



Article Phase Transformation and Zinc Extraction from Zinc Ferrite by Calcium Roasting and Ammonia Leaching Process

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Abstract: Zinc ferrite (ZnFe₂O₄), one of the major forms of zinc in electric arc furnace dust and zinc leaching residue, is very difficult to chemically dissolve in either acidic or alkaline solutions. It is therefore necessary to study the extraction of zinc from zinc ferrite to achieve the full recovery of the zinc resources. In this paper, CaO was used to degrade the structure of zinc ferrite and then the ammonia leaching method was applied to recover zinc. The thermodynamic analysis indicated that the zinc ferrite could react with CaO to form zinc oxide and dicalcium ferrite. The CaO–Fe₂O₃–ZnO phase diagram shows that Ca₂Fe₂O₅ can be in equilibrium with ZnO rather than ZnFe₂O₄ at a higher CaO area. The experimental results indicated that the phase transformation of zinc ferrite, reacted with CaO, to zinc oxide and dicalcium ferrite could be completely achieved with a CaO addition of 2.4:1 (mole ratio). The peaks of zinc ferrite in the X-ray diffraction (XRD) patterns cannot be clearly observed after calcium roasting at 1220 °C for 2 h. The ammonia leaching tests showed that the zinc leaching rate was about 90% under optimal roasting conditions.

Keywords: zinc; zinc ferrite; calcium roasting; ammonia leaching; phase transformation

1. Introduction

According to the United States Geological Survey (USGS) statistics, the world's total zinc reserves are about 250 million tons in 2020 [1]. Zinc is mainly used in the fields of steel, metallurgy, machinery, electricity, chemical industry, light industry, military and medicine [2–5]. The raw materials for zinc extractions include lead-zinc ores, zinc concentrate, and also secondary zinc resources from the smelter output. In the exploitation and consumption of zinc ore, many researchers have focused on the extraction of zinc from zinc-containing wastes, such as zinc-bearing dust in ironmaking and steelmaking [6–11].

Iron and steel enterprises produce a large amount of dust in various processes such as ironmaking, steelmaking and steel rolling, accounting for about 10% of the total steel production. Recently, there has been about 100 million tons of zinc-bearing dust per year in China.

In some steel companies, zinc-bearing dust is put into the sintering process. However, the dust contains high levels of zinc and other harmful elements [12], and direct back-matching causes zinc to be enriched in a continuous cycle, leading to excessive zinc load in the blast furnace, which is harmful to the smooth production and safe longevity. On the other hand, since the quality of zinc-containing dust is far less than that of traditional zinc-making raw materials, the value of zinc extraction is limited and usually cannot be directly used by zinc-making enterprises [13]. The zinc-bearing dust in ironmaking and steelmaking contains a lot of iron and zinc elements, in which the zinc mainly exists in the form of zinc oxide and zinc ferrite ($ZnFe_2O_4$), and iron mainly exists as magnetite (Fe_3O_4), hematite (Fe_2O_3), wustite (FeO) and zinc ferrite austenite phase [8,13–15]. However, zinc ferrite has a spinel structure, which has considerable stability [16–18]; furthermore, zinc



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Copyright: © 2022 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). ferrite is insoluble in water, dilute acid solution, alkali and ammonia solution under normal conditions.

Since the zinc cycle and hazards cannot be avoided in the traditional process mode, the adoption of new specialized disposal processes has become one of the feasible measures. The disposal technologies are the Waelz rotary kiln process, the rotary bottom furnace process, the OxyCup shaft furnace process, the Duisburg Kupferhutte (DK) small blast furnace, etc. [12,19]. The rotary bottom furnace process is mainly used to recover zinc, but the zinc recovery rate is low. Furthermore, some hydrometallurgical processes have been reported to recover zinc from the zinc-containing dust, in which the main phases of zinc are zinc oxides [11,20–25].

At present, the pyrometallurgy process is the main process, and gradually the combined pyrometallurgy-hydrometallurgy process is being used more. A large amount of zinc secondary resources contains high zinc, and the zinc mainly exists in the form of zinc ferrite, and the current recycling utilization rate is extremely low, causing the waste of resources and more serious environmental pollution. There are various hydrometallurgical and pyrometallurgical processes for the decomposition of zinc ferrite, including high-pressure acid leaching, various acids leaching, microwave caustic leaching, alkaline leaching, ammoniac solutions and chloride leaching [17,26]. However, the further treatment of the residues is difficult and causes environmental problems. Furthermore, Na₂CO₃ roasting and sulphation roasting are used to convert zinc ferrite into soluble zinc and iron compounds at high temperatures [27]. However, the residual alkali metals and sulfur increase the difficulty for further treatment of residues. Developing a clean and efficient comprehensive utilization of the secondary zinc resources technology both has huge economic benefits and significant environmental benefits, and is also a way to solve the structural contradictions of zinc resources in China. Therefore, it is particularly important to develop new ways for zinc ferrite comprehensive utilization of zinc-bearing dust and achieve the recycled utilization of zinc in the factory.

In this paper, the extraction of zinc from zinc ferrite by calcium roasting and the ammonia leaching process was investigated. The thermodynamics of chemical reactions and the phase diagram were analyzed. The microstructure and phases composition of the samples were determined by the X-ray diffraction (XRD) and scanning electron microscopeenergy dispersive spectroscopy (SEM-EDS) techniques. The optimized parameters of the process were studied and proposed. This study can provide the technical basis for further research on the high content of the zinc ferrite phase for the comprehensive utilization of zinc dust.

2. Materials and Methods

2.1. Materials

Zinc ferrite is a mineral formed in industrial production and has not been found in nature, thus we used synthetic zinc ferrite as raw material in the following experiments. We prepared the synthetic zinc ferrite as follows [28]. Firstly, zinc oxide and iron oxide were thoroughly mixed with a molar ratio of 1:1. The mixture was placed into a muffle furnace and roasted at 1000 °C for 4 h. Then, the roasted samples were ground to particles and passed through a 200 mesh Taylor standard sieve. The samples were washed in 1 mol/L dilute hydrochloric leaching at a temperature of 40 °C and a leaching time of 1 h. After repeatedly washing three times again, the leaching residue was placed in a muffle furnace for roasting at 1000 °C for 4 h. The synthetic reaction equation is Equation (1):

$$Fe_2O_3(s) + ZnO(s) \rightarrow ZnFe_2O_4(s)$$
 (1)

The XRD patterns of the prepared zinc ferrite sample are shown in Figure 1. The chemical composition of the zinc ferrite sample is listed in Table 1. The chemical composition of zinc ferrite was determined by the chemical analysis method according to a Chinese standard GB/T 8151.1-2012 and GB/T 8151.3-2012 [29,30]. The deviations are less than 0.3 wt.%.



Figure 1. XRD patterns of prepared zinc ferrite.

Table 1. The chemical composition of zinc ferrite sample (wt.%).

Zinc (Zn)	Iron (Fe)	Oxygen (O)
27.11	46.28	26.61

According to the results in Figure 1 and Table 1, the zinc and iron element contents of the zinc ferrite sample are very close to the theoretical content of zinc ferrite (27.12 wt.% and 46.33 wt.%). Thus, it can be inferred that the purity of the prepared zinc ferrite sample was enough for the following experiments.

2.2. Experimental Methods

(1) Roasting experiment: the roasting tests were conducted in a muffle furnace (HF-Kejing, Hefei, China) with silicon carbide heating elements. The synthetic zinc ferrite powder (2.5 g) and analytical purity CaO powder were mixed thoroughly at the designed ratios. The mixture was mixed with a small amount of water and pressed into cylindrical pellets (Φ 10 mm × 10 mm). Then, the dried pellets were placed into the tube furnace, and the tube furnace was heated to the designed temperature and roasted for a planned duration under an air atmosphere. Then, the roasted samples were taken out for subsequent tests and analysis.

(2) Leaching experiment: The roasted samples were ground into particles and sieved with a 200 muse Taylor standard sieve to make the powder less than 74 μ m in size. As illustrated in Figure 2, the leaching experiments were conducted in a 250 mL beaker heated in a water bath at a target temperature [31]. The leaching procedure was initiated by pouring 100 mL of the required concentration ammonia leaching agent into a 250 mL beaker with a plastic film covered. Then, the leaching agent was heated to the experimental temperature with stirring. Time was counted when the samples were added to the leaching beaker. After the required leaching duration ended, the leachate was separated from the residue by vacuum filtration and analyzed. The leaching conditions were kept constant as follows: the total ammonia concentration was 6 mol/L, the mole ratio of NH₄Cl to NH₃•H₂O was 1:1, the leaching temperature was 40 °C, the solid–liquid ratio was 20 g/L, the stirring speed was 350 r/min and the leaching time was 3 h. Each group of analyses was repeated three times and the average value was taken as the final data.



Figure 2. Schematic diagram of leaching experiment equipment. 1—Frequency converter stirrer bracket. 2—Agitator. 3—Stainless steel impeller. 4—Plastic film. 5—Beaker. 6—Stirring blades. 7—Constant temperature water bath. 8—Sample and ammonia mixture. 9—Constant temperature flume.

In addition, the zinc contents of the samples were determined according to a standard GB/T 8151.1-2012 [29]. The phases compositions of the samples were analyzed by X-ray diffraction (TTR-III, D8 Advance, Tokyo, Japan,) with a graphite monochromator and Cu-K α radiation in the range of 10–70° at a scanning rate of 10°/min. The microstructure and elemental distributions of the samples were determined by SEM (TESCAN, MIRA3-LMH, Brno, Czech) with EDS (Oxford X-MAX20, Oxford, UK). The relative deviation is 5% for elements with content higher than 20%, 10% for those with 3–20%, 20% for those with 1–3%, and 50% for those with 1% or less.

The leaching ratio of zinc (φ), which is the maximal theoretical value of zinc leaching rate by ammonia leaching, is calculated as Equation (2):

$$\varphi = \left[\frac{\mathbf{F} \times \mathbf{V}}{\mathbf{M} \times \mathbf{Z}}\right] \times 100\% \tag{2}$$

where F is the concentration of zinc in the filtrate, g/L; V is the volume of filtrate, L; M is the total mass of the sample, g; Z is the zinc content of the sample.

3. Results and Discussion

3.1. Thermodynamic Analysis

In this study, the CaO was added to zinc ferrite to break the crystal structure of zinc ferrite. The thermodynamics of the chemical reaction was analyzed and listed in Equation (3).

$$ZnFe_2O_4 + 2CaO = ZnO + Ca_2Fe_2O_5$$

$$\Delta G = -16.901T - 27880 (J/mol)$$
(3)

Reaction 4 shows that ammonia can form ammonia complexes with zinc oxide.

$$ZnO + nNH_3 \bullet H_2O = [Zn(NH_3)n]^{2+} + 2OH^-$$
 (4)

In addition, the generated $Ca_2Fe_2O_5$ is insoluble in the leaching process, eventually, $Ca_2Fe_2O_5$ can be used as a sintering raw material and steelmaking solvent. After processing, zinc leach liquor can be used as the raw material for zinc electrodeposition.

The phase diagram was used to determine the additional amount of CaO in the roasting process. The phase diagram of the CaO-ZnO-Fe₂O₃ system at 1200 °C was calculated by the FactSage 8.0 software [32] through the "Phase Diagram" module with

the database "FactPS" and "FToxid". As shown in Figure 3, the ZnO and $Ca_2Fe_2O_5$ can be stable coexistence without $ZnFe_2O_4$ at 1200 °C. The dashed line is the theoretical formation of $ZnFe_2O_4$. There is no " $ZnFe_2O_4$ " in the " $ZnO+CaFe_2O_5+CaO$ " area when the mole ratio of $CaO:ZnFe_2O_4$ is about 2:1. Thus, we chose a selection of ratios larger than the theoretical value of 2 in our study.



Figure 3. Isothermal section diagram of CaO-ZnO-Fe₂O₃ at 1200 °C.

3.2. Phases Compositions of the Roasted Samples

The effects of CaO addition on the phase transformation of zinc ferrite were studied at 1220 °C for 2 h. The CaO powder and zinc ferrite were mixed in the molar ratios of 2.0:1 to 2.7:1. To study the phase transformation of zinc ferrite after calcified roasting, the XRD technique was carried out on the roasted samples. The results are shown in Figure 4.

As illustrated in Figure 4, with the increase of CaO addition, the peaks of zinc ferrite gradually become weakened, and the diffraction peaks of calcium ferrite and zinc oxide gradually increased. When the ratio is 2.3:1, zinc ferrite still has obvious diffraction peaks, indicating that zinc ferrite is not completely transformed. When the ratio was 2.4:1, the diffraction peaks of zinc ferrite disappeared, indicating that when the ratio of n (CaO)/n (ZnFe₂O₄) was 2.4:1 and roasted at 1220 °C for 2 h, almost all zinc ferrite react with CaO and convert to ZnO and Ca₂Fe₂O₅.

The reaction of zinc ferrite and calcium oxide is almost completely transformed into ZnO and $Ca_2Fe_2O_5$, and the system reaches equilibrium when $CaO/ZnFe_2O_4$ (mole ratio) is 2.4:1. Therefore, the ratio of CaO addition should be above 2.4:1 to completely transform zinc ferrite. Due to the solid–solid reaction, the diffusion rate is slow, which may be a restrictive link in the reaction. The essence of the reaction is of cation and anion migration replacement, which must contact each other to react; therefore, the CaO addition amount



should be excessive than the theory of consumption. Thus, the suitable addition of CaO should be 2.4:1.

Figure 4. XRD patterns of roasted samples with various CaO addition: (**a**) raw material, (**b**) CaO addition of 2.0:1, (**c**) CaO addition of 2.1:1, (**d**) CaO addition of 2.3:1, (**e**) CaO addition of 2.4:1, (**f**) CaO addition of 2.5:1, (**g**) CaO addition of 2.6:1, (**h**) CaO addition of 2.7:1.

The effects of roasting time on the phase compositions of samples were also studied. The mole ratio of CaO and zinc ferrite was fixed at 2.4:1, the roasting temperature was kept at 1220 °C, roasting time was 0.5 h, 1 h and 2 h. The phase composition of the roasted samples is shown in Figure 5; the peaks of the zinc ferrite phase in the roasting product after roasting for 0.5 h still can be observed, which indicates that zinc ferrite is not completely transformed at this time, but the calcium ferrite phase appeared, which may be due to element migration and displacement in the contact part of the solid–solid reaction, but not completely carried out. However, the diffraction peaks of calcium ferrite and zinc oxide are further enhanced with the prolonged roasting time. This may be explained by the

insufficient roasting time leading to incomplete element migration and replacement during the roasting process. After roasting for 2 h, there are no significant diffraction peaks of zinc ferrite in the product, indicating that the extension of time is conducive to the element migration and replacement reaction of the solid–solid reaction. In general, the element diffusion and migration between the reactants are the limiting factors in the reaction dynamics of the solid–solid reaction [33]. Therefore, to ensure the diffusion of elements, a certain roasting time must be guaranteed, and a roasting time of 2 h is recommended.



Figure 5. The XRD patterns of roasted samples under different roasting times.

According to the phase transformation process of zinc ferrite with the addition of lime, it can be concluded that the formed calcium iron oxide is dicalcium ferrite ($Ca_2Fe_2O_5$).

3.3. Microstructure and Element Distribution of Roasted Samples

In this part, the microstructure of the roasted samples with various roasting durations is observed by scanning electron microscopy (SEM). Figure 6b–d show microstructures of the calcined products at 1220 °C for calcination time of 0.5 h, 1 h and 2 h, respectively. Tables 2–4 list the energy spectrum analysis (EDS) results of the typical regions in the calcium roasted products under the corresponding roasting time, respectively.



Figure 6. Scanning electron microscope (SEM) images of roasted products for different time: (a) without roasting, (b) 0.5 h, (c) 1 h, (d) 2 h.

EDS	5 Points	CaO	Fe ₂ O ₃	ZnO
1	wt.%	40.3	57.5	2.2
	mol.%	65.0	32.6	2.4
2	wt.%	15.7	24.5	59.8
	mol.%	23.9	13.1	63.0

Table 2. Energy spectrum analysis (EDS) analysis of the roasted product with the roasting time of 0.5 h.

Table 3. EDS analysis of the roasted product with the roasting time of 1 h.

EDS	Points	CaO	Fe ₂ O ₃	ZnO	
1	wt.% mol.%	41.6 64.7	51.8 28.3	6.6 7.0	
2	wt.% mol.%	12.7 20.9	35.5 20.5	51.8 58.6	

Table 4. EDS analysis of the roasted product with the roasting time of 2 h.

EDS	6 Points	CaO	Fe ₂ O ₃	ZnO
1	wt.%	40.7	55.1	4.2
	mol.%	64.6	30.8	4.6
2	wt.%	1.8	3.7	94.5
	mol.%	2.6	1.9	95.5

As shown in Figure 6, the roasted products are mainly rod-shaped or granular with clear morphology and certain intercalation of different phases. However, as the roasting time increases, the intercalation relationship decreases. Moreover, when the calcination time was 0.5 h and 1 h, the calcined reconstructed products showed different typical regions, namely the white region and grey region. Combined with the energy spectrum analysis results of these typical regions, it could be seen that the white region was mainly ZnO generated by the reaction. The grey area is mainly a mixture of unreacted zinc ferrite, ZnO and Ca₂Fe₂O₅. The phases are distributed mutually without independent phase formation. It could be inferred that when the roasting time was less than 1 h, the reaction between CaO and ZnFe₂O₄ was not complete. In addition, it can also be seen from Figure 6d that the sizes of ZnO and $Ca_2Fe_2O_5$ crystals are larger when the calcination time is 2 h than that when the calcination time is 0.5 h and 1h. Compared with the chemical compositions in Tables 2-4, the independent ZnO grains can be observed and the embedded relationship of the main phases in the sample becomes simple. It can be inferred that this structure would be favorable to the subsequent leaching extraction of zinc from the roasted samples. Therefore, to ensure the complete transformation of $ZnFe_2O_4$, the roasting time should not be less than 2 h, which is consistent with the XRD results.

Figures 7–9 illustrate the elemental mapping of the roasted products under different roasting durations. Compared with the element's distribution of the samples, it can be seen that the zinc element migrates to the bright white area gathered (ZnO phase zones), while Ca and Fe elements migrate and accumulate in the dark grey area ($Ca_2Fe_2O_5$ phase accumulation area). Meanwhile, with the increase of calcination time, the distribution of Zn elements in the bright white area (ZnO phase) becomes denser, while the distribution of Zn elements in the dark grey area ($Ca_2Fe_2O_5$ phase) becomes sparse. The corresponding distribution of Ca and Fe elements in the dark grey region ($Ca_2Fe_2O_5$ phase) is more sparse, whereas the distribution of Ca and Fe elements in the dark grey region ($Ca_2Fe_2O_5$ phase) becomes denser. Under the same condition of calcium oxide ratio and roasting temperature, prolonging the roasting time could promote element migration, which is also verified by energy spectrum analysis results.



Figure 7. Main element distribution of the roasted product with the roasting time of 0.5 h.

 Te
 10 μm

Figure 8. Main element distribution of the roasted product with the roasting time of 1 h.



Figure 9. Main element distribution of the roasted product with the roasting time of 2 h.

3.4. Ammonia Leaching of Roasted Samples

As we know, the ammonia leaching method is known to be a powerful method used in hydrometallurgical processes. Many metals can be extracted using the mixture of ammonia/ammonium [34–37]. In this study, ammonia leaching is used to extract the zinc from the calcium roasted zinc ferrite samples and verified the results of phase transformations of zinc ferrite.

The effect of the CaO addition is shown in Figure 10. The zinc leaching rate is only about 63% when the CaO addition is 2.1:1. This can be explained by the incomplete conversion of zinc ferrite. When the lime addition is above 2.4:1, the zinc leaching rate is up to about 90%. This also indicates that the phase transformation of zinc ferrite to zinc oxides is almost obtained during the calcium roasting process.



Figure 10. Effect of CaO/ZnFe₂O₄ (mole ratio) on zinc leaching rate.

In this part, the effect of roasting temperature on the zinc leaching rate was studied with the fixed mole ratio of CaO/ZnFe₂O₄ as 2.4:1. The roasting temperature was from 1220 °C to 1250 °C and the roasting time was 120 min. Figure 11 shows that the zinc leaching rate rises sharply to 90.41% at 1230 °C.



Figure 11. Effect of roasting temperature on zinc leaching rate.

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In this study, the calcium roasting-ammonia leaching process was adopted to extract zinc from zinc ferrite. The spinel-type zinc ferrite could be converted into zinc oxide and dicalcium ferrite by calcium roasting. The residue contains mainly dicalcium ferrite, which can be used in the iron ore sintering process in the steel company. Compared to the additions of sodium carbonate and sulfur in other processes, our proposed process achieved the treatment of zinc-bearing solid waste and the recycling utilization of zinc resources in the company.

4. Conclusions

The extraction of zinc from the zinc ferrite by calcium roasting and ammonia leaching process was studied in this paper. The thermodynamic analysis indicated that the $ZnFe_2O_4$ could react with CaO to transform into ZnO and $Ca_2Fe_2O_5$ within suitable roasting conditions. The suitable mole ratio of CaO addition for the complete transformation of zinc ferrite should be not less than 2.4:1. Prolonging the roasting time is beneficial for the formation of zinc oxide and dicalcium ferrite and the growth of zinc oxides phases. The generated zinc oxide crystals became independent and large with the increase in roasting duration. The ammonia leaching was used to extract the zinc from the calcium roasting zinc ferrite samples and verified the results of phase transformations of zinc ferrite. Under the optimal roasting conditions—a CaO/ZnFe₂O₄ mole ratio of 2.4:1, a roasting temperature of 1230 °C and a roasting time of 2 h—the zinc leaching rate was up to 90.41%.

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