Nickel Extraction from Fuel Oil Ash Using Sonoleaching Process

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Introduction
Fly ashes, which contain metals such as vanadium (V) and nickel (Ni), would create many environmental problems, including soil, water and air pollutions [1-3]. Therefore, the extraction of metals from produced fly ashes is very desirable in both environmental and economic aspects [3]. Nowadays, material processing using hydrometallurgical techniques is becoming a good and effective way to recover metals [4]. Although hydrometallurgy has many advantages, including low-cost equipment, non-sensitivity to environmental conditions, and reduced air pollution, it still has problems such as relatively long operating periods [5]. To assist the effect of acid in the leaching process, different methods including ultrasound, H2O2 and microwave have been used [6]. In this study, sulfuric acid has been used as a leaching agent, as well as ultrasonic as an auxiliary agent for the extraction of Ni from the power plants oil-furnace ash. In addition, the response surface method has been used to optimize the factors. In order to better examine the process, kinetic and thermodynamic studies have also been studied.

Experimental Procedure
Preparation of the Fly Ash
A Neka thermal fly ash sample was collected from the Mazandaran, Iran. Aqua regia was prepared for the complete digestion of the sample. Inductively coupled plasma optical emission spectrometry (ICP-OES) (Vista Pro, Varian, Inc., California, USA) was used to analyze chemical elements. The major elements were 4665 ppm V, 1346 ppm Ni, 1059 ppm Fe, 5.25 ppm Cu and 90.45 ppm Al by mass.

Leaching Experiment
In each experiment, a certain amounts of the fuel oil ash and sulfuric acid were put into a 250 mL glass beaker. An ultrasonic probe (UC-5150B, ChromTech, Taiwan) was inserted in the solution during leaching. Furthermore, atomic adsorption (AAS-NORDANTEC990) was used to determine the concentrations of metals in the liquor.

Experimental Design
Design Expert 7.0.0 software was adopted to optimize the leaching parameters. Response Surface Methodology (RSM) was applied to study the effects of three main factors, namely solid/liquid (S/L) ratio (1-4 %w/v) sulfuric acid concentration (1-70 %v/v) and ultrasound power (5-80 W) on Ni recovery. Analysis of Variance (ANOVA) was used to determine the significant models and parameters.

Results and Discussion
Statistical Analysis
A reduced second model was used for the Ni extraction. The correlation between Ni recovery and the three defined factors is shown as follows:
Ni extraction (%) =+16.13 – 3.89A +3.81B +1.71C–0.80BC–3.83B2 (1)
where A, B and C are S/L ratio, sulfuric acid concentration and ultrasound power respectively. ANOVA results showed that at a 95% confidence level, the calculated p-value of the model was less than 0.05. Fig. 1 shows the interaction between S/L ratio and sulfuric concentration at constant 61.6 W ultrasound power. According to it, the Ni extraction was enhanced when acid concentration increased from 15 %v/v to 56 %v/v and decreasing S/L ratio from 3.4 %w/v to 1.6 %w/v.

The goal of optimization was to achieve the highest Ni extraction. The optimal conditions were 1.61 %w/v of S/L ratio, 64.8 %v/v of sulfuric concentration and ultrasound power of 43.6 W. The experimental result fell within the 95% confidence interval (C.I.) of the model value.

**Kinetic and Thermodynamic Study**

The kinetic parameters of the process were analyzed using the shrinking core model. The obtained R² values indicated that the data were fitted with the diffusion-controlled model slightly better than the reaction-controlled model. Using the definition of k based on the Arrhenius equation in the reaction equation, then by plotting ln(r) vs. 1/T, the activation energy was estimated to be 9.57 kJ·mol⁻¹ which is less than 25 kJ·mol⁻¹. This suggests diffusion control in the leaching process.

![Fig.1. Contour plots of the S/L ratio and Acid concentration on Ni extraction.](image)

**Conclusions**

The extraction of nickel from ash was investigated using sonolization method. The recovery of Ni was optimized using RSM. The optimal conditions were 1.61 %w/v of S/L ratio, 64.8 %v/v of sulfuric concentration and ultrasound power of 43.6 W. The results of reaction kinetics analysis showed that the diffusion-control model was favored over the overall reaction-control model. Finally, the activation energy of the reaction was found to be 9.57 kJ·mol⁻¹.