

**Study on the process of preparing cement admixture by desulfurization and smelting
reduction of zinc leaching residue**

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Abstract:

Zinc leaching residue, a bulk solid waste from non-ferrous industries, is produced in excess of 6 million tons annually in China. Exploring effective treatment and resource utilization technologies for zinc leaching residue has become an important topic in the field of environmental engineering. In this study, zinc leaching residue was first desulfurized to improve its suitability as a cement admixture. Subsequently, the desulfurization products were reduced, and the effects of reduction conditions on the reduction rates of zinc, iron and lead were systematically studied. Finally, the properties of cement prepared using zinc leaching residue reduction slag as cement admixture were analyzed. Research has found that a desulfurization rate of more than 95% can be achieved at 1300 °C and a holding time of 10 min. Under the conditions of 1350 °C, 40 min of holding time and a calcium-to-silicon ratio of 0.8, the reduction rates of iron, zinc and lead all reached over 97%. Using zinc leaching residue reduction slag as cement admixture, with appropriate addition amount, the prepared cement meets industry standards. This article provides insights into the co-production of cement from bulk solid waste in the non-ferrous metal industry.

Keywords: Zinc leaching residue; Desulfurization; Valuable metal recovery; Solid waste treatment; Cement admixture

1. Introduction

In recent years, the scale and number of zinc smelting enterprises in China have incrementally increased, accompanied by significant advancements in smelting technology and notable improvements in production output and product quality. Despite these developments, the zinc production process remains inherently complex and is associated with numerous pollution-intensive stages[1–3]. According to the "Guidelines for the Comprehensive Utilization of Bulk Solid Waste" promulgated during the 14th Five-Year Plan, the cumulative storage of smelting waste slag has surpassed 60 billion tons. The burgeoning zinc industry in China is shadowed by the escalating issue of zinc leaching residue accumulation, which poses an urgent and growing environmental concern[4–7]. Addressing the economical and efficient processing of zinc leaching residue represents a formidable challenge and a critical opportunity for the zinc smelting sector.

Currently, the majority of zinc smelting enterprises globally employ the hydrometallurgical process for zinc extraction on a large scale [8]. The principal stages of this process encompass leaching, purification, electrolytic deposition, and the melting and casting of cathode zinc, as

delineated in Fig. 1. The hydrometallurgical zinc smelting process is categorized based on the subsequent treatment methods for zinc leaching residue, which include: conventional leaching, hot acid leaching with the jarosite method, hot acid leaching with the goethite method, and high-pressure leaching with the hematite method [9]. Given the diversity of leaching techniques, the term 'zinc leaching residue' broadly refers to a wide spectrum of zinc smelting solid wastes. This study specifically focuses on the leach residue derived from the conventional leaching process, henceforth termed as 'zinc leaching slag'.

At present, regardless of whether zinc leaching residue is treated via the pyrometallurgy method [10,11] or hydrometallurgy method [12], tailings slag that needs to be further processed will still be generated [13,14]. If it is not properly treated, it will still need to be stockpiled, which will cause serious pollution to the environment [15]. If the extraction of valuable metals from zinc leaching slag and cementation of tailings slag can be realized, the qualitative application of zinc leaching residue can be realized. Xiaoying Liang et al. studied the feasibility of using lead-zinc tailings to replace part of the cementitious material to prepare C30 concrete [16]. Their studies revealed that concrete with partial replacement of cement, fly ash, and ground high-alumina slag satisfied C30 requirements. Kim Taewan et al. studied the effect of zinc oxide addition on the mechanical properties and microstructure of alkali-activated slag cement [17]. The results showed that the addition of 10% zinc oxide increased the compressive strength by approximately 22% at 28 days. The coprocessing technology of solid waste in the cement industry is still in its infancy. Currently, the large-scale zinc leaching residue treatment process cannot meet this requirement. Industrial waste, such as lead-zinc tailings, is usually the main treatment object and can be directly used as a mineralizer, flux or substitute in cement production [18–21]. A process that combines the extraction of valuable metals with complete application has not yet been developed.

The study explores the extraction of valuable metals from zinc leaching residue and the cementation of the resulting tailings. It encompasses desulfurization processes, scrutinizing the impact of different temperatures, durations, and aeration methods on the effectiveness of sulfur removal from zinc leaching residue. Concurrently, the research evaluates the eddy current reduction of desulfurized products, focusing on the influence of reduction conditions—specifically temperature, time, and calcium-to-silicon ratio—on the recovery rates of zinc, iron and lead, to refine the metallurgical process for recovering valuable metals from zinc smelting slag. Additionally, the study investigates the feasibility of incorporating reduced zinc leaching residue as cement admixture in cement production, analyzing the cement properties to provide a theoretical framework for the comprehensive valorization of zinc leaching residue. This approach aims to contribute to the sustainable management of zinc leaching residue by enhancing its full-scale application and minimizing its environmental footprint.

2. Materials and Methods

2.1 Materials

The zinc leaching residue was provided by Henan Yuguang Gold and Lead Co., Ltd. This zinc leaching residue is conventional leaching slag that has not been treated by wet or fire methods. After the zinc leaching residue sample was crushed and dried, it was passed through a 200-mesh experimental sieve to obtain a raw material sample with a particle size less than 80 μ m. X-ray diffraction analysis (XRD) and chemical composition analysis were performed on the samples, and

the results are shown in Figure 2 and Table 1 respectively.

Fig. 2 shows that the main phases of zinc leaching residue were Fe_2O_3 , CaSO_4 , SiO_2 , ZnFe_2O_4 , ZnSO_4 and PbSO_4 . As shown in Table 1, the main elements in zinc leaching residue were Zn, S, Fe, and Si, of which the zinc content was 14.0%, the sulfur content was 8.45%, and the total iron content was 19.7%. Zinc leaching residue also contained small amounts of Mg, Al, Cu, Pb and other elements.

2.2. Experimental procedure

The complete application of recovering valuable metals from zinc leaching residue and preparing cement admixtures from tailings was mainly divided into the following steps. Firstly, an adequate amount of zinc leaching residue was taken and placed in an oven for drying to remove moisture. The dried material was then ground using a rod mill. After grinding, the material was sealed and stored for later use.

Secondly, desulfurization treatment of the zinc leaching residue was conducted due to the sulfur content limitations required for cementitious applications. The desulfurization experiment was carried out using an induction furnace for heating. Zinc leaching residue (m_1) was placed into an alumina crucible, with an external graphite crucible serving as a sleeve. The setup was heated to a specified temperature. Once the temperature became relatively stable, gas was introduced to initiate the desulfurization experiment. Upon completion of the experiment, the alumina crucible was removed from the graphite crucible and immediately poured into a mold. After solidification, the desulfurized zinc leaching residue was recovered, weighed (m_2), and samples were taken to measure the sulfur content within it. The desulfurization rate (σ) is calculated using Equation 1, where c_1 represents the sulfur content in the raw material, and c_2 represents the sulfur content in the desulfurized zinc leaching residue.

$$\sigma = \frac{m_1 \cdot c_1 - m_2 \cdot c_2}{m_1 \cdot c_1} \quad (1)$$

Next, the desulfurization products were subjected to vortex melting reduction to recover iron, zinc and lead. The desulfurized zinc leaching residue (m_3) and calcium oxide were added to a graphite crucible. After heating to the specified temperature, an adequate amount of carbon powder was immediately introduced, followed by stirring at a moderate speed. Once the process was completed, the mixture was immediately poured into a mold. The resulting upper slag (m_4) and lower layer of metal were obtained. The metal reduction rate (μ) is calculated using Equation 2, where c_3 represents the metal content in the desulfurized zinc leaching residue, and c_4 represents the metal content in the zinc leaching residue after the smelting reduction process.

$$\mu = \frac{m_3 \cdot c_3 - m_4 \cdot c_4}{m_3 \cdot c_3} \quad (2)$$

Finally, the zinc leaching residue reduction slag was used as the cement admixture and added to the clinker and gypsum, and the experiments were performed on the basis of the Grade 42.5 cement of the Chinese national standard GB175-2023.

2.3. Characterization methods

The chemical analysis of the samples were performed by powder XRD (CuK α radiation, Bruker D8 Advance, Germany); Elemental analysis of the products was performed by XRF (ZSX

PrimusIV, Japan): The thermal stability of the zinc leaching residue was determined by an STA449F3 synchronous thermal analyzer (Netzsch, GER).

3. Results

3.1 Desulfurization of zinc leaching residue

3.1.1 Effect of temperature on desulfurization

The desulfurization temperature is a very important factor that is related to the stability of sulfates and metal sulfates and directly affects the desulfurization effect. The desulfurization rate and S concentration of the zinc leaching residue are shown in Fig. 3(a). The desulfurization rate gradually increased as the temperature increased. When the temperature was lower than 1200 °C, the sulfate was not completely decomposed, and the desulfurization result was poor. When the temperature reached 1300 °C or higher, a good removal rate can be obtained within 10 min. CaSO_4 can only start to decompose at temperatures above 1200 °C and can be completely decomposed at temperatures above 1500 °C[22]. Therefore, even if the temperature reached 1400 °C, the sulfur content cannot be completely removed, but the residual sulfur content had already fallen below 1%, and a lower sulfide content had little effect on the performance of cement. And raw materials with less than 2% sulfide residual are considered high quality[23], and has little impact on the subsequent reduction stage. Therefore, 1300 °C was the optimal desulfurization temperature.

3.1.2 Effect of time on desulfurization

At 1300 °C without oxygen, the effect of desulfurization time on the desulfurization result was investigated. The desulfurization rates of the zinc leaching residue and the sulfur content in the slag are shown in Fig. 3(b). As the desulfurization time increased, the desulfurization rate increased accordingly. When the desulfurization time was longer than 10 min, the overall removal rate was above 94%. On the basis of comprehensive consideration of the desulfurization effect and energy consumption, the selected time was 10 min.

3.1.3 Effect of oxygen ventilation on desulfurization

Under the conditions of 1300 °C, a holding time of 10 min, and aeration for 5 min, the effect of the oxygen ventilation method on desulfurization performance was investigated. The desulfurization rate and sulfur content of the zinc leaching residue under different oxygen ventilation methods are shown in Fig. 3(c). Bottom blowing is superior to top blowing, with a better desulfurization rate. The desulfurization rate of bottom-blown air (98.74%) was slightly lower than that of bottom-blown oxygen (99.37%), but both methods achieved high desulfurization efficiency. In addition, air is more advantageous in cost, so bottom-blown air was used in the desulfurization experiments. According to the XRD results of the raw material (Fig. 2), the sulfide content was very low, and desulfurization had little to do with the oxidation of sulfide by oxygen. It is speculated that oxygen blowing only agitates the melt and reduces the partial pressure of the product (sulfur dioxide) on the melt surface, thus kinetically accelerating the reaction. The XRD results of the air bottom-blowing zinc leaching residue are shown in Fig. 3(d). The main components of the slag were Fe_3O_4 and ZnFe_2O_4 , and the diffraction peak of sulfate had completely disappeared.

3.1.4 Mechanism of high temperature desulfurization

Thermogravimetric-differential scanning calorimetry (TG-DSC) thermal analysis was performed on the zinc leaching residue, and the results are shown in Figure 4. When the temperature reached 84.5 °C and 369.4 °C, endothermic peaks appeared, and the mass losses were 6.91% and 1.90%, respectively. This stage involves the removal of water due to hygroscopic absorption and the removal of crystal water. The decomposition temperature of zinc sulfate was approximately 900 K [24]. Therefore, the endothermic peak at 737.5 °C may be the decomposition of zinc sulfate, and the mass loss was 4.40%. In theory, the decomposition temperature of lead sulfate can be greater than 1100 K, and the endothermic peak at 1006.0 °C may be the decomposition of lead sulfate, with a mass loss of 10.26%. When the temperature was higher than 1200 °C, the TG curve tended to exhibit weight loss, indicating the decomposition of calcium sulfate. No exothermic peak of metal sulfides was observed at 1300 °C, indicating that the metal sulfide content in the raw material was very low. Therefore, the overall desulfurization reaction is mostly the decomposition of metal sulfates, while the oxidation proportion of metal sulfides is negligible.

3.2 Vortex smelting reduction of desulfurization products

3.2.1 Effect of temperature on reduction effect

First, the effect of temperature on the reduction process was investigated under the experimental conditions of a calcium-silicon ratio of 1.0 and a reaction time of 50 min. The results are shown in Figure 5. As the temperature rises, the reduction rate increases. When the temperature exceeded 1300 °C, the reduction rate did not increase significantly. The temperature with the best reduction rate was 1450 °C, with an iron reduction rate of 97.6%, a zinc reduction rate of 99.6%, and a lead reduction rate of 99.5%. Considering the energy consumption and recovery rate, 1350 °C was selected as the reduction temperature. Fig. 6 shows the XRD pattern. Below 1300 °C, there is more iron in the form of ferrous oxide. When the temperature increased, all the iron, zinc, lead, and arsenic were completely reduced, and only the $\text{Ca}_2\text{Mg}(\text{Si}_2\text{O}_7)$ phase remained.

3.2.2 Effect of time on reduction effect

The effect of reaction time on the reduction was investigated at 1350 °C at the calcium-silicon ratio of 1.0, and the results were shown in Fig. 7. The results showed that with increasing holding time, the reduction rates of Zn and iron significantly increased. At 50 min, the reduction rate of zinc reached 99%, and the reduction rate of iron also reached nearly 92%. According to the XRD results in Fig. 8, when the holding time was less than 30 min, there is still iron in the form of ferrous oxide. It is difficult to detect iron in the form of ferrous oxide after 40 min. Therefore, the optimal reaction time was 50min.

3.2.3 Effect of calcium-silicon ratio on reduction effect

Basicity has a critical impact on iron reduction and has a critical effect on the composition and properties of slag. Under the present raw material and experimental conditions, there are too many uncertain factors regarding the reasonable calculation of the multicomponent basicity of the present slag system. Therefore, this experiment considered only the effect of the calcium-silicon ratio and experimentally explored the effect of monobasicity on the reduction rate.

Fig. 9 shows that when the calcium-silicon ratio was varied from 0.6 to 1.0, the reduction in zinc and iron first tended to increase but then decreased, and the lead reduction rate was always

close at 100%. A better reduction result was obtained when the calcium-to-silicon ratio was 0.8, the holding time was 40 min and the temperature was 1350 °C. The reduction rate of iron reached 97.49%, the reduction rate of zinc reached 99.03%. Therefore, a reasonable calcium-silicon ratio is 0.8.

However, it should be noted that in this set of experiments, as the addition of calcium oxide increased, different degrees of vitrification were observed at calcium–silicon ratios ranging from 0.6-0.9, as shown in Fig. 10. For the study of cementation, the ease of vitrification of tailings is a very favorable property. This shows that as long as the mixture is poured into water, water-quenched slag without a crystalline phase can be obtained in the experiment. The color of the product diagram clearly shows that the phase composition first increased and then decreased, similar to the zinc-iron reduction rate, and the overall product experienced a process from gray–black to white and then to gray–black.

3.3 Vortex smelting reduction of desulfurization products

The functions of adding admixture materials to cement are mainly to increase the cement output, reduce the cement production cost, adjust the cement strength grade, improve the cement performance and quality, comprehensively utilize industrial waste, reduce environmental pollution, and realize the ecology of the cement industry. This section plans to investigate the use of tailings slag after vortex reduction as a cement admixture material to provide a reference for the follow-up work of vortex reduction-cementation complete utilization.

In this test, 4%, 7%, or 10% zinc leaching residue reduction slag was added to the cement. According to the requirements of 42.5 grade cement in the Chinese national standard GB175-2023, zinc leaching residue reduction slag was used as the cement admixture. “GB/T1346-2011 Test methods for water consumption, setting time and stability of cement” is used as the direct basis for the inspection of cement-related specifications, such as water consumption at standard consistency, cement setting time and cement stability, which were used as the inspection standard basis. According to the Chinese national standard GB/T 17671-2021, the strength of cement mortar was determined.

The results of the stability and setting time tests for the cement samples mixed with zinc leaching residue and reduction slag are listed in Table 2. From the aspect of setting characteristics, the pattern reflected by the samples is that the setting time of the samples was not significantly affected by the change in the admixture of zinc leaching residue, but the initial and final setting time of the sample were obviously increased with the increase of gypsum content [25]. The stability of all the samples was satisfactory.

The analysis of the data in Table 3 reveals that (1) when the gypsum dosage is constant, the 3 d compressive strength and flexural strength of the cement both gradually decrease with increasing dosage of zinc leaching residue. In contrast, the adverse effect of zinc leaching residue reduction slag dosage significantly weakened, and the later strength of each sample had a higher growth rate. This phenomenon reflected that although the total dosage of zinc leaching residue reduction slag was relatively low, it still had a clear inhibitory effect on the early hydration process of cement while exhibiting high hydration activity in the later stage. (2) Under the condition of a fixed zinc leaching residue dosage, the promotion effect on the early strength of cement was obvious when the dosage of gypsum increases. When the dosage of gypsum was 6%, the corresponding cement sample’s 3 d compressive strength was increased by 3 MPa and 0.5 MPa compared with the corresponding

strengths of the 3% gypsum cement sample; however, for the 28 d strength, the promoting effect was not as significant, but overall, increasing the gypsum admixture had a positive effect on the strength of the corresponding cement sample.

4. Conclusions

This study provides a novel approach for the resource utilization of zinc leaching residue, demonstrating that zinc leaching residue, after desulfurization and smelting reduction processes, can be utilized as cement admixtures. The desulfurization rate of zinc leaching residue is highly dependent on temperature and holding time, with optimal results achieved at 1300 °C and 10 min. The desulfurized product, after smelting reduction, can achieve a high reduction rate. Under the conditions of 1350 °C, 40 min of holding time and a calcium-to-silicon ratio of 0.8, the reduction rates of iron, zinc and lead all reached over 97%. The setting time, stability, 3 d strength and 28 d strength of the cement prepared with the addition of 6% gypsum and 4%, 7% and 10% zinc leaching residue reduction slag met the standards. The technical route for preparing cement admixtures with zinc leaching residue proposed in this study is feasible, and further research can be conducted on the long-term performance of cement and the feasibility of large-scale production in the future.

Declaration of competing interest

We declare that we have no financial or personal relationships with other people or organizations that can inappropriately influence our work. There is no professional or other personal interest of any nature or kind in any product, service or company.

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Figure and table captions

Fig. 1 Main process of wet zinc smelting

Fig. 2 Phase analysis of zinc leaching residue

Fig. 3 Desulfurization rate and S content of zinc leaching residue at (a) different temperatures (b) time (c) ventilation methods, (d) XRD result of air bottom blowing desulfurization zinc leaching residue

Fig. 4 TG-DSC curve of zinc leaching residue

Fig. 5 Reduction rate of valuable metals at different insulation temperatures

Fig. 6 XRD patterns at different temperatures

Fig. 7 Reduction rate of valuable metals at different holding times

Fig. 8 XRD patterns of the product at different holding times

Fig. 9 Reduction rate of valuable metals at different calcium-silicon ratios

Fig. 10 XRD patterns and product pictures of reduced slag with different calcium-to-silicon ratios

Table 1 Composition analysis of zinc leaching residue (%)

Table 2 Setting time and stability measurement results

Table 3 Measurement results of compressive and flexural strength of cement

Figure and table

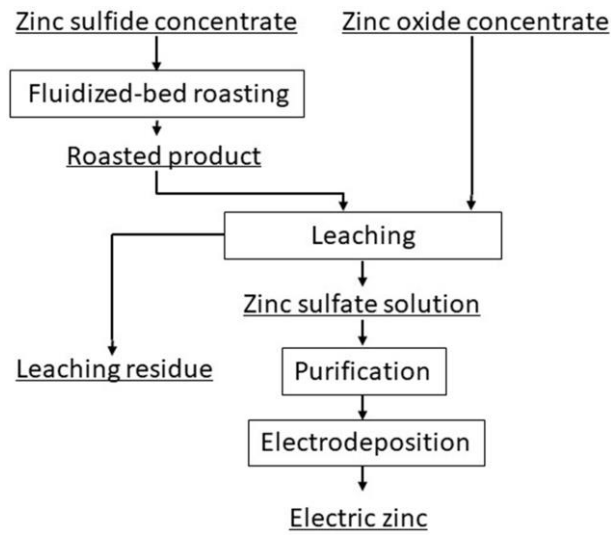


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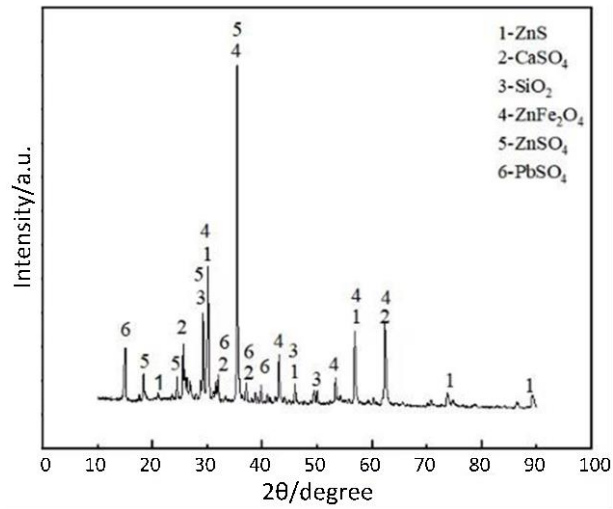


Fig. 2 Phase analysis of zinc leaching residue

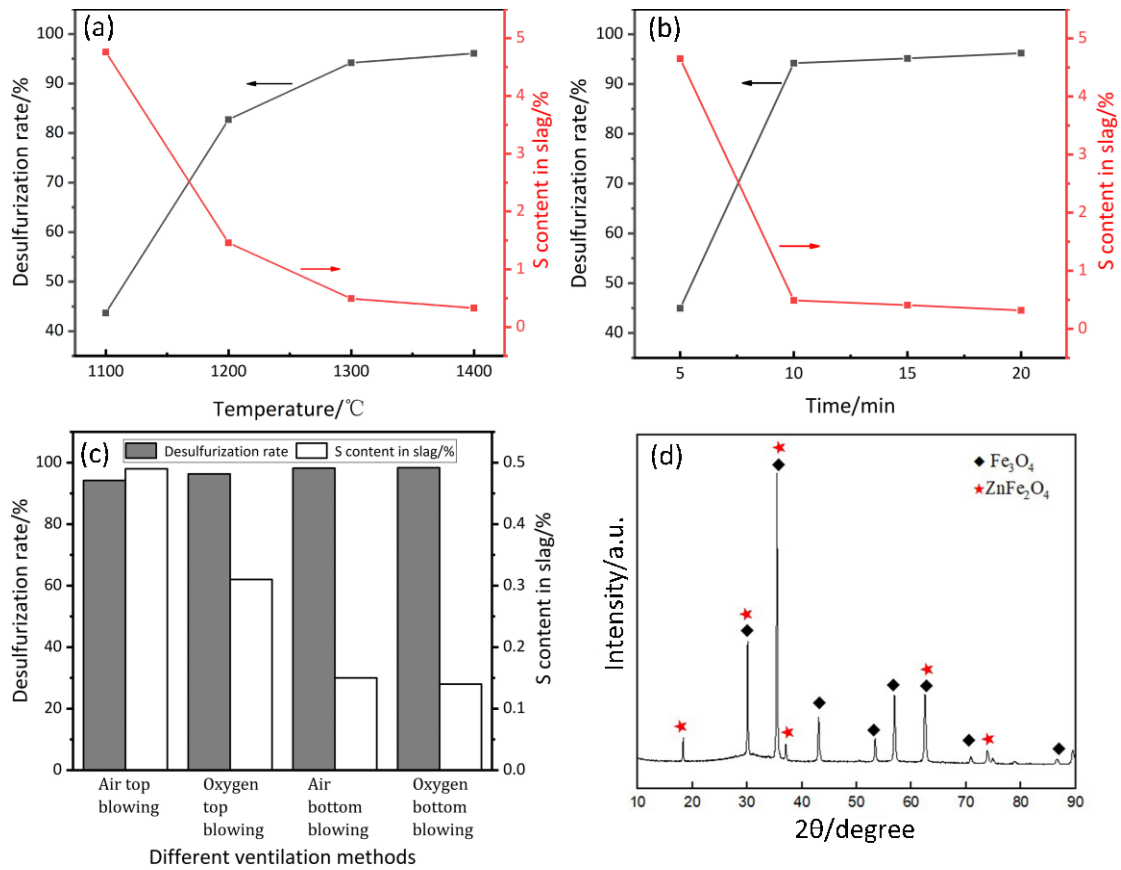


Fig. 3 Desulfurization rate and S content of zinc leaching residue at (a) different temperatures (b) time (c) ventilation methods, (d) XRD result of air bottom blowing desulfurization zinc leaching residue

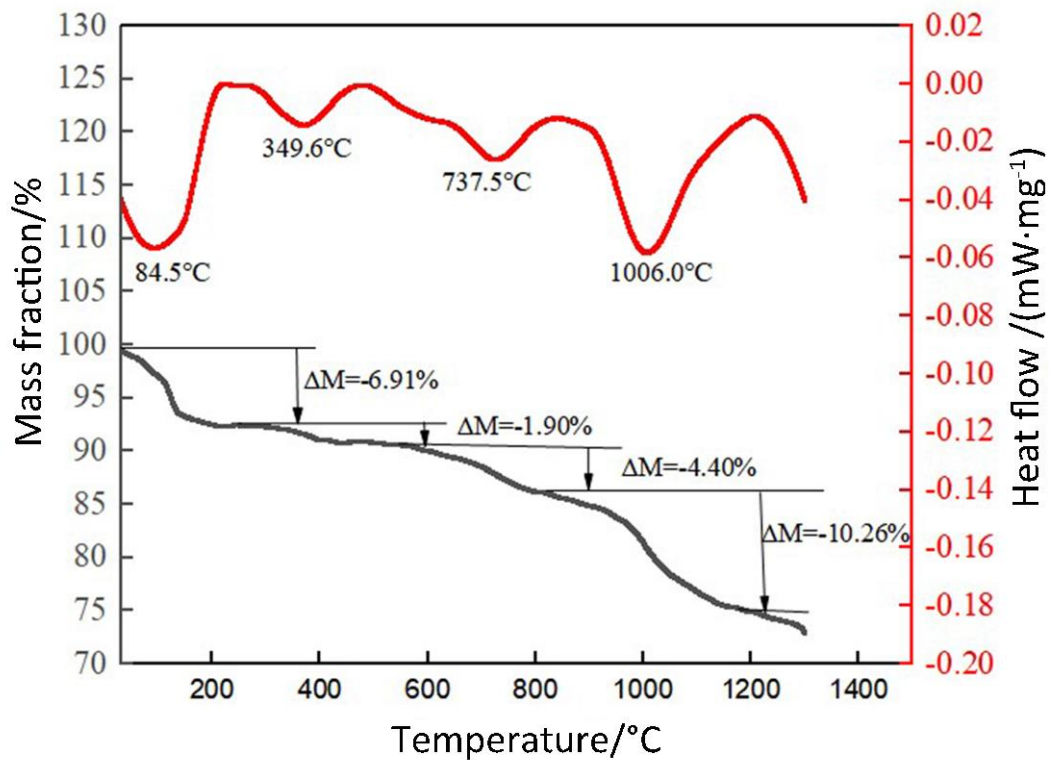


Fig. 4 TG-DSC curve of zinc leaching residue

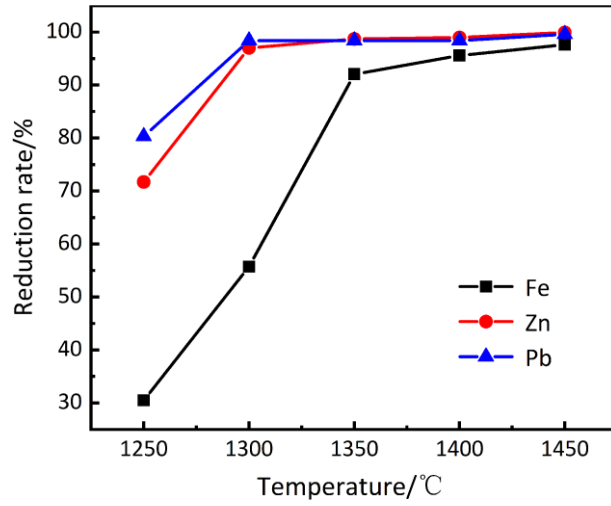


Fig. 5 Reduction rate of valuable metals at different insulation temperatures

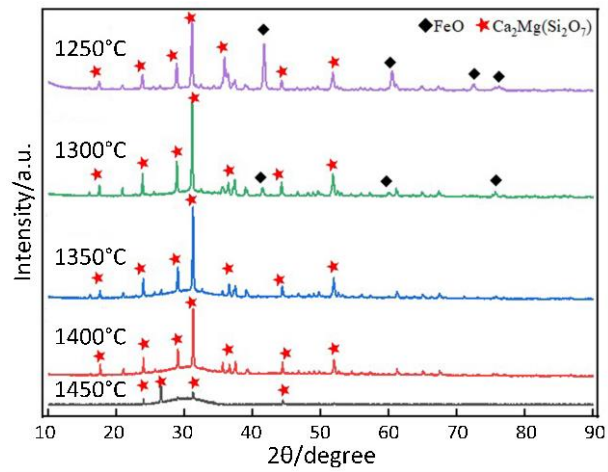


Fig. 6 XRD patterns at different temperatures

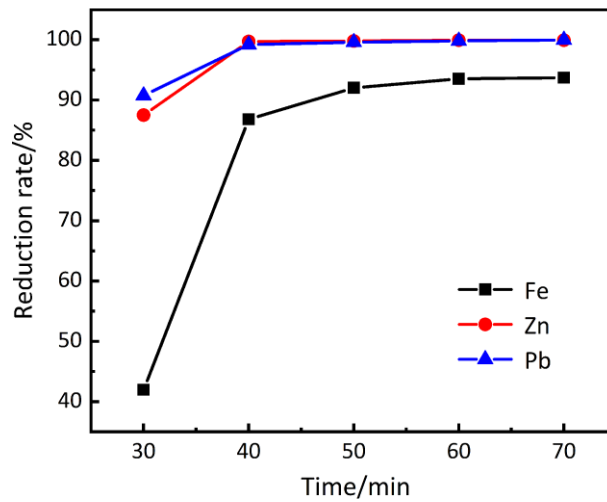


Fig. 7 Reduction rate of valuable metals at different holding times

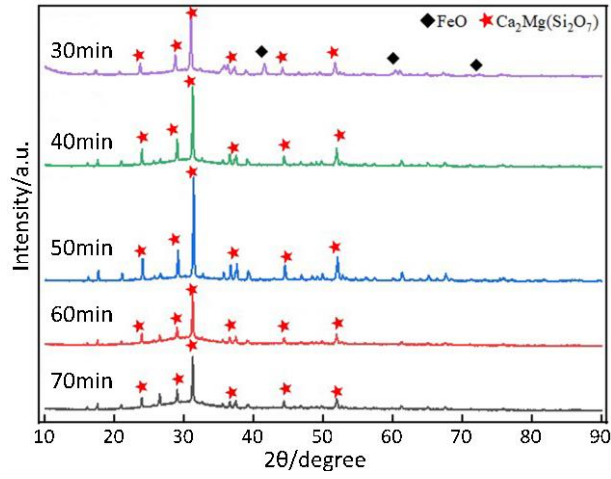


Fig 8 XRD patterns of the product at different holding times

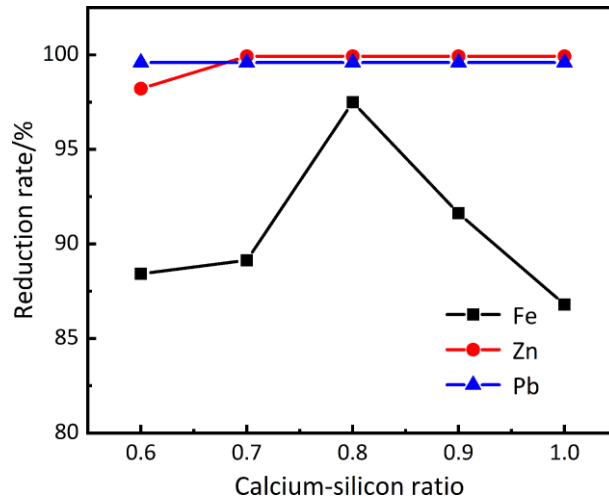


Fig. 9 Reduction rate of valuable metals at different calcium-silicon ratios

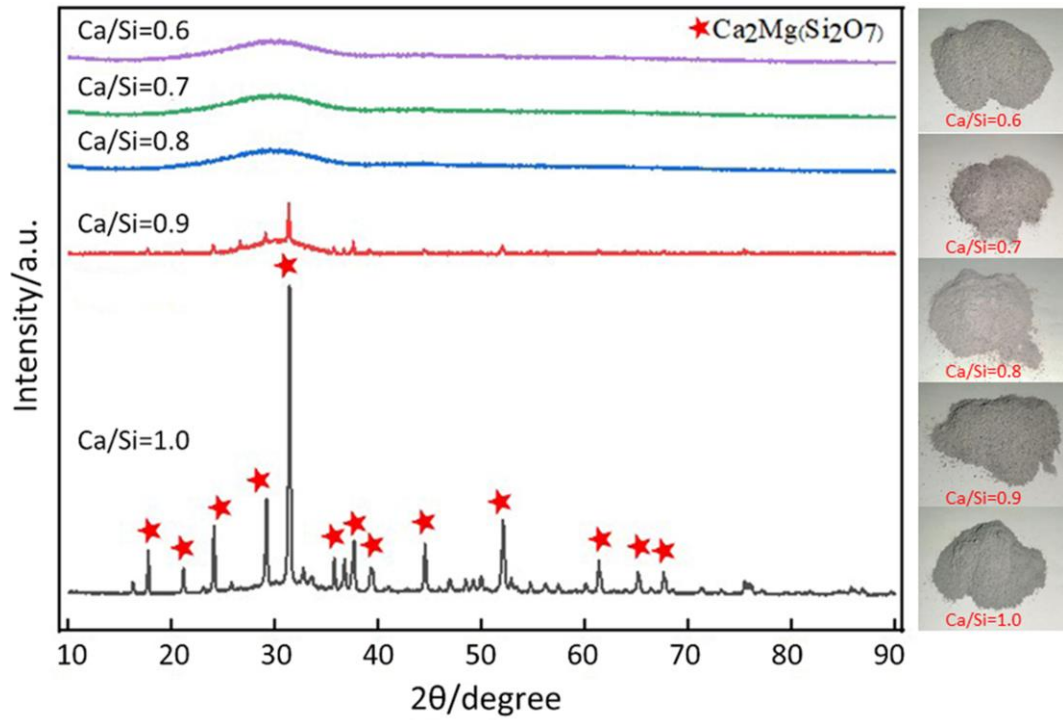


Fig 10 XRD patterns and product pictures of reduced slag with different calcium-to-silicon ratios

Table 1 Composition analysis of zinc leaching residue (%)

Element	Cu	Zn	S	Fe	Pb
Content	0.92	14.0	8.45	19.7	2.49

Table 2 Setting time and stability measurement results

Number	Clinker/%	Gypsum/%	Zinc leaching	Setting time/min		Stability
			residue reduction slag/%	Initial setting	Final setting	
S1	93	3	4	200	230	Qualified
S2	90	3	7	205	235	Qualified
S3	87	3	10	200	230	Qualified
S4	90	6	4	230	260	Qualified
S5	87	6	7	225	255	Qualified
S6	84	6	10	220	255	Qualified

Table 3 Measurement results of compressive and flexural strength of cement

Number	Clinker/%	Gypsum/%	Zinc leaching	3d strength/MPa		28d strength/MPa	
			residue reduction slag/%	Compressive strength	Flexural strength	Compressive strength	Flexural strength
S1	93	3	4	19.01	4.61	46.03	7.84
S2	90	3	7	18.54	4.13	39.59	7.80
S3	87	3	10	17.65	3.92	37.30	7.56
S4	90	6	4	22.07	5.18	42.83	8.02

S5	87	6	7	21.25	4.76	45.45	7.78
S6	84	6	10	20.76	4.51	42.60	7.83

Biographies

SONG WANG is a doctoral student at the School of Metallurgy of Northeastern University. His main research areas include strategic metal recovery, polymer synthesis, pressurized hydrometallurgy, and clean utilization of non-traditional metallurgical resources.

GUOZHI LV is a professor at Northeastern University, a doctor, a doctoral supervisor, and the deputy director of the Department of Metallurgical Engineering. He is also the deputy director of the Institute of Special Metallurgy and Process Engineering, and is mainly responsible for research related to pressurized hydrometallurgical processes. He has presided over three National Natural Science Foundation projects and participated in more than 20 research projects such as the National Natural Science Foundation key projects and the national "863 Program" key projects as the executive director and research backbone.

XUEWEI YANG, male, doctorate, senior engineer of China Building Materials Group. He has been deeply cultivated in industrial projects at home and abroad for many years. He has undertaken the management of multiple general contracting projects of Asia, Africa, and Europe, and has accumulated rich experience in overseas engineering management. International practice, technical standards, and project operation models of developed countries in Europe and the United States Wait for unique insights. In recent years, it has undertaken the development, design, and implementation of the "mining triangle truss automatic inspection and transportation system", "" multi -material hazardous waste incineration compatibility system "and other R & D projects. Related research work, as the first author, published 3 high -level papers in the domestic and foreign metallurgical journals, two volumes of two volumes of metallurgy solid waste building materials industry standards, and two "Belt and Road" series of books. A prize.

TINGAN ZHANG is a second-level professor at Northeastern University, a doctor, a doctoral supervisor, a member of the State Council's academic review group, a recipient of the government's special allowance, and a foreign academician of Russia. He is also the executive director of the China Nonferrous Metals Industry Association and the honorary executive director of the China Nonferrous Metals Society. He is currently the director of the Engineering Center of the Ministry of Education for Nonferrous Metals Metallurgical Process Technology, the dean of the Nonferrous Metals Solid Waste Technology Research Institute of Northeastern University, and the former dean of the School of Materials and Metallurgy of Northeastern University and the director of the library.