



Recovery of iron from waste ferrous sulphate by co-precipitation and magnetic separation



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Abstract: Magnetite concentrate was recovered from ferrous sulphate by co-precipitation and magnetic separation. In co-precipitation process, the effects of reaction conditions on iron recovery were studied, and the optimal reaction parameters are proposed as follows: $n(\text{CaO})/n(\text{Fe}^{2+})$ 1.4:1, reaction temperature 80 °C, ferrous ion concentration 0.4 mol/L, and the final mole ratio of Fe^{3+} to Fe^{2+} in the reaction solution 1.9–2.1. In magnetic separation process, the effects of milling time and magnetic induction intensity on iron recovery were investigated. Wet milling played an important part in breaking the encapsulated magnetic phases. The results showed that the mixed product was wet-milled for 20 min before magnetic separation, the grade and recovery rate of iron in magnetite concentrate were increased from 51.41% and 84.15% to 62.05% and 85.35%, respectively.

Key words: ferrous sulphate; titanium dioxide; magnetite concentrate; co-precipitation; wet milling; magnetic separation

1 Introduction

Titanium dioxide (TiO_2), the most widely used Ti products, can be used as pigment, as filler in paper, plastics and rubber industries and as flux in glass manufacture [1]. The commercial technologies for the manufacture of pigment grade titanium dioxide are the sulfate process and chloride process. In the sulfate process, a large amount of wastes or toxic by-products, such as spent sulfate acid and ferrous sulphate ($\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$), are produced [2,3]. The total ferrous sulphate production in China was more than 7×10^6 t in 2013, and the annual growth rate of production exceeds 10% [4]. Unfortunately, this waste is less marketable and difficult to be utilized because of its high impurity content [5], which causes not only severe environmental problems but also the waste of iron resource. Consequently, the urgent need for proper utilization of $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ has attracted great attention of the researchers in the world.

At present, ferrous sulphate is principally used as raw material to manufacture iron oxide pigments or a

coagulant for water treatment, new methods for utilization of ferrous sulphate are to prepare alkali ferrates, cation-substituted LiFePO_4 and iron(III) tanning salts, but all of these methods are restricted by insufficient market demand [6–14]. In the present study, a novel method to utilize $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ to prepare magnetite concentrate for steel-making was proposed. Many methods were developed to synthesize magnetic particles of magnetite such as co-precipitation, microemulsions, sol–gel syntheses, and hydrothermal or solvothermal reactions [15–18]. However, the most common method for producing synthetic magnetite particles is the co-precipitation of $\text{Fe}^{2+}/\text{Fe}^{3+}$ ions (molar ratio 1:2) by sodium hydroxide or ammonia solution [19].

We focused on the preparation of magnetite concentrate from ferrous sulphate by co-precipitation and magnetic separation. Calcium hydroxide was selected as the precipitator and added to ferrous sulphate solution. Consequently, Fe^{2+} ions in the reaction solution were precipitated in the form of $\text{Fe}(\text{OH})_2$, and then $\text{Fe}(\text{OH})_2$ was converted into Fe_3O_4 by air oxidation and heating. Subsequently, the obtained mixture of magnetite and

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gypsum was wet-milled and then separated by magnetic separator, and magnetite concentrate was obtained. The technical parameters for synthesizing magnetite were optimized. Meanwhile, the effect of wet milling prior to magnetic separation on the iron grade in concentrate was studied. This process has the advantages of a lower cost than the traditional process and a simple processing flow.

2 Experimental

2.1 Materials

The dried ferrous sulphate sample used for this study was obtained from Guangdong Huiyun Titanium Industry Corporation Limited, China. Reagent-grade CaO (Guangdong Xilong Chemical Co., Ltd., China) was used directly without further purification.

2.2 Co-precipitation

Magnetite was prepared by the co-precipitation method. 400 mL of ferrous sulphate solution was added to a 500 mL four-necked flask fitted with a reflux condenser and electric heater, and the concentrations of ferrous ions in solution ranging from 0.2 to 0.6 mol/L were examined. To obtain lime milk, purified water was added to calcium oxide (CaO) with a mass ratio of liquid to solid 3:1 under vigorous stirring. The lime milk with a molar ratio of CaO to Fe^{2+} ranging from 0.8:1 to 1.6:1 was dropwise added into the solution under sufficient stirring. The reactor was heated to a certain temperature ranging from 60 to 90 °C at 10 °C increment, followed by an air flow (1 L/min) bubbled into the solution. The reaction time was maintained between 2 and 4 h at 0.5 h increment. After the reaction, a mixed precipitate of magnetite and gypsum was filtrated and then dried in a vacuum atmosphere at 80 °C.

2.3 Magnetic separation

The obtained mixed product was fully ground in a mortar, and magnetic separation was performed on the slurry of mixed product using a self-designed magnetic separator, as shown in Fig. 1. The magnetic separator is composed of stirring axis, cylinder, permanent magnets and stirring paddle. Stirring axis, cylinder and stirring paddle are made of stainless steel. The cylinder has dimensions of 27 mm in outer diameter, 21 mm in inner diameter and 115 mm in height. The upper end of the stirring axis can be fixed to an electric mixer which can adjust stirring rate, while the lower end is welded to the cylinder cover. The cylinder cover can be screwed to the cylinder surface, and a stirring paddle is welded to the cylinder bottom. The permanent magnets are composed of 4 identical magnet rings that superimpose together, and stuck into the cylinder with the help of cylinder cover. The magnet ring is made of N45H Ru–Fe–B

magnet with dimensions of 20 mm in outer diameter, 12 mm in inner diameter and 25 mm in thickness, and radially magnetized with a maximum surface magnetic induction intensity of 0.509 T. 10 g of mixed product and 400 mL of purified water were added to a 500 mL high-type beaker for magnetic separation, and the stirring rate and time were held constant at 600 r/min and 1 h, respectively. Finally, the magnetic fractions were filtered, dried, weighed and subjected to chemical analysis.

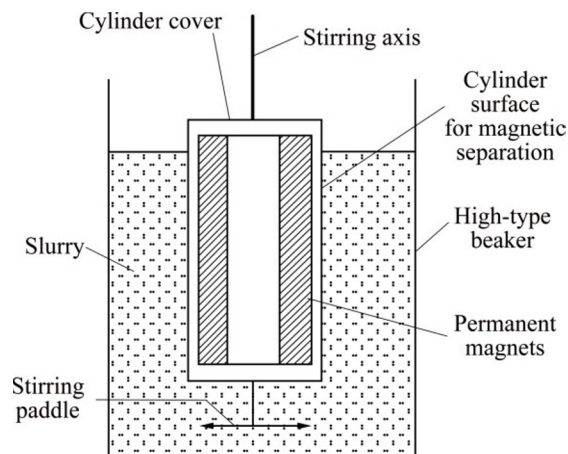


Fig. 1 Schematic diagram of self-designed magnetic separator

Based on the single factor experiments described above, the scale-up magnetite preparation procedure was carried out in a 5 L four-necked flask under the optimum experimental conditions.

4 L ferrous sulphate solution, with ferrous ion concentration of 0.4 mol/L and $n(\text{CaO})/n(\text{Fe}^{2+})$ of 1.4:1, respectively, was placed in the flask under sufficient stirring and treated at 80 °C, followed by an air flow (0.6 m³/h) bubbled into the solution. When the mole ratio of Fe^{3+} to Fe^{2+} in the reaction solution reached 1.9–2.1, the reaction was stopped. Finally, the obtained mixture of magnetite and gypsum was filtrated, dried and ground for the next experiments.

Magnetic separation was performed on a slurry of mixed product (5% in solid) using a low intensity magnetic separator (CRIMM DC CXGd50, Changsha Research Institute of Mining and Metallurgy Co., Ltd., China). A targeted magnetic induction intensity ranging from 0.05 to 0.25 T was used for 10 min magnetic separation. The field was then switched off and the magnetite fraction was washed, filtered, weighed and subjected to various analyses.

The wet milling pretreatment was conducted in a ball mill in a mixture with a mixed product-to-water mass ratio of 1:40 at room temperature and a rotation speed of 130 r/min. The milling time varied from 0 to 40 min, and the treated mixed products were used directly for magnetic separation under the previous best magnetic separation conditions.

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