Research Article

Bing Luo, Tongjiang Peng*, Hongjuan Sun*

Recovery of γ -Fe₂O₃ from copper ore tailings by magnetization roasting and magnetic separation

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Abstract: To comprehensively reuse copper ore tailings, the recovery of γ -Fe₂O₃ from magnetic roasted slag after sulfur release from copper ore tailings followed by magnetic separation is performed. In this work, after analysis of chemical composition and mineralogical phase composition, the effects of parameters in both magnetization roasting and magnetic separation process with respect to roasting temperature, residence time, airflow, particle size distribution, magnetic field intensity, and the ratio of sodium dodecyl sulfonate to roasted slag were investigated. Under optimum parameters, a great number of γ -Fe₂O₃ is recycled with a grade of 66.86% and a yield rate of 67.21%. Meanwhile, the microstructure, phase transformation and magnetic property of copper ore tailings, roasted slag, and magnetic concentrate are carried out.

Keywords: copper ore tailings, γ -Fe₂O₃ recovery, magnetization roasting, magnetic separation

* **Corresponding author: Tongjiang Peng**, Key Laboratory of Ministry of Education for Solid Waste Treatment and Resource Recycle, Southwest University of Science and Technology, Mianyang, Sichuan, 621010, China; Institute of Mineral Materials and Applications, Southwest University of Science and Technology, Mianyang, Sichuan, 621010, China; Center of Forecasting and Analysis, Southwest University of Science and Technology, Mianyang, Sichuan, 621010, China, e-mail: tjpeng@swust.edu.cn

* **Corresponding author: Hongjuan Sun**, School of Environment and Resource, Key Laboratory of Ministry of Education for Solid Waste Treatment and Resource Recycle, Southwest University of Science and Technology, Mianyang, Sichuan, 621010, China; Institute of Mineral Materials and Applications, Southwest University of Science and Technology, Mianyang, Sichuan, 621010, China, e-mail: sunhongjuan@swust.edu.cn

Bing Luo: School of Environment and Resource, Key Laboratory of Ministry of Education for Solid Waste Treatment and Resource Recycle, Southwest University of Science and Technology, Mianyang, Sichuan, 621010, China; Institute of Mineral Materials and Applications, Southwest University of Science and Technology, Mianyang, Sichuan, 621010, China; City College, Southwest University of Science and Technology, Mianyang, Sichuan, 621000, China

1 Introduction

With urgent requirement of the thirteenth 5-Year Plan for circular economy in China, copper ore tailings as one kind of non-ferrous tailings in industrial solid waste have been paid more and more attention to comprehensive disposal and resource utilization. It is estimated that approximately 400 tons of tailings are generated, while every ton of copper produced [1]. Copper ore tailings contain pyrrhotite (Fe_(1-x)S, where $0 \leq x < 0.223$), which has three homogeneous polychromatic variants of iron sulfide mineral and tends to produce acid mine drainage because of oxidation [2,3]. To effectively prevent the environmental risk of the copper ore tailings and recycle the sulfur resource in the copper ore tailings, the sulfur released as SO₂ can be collected to produce sulfuric acid by oxidation roasting as mentioned in the previous study [4]. Meanwhile, the iron units are concentrated in the roasted slag and existed in the form of γ -Fe₂O₃. Magnetite is a defective spinel structure where Fe³⁺ occupies both of A tetrahedral position and of B tetrahedral position with a certain amount of vacancies [5,6]. In addition, γ -Fe₂O₃ is widely used as a magnetic material with advantages such as stabilization, nontoxicity, and low consumption, which has a wide range of application prospects [7]. Therefore, the recovery and utilization of γ -Fe₂O₃ in roasted copper ore tailings are of great importance to save iron resources and promote copper ore tailings more sustainable and respectful to ecological environment.

As we all know, both iron concentrate (Fe₃O₄) and sponge iron (Fe) have some magnetic properties; therefore, magnetic separation is an effective way to recover iron where Fe₃O₄ and Fe are used for iron smelting and steel making [8–11], respectively. Currently, the recovery of Fe₃O₄ and Fe was studied by many researchers using magnetic separation from various metal dressing tailings or other solid wastes rich in iron [12–14]. However, not all iron with a certain phase in tailings and solid wastes are Fe₃O₄ or Fe. It requires magnetizing roasting and direct reduction to modify nonmagnetic iron to magnetic

iron [15–18]. The modified conditions with iron-bearing minerals are summarized in Table 1. However, the current studies about the recovery of $\gamma\text{-Fe}_2\text{O}_3$ remain unsolved. Taking advantage of magnetic properties of $\gamma\text{-Fe}_2\text{O}_3$ will greatly facilitate the recovery of iron in the roasted slag.

Therefore, sulfur release was studied first in previous study [4]. In this paper, the magnetizing roasting of the copper ore tailings followed by magnetic separation is further investigated to recover $\gamma\text{-Fe}_2\text{O}_3$ from the roasted slag. The roles of roasting temperature, residence time, airflow, particle size, magnetic field intensity, and the ratio of sodium dodecyl sulfonate (SDS) to roasted slag were established. Furthermore, the characteristics of magnetic concentrate were discussed as well. More usefully, it born the potential to comprehensively use copper ore tailings and reduce environmental risks.

2 Materials and methods

2.1 Materials

Copper ore tailings were sampled from Liwu copper of Jiulong County in Ganzi Prefecture of Sichuan Province, China. Copper ore tailings were dried at $105 \pm 2^\circ\text{C}$ for 8 h, milled to 100 meshes, and stored, sequentially. The mineralogical phase compositions of the copper ore tailings were determined by X-ray diffraction (XRD, The Netherlands), and the results are shown in Figure 1. The chemical composition of the copper ore tailings was tested by X-ray fluorescence (XRF, The Netherlands), and the results are shown in Table 2.

The main reagent SDS was chemically pure, which was bought from Nanjing Chemical Reagent Co Ltd, China.

The main instruments were LTKC-6-16A programmable energy-saving tubular furnace (Hangzhou Blue Sky Instrument, Co Ltd, China) and XCRQ-50*70 magnetic tube (Wuhan prospecting machinery Factory, Co Ltd, China).

Figure 1: XRD pattern of the copper ore tailings sample.

2.2 Experimental procedures

Six group experiments were carried out to optimize different parameters in both magnetization roasting and magnetic separation on $\gamma\text{-Fe}_2\text{O}_3$ recovery from copper ore tailings, including the ratio of coal to iron ore tailings, roasting temperature, roasting time, airflow, particle size, magnetic field, and the ratio of SDS. The sample was located in a programmable energy-saving tubular furnace (LTKC-6-16A) to magnetizing roast. The magnetized sample was weighed about 20 g in low-intensity magnetic separation first. Subsequently, the best parameters of magnetizing roast were confirmed. Under the optimized parameters, 100 g magnetized sample and 4 g SDS were mixed evenly and then transferred into a 1,000 mL beaker with about 500 mL water and an appropriate amount of alcohol. The magnetic mixture was stirred well and allowed to stand for 10 min. Subsequently, the magnetic mixture was put in the magnetic tube (XCRQ-50*70), in which the optimized magnetic field intensity was investigated by changing current intensity.

Table 1: The modified conditions with iron-bearing minerals

Iron-bearing minerals	Modified condition	Recovery of iron
Fe_2SiO_4	With weak oxidation modification roasting in $\text{CO}\text{--}\text{CO}_2$ mixed gases, FeO in Fe_2SiO_4 was oxidized to Fe_3O_4	Grinding and magnetic separation
Fe_2O_3	Under high temperature (usually $>1,000^\circ\text{C}$), it was reduced to Fe_3O_4 by reducing agent (pulverized coal)	Magnetic separation
$\text{Fe}_{(1-x)}\text{S}$	In coal-based direct reduction with non-coking coal, the final product was Fe , which was used for steelmaking and as a charge of blast furnaces	Magnetic separation

Table 2: Chemical composition of the copper ore tailings (wt%)

Components	Fe ₂ O ₃	SO ₃	SiO ₂	Al ₂ O ₃	Na ₂ O	ZnO	CaO	K ₂ O	MgO
Content	36.35	27.82	22.81	6.03	2.12	1.61	1.16	0.93	0.47
Components	TiO ₂	CuO	P ₂ O ₅	Co ₃ O ₄	PbO	ZrO ₂	MnO	SrO	Total
Content	0.25	0.22	0.10	0.05	0.03	0.02	0.02	0.01	100

2.3 Analytical methods

The mineralogical phase compositions of both raw tailings and roasted samples were tested by X-ray diffraction (XRD, X'Pert pro + 3 kW, The Netherlands, $\lambda = 0.15406$ nm), using Cu K α radiation with continuous scanning step of 0.02° in 2θ ranging from 3 to 80° . The chemical composition of the copper ore tailings was obtained by X-ray fluorescence Spectrometer (XRF- Axios, The Netherlands, wavelength dispersive type, 2.4 kW) while the content ranges from 0.01 to 100% and the sample diameter is 32 mm, using Rh target with $\theta/2\theta$ scanning mode of the goniometer according to JY/T 016-1996. The magnetic properties of magnetic concentrate and copper ore tailings were investigated using a vibrating sample magnetometer (VSM BKT-4500Z, China). The microstructures and elemental analysis of copper ore tailings, roasted slag, and magnetic concentrate were characterized by scanning electron microscopy (Leica Cambridge LTD, 15 kV), with magnifying multiple ranges from 3,000 to 200. The content of γ -Fe₂O₃ was determined by chemical titration. The grade of γ -Fe₂O₃ was equal to the content of γ -Fe₂O₃.

The yield of γ -Fe₂O₃ was calculated as follows:

$$i = \frac{Q_i}{Q_y} \times 100\%, \quad (1)$$

where i is the yield of γ -Fe₂O₃; Q_i is the magnetic concentrate mass; and Q_y is the roasted slag mass.

Ethical approval: The research conducted is not related to either human or animal use.

3 Results and discussion

3.1 Characterization of the copper ore tailings

From Figure 1, the main metal phase in samples was pyrrhotite and lepidocrocite. After pyrrhotite and lepidocrocite were converted to iron in certain ways [12,14,16],

iron could be recycled as an important metal resource for further use. As shown in Table 2, the content of Fe₂O₃ reached 36.35 wt%. When the copper ore tailings were roasted from 600 to 1,200°C, the roasted slag was magnetic which contained γ -Fe₂O₃ [4]. Therefore, it was necessary and meaningful to roast to release sulfur first and recycle γ -Fe₂O₃ subsequently.

3.2 γ -Fe₂O₃ recovery

3.2.1 Effect of the roasting temperature on γ -Fe₂O₃ recovery

Roasting temperature was the most important factor that affects sulfur release and magnetization. Sulfur compounds were easy to decompose and release sulfur with the increasing roasting temperature ranging from 1,000 to 1,350°C [19]. After roasting at 1,200°C with the duration time from 20 to 60 min, the sulfur release rate reached to 99.78% and the residual S content reduced to 0.06% [4]. In this study, roasting temperature was set as 1,000, 1,050, 1,100, 1,150, and 1,200°C, and roasted time was 10 min, with the airflow of 5 L min⁻¹. The magnetic field intensity was 160 mT. Results are shown in Figure 2.

As shown in Figure 2, with the roasting temperature increasing, the grade and yield of γ -Fe₂O₃ increased from 40.15 to 45.37% and from 7.69 to 52.06%, respectively. From 1,000 to 1,200°C, the grade of γ -Fe₂O₃ increased with the increase in roasting temperature. The reaction between Fe_(1-x)S and O₂ was dramatic because of sulfur release [20]. While at 1,050°C, the reaction was accelerated suddenly, which promotes the oxidation of Fe₃O₄ to γ -Fe₂O₃. When high roasting temperatures exceeded 1,200°C, the sintered sample would melt. Then, it was unfavorable to separate magnetic particle from the roasted slag.

3.2.2 Effect of residence time on γ -Fe₂O₃ recovery

Copper ore tailings were roasted at 1,200°C for different roasting time, with the airflow of 5 L min⁻¹. The magnetic

Figure 2: Effect of roasting temperature on $\gamma\text{-Fe}_2\text{O}_3$ recovery.

field intensity was 160 mT. Results are shown in Figure 3. From Figure 3, neither the grade nor the yield was in linear relationship with roasting time. After 10 min, the yield of $\gamma\text{-Fe}_2\text{O}_3$ increased even more and achieved a peak of 73.52%, whereas the grade of $\gamma\text{-Fe}_2\text{O}_3$ was slow, only 49.96%. It indicated that some non-magnetic particles, which attached to magnetic concentrate, were separated together in low-intensity magnetic separation [21].

3.2.3 Effect of airflow on $\gamma\text{-Fe}_2\text{O}_3$ recovery

As an oxidant, air not only affected the rate of sulfur release but also had an influence on the magnetization roasting [22]. Copper ore tailings were roasted at 1,200°C for 30 min with different airflow, and the magnetic field intensity was 160 mT. As shown in Figure 4, whether the

Figure 3: Effect of roasting time on $\gamma\text{-Fe}_2\text{O}_3$ recovery.

Figure 4: Effect of airflow on $\gamma\text{-Fe}_2\text{O}_3$ recovery.

airflow was increased or decreased, both the grade and the yield of $\gamma\text{-Fe}_2\text{O}_3$ decreased. When airflow was low, iron atoms released from pyrrhotite lattice could not combine with oxygen atom completely because of sulfur atom undergoing external diffusion and desorbing as SO_x [23]. When airflow was high, the excess air may decrease the roasting temperature and take away some powder with iron [24]. In this experiment, the optimum airflow was confirmed 5 L min^{-1} .

3.2.4 Effect of particle size distribution on $\gamma\text{-Fe}_2\text{O}_3$ recovery

Copper ore tailings were roasted at 1,200°C for 30 min, with the airflow of 5 L min^{-1} . According to the approximate distribution of particle size in copper ore tailings, standard sieves were selected as 100, 120, 140, 160, and 180 meshes, respectively. The roasted samples were milled and sifted in turn. The magnetic field intensity was 160 mT. From Figure 5, the grade and the yield of $\gamma\text{-Fe}_2\text{O}_3$ increased to 51.20 and 76.25%, respectively, whereas the particle size was 120 μm . As a result of complex embedded features of roasted samples, magnetic separation was not carried out effectively with improper particle [25]. When the particle size was large, the non-magnetic particle would be attached to the magnetic particle and would be magnetized [26], which lead to the decrease in the grade and the increase in the yield simultaneously.

3.2.5 Effect of magnetic field intensity on $\gamma\text{-Fe}_2\text{O}_3$ recovery

Copper ore tailings were roasted under optimal conditions and milled to 120 meshes. Subsequently, the

Figure 5: Effect of size of particles on $\gamma\text{-Fe}_2\text{O}_3$ recovery.

Figure 6: Effect of magnetic field intensity on $\gamma\text{-Fe}_2\text{O}_3$ recovery.

samples were separated in different magnetic intensities selected as 160, 180, 200, 220, 240, and 250 mT, respectively. As shown in Figure 6, the grade and yield increased with magnetic intensity ranging from 160 to 220 mT. Although the magnetic intensity was low, magnetic particles were easily carried away by flowing water under the effects of gravity and scouring, which resulted in the loss of magnetic particles and decrease in grade and yield of $\gamma\text{-Fe}_2\text{O}_3$. Meanwhile, although the magnetic intensity was excessive, gangue particles were absorbed on magnetic particles and were separated together [27,28]. Therefore, the grade and yield of $\gamma\text{-Fe}_2\text{O}_3$ increased to 63.59 and 78.56% with the magnetic intensity 220 mT, respectively.

3.2.6 Effect of the ratio of SDS to roasted slag on $\gamma\text{-Fe}_2\text{O}_3$ recovery

Because of the aggregation between magnetic and non-magnetic particles, the yield was always higher than the grade from the above experimental results. To separate magnetic and non-magnetic particles before magnetic separation, as a kind of surfactant, SDS could be used to disperse the magnetic and non-magnetic particles with certain levels [29]. The magnetic field intensity was 220 mT, with the proportion of SDS and samples at 1:100, 1:50, 3:100, 1:25, and 1:20. Results are shown in Figure 7. It revealed that the grade was increased and the yield was decreased with increasing SDS. When the ratio of SDS to roasted slag elevated from 1:100 to 1:25, the yield decreased dramatically from 72.63 to 67.21% with a corresponding grade ranging from 64.18 to 66.86%. The results showed that SDS could promote to strip and separate magnetic and non-magnetic particles. Therefore, the optimum ratio of SDS to roasted slag was suggested at 1:25.

3.3 Analysis of roasted slag

3.3.1 Phase transformation

Owing to the characteristics of pyrrhotite with more sulfur and less iron, the purpose of magnetization roasting was to release sulfur first [4] and then to oxidize magnetite to maghemite. The possible reactions during magnetization roasting were expressed as follows:

Equation (1) occurs below 1,000°C. Because of the presence of sulfur, the performance of iron products

Figure 7: Effect of the ratio of SDS to roasted slag on $\gamma\text{-Fe}_2\text{O}_3$ recovery.

would be affected. Therefore, it was necessary to raise the temperature to release the sulfur before the recovery of iron. When the temperature reached $1,000^\circ\text{C}$, magnetite appeared as shown in Figure 8. With the increasing temperature, Fe_3O_4 was oxidized to $\gamma\text{-Fe}_2\text{O}_3$.

To investigate the transformation of iron, the roasted slag with the roasting temperature from $1,000$ to $1,200^\circ\text{C}$ for 30 min at airflow 5 L min^{-1} was sampled and analyzed for mineral phase. From Figure 8, Fe_3O_4 appeared which was consistent with equation (2) and could also make the low-intensity magnetic separation easy to realize. Meanwhile, compared with the XRD spectra of copper ore tailings, the pattern of pyrrhotite disappeared, which indicated that pyrrhotite decomposed and was oxidized. Moreover, the pattern of Fe_2O_3 also showed up, which was corresponding to equation (1).

3.3.2 SEM-EDS analysis

The SEM images of copper ore tailings and roasted slag ($1,000^\circ\text{C}$ for 30 min, airflow 5 L min^{-1}) are shown in Figure 9. Meanwhile, the energy spectrum of A/B area (Figure 9a and b) was also presented together. From Figure 9b, the roasted slag was light sintered. In the roasting process, crystal water of samples evaporated gradually and SO_2 generated during the reaction volatilize [26]. This may have resulted in the appearance of porous and the increase in granularity [16]. Therefore, it was necessary and significant to grind and mill the sample to a certain extent. From the energy spectrum of roasted slag, S was still present in samples, which indicated that the sulfur release reaction was not complete. Therefore, the roasting temperature should be raised appropriately to release residual sulfur before the recovery of magnetic concentrate.

3.4 Analysis of magnetic concentrate

3.4.1 XRD analysis

The magnetic concentrate was separated from the roasted slag as above, and Figure 10 presents its XRD pattern. From Figure 10, maghemite was the major metallic mineral phases in magnetic concentrate. Moreover, quartz fine-disseminated with maghemite was separated together. According to the report, magnetic concentrate could be sold directly when the content of total Fe and S exceed 65% and was less than 0.1% [21], respectively. Obviously, the content of

Figure 8: XRD spectra of roasted slag at different roasting temperature.

$\gamma\text{-Fe}_2\text{O}_3$ was 66.86% with sulfur 0.08% from Table 3. Therefore, the magnetic concentrate powder could be used in the powder metallurgy industry.

3.4.2 SEM-EDS analysis

Figure 11 shows the SEM image of magnetic and the energy spectrum of C area. From Figure 11, some magnetic concentrate were spherical and granular. Meanwhile, coarser particle in magnetic fraction and finer sized particles at the non-magnetic aggregated together. Fe, O, and Si were included in C area, which was corresponding to the XRD analysis.

3.4.3 Magnetic property

The saturation magnetization (M_s) of copper ore tailings, roasted slag ($1,200^\circ\text{C}$ for 30 min, airflow 5 L min^{-1}), and magnetic concentrate at above optimum conditions were studied in VSM, and the results are shown in Figure 12. It

Figure 9: The SEM images and energy spectrum of A/B area in copper ore tailings and roasted slag. (a) Copper ore tailings; (b) roasted slag at 1,000°C for 30 min, airflow 5 L min⁻¹.

was obvious that M_s of copper ore tailings increases from 4.85 to 11.55 emu/g after magnetizing roasting, which indicated that the magnetic properties may vary more or less under different roasting conditions [30]. Moreover, the maximum M_s of the magnetic concentrate was 12.94 emu/g. These results were attributed to the conversion of weak $Fe_{(1-x)}S$ to maghemite in magnetizing roasting and the segregation of magnetic particles and non-magnetic particles by wet magnetic separation [31].

3.5 The process steps in separation and recovery of $\gamma\text{-Fe}_2\text{O}_3$

The process of separation and recovery of $\gamma\text{-Fe}_2\text{O}_3$ is summarized in Figure 13. The pyrrhotite rich in copper

Figure 10: XRD spectra of magnetic concentrate.

Table 3: Chemical composition of the magnetic concentrate (wt%)

Components	Fe_2O_3	SiO_2	Al_2O_3	MgO	ZnO	CaO	Na_2O	K_2O	CuO	Co_3O_4
Content	66.86	17.30	7.08	2.37	1.76	1.14	1.07	0.92	0.92	0.37
Components	TiO_2	P_2O_5	SO_3	PbO	BaO	MnO	ZrO_2	Rb_2O	As_2O_3	Total
Content	0.30	0.12	0.08	0.04	0.03	0.03	0.02	0.01	0.01	100%

ore tailings was oxidized to $\gamma\text{-Fe}_2\text{O}_3$ which was highly magnetic in magnetizing roasting. Meanwhile, sulfur in the form of SO_2 was released from the inside of pyrrhotite to the surface through slits. After magnetizing roasting, magnetic particles and non-magnetic particles were coated with each other. Therefore, it was imperative to crush and mill roasted slag to disperse $\gamma\text{-Fe}_2\text{O}_3$ and Si–Ca–Mg–Al minerals. Subsequently, magnetic $\gamma\text{-Fe}_2\text{O}_3$ was separated in magnetic separation tube.

4 Conclusions

- (1) Major mineral phases of copper ore tailings were quartz, biotite, clinocllore, pyrrhotite, and lepidocrocite. The content of total Fe was 36.35%. Most of the iron existed in non-magnetic characteristics, which were hard to separate.
- (2) With six groups' experiments, the optimal roasting and magnetization separation conditions were determined as follows: roasting at $1,200^\circ\text{C}$ for 30 min with airflow 5 L min^{-1} and the ratio of SDS to roasted slag ($120\text{ }\mu\text{m}$) is 1:25 in magnetic field intensity 220 mT. Under these conditions, the yield was 67.21%, the content of $\gamma\text{-Fe}_2\text{O}_3$ separated from roasted slag was

Figure 12: Magnetization curve of copper ore tailings, roasted slag, and magnetic concentrate.

66.86% with sulfur 0.08%, and the M_s of magnetic concentrate was 12.94 emu/g.

- (3) As for tailings or waste rich in sulfur and iron, the recycle process was demonstrated to be feasible, where sulfur release from copper ore tailings with optimal roasting conditions first and iron recovery from roasted slag subsequently.

Figure 11: The SEM images and energy spectrum of C area in magnetic concentrate.

Figure 13: The diagram of the process steps in separation and recovery of $\gamma\text{-Fe}_2\text{O}_3$ from copper ore tailings.

(4) The technology of magnetization roasting and wet magnetic separation to recycle $\gamma\text{-Fe}_2\text{O}_3$ could be extended to other iron-bearing tailings or waste, including iron ore tailings, pyrite-containing tailings, pyrrhotite-containing tailings, and so on.

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