Experimental Study on Influence of Al\(_2\)O\(_3\), CaO and SiO\(_2\) on Preparation of Zinc Ferrite

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Abstract: Gossan ore of sulfide zinc deposit contains abundant zinc, iron, and other metal elements, which is a significant resource with complex components and can be utilized. In this study, a new technology of preparing zinc ferrite from zinc sulfide deposit gossan was proposed. The effects of Al\(_2\)O\(_3\), CaO, and SiO\(_2\) in gossan on the formation of zinc ferrite were investigated by X-ray diffraction (XRD), scanning electron microscopy (SEM), and specific surface area and pore size analysis (BET). The results show that the presence of Al\(_2\)O\(_3\) and CaO could hinder the formation of zinc ferrite, while silica had no effect on the formation of zinc ferrite. Under the conditions of the molar ratio of ZnO and Fe\(_2\)O\(_3\) to Al\(_2\)O\(_3\), CaO, and SiO\(_2\) of 1:1:1, an activation time of 60 min, and a roasting temperature of 750 °C for 120 min, the products, which had good crystallinity, smooth particle surface, and uniform particle size could be obtained. In addition, compared to the roasted products with Al\(_2\)O\(_3\) and CaO, the specific surface area, pore volume, and pore size of the products with SiO\(_2\) were the largest.

Keywords: zinc sulfide deposit gossan; zinc ferrite; behavior characteristics; phase transformation; microstructure

1. Introduction

Gossan is a kind of ore mainly composed of iron oxide, manganese oxide, calcium oxide, silicon oxide, alumina, hydrated oxide, sulfate, alum, and clay, which is often distributed in the upper part of primary sulfide deposits [1,2]. Clay minerals, limonite, and colloids in gossan can absorb some valuable elements such as gold, silver, and copper. However, the main component of gossan is oxidized ore, in which the metal content is low, meaning it was difficult to recover and utilize these valuable elements by traditional beneficiation and metallurgy technology. At present, researchers worldwide have studied the recovery and utilization of gossan ore. Sánchez et al. [3] used the conventional cyanidation process to treat the gold silver gossan ore, a gold leaching rate of 75%–80% was obtained, and the silver leaching rate could be increased from 40%–45% to 75% after the sample was pretreated with sulfide solution. Celepo et al. [4] explored the pretreatment of alkaline solution before cyanide leaching for gold silver gossan ore. The leaching rate of gold and silver increased from 76% and 23% to 87% and 90%, respectively. Li et al. [5] conducted a cyanidation leaching experiment on a gossan gold silver ore from Qinghai Province. Under certain leaching conditions, the leaching rates of gold and silver in the ore reached 93% and 83%, respectively. Chen et al. [6] used roasting acid leaching cyanidation leaching processes to recover copper, gold, and iron from Qinghai Derni gossan ore and high sulfur semi oxide ore. The results showed that under the conditions of mass ratio of oxide ore and high sulfur semi oxide ore of 1:1, ore size of −75 µm accounting for 81.5%, a roasting
temperature of 580 °C for 2 h, a copper leaching rate of 88.26% was obtained; the leaching rate of gold from acid leaching residue was 85.43% by sodium cyanide solution rolling flask leaching. Moreover, in the early stage, the authors carried out a series of studies on the utilization of zinc and iron in gossan. The results showed that a zinc concentrate with a zinc grade of 13.35%, at a zinc recovery of 53.45%, and an iron concentrate with an iron grade of 40.25%, at an iron recovery of 52.19%, were obtained by gravity separation and magnetic separation, respectively [7,8]. At present, the utilization of gossan resources worldwide has few options, is low level, and there is a serious waste of resources. Therefore, it was particularly necessary to conduct in-depth and systematic research on this special resource [9–11].

The main zinc bearing iron ores in gossan ore of sulfide zinc deposit are limonite, siderite, smithsonite, etc. these ores could be decomposed under a high temperature roasting environment, and finally transformed into iron oxide, zinc oxide, and other minerals. The results showed that zinc ferrite could be prepared by grinding and mixing pure iron oxide and zinc oxide in proportion and roasting at 900 °C for 2–4 h [12]. Zinc ferrite showed good catalytic and photocatalytic activity and photoelectric conversion performances [13–16], and had the advantages of high temperature resistance, corrosion resistance, and non-toxicity [17–24]. Zinc ferrite, used for energy storage, gas sensing, catalytic dehydrogenation, coating materials, and as a high temperature gas desulfurization agent, had broad application prospects [25–28], which was another way to realize the efficient utilization of this type of gossan resources. In the previous study [29], the authors found that the main mineral of gossan ore was a zinc-iron mineral. However, Al, Ca, Si, and other elemental compounds also occurred in gossan ores, and inevitably participated in the reaction of zinc and iron minerals in ores, which may change the reaction process and mineral phase transformation of zinc and iron minerals. Therefore, in this study, pure minerals such as iron oxide, zinc oxide, alumina, calcium oxide, and silicon oxide were used to study the reaction behaviors of zinc iron minerals and the formation law of zinc ferrite in the pure mineral system. The effects of adding alumina, calcium oxide, and silicon oxide on the synthesis of zinc ferrite from iron oxide and zinc oxide were mainly investigated under the condition of high temperature roasting, which laid a theoretical foundation for the follow-up study on the new technology of preparing zinc ferrite by mineral phase transformation of gossan ore in zinc sulfide deposit.

2. Materials and Methods

The reagents used in the test were all analytically pure. Zinc ferrite was prepared from pure minerals ZnO and Fe₂O₃ by a high-temperature roasting method. Other oxides used were Al₂O₃, CaO, and SiO₂. The main reaction principle of producing zinc ferrite is shown in Equation (1).

\[ \text{ZnO} + \text{Fe}_2\text{O}_3 \rightarrow \text{ZnFe}_2\text{O}_4 \]  

(1)

The test process was as follows:

1. According to the molar ratio of reactants, the appropriate amount of analytically pure zinc oxide and iron oxide, as well as different amounts of alumina, calcium oxide, and silicon oxide were weighed and mixed by ultrasonic oscillation, and then poured into the zirconium tank of planetary mill.
2. Put the zirconium ball into the zirconium tank, cover the tank cover, put it into the slot symmetrically, set the rotation speed and time of the planetary mill, and carry out the mechanical activation pretreatment of the material at different times.
3. The pretreated materials were taken out and put into the ceramic crucible. The roasting test was conducted under different roasting times and temperatures, and finally the zinc ferrite product was obtained.

X-ray diffraction (XRD, D8 Advance, Bruker, Germany) was used to analyze the phase of the roasted product (maximum output power 3000 W, Cu target, scanning range of 5°–90°). A scanning electron microscope (SEM, su8020, Hitachi, Japan) was used to analyze
the surface morphology of the products (the accelerating voltage was 0.1–30 kV). A specific surface area and pore size distribution of the samples were analyzed by Specific surface analyzer (BET, 3flex3.01, Mike instruments, USA).

3. Results and Discussion

The effects of alumina (Al₂O₃), calcium oxide (CaO), and silicon oxide (SiO₂) on the formation of zinc ferrite from zinc oxide and iron oxide, and the phase transformation characteristics of minerals under a different reactant ratio, activation time, roasting temperature, and roasting time were investigated. Concurrently, the microstructure, specific surface area, and pore size of the roasted products were studied.

3.1. Effect of Al₂O₃ on Preparation of Zinc Ferrite

3.1.1. XRD Analysis of Roasted Products

The content and XRD analysis of roasted products are shown in Table 1 and Figure 1, respectively.

Table 1 and Figure 1a show that the main products were Fe₂O₃, ZnFe₂O₄ (zinc ferrite), and ZnAl₂O₄ (zinc aluminate). With an increase in alumina content, the intensity of diffraction peaks of iron oxide and zinc aluminate gradually increased, while that of zinc ferrite gradually decreased. Concurrently, when the mixture ratio increased from 1:1:0.5 to 1:1:1.5, the content of zinc aluminate increased from 1.8% to 2.7%, the content of zinc ferrite decreased from 91.0% to 84.5%, and the content of residual iron oxide increased from 6.6% to 12.1%, which was also consistent with the intensity of the diffraction peak in Figure 1a. In summary, when the mixture of ZnO, Fe₂O₃, and Al₂O₃ was roasted, alumina could preferentially combine with zinc oxide to form zinc aluminate. Hence, the existence of alumina could hinder the formation of zinc ferrite. Table 1 and Figure 1b indicate that the products of the ZnO, Fe₂O₃, and Al₂O₃ mixture roasted at 750 °C were Fe₂O₃, ZnFe₂O₄, and ZnAl₂O₄. With an increase in activation time, the diffraction peak intensity of zinc aluminate increased, and the amount of zinc aluminate increased from 1.5% to 3.6%. The content of zinc ferrite and zinc aluminate in the roasted products had been changed at a different activation time, which indicated that the activation time could affect the reaction of the mixture. Figure 1c demonstrates that there were diffraction peaks of ZnO, Fe₂O₃, ZnAl₂O₄, and ZnFe₂O₄ in the roasted product at 650 °C. With an increase in roasting temperature, the diffraction peak of alumina disappeared, the diffraction peak intensity of zinc ferrite decreased, and the diffraction peak intensity of zinc aluminate increased. According to Table 1, the content of zinc ferrite was 84.5% at 750 °C and 54.0% at 950 °C. There was a diffraction peak of ferric aluminate in the product when the roasting temperature was 950 °C, while the diffraction peak of ferric oxide was stronger, and the diffraction peak of zinc ferrite was weaker. When the roasting temperature increased from 650 °C to 750 °C, the content of zinc aluminate increased from 0.7% to 7.4%. When the roasting temperature reached 950 °C, the diffraction peak of iron aluminate appeared with a content of 13.2%. With an increase in temperature, alumina could react with zinc oxide to produce more zinc aluminate.

Figure 1d exhibits that the roasted products were mainly Fe₂O₃, ZnAl₂O₄, and ZnFe₂O₄. With an increase in roasting time, the diffraction peak intensity of zinc ferrite increased, and the diffraction peak intensity reached a maximum at 120 min. After that, the diffraction peak intensity of zinc ferrite remained mostly unchanged, while the diffraction peak intensity of zinc aluminate did not change significantly. It can be seen from Table 1 that when the roasting time was 120 min, the highest content of zinc ferrite in the product reached 86.0%, while the content of zinc aluminate was about 2.6%–2.7%. When the roasting time was 60 min, a ferric aluminate of 1% could be obtained; when the roasting time was 240 min, the remaining ferric oxide content increased to 13.9%. In summary, the roasting time had little effect on the formation of zinc ferrite, and the amount of zinc ferrite was the largest at 120 min. The zinc aluminate was mainly produced by alumina,
which could produce zinc aluminate within 60 min, and it was relatively less affected by roasting time.

Table 1. The contents of products under a different molar ratio of ZnO, Fe$_2$O$_3$, and Al$_2$O$_3$ (%).

| Reactant Ratio | Activation Time/min | Roasting Temperature /°C | Roasting Time/min | ZnO | Fe$_2$O$_3$ | Al$_2$O$_3$ | ZnFe$_2$O$_4$ | ZnAl$_2$O$_4$ |
|----------------|---------------------|--------------------------|------------------|-----|------------|------------|-------------|-------------|
| 1:1:0.5        |                     |                          |                  | 0.6 | 6.6        | -          | 91.0        | 1.8         |
| 1:1:1          | 60                  | 750                      | 120              | 0.5 | 9.4        | -          | 88.1        | 2.0         |
| 1:1:1.5        | 0.7                 |                          |                  | -   | 47.9       | -          | 50.4        | 1.7         |

| Reactant Ratio | Activation Time/min | Roasting Temperature /°C | Roasting Time/min | ZnO | Fe$_2$O$_3$ | Al$_2$O$_3$ | ZnFe$_2$O$_4$ | ZnAl$_2$O$_4$ |
|----------------|---------------------|--------------------------|------------------|-----|------------|------------|-------------|-------------|
| 1:1:1          | 60                  | 750                      | 120              | 0.5 | 9.4        | -          | 88.1        | 2.0         |
| 1:1:1.5        | 0.7                 |                          |                  | -   | 47.9       | -          | 50.4        | 1.7         |

Figure 1. XRD patterns of roasted products under different conditions: (a) different ratio; (b) activation time; (c) roasting temperature; (d) roasting time.
3.1.2. SEM Analysis

To study the effect of alumina on the microstructure of roasted products, the effects of different roasting times and temperatures on the microstructure of roasted products were studied under the conditions of the molar ratio of $\text{ZnO}$, $\text{Fe}_{2}\text{O}_{3}$, and $\text{Al}_{2}\text{O}_{3}$ of 1:1:1 and an activation time of 60 min. The results are shown in Figure 2.

![SEM images of roasted products](image)

**Figure 2.** Microstructure of roasted products at different roasting times and temperatures: (a-1,a-2) and (b-1,b-2) denote the SEM images at a roasting temperature of 750 °C, a roasting time of 120 min and 240 min, respectively; (c-1,c-2) and (d-1,d-2) denote the SEM images at a roasting time of 120 min, a roasting temperature of 850 °C and 950 °C, respectively.

Figure 2(a-1,a-2,b-1,b-2) indicate that when the roasting time increased from 120 min to 240 min, the morphology of the particles changed only a little, with a particle size
of less than 3 \( \mu m \), and the particle surface was smooth, with partial agglomeration. Figure 2(c-1,c-2,d-1,d-2) exhibit that the size and morphology of the products changed greatly under the same roasting time and different roasting temperatures. When the roasting temperature was 850 \( ^\circ C \), the particle size was relatively uniform, but there were still some large particles with the characteristics of spherical, ellipsoidal, or irregular shape. The particle surface was smooth and some were aggregated. The particle aggregation was obvious when the roasting temperature was 950 \( ^\circ C \).

3.1.3. BET Analysis

The roasted product was prepared under the following conditions of the molar ratio of \( \text{ZnO, Fe}_2\text{O}_3, \) and \( \text{Al}_2\text{O}_3 \) of 1:1:1, an activation time of 60 min, and a roasting temperature of 750 \( ^\circ C \) for 120 min. The adsorption desorption curve is shown in Figure 3.

![Figure 3. Adsorption desorption curve (a) and pore size distribution (b) of roasted product.](image)

Figure 3a shows that the adsorption isotherms of zinc ferrite formed under the influence of alumina also belonged to three types of adsorption isotherms. According to Figure 3b, the pore size of the mesoporous was about 3–50 nm, and some of the pores were micropores of less than 5 nm. Moreover, the BET specific surface area of the sample prepared under the influence of alumina was 49.43 m\(^2\cdot\)g\(^{-1}\), the total pore volume was 0.12 m\(^3\cdot\)g\(^{-1}\), and the average pore size was 9.54 nm.

3.2. Effect of CaO on Preparation of Zinc Ferrite

3.2.1. XRD Analysis of Roasted Products

XRD analysis of roasted products under different factors (molar ratio of \( \text{ZnO, Fe}_2\text{O}_3, \) and \( \text{CaO}, \) activation time, roasting temperature, roasting time) is shown in Figure 4.

Figure 4a shows that when the molar ratio of \( \text{ZnO to Fe}_2\text{O}_3 \) was 1:1, the roasted products were mainly zinc ferrite and calcium ferrite, and some zinc oxide and iron oxide were not completely reacted. With an increase in \( \text{CaO} \) content, the diffraction peak intensity of zinc ferrite first increased and then decreased. Figure 4b shows that when the activation time was 120 min, the diffraction peak intensity of zinc ferrite was the highest, while the diffraction peaks of \( \text{ZnO and Fe}_2\text{O}_3 \) disappeared. Figure 4c shows that with an increase in roasting temperature, the diffraction peaks of \( \text{ZnO and Fe}_2\text{O}_3 \) disappeared, the diffraction peak intensity of zinc ferrite increased gradually, and the diffraction peak of zinc ferrite was the strongest at 950 \( ^\circ C \). Figure 4d indicates that the roasting product was mainly zinc ferrite. With an increase in roasting time, the diffraction peak intensity of zinc ferrite increased, and the diffraction peak was the strongest when the roasting time was 240 min. In summary, \( \text{CaO} \) could react with \( \text{Fe}_2\text{O}_3 \) to form calcium ferrite, \( \text{ZnO} \) could not completely react with \( \text{Fe}_2\text{O}_3 \) to form zinc ferrite, \( \text{CaO} \) could hinder the formation of zinc ferrite, and roasting temperature was the main factor affecting the reaction.
Figure 4. XRD patterns of roasted products under different conditions: (a) different ratio; (b) activation time; (c) roasting temperature; (d) roasting time.

3.2.2. SEM Analysis

To study the effect of calcium oxide on the microstructure of roasted products, the effects of different roasting times and temperatures on the microstructure of roasted products were studied under the conditions of the molar ratio of ZnO, Fe$_2$O$_3$, and CaO of 1:1:1 and an activation time of 60 min. The results are shown in Figure 5.

Figure 5(a-1,a-2,b-1,b-2) present that under the same roasting temperature, when the roasting time increased from 120 min to 240 min, the particle size of the roasted product only changed a little, with the size range of 1–3 µm. The particle surface was smooth, but the agglomeration phenomenon between particles was obvious. Figure 5(c-1,c-2,d-1,d-2) show that when the roasting time was 120 min, the roasting temperature increased from 850 °C to 950 °C, the particle size of the product was more uniform, and the agglomeration phenomenon of the product was also obvious. In summary, a product with better crystallinity and a more uniform particle size could be obtained under the conditions of a roasting time of 120 min and a roasting temperature of 750–850 °C.
Figure 5. Microstructure of roasted products at different roasting times and temperatures: (a-1,a-2) and (b-1,b-2) denote the SEM images at a roasting temperature of 750 °C, a roasting time of 120 min and 240 min, respectively; (c-1,c-2) and (d-1,d-2) denote the SEM images at a roasting time of 120 min, a roasting temperature 850 °C and 950 °C, respectively.

3.2.3. BET Analysis

The roasted product was prepared under the following conditions of the molar ratio of ZnO, Fe₂O₃, and CaO of 1:1:1, an activation time of 60 min, and a roasting temperature of 750 °C for 120 min. The adsorption desorption curve is shown in Figure 6.
Figure 6a shows that the adsorption isotherms of zinc ferrite formed under the influence of calcium oxide also belonged to three types of adsorption isotherms. According to Figure 6b, the pore size of the mesoporous was about 10–50 nm, and some of the pores were micropores of less than 5 nm. Moreover, the BET specific surface area of the sample prepared under the influence of calcium oxide was 59.69 m²·g⁻¹, the total pore volume was 0.08 m³·g⁻¹, and the average pore size was 5.51 nm.

3.3. Effect of SiO₂ on Preparation of Zinc Ferrite

3.3.1. XRD Analysis of Roasted Products

XRD analysis of roasted products under different factors (molar ratio of ZnO, Fe₂O₃, and SiO₂, activation time, roasting temperature, roasting time) is shown in Figure 7.
Figure 7a shows that when the molar ratio of ZnO to Fe$_2$O$_3$ was 1:1, zinc ferrite was the main component in the roasted product. With the increase in SiO$_2$ content, the diffraction peak intensity of ZnFe$_2$O$_4$ hardly changed. When the content of ZnO in the mixture was insufficient, the diffraction peak intensity of the zinc ferrite product was obviously lower than other conditions. Therefore, the change of SiO$_2$ content in the mixture did not affect the formation of zinc ferrite. Figure 7b shows that the roasted product at 750 °C was zinc ferrite. With the activation time increasing from 30 min to 90 min, the diffraction peak intensity of zinc ferrite obviously did not change. When the activation time was 120 min, the diffraction peak intensity of zinc ferrite was relatively large. Figure 7c shows that ZnO, Fe$_2$O$_3$, and ZnFe$_2$O$_4$ were the main products at the roasting temperature of 650 °C. With an increase in roasting temperature, the diffraction peaks of zinc oxide and iron oxide disappeared, and the diffraction peak intensity of zinc ferrite increased gradually. When the roasting temperature was 950 °C, the diffraction peak of zinc ferrite was the strongest. The roasting temperature had a significant influence on the formation of zinc ferrite from the mixture of ZnO, Fe$_2$O$_3$, and SiO$_2$. When the roasting temperature was 650 °C, zinc oxide and iron oxide did not form zinc ferrite. When the roasting temperature was 950 °C, the content of zinc ferrite was the most. It can be seen from Figure 7d that the main product of the mixture after the reaction was ZnFe$_2$O$_4$. With the increase in roasting time, the diffraction peak intensity of ZnFe$_2$O$_4$ increased, and the diffraction peak intensity was the strongest at 120 min. After that, the diffraction peak intensity of ZnFe$_2$O$_4$ was basically unchanged. In summary, the change of SiO$_2$ content in the mixture could not affect the formation of zinc ferrite. Roasting temperature was a significant factor affecting the formation of zinc ferrite from the mixture of zinc oxide, iron oxide, and silicon oxide.

3.3.2. SEM Analysis

To study the effect of silicon oxide on the microstructure of roasted products, the effects of different roasting times and temperatures on the microstructure of roasted products were studied under the conditions of the molar ratio of ZnO, Fe$_2$O$_3$, and SiO$_2$ of 1:1:1 and an activation time of 60 min. The results are shown in Figure 8.

Figure 8(a-1,a-2,b-1,b-2) show that under certain conditions of the roasting temperature of 750 °C for 120 min, the particle size distribution of the product was more uniform, and the content of fine particles was higher. With the roasting time extended to 240 min, more large particles with a diameter of 2–5 µm appeared in the product, this was because SiO$_2$ agglomerated into large pieces in a high temperature environment for a long time, which made the surrounding small particles of zinc ferrite stick together. Hence, the roasting time of 120 min was more favorable for the formation of zinc ferrite. Figure 8(c-1,c-2,d-1,d-2) show that the overall distribution of zinc ferrite particles prepared was relatively uniform when the roasting time was 120 min; however, when the temperature rose from 850 °C to 950 °C, some large particles appeared, and the surface of zinc ferrite particles became rough. It can be seen that under the condition of the existence of silica, a roasting time of 120 min, and a roasting temperature of 750–850 °C, the product had the characteristics of uniform particle size and good morphology.
Figure 8. Microstructure of roasted products at different roasting times and temperatures: (a-1,a-2) and (b-1,b-2) denote the SEM images at roasting a temperature of 750 °C, a roasting time of 120 min and 240 min, respectively; (c-1,c-2) and (d-1,d-2) denote the SEM images at a roasting time of 120 min, a roasting temperature of 850 °C and 950 °C, respectively.

3.3.3. BET Analysis

The roasted product was prepared under the following conditions of the molar ratio of ZnO, Fe₂O₃, and SiO₂ of 1:1:1, an activation time of 60 min, and a roasting temperature of 750 °C for 120 min. The adsorption desorption curve is shown in Figure 9.
Figure 9. Adsorption desorption curve (a) and pore size distribution (b) of roasted product.

Figure 9a shows that the adsorption isotherms of zinc ferrite formed under the influence of silicon oxide also belonged to three types of adsorption isotherms. According to Figure 9b, the pore size of the mesoporous was about 10–50 nm, and some of the pores were micropores of less than 5 nm. Moreover, the BET specific surface area of the sample prepared under the influence of silicon oxide was 72.33 m²·g⁻¹, the total pore volume was 0.34 m³·g⁻¹, and the average pore size was 17.95 nm.

4. Conclusions

The development and utilization of gossan ore, and the preparation of zinc ferrite by directional phase control of gossan ore from zinc sulfide deposit is of great theoretical and practical significance. In this study, the effect of adding alumina, calcium oxide, and silicon oxide on the synthesis of zinc ferrite from iron oxide and zinc oxide under the conditions of high temperature roasting has been proven. In the roasting process of alumina, calcium oxide, and silicon oxide, mixed with zinc oxide and iron oxide respectively, alumina could be preferentially reacted with zinc oxide to form zinc aluminate. The existence of calcium oxide had an obvious hindrance on the formation of zinc ferrite, while the change of silicon oxide content could not affect the formation of zinc ferrite. The ratio of reactants, activation time, and roasting time had little influence on the reaction, while the roasting temperature had a great influence on the reaction. Under the conditions of the molar ratio of alumina, calcium oxide, and silicon oxide to zinc oxide and iron oxide of 1:1:1, an activation time of 60 min, and a roasting temperature of 750 °C for 120 min, the product had the characteristics of the largest amount, uniform particle size, and good morphology.

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