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Magnetic Separation of Impurities from Hydrometallurgy Solutions and Waste Water Using Magnetic Iron Ore Seeding

Haisheng Han, Wenjuan Sun, Wei Sun and Yuehua Hu

Abstract

The removal of iron ion from leaching solution is critical for the recovery of value metals, with the method of choice commonly being crystallization (precipitation). This paper summarized the new improvements in iron removal by precipitation methods in recent years and proposed a novel process, magnetic seeding and separation. The new process can promote iron precipitate aggregation and growth on the surface of the magnetic iron ore seeds. A core-shell structure was formed of iron precipitate and magnetic iron ore seeds, which can be magnetized and coalesced in magnetic field, accelerating the solid-liquid separation. The efficient magnetic flocculation and separation offset the poor settleability and filterability of the residues, contributing to the development of the hydrometallurgy process. Moreover, magnetic seeding and separation was also used for the removal of organic and inorganic contaminants from wastewater, significantly improving the purification efficiency. Therefore, iron ore not only played an important role in mining and steel manufacture, but also can be used to solve some problems in crossing fields.

Keywords: leaching solution, goethite process, iron removal, magnetic seed

1. Introduction

Iron is one of the most abundant elements in the earth's crust. It always coexists with metals in the ore, mainly exists in the form of hematite, magnetite and muscovite on the surface of particles or in the inclusions inside crystals [1]. In hydrometallurgy, iron, although is converted into insoluble precipitates and removed in advance by sulfation roasting, soda roasting, acid leaching, etc. during ore pretreatment, still inevitably goes to the aqueous solution with the dissolution of the target metal during the leaching process [2–4]. The classical methods for removing iron in the leaching solution are precipitation, extraction, ion exchange, displacement, and electrowinning [4]. The commonly used method is the precipitation method, which separates iron ions by converting to iron precipitation compounds. According to the different iron precipitation compounds, it can be divided into jarosite [5–6], hematite [7], iron(III) oxide-hydroxide [8] and goethite [9–10] method, etc. The jarosite method produces a large amount of low-grade iron-bearing slag in the application, which is difficult to handle, consumes a large amount of sulfate, and

causes certain environmental problems [5–6]; the hematite method needs to be carried out under high temperature and pressure, which consumes large energy and high CAPEX (capital expenditure) [7]. The filtration efficiency of $\text{Fe}(\text{OH})_3$ colloid precipitation method is low, and it is easy to adsorb a large amount of other valuable metals, causing large metal loss [8].

The goethite method is widely used in hydrometallurgical plants for zinc, copper and nickel as the main process for removing iron because of its low CAPEX and environmentally friendly products [9–10]. In order to ensure the effect and efficiency of iron removal, the goethite process must strictly control the concentration of Fe^{3+} below 1 g/L, and thus developed the two commonly used processes - VM method and EZ method [8–9, 11]. The former firstly reduces all the iron ions to Fe^{2+} , and then slowly oxidizes the Fe^{2+} to Fe^{3+} under hydrolysis conditions to control the content of Fe^{3+} [9], and the latter slowly adds the concentrated pressure leachate containing Fe^{3+} in the precipitation vessel with addition rate of less than the Fe^{3+} hydrolysis rate, thereby forming goethite precipitation [11]. The pH in goethite process is common lower than 4.0, and calcium hydroxide or calcium carbonate is usually used as neutralizer, which will result in a large amount of calcium sulfate mixed with the goethite residue [12]. These mixed residues reduce the filtration efficiency and cause the loss of valuable metals such as Zn and Ni [5, 13–14]. In addition, the residue mixture accumulated in the tailings pond contains heavy metals such as Pb, As, and Cr, which causes pollution of local water and soil. Therefore, improving filtration performance and reducing the loss of valuable metals are two problems that need to be solved urgently in the traditional goethite precipitation method.

This article summarizes the new improvements in iron removal by precipitation methods in recent years, and on this basis, proposes a novel iron removal process - magnetic seeding and separation. A core-shell structure is formed by precipitating and growing iron on the magnetic seeds surface, and achieves high-efficiency solid-liquid separation by magnetic separation. The new process remarkably reduces the loss of valuable metals in iron removal. Magnetic seeding and separation processes have not only been successfully used in the removal of iron from hydrometallurgical leachate, but also shown good application prospects in wastewater and soil pollution treatment.

2. Iron removal in nickel and zinc leaching solution

2.1 Magnetite precipitate process

Magnetic flocculation and separation based on the magnetic difference of materials can easily separate magnetic solids from mixtures. It shows higher selectivity and efficiency than centrifugation and filtration, and has been widely used in water treatment, biotechnology and minerals separation [15–18]. As is shown in **Figure 1**, Han et al. [3] studied the feasibility of magnetite precipitation in the hydrometallurgical nickel leaching solution. Under lower oxidation potential, at pH 2.0–2.2 and 90–100°C, the iron ions in the leachate may slowly oxidize and partially precipitate in the form of magnetite. Magnetic flocculation and separation can effectively separate the precipitate from the solution. Unfortunately, the iron precipitation from solution is still dominated by goethite, the magnetite composition is relatively small, and it is difficult to truly achieve effective magnetic separation in industrial applications. But this research of magnetite process of iron removal provides other new ideas of magnetic separation.

2.2 Induced crystallization goethite process

The goethite process can be divided into four stages: (a) hydrolysis to monomers and dimers; (b) the reversible stage involving rapid growth to small polymers; (c) formation of slowly reacting large polymers; and (d) precipitation of a solid phase [19–20]. The goethite precipitation system is a complex system, and the presence and content of different components and iron phases have a greater impact on the precipitation and filtration performance of goethite. As shown in **Figure 2**, the pH and temperature conditions of the sulfate-containing solution determine the existence and content of different iron phases such as hematite, goethite, iron hydroxide and hydroxyl salt [21]. The goethite residues that cause

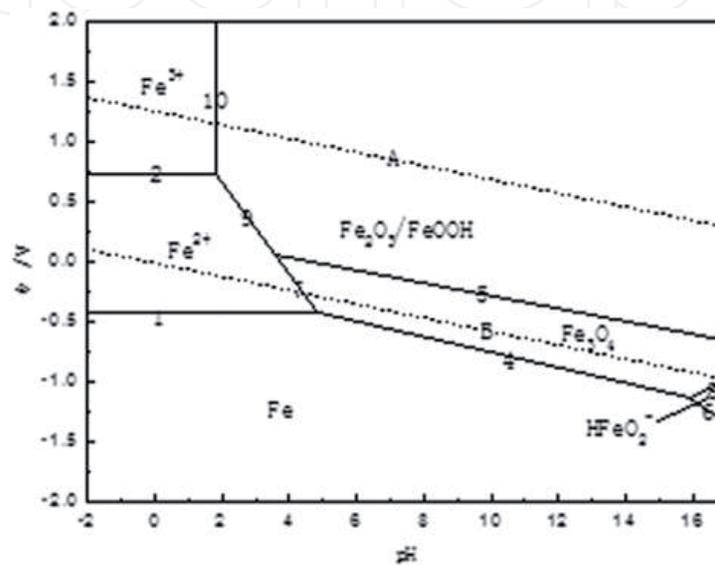


Figure 1.
 Potential/pH diagram for Fe-H₂O system at 100°C.

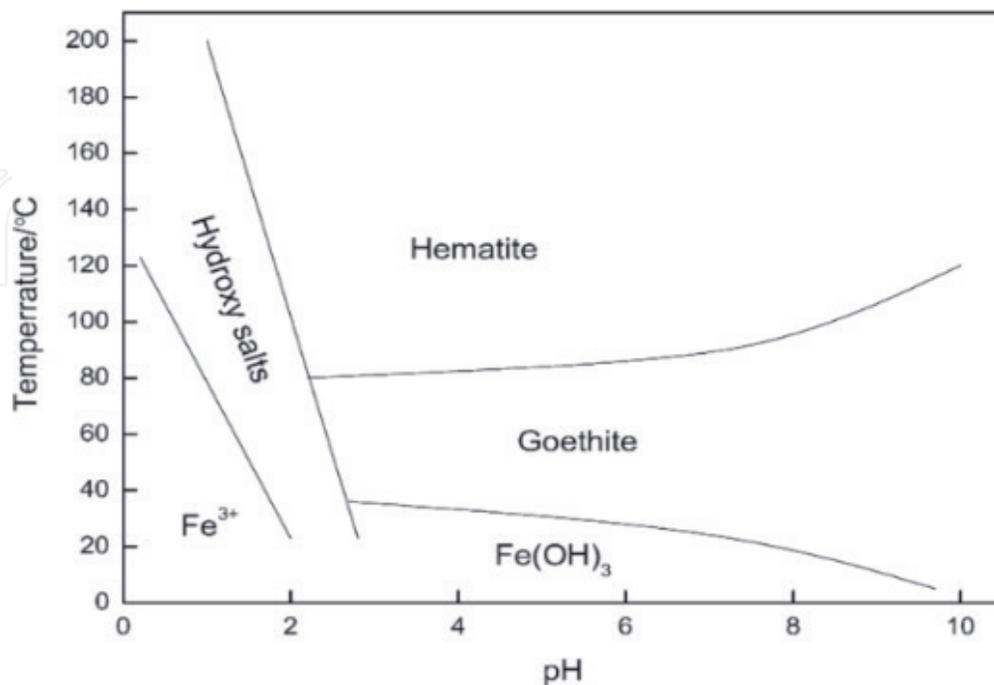


Figure 2.
 Temperature and pH conditions for the precipitation of hematite, goethite, ferric hydroxide, and hydroxy salts (including jarosites) from 0.5 M ferric sulfate solution [21, 23].

filtration difficulties and metal loss are composed of amorphous iron phase, six-line ferrihydrite, poor crystalline goethite, solid solution jarosite phase and silica [2, 22]. Therefore, the crystallinity, size and content of the goethite particles can be controlled by adjusting the pH, thereby improving the separation performance and the loss of valuable metals.

Yue and Han [23] study that as the pH value decreases from 5.0 to 2.0, as shown in **Figure 3**, the crystallinity of goethite decreases, the goethite particles tend to agglomerate, the particle size increases significantly, and the filterability of the precipitate improves. Nickel is lost in the iron precipitate by being incorporated into the crystal lattice and adsorbed on the surface of the goethite particles, and the nickel adsorption loss are related to the specific surface area of the goethite particles. When goethite is in an intermediate transition state at low pH (2.5–3.3), which is between the crystalline state and the colloidal state, the loss of nickel is the least. However, the improvement by only adjusting the pH of the goethite precipitation process is minimal. Chang et al. [24] carefully reduced the pH from 4.0 to 2.5, and the loss of nickel is only reduced by about 10% in the iron precipitation. Moreover, it is not realistic to achieve such detailed condition control in actual industrial applications.

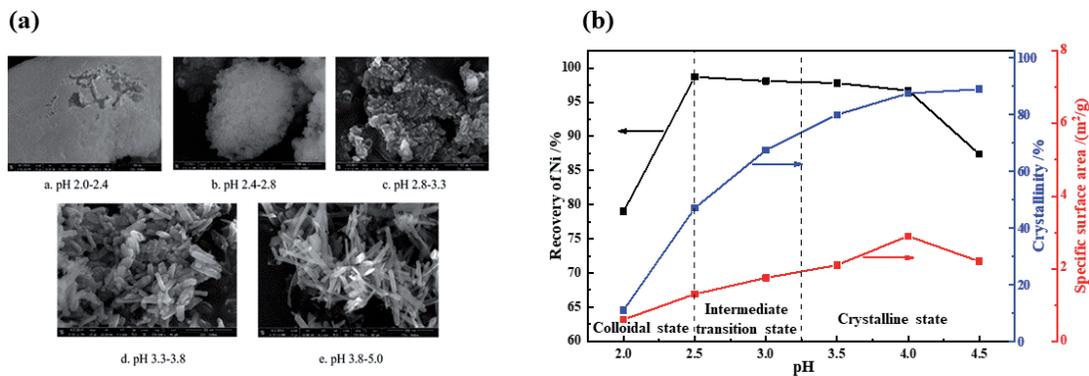


Figure 3. (a) SEM images of the goethite precipitate at different pHs and (b) pH effect for the nickel loss, the crystallinity, and the specific surface area of the precipitate [23, 25].

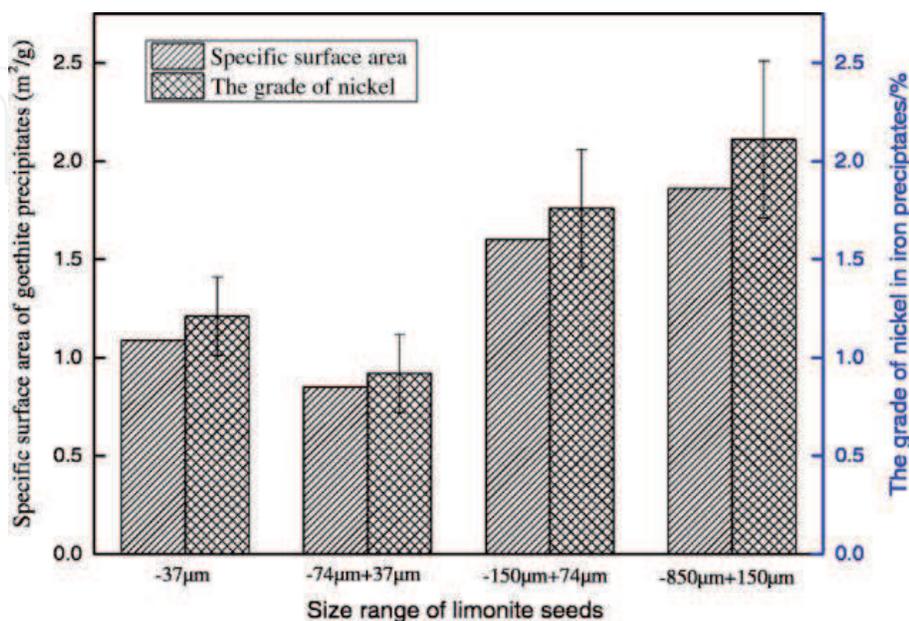


Figure 4. The specific surface area and the nickel grade of iron precipitates with limonite seeds in different size ranges (2 g/L limonite seeds, pH 2.1–2.5, 85°C) [30].

The traditional goethite precipitation method needs to overcome high barriers to the formation of crystals, and often requires a few days of reaction time. The amorphous iron phase appears at this stage, making precipitation separation difficult. Seed induced crystallization can make crystals precipitate and crystallize from the solution at lower solution saturation, pH value and temperature, and has been widely used in the preparation and production of drugs and nanomaterials [26–29]. Han [30] choose natural limonite as the seed crystal of goethite and induce crystallization to improve the problem of poor filterability at the low pH goethite precipitate. As is shown in **Figure 4**, by adding limonite seeds, the particle size of the goethite precipitate is significantly increased. The goethite particles in the particle size range of 37–74 μm have the largest yield and the smallest specific surface area, which can result iron precipitates with a nickel grade of <1%. However, the reduction of metal loss and improvement of filterability are difficult to achieve at the same time by pH control and induced crystallization, one of them must be sacrificed. The intermediate transition state goethite with good filtration performance and minimum metal loss is difficult to accurately induce formation in the actual field industry. It is a need to find other ideas to achieve qualitative progress.

3. Magnetic iron seeding and separation

Han et al. [25, 31–32] combined seed induced crystallization and magnetic separation, and proposed a novel magnetic seeding and separation process, as shown in **Figure 5**. Before the iron is precipitated as goethite, fine-grained maghemite or magnetite particles are added to the leaching solution to make the goethite precipitate and grow on the surface of the magnetic particles, thereby avoiding mixing with the calcium sulfate precipitation in the solution. The iron precipitates on the surface of the goethite to form large magnetic particles with a core-shell structure, and the precipitates are efficiently settled and separated by magnetic separation. The results show that the iron content in the dry iron residue is more than 52% and the Ni content is less than 0.6%, which can be used in industrial applications to deal with a large amount of iron precipitation. After the calcium sulfate precipitation is roasted, 99% of S and As can be removed, and the roasting residue can be respectively used as raw materials for ironmaking and building materials.

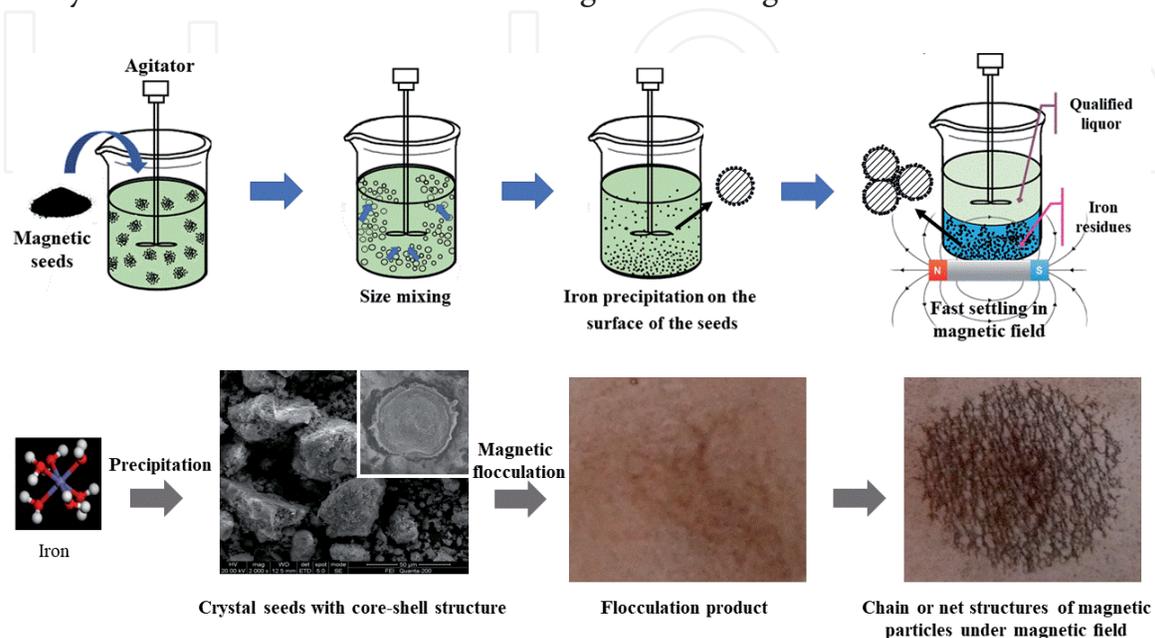


Figure 5. The process of iron precipitation on the magnetic seeds and the magnetic flocculation in magnetic field [3, 25].

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