

## Leaching of nickel laterite ore assisted by microwave technique

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**Abstract:** The application of microwave technique in the hydrometallurgy of nickel laterite ores was described. The mixture of nickel laterite ores and sulfuric acid was pre-treated by microwave irradiation. The dissolving of nickel was conducted in hot water at the atmospheric pressure. The effects of factors, such as microwave power, microwave irradiation time, and sulfuric acid dosage, were investigated. In microwave field, the migration of ionic species and/or rotation of dipolar species promote the liquid–solid reaction process due to the increased contact area of reactants and leaching reaction rate constant. Thanks to the strengthening action of microwave, the microwave-assisted leaching process has its advantages, such as higher extracting rate than conventional atmospheric leaching, and no need for high-pressure operation as high pressure acid leaching (HPAL). The ferric iron in leaching solution could be effectively removed by sodium jarosite process with a little loss of nickel in the jarosite precipitate.

**Key words:** nickel laterite ore; leaching; microwave; jarosite; pre-treatment

### 1 Introduction

For a hydrometallurgical process of nickel laterites, conventionally, high pressure acid leaching (HPAL) process has been the preferred route due to precipitation of iron as hematite during leaching[1]. And many studies on the pressure leaching have been carried out [2–5]. Recently, leaching at atmospheric pressure has gained much attention due to higher capital cost of pressure acid leaching at commercial level[6–7]. From these studies, however, the acid leaching of laterites at atmospheric pressure shows its disadvantages, such as slower leaching kinetics, higher acid consumption and high residual acid concentration. Therefore, a new technology should be applied for trying to solve the problem.

Microwave heating offers a number of advantages with its unique characteristics[8–9], which include short processing time, direct, selective and volumetric heating, and a more controllable heating process. For many years, numerous studies on microwave assisted leaching have been conducted[10–12]. And detailed reviews on application of microwave energy for treatment of metal ores and microwave assisted leaching have been reported in Refs.[13–14], which show a great potential of application of microwave technique in atmospheric

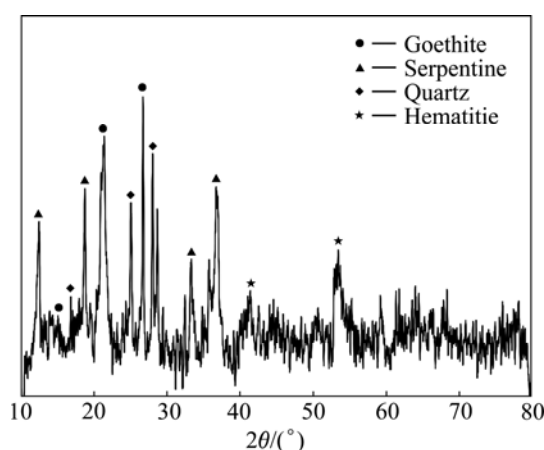
pressure leaching of laterite.

In a leaching system, however, strengthening action of microwave is weakened by water due to its good property for microwave energy absorption. To examine the strengthening action of microwave, the effect of the microwave pre-heating of the mixture of laterite and sulfuric acid on nickel extraction was studied in the present work. And the iron removal of leaching solution as the further treatment of the process was investigated.

### 2 Experimental

The raw material used in this study was nickel laterite, which was obtained from Philippine. From the X-ray diffraction analysis, the main phases presented in the laterite were goethite ( $\text{FeOOH}$ ), serpentine ( $\text{Mg}_3\text{Si}_2\text{O}_5(\text{OH})_4$ ), hematite ( $\text{Fe}_2\text{O}_3$ ) and quartz ( $\text{SiO}_2$ ) (Fig.1). Chemical analysis of the laterite shows the following elemental contents: Ni 1.54% and Fe 50.2% (mass fraction). This indicates that it is limonite laterite.

As a typical experimental procedure, the mixture of 30.00 g nickel laterite ores and sulfuric acid with predetermined mass ratio of acid to ore was pre-treated by microwave irradiation in a domestic microwave oven for pre-set time. Then, the dissolving of nickel from the



**Fig.1** X-ray diffraction pattern of laterite ore sample

product in the microwave treatment process was conducted in 300 mL water at 90 °C for 90 min. At last, the aqueous iron (mainly ferric ion) in the leaching solution was removed by precipitation as sodium jarosite. As a typical precipitation procedure, 6.00 g natrojarosite as seed crystal was added after the leaching solution was heated to pre-set temperature; the solution pH value was monitored by a digital pH meter; as iron precipitated as jarosite, the solution pH value decreased; then the  $\text{Na}_2\text{CO}_3$  was added as pH value adjustor and sodium source for natrojarosite.

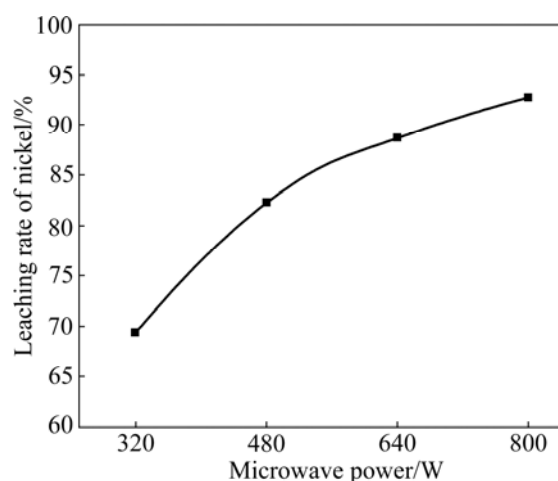
During the test processes, samples were obtained at predetermined time. The solution samples were analyzed for Ni and Fe using inductively coupled plasma (ICP). The characterization of the solid samples was done by X-ray diffractometry and scanning electron microscopy (SEM).

### 3 Results and discussion

#### 3.1 Effect of microwave power on nickel extraction

To determine the various effects of process factors, tests were performed under the typical conditions as follows. Microwave power was 800 W; microwave irradiation time was 6 min; and the mass ratio of acid to ore was 2.0. The effect of microwave power on leaching rate of nickel is shown in Fig.2. Obviously, the increasing of microwave power leads to higher leaching rate of nickel.

In microwave field, the migration of ionic species and/or rotation of dipolar species promote the liquid–solid reaction process due to the increased contact area of reactants and the leaching reaction rate constant. The leaching rate of nickel reaches 92% when the microwave power is set as 800 W. According to the results, if microwave power is continually increased, some unfavorable phenomena occur, such as slurry sputtering. Therefore, the optimum microwave power is

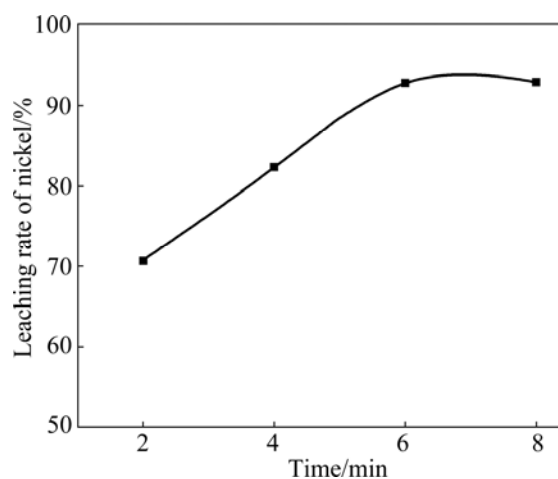


**Fig.2** Effect of microwave power on nickel extraction

set as 800 W under the applied conditions.

#### 3.2 Effect of microwave irradiation time on nickel extraction

Tests with various microwave irradiation time were conducted under the typical conditions mentioned in Section 3.1. The results of the tests are shown in Fig.3. It can be seen from Fig.3 that the reactions do not proceed completely with a limited reaction time, and the leaching rate of nickel is only about 70% after leaching for 2 min.

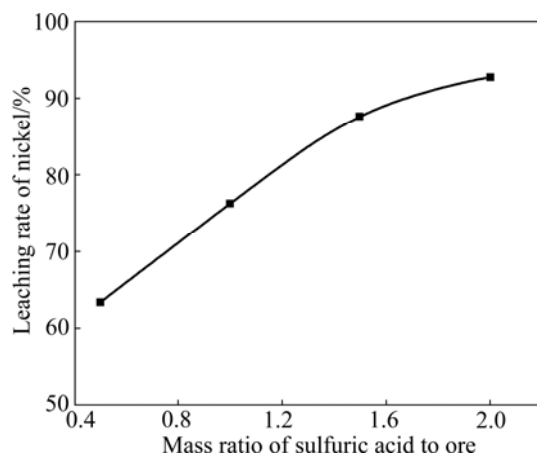


**Fig.3** Effect of microwave irradiation time on nickel extraction

With prolonging the microwave irradiation time, the leaching rate increases. The leaching rate of nickel is up to 92% after microwave heating for 6 min. The continuous increasing of microwave irradiation time has no remarkable influence on the increase of leaching rate of nickel. The leaching rate of nickel has only a little difference between microwave time set as 6 min (92.76%) and 8 min (92.97%). Therefore, the optimum microwave time is 6 min.

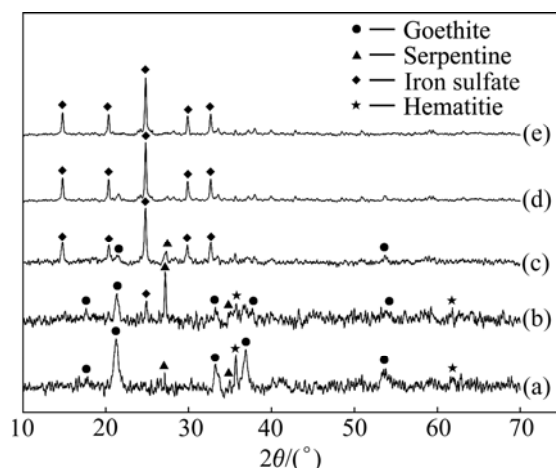
### 3.3 Effect of mass ratio of acid to ore on nickel extraction

To determine the effect of the mass ratio of acid to ore on nickel extraction, tests with various mass ratios of acid to ore were conducted under the typical conditions mentioned in Section 3.1. And the results are shown in Fig.4.



**Fig.4** Effect of mass ratio of acid to ore on leaching rate of nickel

The mass ratio of acid to ore has an approximate linear relation with leaching rate of nickel. Obviously, sufficient acid is necessary to obtain high leaching rate of nickel. The X-ray diffraction results of the products during the microwave treatment process with different mass ratios of acid to ore are shown in Fig.5. Under the conditions of the mass ratio of acid to ore of 0.5 and 1.0, goethite is still found in the residue. With the increase of acid dosage, goethite in the laterite is destroyed and dissolved as well as hematite. With the mass ratio of acid to ore increasing, the main phase in the product is  $\text{Fe}_2(\text{SO}_4)_3$  that can be easily dissolved with nickel sulfate



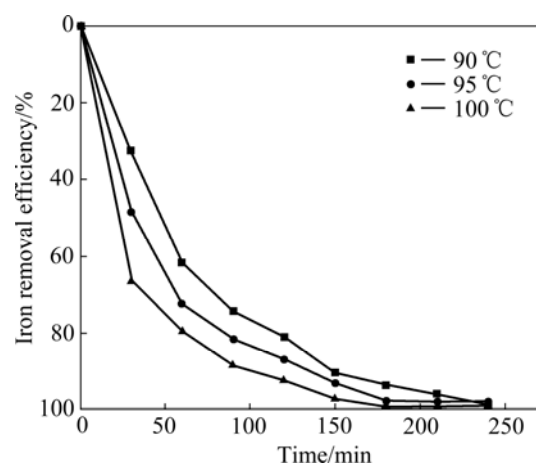
**Fig.5** X-ray diffraction patterns of products during microwave treatment process with different mass ratios of acid to ore: (a) Raw; (b) 0.5; (c) 1.0; (d) 1.5; (e) 2.0

in the following water leaching.

### 3.4 Effect of temperature on iron removal efficiency

During the microwave sintering–atmospheric leaching process of laterites, both nickel and iron were dissolved in the solution, which led to very high iron contents in the solution. In order to recover nickel effectively, leaching solution must be treated by iron removal. Aqueous iron exists in form of ferric ion ( $\text{Fe}^{3+}$ ) in the solution, so, atmospheric natrojarosite method was selected for iron removal.

At room temperature, the formation of natrojarosite is very slow, which needs 1–6 months. When the temperature reaches 100 °C, natrojarosite can be precipitated in a few hours. While the temperature exceeds 180 °C, natrojarosite begins to decompose in the acid solution. According to the effect of temperature on iron removal practice, the temperature range was selected as 90–100 °C. During the precipitation of jarosite, pH of solution value was approximately maintained as 2.0. The effect of temperature on iron removal efficiency is shown in Fig.6.



**Fig.6** Effect of temperature on iron removal efficiency

When the temperature is 90 °C, the rate of iron precipitation is very low. The removal of iron is only 61.58% after 60 min. At the time of 120 min and 180 min, it is 81% and 93.15%, respectively. The filterability of the jarosite residues is not good. The rate of iron precipitation becomes high and removal rate of iron increases continuously. While the temperature is 100 °C, the removal rate of iron reaches 79.48% after 60 min reaction. And it is 98.58% for 180 min. The precipitation reaction is mostly completed. Under the temperatures of 90, 95 and 100 °C with the pH value of 2.0, the fractions of nickel lost in the precipitates are 1.40%, 1.57% and 1.61%, respectively.

### 3.5 Effect of pH value on iron rejection

Precipitation tests at various pH values were

conducted at 100 °C. The effect of pH value on iron removal is shown in Fig.7. With the increase of acidity, both stability of natrojarosite and precipitation rate of iron decrease. When pH value is 1.5, iron removal efficiency is 78.42% and 99% with reaction time of 60 min and 180 min, respectively. When the value increases to 2.5, colloidal precipitation of ferric hydroxide is formed, so iron removal efficiency decreases. The iron removal is only 94.72% with reaction time of 240 min. While the iron removal efficiency is 96.86% with reaction time of 150 min at pH value of 1.5. The X-ray diffraction results of the samples precipitated at different pH values are shown in Fig.8.

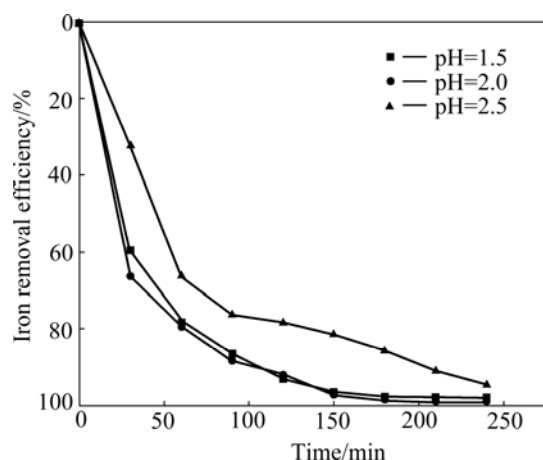


Fig.7 Effect of pH value on iron removal efficiency

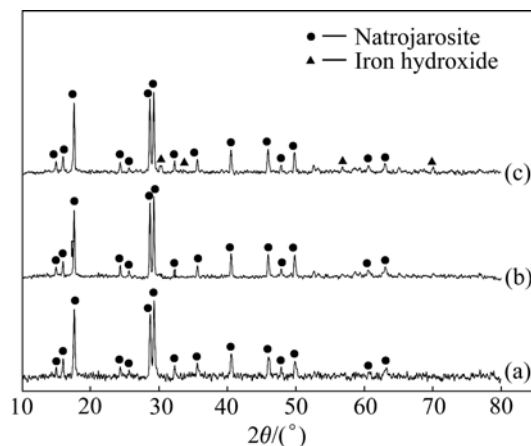


Fig.8 X-ray diffraction patterns of samples precipitated at different pH values: (a) 1.5; (b) 2.0; (c) 2.5

Fig.8 indicates that the phase of precipitation is natrojarosite at pH 1.5 and pH 2.0. The phase of precipitation is mainly natrojarosite when pH is 2.5, however, iron hydroxide ( $\text{Fe}(\text{OH})_3$ ) is found in it. Iron hydroxide colloid entrains metal ions, which leads to loss of nickel ion and filtration difficulty. When the pH values are 1.5, 2.0 and 2.5, respectively, the rates of nickel lost in the precipitates are 1.65%, 1.61% and 10.44%, respectively. Therefore, a high pH value is not

suitable for iron removal from the leaching solution. The characteristic of the precipitation was analyzed by scanning electron microscopy (SEM) (Fig.9). The petaloid jarosite precipitates on the seed particles.

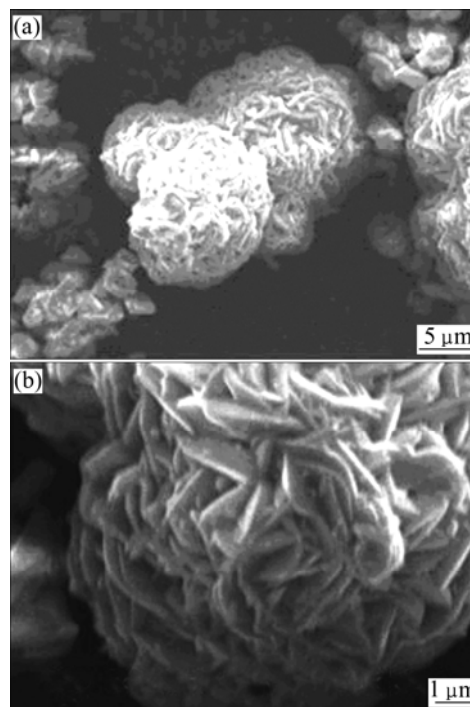


Fig.9 SEM images of jarosite precipitation sample

## 4 Conclusions

1) For the microwave pre-heating and leaching of nickel laterite ores in laboratory scale, the optimum conditions of microwave pre-heating are as follows: microwave power is 800 W; microwave heating time is 6 min; and the mass ratio of acid to ore is 2.0. 92.76% of nickel in the ores is dissolved in water at atmospheric pressure.

2) When the conditions are controlled at temperature of 100 °C and pH value is approximately 2.0, aqueous ferric iron in the leaching solution can be effectively by precipitating iron as jarosite. And the rate of aqueous nickel lost in the precipitates is less than 2.0%.

3) Under the applied conditions, although the microwave pre-treatment process does not show a selective effect for nickel extraction, it can effectively shorten the reaction time and reduce the residual acid concentration in leaching solution.

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