Enrichment of iron element from sulfur-containing iron tailings by S-HGMS technology

Ya-qian Zhou^a, Rui-ming Yang^a, Peng-hui Guo^a, Su-qin Li^{a, *}, and Yi Xing^{b, *}

a. School of Metallurgical and Ecological Engineering, University of Science and Technology Beijing, Beijing 100083, China b. School of Energy and Environment, University of Science and Technology Beijing, Beijing 100083, China

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Abstract

Comprehensive utilization of sulfur-containing iron tailings (SIT) not only solves environmental problems but also creates certain economic value. The iron element from SIT was enriched by the superconducting high gradient magnetic separation (S-HGMS) technology in this study. In the experiments, the total iron content (TFe) was increased from 26.3% to 60.5% with the total sulfur content (TS) of 5.9% under the optimal parameters, i.e., a magnetic flux density of 0.4 T, a slurry flow rate of 1500 mL/min. The high-quality sulfur-containing material with TFe of more than 60% was obtained, which can be used for preparing high-sulfur free cutting steel. The S-HGMS technology can realize the resource utilization of iron tailings with high added value.

Key words: S-HGMS, sulfur-containing iron tailings, separation and purification, sulfur-containing material, resource utilization

1. INTRODUCTION

Iron tailings are produced in the steel industry as the main by-product [1]. In China, the total accumulation of iron tailings has exceeded 7.5 billion tons, and the annual emission is about 500 million tons [2]. The stacking of iron tailings occupies vast areas of land and causes environmental pollution [3]. Especially, the SIT considerably damages the atmosphere, water source, soil and biological community. Due to the extensive mining methods in the past, a large amount of iron element remains in the SIT produced, which has enormous economic value. Therefore, the efficient enrichment of valuable elements from the SIT and reduction of sulfur pollution has attracted the attention of many researchers.

Currently, magnetic separation is the primary purification method of iron from SIT [4]. However, the traditional magnetic separation technologies have several shortcomings, such as low separation efficiency, large energy consumption, complex process, etc. Moreover, the tailings contain a large number of micro-fine magnetic particles, the separation process such as gravity separation and flotation are also difficult to achieve satisfactory results [5]. These methods substantially affect the comprehensive utilization efficiencies of SIT. The advent of S-HGMS technology effectively solves the deficiency of traditional technology.

The S-HGMS is a green and efficient separation technology, generating a stable high-intensity magnetic field [6-7]. Its energy consumption is only one-tenth of that of electromagnetic separation technology, and it has the advantages of simple equipment and convenient operation [8]. In addition, comparing with permanent magnet materials, S-HGMS technology can provide a wider range of magnetic fields. When large amounts of tailings slurry through the magnetic field area in a small space, the micro-fine magnetic particles that cannot be captured by the traditional beneficiation technology are adsorbed by the steel wool. This technology significantly improves the recovery of the iron element [9-10].

Based on a large amount of successful experience accumulated by our research group in the application of S-HGMS technology ^[11], we tried to develop a novel approach to enriching the iron element from the SIT, so as to realize the resource utilization of solid waste.

2. EXPERIMENTAL

2.1. The raw material

The raw material is the SIT from Shougang Group Co. Ltd. Based on a series of analysis methods, such as XRF, XRD and SEM, the chemical compositions and mineralogical microstructure were analyzed. Subsequently, the XRF analysis results of the tailings are clearly shown in Table 1, the contents of Fe and S which are 26.34% and 12.00%, respectively. Meanwhile, the XRD analysis results are presented in Fig.1(a), the iron in the SIT mainly exists in the form of magnetite (Fe₃O₄), pyrrhotite ($Fe_{1-x}S$) and pyrite (FeS_2). In addition, other minerals include $(Na_{Ca})_2(Mg_{Mn_{Fe}})_5Si_8O_{22}(OH)_2$, Mg₂Al₄Si₅O₁₈ and Na(Zn_{.8}Fe_{.2})PO₄. Then, the SEM analysis results are showed in Fig.1(b), large and micro-fine particles that simultaneously exist in the tailings with significantly different sizes.

^{*} Corresponding author: lisuqin@metall.ustb.edu.cn

CHEMICAL COMPOSITION OF SULFER-CONTAIMING IRON TAILINGS.							
Chemical composition	Fe	Si	S	Mg	Ca	AL	Other
Content (%)	26.34	12.00	12.49	4.45	3.00	2.82	38.9
		THE	TABLE2 Main Parameter	S OF MAGNET.			
Item				Specification			
Central magnetic flux density				5.5T			
Magnetic field gradient				201/m			
Room temperature aperture				200mm 1520			
Equipment neight				1520mm 1480mm			
Weight				148011111 Ot			
Working temperature				4 2K			
Power consumption				8kW			
		▲ Fe ₃ O ₄ ♥ Fe ³ ♥ Fe ³ ₂ ♥ (Na,Ca) ₂ (Mg,Mn, ★ Mg ₂ Al ₂ Si ₃ O ₁₈ ■ Na(Zn ₃ Fe ₂)PO ₄	(a) Fe) ₅ Si ₈ O ₂₂ (OH) ₂			(b)	

 TABLE 1

 CHEMICAL COMPOSITION OF SULFER-CONTAIMING IRON TAILING:

(b) SEM image of the raw tailings

Fig. 1. Compositions and microstructure of the raw tailings.

2.2. Experimental system

The main experimental equipment of S-HGMS was developed by the Institute of High Energy Physics of the Chinese Academy of Sciences. The principal parameters of the equipment were summarized in Table 2. In the process of magnetic separation, the characteristics of magnetic medium are normally depended on steel wool ^[12]. The steel wool with wire diameter of 0.06-0.08 mm were put into two structural units with the total volume of 390 cm² to enhance the magnetic field range and the gradient of the magnetic separation system ^[13]. The recovery of iron element was regulated by controlling the background magnetic induction and slurry flow rate.

(a) XRD pattern of the raw tailings;

After crushing and screening, slurry of SIT with a specific concentration was fed into the S-HGMS separator under the condition of continuous agitation. The addition of steel wool produced a high gradient magnetic field in the separator. Afterwards, the magnetic particles were recovered efficiently, and the non-magnetic particles flowed out through the magnetic field with the slurry. Finally, the slurry particles adsorbed by steel wool was detected after drying. The schematic diagram of the superconducting magnetic separator and filters are illustrated in Fig. 2.

2.3. Ancillary devices

The auxiliary equipment used included ball

mill(PM10L), constant temperature oven (JXX1-277607), peristaltic pump (WT600-1F), analytical balance (AUY220), X-ray camera (D/MAX-RB), Scanning Electron Microscope (SUPRATM55), constant temperature magnetic stirrer (HJ-3), etc.



Fig. 2. Schematic diagram of S-HGMS

3. RESULTS AND DISCUSSION

3.1. Effect of magnetic flux density

Magnetic flux density was an essential factor affecting magnetic separation efficiency. The different strengths from 0.2 to 5 T were attempted with a slurry flow rate of



Fig. 3. The effect of magnetic flux density on separation efficiency.

500 mL/min, a steel wool filling volume of 10 g and a particle size of <45 μ m. The test outcomes were shown in Fig. 3. The iron element of the tailings significantly increased after passing the magnetic field. The best separation efficiency was obtained at 0.4 T, and the TFe reached 41.8%. Then, the TFe decreased with the increase of magnetic flux density.

When the magnetic flux density was excessively high, the magnetic force of the slurry particles was much greater than the fluid resistance. Meanwhile, the occurrence of magnetic flocculation phenomenon led to other non-magnetic particles were adsorbed on the steel wool. It would directly reduce the efficiency of magnetic separation. Compared with the traditional magnetic separation technologies, S-HGMS technology can provide a wider range of stable magnetic field. Under the condition of low magnetic flux density, the micro-fine magnetic particles in tailings can be effectively collected and the weak magnetic particles with high impurity content can be removed. Hence, 0.4 T was used as the best magnetic flux density in subsequent experiments.

3.2. Effect of slurry flow rate

In order to investigate the effects of slurry flow rate on separation efficiency, the slurry flow rate was established at 500~2000 mL/min under certain conditions, namely a magnetic flux density of 0.4 T, a steel wool filling volume of 10 g and a particle size of <45 μ m. Moreover, the TFe rose as the increase of slurry flow rate and reached a maximum value of 42.9% at the slurry flow rate of 1500ml/min, as illustrated in Fig.4. Then, declined with its further increase.

This was attributed to the magnetic force of iron-containing particles in the slurry is less than the fluid resistance at this time. Some of the magnetic particles adsorbed on the outer layer of the steel wool are washed away by the slurry. Furthermore, the residence time of the slurry in the magnetic separation device is too short, leading to the decline of iron recovery. Therefore, 1500 ml/min was used as the best slurry flow rate in the subsequent experiments.

3.3. Effect of steel wool filling volume



Fig. 4. The effect of slurry flow rate on separation efficiency.



Fig. 5. The effect of steel wool filling volume on separation efficiency.

Apart from the slurry flow rate and magnetic flux

density, the steel wool filling volume could significantly influence the separation efficiency. Moreover, the steel wool filling volume was fixed at 2.5 ~20 g, and the particle size of the tailings was less than 45 μ m. It proceeded to select the best values of the previous influencing factors as the experimental conditions. Fig.5 shows that the separation effect decreased with the increase of the volume of steel wool. The most optimal recovery effect took place when the steel wool filling volume had a minimum value of 2.5 g, and the TFe reached 46.7%.

This is because when the steel wool was excessive, the slurry flow resistance increased. As the non-magnetic particles cannot smoothly pass through the steel wool which caused mechanical inclusion. Therefore, the follow-up experiment used a steel wool filling volume for 2.5 g.

3.4. Effect of particle size

In order to explore the effect of particle size on purification efficiency, the SIT was ground and screened to make the particle size less than 250 μ m, 150 μ m, 75 μ m and 45 μ m, respectively, and other factors were fastened on the optimal value. It could be seen from Fig.6 that the finer the particle size of the tailings, the better the separation and purification effect of iron element.

The smaller particle size can separate the magnetic minerals from other impurities, reducing the number of minerals associated with them and improving purification efficiency. Given the treatment effect and the difficulty of screening and crushing, the appropriate particle size of less than 45 μ m was selected.

3.5. Further purification of SIT

Considering that the recovered tailings may be enriched again after passing through the magnetic field, the 60 g tailings passed through the magnetic field three times under the best parameters. As showed in Fig.7, the TFe was increased to 60.5% and TS was reduced to 5.9%. Moreover, the multiple S-HGMS treatments can effectively improve the iron grade of tailings. Therefore,



Fig. 6. The effect of particle size on separation efficiency.



in the actual production process, passing the slurry through the magnetic field several more times to achieve a better separation and purification effect can be considered.

The sulfur-containing material that had been recovered was analyzed by XRD and SEM. As can be seen from the Fig.8(a), under the condition of less than 45 μ m, the S-HGMS technology can effectively enrich magnetite (Fe₃O₄) and pyrite (Fe_{1-x}S), as well as remove other impurities. Meanwhile, there were not only large particles, but also agglomerated small particles in the treated tailings, as illustrated in Fig.8(b). This indicated that S-HGMS technology is also effective in recovering micro-fine magnetic particles and has a higher separation efficiency than traditional magnetic separation.



Fig.7. The purification effect of iron and sulfur in different treatment stages



(a)XRD pattern of the as-prepared sulfur-containing material; (b) SEM image of the as-prepared sulfur-containing material. Fig.8. Compositions and microstructure of the as-prepared sulfur-containing material

4. CONCLUSIONS

The S-HGMS technology has been successfully applied to the purification of SIT. The iron elements in SIT can be efficiently enriched to prepare the sulfur-containing material. The appropriate parameters are as follows: a magnetic flux density of 0.4 T, a slurry flow rate of 1500 mL/min, a particle size of <45 μ m and a steel wool filling volume of 2.5g. Under this conditions, the content of TFe was increased to 60.5% from 26.3% with the sulfur content of 5.9%, and it can be used for

preparing high-sulfur free cutting steel. The S-HGMS technology has the advantages of a small occupation area, low energy consumption and efficient separation process. It would provide a new idea of separation and purification to realize the green and efficient recovery of solid waste.

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REFERENCES

- D.Q. Deng, G.D. Cao, and Y.X. Zhang. "Experimental Study on the Fine Iron Ore Tailing Containing Gypsum as Backfill Material," *Advances in Materials Science and Engineering*, vol. 2021, pp. 1-9, 2021.
- [2] Y. Y. Li, X. Y. Tian, X. He, et al., "Comprehensive Reutilization of Iron in Iron Ore Tailings: Preparation and Characterization of Magnetic Flocculants," *Environmental Science and Pollution Research*, vol. 27, no. 9, pp. 37011–37021, 2020.
- [3] D. Kossoff, W. E. Dubbin, M. Alfredsson, et al., "Mine Tailings Dams: Characteristics, Failure, Environmental Impacts, and Remediation," *Applied Geochemistry*, vol.51, pp. 229-245, 2014.
- [4] A. L. Arol and A. Aydogan, "Recovery Enhancement of Magnetite Fines in Magnetic Separation," *Colloids & Surfaces A Physicochemical & Engineering Aspects*, vol. 232, no. 2-3, pp. 151-154, 2004.
- [5] F. Mishima, S. Takeda, M. Fukushima, et al., "A Superconducting Magnetic Separation System of Ferromagnetic Fine Particles from a Viscous Fluid," *Physica C: Superconductivity and Its Applications*, vol. 463-465, pp. 1302-1305, 2007.
- [6] J. Xu, D. Xiong, S. Song, et al., "Superconducting Pulsating High Gradient Magnetic Separation for Fine Weakly Magnetic Ores:

Cases of Kaolin and Chalcopyrite," *Results in Physics*, vol. 10, pp. 837-840, 2018.

- [7] Y. G. Kim, J. B. Song, G. Y. Dong, et al., "Purification of Chemical Mechanical Polishing Wastewater via Superconducting High Gradient Magnetic Separation System with Optimal Coagulation Process". *IEEE Transactions on Applied Superconductivity*, vol. 25, no. 3, pp. 1-5, 2015.
- [8] J. B. Song, K. L. Kim, D. Yang, et al., "High-Tc Superconducting High Gradient Magnetic Separator Using Solid Nitrogen Cooling System for Purification of CMP Wastewater," *IEEE Transactions* on Applied Superconductivity, vol. 23, no. 3, 2013.
- [9] C. Q. Yang, S. Q Li, Z. J. Guo, et al., "Application and Prospect of Superconducting High Gradient Magnetic Separation in Disposal of Micro-Fine Tailings," *Materials Science and Engineering*, vol. 275, pp. 012044, 2017.
- [10] C. T. Yavuz, A. Prakash, J. T. Mayo, et al., "Magnetic Separations: From Steel Plants to Biotechnology," *Chemical Engineering Science*, vol. 64, no. 10, pp. 2510-2521, 2009.
- [11] C. Q. Yang, S. Q. Li, C. Q Zhang, et al., "Application of Superconducting High Gradient Magnetic Separation Technology On Silica Extraction from Iron Ore Beneficiation Tailings," *Mineral Processing & Extractive Metallurgy Review*, vol. 39, no. 2, pp. 44-49, 2017.
- [12] X. Y. Zheng, Y. N. Wang, D. F. Lu, et al., "Theoretical and Experimental Study on Elliptic Matrices in the Transversal High Gradient Magnetic Separation," *Minerals Engineering*, vol. 111, pp. 68-78, 2017.
- [13] Y. G. Kim, J. B. Song, D. G. Yang, et al., "Effects of filter shapes on the capture efficiency of a superconducting high-gradient magnetic separation system," *Superconductor Science & Technology*, vol. 26, no. 8, 2013.