



Process development for selective precipitation of valuable metals and simultaneous synthesis of single-phase spinel ferrites from saprolite-limonite laterite leach liquors



Jian-ming Gao^{a,b,c,**}, Mei Zhang^{b,c}, Fangqin Cheng^{a,b}, Min Guo^{b,c,*}

^a Institute of Resources and Environmental Engineering, State Environmental Protection Key Laboratory of Efficient Utilization Technology of Coal Waste Resources, Shanxi University, Taiyuan 030006, PR China

^b Shanxi Collaborative Innovation Center of High Value-added Utilization of Coal-related Wastes, Taiyuan 030006, PR China

^c School of Metallurgical and Ecological Engineering, University of Science and Technology Beijing, Beijing 100083, PR China

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ABSTRACT

To comprehensively utilize the laterite resources, the present study focused on the selective recovery of valuable metals Ni, Co or Al, Cr, and concurrent synthesis of spinel ferrites from saprolite-limonite laterite leach liquors by a one-step selective precipitation method. The precipitation behaviors were investigated in detail, as were factors such as precipitant reagent, pH value, precipitation method, and initial metal ion concentration. In optimal conditions, about 93.0% Ni and 92.0% Co could be enriched in the filtrate by using ammonia (aqueous NH₃) solution as precipitant reagent while the recoveries of Al and Cr were 92.0% and 90.0% by using sodium hydroxide (NaOH) solution. After the separation and selective precipitation processes, the precipitates were utilized to synthesize spinel ferrites and the effects of saprolite to limonite laterite mass ratio ($M_{S/L}$) and calcination temperature on the product purity are discussed. Single phase multi-metal doped magnesium ferrites with high purity could be synthesized when the precipitates were obtained using $M_{S/L}$ ratio of 9:1 and 7:3 by using aqueous NH₃ and NaOH solutions as precipitant reagents, and then calcined at 1000 °C for 2 h, respectively. The overall metal recoveries from laterite blends using aqueous NH₃ and NaOH precipitant reagents were 89.2% and 85.4%, respectively.

1. Introduction

With the depletion of sulfide nickel mineral resources and rising demand for nickel, extraction of valuable metals such as Ni and Co from nickel laterite ores have been paid greater attention although such ores have lower grades (0.75–3%) and are more difficult to treat compared with sulfide nickel ores (Oxley and Barcza, 2013; Fan and Gerson, 2013). Recently, various hydrometallurgical methods have been utilized to treat nickel laterite ore, including reduction roasting-ammonia leaching (RRAL) (Ma et al., 2013), high pressure acid leaching (HPAL) (Georgiou and Papangelakis, 1998; Rubisov et al., 2000), atmospheric acid leaching (AL) (McDonald and Whittington, 2008a; McDonald and Whittington, 2008b), sulfation-roasting-leaching (Guo et al., 2009; Li et al., 2010a), heap leaching (Nosrati et al., 2012; Nosrati et al., 2013) and carbonyl process (Terekhov and Emmanuel, 2013). Among these processes, atmospheric acid leaching is an attractive and promising technology for low grade nickel laterite ore with some advantages of simple flowsheet, easy operation, high leaching efficiency and short

leaching time (Luo et al., 2010; Wang et al., 2012). After the leaching operation, the laterite leach liquor contains the target metal ions Ni and Co, as well as many impurity ions such as Mn, Mg, Al, Cr and Fe etc. The contents of some impurity ions can be higher than that of Ni ions in the leach liquors.

Up to now, many efforts have focused on separation and removal of impurities from laterite leach liquors by using chemical precipitation and solvent extraction methods (Guimaraes et al., 2014; Zhu et al., 2014; Cheng et al., 2010a; Cheng et al., 2010b; Hutton-Ashkenny et al., 2015). Compared with the solvent extraction method which requires specific solvent extractants and a complicated extraction process, chemical precipitation is usually utilized to first remove the main impurity ions (Fe ions and/or Al, Cr ions) from the laterite leach liquors (Wang et al., 2011; Chang et al., 2010; Zhang et al., 2015; Shi, 2014; Zhu et al., 2010). The use of chemical precipitation for separation and purification of the valuable metals can result in various problems. First, in acidic solutions, the generated iron hydroxide or other insoluble iron compounds can be colloidal, so, the removal of impurity Fe(III) ions may

* Corresponding author at: School of Metallurgical and Ecological Engineering, University of Science and Technology Beijing, Beijing 100083, PR China.

** Corresponding author.

E-mail address: guomin@ustb.edu.cn (M. Guo).

not be as efficient, leading to difficulty in separation and unsatisfactory purification of leach liquors. Second, the ability to wash iron(III) hydroxide or other insoluble iron compounds can be poor, resulting in the loss of valuable metal ions during the separation process. Chang et al. (2010) discussed the removal of iron from acidic leach liquor by formation of goethite precipitate. They found that during the precipitation process, some nickel loss was unavoidable. Third, from the separation and purification process, the iron(III) precipitate obtained is not utilized, resulting in a waste of resources. Due to these reasons, others have tried to recover most metals from laterite leach liquors. Zhao et al. (2013) prepared Fe_2O_3 , Ni-Co oxides and MgO from the laterite leach liquor using a step-by-step precipitation method based upon the different pH value ranges for the formation of each metal hydroxide. Qi et al. (Meng et al., 2015) recovered MnO_2 , MgO and Ni-Co residues from synthetic nickel laterite leach liquors by using alkaline oxidation and hydrochloric acid leaching. In these studies, most of the metal ions in the laterite leach liquors were separated. However, it was found that the purity of the as-prepared products or the metal recovery was not high due to the complicated separation processes.

In the present study, approaches to the cooperative utilization of metal ions in the laterite leach liquor have been proposed. In our previous work (Gao et al., 2014a, 2014b), hydrothermal coprecipitation method was adopted to separate Ni, Co, Mn and Fe from Mg in a saprolite laterite leach liquor, and synthesize metal doped nickel ferrite and magnesium hydroxide powders after the solid liquid separation process. Similarly, Li et al. (2009, 2010b, 2011) developed a pathway to synthesize $\text{LiNi}_{0.8}\text{Co}_{0.1}\text{Mn}_{0.1}\text{O}_2$ and LiFePO_4 cathode materials for lithium ion batteries from laterite leach liquors by using a selective precipitation method with phosphate as the precipitant reagent. Overall, in comparison with other step-by-step processes, the treatment processes described above are simpler. Moreover, the valuable metals are utilized comprehensively and potentially to obtain functional materials. However, these treatment methods require rigorous experimental conditions such as multiple hydrothermal reactions at 200 °C for longer time periods (over 10 h), or the addition of analytical pure reagents such as lithium, nickel and cobalt, resulting in high costs. Therefore, the simplification of the treatment processes for practical applications, and the cooperative utilization of valuable metals Fe, Ni, Co, Mg, Mn etc. in the laterite leach liquors is a research topic of significant interest.

Spinel ferrites such as NiFe_2O_4 , and MgFe_2O_4 (formula of MFe_2O_4), have attracted considerable interest due to their novel magnetic and electric properties, and applications in the fields of ferrofluids, catalysts, magnetic high-density storage, etc. The world's consumption of soft magnetic ferrite is about 700,000 tons in 2015, and increasing year by year, with 70% of these materials being produced in China. Generally, spinel ferrites (MFe_2O_4) are synthesized from analytical pure reagents according to the stoichiometric ratio of 2.0. In our previous

work (Gao et al., 2014c), single phase metal doped magnesium ferrite could be synthesized from a saprolite and limonite laterite blend leach liquor using a coprecipitation method. The cooperative utilization of Ni, Co, Mn, Mg and Fe in the laterite leach liquors assisted the synthesis of Ni-Co-Mn doped magnesium ferrite in a single step. It was also found that Al and Cr could be separated from the precipitate by adjusting the pH value of the solution. Moreover, the mass ratio of saprolite to limonite laterite ore ($M_{S/L}$), which directly influence the relative amount of the metal ions in the laterite leach liquors, played a key role in determining the resultant purity of as-prepared spinel ferrite. However, the metal ion precipitation behaviors and the separation efficiencies were examined qualitatively rather than determined quantitatively, and the effect of choice of precipitant reagent used in the treatment process was not studied.

In this paper, a selective precipitation method with ammonia (aqueous NH_3) solution and sodium hydroxide (NaOH) solution as precipitant reagents enabled selective recovery of valuable metals Ni, Co or Al, Cr, respectively and the synthesis of spinel ferrites from saprolite and limonite laterite leach liquors. The effects of these precipitant reagents, pH value, precipitation method and initial metal ion concentration on the recovery and precipitation percentage of each metal ion were studied in detail. After solid-liquid separation, the precipitates were used for preparation of spinel ferrites. To synthesize single phase spinel ferrites, the effects of $M_{S/L}$ and calcination temperature on the purity of as-prepared samples were systematically examined. From this work, general flowsheets for metals recovery from laterite ores were quantitatively summarized. The approaches described might provide simple pathways for the sustainable and comprehensive utilization of laterite ore resources.

2. Experimental

2.1. Materials and reagents

The saprolite and limonite laterite ores studied in this paper were supplied by Beijing Research Institute of Mining and Metallurgy. Analytical reagents aqueous NH_3 and NaOH were purchased from Beijing Chemical Reagent Company and used without any treatment.

2.2. Experimental process

The detailed flow chart used for this study is shown in Fig. 1. The experiments were conducted step by step according to the following two approaches (i) and (ii).

The laterite leach liquors were obtained as follows: the saprolite laterite and limonite laterite with different saprolite to limonite laterite ore mass ratios ($M_{S/L}$, g g^{-1}) were blended to react with certain amount of HCl solution (concentration of HCl 2.50 mol L^{-1} ; liquid to solid

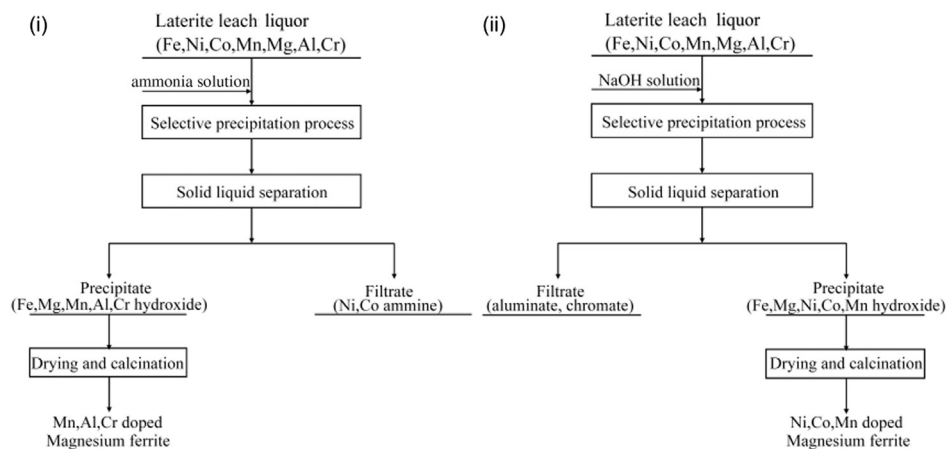


Fig. 1. Detailed flow chart for the cooperative utilization of valuable metals from the laterite leach liquors.

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