Probing Study on Separating Pb, Zn, and Fe from Lead Slag by Coal-based Direct Reduction

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Water-quenched lead slags are classified as hazardous materials because they contain a certain amount of heavy metals. In this study, coal-based direct reduction followed by magnetic separation was presented to separate Pb, Zn, and Fe from lead slag. Lead slag was mixed with coal and additive and treated in a laboratory tube furnace. In the process of direct reduction, Pb and Zn compounds were reduced to elementary substances and entered into flue gas as dust. Fe was reduced to metallic iron and separated from roasted slag by magnetic separation. The effects of roasting temperature, roasting time, and usage of coal and additive were experimentally determined. The following optimum process parameters were proposed: weight ratios of slag, coal ratio of 30%, additive ratio of 15%, and roasting at 1 200°C for 90 min. Under these conditions, a magnetic concentrate containing 93.73% total iron and recovery rate of 80.32% was obtained. In the process of direct reduction, the Pb and Zn volatilization rates were 97.48% and 97.19%, respectively. Lead slag and roasted slags were subjected to scanning electron microscopy and X-ray diffraction to identify the reduction mechanism of Pb, Zn, and Fe during direct reduction.

KEY WORDS: lead slag; direct reduction; magnetic separation; volatilization; metallic iron.

1. Introduction

Water-quenched lead slags are produced from the treatment process of high-temperature smelting lead slags in lead and zinc metallurgy industries.1,2) Approximately 20 million tons of water-quenched lead slag is found in China, and over 1 million tons is generated annually. Lead slag is a hazardous waste containing heavy metals, such as Pb, Zn, Sb, and Cr.3,4) In China, lead slag is included in the National Dangerous Wastes Catalogue. If slag is dumped openly, toxic heavy metals are released into soil and groundwater by various agents, including rain and wind, and are hardly decomposed.5,6) As a consequence, organisms and environments become chronically and irreversibly polluted. Therefore, lead slag is harmful to living organisms,7) and its harmful effects have been extensively investigated all over the world.8–10) The results indicate Pb, Zn, and other heavy metals are absorbed by organisms via the food chain and are accounted for undesirable biochemical and physiological changes in the heart, liver, and blood.11) The recovery of valuable metals from lead slag not only allows the recycling of disused resources but also minimizes their negative effects on environments and organisms.

Nowadays, the main ways of disposing lead slag are recovery of valuable metals and their application as construction materials. Atzeni et al. and Albitar et al. have studied the possibility of using granulated lead slags replacement of sand in mortars and concretes. Atzeni et al. proved that mix design does not impair the mechanical strength of concrete. Albitar et al. indicated that incorporating 75% of granulated lead smelter slag to replace fly ash as a filler can significantly improve the compressive strength of hardened concrete. Therefore, these slags can be used as construction materials.12,13) However, this method fails to achieve the maximum value of comprehensive lead slag utilization. The valuable metals such as Pb, Zn, Cu, and Fe are still in concrete.14) On the other hand, concrete which contains the toxic heavy metals, may cause secondary pollution to the environment. In the Flemish region of Belgium, the government imposed a law to manage the use of waste materials as secondary raw materials, especially in building applications. According to the legislation, waste materials can only be used as secondary raw materials if they satisfy the required limit value of total heavy metals concentrations.15) Therefore, valuable metals should be recovered through an environmentally friendly mechanism of lead slag disposal.

Recycling valuable metals from lead slag has been widely explored. Guo et al. used indigenous moderate thermophilic bacteria to bioleach lead slag and found that >80% of As, Cu, Mn, and Zn are mobilized from lead slag under optimum conditions.16) This finding demonstrates that bioleaching can be applied to recover valuable metals and remove toxic elements from lead slag. However, this process has characteristics of long processing duration, yields low processing capacity, and involves strict leaching conditions.
Thus, this process is unsuitable for large-scale industrial production. Seignez et al. examined the recovery of Pb from lead slag by wet leaching process, and observed that Pb is strongly released under open-flow conditions, but Fe, Zn, and other valuable metals are insufficiently recycled. Zheng et al. converted Zn in lead slag to ZnS through mineralogical reconstruction with pyrite and carbon in the presence of sodium carbonate and subsequently separated ZnS from the treated slag by conventional ore beneficiation process. Their flotation test revealed that the obtained concentrate contains 32.76% Zn and the total recovery rate of Zn is 88.17%. Wang et al. applied fuming process to treat lead slag and found the Zn volatilization rate can up to 83%. Nevertheless, only one or two metals have been recycled via these processes and most valuable metals have not been recovered from lead slag. Refractory iron ores and solid wastes are always disposed through coal-based direct reduction followed by magnetic separation, which has been comprehensively investigated. In the process of coal-based direct reduction followed by magnetic separation with coal as a reductant, metals are reduced to elementary substances at high temperatures. Metallic iron can be separated from roasted slags by magnetic separation, and volatile metals vaporize into flue gas as dust. Therefore, coal-based direct reduction followed by magnetic separation process is suitable for the disposal of lead slag and recovery of valuable metals.

2. Experimental

2.1. Materials

Water-quenched lead slag was collected from Hechi in Guangxi province of China, which was generated as a waste material during the Pb production. The lead slag, a granulated material with particle sizes < 6 mm, was crushed and screened to produce < 2 mm particles as testing samples.

The chemical compositions of the samples were characterized through X-ray fluorescence spectrometry (Rigaku, model ZSX Primus II). As shown in Table 1, Fe (20.1%), Zn (4.35%), Ca (9.63%), Al (4.91%), Na (3.45%), and Pb (0.739%) are the main metallic elements and O (39.1%) and Si (12.6%) are the main non-metallic elements. The phases of lead slag were detected by X-ray diffraction (XRD). XRD results (Fig. 1) revealed that the main crystalline phases of the samples are maghemite, magnetite, magnesiostilite, zinc ferrite, zinc aluminum ferrite, wollastonite, andradite, and small amounts of lead silicate. The composition and texture of the slag particles were evaluated by scanning electron microscopy (SEM)-energy-dispersive X-ray spectroscopy (EDS). Figure 2(a) illustrates the backscattering SEM images of lead slag, with two regions exhibiting different tonalities in the sample. The EDS spectrum in Fig. 2(b) indicates that the white spots are Fe and Zn phases and the light gray regions are gangue and some Fe, Zn, and Al phases. These properties showed that lead slag exhibits complex association and dissemination. These findings suggest that valuable metals, such as Pb, Zn, and Fe, are difficult to be recovered by traditional beneficitation methods.

The coal used in this study was lignite, and its compositions are listed in Table 2. Fixed carbon (48.29%) was the main active reagent in direct reduction. The size analysis of the lignite used for the experiments are shown in Table 3. The size of lignite are main −0.6 mm. Analytical-grade lime (CaO) was used as a flux. The size analysis of lead slag are shown in Table 4.

2.2. Methods

Experiments were conducted in a tubular resistance furnace (MXG-1400) with a maximum operating temperature of 1 400°C. The tube was composed of corundum with an internal diameter of 50 mm. The temperature of the tube furnace was maintained within ±1°C of the set temperature. Lead slag (25 g) was thoroughly mixed with lignite and CaO at different ratios. The mixture was placed in a corundum crucible in the tube furnace at various temperatures and times without purge gas to the tube furnace during roasting. Afterward, the crucible was removed from the furnace and cooled in a desiccator. The roasted slags were weighed, ground, and analyzed via a chemical method to analyze the residual Pb and Zn concentrations and the percentage Fe metallization. The volatilization rates of Pb and Zn were calculated as follows:

$$V = \frac{(M_0 - M_f)}{M_0} \times 100\%$$

where $M_0$ is the mass of lead slag, $C_0$ is the Pb or Zn content in lead slag, $M_f$ is the mass of the roasted sample, and $C_1$ is

| Elements | Content | Elements | Content |
|----------|---------|----------|---------|
| Fe       | 20.1    | K        | 0.361   |
| Pb       | 0.739   | Mg       | 0.606   |
| Zn       | 4.35    | Ti       | 0.349   |
| Cu       | 0.179   | Mn       | 0.426   |
| Sb       | 1.78    | Sn       | 0.395   |
| Si       | 12.6    | Sr       | 0.0288  |
| P        | 0.483   | Ba       | 0.218   |
| S        | 0.0438  | As       | 0.154   |
| Na       | 3.45    | Co       | 0.0265  |
| Ca       | 9.63    | Cr       | 0.0326  |
| Al       | 4.91    | O        | 39.1    |

Fig. 1. X-ray diffraction pattern of the lead slag.
the content of Pb or Zn in the roasted slag. The percentage metallization of Fe was calculated as follows:

$$ M = \frac{m_{Fe}}{m_{Fe} + T_{Fe}} \times 100\% $$

where $m_{Fe}$ is the content of metallic iron in the roasted sample and $T_{Fe}$ is the content of total iron in lead slag.

The primary compositions of volatiles in the process of direct reduction were PbOx and ZnO, which can be separated via a chemical method to obtain high-grade PbOx and ZnO.

The roasted slags underwent wet grinding and two-stage wet magnetic separation with a Davis tube. The magnetic field intensities of wet magnetic separation were 143 kA/m of the first stage and 64 kA/m of the second stage, which were determined by the experimental results. The total Fe content and metallized iron content in magnetic concentrate were analyzed by the chemical method.

The samples were observed by using an XRD (Rigaku DMAX-RB, Cu Kα) and a high-power optical microscope (Leica 4000 P). They were further analyzed by SEM and EDS (Carl Zeiss EVO 18).

### 2.3. Experimental Mechanisms

In this study, lignite acted as reducing agent, and the purpose of coal-based direct reduction is to reduce ferrous minerals to metallic iron, Zn bearing minerals to metallic Zn, and Pb bearing minerals to metallic Pb. The software HSC 6.0 was used to calculate the equilibrium of phase in Gibbs free energy minimization. The reaction mechanism can be expressed with the following equations:
As Fig. 3 shows, the correlation of standard free energy with temperature in Eqs. (2-1) to (2-9). Accordingly, a higher temperature corresponds to lower standard free energy. This finding indicates that reduction reaction possibly occurs. The change in standard free energy for the direct reduction of ZnO to Zn is above 950 °C and the starting temperatures of other reactions are below 720 °C. Therefore, direct reduction occurs at a certain temperature. In Eq. (2-8) compared with Eq. (2-9) in Fig. 3, the addition of CaO can decrease the starting temperature of the reduction of Pb2SiO4 to Pb, which can promote reduction reaction. On the basis of these results, we focused on four factors affecting recovery process: roasting temperature, roasting time, lignite ratio (weight percentage of coal to lead slag), and CaO ratio (weight percentage of CaO to slag).

3. Results and Discussion

3.1. Direct Reduction and Separation Process

3.1.1. Effect of Roasting Temperature

The effect of roasting temperature on iron recovery and reductant dose on the volatilization rates of Pb and Zn are shown in Fig. 4. The mixtures consisting of lead slag, lignite dose of 20%, and CaO with a ratio of 12% were roasted for 60 min at various temperatures.

Roasting temperature is a key factor in metal reduction and Pb and Zn volatilization. In Fig. 4, the total iron grade and recovery rate of magnetic concentrates increased gradually as roasting temperature increased. The total iron grade of magnetic concentrates slightly changed as temperature increased to > 1200 °C. The Fe metallization rate increased gradually as temperature increased from 1100 °C to 1200 °C and then decreased slightly at > 1200 °C. The Pb and Zn volatilization rates increased rapidly when temperature increased from 1100 °C to 1250 °C and increased slightly when temperature increased to > 1250 °C. Overall, the appropriate roasting temperature could be obtained in the range of 1200 °C to 1250 °C. In Fig. 3, the reduction reactions 2-1 to 2-9 can be improved as temperature increased, but as the roasting temperature exceeded 1250 °C, a distinct sintering phenomenon occurred in the roasted slags and the products were difficult to be crushed. Considering energy consumption, the optimum roasting temperature was 1200 °C.

3.1.2. Effect of Lignite Ratio

Various lignite ratios were examined to optimize the amount of lignite addition. The following parameters were kept constant: CaO ratio of 12% and the mixtures were roasted at 1200 °C for 60 min. The effect of lignite is shown in Fig. 5.

Fig. 3. Correlation of standard free energy (ΔG° f) with temperature in Eqs. (2-1) to (2-9).

Fig. 4. Effect of roasting temperature.
In Fig. 5, the total iron grade, recovery rate, Fe metallization rate, and Zn volatilization rate were apparently increased when the lignite ratio increased from 10% to 25% and it changed slightly as the lignite ratio was >25%. Iron grade decreased at the lignite ratio of >30%. Therefore, the highest total iron grade was obtained at a lignite ratio of 30% and the Pb volatilization rate decreased when the lignite ratio was >25%. Increasing the ratio of lignite can strengthen the reducing atmosphere and more Fe is reduced to metallic iron. A lower quantity of low melting point material is produced. As a result, the diffusion of Fe and Pb ions to the surface of the metallic iron particles is hindered and the growth of the metallic iron particles is inhibited. This leads to the formation of many small Fe and Pb particles.\textsuperscript{28,29) Most of the small iron particles and Pb simple substance are connected with each other to form a network with gangue trapped within the particles.

Above all, Lignite as a reductant in direct reduction process reduced the metals to their elementary substances. So the optimum lignite ratio was 30%.

3.1.3. Effect of CaO Ratio

Various CaO ratios were investigated at a roasting temperature of 1200°C, lignite ratio of 30%, and roasting time of 60 min to investigate the effect of CaO ratio on the direct reduction process. The results are presented in Fig. 6.

As shown in Fig. 6, the iron metallization rate and the Pb and Zn volatilization rates increased as the CaO ratio increased. At CaO ratio of >15%, the iron metallization rate and the Pb volatilization rate decreased significantly, whereas the Zn volatilization rate was slightly changed. The total iron grade and recovery rate of magnetic concentrate were also increased as the CaO ratio increased. The recovery rate decreased and the grade of concentrate slightly changed at a CaO ratio of >12%. Increasing the ratio of CaO helps to improve the activity of ZnO, thus promoting the volatilization rate of Zn. Due to the activity of PbO can increase with ZnO content, Pb volatilization rate decreased as the volatilization rate of ZnO increasing. In other hand, the melting point of the slag increased and the fluidity reduced by adding excess of CaO. Hence, excess CaO ratio negatively affects Pb volatilization and iron reduction. Overall, 15% is the optimum ratio of CaO.

3.1.4. Effect of Roasting Time

The lignite ratio of 30% and the CaO ratio of 15% were kept to investigate the effect of roasting time on direct reduction. The mixtures were roasted at 1200°C for different times. The results are shown in Fig. 7.

Roasting time significantly affected the reactivity of metals in lead slag. At a roasting time of less than 60 min, all of the indicators were significantly increased as roasting time was prolonged. The recovery rate and total iron grade of magnetic concentrate slightly changed when the roasting time was >90 min. The Pb volatilization rate and the Fe metallization rate were significantly decreased after roasting time >90 min. The Zn volatilization rate continuously increased until roasting time was >120 min, then, the Zn volatilization rate slightly changed. Therefore, the suitable roasting time was 90 min.

As mentioned above, the following optimum parameters were obtained: lignite as a reductant, CaO as an additive, lignite ratio of 30%, CaO ratio of 15%, reduction roasting at 1200°C for 90 min, and separation of metallic iron by wet grinding and two-stage wet magnetic separation. Under these conditions, a magnetic concentrate containing 93.73% total iron and recovery rate of 80.32% were obtained. The Pb volatilization rate was 97.48% and the Zn volatilization rate was 97.19% in direct reduction process.
3.2. Phase Transformations of Lead, Zinc and Iron in Lead Slag during Direct Reduction Process

3.2.1. Volatilization Dust Analysis

The collected volatilization dust was detected by XRD and atomic absorption spectroscopy (AAS). In Fig. 8 and Table 3, the main phases in volatilization dust are ZnO, PbO, Pb2O, and elementary Pb. The main elements are Pb and Zn in volatilization dust. The AAS results (Table 3) indicate that Pb and Zn are 10.49% and 61.37%, respectively. Therefore, the content of PbO, Pb2O, and ZnO are over 71.86%. The flue gas dust obtained by direct reduction can be high-purity dust, which could be regarded as a raw material for industrial applications. The volatilization temperatures of Pb and Zn are 627°C and 907°C, respectively. The melting points of PbO and ZnO are 883°C and 1 975°C, respectively. So Pb and Zn in their elementary substance forms are more easily volatilized than oxide. Pb and Zn in lead slag are also reduced to their elementary substances and then entered into flue gas as dust in direct reduction process. Hence, the transfer behavior of lead and zinc in lead slag was observed as follows: Pb2SiO4 → Pb → PbO and ZnFe2O4 → ZnO → Zn → ZnO.

3.2.2. Reduction Behavior of Iron Compounds in Direct Reduction Process

Lead slag was roasted at 1 200°C at different roasting times of 30 min to 180 min with a lignite ratio of 30% and a CaO ratio of 15% to reveal the reduction behavior of lead slag during direct reduction. The roasted slags were analyzed by XRD and SEM. The XRD patterns of the roasted slags are shown in Fig. 9. The main mineral phases were iron, gehlenite, lead oxide, and calcium lead oxide. The diffraction peak intensities of iron and gehlenite increased as the roasting time was extended. The phases of the raw ore and the roasted slags differed. In particular, the main phases of the raw ore disappeared and many phases of the roasted slags were generated. Therefore, complex chemical reactions occur during coal-based direct reduction process. Zinc ferrite is reduced to zinc oxide and magnetite. Magnesioferrite is reduced to magnetite and magnesium silicate. Magnetite and maghemite are reduced to iron. Calcium aluminate, wollastonite, and calcium aluminum silicate as gangue minerals generated gehlenite in direct reduction process. Metallic iron is easily recovered by low-intensity magnetic separation after grinding. Pb and Zn in lead slag are reduced to their elementary substance forms and entered into flue gas as dust. Therefore, Pb, Zn, and Fe were efficiently separated from lead slag. The diffraction peak intensity of calcium lead oxide was significantly enhanced when roasting time was >90 min, so calcium lead oxide was generated as extend the roasting time for more than 90 min, and it restricted the volatilization of Pb. In Fig. 7, the Pb volatilization rate decreased when the roasting time was >90 min. Gehlenite was the main gangue mineral in the roasted slags, which was generated by calcium aluminate, wollastonite, and calcium aluminum silicate at high temperature.

As shown in Fig. 10, the XRD patterns of the magnetic concentrate of the roasted slag by wet magnetic separation. In Fig. 10, the main phases in the magnetic concentrate are metallic iron and gehlenite, anorthite, quartz, and forsterite as gangue. Fe is reduced to metallic iron during direct reduction. Furthermore, metallic iron can be separated by magnetic separation.

In Fig. 11, the SEM image and EDS results of the roasted sample under the following conditions: mixture roasted at 1 200°C for 60 min, lignite ratio of 30%, and CaO ratio of 15%. Three main kinds of tonalities are observed in the

![Fig. 8. X-ray diffraction pattern for volatilization dust.](image)

![Fig. 9. X-ray diffraction of raw ore and roasted slags in different roasting times.](image)

![Fig. 10. X-ray diffraction of magnetic concentrate.](image)
SEM image. Therefore, the EDS test points were selected from three different kinds of tonalities. The EDS results indicate that the main elements in several areas, such as Point-1, are Fe and C, and these areas were brighter than other areas. C was coated as a conductive material on the samples. Considering the iron phase in the magnetic concentrate shown in Fig. 10, which can be regarded as metallic iron. The main elements in other areas, such as Point-2 and Point-3, are Ca, Na, Mg, Si, Al, O, and Fe. These phases can be considered as gangue and unreduced Fe. The main metallic iron distributed in the roasted slags yielded sizes of approximately 20 \( \mu \text{m} \) to 60 \( \mu \text{m} \) at the edge of the grains. Metallic iron particles possess clear boundaries between gangue grains and major iron particles, and these particles are not embedded in gangue grains. Therefore, metallic iron particles are easily separated by grinding. The reduction behaviors of iron in lead slag were as follows: \( \text{ZnFe}_2\text{O}_4 \rightarrow \text{Fe}_3\text{O}_4 \), \( \text{MgFe}_2\text{O}_4 \rightarrow \text{Fe}_3\text{O}_4 \), \( \text{Fe}_3\text{O}_4 \rightarrow \text{FeO} \rightarrow \text{Fe} \), and \( \text{Fe}_2\text{O}_3 \rightarrow \text{FeO} \rightarrow \text{Fe} \).

Metallic iron initiation and growth were observed under a high-power optical microscope (Fig. 12). Lead slag was mixed with 15% CaO and 30% lignite and was roasted at

![Fig. 11. SEM image and EDS results of roasted sample.](image1)

![Fig. 12. Optical microscopy images of roasted slags at different roasting times.](image2)
1 200°C for 30, 60, 90, and 120 min. Based on Fe in Fig. 11, the bright white areas in Fig. 12 are metallic iron. Image (a) shows that the Fe compounds were reduced to metallic iron, and the metallic iron particles were distributed among the roasted slags with different sizes. Many iron particles were connected on the edge of the samples and formed strips. This finding indicated that reduction reactions may occur from the outside to the core, and the reduced iron attached to metallic iron on the outside. Many metallic iron particles distributed in the roasted slags with small sizes were not easily separated from gangue by grinding. Image (b) illustrates that the particles of metallic iron continuously aggregate and grow. Many joined crystals were also formed at this roasting time. Image (c) reveals that more of the joined crystals disappeared, and metallic iron was mostly present in the form of near-spherical particles. The size of the iron particles ranged from 50 μm to 100 μm and thus were easily separated through grinding-magnetic separation. Image (d) indicates that near-spherical iron particles continuously grow and become connected to form large iron particles and combined crystals in the form of thick strips. However, these iron particles are not easily separated from gangue by grinding. Larger iron particles may increase grinding consumption and mix with some small gangue particles via crumbling during grinding.

In conclusion, metallic iron particles aggregated and grew to form combined strip crystals as roasting time increased (Fig. 12). At a roasting time of 90 min, the metallic iron appeared as near-spherical particles and became loosely scattered on the roasted slag. The clear boundaries between metallic iron particles and gangue were easily observed. Metallic iron particles were not embedded in gangue. By comparison, the iron particles became too large to separate from the roasted slags in appropriate sizes by grinding when the roasting time was extended to 120 min. Therefore, the optimum roasting time was 90 min.

4. Conclusions

The following conclusions can be drawn from the present study:

(1) The process of coal-based direct reduction followed by magnetic separation was conducted to extract metallic iron and separate Pb and Zn from lead slag. Metallic iron powder with 93.73% total iron and recovery rate of 80.32% was obtained under the following optimum conditions: lignite as a reductant, CaO as an additive, lignite ratio of 30%, CaO ratio of 15%, and reduction roasting at 1 200°C for 90 min.

(2) Pb and Zn in lead slag were reduced to simple substances and entered into flue gas as dust with Pb volatilization rate of 97.48% and Zn volatilization rate of 97.19% under the optimum conditions in the process of coal-based direct reduction. The elemental contents of Pb and Zn in the flue gas dust were 10.49% and 61.37%, respectively. The transfer behaviors of Pb and Zn in lead slag were as follows: PbSiO₄ → Pb → PbO and ZnFe₂O₄ → ZnO → Zn → ZnO.

(3) Fe-containing compounds were reduced to metallic iron, which can be separated through secondary wet grinding and two-stage wet magnetic separation. The reduction behaviors of Fe in lead slag were as follows: ZnFe₂O₄ → Fe₂O₃, MgFe₂O₄ → Fe₂O₃, Fe₂O₃ → FeO → Fe, and FeO → Fe → Fe.

(4) In the process of coal-based direct reduction, the metallic iron particles aggregated and grew as roasting time increased. As a result, combined crystals in the shape of strips were formed. The metallic iron appeared as near-spherical particles and became loosely scattered on the roasted slag with clear boundaries when the roasting time was extended to 90 min. Therefore, metallic iron particles in this roasted slag were easily separated from gangue by grinding.

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Table 5. AAS results of volatilization dust (mass%).

| Elements | Pb  | Zn  |
|----------|-----|-----|
| Content  | 10.49 | 61.37 |

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