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MINERAL DRESSING

Post-Leaching of Silver from a Non-Sulfide Lead–Zinc Ore Flotation Tailing Leach Residue in a Copper–Ammonium Thiosulfate Solution: A Fuzzy Logic Prediction

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Abstract—The post-leaching of silver (Ag) from a non-sulfide lead–zinc (Pb–Zn) ore flotation tailing leach residue in a copper–ammonium thiosulfate solution was investigated. Ag (89.7%) was extracted into the leaching solution under the following conditions: 30 g/l ammonium thiosulfate, 0.5 g/l copper sulfate, 25 °C leaching temperature and 4 h leaching time. On the basis of the experimental results, a fuzzy logic prediction was made. Ammonium thiosulfate, copper sulfate and leaching period were chosen as predictive criteria in this step. The fuzzy prediction model was found to be very consistent with the experimental data (R^2:0.9657). Based on these findings, the application of the fuzzy logic prediction approach to the silver dissolution from the leach residue could be considered.

Keywords: Silver, ammonium thiosulfate, copper sulfate, flotation tailing, fuzzy logic prediction.

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INTRODUCTION

Silver is a vital component of many industrial processes and produced in large quantities as a byproduct of the extraction of base metal ores such as copper, lead, and zinc. Due to its high acid resistance, high melting point, excellent electrical conductivity, and high ductility, it is widely used in electronic, electrical, aerospace, and other industries [1]. According to the US Geological Survey (USGS), yearly global silver production will decline by 6% to an estimated 25.000 t in 2020. Silver production from mines in China, Mexico, and Peru declined marginally in the first half of the year due to shutdowns in response to the COVID-19 outbreak [2]. With an increasing demand for scarce raw materials, silver recovery from secondary sources has gained substantial interest in recent years. For example, technologies for the chemical treatment of silver from waste streams such as wastewater and scrap materials have been developed in urban mining.

Silver is found in a variety of materials, including gold-silver ores, used catalysts, fibers, and printed circuit boards (PCBs) [3–5]. Although several procedures for the leaching of silver from raw ores and secondary sources have been reported, the most effective method is cyanide leaching. However, strict environmental regulations have recently prompted the exploration of thiosulfate as a possible substitute for cyanide in the leaching of precious metal deposits [6–10]. Due to the strong compounds formed with silver, the thiosulfate solution has also been considered an effective method.

Numerous studies have been conducted to identify alternate lixiviants and leaching systems, including silver sulfide in a ferricyanide-cyanide solution, as well as silver sulfide in $FeCl_3$–HCl and $Fe_2(SO_4)_3$–H_2SO_4 solutions [11–13]. Thiosulfate is getting growing attention as an alternative to the cyanide process, such as the Patera process [14], and is most likely one of the most plausible substitutes for cyanides due to its low cost and nontoxicity.

When the silver embedded in the ores is scattered, the complexation of silver and thiosulfate is adequate [15, 16]. Ag and Au, as well as base metals, are heavily dependent on sulfide ores. Numerous investigations published recently have demonstrated that sulfide minerals and concentrates dissolve to varying degrees in thiosulfate-ammonia environments [17–21]. The researchers investigated the mechanochemical pretreatment and mechanical activation of silver, gold (Au), and bismuth (Bi) from sulfide concentrate [19]. The extraction of silver selectively from sulfide concentrations was optimized [20]. Thermodynamic evaluations of the leaching solution were conducted to determine their ability to remove Ag, Pb and Au [21]. Previous research has demonstrated that it is possible to recover more than 90% of Ag from ores or concentrates.

Cyanidation's environmental rules and the rising price of silver have generated considerable interest in hydrometallurgical techniques for processing silver-bearing raw ores and secondary resources. Increased cyanide content results in a rise in silver and mercury dissolution, posing an environmental hazard [22]. Yiqi et al. 2011 [23] examined the extraction of silver from a synthetic silver sulfide powder in the absence of ammonia using a cupric-thiosulfate method. The silver extraction rate was 95.1% when a 0.12 M thiosulfate solution was used in the presence of 0.05 mol/l cupric, according to the study.

Therefore, it is of great significance to study the dissolution of silver from lead-zinc flotation rejects in a copper-ammonium thiosulfate solution to expand its application range. The primary objective of this study is to investigate the effect of leaching parameters such as ammonium thiosulfate concentration, copper sulfate concentration, leaching time, and leaching temperature on the dissolution of silver from a non-sulfide ore flotation reject leach residue, which has not investigated before. Additionally, the paper evaluates the prediction of silver dissolution using a fuzzy logic approach, one of the most efficient artificial intelligence techniques for consistently and flexibly resolving complicated and confusing situations. Our prior research has a wealth of additional information about the sample [24, 25].

1. MATERIALS AND METHODS

Orex Mining Co. in Kayseri, Turkey supplied the tailing sample. After a two-step leaching procedure utilizing sulfuric acid and a solution of sodium hydroxide and potassium sodium tartrate, the leach residue was collected for post-leaching tests using a copper-ammonium thiosulfate system. At ALS Mineral Laboratory in Izmir, Turkey, the elemental composition of the collected residue was determined using inductively coupled plasma mass spectrometry (ICP-MS). The chemical analysis determined that the residue contained 194 ppm Ag, 270 ppm Cu, 100 ppm Cr, 18900 ppm Pb, 17950 ppm Zn, 14900 ppm As, 10200 ppm Ba, and 29.2% Fe. Al (3.11%), K (1.2%), Ca (1.05%), Na (0.98%), and Mg (0.41%) were determined to be the alkali metals in the residue. The residue's mineralogical composition was determined using the Bruker D8 Discover (XRD) and the position of the $2\theta = 10 - 70^\circ$ radiation produced at 40 mA and 45 kV was regulated using a silicon reference. Mineral identifications were performed using the ICDD PDF-2/Minerals database. Major minerals in the residue were, %: quartz (SiO_2)—45.7, anglesite ($PbSO_4$)—15.9, corkite ($PbFe_3[(PO_4)(SO_4)(OH_6)]$)—12.7, dolomite ($CaMg(CO_3)_2$)—7.2, goethite ($FeO(OH)$)—9.5; while minor mineral phases included, %: calcite ($CaCO_3$)—4, smithsonite ($ZnCO_3$)—3, cerussite ($PbCO_3$)—2.

The post-leaching study was conducted in a 100-ml glass vessel equipped with a temperature-controlled heating mantle and magnetic stirrers (MTOPS). Using a Hach 40D pH meter, the pH of the slurry was checked on a regular basis. To decrease evaporative loss, the vessel was wrapped with aluminum foil. Ammonium thiosulfate (50 ml) and copper sulfate were added to the vessel in predetermined concentrations for each leaching experiment. After heating the solution to the desired temperature, the sample was added to the vessel to initiate the leaching process. After the leaching process was complete, the magnetic stirrer and temperature controllers were turned off and the vessel cooled. Filtration and slurry washing occurred using deionized water. The residue was dried using a 100 °C oven for 24 h. Weighing and processing the dried residue in hot aqua regia (3:1 of $HCl:HNO_3$). An atomic absorption spectrophotometer was used to determine the amounts of silver in the solution (AAS-PinAAcle 900F PerkinElmer). Based on the metal composition of the leached residue, the metal dissolution percentage was calculated. Analytical grade nitric acid (HNO_3, Merck), hydrochloric acid (HCl, Merck), ammonium thiosulfate ($NH4S_2O_3$, Acros organics), and copper sulfate ($CuSO_4$, Detsan) were used. The stirring speed was kept constant at 400 rpm throughout the leaching tests. The majority of leaching experiments were duplicated to determine the repeatability of the results.

2. FUZZY SET THEORY

Fuzzy Logic is a problem-solving methodology that is capable of handling both linguistic and numerical data in real-time. This technique simplifies the control of a complicated system. In contrast to traditional logic, fuzzy logic makes use of linguistic phrases such as true or false, black or white, and on or off. While an object in conventional logic can only have a value of zero or one, a statement in fuzzy logic can have any actual value between 0 and 1. The fuzzy logic process is summarized in [26]. The main steps are as follows.

1. Determination of the fuzzy linguistic parameters for the input and output. The first stage in developing a fuzzy logic model is to choose appropriate inputs. These input variables should be capable of representing the system in its entirety.

2. Using input membership functions, fuzzify the inputs. The fuzzification method uses membership functions to convert the crisp inputs to a fuzzy set membership degree [27]. These membership functions should encompass the entirety of discourse and be capable of representing either a linguistic variable or a fuzzy set. Trapezoidal, triangular, and Gaussian membership functions are frequently utilized. Triangular and trapezoidal shapes are the most frequently utilized because they are simple to represent the user's concept and need less calculating time [26]. Three parameters define a triangle membership function (a_1, a_2, a_3):

$$\mu_A(x) = \begin{cases} 0, & x < a_1, \\ \dfrac{x - a_1}{a_2 - a_1}, & a_1 \le x \le a_2, \\ \dfrac{a_3 - x}{a_3 - a_2}, & a_2 \le x \le a_3, \\ 0, & x > a_3. \end{cases}$$

Figure 1 shows a **s**chematic view of a triangular membership function [28].

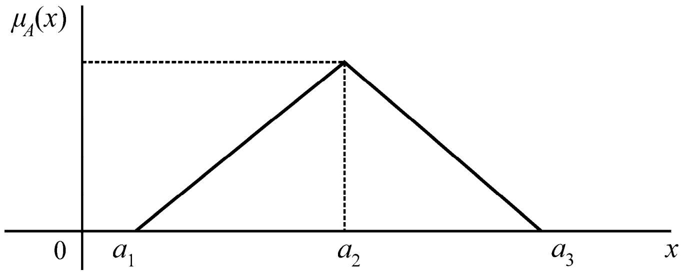

Fig. 1. Triangular membership function.

3. Establishing fuzzy rules. It is composed of numerous if-then statements. The rule's "If" section is referred to as the antecedent, while the "Then" section is referred to as the result. The following fuzzy rules are presented:

If input 1 — MF1, input 2 — MF2, and/or…, the output is then output MF,

4. Defuzzification of the distribution of output values. The fuzzy variables are turned into crisp sets in this stage. This step is critical since the crisp values can only be utilized as inputs in the real world's other systems. This is typically only required when the Mamdani Fuzzy Model is utilized to construct a controller. Larsen, Tagaki–Sugeno, and Tsukamoto are other fuzzy inference systems. Unlike the Mamdani model, the outputs of the other two models are recognized using a specific function, and so the result is crisp rather than fuzzy. This is irrational, as a fuzzy model should be capable of properly spreading the fuzziness from the inputs to the outputs. The literature contains a variety of defuzzification techniques, including the centroid of the area, the bisector of the area, the mean of the maximum, the smallest of the minimum, and the largest of the maximum. Due to their inherent bias, the final two defuzzification procedures are rarely utilized. The method that is most frequently employed is the centroid of the area method.

This method asserts a single crisp integer by utilizing the output dispersion and determining its center of mass. It is as follows:

$$z = \frac{\sum_{j=1}^{q} z_j u_c(z_j)}{\sum_{j=1}^{q} u_c(z_j)},$$

where u_c is the membership in class c at value z_j; z is the center of mass [29].

3. RESULTS AND DISCUSSION

Experiments were conducted using a concentration of 20 g/l ammonium or sodium thiosulfate and 0.5 g/l copper sulfate at a pH of 10.5. These trials were carried out at a temperature of 25 °C. Figure 2 illustrates leaching times ranging from 1 to 6 h.

Silver and copper were discovered in the ammonium thiosulfate leaching process in the form of amine and thiosulfate complexes. According to equations:

$$Ag + S_2O_3^{2-} + [Cu(NH_3)_4]^{2+} \rightarrow [Ag(S_2O_3)_2]^{3-} + 2NH_3 + [Cu(NH_3)_2]^{+}, \ \Delta G = -581.2 \text{ kJ/mol},$$

$$Ag + 5S_2O_3^{2-} + [Cu(NH_3)_4]^{2+} \rightarrow [Ag(S_2O_3)_2]^{3-} + 4NH_3 + [Cu(S_2O_3)_3]^{5-}, \ \Delta G = -16.1 \text{ kJ/mol},$$

where ΔG is the Gibbs energy, the cupric-tetramine complex $[Cu(NH_3)_4]^{2+}$ acts as the main oxidant [17, 30].

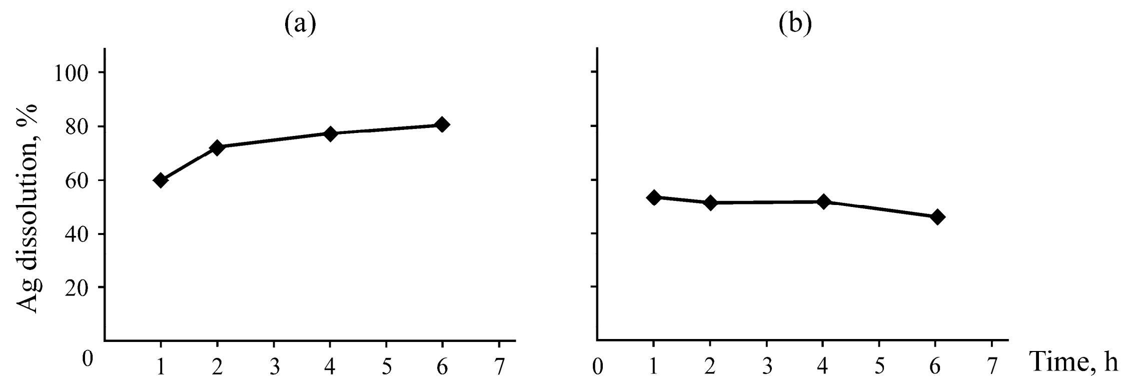

Fig. 2. Effect of (a) ammonium thiosulfate (20 g/l) and (b) sodium thiosulfate (20 g/l) on Ag dissolution.

When the leaching duration was increased from 1 to 4 hours, the dissolution of Ag was dramatically increased from 59.8 to 77.1% using ammonium thiosulfate (Fig. 2a). It can be seen that longer leaching durations promote Ag dissolution, but shorter durations result in poor dissolution percentages, which can be related to the requirement for further leaching time due to insufficient thiosulfate concentrations. According to Bae et al. 2020 [31], the greatest dissolution of Ag may be reached by increasing the leaching period to 4 h and decreasing the solid-to-liquid ratio from 0.5 to 0.2. In comparison to ammonium thiosulfate, the dissolution of Ag in sodium thiosulfate solution (Fig. 2b) remained relatively stable at around 51% up to 4 h before dropping significantly to 45.5% when the leaching time was increased to 6 h. This is most likely due to the low concentration of hydrogen ions, which is insufficient to form a complex. Ammonium thiosulfate was found to be a more acceptable leaching agent than sodium thiosulfate in terms of the percentage of Ag dissolved in the leach residue under the conditions investigated.

The effect of ammonium thiosulfate concentration on the dissolution of Ag as a function of time is shown in Fig. 3 using a liquid-to-solid ratio of 10:1, a leaching temperature of 25 °C, $CuSO_4$ concentration of 0.5 g/l and pH 10.5.

The dissolution of Ag rises with increasing ammonium thiosulfate concentration, which is consistent with the results of Bae et al. 2020 [31]. As shown in Fig. 3a, Ag dissolution rose rapidly up to 4 h when 30 g/l ammonium thiosulfate was used and subsequently remained reasonably steady; in particular, 87.6% Ag dissolution was seen after 4 h at 30 g/l ammonium thiosulfate. There was no significant effect of leaching duration on Ag dissolution at 40 and 50 g/l ammonium thiosulfate concentrations, as Ag dissolution was less influenced than that at 30 g/l ammonium thiosulfate concentration for the initial 1 h post-leaching of Ag (Fig. 3b, c).

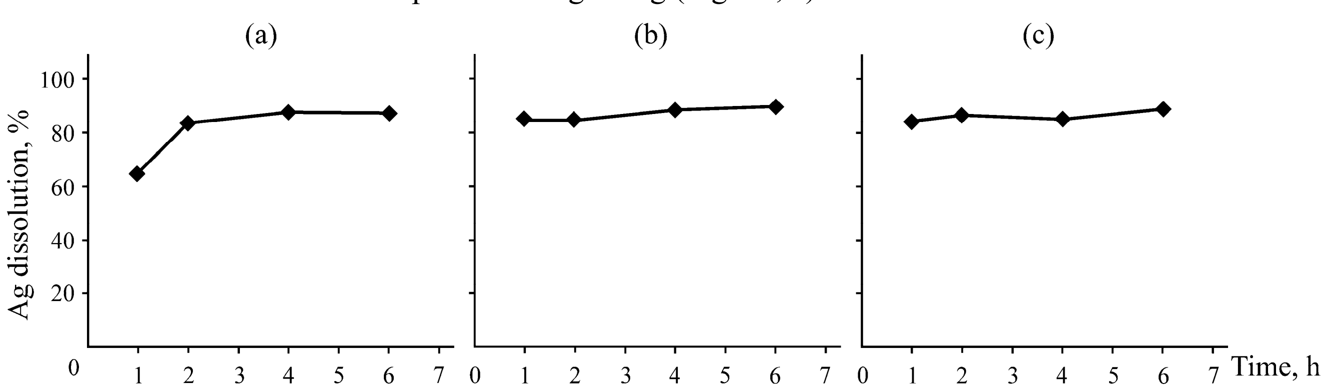

Fig. 3. Effect of ammonium thiosulfate on Ag dissolution: (a) 30 g/l, (b) 40 g/l, and (c) 50 g/l of ammonium thiosulfate.

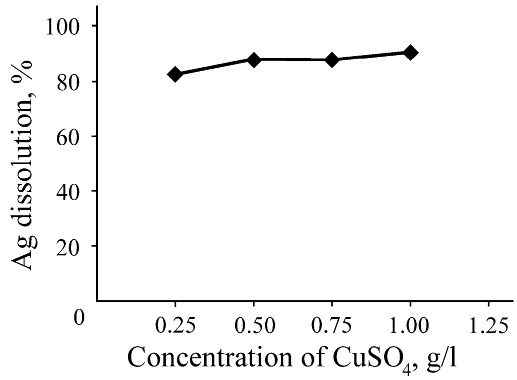

Fig. 4. Effect of copper sulfate on Ag dissolution.

Ficeriova et al. 2005 [19] reported that silver dissolution from complicated sulfide concentrates using ammonium thiosulfate without any mechanochemical preparation was 52% within 1 h of leaching using 74 g/l ammonium thiosulfate, 10 g/l of $CuSO_4$ at 70 °C and pH 5. As a result of these observations, 30 g/l of ammonium thiosulfate and a 4-hour leaching time are sufficient to dissolve over 85% Ag in this solution. Additionally, it was observed that ammonium thiosulfate dissolved less than 90% of the Ag. This is most likely due to Ag being trapped within the quartz crystal phases.

The dissolution of Ag is shown in Fig. 4 as a function of the $CuSO_4$ concentration. These investigations were conducted at a liquid-to-solid ratio of 10:1 and a temperature of 25 °C for 4 h with a concentration of 30 g/l ammonium thiosulfate, pH 10.5. The dissolution of Ag increased slightly from 82.5 to 87.6% up to a $CuSO_4$ concentration of 0.5 g/l and thereafter gradually increased. According to Bae et al. 2020 [31], silver dissolution from concentrated sulfide ore increased in proportion to the concentration of copper sulfate. Ag dissolution of 90.6% was accomplished at a $CuSO_4$ concentration of 1 g/l. The dissolution of Ag was determined to be 87.8% at a $CuSO_4$ concentration of 0.75 g/l. The dissolution of Ag was found to be less impacted by changes in $CuSO_4$ concentration than by changes in ammonium thiosulfate concentration. As a result, the concentration of 0.5 g/l $CuSO_4$ was found to be appropriate for Ag post-leaching in this investigation.

Figure 6 illustrates the influence of temperature on Ag dissolution in ammonium thiosulfate solutions. The temperature of the leaching solution was increased from 25 to 60 °C for 4 h, with a liquid-to-solid ratio of 10:1 and pH 10.5 using 30 g/l of ammonium thiosulfate and 0.5 g/l of copper sulfate. The Ag dissolution increased gradually up to the reaction temperature of 40 °C and thereafter remained essentially constant. At 25 °C for 4 h, the present results suggested that 87.6% Ag dissolution was obtained. In contrast to our findings, Li et al. 2018 [32] discovered that Ag recovery reduces when leaching temperature increases from 20 to 70 °C, when 75 g/l of sodium thiosulfate, a 2-hour leaching duration, and pH 5 are used. The optimal temperature for post-leaching was determined to be 25 °C based on the experimental results.

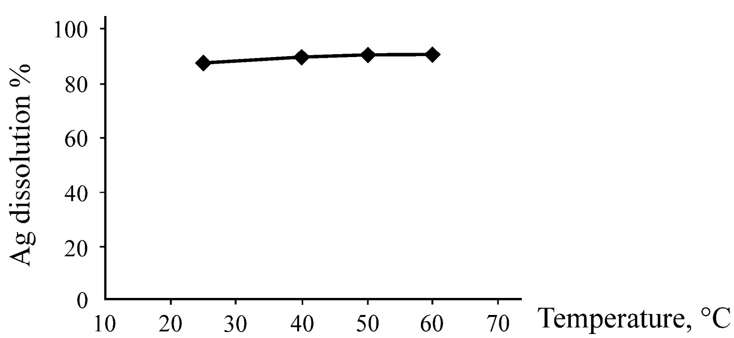

Fig. 5. Effect of leaching temperature on Ag dissolution.

Table 1. Linguistic expression and membership function parameters of fuzzy logic model

Parameter	Description	Membership function parameters	Linguistic expression level
Input	Leaching time, h	[1 2 4] [2 4 6]	Low High
	Ammonium thiosulfate concentration, g/l	[20 30 40] [30 40 50]	Low High
	Copper sulfate concentration, g/l	[0.25 0.50 0.75] [0.50 0.75 1.00]	Low High
Output	Silver dissolution, %	[65.0 82.5 87.6] [82.5 87.6 90.6]	Low High

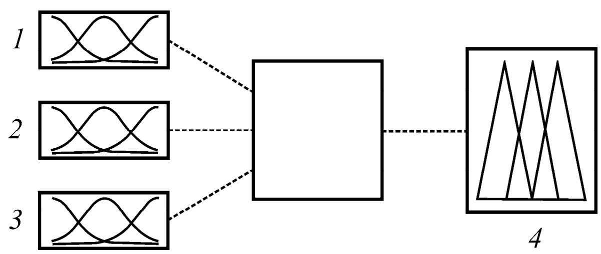

Fig. 6. Structure of the fuzzy logic model: *1*—leaching time; *2*—ammonium thiosulfate concentration; *3*—copper sulfate concentration; *4*—silver dissolution.

The initial step for the fuzzy logic model was to identify the membership functions of the input and output parameters. Table 1 contains the linguistic expression and membership function parameters for the input and output parameters.

According to the experimental results, the following parameters were selected for a fuzzy logic model: triangular membership function, Mamdani inference system, and center of the area defuzzification approach. Figure 6 illustrates the structure of the fuzzy logic model.

Figure 7 illustrates the graphical membership functions for the input and output parameters. MATLAB R2015a software was used to implement the fuzzy logic.

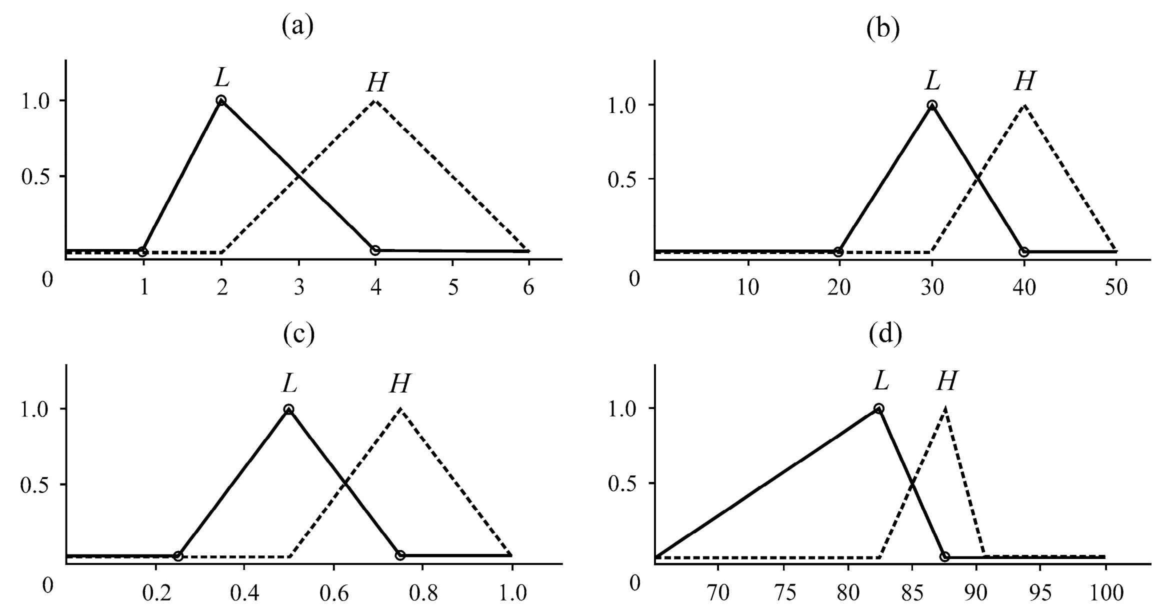

Fig. 7. Membership functions of (a) leaching time; (b) ammonium thiosulfate concentration; (c) copper sulfate concentration; (d) silver dissolution (*L*—low level, *H*—high level).

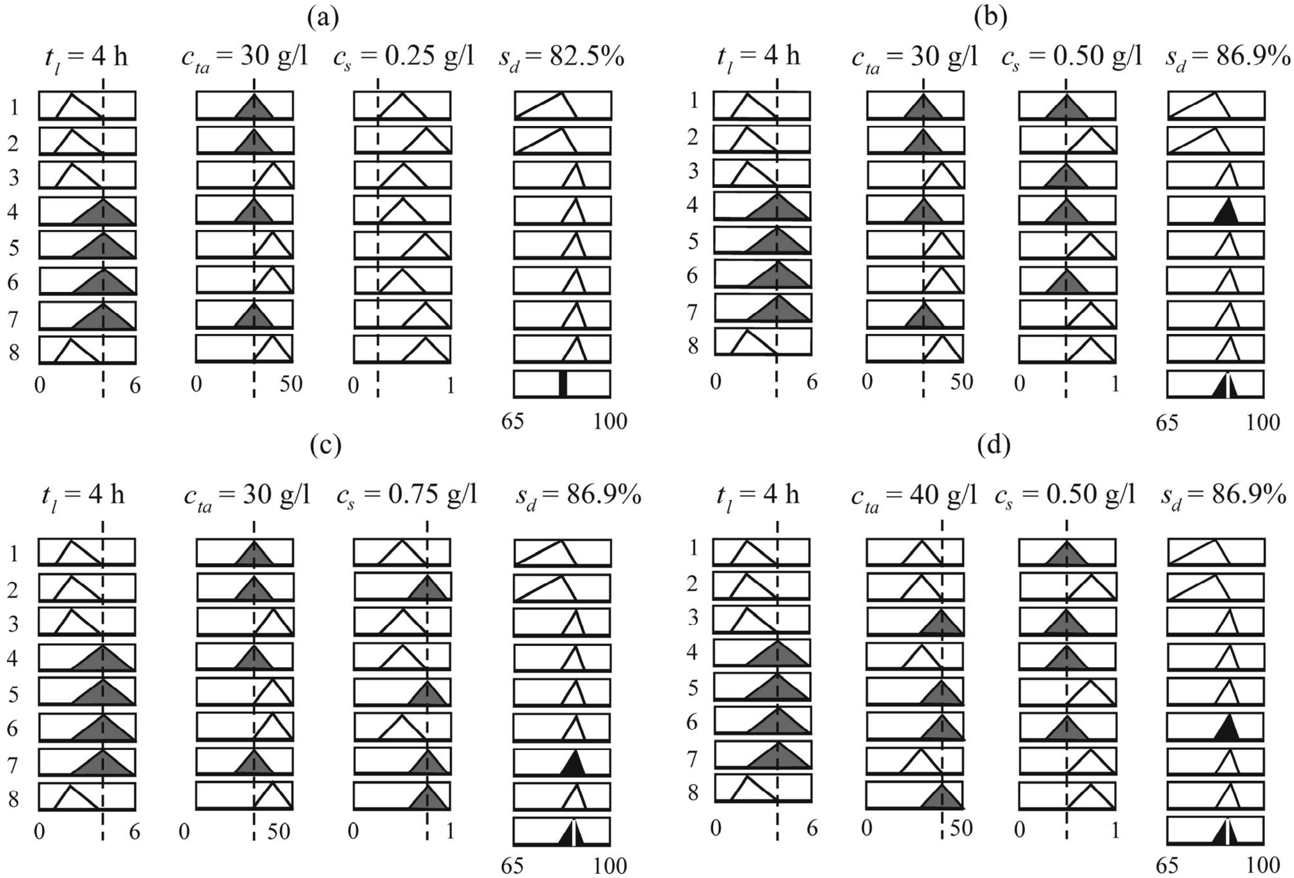

Fig. 8. Graphical representation of fuzzy experimental results 1 (a), 2 (b), 3 (c), 4 (d): t_l—leaching time; c_{ta}—concentration of ammonium thiosulfate; c_s—concentration of copper sulfate; s_d—silver dissolution.

The fuzzy model is fed six language terms as inputs (leaching time, ATS concentration, and copper sulfate input parameters each have two linguistic expressions). As a result, eight fuzzy rules were created.

The fuzzy logic method for predicting silver dissolution comprises four steps. It takes crisp input parameters such as leaching duration, ATS concentrations, and copper sulfate and converts these to fuzzy inputs via membership functions (low or high level). The fuzzy rules were used to evaluate these fuzzy inputs. Following that, fuzzy outputs were generated. Finally, we combined the fuzzy outputs into a single crisp output (silver dissolution). To assess the proposed fuzzy model's performance, a comparison of anticipated and experimental values was made.

Fig. 8 illustrates the fuzzy findings graphically. Silver dissolution was predicted using various combinations of input factors and the results are shown in Fig. 9.

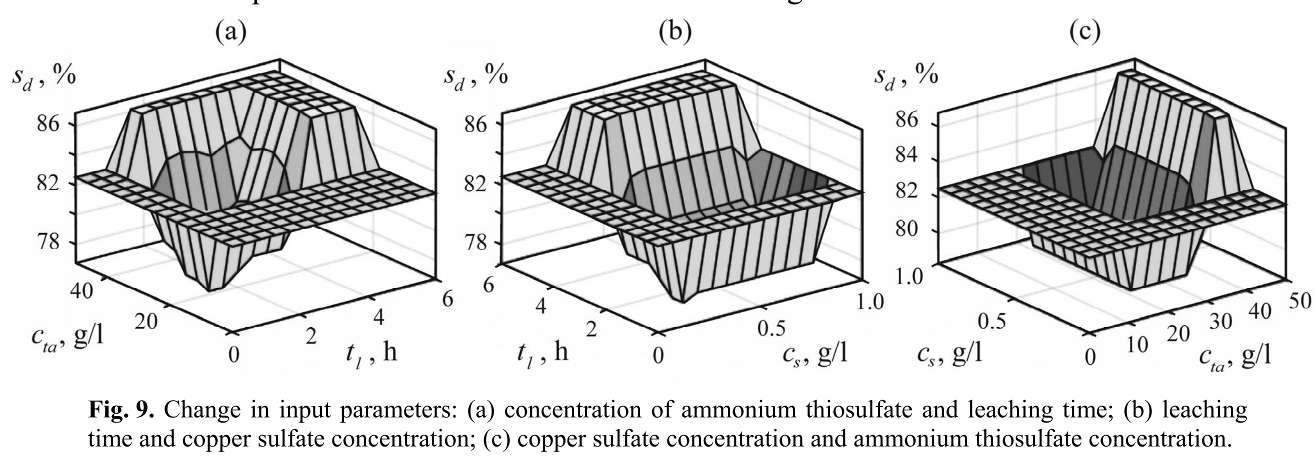

Fig. 9. Change in input parameters: (a) concentration of ammonium thiosulfate and leaching time; (b) leaching time and copper sulfate concentration; (c) copper sulfate concentration and ammonium thiosulfate concentration.

Table 2. Comparison between the fuzzy model and experimental data

Experiment no.		Input parameters		Output parameters	
		Concentration, g/l		Silver dissolution, %	Fuzzy logic result, %
		Ammonium thiosulfate (ATS)	Copper sulfate		
1		30	0.25	82.5	82.5
2		30	0.50	87.6	86.9
3		30	0.75	87.8	86.9
4		40	0.50	88.8	86.9

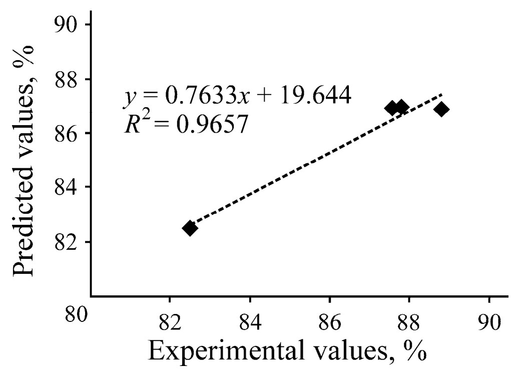

Fig. 10. Relationship between predicted and experimental values of silver dissolution.

Table 2 compares certain predicted fuzzy logic results to experimental values and a good correlation of results is shown. The leaching time was 4 h.

Thus, the fuzzy model can provide a credible estimation of silver dissolution with a success rate of 0.9657 (Fig. 10).

CONCLUSIONS

The hydrometallurgical method of post-leaching silver from a non-sulfide type ore flotation tailing leach residue in a copper–ammonium thiosulfate solution was examined. To validate the experimental results, MATLAB software was used to do a fuzzy logic prediction.

The post-leaching results indicated that a relatively high percentage (87.6%) of Ag was dissolved from the residue using a copper-ammonium thiosulfate solution under the following conditions: 30 g/l of ammonium thiosulfate, 0.5 g/l of $CuSO_4$, leaching temperature of 25 °C, and leaching time of 4 h. The experimental results suggested that the most critical parameters for silver dissolution were ammonium thiosulfate concentration, copper sulfate concentration, and leaching duration.

The fuzzy prediction method was used for the three most critical factors. The experimental values were found to be consistent with the theoretical values, and the R^2 value was determined to be 0.9657. As a result of the high R^2 value, the fuzzy logic method could be utilized to forecast silver dissolution from the residue.

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