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Research article

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The novelty of silver extraction by leaching in acetic acid with hydrogen peroxide as an organic alternative lixiviant for cyanide

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ABSTRACT

In this article, the dissolution kinetics of pure metallic silver in acetic acid with a hydrogen peroxide solution were carried out. The effects of stirring speed, acetic acid concentration, hydrogen peroxide concentration, and temperature were examined. The results show that increasing the stirring speed decomposes the hydrogen peroxide and negatively affects the dissolution rate of silver. In addition, an acetic acid concentration in the range of 0.25–1 M has a positive effect and a negative effect in the range of 1–3 M. A hydrogen peroxide concentration in the range of 0.5–2 M has a significantly positive effect on the dissolution rate, while it has a negative effect in the range of 2–3 M. The temperature in the range of 61.5–70 °C has a negative effect due to the decomposition of hydrogen peroxide. The shrinking core model was applied to all parameters to obtain the dissolution kinetics. The dissolution process of silver was controlled by the surface reaction-controlled shrinking core model, i.e. 1-(1-X)^{1/3} = kst, with an activation energy of 28.80 kJ/mol.

1. Introduction

Silver is considered one of the most valuable or noble metals and occurs together with copper, lead, zinc and always gold ores [1]. In mineral sulfides, silver occurs mainly together with galena (PbS), chalcopyrite (CuFeS₂), and sphalerite (ZnS). For a long time, cyanidation was the most common technique for extracting silver and gold from their ores [2]. Recently, silver can be extracted using ammonia thiosulfates, glycine, and glucose [3,4]. The chemical reaction of cyanidation is very complex and depends on the oxidation rate and mineral composition, as shown in Eq. (1) [5]. The most important parameters for the silver cyanidation reaction are cyanide ions (CN⁻), hydroxyl ions (OH⁻), and dissolved oxygen (DO) [6,7].

$$4Ag + 8N^{-} + O_2 + 2H_2O \rightarrow 4Ag(CN)_2^{-} + 4OH^{-}$$

(1)

Flotation is a physicochemical method often used to upgrade low-grade silver in sulfide ore before the pretreatment and leaching process [8–10]. The silver mineral can be recovered by selective ore bulk flotation depending on the amount of minerals of interest to the ore [11]. Sulfide minerals of silver exhibit slow leaching or extraction rates, are called refractory silver ore and require a high cyanide concentration and a long leaching time to increase the recovery [12]. The pretreatment of refractory ore with lead nitrate was studied, and it was observed that the recovery of silver increased when the lead nitrate was added directly to the leaching process [13].

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(3)



Fig. 1. (a) Pourbaix diagram, *Eh* vs. pH calculated at 25 °C, conc. of dissolved Ag species = $10^{-2} \text{ mol } L^{-1}$. (b) Pourbaix diagram in cyanide solution, E vs. pH calculated at 25 °C, conc. of dissolved Ag species = $10^{-4} \text{ mol } L^{-1}$ and $CN^- = 10^{-3} \text{ mol } L^{-1}$.



Fig. 2. The granular of metallic silver.

In addition, the results of hydrogen peroxide on the cyanidation rate showed that at high concentrations, hydrogen peroxide has a negative impact on the extraction rate due to the formation of a silver oxide layer on the silver particles based on pourbaix diagrams, as shown in Fig. 1(a) and (b) [6,14]. The increase in dissolved oxygen in the leaching solution improves the redox potential of the reaction, according to the Nernst equation [13,15,16].

Hydrogen peroxide increases the concentration of dissolved oxygen and the redox potential of the reaction, as shown in Eq. (2) and Eq. (3) [16,17].

$$Ag_s \rightarrow Ag^+ + e^- \quad E^0 = -0.796 \text{ V at } 25^{\circ}\text{C}$$
 (2)

$$H_2O_2 + 2e^- \rightarrow 2OH^- E^0 = 1.007 V$$

Extensive work on the kinetics of dissolution of metallic silver by cyanidation, nitric acid, thiosulfate, and thiourea was conducted [2,18,19]. The kinetics of silver with aqueous cyanide and oxygen solution were studied, and the process was controlled by a diffusion-controlled model with an activation energy of 10 kJ/mol at a temperature of 24–110 °C [20]. The effects of oxygen partial pressure, thiosulfate concentration, and temperature on the dissolution kinetics of silver leaching with sodium thiosulfate were studied. The results showed that the activation energy was 1.912 kJ/mol at a temperature in the range of 288–328 K [21]. Also, the dissolution kinetics of silver in precious metal scraps using HNO₃ were studied. The influence of stirring speed, particle size, temperature, and acid concentration on the dissolution rate was investigated. The reaction was controlled by the shrinking core model through internal diffusion, with an activation energy of 33.95 kJ/mol forAg-Au_{0.04}-Cu_{0.10} and 68.87 kJ/mol for Ag-Cu_{0.23} [22]. In addition, the dissolution kinetics of silver were studied using an alkaline sodium thiosulfate-potassium ferricyanide leaching system. The reaction was controlled by a diffusion process with an activation energy of 17.96 kJ/mol [23]. The reaction kinetics and mechanism of the reaction of silver with thiourea in alkaline media were studied, and the results showed that silver could be formed as Ag (OH)⁻⁴ in strongly alkaline media by dissolving the electrolysis of a silver foil anode in a 1.2 M NaOH solution can, as shown in

Fig. 1 [24]. The kinetics of silver dissolution in sodium thiosulfate solution were also studied. The reaction was controlled by a diffusion process with an activation energy of 3.15 kJ/mol [25]. Furthermore, the kinetics of metallic silver were studied using the O₂-thiosulfate system. The influence of temperature, stirring speed, oxygen partial pressure, thiosulfate concentration, and hydronium concentration was investigated. The activation energy was calculated to be 4.5 kJ/mol, and the kinetics were controlled by the mass transfer of oxygen at the solid-liquid interface [26].

Acetic acid (HAc) is used to extract metals such as lead, cadmium and nickel in hydrometallurgical applications [27,28]. This work investigates the kinetics of silver dissolution in acetic acid using the hydrogen peroxide system as an alternative lixiviant.

2. Materials and methods

2.1. Material

Granulated metallic silver (99.99 %) was used for the experiments in the study, as shown in Fig. 2. The sample was sieved, sorted, and then given a closed weight of 0.215 \pm 0.002 g in the range of -4.0 + 3.35 mm.

2.2. Methods

For the experimental tests, a beaker with a capacity of 1 L was used and then placed in the controlled water bath, and a Tefloncoated mechanical stirrer was also used for mixing. The effect of the stirring speed of (50–300 rpm), the temperature of (40–70.1 °C), the acetic acid concentration of (0.25–3 M) and the hydrogen peroxide concentration of (0.5–3 M) was studied. For each test, granular metallic silver was leached in a 0.5 L solution of H₂O₂/HAc for 330 min. An atomic absorption spectrophotometer (AAS) was used to analyse the dissolved silver. A sample of the leaching solution was taken at regular intervals, the actual volume was calculated and corrected using Eq. (4) [29]. All experimental tests were carried out using distilled water and high-quality reagents.

$$X_{M,i} = \frac{\left(V - \sum_{i=1}^{i-1} v_i\right) C_{M,i} + \sum_{i=1}^{i-1} V_i C_{M,i}}{m\left(\frac{c_m}{100}\right)}$$
(4)

where, *V* is the initial volume (mL) of the solution, v_i is the volume of the sample (mL) taken at each time, $C_{m,i}$ is the concentration of (Ag) in sample *i* (mg/L), m the initial mass of dry silver in g added to the leaching system and c_m is the concentration of M in the silver sample (wt.% dried solids).

3. Results and discussions

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Industrially, silver acetate can be produced by reacting silver carbonate with glacial acetic acid, as shown in Eq. (5) [30]. The solubility of silver acetate in water at 25 °C is 10.4 g/L [31].

$$2CH_3CO_2H + Ag_5CO_3 \rightarrow 2AgO_5CCH_3 + H_2O + CO_2$$
(5)

The mechanism of silver dissolution in an acetic acid-hydrogen peroxide system can be described in Eq. (6) and Eq. (8) [14,32]:

$$Ag \rightarrow Ag^+ + e^- \quad E_0 = -0.88 \text{ V at } 25^{\circ}\text{C}$$
 (6)

$$H_2O_2 + 2H^+ + 2e^- \rightarrow 2H_2O \quad E_0 = 1.77 \text{ V} \quad \text{at } 25^\circ C \tag{7}$$

Acetic acid is an organic acid with a low ionisation constant:

$$CH_3COOH \leftrightarrow H^+ + CH_3COO^- \quad K_a = 1.75 \times 10^{-5} \text{ at } 25^{\circ}C$$
(8)

Hydrogen peroxide can roll as a reducing agent as seen in Eq. 9

$$H_2O_2 \to O_2 + 2H^+ + 2e^- \tag{9}$$

Eq. (10) is the overall result of Eq. (7) and Eq. (9) and it can be written as follows:

$$2H_2O_2 \rightarrow O_2 + 2H_2O \tag{10}$$

in the decomposition of H_2O_2 , oxygen adsorbs on the metal surface and leads to electron transfer into the solution. The redox potential of 1.77 V of hydrogen peroxide in an acidic solution is quite enough to oxidize many metals [32]. Furthermore, hydrogen peroxide decomposes into the form of water and O_2 , as shown in Eq. (11) [33].

$$H_2O_{2(aq)} \rightarrow H_2O_{(aq)} + \frac{1}{2}O_2(g)$$
(11)

In this case, the dissolution of silver in an acetic acid-hydrogen peroxide system is an electrochemical process. It can be described by the following reactions, as shown in Eq. (12) and Eq. (13) (Liebermann., 1988):

(12)

(13)



Fig. 3. Effect of stirring speed on the silver dissolution at 60.1 °C, 1 M acetic acid and 1 M H₂O₂.



Fig. 4. Effect of acetic acid concentration on silver dissolution at 1 M H₂O₂, 50 rpm and 61.5 °C.

 $2Ag + 2CH_3COOH + H_2O_2 \rightarrow 2CH_3COOAg + 2H_2O$

 $CH_3COOAg_{(s)} \leftrightarrow Ag^+ + CH_3OO^- K_{sp} = 2.3 * 10^{-3} \text{ at } 25^{\circ}C$

3.1. The effect of stirring speed

The effect of stirring speed on the silver dissolution in the range of 50–300 rpm is shown in Fig. 3. The rate of dissolution of silver decreases as the stirring speed increases because hydrogen peroxide decomposes at high rates of stirring speed, as shown in Eq. (11). Furthermore, the result at 200 rpm is similar to the result at 300 rpm. Wadsworth (2000) investigated a study on the surface processes of silver and gold cyanidation. It was observed that the higher stirring speeds are expected to increase the rate of desorption of the peroxide ion from the metal surface, limiting the extent of catalytic decomposition [34]. However, Adebayo and Ipinmoroti (2003) [35] found that the negativity of the stirring speed is not related to the decomposition but to the contact of the pyrite particles with the peroxide.

3.2. The effect of acetic acid concentration

Fig. 4 shows the dissolution of silver as a function of acetic acid concentration in the range of 0.25–3 M. The results show that the silver extraction rate increases at an acetic acid concentration in the range of 0.25–1 M and decreases at a concentration above 1 M. As shown in Fig. 4, the extraction percentage at 3 M is almost the same as that at 0.5 M. The reason is attributed to the Eh potential of the



Fig. 5. The Ag_2O_3 layer on the metallic silver.



Fig. 6. Effect of H_2O_2 concentration on silver dissolution at 1 M acetic acid, 50 rpm and 61.5 °C.



Fig. 7. Effect of temperature on silver dissolution at 1 M acetic acid, 50 rpm and 1 M H_2O_2 .



Fig. 8. The variation in $1-(1-X)^{1/3}$ with time at various stirring speeds.



Fig. 9. Determination of the reaction order with respect to stirring speed.

reaction since the increase in an acetic acid concentration above 1 M leads to an increase in the Eh potential of the reaction to Eh > 1.5 V, and then a black layer of Ag₂O₃ appears on the surface of the silver particle. as shown in Fig. 5 and Pourbaix diagram in Fig. 1 [19, 36]. Xue and Osseo-Asare (1985) [37] studied the equilibrium relationship in the Au–CN–H₂O and Ag–CN–H₂O systems. It has been found that metallic gold cannot be oxidized by dissolved oxygen or strong acids at all pH values. However, metallic silver can only oxidize in the presence of a strong oxidizing agent.

3.3. Effect of hydrogen peroxide concentration

Fig. 6 shows the dissolution of silver as a function of hydrogen peroxide concentration in the range of 0.5-3 M. The results show that increasing the hydrogen peroxide concentration in the range of 0.5-2 M increases the dissolution of silver. However, at a concentration of 2–3 M, the dissolution rate decreases rapidly and is above 1.5 V due to the increasing reaction potential. This potential value leads to the passivation or precipitation of Ag₂O₃ on the surface of silver particle, as shown in Fig. 5. Similar results were also reported by Xue and Osseo-Asare (2001) [38] in alkaline media using cyanide.

3.4. Effect of temperature

The result in Fig. 7 shows the effect of temperature in the range of 40–61.5 $^{\circ}$ C on silver dissolution rate. The dissolution rate decreases after 30 min at a temperature above 66.5 $^{\circ}$ C, as hydrogen peroxide typically decomposes at a temperature above 60 $^{\circ}$ C [39, 40].



Fig. 10. The variation in $1-(1-X)^{1/3}$ with time at various acetic acid concentration.



Fig. 11. Determination of the reaction order with respect to acetic acid concentration.



Fig. 12. The variation in $1-(1-X)^{1/3}$ with time at various hydrogen peroxide concentration.



Fig. 13. Determination of the reaction order with respect to hydrogen peroxide concentration.



Fig. 14. The variation in $1-(1-X)^{1/3}$ with time at various values of temperature.

3.5. Kinetics of dissolution

The shrinking core model for the surface chemical reaction was applied to the results of stirring speed, acetic acid concentration, hydrogen peroxide concentration, and temperature effect.

$$k_s = 1 - (1 - x)^{1/3}$$

(14)

Where *X* is the fraction of dissolved silver, k_s is the apparent rate constant and *t* is the time [39].

Fig. 8 was linearised via Eq. (14), and then the ks values were determined for each stirring speed to obtain the slope of the line. The chemical reaction order was determined to be -0.78, as shown in Fig. 9.

The above procedure was also applied to the effect of acetic acid concentration, as shown in Fig. 10. There are two reaction orders for the concentration in the range of 0.25–1 M and the concentration in the range of 1–3 M to be 1.12 and 0.536, respectively, as shown in Fig. 11.

Fig. 12 presents the variation in 1-(1-X)^{1/3} with time at various hydrogen peroxide concentrations. The chemical reaction orders of 0.73 and -6.96 were determined for the hydrogen peroxide concentration in the range of 0.75–2 M and concentration in the range of 2–3 M, respectively, as shown in Fig. 13.

The variation of $1 \cdot (1 \cdot X)^{1/3}$ with time at different temperature values is presented in Fig. 14.

In the calculation of activation energy, the *ks* values of temperature were used with the temperature values *K* to draw the Arrhenius diagram as shown in Fig. 15. Since the hydrogen peroxide begins to decompose at a high temperature of more than 61.5 °C, the results of 66 and 70 were ignored. The activation energy was calculated to be 28.80 kJ/mol via Arrhenius's equation [$k = Ae^{-\frac{Ea}{RT}}$]. The



Fig. 15. Arrhenius plot of data presented.

activation energy of silver dissolution in the nitric acid solution was calculated to be 12.1 kJ/mol [39].

4. Conclusion

In this study, the dissolution of pure metallic silver in acetic acid with a hydrogen peroxide solution was investigated as a fundamental study. The effect of stirring speed, temperature, H_2O_2 , and acetic acid concentration on the dissolution rate of silver was examined to investigate the dissolution kinetics. The results show that at a high concentration of hydrogen peroxide or acetic acid, the dissolution rate decreases. The dissolution process is controlled by a chemical surface reaction model with an activation energy of 28.80 kJ/mol.

CRediT authorship contribution statement

Salih Aydoğan: Supervision, Conceptualization. Mahmoud Motasim: Writing – original draft, Writing – review & editing, Software, Resources, Conceptualization. Babiker Ali: Validation, Methodology.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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