



Synchronous extraction of nickel and copper from a mixed oxide-sulfide nickel ore in a low-temperature roasting system

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ABSTRACT

Mixed oxide-sulfide nickel ore is becoming the major source of nickel produced in the nickel smelting enterprise due to the increasing global demand of nickel and the depletion of high-grade nickel sulfide ore. Low temperature roasting technique by ammonium sulfate is a potential cleaner production process with high metal recovery, high reaction selectivity and low energy consumption. In this work, Chinese mixed oxide-sulfide nickel ores were roasted by ammonium sulfate at low temperatures of 300–500 °C to extract valuable metals directly and synchronously. Some influence factors including roasting temperature, ammonium-to-ore mole ratio, roasting time and particle size of the ore samples were studied in the roasting process. Sodium sulfate was added to improve the extraction of nickel by converting into Na-pyrosulfate. The results showed that more than 97% Cu, 97% Ni and 82% Fe are extracted, under the conditions of roasting temperature 450 °C, roasting time 4 h, ammonium-to-ore mole ratio 2.2:1, particle size –160 + 180 mesh, sodium sulfate dosage 3 g. The evolution behavior and mechanism of various minerals were elucidated using X-ray Diffraction, Thermogravimetric Analysis, Differential Thermal Analysis, Scanning Electron Microscope and Energy Dispersive Spectrometer. It is found that pyrite (FeS₂), pyrrhotite (Fe₇S₈), pentlandite ((Fe,Ni)₉S₈) and chalcopyrite (CuFeS₂) can react sufficiently with ammonium sulfate to form the corresponding metallic ammonium sulfate salts. Ammonium ferric sulfate can be decomposed to produce insoluble iron oxide by increasing the roasting temperature to higher than 500 °C to realize the separation from copper and nickel in the leaching liquid. The ammonium sulfate roasting-water leaching technique provides an effective approach for the extraction of nickel and copper from Chinese low grade mixed oxide-sulfide nickel ores.

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1. Introduction

As an important strategic metal, nickel is mainly used in the production of stainless steel and nonferrous alloys due to its high impact strength and ductility properties (Guo et al., 2011). These products are important materials for manufacturing aircraft, tanks, submarines, radars and missiles. China is one of the countries with rich nickel resources and is also the largest producer and consumer of nickel (Xu, 2008), it is estimated that more than 43% of world

nickel is consumed by China (Huang et al., 2014). Nickel sulfide ore, as the main nickel-containing resource, accounting for approximately 91% of the nickel total reserves (Watling, 2008), is mainly distributed in Gansu, Xinjiang and Yunnan provinces. Generally, nickel sulfide ore contains copper, cobalt, and numerous platinum group elements, all of which leads to the significant exploitation values of the ore (Cameron et al., 2009). Nickel sulfide ore is typically processed by flotation method to produce concentrates which are then subjected to high-temperature processing (Gericke and Govender, 2011). Because of the rapid rise at the rate of approximately 4.7% per year in stainless steel demand (Norgate and Jahanshahi, 2011), nickel sulfide ore has been rapidly exploited in recent years, accelerating the depletion of nickel sulfide ore, so that mixed oxide-sulfide nickel ore is becoming the major source of the

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nickel product in nickel smelting enterprise.

Mixed oxide-sulfide nickel ore is a thick mixed ore belt located between the primary sulfide nickel ore lean ore and upper oxidized ore belt, accounting for approximately 2/3 of total Chinese nickel reserves. Several typical characteristics of mixed oxide-sulfide nickel ore are different from those of traditional sulfide nickel ore, namely, low metal content, high alkali gangue content, complex chemical compositions and mineral structures. The content of metallic minerals including pyrite, pyrrhotite, pentlandite, violarite, chalcopyrite is lower than 10% of the total minerals, while gangue minerals, mainly containing serpentine, chlorite, talc, olivine and other silicate minerals, account for over 90%. Valuable metals of nickel and copper are dispersed in different minerals such as sulfide ore, oxide ore and silicate gangue, which causes the minerals to show different structure and properties. When mixed oxide-sulfide nickel ore is treated by the existing process for high-grade nickel sulfide ore, approximately half of the non-ferrous metals including nickel, copper and platinum group metals are lost (Kosyakov et al., 1995), resulting in low recovery of valuable metals and poor economic benefit (Senior and Thomas, 2005).

Research on the metallurgical technologies of mixed oxide-sulfide nickel ore has been somewhat rare, with only a small amount of published experimental data regarding the direct extraction of valuable metals by bioleaching. Heap bioleaching (Qin et al., 2009) and column bioleaching (Zhen et al., 2009) were used to process low-grade nickel sulfide ore containing rather high levels of olivine, chlorite and antigorite (MgO 30–35%) present in the main gangue minerals, where it was found that more than 80% of Ni and Co was extracted, and a recovery of approximately 30% Cu was achieved. Although the bioleaching of mixed oxide-sulfide nickel ore is technically feasible, the economic viability of process was restricted by high acid-consumption of high gangue minerals and low extraction rate of valuable metals, with much research effort still required to advance to industrial application of the bioleaching process. It is very necessary to develop an economically, technically and environmentally viable processing techniques suited to the long exploitation of mixed oxide-sulfide nickel ore.

The technique of low-temperature roasting with ammonium sulfate has been regarded as a promising and cleaner process for direct extraction of valuable metals from low-grade laterite ores (Li et al., 2017), exhibiting many excellent characteristics including high metal recovery, high reaction selectivity, low energy consumption and low cost. The mixture of ores and ammonium sulfate was roasted in the temperature range of 200–600 °C and it was found that metal oxide minerals could effectively react with ammonium sulfate to form soluble metal sulfates and then be separated from slag by water leaching process.

In this study, ammonium sulfate roasting technique was used to process a typical Chinese mixed oxide-sulfide nickel ore to extract copper and nickel directly in a relatively low temperature range (300–500 °C) under the condition of inletting oxygen gas. The reaction system of valuable metal sulfide mineral phases and ammonium sulfate is novel, and no relevant report is available until now. The gas of SO₃ and NH₃ generated in the roasting process can be recovered in the form of ammonium sulfate crystals for recycling to achieve cleaner production. The effects of different variables, such as the roasting temperature, roasting time, particle size of ore samples, ammonium-to-ore mass ratio and sodium sulfate addition during roasting process on the conversion of nickel and copper were evaluated. X-ray Diffraction (XRD), Thermogravimetric Analysis/Differential Thermal Analysis (TG/DTA), Scanning Electron Microscope (SEM) and Energy Dispersive Spectrometer (EDS) were applied to test roasting samples to reveal the transformation behavior and mechanism of valuable metal sulfide minerals.

2. Materials and methods

2.1. Materials

The mixed oxide-sulfide nickel ore used in this study was taken from Xinjiang province, China. The ore samples were ground and sieved into six narrow size fractions: +120, –120 + 140, –140 + 160, –160 + 180, –180 + 200 and –200 mesh. Unless otherwise stated, roasting experiments were performed with the –160 + 180 mesh. The chemical compositions of each size fraction are shown in Table 1. The ore contains 41–45% of iron, 29–32% of sulfur and 10–14% of silicon, and the main valuable metals contained in the ore are nickel and copper with their contents in the range of 2–4%.

The mineralogical phase identification of the ore samples was carried out using XRD (Rint-2000, Rigaku), and the results in Fig. 1 indicate that the major crystalline minerals in mixed oxide-sulfide nickel ore are pyrite (FeS₂), pyrrhotite (Fe₇S₈), pentlandite ((Fe,Ni)₉S₈), chalcopyrite (CuFeS₂) and quartz (SiO₂). It can be confirmed that pentlandite is the dominant nickel bearing phase, and chalcopyrite is the dominant copper bearing phase. Due to the low concentration of nickel oxide and nickel silicate in the ore body, their diffraction peaks are relatively weak and cannot be observed in the XRD spectrum.

2.2. Methods and procedure

The roasting experiments were conducted at the laboratory scale (10 g). After being dried at 80 °C for 12 h, the ore samples were mixed equally with a calculated amount of ammonium sulfate, the mixture was loaded in a ceramic crucible and heated in a vertical tube furnace controlled using a programmable temperature controller with the precision of ±1 °C. Oxygen gas was introduced from the bottom of tube, and the gas generated during the roasting process was absorbed by water in a bottle connected from the top of tube. The roasting temperature was set at the desired values ranging from 300 to 500 °C, at an interval of 50 °C. After reaching the predetermined temperature, the mixture would be maintained at this temperature for a period of time. After cooling to room temperature, the roasted samples were leached with purified water at 80 °C for 40 min under the solid/liquid ratio of 1:4 g/mL and the stirring speed of 450 r/min, where the sulfate salts formed in the roasting process were dissolved into water. Filtration was conducted to collect the liquid samples for measuring the concentration of the main metal ions in order to calculate their extractions according to Eq. (1). The iron concentration was determined by standard potassium dichromate titration, the concentration of nickel and copper was analyzed using UV–vis spectrophotometer. The solid residues were washed thoroughly several times with purified water and then dried for XRD and SEM analysis.

$$\eta_i = \frac{c_i M_i V}{m w_i} \times 100\% \quad (1)$$

Table 1

The main chemical compositions of ore samples at different size fractions (wt.%).

Particle size (mesh)	Ni	Cu	TFe	S	Al ₂ O ₃	MgO	SiO ₂
+120	2.26	2.99	41.54	29.3	3.12	2.21	13.05
–120 + 140	2.33	3.16	42.32	30.5	2.85	2.17	11.65
–140 + 160	2.38	3.23	42.47	31.6	2.80	1.98	11.38
–160 + 180	2.43	2.96	42.64	30.6	3.05	1.90	11.40
–180 + 200	2.33	2.83	43.20	29.9	2.91	1.98	11.93
–200	2.34	2.65	44.30	31.2	2.60	1.79	10.80

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