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Kinetics of ultrasound-assisted silver leaching from sintering dust using thiourea

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Abstract: In the process of steel manufacture, up to tens of millions of tons of sintering dusts are produced annually in China, which contain noble metals such as Ag. Therefore, the recovery of Ag from sintering dust could be a potential economic and environmental activity. The present work attempts to use ultrasound for leaching silver from the water-leaching residue of sintering dust (WLR-SD) by acidic thiourea at atmospheric pressure. The effects of particle size, leaching temperature, and thiourea concentration were assessed on the leaching efficiency. Mineralogical changes of the WLR-SD were estimated using X-ray fluorescence, X-ray diffraction, and scanning electron microscope. The results have shown that under the conditions used, up to 94% silver could be leached. The leaching kinetics was estimated to follow the first-order kinetic model, and the activation energy was estimated to be 20.7 kJ/mol, confirming an internal diffusion-controlled leaching process. The order of the reaction with respect to the thiourea concentration was estimated to be 0.68.

Keywords: kinetics; silver; sintering dust; ultrasound-assisted leaching.

1 Introduction

One of the main pollution sources of iron-making process is the generation of large volumes of sintering dust, which are separated using cloth filter bag and electrostatic precipitators [1, 2]. The popular method adopted by steel manufacturers is to sinter dusts, which are fed back into the blast furnace to eliminate dust emission [3]. The sintering dusts are high in lead, zinc, alkali metal, and other valuable elements. The recycling of the sintering dust back to the blast furnace is known to affect steel quality as well as enhance corrosion, leading to increased manufacturing cost [4, 5]. An alternative method that is practiced industrially is solid waste pile up, which is a serious environmental concern. Recently, there is an increasing emphasis on recycling waste resources due to potential financial benefits and to overcome environmental concerns. Hence, sintering dust is a potential candidate as it possesses abundant amounts of valuable metals such as iron, potassium, lead, and some silver. A comprehensive research on the recovery of valuable metals is recently reported in China [6, 7]. However, the extraction of silver from the dusts has rarely been reported.

Silver is a trace element. The average content of silver in the earth's crust is just 1×10^{-5}%, which is only 20–30 times higher than gold, and about two-thirds of the silver resources are usually associated with nonferrous and noble metals [8]. Because of its unique electric, optic, and magnetic characteristics, silver has widespread application, which includes dental alloys, conductor, solder and brazing alloys, high-capacity silver-zinc and silver-cadmium batteries, contactor, and safety switch, besides its well-known use for jewelry and decorative purposes [9–11]. Hence, the present work considers the recovery of silver from sinter dust by using ultrasound-induced thiourea leaching.

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It is well known that cyanide leaching has been widely used to recycle silver from their ores for the past 100 years. A well-known equation describes the stoichiometry of the reactions as follows [12]:

$$4Ag+8CN^{-}+O_2+2H_2O=4Ag(CN)_2^{-}+4OH^{-}, \tag{1}$$

which is known to produce a large volume of cyanide wastewater, a serious health and safety executive (HSE) issue [13]. As an alternative, thiosulfate is a nontoxic and low-priced reagent; however, it demands a larger volume, rendering it commercially unfeasible [14, 15]. Thiocyanate is also considered as a potential leachant because of its stability in weak acid solution and low toxicity; nevertheless, the process demands using it at high concentrations [16]. To overcome the disadvantages associated with various solvents, thiourea is considered as a potential solvent because of its high reaction kinetics, low cost, and less environmental impact [17, 18].

Ultrasound-assisted leaching is well known to enhance hydrometallurgy, as it facilitates the release of metals in a short duration using acids at low concentration [19], at atmospheric pressure, and at room temperature [20]. It has been widely applied to the assisted extraction of valuable metals from rare or precious metal ores, electronic waste, and sludge waste, exhibiting good results [21–26]. Calle et al. [27] have used ultrasound to extract gold and silver from the environment with thiourea, and they have reported efficient removal at a shorter duration, with less harmful waste generation. Öncel et al. [11] have reported ultrasound-assisted leaching of silver from solid waste using sulfuric acid-thiourea solution with a recovery efficiency in excess of 99%. Sayana et al. [28] have reported a 20% increase in the TiO_2 recovery with ultrasound-assisted leaching of Ti from red mud with sulfuric acid.

The present work attempts to leach silver using high selective environmentally friendly thiourea from the water-leaching residue of sintering dust (WLR-SD). According to the literature [29], ferric ion was reported to be the most effective oxidant, applied along with sulfuric acid to adjust the pH level. The effects of the concentration of thiourea, the particle size, the temperature, and the duration of leaching were investigated on the leaching kinetics.

2 Materials and methods

2.1 Materials

The composition of the WLR-SD was determined by X-ray fluorescence for high content elements and inductively coupled plasma-atomic emission spectrometry (ICP-AES; Thermo Fisher, iCAP6300, USA) for low content elements, and the results are shown in Table 1. The crystalline phases of the samples were investigated by X-ray powder diffraction (Rigaku D/Max 2500/PC, Japan). As Figure 1 shows, hematite (Fe_2O_3), magnetite (Fe_3O_4), cotunnite ($PbCl_2$), laurionite (PbOHCl), anglesite ($PbSO_4$), quartz (SiO_2), and calcia (CaO) are the main crystals in the mineral phases of the sample. The scanning

Table 1: Chemical composition of WLR-SD (mass fraction, %).

Elements	Fe	Pb	Ca	Si	S	Cl	F	Mg	Al	Ag
Content %	37.15	17.74	4.62	2.51	2.06	1.97	1.40	0.78	0.73	688 g/t

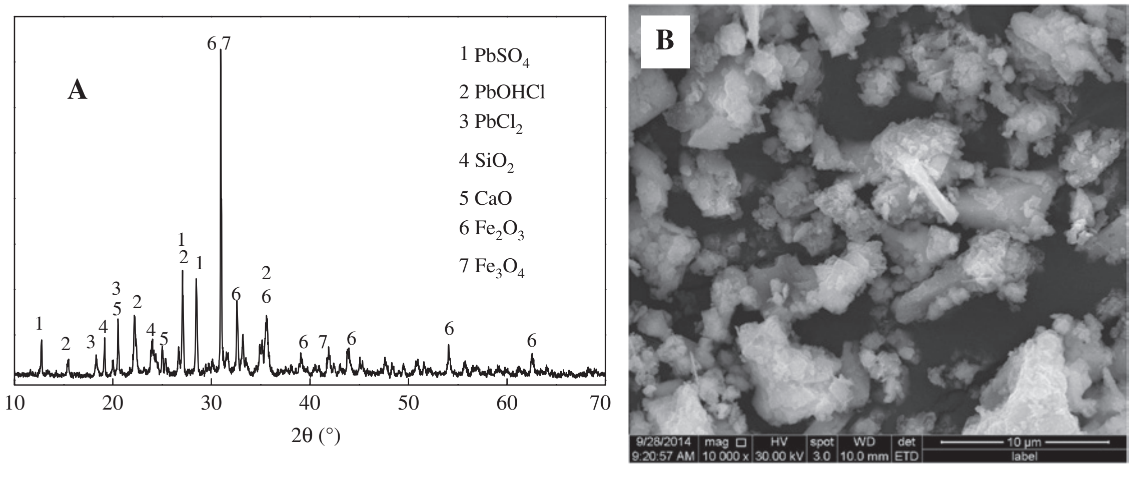

Figure 1: X-ray diffraction pattern (A) and scanning electron microscope image (B) of WLR-SD.

electron microscope (SEM) image of the WLR-SD was observed by an FEI Quanta650 SEM (USA) as shown in Figure 1B.

2.2 Equipment and procedure

The schematic illustration of the experimental setup is shown in Figure 2. The leaching of silver from WLR-SD was performed under atmospheric pressure, in a beaker of 1000 ml volume, which was placed on a thermostatically controlled water bath equipped with magnetic stirring, within a precision of ±1°C. Ultrasound was produced by an ultrasonic generator (Nanjing Han Zhou Technology Co., Ltd, China) placed inside the beaker through an immersed titanium probe, with a frequency of 20 kHz and a variable power of 0–1000 W. The pH level inside the beaker was measured by a calibrated PHS-3C pH meter (Shanghai REX Instrument Factory, China).

Prior to use, the sintering dust was leached with tap water at room temperature for 60 min at a pulp density of 200 g/l with a mechanical stirrer at 400 rpm to remove the soluble substances. After filtration, the residue was dried and sieved.

Fifty grams of the WLR-SD was added at one time to the agitated acidic thiourea solution (pH 1.5–2.0) of desired concentration once the temperature reached the set point. The mixture was stirred at 400 rpm, using 0.5 mol/l sulfuric acid for pH adjustment in the process. Air was bubbled beneath the probe tip by means of a ring distributor; its flow rate was adjusted by a flow meter. After the process of leaching, the filtrate and the filtration residue were analyzed for silver using ICP-AES. The leaching efficiency of silver was calculated using the following equation:

$$\text{Leaching efficiency (pct)} = (C_{Ag} \times 10^{-6} \times 400)/(\omega_{Ag} \times 50), \tag{2}$$

where C_{Ag} (μg/ml) is the time-dependent concentration of silver measured by the ICP-AES, ω_{Ag} is the percentage content of silver in the WLR-SD, 400 (ml) is the total volume of leaching solution, and 50 (g) is the weight of the WLR-SD added in the beaker.

All the experiments were performed with grains of the size 96–80 μm, except those relating to the assessment of the effect of particle size on the reaction kinetics. The ultrasonic power, solid/liquid ratio, magnetic stirring speed, and airflow rate were fixed at 200 W, 50/400 g/ml, 400 rpm, and 20 ml/min, respectively.

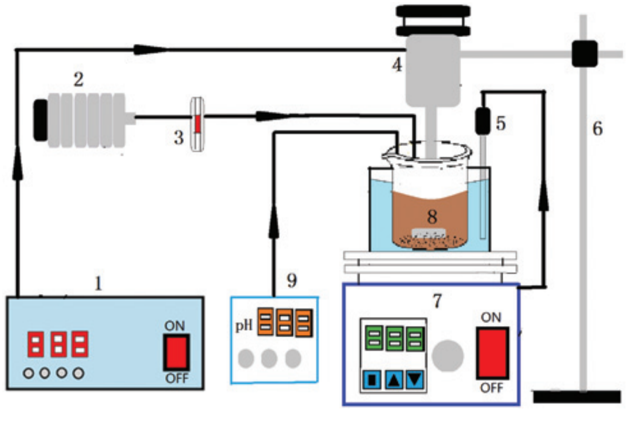

Figure 2: Schematic of experimental setup. (1) Ultrasound generator, (2) small-scale air compressor, (3) gas flowmeter, (4) ultrasound probe magnetic mixer, (5) temperature probe, (6) stand, (7) thermostatic bath, (8) magnetic stirrer, (9) pH meter.

3 Results and discussion

3.1 Chemical reaction

The reaction of silver in the acidic solutions of thiourea can be written as follows:

$$Ag + 3CS(NH_2)_2 + Fe^{3+} = Ag[CS(NH_2)_2]_3^{+} + Fe^{2+}. \tag{3}$$

In the previously mentioned reaction, the ferric iron was provided by the WLR-SD. The effects of variables such as the particle size, the leaching temperature, and the thiourea concentration on the leaching rate were assessed.

3.2 Effect of leaching parameters on silver recovery yield

To obtain the kinetic parameters for the solver leaching process, the effects of variables such as particle size, leaching temperature, and thiourea concentration on the leaching rate were assessed.

3.2.1 Effect of particle size

The effect of different fractions of particle sizes was assessed at a thiourea mass concentration of 12 g/l and a reaction temperature of 45°C, covering a leaching duration of 10–120 min, and the results are shown in Figure 3.

An increase in the leaching efficiency was observed with a decrease in the particle size. The lowest of the particle size resulted in a leaching efficiency in excess of 95%. A decrease in the particle size would increase the surface area; in turn, the interfacial area between the solids and the liquid would contribute to a faster leaching rate. In

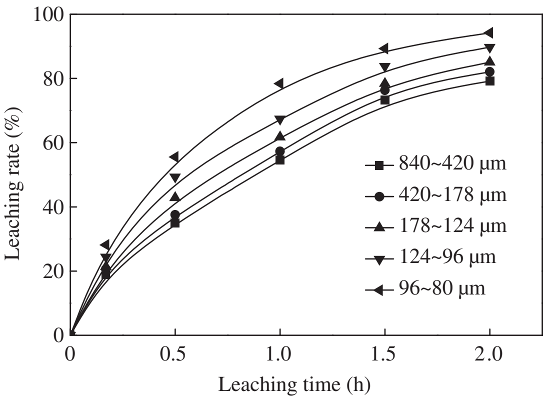

Figure 3: Effect of particle size on silver leaching efficiency.

addition, the rate of leaching would enhance because of the ultrasound cavitation contribution to the generation of fracture growth as well as to the reduction in external mass transfer resistance. Hence, a choice of particle size from the reaction kinetics point of view could be "the smaller, the better".

3.2.2 Effect of temperature

The effect of reaction temperature on the leaching efficiency of silver is shown in Figure 4, covering a temperature range of 25–45°C, with the thiourea mass concentration and the particle size fraction held constant at 12 g/l and 96–80 µm, respectively.

Figure 4 shows an increase in the leaching efficiency with an increase in the leaching temperature. An increase in the rate of reaction with an increase in the temperature is well known, and the experimental results concur with the known fact. An increase in temperature is expected to exponentially increase the rate constant of a reaction and, hence, the rate of reaction. Although the rate of leaching could be increased with an increase in temperature beyond 45°C, the temperature stability of the thiourea restricts higher leaching temperature as it decomposes to formamidine disulfide [18].

3.2.3 Effect of thiourea mass concentrations

The effect of thiourea mass concentration in the range of 4–12 g/l was assessed with a leaching temperature of 45°C, and the particle size was held constant at 96–80 µm.

Figure 5 shows an increase in leaching efficiency with an increase in the mass concentration of thiourea in acidic conditions. The increase in the leaching efficiency could be attributed to the increased rate of leaching due to the higher concentration of the thiourea. An increase in the rate of reaction with an increase in the concentration of the reactant is well known, and the present results concur well with the well-known fact. The order of the leaching reaction with respect to the thiourea concentration is estimated and presented in the later part of the paper.

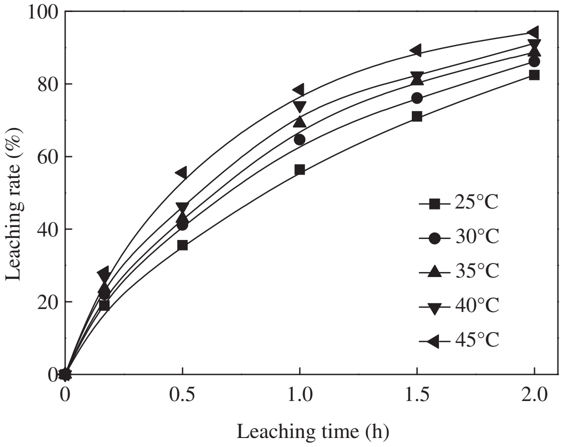

Figure 4: Effect of temperature on silver leaching efficiency.

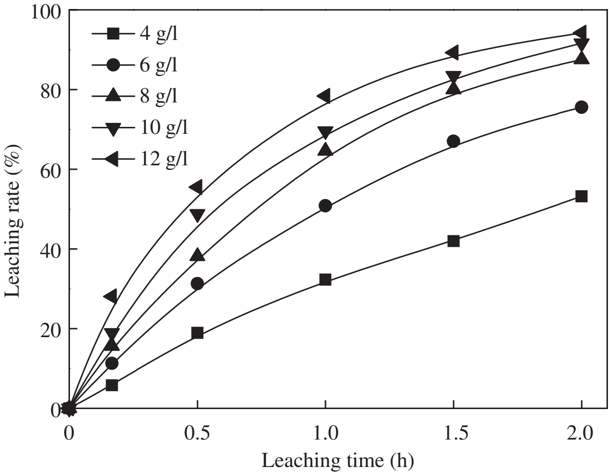

Figure 5: Effect of thiourea mass concentration on silver leaching efficiency.

3.3 Kinetics analysis

To determine the relationship between the silver recovery yield and the operating parameters, the kinetics equation is studied based on the previously mentioned data. Several models have been studied first to see their suitability for the data of this work. The models include $1\text{-}(1\text{-}x)^{1/3}$, $1\text{-}2x/3\text{-}(1\text{-}x)^{2/3}$. However, the results show that these models do not fit the experimental data well.

To find a better fit to the data of this work and to understand the leaching mechanism, Sharp's method of reduced half time was used to select the kinetic models [30, 31].

$$F(\alpha)=A(t/t_{0.5}), \tag{4}$$

where α is the degree of reaction, $t_{0.5}$ is the duration for 50% conversion, and constant A depends on a function $F(\alpha)$. It is possible to determine the values of the half-time reaction and the time for different degrees of leaching for each temperature and thiourea mass concentration. Therefore, an estimate of $(t/t_{0.5})$ for different kinetic models can be compared with the experimental results to identify the best kinetic function for linearization of the results [30, 31].

The experimental data in the present work are found to match closely with the following kinetic function:

$$-\ln(1-\alpha)=kt, \qquad (5)$$

where α is the degree of reaction, k is the apparent kinetic constant, and t is the leaching duration.

The plots of -ln(1-α) versus time under different leaching conditions are shown in Figures 6–8. The figures clearly indicate that the data fit satisfactorily with the kinetic model.

3.3.1 Calculation of activation energy

The activation energy of silver leaching from WLR-SD was estimated using the Arrhenius equation $k=A\exp(Ea/RT)$, using the rate constants estimated from the slopes of the lines in Figure 7. The values of rate constants at different reaction temperatures (k_t) are presented in Table 2.

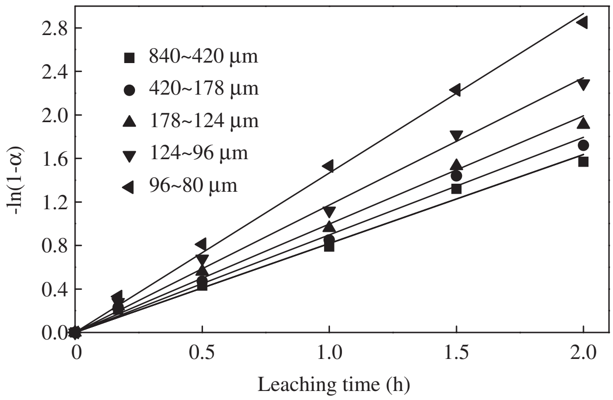

Figure 6: The variation in -ln(1-α) with time for different particle sizes.

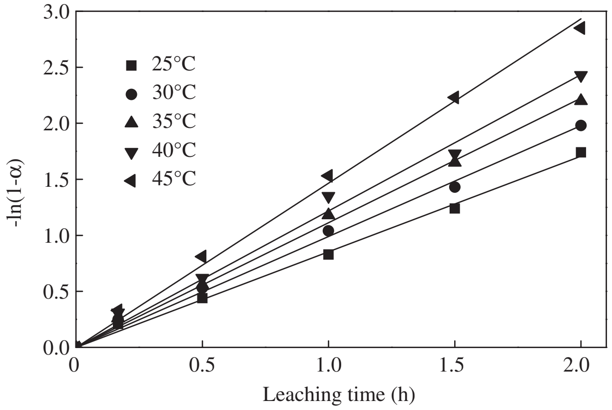

Figure 7: The variation in -ln(1-α) with time for different temperatures.

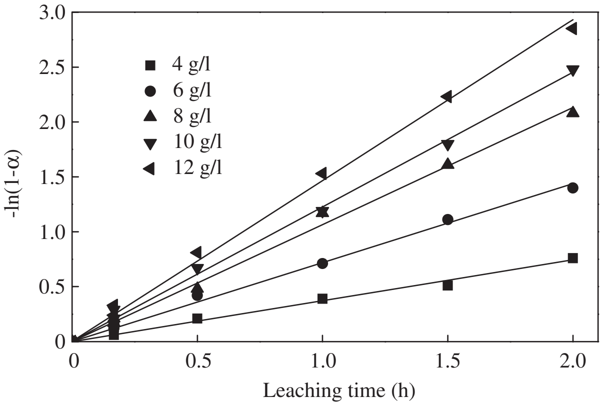

Figure 8: The variation in -ln(1-α) with time for different thiourea concentrations.

Table 2: Rate constants at different temperatures (k_t) and correlation coefficients.

T/K	k_t	R^2
298	0.8535	0.9983
303	0.9899	0.9977
308	1.113	0.9986
313	1.218	0.9956
318	1.466	0.9982

The plot of the linearized form of rate constant with the inverse of temperature shows a good fit (Figure 9).

The activation energy, E_a, and the constant A are estimated to be 20.7 kJ/mol and 8.18, respectively.

The leaching of silver in acidic thiourea solutions includes a heterogeneous reaction as represented by Eq. (3). In the leaching process, solid particles almost remain unchanged in size during the heterogeneous reaction because the sample contains a large amount of insolubles, which remain as a nonflaking residual called solid layer.

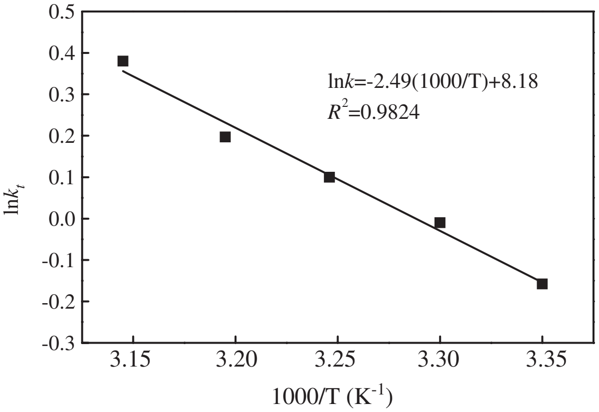

Figure 9: Arrhenius plot for the indium extraction from sphalerite concentrate.

Table 3: Rate constants at different thiourea mass concentrations (k_c) and correlation coefficients.

Concentration (g/l)	k_c	R^2
4	0.3721	0.9912
6	0.7191	0.9975
8	1.065	0.9977
10	1.227	0.9986
12	1.466	0.9982

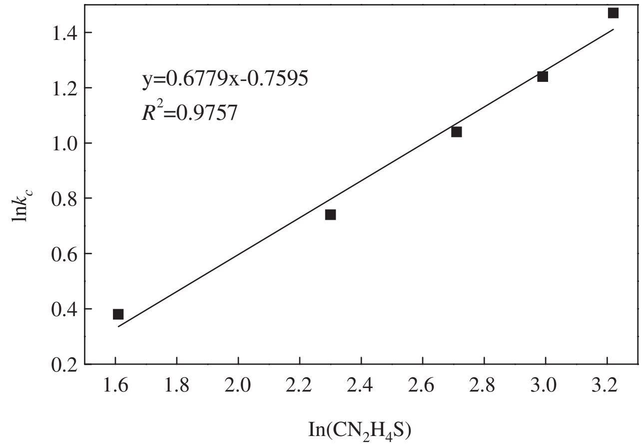

Figure 10: Estimation of reaction order with respect to thiourea concentration.

The experiment data indicated that the influence of thiourea concentration is more significant than the temperature on silver leaching rate. Diffusion velocity control equation can be represented as $\frac{dn}{dt}=\frac{D_{Ag}A}{\delta}(c_1\text{-}c_s)$ where $\frac{dn}{dt}$ is the diffusion velocity, D_{Ag} is the diffusion coefficient of $[Ag(SC(NH_2)_2)_3]^+$, n is the amount of diffusion substance, δ is the thickness of the diffusion layer, c_1 is the concentration of reactant in the solution, c_s is the concentration of reactant on the solid surface, and A is the surface area. Leaching reaction was greatly influenced by reactant concentration and thickness of diffusion layer. We can originally judge that the rate-determining step in the reaction of acidic thiourea leaching silver is diffusion in this work.

In addition, the energy value falls in the range of 8–30 kJ/mol for a typical inner diffusion-controlled process, further confirming that the leaching process is inner diffusion controlled [32]. Batric and Thom [33] have reported an activation energy of 22.6 kJ/mol for the leaching of silver with thiourea in the presence of ferric sulfate, indicating the presence of diffusion control, whereas Acma et al. [34] obtained an activation energy of 27 kJ/mol for the extraction of silver from the refractory type Gümüşköy-Pasa (Turkey) ores by thiourea leaching and stated that leaching is the diffusion of the Ag-thiourea complex in the thiourea through the reacted layer surrounding the unreacted core of the solution.

3.3.2 Reaction order

The rate constants estimated at different thiourea concentrations (k_c) using Eq. (5) for silver leaching are presented in Table 3.

The correlation coefficient (R^2) shows the model in good agreement with the experimental data. To estimate the order of reaction with respect to thiourea concentration, a linearized plot of concentration of thiourea versus the rate constant was plotted and is shown in Figure 8. The R^2 value presents the goodness of the fit and the appropriateness of the estimated order of reaction. As shown in Figure 5, the results on the effect of thiourea concentration were applied to the selected kinetic model. To create the plot of $\ln k_c$ versus ln $[CN_2H_4S]$ in Figure 10, the reaction order for silver with thiourea concentration is 0.68.

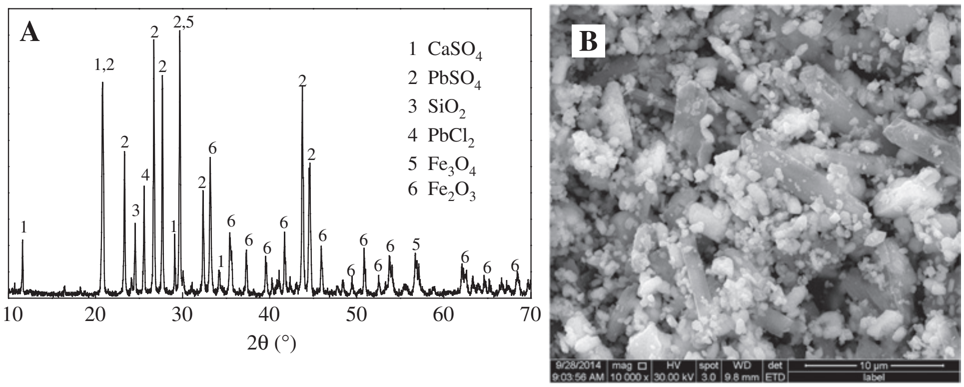

Figure 11: X-ray diffraction pattern (A) and scanning electron microscope image (B) of leached WLR-SD.

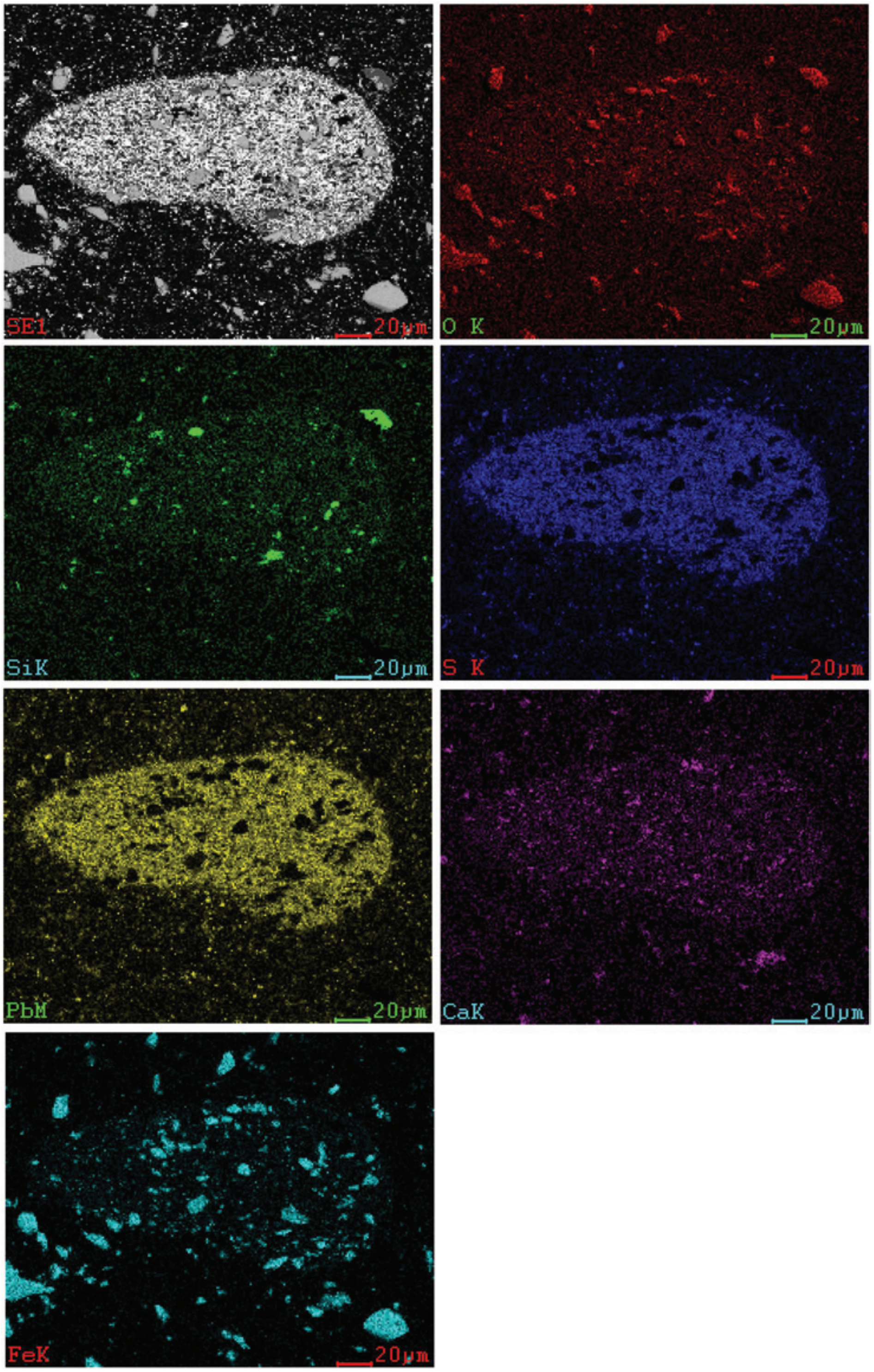

Figure 12: SE image of WLR-SD leached residues and along with the corresponding O, Si, S, Pb, Ca, and Fe maps.

3.4 Mineralogy and morphology of the residues

The X-ray diffraction patterns (Figure 11A) of the leaching residue show that the phases are $CaSO_4$, $PbSO_4$, $PbCl_2$, SiO_2, Fe_3O_4, and Fe_2O_3. Compared with those in Figure 1, Pb(OH)Cl and CaO are converted into $PbCl_2$ and $CaSO_4$, respectively.

As shown in Figure 11B, after the leaching, the sample is distinguished by its smooth surface because of the

Table 4: Compositions of leached WLR-SD.

Elements	Fe	Pb	S	Si	Ca	Cl	F	Mg	Al	Ag
Content %	31.23	25.86	5.75	1.97	2.35	0.18	–	0.32	0.33	41 g/t

cleanup activity of the solid surface by the ultrasonic radiation.

In particular, backscattered micrographs together with the corresponding O, Si, S, Pb, Ca, and Fe maps are reported in Figure 12 for the leached residue sample. The maps revealed that iron and, more important, lead are significantly concentrated inside them. The result is consistent with the X-ray analysis and the ICP-AES analysis of the leaching residue, as shown in Table 4.

The result indicated that the process of leaching had significantly lowered the Ag content in the residue. In addition, a small amount of Fe is also dissolved into the solution by leaching.

3.5 Mechanisms analysis

In this study, the acidic thiourea extraction augmented by ultrasound was investigated to assess the extraction efficiency of silver from WLR-SD. Thiourea is potentially capable of forming coordination bonds with silver through the lone pairs of electron on nitrogen and sulfur atoms. It can greatly reduce the silver oxidation reduction potential, making the silver easy to oxidize and dissolve into the solution. It was clear that both redox reaction and acoustic cavitation occurred during the extraction and were important factors affecting silver extraction.

The leaching process in this experiment contains three chemical reactions, and the last two reactions were taking place circularly. In the early stages of the first reaction, a small quantity of Fe_2O_3 existing in experimental sample is dissolved at pH 1.5–2 sulfuric acid solution to liberate Fe^{3+}, as described in Eq. (6):

$$Fe_2O_3+3H_2SO_4=Fe_2(SO4)_3+3H_2O. \tag{6}$$

The second reaction shows that by using ferric sulfate as an oxidant, thiourea ($SC(NH_2)_2$, or Tu) dissolves in the acid solution to form a relatively stable aqueous species, which react with the silver-bearing ion to produce a stable cationic complex, as described in Eq. (7). This key reaction is more important as it determines the extraction efficiency.

$$Ag+3SC(NH_2)_2+Fe^{3+}=Ag(SC(NH_2)_2)_3^{+}+Fe^{2+}. \tag{7}$$

It is well known that in the aqueous medium, ultrasound produces OH radical, which is a strong oxidant necessary for the oxidation of the silver-bearing phase existing in the sample. Dissolved O_2 transferred from air bubbles during the sonication participates in the production of OH radicals, and the OH radicals combine to form H_2O_2. Thus, the ferrous iron in Eq. (7) is oxidized to ferric iron, and promoting Eq. (7) to continue, the last reaction can be described in Eq. (8):

$$2Fe^{2+}+H_2O_2+2H^{+}=2Fe^{3+}+2H_2O. \tag{8}$$

The process of silver liberation by ultrasound is illustrated in Figure 13. The large amount of cavitation bubbles produced collapse and imploded instantaneously, generating localized temperatures as high as 5000°F, pressure of 100 MPa, and intense shear stress, which damage the solid surface and facilitate the removal of the passive film formation through chemical reactions [24, 26]. In addition, the ultrasound can remove the adsorbed impurities, which exist in microcrack gap so that the mineral surface can maintain high activity. The removal of silver attached to the surface of the silver-bearing particles results in increased contact area, which facilitates the increased rate of leaching and hence a higher leaching efficiency.

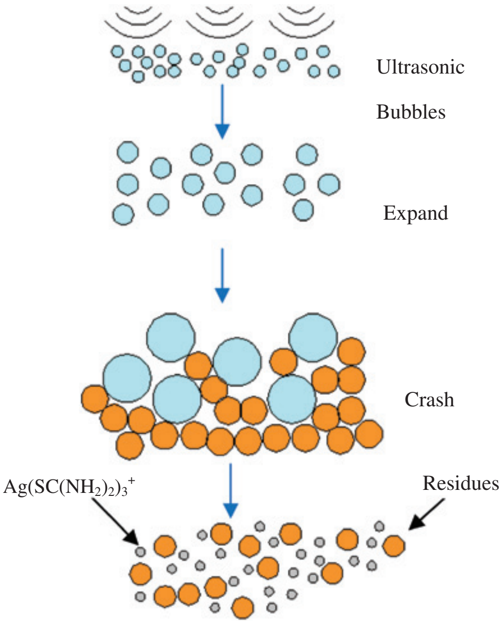

Figure 13: Sketches of mechanism of ultrasound-assisted leaching of Ag from WLR-SD.

4 Conclusions

The following conclusions could be derived for the ultrasound-augmented leaching of WLR-SD using thiourea.

The leaching efficiency was found to increase with an increase in the duration of leaching. The rate of extraction was found to increase with a decrease in the particle size, temperature, and concentration of the thiourea, resulting in decreased leaching efficiency. A maximum leaching efficiency of 94.2% could be achieved at the following combination of the experimental parameters: particle size, 96–80 µm; thiourea mass concentration, 12 g/l; and leaching duration at 45°C, 120 min.

The kinetics of leaching was found to follow the first-order kinetics, and the rate constant was found to increase with the increase in the leaching temperature and the concentration of the thiourea. The activation energy was estimated to be 22.6 kJ/mol, confirming the leaching to be an internal diffusion-controlled process. The rate of reaction with respect to thiourea mass concentration was estimated to be 0.68.

The ultrasound-augmented leaching of silver using thiourea confirmed better leaching efficiency at a faster rate, encouraging commercial adoption.

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