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Nickel extraction from low grade laterite by agitation leaching at atmospheric pressure

Fatahi Mohammadreza *, Noaparast Mohammad, Shafaei Seyyed Ziaeddin

School of Mining Engineering, College of Engineering, University of Tehran, Tehran 14395-515, Iran

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ABSTRACT

This research work deals with the extraction of nickel from a low grade nickel laterite ore, taken from a deposit located in southwestern of Iran, through agitation leaching at atmospheric pressure. The assaying and mineralogical studies carried out on the nickel laterite sample, showed the 0.88% Ni, and principally consisted of oxide and silicate crystalline phases i.e. dolomite, quartz, magnetite, and goethite. Among numerous factors affecting such process, four major parameters i.e. temperature, agitator speed (r/min), leaching agents and their concentration were considered in a two-level full factorial experimental design. The agitation leach tests showed that the ore could be leached at atmospheric pressure with sulfuric acid while citric acid was almost unpromising. Analysis of variance (ANOVA) using DX7 software was employed to identify effective parameters. Sulfuric acid concentration and temperature were the most effective parameters on Ni extraction. Furthermore, the factorial models for experiment responses were developed. The results showed 83% Ni extraction after 4 h leaching, under optimized conditions i.e. temperature at 95 °C, acid concentration at 5 N and agitator speed at 1000 r/min. This study revealed that factorial experimental design can be implemented to identify effective parameters on the agitation leaching process of nickel laterite.

1. Introduction

Owing to its corrosion resistance, nickel is an important metal in stainless steel and super alloys production industries. Nickel occurs in nature as sulfides and oxides [1]. Nickel sulfides, from which pentlandite $(Ni,Fe)_9S_8$ is the main mineral, comprise only about 27% world nickel reserves and has covered 60% of global production up to now. With continuous depletion of these generally high grade deposits and increasing demand, oxides, in form of nickel laterites, has become more attractive and it is estimated that more than half of the future nickel demand will be supplied by them [2].

Sulfide ores are amenable to conventional processing routes like magnetic separation and flotation to produce a concentrate for further treatment by pyrometallurgical processes resulting in final products such as ferronickel and matte. Despite sulfides, nickel in laterites is not present as an independent mineral and substitutes in iron bearing minerals mainly Goethite [FeO(OH)] [3]. Due to this fact, common beneficiation methods are almost inefficient and inherently pricey and energy consuming hydrometallurgical routes like high pressure acid leaching (HPAL) are in use.

Leaching at atmospheric pressure (AL) has been recently receiving more attention due to higher capital cost and materials of construction problems of high pressure acid leaching at commercial level [4]. The typical AL leaching process involves direct leaching of nickel laterite ores with both inorganic and/or organic acids by either agitation or heap leaching. In this process metals within the ore are liberated in the acidic environment, then the metal rich leach solution selectively precipitated for metal recovery by various methods including hydroxide precipitation, sulfide precipitation or hybrid methods [5]. Earlier studies indicated that nickel extraction from lateritic ores was strongly dependent on types of nickel bearing mineral, leaching temperature and sulfuric acid concentration [6]. Further studies were carried out to examine the atmospheric leaching (AL) of nickel laterite ores with sulfuric acid and chloride and bio-technologies [7–8]. Luo et al. investigated saprolitic laterite leaching using acid sulfuric at atmospheric pressure to determine process kinetics [9]. Li et al. used an acidic thiosulfate solution for leaching of limonitic laterite ore [10]. Girgin et al. examined dissolution behavior of a Turkish lateritic nickel ore [11]. Büyükakinci et al. determined the optimum conditions for agitation leaching by studying various parameters on

* Corresponding author. Tel.: +98 917 3200944.
E-mail address: mohammadreza.fatahi@yahoo.com (F. Mohammadreza).

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Nontronitic and limonitic type laterites [12]. Moreover, some new approaches for biohydrometallurgical processing of laterites have been introduced recently [13–14]. Senanayake et al. carried out comprehensive researches on reductive dissolution of nickel laterites [15]. The main nickel and iron oxide reaction with sulfuric acid can be described by reactions (1) and (2) [16]:

$$NiO + 3H_2SO_4 \rightarrow Fe_2SO_4 + 4H_2O \quad (1)$$

$$2Fe + 3O(OH) + 3H_2SO_4 \rightarrow NiSO_4 + H_2O \quad (2)$$

This study aims to investigate Ni extraction through agitation leaching of a low grade laterite ore at atmospheric pressure. Two-level full factorial experimental design was implemented to recognize the influence of some parameters affecting acidic leaching process. The parameters selected in this investigation were temperature (°C), acid type, acid concentration (N) and agitator speed (r/min).

2. Materials and methods

2.1. Sample characterization

Low grade limonitic laterites sample containing 0.88% Ni, 0.06% Co, and 29.89% Fe from Chahegheyb nickel deposit, located in Fars province, southwestern of Iran, was supplied during the experiments. After primary and secondary crushing by jaw and roll crushers respectively, representative samples were ground in a rod mill and passed a 100 microns sieve. Mineralogical studies of ore samples were carried out with a combination of X-ray fluorescence (XRF), Semi quantitative X-ray diffraction (SQXRD) and microscopic study of both thin and polish sections. To prepare the leaching experiments feed, samples were ground for 10 min, using 10 min, and sieved to a mean particle size of 100 microns. The results of XRD and chemical composition analysis of the ore sample are presented in Tables 1 and 2 respectively. Fig. 1 depicts the XRD pattern of the Ni bearing sample which mainly consists of the major oxide and silicate crystalline phases i.e. dolomite, quartz, magnetite, and goethite.

2.2. Design of experiments

2.2.1. Full factorial design

Factorial designs are widely used to investigate the effects of experimental factors and their interactions, that is, how the effect of one factor varies with the level of the other factors in a response. The advantages of factorial experiments include the relatively low cost, a reduced number of experiments, and increased possibilities to evaluate interactions among the variables. The most popular first-order design is the two-level full (or fractional) factorial, in which each factor is experimentally studied at only two levels that are expressed in coded form: −1 for low level and +1 for high level. The full factorial experimental design consists of a 2*k* experiment (*k* factors, each experiment at two levels), which is very useful for either preliminary studies or in initial optimization steps, while fractional designs are almost mandatory when the problem involves a large number of factors [17].

In several investigations, factorial design has been used to determine important operating parameters. Kar et al. used design of experiments to study the extraction of nickel from lateritic ore by sulphatization using sulphuric acid [18]. Massacci et al. used this method for leaching of zinc sulphide, Ubaldini et al. applied a complete factorial design for column leaching of quartz sands to remove iron, Vegliò et al. practiced a two-level fractional factorial design to study the effect of some process factors on reductive acid leaching of a manganese dioxide ore [19–21]. These investigations have confirmed that factorial design can be an economical and effective method to deal with various processes.

Table 1
Mineralogical composition of the sample using XRD method.

Minerals	Formula	Percent (%)
Dolomite	$MgCa(CO_3)_2$	26.5
Quartz	SiO_2	21.1
Magnetite	Fe_3O_4	15.2
Goethite	FeO(OH)	9.3
Montmorillonite	$Na_{0.3}(Al\ Mg)_2Si_4O_{10}(OH)_2$	8.3
Hematite	Fe_2O_3	7.1
Sanidine	$K(Si_3Al)O_8$	6.3
Biotite	$Na\text{-}Mg\text{-}Al\text{-}Si_4O_{11}$	6.2
Total		100.0

Table 2
Chemical composition of laterite material (% by weight).

Constituent	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	L.O.I
Content	23.07	3.43	55.19	4.62	4.13	9.56

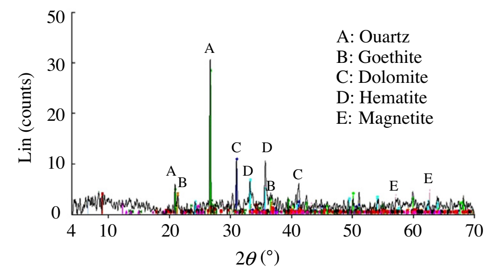

Fig. 1. XRD pattern of the nickel laterite sample.

2.3. Experimental design

The experiments were designed with software DX7. Two-level full factorial design with 3 replicates for each center point was applied in experimental design. Effect of 4 factors consisting: (i) type of leaching agent (acid), (ii) temperature, (iii) acid dosage and (iv) agitator speed were considered in this study and consequently 22 experiments were performed under following ranges:

- Leaching agent: sulfuric acid and citric acid
- Acid dosage: 3–5 N.
- Temperature: 30–8 °C.
- Agitator speed: 500–1000 r/min.

22 sets of tests with appropriate combination of sulfuric acid (X_1), citric acid (X_2), temperature (X_3) and agitator speed (X_4) were designed. The factors and coded/actual values are given in Table 3.

2.4. Apparatus and experimental procedure and sampling

The leaching experiments were conducted in a 1000 mL 3-neck flask equipped reactor. A magnetically stirred hot plate (Multi

Table 3
Coded and actual levels of independent variables used in factorial design.

Variable	Symbol	Coded variable level		
		Low	Center	High
		−1	0	1
Sulfuric acid (N)	X_1	3	4	5
Citric acid (N)	X_2	3	4	5
Temperature (°C)	X_3	30	55	80
Agitator speed (r/min)	X_4	500	750	1000