM of 7:100. Furthermore, the transformation of metals during magnetizing roasting was investigated by analyzing the microstructure and mineralogical changes during the magnetizing roasting process. With this method, the processes of iron recovery and other metals enrichment from RM using magnetizing roasting followed by magnetic separation was proposed for the comprehensive utilization of lead-zinc tailings.

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1. Introduction

Approximately 29 million tons lead-zinc tailings were produced in China in 2009 according the pollutant generation and discharge coefficients of lead and zinc mine exploitation (National Census of Pollution Source Compilation Committee, 2011). Lead-zinc tailings contain many heavy metals and sulfides, which produce acid mine drainage (AMD) and cause serious pollution of the environment. To eliminate the environmental risk of the lead-zinc tailings and reuse the resources in the lead-zinc tailings, systematic research was conducted on the recovery of sulfur, iron and rare metals such as Ag and Ga from lead-zinc tailings by different processes including roasting, magnetic separation, leaching and solvent extraction. The recovery of sulfur from lead-zinc tailings was examined in the previous study, and the results indicated that the technology for the recovery of sulfur from lead-zinc tailings was feasible using oxidation roasting (Lei et al., 2015). However, as the waste product

* Corresponding author. E-mail address: yanbo2007@gig.ac.cn (B. Yan). from the roasting, the cinders not only occupy a great area of land but also lead to a dust problem in the surrounding environment because of their fine particle size ($-75 \mu m$) (Alp et al., 2009). In addition, large quantities of the cinders containing many hazardous heavy metals are landfilled or dumped as solid wastes, which pose a potential risk to the environment and human health in the long term (Edraki et al., 2014).

Many measures such as phytoremediation technology (Luo et al., 2016) and cement production (Onuaguluchi and Eren, 2016) have been taken to avoid environmental pollution and to recover a resource from the cinder. However, only low value-added products can be obtained (He et al., 2010). To achieve a great increase in its added value, the recovery of iron and other valuable metals including lead, silver and gallium from the cinders that contain many valuable components is necessary and important. To reduce the waste product in the processes and increase efficiency in the recovery of lead-zinc tailings, a sustainable technical process should be developed. Many physical processes have been developed such as gravity separation (Abdrakhimov et al., 2006) and froth flotation (Liu et al., 2007). However, the fine size distribution of pyrite cinders makes gravity separation difficult, and the low



recovery and the ecological and environmental problems of froth flotation hinder its extensive application (He et al., 2010). At present, magnetizing roasting and direct reduction are the two effective ways to recover iron from solid wastes (Li et al., 2010; Yang et al., 2011). Owing to its shorter roasting time, lower roasting temperature, and lower cost, magnetizing roasting is more widely used than direct reduction (Li et al., 2010). However, the current studies on magnetizing roasting mainly focus on the recovery of iron but pay little attention to the changes in other valuable metals such as Ag, Ga and Pb in the cinder during magnetizing roasting. In fact, in the process of magnetizing roasting, when the iron oxide transforms to a magnetite form, the other metals are also changed to different forms. Taking advantage of these transformations is helpful in the recovery of valuable metals during the recovery procedure of iron in the cinder.

To comprehensively use the lead-zinc tailings and avoid environmental pollution, the magnetizing roasting of the cinder was studied to recover iron from a magnetic concentrate and enrich other metals in magnetic tailing in this study. The transformation and enrichment characteristics of metals in the magnetizing roasting were investigated by studying the effects of roasting temperature and amount of coal added on the iron recovery and metal enrichment. It is expected that this study will provide a feasible method for reducing the waste product and avoiding the pollution of lead-zinc tailings in the processes of the metals recovery from lead-zinc tailings.

2. Materials and methods

2.1. Materials

The raw material (RM) used in this study was the roasted leadzinc tailings, which was the cinder obtained from the oxidation roasting of the lead-zinc tailings in the Fankou lead and zinc mine (Lei et al., 2015). The main mineral in the RM is hematite, which was generated from the oxidation of pyrite in the lead-zinc tailings. The samples were ground to below 200 mesh and dried at 105 ± 2 °C for 24 h. The chemical composition of RM is given in Table 1. As listed in Table 1, the total iron content was approximately 29.43%; however, the contents of SiO₂ and CaO, which were detrimental to the metal recovery, were 15.45% and 13.29%, respectively. More importantly, owing to their high added value, the valuable metals such as Ag and Ga should not be ignored. Coal was used as the reducing agent of magnetizing roasting in the experiments. The main active reagent in the magnetizing roasting was fixed carbon (55.91%), which played the role of a reducing agent in the reduction reaction.

2.2. Experimental procedures

The RM and the coal were thoroughly mixed at a ratio of coal to RM of 3:100, 5:100, 6:100, 7:100, 10:100 and 14:100. Then, the mixture was put into a porcelain crucible with a sealed cover and roasted in a muffle furnace at a required temperature (650, 680, 700, 720, 750 and 800 °C). After a given time, the roasted samples were rapidly taken out, quickly quenched with water, and then wet ground in an agate mortar. Next, the samples were separated by a

Table	1
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The composition of the cinder.

Components	SiO ₂	CaO	Al ₂ O ₃	MgO	Na ₂ O	K ₂ O
Content (%)	15.45	13.29	5.29	1.02	0.46	1.90
Components	T Fe	PbO	ZnO	MnO	Ag ^a	Ga ^a
Content (%)	29.43	0.85	0.76	0.13	39.82	91.21

^a The content unit is g/t.

magnetic separator (Model: XCGS-50) at 0.3 T for roughing and 0.1 T for concentrating.

To investigate the iron transformation during magnetic roasting, the iron recovery rate was calculated as follows:

$$R = \frac{c_c \cdot m_c}{c_0 \cdot m_0} \times 100\%$$

where *R* is the iron recovery rate, c_c is the iron content in the magnetic concentrate, m_c is the mass of the magnetic concentrate, c_0 is the iron content in the RM, and m_0 is the mass of the RM. To measure the enrichment effect of the valuable metals in the magnetic tailing, the enrichment ratio, which is the ratio of the metal content in the magnetic tailing to the metal content in the RM, was used. The enrichment ratio was calculated as follows:

$$E = c_t/c_0$$

where *E* is the enrichment ratio of the metal, c_t is the metal content in the magnetic tailings, and c_0 is the metal content in the RM.

2.3. Analytical methods

The mineral phases of zinc in the magnetic concentrate and tailings were determined using the chemical analysis method. In this method, a sequence of extractants (H₂O, ammonium acetate, acetic acid and saturated bromine water) was used to extract the different mineral phases of zinc (ZnSO₄, ZnO, ZnSiO₃ and ZnS). The residue was analyzed to determine the ZnFe₂O₄ content.

To determine the metals in the magnetic concentrate and tailings, the solid samples were digested with a Microwave Digestion System (WX-8000; Shanghai Yi-Yao Instruments, Shanghai, China) according to method 3052 (US EPA, 1996). The metals were determined using an atomic absorption spectrophotometer (AAS, Hitachi ZA3000). All the above experimental steps were performed in triplicate, and the average of these results was used as the final value. The microstructures of the samples were observed by scanning electron microscopy (SEM). The compositional analyses were carried out using an energy dispersion system (EDS) with the SEM. The mineralogical phase analysis was carried out using an X-ray diffractometer (XRD, MiniFlex 600, Rigaku). XPS analyses were performed using an X-ray photoelectron spectrometer (XPS, ESCALAB 250).

HSC Chemistry 5.0 software (Outokumpu Research Oy, Pori, Finland) was employed to carry out conventional thermodynamic calculations based on the minimization of the Gibbs free energy. These calculations were used to evaluate the possible influence of the reaction temperature and the amount of coal on the speciation of iron and other metals during magnetizing roasting.

3. Result and discussion

3.1. Transformation of iron in the magnetizing roasting

3.1.1. Mechanism of magnetizing roasting

As the reducing agent, the carbon in the coal played an important role in the magnetizing roasting. The main chemical reactions are as follows:

$$C(s) + O_2(g) = 2CO(g)$$
 (1)

$$3Fe_2O_3(s) + C(s) = 2Fe_3O_4(s) + CO(g)$$
 (2)

$$3Fe_2O_3(s) + CO(g) = 2Fe_3O_4(s) + CO_2(g)$$
(3)



Fig. 1. Effects of CO and temperature on the products of the reduction reactions.



Fig. 2. Effect of the roasting temperature on iron recovery.

$$C(s) + CO_2(g) = 2CO(g) \tag{4}$$

Due to the limited contact between the solid Fe_2O_3 and the solid carbon and the easy diffusion of CO gases from the carbon to the surface of Fe_2O_3 , the main reduction reactions in the roasting are the reaction between the solid Fe_2O_3 and the CO gas. However, the following reactions may occur when more CO is available than the required in magnetizing roasting(Yu and Qi, 2011):

$$Fe_3O_4 + CO(g) = 3FeO + CO_2(g)$$
⁽⁵⁾

$$FeO + CO(g) = Fe + CO_2(g)$$
(6)

$$1/4Fe_{3}O_{4} + CO(g) = 3/4Fe + CO_{2}(g)$$
(7)

To investigate the effects of CO and temperature on the products of the reduction reactions, the Van't Hoff equation and reaction quotient equation were used:

$$\Delta G = \Delta G^{\theta} + RT lnQ \tag{8}$$

$$Q = \frac{P_{CO_2} / P^{\theta}}{P_{CO} / P^{\theta}}$$
(9)

where ΔG^{θ} is the standard Gibbs free energy change of the reaction, J mol⁻¹; ΔG is the Gibbs free energy change of the reaction, J mol⁻¹; R is the ideal gas constant, 8.314 J mol⁻¹ K⁻¹; T is the temperature of the reaction, K; Q is the reaction quotient; and P_{CO} and P_{CO_2} are partial pressure of CO and CO₂, respectively.

When the reaction is at equilibrium, ΔG is equal to zero, and the following equations can be derived:

$$\frac{1 - P_{CO}}{P_{CO}} = \exp\left(-\frac{\Delta G^{\theta}}{RT}\right)$$
(10)

$$P_{CO} = \frac{1}{1 + \exp\left(-\frac{\Delta G^{\theta}}{RT}\right)} \tag{11}$$

According to Reactions (5)–(7) and Reaction (3), as well as Equation (11), the effects of CO and temperature on the products of the reduction reactions can be depicted as Fig. 1 (Yu and Qi, 2011). The reduction of Fe₂O₃ to magnetite Fe₃O₄ is easy because the equilibrium concentration of CO is very low at any temperature in Reaction (3). However, a high concentration of CO should be avoided because it will lead to the generation of Fe. Moreover, when the temperature is too high, the over-reduction of Fe₃O₄ to FeO may occur. Thus, the temperature and the amount of coal are two key factors influencing iron transformation. Therefore, the effects of the roasting temperature and the amount of coal added on the iron transformation were studied.

3.1.2. Effect of the roasting temperature on the iron transformation

The experiments investigating the effects of the roasting temperature were conducted at temperatures ranging from 650 to $800 \degree C$ for 30 min with a ratio of coal to RM of 7:100. To obtain the result of the iron transformation, the samples after roasting were



w material

Fig. 3. SEM image of the RM and the roasted sample.



Fig. 4. Effect of the ratio of coal to RM on iron recovery.

ground and separated by a magnetic separator. The iron content in the magnetic concentrate and iron recovery rate were used to measure the efficiency of iron transformation. As shown in Fig. 2, the iron content in the magnetic concentrate and the iron recovery rate increased with increase in the roasting temperature from 650 to 720 °C. This result was likely because the low roasting temperature hindered the reaction between Fe₂O₃ and CO, resulting in the incompletion of the reduction reaction. When the roasting temperature increased, the reaction was accelerated, which promoted the generation of Fe₃O₄. As the roasting temperature increased to 720 °C, the iron content in the magnetic concentrate was 62.14%, and the iron recovery rate reached 82.16%. It indicated that most of the Fe₂O₃ in the RM was transformed to the Fe₃O₄. However, when the roasting temperature was higher than 720 °C, the iron content in the magnetic concentrate and the iron recovery rate slowly decreased. This effect could be explained by the fact that the excessively high temperature led to the over-reduction, which agreed well with the depiction in Fig. 1. As a result, the formation of



Fig. 6. Ellingham diagram of the reduction of metals in the presence of CO.

wustite (Guo et al., 2004; Uwadiale and Whewell, 1988), which is difficult to recycle owing to its weakly magnetic properties, hindered the separation of iron from the gangue minerals. In order to further investigate the microstructures changes of the samples during magnetizing roasting, the SEM images of the samples were analyzed. As shown in Fig. 3, compared with the RM, the roasted sample was lightly sintered. Some components of the material would reach a molten state at a high temperature and result in the formation of agglomeration, which decreased the porosity of the materials and blocked the diffusion of CO to the surface of Fe₂O₃ (Zhang et al., 2012).

3.1.3. Effect of the ratio of coal to RM on the iron transformation

The experiments investigating the effects of the ratio of coal to RM were conducted at 720 °C for 30 min with different coal ratios. After roasting, the samples were ground and



Fig. 5. The XPS spectrum of Fe 2p peaks of the RM (a), roasted sample (b), magnetic concentrate (c) and magnetic tailing (d).



Fig. 7. Effect of the roasting temperature on the enrichment ratio of metals.

separated by a magnetic separator. As shown in Fig. 4, the iron content in the magnetic concentrate and the iron recovery rate rapidly increased as the ratio of coal to RM increased from 3:100 to 7:100 and then slightly decreased as the ratio of coal continued to increase. The low iron recovery rate at a low addition of coal could be explained by the fact that the carbon was insufficient and the reduction reaction was incomplete. However, when the coal was too much, as shown in Fig. 1, Fe₃O₄ would be over-reduced and transformed to ferrous oxide, which is weakly magnetic, resulting in decreases of both the recovery rate and the iron content in the magnetic concentrate. Therefore, the high ratio of coal to RM is unfavorable for the iron transformation process.

To further verify the transformation of Fe, the XPS spectra of Fe 2p peaks of the RM, roasted sample, magnetic concentrate and magnetic tailing are presented in Fig. 5. The XPS spectrum of the RM shows that the binding energy of Fe $2p_{3/2}$ is 710.7 eV and the Fe $2p_{3/2}$ peak has an associated satellite peak (718.6 eV). It is obvious that the form of Fe in the RM is Fe₂O₃. However, the XPS spectra of the roasted sample and magnetic concentrate do not have satellite peaks, which means that the form of Fe in the roasted sample and magnetic transformed to Fe₃O₄ (Muhler et al., 1992; Yamashita and Hayes, 2008).



Fig. 8. The mineral phase of Zn in the products.

3.2. Enrichment characteristics of other metals in the magnetizing roasting

3.2.1. The transformation of other metals in the magnetizing roasting

To investigate the transformation of the other valuable metals. the possible reduction reactions were analyzed through an Ellingham diagram (Castro et al., 2004). Fig. 6 shows the ΔG values for the oxidation of some metals as a function of temperature. In Fig. 6, the oxidation of the metal is easy when the ΔG is negative, which means that the corresponding metal oxide is difficult to be reduced. Because carbon was used as a reducing agent, the reduction of the metal would occur to the right of the point where the carbon and the metal oxidation lines cross. It can be seen from Fig. 6 that the curves of Fe₃O₄, Pb and Ag were above the curves of C and CO, namely, Fe₂O₃, PbO and Ag₂O were easy to be reduced. On the contrary, MnO, Ga₂O₃ and ZnO were difficult to be reduced. Besides the reduction reaction between the metals and the reductant, some of the metals might react with iron compounds and produce magnetic materials, which would make it difficult to separate from iron by magnetic separation. To investigate the transformation of different metals during magnetizing roasting, the effects of the ratio of coal to RM and the roasting temperature were investigated.

3.2.2. Effect of the roasting temperature on the metal enrichment

The transformation of metals during the magnetizing roasting can be revealed by the enrichment efficiencies of the metals in the magnetic tailing. The effects of the roasting temperature on the enrichment ratios of the metals are shown in Fig. 7. The results in Fig. 7 indicate that the enrichment ratios of Ag, Ga, Pb and Mn increased as the roasting temperature increased from 650 to 720 °C and then gradually decreased. This trend was similar to the trends of the iron recovery rate and the iron content in the magnetic concentrate at different roasting temperatures. It can be explained by the fact that the more production of the Fe₃O₄ during magnetizing roasting facilitates the separation of the nonmagnetic metals from iron during magnetic separation. When the roasting temperature was 720 °C, the enrichment ratios of Ag, Ga, Pb and Mn were 1.64, 1.39, 1.35 and 1.24, respectively. That is, Ag, Ga, Pb and Mn could be enriched in the magnetic tailing. However, in contrast to Ag, Ga, Pb and Mn, the enrichment ratio of Zn in the magnetic tailing decreased as the roasting temperature increased. The enrichment ratio of Zn was less than 1 at temperatures ranging from 650 to 800 °C. It could be deduced that the Zn reacted with



Fig. 9. Effect of the ratio of coal to RM on the enrichment ratio of metals.



Fig. 10. SEM image and the corresponding EDS spectrum of the magnetic concentrate and the magnetic tailing.

iron compounds and produced magnetic materials in the magnetizing roasting or was combined with the iron compounds, which made it difficult to separate.

To investigate the transformation of Zn during the magnetizing roasting, the main phases of Zn in the magnetic concentrate and tailings were analyzed. As shown in Fig. 8, the proportion of ZnFe₂O₄ was significantly larger than the proportions of the other zinc phases (ZnSiO₃, ZnO and ZnS). Moreover, the magnetic concentrate contained more ZnFe₂O₄ than the magnetic tailing, which might be because ZnFe₂O₄ is a magnetic material (Deraz and Alarifi, 2012) and exists in the form of solid solution of zinc ferrite (ZnFe₂O₄) and magnetite (Fe₃O₄) (Graydon and Kirk, 1988). As a result, the zinc tended to stay in the magnetic concentrate rather than magnetic tailings after magnetic separation. Therefore, the high temperature should be avoided in the magnetic roasting

because of the easier reaction between zinc and iron compounds at a higher temperature.

3.2.3. Effect of the ratio of coal to RM on the metal enrichment

The effects of the ratio of coal to RM on the enrichment ratios of the metals are shown in Fig. 9. As shown in Fig. 9, the enrichment ratios of Ag, Ga, Pb and Mn increased as the ratio of coal to RM increased from 3:100 to 7:100 and then slightly decreased as the ratio continued to increase. The trend was similar to the trends of the iron recovery rate and the iron content in the magnetic concentrate at different ratios of coal to RM. Besides, Ag, Ga, Pb and Mn tended to stay in the magnetic tailings according to the enrichment ratios of the metals. However, the enrichment ratio of Zn increased as the ratio of coal to RM increased from 3:100 to 14:100. Moreover, Zn tended to stay in



Fig. 11. The processes of iron recovery and other metal enrichment using magnetizing roasting and magnetic separation.



Fig. 12. XRD pattern of RM, roasted sample, magnetic concentrate and magnetic tailing.

the magnetic concentrate at the ratio of coal to RM ranging from 3:100 to 10:100 and tended to stay in the magnetic tailings as the ratio continued to increase to 14:100. It may be attributed to the transformation of the zinc ferrite during the magnetizing roasting. When the ratio of coal to RM was high, the zinc ferrite might be reduced to ZnO and Fe_3O_4 (Yan et al., 2013). The reaction with CO could occur as follows (Peng et al., 2012; Wu et al., 2014):

 $3ZnFe_2O_4(s) + CO(g) = 3ZnO(s) + 2Fe_3O_4(s) + CO_2(g)$

It can be deduced from the reaction that the reduction of zinc ferrite was promoted by the increasing coal and caused the separation of zinc from iron after magnetic separation.

In order to further analyze the transformation of metals during the magnetizing roasting, the SEM image and the corresponding EDS spectrum of the magnetic concentrate and the magnetic tailing are shown in Fig. 10. As shown in Fig. 10, iron was the main metal element and other elements such as Si, Pb and Ca were the minority in the magnetic concentrate. It could be also seen that the iron in the magnetic tailing clearly decreased and the Pb significantly increased, which is consistent with the results of the transformation of the metals.

3.3. The processes of iron recovery and other metals enrichment

A simplified process flowsheet of iron recovery and other metals

enrichment using magnetizing roasting and magnetic separation is shown in Fig. 11. The RM was first mixed with the coal at the ratio of coal to RM of 7:100 and then roasted at the temperature of 720 °C. After that, the roasted sample was separated by a magnetic separator. The iron was recovered from the magnetic concentrate and the other metals were enriched in the magnetic tailing.

To analyze the products in the processes, the XRD patterns of RM, roasted sample, magnetic concentrate and magnetic tailing are depicted in Fig. 12. As depicted in Fig. 12, the main phase of the RM was Fe₂O₃, whereas the main phase of the roasted sample was Fe₃O₄, which indicated that almost all Fe₂O₃ was transformed to Fe₃O₄ after magnetizing roasting. Additionally, it can be seen from the XRD patterns of magnetic concentrate and magnetic tailing that most Fe₃O₄ was transferred to the magnetic concentrate, whereas the gangue minerals were left in the magnetic tailing.

The metal contents of the magnetic concentrate and tailings are listed in Table 2. As shown in Table 2, the iron content in the magnetic concentrate reached 62.14%, which was significantly higher than the iron content in the magnetic tailing. The grade of

Table 2	
The metal content of the magnetic concentrate and tai	ling.

Metals	Ag (g/t)	Ga (g/t)	Fe (%)	Pb (%)	Zn (%)	Mn (%)
RM	34.38	84.72	29.34	0.807	0.609	0.104
Magnetic concentrate	17.38	57.43	62.14	0.542	0.796	0.089
Magnetic tailing	56.33	117.6	9.54	1.09	0.571	0.129

iron concentrate could meet the requirement of the Code for Design of Blast Furnace Ironmaking Technology (GB 50427-2008) in China (China Metallurgical Construction Association, 2008). Thus, the magnetic concentrate could be reused for ironmaking. Moreover, the contents of Ag, Ga, Pb, and Mn in the magnetic tailing were significantly higher than those in the magnetic concentrate. Therefore, the further recovery of valuable metals from the magnetic tailing became necessary and possible.

The results of the metal transformation during magnetizing roasting demonstrated the feasibility of the flowsheet (Fig. 11). However, in order to carry out the pilot-scale study and to further improve the iron recovery and other metal enrichment rates, a further research is needed to determine the optimum parameters of the magnetic separation process.

4. Conclusions

The transformation and enrichment characteristics of metals in RM was affected by the roasting temperature and the amount of coal added in the magnetizing roasting. Most of iron in the RM was transformed from Fe₂O₃ to Fe₃O₄ at the roasting temperature of 720 °C and the ratio of coal to RM of 7:100. After magnetic separation, the iron recovery rate reached 82.16%, and the iron content in the magnetic concentrate reached 62.14%. The investigation of the enrichment characteristics of the metals in the magnetizing showed that Ag, Ga, Pb and Mn could be enriched in the magnetic tailing, with enrichment ratios of 1.64, 1.39, 1.35 and 1.24, respectively, under the conditions of a roasting temperature of 720 °C and a ratio of coal to RM of 7:100. The processes of iron recovery and other metals enrichment from RM using magnetizing roasting and magnetic separation was proposed for the comprehensive utilization of lead-zinc tailings. The results of the analysis of the products showed that the Ag, Ga, Pb and Mn could be enriched for further recovery and that the magnetic concentrate could be reused for ironmaking. The study demonstrated the feasibility of the technology for the recovery of iron and the enrichment of valuable metals from the cinder after oxidation roasting of lead-zinc tailings using magnetizing roasting followed by magnetic separation.

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