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Leaching optimization of copper from converter slag ash

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ABSTRACT

The main objective of this work was to determine the adequacy of Response Surface Methodology (RSM) in optimizing conditions for copper solubilization from a converter slag using sulfuric acid and to test the polynomial model associated with RSM. RSM was applied for optimization of the main factors, including initial pH, initial temperature, and pulp density. The maximum copper recovery was 100% under the optimal conditions of initial pH of 3, 1.4 g/100mL initial density, and 55°C. The confirmation test showed that the model can predict conditions accurately.

Introduction

The ashes generated by industrial units from the burning of fossil fuels like oil and coal represent a common environmental problem. Disposal methods can also cause problems, such as the leakage of acid liquids and the dusting and pollution of ground water with heavy metals [1,2]. Steelmaking operations are particularly concerned by this problem due to the production of slag ash that is useful as a building material in civil engineering [3,4]. However, steelmaking slag contains potential toxic elements that can be released into the environment, making its use potentially problematic. Many studies have been done to determine the optimal method for recovering valuable metals such as copper and iron from slag [3,4]. Hydrometallurgy is a process in which leaching is used to recover metal [5]. Various reagents have been used in chemical leaching. These include hydrochloric acid, nitric acid, ferric chloride, sulfuric acid, hydrogen peroxide, thiourea, potassium isocyanate, potassium iodide and iodine, thiosulphates, and cyanides[6,7]. Compared with pyrometallurgy, hydrometallurgy has a high recovery percentage, is cleaner for the environment, has a low gas emission and temperature, and is more economic for metal extraction from waste[8]. Different parameters exercise effects on the hydrometallurgic process. To optimize the leaching process, a methodology is required[9]. The conventional method for optimization is the one-factor-at-a-time method that evaluates changing one independent variable at a time while the other variables remain fixed [10]. Statistical optimization is a better method because it allows for evaluation of the effects of the factors, understanding the interactions among the factors, reducing the total number of experiments, building models, and searching for the optimal conditions [11,12]. Response surface methodology (RSM) is based on statistical optimization in which the optimal conditions of a multivariable system are determined [13]. The process consists of leaching slag using sulfuric acid and optimizing three main factors, namely initial pH, temperature, and pulp density, using RSM.

Materials and Methods

Slag sample

The converter slag used in this study was collected from disposal sites of waste materials from the Esfahan Steel Company, Iran. It was ground and passed through a No. 200 sieve to yield particles less than 75 mm. This prepared sample was the basis for all samples used in the experiments. X-ray fluorescence was used to analyze the chemical composition in the slag sample, which is shown in Table 1. All chemical materials were analytical grade reagents, and all aqueous solutions were prepared using distilled water.

Analytical methods

Leaching was done in 250 mL Erlenmeyer flasks. The flasks were shaken in an orbital shaker at 150 rpm. The pH and Eh of the medium were monitored during the leaching process using a portable pH/Eh meter. Atomic absorption spectroscopy was used to analyze the metals in the leaching process after filtration of the solution through a Whatman no. 42 filter paper.

Table 1. Chemical composition of convertor slag sample.

Element	Value (%)
Cu	4.2
Ni	1.98
Fe	38.8
Ca	4.01
Mg	2.6
Co	0.48
Al	0.08
SiO_2	34.3

Experimental design and optimization

The experimental design of the study was performed using Design-Expert 7.1.4 software. The central composite design (CCD) based on RSM was used extensively to introduce this polynomial model. According to the CCD method, the total number of experiments was obtained using 2k + nα + n0, where k is the number of independent variables, nα represents the axial points, and n0 is the center point [14,15] . In this study, there were three independent variables: initial pH, temperature, and pulp density. The center point was 6, which led to a total of 20 runs. The behavior of the system is explained by the polynomial empirical model.

$$y = \beta_0 + \sum_{i=1}^{k} \beta_i X_i + \sum_{i=1}^{k} \beta_{ii} X_i^2 + \sum_{i=1}^{k} \sum_{j=1}^{k} \beta_{ij} X_i X_j + \varepsilon \quad (1)$$

Where y is the expected value of the response variable, and are the model parameters, and Xi and Xj are the coded factors evaluated. In this study, y represents the copper recovery. The variables of Xi were coded as xi according to the following equation [16]:

$$x_i = \frac{(x_i - x_0)}{\Delta x}, i = 1,2,3,\ldots,k \quad (2)$$

where xi is a dimensionless coded value, Xi is the real value, X0 is the value of Xi at the center point, and ΔXi is the step change of the real value of the variable i. Each factor was varied at five different levels based on the CCD shown in Table 2. The analysis of variance (ANOVA) of the polynomial model was carried out to evaluate the significant parameters in this study, and a p-value of 0.05 was considered significant. In order to verify the optimal conditions, an experiment at the obtained optimal conditions using RSM was performed and the result was compared with predicted result.

Table 2. Variables levels used for experimental design.

Factor	Code	Unit	Low axial (-α)	Low factorial (-1)	Centre point (0)	High factorial (+1)	High axial (+α)
Initial pH	A	-	1.5	1.8	2.2	1.7	3
Initial density	B	g/l00ml	0.5	1.4	2.75	4.0	5
Temperature	C	°C	25.5	33	44	55	62.5

Results

Fig. 1 shows the leaching of copper at different time points at a pulp density 0.5 g/100ml, a temperature of 35 °C, and an initial pH of 6. The pH and Eh variations with time are shown in Fig. 2. The experimental conditions and results are shown in Table 3. In order to analyze copper recovery, an analysis of variance (ANOVA) test was performed and the results are shown in Table 4. Fig. 3a and b shows the two dimensional contour plot between different parameters for copper recovery.

Discussion

Leaching recovery at different time points

The leaching of copper at different time points and certain conditions was represented in Fig. 1. With regard, the recovery increased with time until 90 min, after which the recovery was approximately constant. This constant recovery could be due to intra-particle limited diffusion, formation of a product layer, depletion of easily available reacting species, or a combination of these phenomena. The ultimate metal recovery for copper after 120 min was 67% with these parameters.

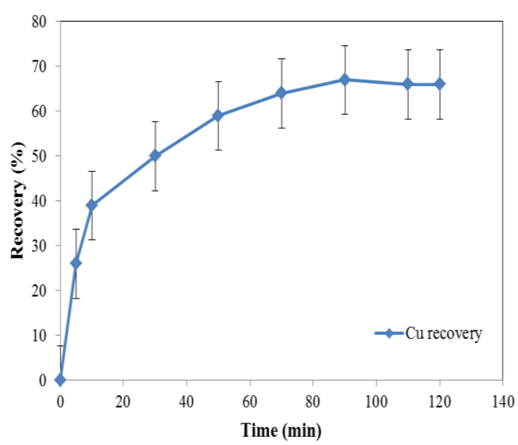

Figure 1. Metals leaching recovery versus time

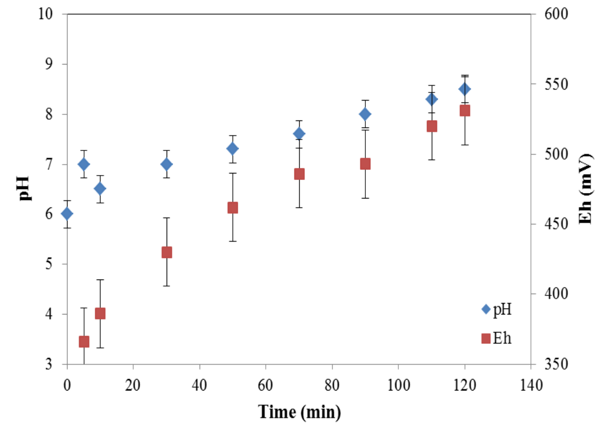

Figure 2. pH and Eh variation with the time

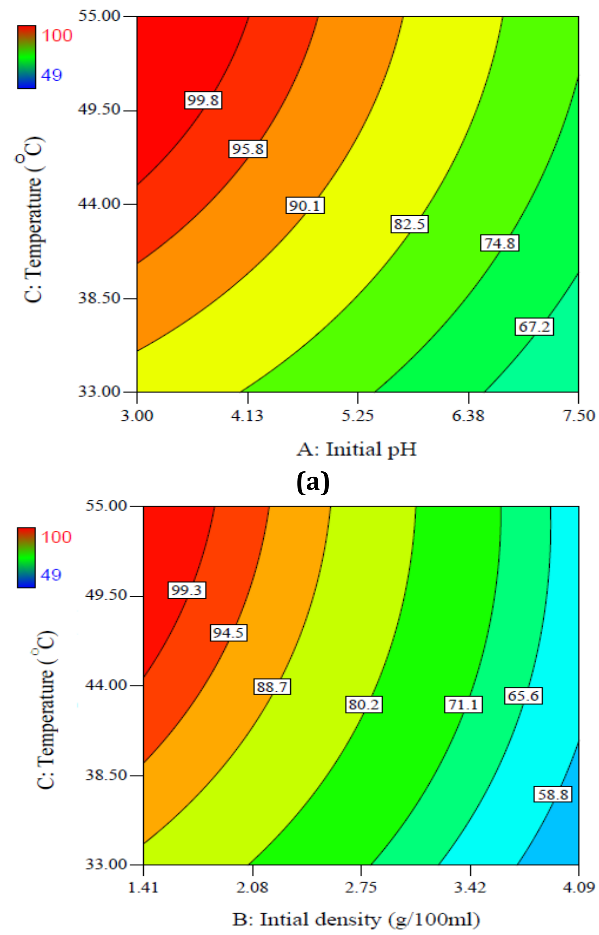

Figure 3. Contour plots of the interaction effects for Cu recovery: (a) temperature and initial pH (b) temperature and initial density.

pH and Eh variations

Fig. 2 displays the pH and Eh variations with time. Due to acid consumption during the leaching process, the pH increased over time. After 120 min, the final pH was 8.5. Eh is related to the potential to extract ions from the slag. The highest Eh was 531 mV, which was observed after 120 min.

Statistical analysis

The quadratic model was used to predict copper recovery. The p-values for copper recovery were less than 0.05, indicating statistical significance (see table 1 and 2).

The R-squared for copper recovery was 0.92, which implies that the model is a good fit with the experimental data. The coefficient of variation (CV) is defined as the ratio of the standard deviation to the mean and measures the reproducibility of the model. If the CV is <10%, the model can be considered reasonably reproducible [16]. The results showed that both models had CV values of <10% [16].The adequate precision, which represents the signal to noise ratio and indicates reliability at a value >4, was 16.7.

Fitted statistical models for metals recovery

The quadratic empirical relationship between copper recovery and the variables obtained from Design-Expert 7.1.4 software is shown in the following equation:

$$\text{Copper recovery (\%)} = 77.41 - 6.70\times A - 11.75\times B + 6.42\times C + 7.50\times AB - 0.50\times AC - 2.25\times BC - 2.57\times A^2 - 1.87\times B^2 - 2.75\times C^2 \quad (3)$$

where A is the initial pH, B is the initial density, and C is the temperature. Fig. 3 shows the predicted values versus the actual data. The points around the model line show a satisfactory correlation between the experimental data and the predicted values [9].

Contour plots in metals recovery

As shown in Fig. 3a, the copper recovery increased as the initial pH decreased and the temperature increased at a constant of initial density of 1.4 g/100ml. With an initial pH of 3 and temperature of 50°C, maximum copper recovery was reached with 100% recovery. Decreasing the pH resulted in increased proton concentration in the leaching medium as the oxidant agent; therefore, recovery was increased.

Fig. 3b shows the interaction between initial density and temperature effects on copper recovery at a constant initial pH of 3. According to the model, decreasing initial density and increasing temperature caused increased copper recovery. At low initial densities, the mass transfer limitation decreased and the recovery of metals was improved. As shown in Fig. 3b, a maximum recovery of 100% copper was observed with an initial density of 1.4 g/100mL and temperature of 50°C at the constant initial pH of 3.

Optimization process

Optimization was based on the maximization of copper recovery. It should be noted that the goal of optimization is to find a set of conditions that will meet all of the recovery goals. In numerical optimization, a minimum and maximum level must be provided for each parameter. The optimal conditions proposed by the model were initial pH 3, temperature 55°C, and initial density 1.4 g/100mL, with which a maximum recovery of copper of 100% was achieved.

Confirmatory experiments

To test the validity of the optimized model conditions, an experiment was carried out with the values suggested by the model (data not shown). Under the predicted optimized conditions, the experimental recovery result for copper was 100%.

Conclusions

The leaching of converter slag sample using sulfuric acid was studied. The recovery of copper was optimized using RSM. Under the optimal conditions, which were initial pH 3, temperature 55°C, and initial density 1.4 g/100 ml, the recovery of copper reached 100%. The results showed that the R-squared for copper recovery was 0.92 and the p-values for copper recovery were less than 0.05, indicating that the models can significantly predict conditions with a 95% confidence level.

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