Effect of Microwave Heating on the Leaching of Lateritic Nickel Ore in Perchloric Acid

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Summary: In this study, the leaching conditions of Sivrihisar (Adatepe) limonite type lateritic ore in acidic medium were investigated. Leaching experiments were carried out using conventional and microwave-assisted method. The effects of stirring speed, leaching temperature, perchloric acid concentration, solid/liquid ratio and particle size on conventional leaching were determined. Microwave-assisted leaching was carried out by using the optimum results of the conventional leaching. The pre-heating process was applied on different microwave powers (0, 90, 180, 360 and 600 W) and pre-processing time (0, 1, 3, 5, 7, 10, 15 and 20 min). These experimental results demonstrated that acid leaching was a convenient method for Ni extraction from lateritic ore. The higher dissolution and the higher Ni recoveries in the microwave-assisted leaching process were obtained in less leach time.

Keywords: Nickel, Conventional leaching, Extraction, Microwave-assisted leaching.

Introduction

Laterites are formed by weathering of ultramafic rocks. Lateritic ore essentially may be classified as either limonite or serpentinite ore types. Approximately 70% of the world's nickel reserves are contained within laterite ores, with the remaining nickel associated with sulphide deposits [1]. The production of nickel from lateritic ores includes pyrometallurgical and hydrometallurgical processes [2]. Several authors studied hydrometallurgical methods of processing of lateritic nickel ores in various leaching media [2-9].

In microwave heating systems, electromagnetic energy transforms to heat in materials directly. Therefore, contrary to the conventional systems, in microwave heating the heat moves inside of the material to outside. The inner parts of the material are hotter than its surface, so evaporation of the water from inside to outside and the diffusion is simpler. During drying of the material the shell formation is not observed and the water evaporates easily. Microwave energy is usually used in preheating, baking, blanching, cooking, pasteurization, sterilization, drying and dehydration [10, 11].

Electrical and magnetic fields occur in microwaves in the form of electromagnetic energy [12]. The microwave energy is a non-ionized electromagnetic radiation with a frequency of 300 MHz to 300 GHz. Microwave causes the moving of molecules or ions by providing reflection and absorbance. For this reason, when they are absorbed by any material, they produce some heat [13]. In recent years, microwave pre-processing and enrichment of mineral processing operations for both as a method of reducing the cost and size reduction processes are used as pre-heat treatment. With the use of microwave heating process, minerals or ores should be heated selectively and rapidly compared to other heating devices [14]. Some minerals may get heat by adsorption of microwave although some minerals within the rocks refuse microwave [15, 16]. Microwave pre-heat could cause decreases in rock strength and specific fracture rates are increased [17, 18]. In recent years, the microwave treatment of minerals has become a major focus of interest and carried out some extensive studies on the microwave processes. These studies include: microwave assisted ore milling, microwave assisted carbothermic reduction of metal oxides, microwave assisted mineral leaching, microwave assisted drying and microwave assisted waste management issues [19].

One of the most important of these studies is microwave-assisted acid leaching. According to some researchers, compared to conventional leaching process, microwave-assisted leaching increases the metal extraction and reduces the leaching time. Krishnan *et al.* [20] investigated the use of microwave energy for the extraction of zinc from sulphide concentrate and compared the conventional acid leaching with microwave assisted leaching. They expressed that microwave-assisted leaching process was more efficient. Kuslu and Bayramoglu [21] studied the radiation effect to pyrite extraction in laboratory-scale work. As a result of this study, the activation energy of this leaching process was calculated as 18.72 kJ/mole and expressed that this value was nearly half of the conventional heating. In another work, microwave-assisted acidic ferric chloride leaching of copper sulfide concentrate was studied and 99% of copper extraction was obtained in 40-45 min microwave-heating. However, it was expressed that 2h of heating was needed in conventional heating to reach the same extraction level [22]. In another microwave-assisted leaching study, the experiments were carried out using lateritic nickel ore in sulphuric acid medium and it was concluded that nickel extraction increased efficiently [23, 24].

In this study, conventional and microwave-assisted leaching conditions of Sivrihisar (Adatepe) laterite ore, in perchloric acid medium were investigated.

Experimental

Approximately 100 kg of limonite type lateritic nickel ore obtained from the different places of the Adatepe (Sivrihisar) deposit were reduced to 10 kg by conic methods. This 10 kg of sample were crushed by using jaw crusher and then ground by using rod mill. At this stage, the samples were separated into 4 particle size fractions (-212+150, -150+106, -106+75 and -75+53 µm) for experiments. The results of elemental analysis for each fraction are given in Table-1 [25].

Leaching experiments were carried out using concentrated $HClO_4$ solution. Stirring speed, acid concentration, temperature, solid/liquid ratio and particle size of the ore were selected as experimental parameters. A glass vessel of 1 L was used as a leaching reactor put in a temperature-controlled water bath. Leaching reactor was closed by a rubber cover during experiment. The temperature ranged from 40 to 96°C (± 0.2°C) of the leach solution in the reactor was provided by a thermostatically controlled water bath. The leach solution was stirred by Heidolph RZR 2021 model mechanical stirrer with teflon lined impeller. Microwave-assisted leaching was carried out by using the optimum results of the conventional leaching. The pre-heating process was applied on different microwave powers (0, 90, 180, 360 and 600 W) and pre-processing time (1, 3, 5, 7, 10, 15 and 20 min). For the microwave processing of the ore sample, kitchen-type microwave oven (at 2.45 GHz frequency) adjusted to different powers were used. The flow chart for the experiments is given in Fig. 1.

Table-1: Results of the elemental analysis of the lateritic nickel ore obtained from Adatepe-Sivrihisar.

Particle size fractions [µm]	Elements [W%]			
	Ni	Fe	Co	Mn
-212 + 150	1.43	39.93	0.043	0.064
-150 + 106	1.37	22.70	0.039	0.045
-106 + 75	1.29	25.83	0.033	0.036
-75 + 53	1.30	18.67	0.027	0.024

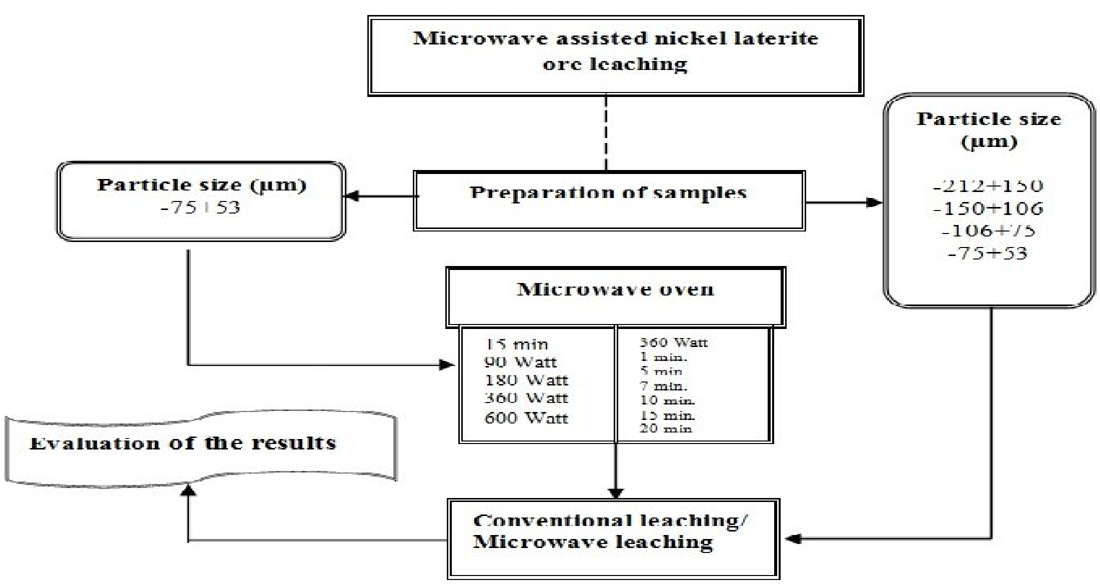

Fig. 1: The flow chart of experimental studies.

Results and Discussion

Conventional leaching of lateritic nickel ore in perchloric acid

Effect of stirring speed

The effect of stirring speed on the nickel dissolution percentage from the lateritic nickel ore were investigated in 0.5 M $HClO_4$ solution at 50°C in the range of 0 to 400 rpm. The results presented in Fig. 2 show that the dissolution percentage of nickel is independent of the stirring speed.

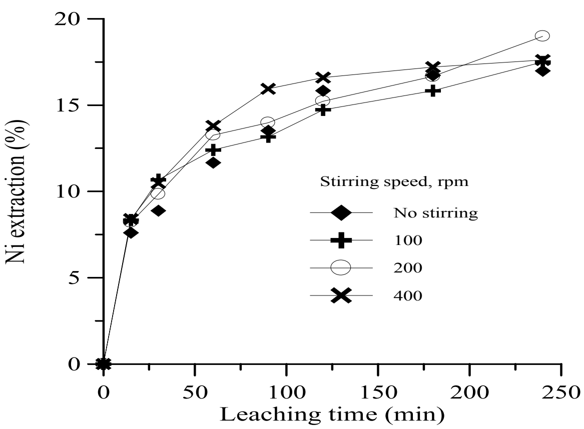

Fig. 2: The effect of stirring speed on the nickel extraction.

Effect of acid concentration

The effect of $HClO_4$ concentration (0.5-2.0 M) on the Ni extraction using -106+75 µm size fraction of 10 g/dm^3 the ore was investigated at 50°C. The dissolution recovery of the ore as a function of leaching time for the different acid concentrations is presented in Fig. 3. As shown in Fig. 3, the nickel extraction of lateritic nickel ore increased with increasing perchloric acid concentration. The nickel dissolution after 240 min leaching time in 0.5-2.0 M perchloric acid solutions was found to be 16.98% and 29.08 %, respectively.

Effect of temperature

The leaching process was carried out in the temperature ranging from 50 to 90 °C with 2.0 M perchloric acid solution using -106+75 µm size fraction of the ore. The effect of temperature on nickel extraction from the lateritic nickel ore are presented in Fig. 4. It can be seen from the Fig. 4 that the extraction of Ni is increased with increased temperature. While, the extraction of Ni was 29.08 % at 50°C after 240 min and increased to 50 % at about 80°C. To determine the other leaching parameters, 80 °C was chosen as the optimum leaching temperature. The selection of 80°C is also advantageous since it is lower than the boiling point of solution. By this way, the leaching process will be economic to prevent the evaporation.

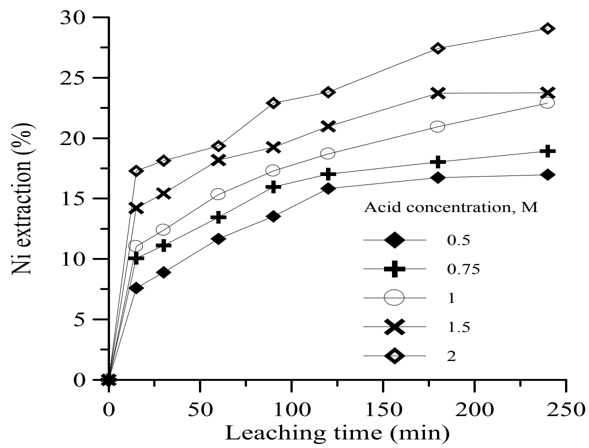

Fig. 3: The effect of $HClO_4$ concentration on the nickel extraction.

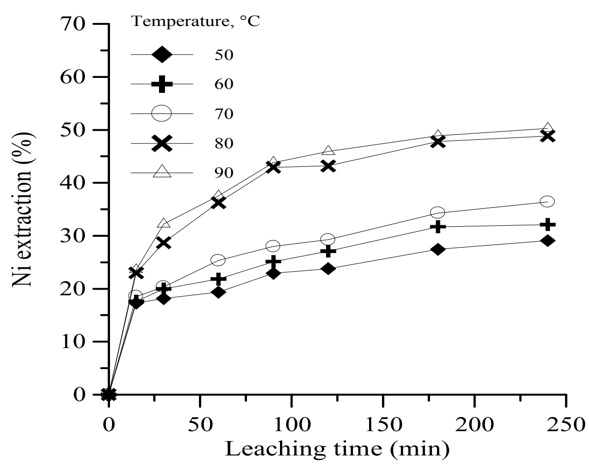

Fig. 4: The effect of temperature on nickel extraction.

Effect of solid/liquid ratio

The effect of solid/liquid ratio on the dissolution process was studied using different ratios in the range of 5-20/500 g/mL. The obtained results were given in Fig. 5. It was found that high dissolution occurred at low solid/liquid ratios. The best solid/liquid ratio to dissolve nickel from lateritic ore was at 5/500 g/mL.

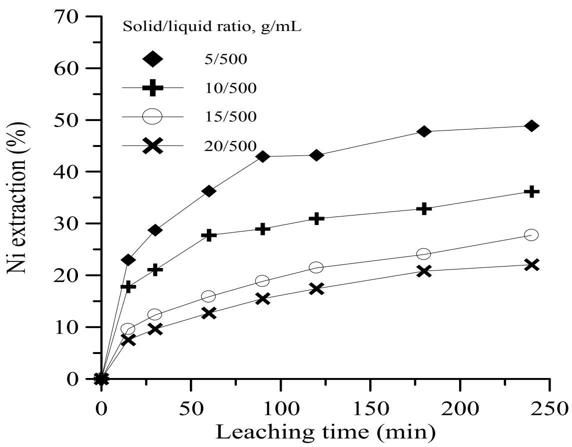

Fig. 5: The effect of solid/liquid ratio on nickel extraction.

Effect of particle size

The experiments were carried out with the four different particle size fractions (-212+150, -150+106, -106+75 and -75+53 µm) in 2.0 M $HClO_4$ solution at 80°C. The results are presented in Fig. 6. Generally speaking the nickel extraction was increased with decreasing of particle size.

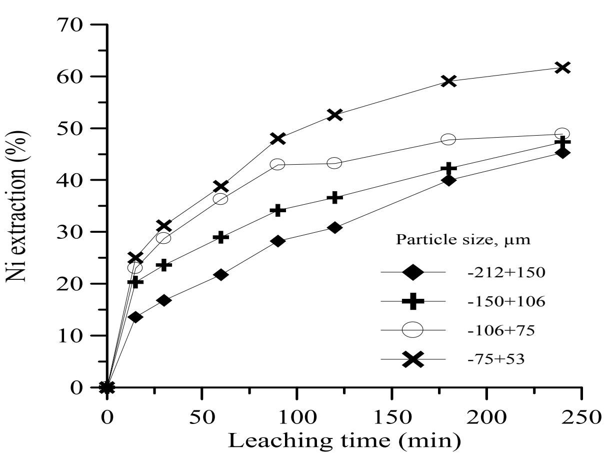

Fig. 6: The effect of particle size on nickel extraction**.**

Microwave leaching of lateritic nickel ore in perchloric acid

Microwave-assisted leaching experiments were carried out using the optimum results of the conventional leaching. The pre-heating process consists of different microwave powers (0, 90, 180, 360 and 600 W) and pre-heating time (0, 1, 3, 5, 7, 10, 15 and 20 min).

Effect of microwave power on nickel extraction

In these experiments, the sample in the fraction of -75+53 µm was pre-heated during 15 minutes at different microwave powers (0, 90, 180, 360 and 600 W). All of the microwave-assisted leaching experiments were carried out using pre-heated samples at determined optimum conventional leaching conditions (2 M $HClO_4$, leaching time 240 min, temperature 80°C, solid/liquid ratio 5/500 g/cm^3, no stirring). The experimental results were shown in Fig. 7.

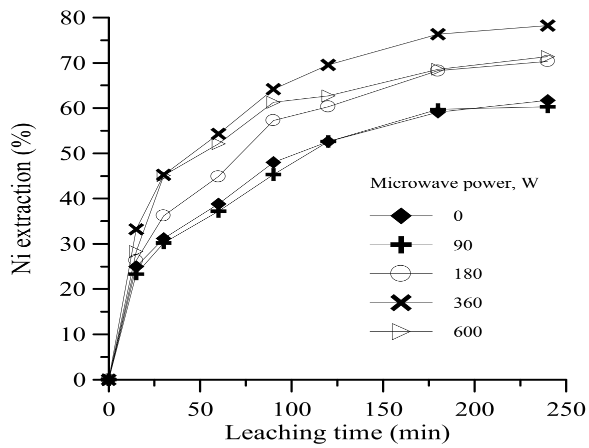

Fig. 7: The effect of microwave power on nickel extraction.

As shown in Fig. 7, in conventional leaching 62% Ni extraction was obtained in 240 min leaching time. Also no difference was observed using 90 W microwave power compared to conventional leaching. As a result of the microwave-assisted leaching experiment, Ni extraction was increased with increasing microwave power until 360 W. The optimum Ni extraction was obtained using 360 W microwave powers in 240 min leaching time.

Effect of microwave pre-heating time on nickel extraction

In these experiments, the sample in the fraction of -75+53 µm was pre-heated at different times (0, 1, 3, 5, 7, 10, 15 and 20 min) using 360 W microwave power. All of the microwave-assisted leaching experiments were carried out using pre-heated samples at determined optimum conventional leaching conditions (2 M $HClO_4$, leaching time 240 min, temperature 80°C, solid/liquid ratio 5/500 g/mL, no stirring). The experimental results were shown in Fig. 8.

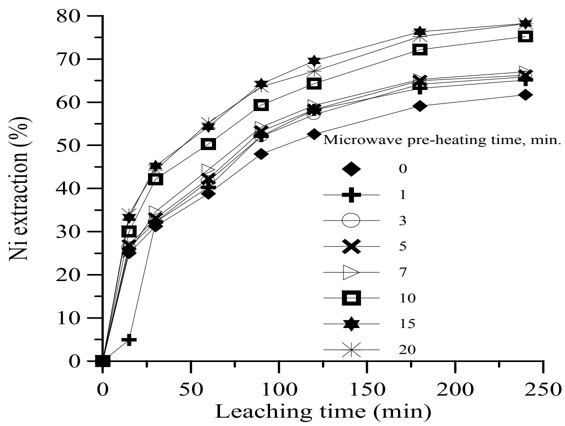

Fig. 8: The effect of microwave pre-heating time on nickel extraction.

As a result of the microwave-assisted leaching experiment, Ni extraction was increased with increasing pre-heating time. The maximum extraction (80% Ni) was obtained at 15 min pre-heating time in 240 min leaching.

As a result of the stirring speed experiments, it was shown that nickel extraction was nearly independent from stirring. Although a kinetic investigation wasn't performed, the negligible effect of stirring speed on laterite leaching could be stem from dissolution mechanism. Georgiou and Papangelakis [26] expressed that stirring speed had a negligible effect on the rate of nickel dissolution from limonitic laterite. In addition, dissolution process was fitted to ash diffusion control in shrinking core model. In this study, acid solution was made using $HClO_4$ which was a strong acid and its use in the leaching studies are uncommon according to literature examination. Due to non oxidative property of $HClO_4$ solution, in the studied leaching conditions of laterite, we didn't take advantage of $HClO_4$ as an oxidizing reagent. In the effect of particle size on nickel extraction experiments, the reason of the higher dissolution percentage of the finer size fraction is due to the higher specific surface area of the ground sample. The higher the specific surface area of the ore is the higher the Ni extraction. Microwave heating has a lot of advantages such as short processing time, direct, selective and volumetric heating, and a more controllable heating process [24]. In this study, it was expressed that microwave pre-heating time and microwave power have favorable effect on nickel extraction from lateritic nickel ore.

Since the laterite was limonitic type the main iron mineral was goethite (FeOOH). Nickel is a main substituent in the goethite lattice. It is also in the magnesium silicate particles. The probable reactions in the leaching process could be suggested as follows;

$$FeOOH + 3H^{+} = Fe^{3+} + 2H_2O \quad (1)$$

$$NiO + 2H^{+} = Ni^{2+} + H_2O \quad (2)$$

$$(Mg, Ni)\,Si_3O_8(OH)_3 + 7H^{+} + H_2O = Ni^{2+} + Mg^{2+} + 3H_4SiO_4 \quad (3)$$

Conclusion

In this study, microwave-assisted leaching conditions for lateritic nickel ore were determined. In the first stage of the study, experimental parameters such as stirring speed, leaching temperature, perchloric acid concentration, solid /liquid ratio and particle size were studied on the conventional Ni leaching. In the second stage of the study, microwave-assisted leaching experiments were carried out at determined optimum conventional leaching conditions. As a result of the experimental study, it was determined that stirring speed had no effect on Ni extraction. While Ni extraction increased with increasing $HClO_4$ concentration and temperature, decreased with increasing solid/liquid ratio and particle size. According to microwave-assisted leaching it was observed that Ni extraction increased with increasing microwave power and pre-heating time. Consequently, the maximum extraction (80% Ni) was obtained.

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