Introduction of the extraction ofnickel from lateritic ore by sulphatization usingsulphuric acid

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ABSTRACT

Experimental method were designed to optimize the sulphatization of a mechanical mixture of chemicallypure nickel and iron oxide and a lateritic ore using sulphuric acid. The significance ofvarious parameters and their selectivity for nickel extraction over iron have been established. Theinteraction among the operating parameters has been introduced using statistically designed experimental methodand 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 afunction of response variables. The accuracy of the equation has been verified by Fisher'sadequacy 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 optimised using the steepest ascent method.

INTRODUCTION

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

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

This process is applicable only for lowmagnesia ores in order to minimize acid consumption. The modified process known asthe 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 sulphationroasting using sulphur dioxide–air mixtures [10], to convert nickel oxide to nickelsulphate. Depending on the extent of iron dissolution, the sulphation process could beselective or non-selective. In a selective process, the consumption of sulphuric acid orsulphur dioxide was less [11], with high nickel extraction and low iron dissolution. Inanother study [12,13], nickel oxide was preferentially sulphated by utilising the higherthermal stability of nickel sulphate in comparison to iron sulphate by addition of

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lessthan the stoichiometric amount of sulphuric acid.

In the present investigation, attempts have been made to study the preferentialsulphatization of nickel over iron by roasting the pug. In this connection, experimental methodwere designed statistically to optimize the process parameters. The statistical approachallows the development of a reliable quantitative approach to express extraction as aresponse variable, on the basis of a number of experimental method.

METHODS

The natural material used in the study was a - 106 ㎛ limonitic lateritic ore with low magnesia; the chemical analysis is shown in Table 1. A mechanical mixture wasalso prepared by pulverizing analaR grade nickel and iron oxide in acetone mediummanually for 2 h. The chemical composition of the mechanical mixture was similar tothat of the lateritic ore(NiO - 1.67% and Fe O - 66.5%).

Table 1

Assay composition of lateritic ore: size fraction — 106 mm

Constituents	Percentage
Fe_2O_3	66.5
NiO	1.67
CoO	0.089
SiO_2	5.60
MgO	1.51
CaO	4.65
Al_2O_3	4.85
Moisture	1.50
L.O.I	12.1
Acid insolubles	1.50

Ten grams of the ore - 106 ㎛. and a calculated amount of sulphuric acid weremixed thoroughly in a silica crucible. The crucible with the charge mix was placed in astainless steel reactor. The reactor was inserted in a muffle furnace and the material washeated at 4508C for 30 min [14]; the lumpy mass, thus formed, was ground to a finersize. The ground material was roasted at elevated temperature for a desired time. Thesulphated mass was cooled to room temperature and leached with water at 70±2℃. Allthe above experimental method were carried out in duplicate to establish the accuracy of theprocedure. Eight such trial runs with variable parameters, i.e. temperature, acid concentration,and time, and three base level experimental method were conducted to establish theconsistency 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 thesolution was analysed by atomic absorption spectrophotometry (AAS). and iron bystandard volumetric methods.

RESULTS AND DISCUSSION

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

Reactions occurring during pugging:

$$2[FeO(OH)] + 3H_2SO_4 \rightarrow Fe_2(SO_4)_3 + 4H_2O \quad (1)$$
$$NiO + H_2SO_4 \rightarrow NiSO_4 + H_2O \quad (2)$$

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

$$2[FeO(OH)] \rightarrow Fe_2O_3 + H_2O \quad (3)$$

Reactions occurring during roasting :

$$Fe_2(SO_4)_3 \rightarrow Fe_2O_3 + 3SO_3,\ \triangle G_{973K} = -5.80 \text{ Kcal/mol} \quad (4)$$
$$NiO + SO_3 \rightarrow NiSO_4,\ \triangle G_{973K} = -13.3 \text{ Kcal/mol} \quad (5)$$

In order to obtain the optimum condition for the sulphatization process, a full factorial design of the type n^khas 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 theregression 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 , and b_3 are the regression interaction coefficients of the concernedvariables, and x_1 , x_2 , and x_3 are the variables effecting the process.[15]

CONCLUSION

Statistical design of experimental method for the extraction of nickel is an efficient techniqueto quantify the effect of variable parameters.Temperature is the most significant parameter affecting nickel extraction, followed byacid concentration.The ore required a higher roasting temperature and more acid to dissolve nickeleffectively than the mechanical mixture of nickel and iron oxides.It was observed that around 75% nickel could be extracted selectively over iron, fromthe laterite ore, at a temperature of 6508C, with 30 wt.% sulphuric acid and 15-minroasting time.The nickel extraction was improved to 93% by optimising the process variables,using the steepest ascent method, at 7108C temperature, 38 wt.% sulphuric acid and13-min roasting time.[15]

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