



# The extraction of valuable metals and phase transformation and formation mechanism in roasting–water leaching process of laterite with ammonium sulfate



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## ARTICLE INFO

### Article history:

Received 14 March 2016

Received in revised form

12 October 2016

Accepted 12 October 2016

Available online 13 October 2016

### Keywords:

Nickel laterite

Ammonium sulfate

Roasting

Valuable metals

Phase transformation

## ABSTRACT

Ammonium sulfate roasting–water leaching technology is a potential cleaner production process with low energy consumption, minor equipment corrosion and high selectivity contrasted with the traditional metallurgy process. Thermodynamic analysis showed that the main mineral of serpentine and magnesium iron ore in raw ore could react with ammonium sulfate, and the analysis through XRD and EDS also proved that most mineral phases were  $(\text{NH}_4)_3\text{Fe}(\text{SO}_4)_3$ ,  $(\text{NH}_4)_2\text{Mg}_2(\text{SO}_4)_3$ ,  $\text{Fe}_2\text{O}_3$ , and  $\text{SiO}_2$  as roasting products. With the decomposition of original mineral in roasting process, valuable metals of nickel, cobalt and manganese disseminated in other host minerals could be released to react with ammonium sulfate and produce metal sulfate which could be leached into the water. However, the iron mainly existed in iron oxide was water-insoluble and remained in residues. The conditions of the ammonium sulfate roasting–water leaching process, such as ammonium sulfate dosage, roasting temperature, roasting time, leaching temperature, and leaching time have been investigated here. The roasting temperature (only 400 °C) is lower than other pyrometallurgical process, which leads to less energy consumption and equipment corrosion. The leaching of Ni, Co, Mn and Fe could be 90.8%, 85.4%, 86.7% and 9.98% at the optimal condition, and the selectivity extraction can be achieved.

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## 1. Introduction

Nickel is a strategic metal and mainly used in the preparation of stainless steel and non-ferrous alloys with properties of high impact strength, strong corrosion resistance and other special properties of electrical, thermal and magnetic. Native nickel mainly comes from sulfide ores due to its low extraction cost in the past decades. The increasing global demand and fluctuating price of these target metals in commodities market and the future depletion of sulfide ores have motivated one to look for alternative strategies for laterite ores, so nickel laterites has become an increasingly important source of nickel (Tong et al., 2013; Marrero et al., 2015), which account for the majority (70%) of world's nickel reserves. However, only 42% of world's nickel production comes from laterites (Quast et al., 2013; Xu et al., 2013). The development of laterite ores has so far been hindered by the economic factor, the

energy costs for processing these ores are 2–3 times higher than that for the sulfide ores (Park and Nam, 2008; Norgate and Jahanshahi, 2011; Quaioco et al., 2014). Therefore, it is necessary to develop technically and economically viable processing techniques for laterite ore in the long term.

Processing of nickel laterite ores utilizes a variety of technologies, mainly including hydrometallurgical process and pyrometallurgical process. Sulphuric acid high pressure leaching (HPAL) process has been the preferred route to extract nickel and cobalt from laterites (mostly limonite) at 250–255 °C in a titanium-clad autoclave via the injection of steam and sulphuric acid, and the iron and aluminum hydrolyze and precipitate as hematite and a range of mixed alunite/jarosite phases, depending on the leaching conditions (Whittington and Muir, 2000). The nitric acid pressure leaching (NAPL) technology was patented and developed to treat laterite ores in China (Ma et al., 2015). The major drawbacks of HPAL process or NAPL process were higher capital and operating cost, more neutralization of acid and construction problems of HPAL or NAPL at commercial level (Büyükkacinci and Topkaya, 2009; Gao and Liu, 2013). Atmospheric leaching (AL) at lower temperature and in open vessels avoids the need for expensive HPAL autoclaves

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which was reported in literature (McDonald and Whittington, 2008a,b). At atmospheric pressure, nickel was extracted from low grade laterite by agitation leaching (Mohammadreza et al., 2014) and with vitriol (Wu, 2014), which are potential process. To know more AL process, leaching mechanisms, kinetics and rheological studies were also needed to research (MacCarthy et al., 2016). The leach liquor from AL is likely to contain significant concentrations of soluble iron and aluminum, methods must be developed to selectively reject these metals from solution or selectively extract nickel and cobalt (Willis, 2007). For the selective leaching of Ni and Co, a novel approach with the addition of ferric chloride was presented (Zhang et al., 2016). As above mentioned, AL process was improved significantly.

Conventionally, pyrometallurgical process was adapted to take this nickel intermediate giving them significant potential benefits, such as nickel ferric alloy come from reduction roasting (Forster et al., 2016; Pickles et al., 2014) and chloridizing segregation (Liu et al., 2010), nickel-iron sulphide concentrate (Harris et al., 2011), and metal salts (Fan et al., 2010), etc.. There are also potential environmental benefits with significantly lower energy consumption and lower greenhouse gases emission per tonne of nickel (Kotzé, 2002). The drawbacks of this type of process are only suitable for high grade nickel laterite ores (saprolicite) and higher capital. Therefore, hydro-pyro integration in the processing of nickel laterites could potentially be the way to process laterite with complexion of their own merits (Oxley and Barcza, 2013).

The process of ammonium sulfate roasting-water leaching is a promising and cleaner method to deal with low-grade complex ores, which is low cost, high extraction rate and high selectivity. The technology of ammonium sulfate roasting-water leaching is that the mixture of ores and  $(\text{NH}_4)_2\text{SO}_4$  is roasted in a relatively low temperature range (200–600 °C) in the tube furnace where the valuable metal oxide can be transformed into metal sulfate. And then hot water is used to leach sulfate and ammonium salts, which can make a valuable metal sulfate into the aqueous solution and then be separated from slag. Contrasted with other roasting pyrometallurgical process, such as smelting (above 1600 °C) (Kotzé, 2002), sulphurdization roasting (450–1000 °C) (Harris et al., 2011), reduction roasting (about 800 °C) (Forster et al., 2016), chlorination roasting (600–1000 °C) (Li et al., 2016), ammonium sulfate roasting-water leaching process can save a lot of energy. Meanwhile, because the impurity metals seldom react with  $(\text{NH}_4)_2\text{SO}_4$  and iron partly hydrolyze in water leaching, it can effectively reduce the impurity of metals leaching (Zeng et al., 2012; Liu et al., 2012a, 2012b).

In this study, the process of ammonium sulfate roasting-water leaching was applied to deal with the low grade nickel laterite. Different from other literature (Liu et al., 2012a, 2012b) which mainly researched the roasting conditions and dissolution kinetics, herein, the thermodynamics calculation and mineral phase analysis through XRD and EDS were researched comprehensively to investigate the mechanism of the whole process in this paper. And the conditions of  $(\text{NH}_4)_2\text{SO}_4$  dosage, roasting temperature, roasting time, leaching temperature and leaching time were also optimized.

## 2. Experimental and materials

### 2.1. Materials

The ores in this study was sourced from Yunnan province in China, where the laterite mainly consists of three layers in order of increasing depth: a hematite cap, a limonitic laterite deposit and finally saprolicite ore. The ore was milled to a size of –100 mesh using a vibratory mill after dried at 100 °C for 12 h prior to experimentation. The typical chemical analysis of these ores is

shown in Table 1. The average nickel, cobalt and iron contents were 1.15%, 0.08% and 14.06%, respectively.

The main minerals, which are evident from the X-ray diffraction pattern (Fig. 1), are serpentine ( $\text{Mg}_3[\text{Si}_2\text{O}_5(\text{OH})_4]$ ), magnesium iron ore ( $\text{Fe}_2\text{MgO}_4$ ), silica ( $\text{SiO}_2$ ) and iron oxide ( $\text{Fe}_3\text{O}_4$ ,  $\text{Fe}_2\text{O}_3$ ). In considering the chemical composition of raw ore shown in Table 1, the ore tested may be classified as a saprolicite laterite ore.

### 2.2. Methods and procedure

The ore was weighed 8 g and mixed with solid ammonium sulfate in the desired stoichiometry in each experiment. The mixture was pelletized with the diameter range of 2–3 cm. Roasting experiments were conducted in a fixed horizontal electric tube furnace where the temperature of the sample was monitored using a Chromel-Alumel thermocouple inserted into the bed. At the end of the tube, exhaust gas was absorbed in a bottle. After each roasting experiment, the roasted ore was leached with hot deionized water to transfer all soluble sulfate salt into solution. The resultant slurry was filtered through a Buchner funnel and washed with deionized water. The filtrate and filter cake were then separately collected and analyzed to determine the percentage of each metal leached. Different parameters of ammonium sulfate dosage, roasting temperature, roasting time, leaching temperature and leaching time were studied, in a way that one parameter was tested while keeping other parameters constant. The flow sheet is shown in Fig. 2.

### 2.3. Product analysis

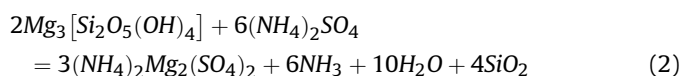
The spectrophotometry method was employed for Fe analysis with LIV-b300 UV/VIS spectrophotometer. Nickel, cobalt and manganese were analyzed with the help of atomic absorption techniques using WFX-1380 Atomic absorption spectrophotometer. A computerized X-ray diffraction equipment (D8 Advance X-ray diffraction) using CuKalpha was used to identify components of raw ore and leaching residues from 5° to 85°, and the scan speed was 0.5°/min. EDS analysis of raw ore, roasted ores and leaching residues were conducted by Hitachi SU8000 Scanning electron microscope and energy spectrum. The specific surface area was determined through BET nitrogen-helium absorption method.

## 3. Thermodynamics analysis of the roasting process

The decomposition reaction of ammonium sulfate is as follows:



XRD diagram of raw ore in Fig. 1 shows that the nickel laterite ore used in this study is main saprolicite laterite ore. So the main equations of the reaction between minerals and ammonium sulfate, and their calculation formulas of Gibbs free energy (Ye and Hu, 2002; Speight, 2004) can be expressed as follows:



$$\Delta_r H_{m,1}^\theta = -1807071 - 29.35T + 14.81 \times 10^{-3}T^2 - 151.03 \times 10^5 T^{-1}$$

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