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Bacterial leaching of nickel laterites using chemolithotrophic microorganisms: Process optimisation using response surface methodology and central composite rotatable design

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ARTICLE INFO

Article history: Received 8 September 2008 Received in revised form 12 February 2009 Accepted 17 May 2009 Available online 22 May 2009

Keywords:
Nickel laterites
Chemolithotrophic microorganisms
Optimisation
RSM
CCRD
ANOVA

ABSTRACT

The depletion of easily processed nickel sulphides and demand for the nickel metal pose a challenge of finding new effective methods for nickel recovery from low grade ores. Solving these problems successfully requires optimisation of the processes. In this study, a statistically-based optimisation strategy has been used in the optimisation of pH, pulp density and particle sizes during the bacterial leaching of nickel laterites using a mixed culture of chemolithotrophic bacteria (Acidithiobacillus ferrooxidans, Acidithiobacillus caldus and Leptospirillum ferroxidans). The central composite rotatable design (CCRD) was used to collect the data for fitting the second order response. A mathematical model equation was then derived by computer simulation programming applying least squares method using MATLAB R2006a. This second order model representing the nickel recovery from nickel laterite ore is expressed as a function of the three variables (pH, particle size, and pulp density). A statistical analysis (ANOVA) was carried out to study the effects of the individual variables as well as their combined interactive effects on the recovery of nickel. The results showed that the effects of the individual variables and their quadratic terms were statistically significant whilst the interactions among the variables were statistically insignificant. Response surface plots drawn for spatial representation of the model showed that the nickel recovery depends more on particle size than on pH and pulp density. Using the model, optimised values of 2.6% pulp density, initial pH of 2.0 and 63 µm particle size resulted in a nickel recovery of 79.8%. Confirmatory test at these optimum conditions resulted in a nickel recovery of 74.1%; thus verifying that the model is valid and plausibly fits the experimental data with a marginal error of 7.7%. The significance of this study is that it has opened up an opportunity for the potential application of chemolithotrophic microorganisms for the commercial processing of the difficult-to-process low grade nickel laterite ores. Some of the examples where this process may be applied include silicate ores, oxidic converter furnace slags and refractory oxides.

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1. Introduction

With an ever increasing demand for nickel and daily depletion of high grade nickel sulphide ore reserves, research is necessary on how to process the more abundant nickel laterite ore reserves. Nickel laterite ores have complex chemical composition, low nickel content and are difficult to treat by conventional methods. Fortunately, microbiological leaching of low grade ores offers many advantages over other conventional methods due to its relative simplicity, requiring mild operating conditions, low capital costs, low energy input, relatively unskilled labour and being environmentally friendly, among others (Acevedo, 2000).

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Current worldwide research in the bacterial leaching of nickel laterite ores remain focussed essentially on the use of heterotrophic microorganisms (Alibhai et al., 1993; Valix et al., 2001a,b; Le et al., 2006). Recently the use of chemolithotrophic microorganisms in the bacterial leaching of nickel laterite ore has been explored (Simate and Ndlovu, 2007; Simate and Ndlovu, 2008). The recoveries obtained from these studies with chemolithotrophic microorganisms, however, are very low for commercial applications. In view of these low recoveries, studies on modelling and optimisation to increase the efficiency of these processes is very important. Therefore, the objective of this study was to determine the conditions that maximise the recovery of nickel. Increase in efficiency depends mainly on optimisation strategies used in a specific process (Edgar and Himmelblau, 1988). Optimisation can be defined as a process of improving an existing situation, device, or system such as a metallurgical process. It consists of finding the best solution to the process within the given constraints. In fact, process optimisation is

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essential for gaining and maintaining a competitive edge in today's world of intense financial competition.

There are essentially two types of optimisation that a metallurgical engineer needs to consider; the first which is termed topological optimisation deals with the topology or the arrangement of the process equipment. The second type termed parametric optimisation is concerned with the operating variables such as pH, temperature, pressure, particle sizes, and pulp density of streams for a given piece of equipment or process (Edgar and Himmelblau, 1988).

Unfortunately several popular optimisation methods usually do not work very well (Öberg and Deming, 2000). They either rely on the classical one parameter at a time approach that ignores the combined interactions between physicochemical parameters or are theoretical in nature. In this particular study, parametric optimisation was considered using a statistically-based optimisation strategy called response surface methodology (RSM) to determine the optimum conditions of pH, pulp density and particle size for bacterial leaching of nickel laterites. RSM is the most popular technique used to find the optimal conditions by using quadratic polynomial model and is applied as a consequence of a screening or diagnostic experiment (Myers and Montgomery, 2002). The central composite rotatable design (CCRD) was used to collect the data for fitting the second order response. The CCRD requires much fewer tests than the full factorial and has been shown to be sufficient to describe the majority of steadystate responses (Obeng et al., 2005).

To simplify the calculations and for uniform comparison, factors were studied with their codified values. The five levels of each factor shown in real values calculated using the relationships in Table 1 are shown in Table 2. The experimental results were analysed statistically by the analysis of variance (ANOVA) using Fisher's variance ratio test (F-test); standard errors of model coefficient (Student's t-test), the coefficient of determination (R^2) and the absolute average deviation (AAD).

2. Materials and methods

2.1. Ore samples and preparation

The ore was crushed and sized using the screen sizes given in Table 2. Table 3 shows the abundances of various constituent minerals of the laterite ore sample. Table 4 indicates the typical chemical composition of various oxides in the laterite ore in this study.

2.2. Microbes

A mixed culture of chemolithotrophic microorganisms (*Acidithiobacilllus ferrooxidans*, *Acidithiobacillus caldus* and *Leptospirillum ferroxidans*) used in the experiments was provided by Mintek, South Africa. Previous studies by Giaveno and Donati (2001) have shown that mixed cultures are more efficient than pure cultures because of the cooperation of the microorganisms involved in the mixed cultures. It is also expected that due to competition for oxygen by iron oxidising bacteria a reductive dissolution of nickel laterites by ferric iron in

Table 1Relationship between coded and actual values of the variables (Napier-Munn, 2000).

| Code | Actual value of a factor |
|------------|--|
| $-\lambda$ | x_{\min} |
| -1 | $\frac{(x_{max} + x_{min})}{2} = \frac{(x_{max} - x_{min})}{2\lambda}$ |
| 0 | $\frac{(x_{max} + x_{min})}{2}$ |
| +1 | $\frac{(x_{max} + x_{min})}{2} + \frac{(x_{max} - x_{min})}{2\lambda}$ |
| $+\lambda$ | x_{\max} |

 x_{max} and x_{min} are the maximum and minimum values of the natural variables respectively, $\lambda = (2^{k-q})^{1/4}$ for a CCRD, k = number of factors studied, $-q = \text{fraction of the number of factors (where <math>q = 0$ for full factorial design).

Table 2Observed and predicted values for the nickel recovery.

| Standard | Actua | Actual levels of variables | | | % recoveries | |
|--------------|-------|----------------------------|---------------|----------|--------------|--|
| runs | рН | Pulp density | Particle size | Observed | Predicted | |
| Factorial po | oints | | | | | |
| 1 | 1.5 | 5 | 38-75 μm | 71.7 | 74.5 | |
| 2 | 3.0 | 5 | 38-75 μm | 63.4 | 69.6 | |
| 3 | 1.5 | 12 | 38-75 μm | 63.1 | 63.1 | |
| 4 | 3.0 | 12 | 38-75 μm | 54.4 | 58.2 | |
| 5 | 1.5 | 5 | 106-150 μm | 68.3 | 64.6 | |
| 6 | 3.0 | 5 | 106-150 μm | 52.4 | 59.7 | |
| 7 | 1.5 | 12 | 106-150 μm | 50.5 | 53.2 | |
| 8 | 3.0 | 12 | 106–150 μm | 42.9 | 48.3 | |
| Axial points | 5 | | | | | |
| 9 | 1.0 | 9 | 75–106 μm | 67.7 | 69.6 | |
| 10 | 3.5 | 9 | 75-106 μm | 71.7 | 61.2 | |
| 11 | 2.3 | 2 | 75-106 μm | 85.1 | 80.6 | |
| 12 | 2.3 | 15 | 75-106 μm | 65.5 | 61.4 | |
| 13 | 2.3 | 9 | <38 μm | 60.6 | 56.0 | |
| 14 | 2.3 | 9 | 150–212 μm | 43.3 | 39.3 | |
| Centre poin | ts | | | | | |
| 15 | 2.3 | 9 | 75–106 μm | 66.8 | 61.0 | |
| 16 | 2.3 | 9 | 75–106 μm | 68.3 | 61.0 | |
| 17 | 2.3 | 9 | 75–106 μm | 55.4 | 61.0 | |
| 18 | 2.3 | 9 | 75–106 μm | 59.5 | 61.0 | |
| 19 | 2.3 | 9 | 75–106 μm | 60.4 | 61.0 | |
| 20 | 2.3 | 9 | 75–106 μm | 53.9 | 61.0 | |

nickel laterites by bacteria would take place producing ferrous ions that could be oxidised by the iron oxidising bacteria, thus destabilising the structure of the nickel laterites (Bridge and Johnson, 1998). The bacteria were cultured in standard 9K nutrient medium (Silverman and Lundgren, 1959) with a supplement of elemental sulphur.

2.3. Experimental design for the response surface methodology and central composite design

In previous studies (Simate and Ndlovu, 2007; Simate and Ndlovu, 2008), it was identified that pH, particle size, pulp density and substrate type were statistically significant operating parameters, while bacterial inoculum size was not statistically significant in the recovery of nickel. Follow up studies also showed that using sulphur substrate as an energy source for bacteria resulted in better nickel recoveries as compared to using pyrite (Simate et al., 2009). Response surface methodology and central composite rotatable design have been used in this study in an attempt to determine the optimal conditions of pH, pulp density and particle size for bacterial leaching of nickel laterites. The five levels of each factor shown in real and coded values calculated using the relationships in Table 1 are shown in Table 2.

Sulphur substrate having been significant in the previous study was taken as a held-constant qualitative factor. The major objective of the response surface methodology was to develop the optimum conditions so as to maximize the solubilisation of nickel laterites using a mixed culture of chemolithotrophic bacteria (*Acidithiobacillus*

Table 3Relative abundances of minerals in the nickel laterite ore sample.

| Mineral phase | Occurrence |
|---|-------------|
| Nickeliferrous serpentine [(Mg) ₃ Si ₂ O ₅ (OH) ₄] | Minor |
| Goethite, hematite [(Fe,Ni)O(OH), Fe ₂ O ₃ .H ₂ O] | Major |
| Titaniferrous pyrite [(Fe,Ti)S ₂] | Trace |
| Nickeliferrous pyrite [(Fe,Ni) S ₂] | Trace |
| Talc $[Mg_6Si_8O_{20}(OH)_4]$ | Trace |
| Saponite [(Ca _{0.25} (Mg,Fe) ₃ (Si,Al) ₄ O ₁₀ (OH) ₂ .nH ₂ O] | Trace |
| Tremolite [(Ca ₂ Mg ₅ Si ₈ O ₂₂ (OH) ₂] | Trace |
| Quartz [SiO ₂] | Predominant |

Predominant: >50% by mass; Major: 20–50% by mass; Minor: 5–10% mass; Trace: <5% by mass.

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