

## Introduction of the extraction of nickel from lateritic ore by sulphatization using sulphuric acid

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### ABSTRACT

Experimental methods were designed to optimize the sulphatization of a mechanical mixture of chemically pure nickel and iron oxide and a lateritic ore using sulphuric acid. The significance of various parameters and their selectivity for nickel extraction over iron have been established. The interaction among the operating parameters has been introduced using statistically designed experimental methods and the data, thus obtained for the pure system, was correlated with that for the lateritic ore. Regression equations were formulated for both systems and the extraction was represented as a function of response variables. The accuracy of the equation has been verified by Fisher's adequacy test. It was observed that the coefficient of temperature was the most dominant, followed by acid concentration. Interestingly, the time factor induces a small effect on extraction. The process has been further optimized using the steepest ascent method.

### INTRODUCTION

In India, low grade lateritic ore is the only source to meet the demand for nickel metal. The total Indian nickel ore reserves is estimated to be 138 million tonnes, out of which 121 million tonnes are located in the Sukinda region of Orissa. The mineralogical characterization of the ore indicates that the valuable metals are present in their oxidic form and thus, conversion of these oxides to water-soluble sulphate forms the basis for some metallurgical processes.[1,2]

The conversion of nickel oxide to its water-soluble sulphate is carried out in a number of ways. One of the processes is high pressure sulphuric acid leaching, which is the basis for the Moa-Bay process.[3-5]

This process is applicable only for low magnesia ores in order to minimize acid consumption. The modified process known as the AMAX process[6-8] is applicable to a wide range of oxidic nickel ores.

There are other processes, such as the pug-roast leach process [9], and sulphation roasting using sulphur dioxide-air mixtures [10], to convert nickel oxide to nickel sulphate. Depending on the extent of iron dissolution, the sulphation process could be selective or non-selective. In a selective process, the consumption of sulphuric acid or sulphur dioxide was less [11], with high nickel extraction and low iron dissolution. In another study [12,13], nickel oxide was preferentially sulphated by utilising the higher thermal stability of nickel sulphate in comparison to iron sulphate by addition of

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less than the stoichiometric amount of sulphuric acid.

In the present investigation, attempts have been made to study the preferential sulphatization of nickel over iron by roasting the pug. In this connection, experimental methods were designed statistically to optimize the process parameters. The statistical approach allows the development of a reliable quantitative approach to express extraction as a response variable, on the basis of a number of experimental methods.

## METHODS

The natural material used in the study was a - 106  $\mu\text{m}$  limonitic lateritic ore with low magnesia; the chemical analysis is shown in Table 1. A mechanical mixture was also prepared by pulverizing analar grade nickel and iron oxide in acetone medium manually for 2 h. The chemical composition of the mechanical mixture was similar to that of the lateritic ore (NiO - 1.67% and FeO - 66.5%).

Table 1

Assay composition of lateritic ore: size fraction — 106  $\mu\text{m}$

Constituents	Percentage
Fe <sub>2</sub> O <sub>3</sub>	66.5
NiO	1.67
CoO	0.089
SiO <sub>2</sub>	5.60
MgO	1.51
CaO	4.65
Al <sub>2</sub> O <sub>3</sub>	4.85
Moisture	1.50
L.O.I	12.1
Acid insolubles	1.50

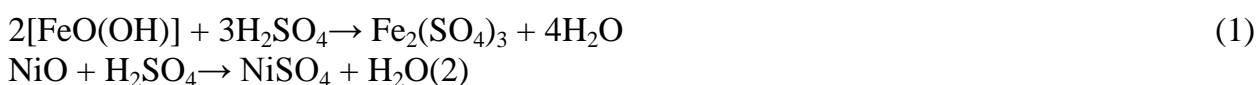
Ten grams of the ore - 106  $\mu\text{m}$ . and a calculated amount of sulphuric acid were mixed thoroughly in a silica crucible. The crucible with the charge mix was placed in a stainless steel reactor. The reactor was inserted in a muffle furnace and the material was heated at 450°C for 30 min [14]; the lumpy mass, thus formed, was ground to a fine size. The ground material was roasted at elevated temperature for a desired time. The sulphated mass was cooled to room temperature and leached with water at  $70 \pm 2^\circ\text{C}$ . All the above experimental methods were carried out in duplicate to establish the accuracy of the procedure. Eight such trial runs with variable parameters, i.e. temperature, acid concentration, and time, and three base level experimental methods were conducted to establish the consistency of the roasting data. A mass balance was performed for each experiment, based on total nickel and iron present in the feed, the leached solution and the residue. Based on the mass balance, the percentage extraction was calculated. The nickel in the solution was analysed by atomic absorption spectrophotometry (AAS). and iron

by standard volumetric methods.

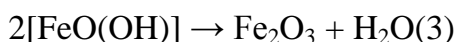
## RESULTS AND DISCUSSION

The process of sulphatization involves pugging of the ore with a sub-stoichiometric amount of sulphuric acid at room temperature and roasting the pug at higher temperature to extract nickel selectively over iron. During pugging with  $H_2SO_4$ , both iron and nickel were partially converted to the sulphate form. The pug was then subjected to roasting at a higher temperature where iron sulphate decomposed to iron oxide, releasing  $SO_3$  gas into the roasting atmosphere. The  $SO_3$  gas subsequently reacted with nickel oxide to form nickel sulphate. The basic chemical reactions involved were as follows. [15]

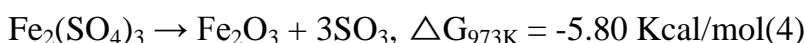
Reactions occurring during pugging:



The unreacted goethite is converted to hematite during the pre-roasting stage.



Reactions occurring during roasting :



In order to obtain the optimum condition for the sulphatization process, a full factorial design of the type  $n^k$  has been used, where  $n$  = no. of levels and  $k$  = no. of factors under verification (here  $n = 2$  and  $k = 3$ ). Thus, the total number of trial experimental method needed for a investigation is  $2^3$ . If  $Y$  is the response variable, then the regression equation with three parameters and their interaction with each other is given by:

$$Y = b_0 + b_1x_1 + b_2x_2 + b_3x_3 + b_{12}x_1x_2 + b_{23}x_2x_3 + b_{31}x_3x_1 + b_{123}x_1x_2x_3 \quad (6)$$

where  $b_0, b_1, b_2, b_3$  are the regression interaction coefficients of the concerned variables, and  $x_1, x_2, x_3$  are the variables effecting the process. [15]

## CONCLUSION

Statistical design of experimental method for the extraction of nickel is an efficient technique to quantify the effect of variable parameters. Temperature is the most significant parameter affecting nickel extraction, followed by acid concentration. The ore required a higher roasting temperature and more acid to dissolve nickel effectively than the mechanical mixture of nickel and iron oxides. It was observed that around 75% nickel

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could be extracted selectively over iron, from the laterite ore, at a temperature of 650°C, with 30 wt.% sulphuric acid and 15-min roasting time. The nickel extraction was improved to 93% by optimising the process variables, using the steepest ascent method, at 710°C temperature, 38 wt.% sulphuric acid and 13-min roasting time. [15]

#### ACKNOWLEDGEMENT

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