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## **Synopsis**

Alkaline sulphide leaching was adopted to extract tellurium from tellurium-bearing gold concentrate for the first time. The tellurium leaching kinetics were studied. The influence of average particle size, stirring speed, leaching temperature,  $Na_2S$  concentration, and NaOH concentration on the tellurium extraction were investigated. It was found that tellurium extraction could reach 79.14% under the optimum conditions: 17.77  $\mu$ m average particle size, 500 r/min stirring speed, 80 g/L  $Na_2S$  concentration, 30g/L NaOH concentration, 80°C leaching temperature, 4:1 liquid to solid ratio, and 180 minutes leaching time. The interface transfer and diffusion through the product layer or insoluble substance layer were the rate-controlling steps. The leaching kinetics equation was established, and the activation energy was 34.384 kJ/mol.

#### Keywords

Gold concentrate; tellurium leaching; alkaline suphide leaching; kinetics.

## Introduction

Tellurium, a scarce and valuable element with relatively low abundance of 0.001–0.005 g/t in the Earth's crust (Kavlak and Graedel, 2013; Makuei and Senanayake, 2018), is widely used in the electronics industry, metallurgy, communications, aerospace, energy, medicine, and other fields. The bismuth or antimony compounds of tellurium are excellent refrigerating materials which can be employed in radars and underwater missiles (Siciliano et al., 2009). Lead and bismuth tellurides are often used to make photosensitive materials (Tsiulyanu et al., 2005). The cadmium telluride photovoltaic panel is one of the most common commercially available solar panels (Bhandari et al., 2015; Fadaam et al., 2019). At present the market demand for tellurium continues to increase year by year (Candelise et al., 2012; Zhong et al., 2018), but annual tellurium production is far behind the market demand (Yu et al., 2018).

Currently, about 90% of tellurium production is from copper anode slimes from the electrolytic refining of blister copper, and the remainder as a by-product of bismuth and lead processing (Makuei and Senanayake, 2018). Due to heavy losses of tellurium during mineral processing and smelting, the current routes for tellurium production as a metallurgical by-product cannot meet the demand. Therefore, the direct recovery of tellurium from tellurium-bearing ores has attracted increasing attention. Since primary tellurium deposits are very rare in nature, current research is focused mainly on the extraction of tellurium from daphyllite ( $Bi_2Te_2S$ ) (Jiang, 2000; Lei and Xie, 2012). However, there are few studies on the extraction of tellurium from other tellurium-bearing ores, and even fewer on gold-telluride ores.

The gold-tellurium series minerals, second only to the gold-silver series, are important carriers of gold and silver. Among the 27 known types of gold-bearing minerals exploited, there are 11 gold-telluride minerals, including calaverite and petzite (Chen *et al.*, 1999). The leaching rates of gold and silver in tellurium-bearing gold ore are low because telluride is difficult to dissolve in cyanide media and the TeO<sub>2</sub> formed by the dissolution forms a layer on the gold surface, preventing the gold from contacting with the lixiviant. Tellurium leaching prior to cyanidation of gold and silver can not only effectively recover tellurium from this type of ore, but also improve the leaching rate of gold and silver (Ellis and Deschênes, 2016). Therefore, it is necessary to give more attention to the direct extraction of tellurium from tellurium-bearing gold ores.

A range of techniques, including sulphation roasting (Xu, Li, and Guo, 2014), soda roasting (Chen and Li, 2008), pressure sulphate leaching (Zhang, Wang, and Peng, 2007), pressure alkaline leaching (Fan *et al.*, 2013), and bioleaching (Rajwade and Paknikar, 2003) have been reported for recovering

tellurium from metallurgical by-products. However, these techniques have many disadvantages, such as harsh leaching conditions, excessive consumption of reagents, and serious corrosion of equipment. The development of a reasonable and efficient method to extract tellurium is therefore required. Guo *et al.*, (2017) used alkaline sulphide leaching to recover tellurium from high-tellurium materials obtained from the basic refining of crude bismuth, and tellurium was enriched from 11.60% to 91.24%. Additionally, the alkaline sulphide leaching method can recover arsenic and antimony (Awe *et al.*, 2013; Curreli *et al.*, 2009; Li *et al.*, 2011). However, little attention has been paid to using the method to recover tellurium from tellurium-bearing gold ores.

In this work, tellurium was extracted directly from tellurium-bearing gold flotation concentrate by alkaline sulphide leaching for the first time. The effects of different leaching conditions on the leaching efficiency of tellurium were investigated, and the kinetics of tellurium leaching are discussed on this basis. This study will provide a technological and theoretical basis for the efficient extraction of tellurium from tellurium-bearing gold ores.

## **Experimental**

#### Materials

The tellurium-bearing gold flotation concentrate was obtained from a tellurium-type gold mine in Qinling Mountains, Shannxi Province, China. Table I shows the chemical composition of the samples and Table II shows the main mineral content. The distribution of major elements in the minerals is shown in Table III.

	Table I  Chemical composition of tellurium-bearing gold concentrate (g/t)					
Au	Ag	Те	Pb (%)			
89.30	93.16	243.72	6.80			

Table II					
Main minerals in tellurium-bearing gold concentrate (%)					
Gold	Calaverite	Petzite	Hessite	Altaite	Pyrite
0.03	0.04	0.12	0.02	0.08	76.05
Chalcopyrite	Bornite	Covellite	Sphalerite	Pyrrhotite	Galena
0.43	0.01	0.03	0.02	1.45	0.21
Potassium feldspar	Plagioclase	Calcite	Dolomite	Rutile	Muscovite
1.02	0.15	0.35	0.09	0.37	10.01

Table III  Distribution of major elements between minerals				
Mineral	Te (%)	Au (%)	Ag (%)	Pb (%)
Petzite	45.00	88.83	89.48	0.00
Altaite	46.43	0.00	0.00	0.21
Hessite	6.93	0.00	9.19	0.00
Calaverite	1.63	3.22	0.00	0.00
Gold	0.00	7.95	1.33	0.00
Galena	0.00	0.00	0.00	99.79

Table II shows that the main metal sulphide minerals are pyrite, pyrrhotite, chalcopyrite, and galena whereas the main nonmetallic minerals are muscovite, quartz, and potassium feldspar. Table III shows that most of the tellurium is contained in the petzite and altaite, and a small amount in the hessite and calaverite.

## Experimental procedure

The tellurium samples were ground in a laboratory planetary ball mill. Particle size distribution analysis of the ground samples was performed using a Malvern laser particle-size analyser (Mastersize 2000). Five size fractions with volumetric average particle sizes of 56.58, 44.50, 28.67, 17.77, and 10.23 µm were obtained to study the effect of particle size on tellurium recovery. The PSDs of these fractions are shown in Figure 1 (a, b, c, d, and e respectively).

The leaching experiments were carried out in a 500 mL four-necked round-bottomed glass reactor fitted with a digitally controlled mechanical stirrer, condenser tubes, and mercury thermometers. Na<sub>2</sub>S-9H<sub>2</sub>O and NaOH of analytical grade and deionized water were used throughout the experiments. The leaching solution was prepared by dissolving sodium sulphide and sodium hydroxide in deionized water. The lixiviant (200 mL) was first added to the reactor, and the reactor was heated to the desired temperature in a superthermostatic water bath. Ground sample (50 g) was added when the desired stirring rate was reached. Samples of the leaching solution (2 mL) were taken at different time intervals, filtered, and submitted for tellurium analysis. The sample loss was compensated by adding 2 mL of fresh leaching solution. After leaching, the slurry was filtered and the residue was washed, dried, and weighed. The contents of main minerals and distribution of major elements in the tellurium samples were analysed by mineral liberation analyser (MLA650, FEI, USA). Tellurium in the solution was determined by ICP-AES (PS-6, Baird Corp, USA).

## Results and discussion

## Alkaline sulphide leaching of tellurium

## The effect of average particle size

Samples with average particle sizes of 57.58, 44.52, 28.66, 17.77, and 10.23 µm were leached in a solution containing 80 g/L Na<sub>2</sub>S plus 30 g/L NaOH at 80°C using a stirring speed of 500 r/min. The results (Figure 2) show that the leaching rate increases with decreasing particle size. A tellurium extraction of 58.56% was obtained for the 57.58 µm size fraction after leaching for 180 minutes, while 75.77% was obtained for the 17.77 µm size fraction. This could be caused by the increase in specific surface area with the decrease in particle size (Xiao *et al.*, 2019; Zheng and Chen, 2014a), so that the mass transfer process is enhanced. In addition, solid particles are also activated during grinding and the occluded tellurium is exposed, enhancing contact with the lixiviant. Considering the grinding energy consumption and subsequent solid-liquid separation process, 17.77 µm is recommended for the average mineral particle size.

## The effect of stirring speed

The effect of stirring speed on tellurium leaching was studied using speeds from 200 to 600 r/min. Other conditions were the same as those in the previous section. As shown in Figure 3, the reaction rate was very fast in the first 30 minutes at any

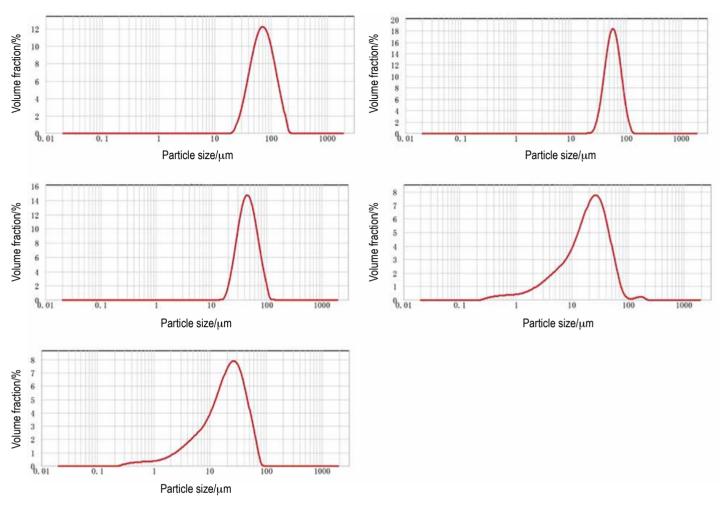


Figure 1 – Particle size distributions of the size fractions. (a) 56.58 μm, (b) 44.50 μm, (c) 28.67 μm, (d) 17.77 μm, (e) 10.23 μm

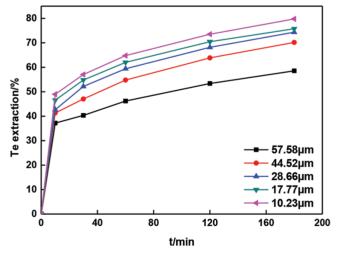


Figure 2—The effect of average particle size on tellurium extraction (stirring speed 500 r/min,  $\rm Na_2S$  concentration 80 g/L, NaOH concentration 30 g/L, leaching temperature 80°C)

stirring speed. After leaching for 30 minutes, tellurium extraction continued to increase, but the rate of extraction gradually slowed. When the stirring speed was increased from 200 r/min to 600 r/min, the increase of tellurium extraction was only 7.45–11.89%. In the solid-liquid reaction process, when liquid film diffusion is

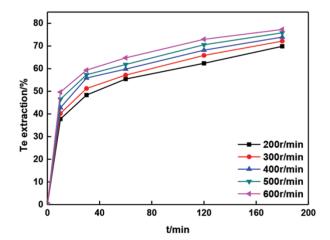


Figure 3—The effect of stirring speed on tellurium extraction (average particle size 17.77  $\mu$ m, Na<sub>2</sub>S concentration 80 g/L, NaOH concentration 30 g/L, leaching temperature 80°C)

the controlling step, the stirring speed has a great influence on the reaction rate (Tavakoli and Dreisinger, 2014). Therefore, the leaching of tellurium is probably not controlled by the external diffusion. A stirring speed of 500 r/min was chosen for the following experiments.

## The effect of leaching temperature

The effect of leaching temperature on tellurium extraction was investigated in the range of 20–90°C. Figure 4 shows the significant effect of temperature on the leaching kinetics. The tellurium extraction increased from 35.60% to 78.23% as the temperature was increased from 20°C to 90°C. This is expected from the exponential dependence of the rate constant in the Arrhenius equation (Zheng and Chen, 2014b). Furthermore, the energy available for atomic and molecular collisions increases with increasing temperature (Rao *et al.*, 2015). Only a minimal increase in extraction was observed at temperatures higher than 80°C, therefore 80°C was selected as the optimum leaching temperature.

# The effect of Na<sub>2</sub>S concentration

The effect of  $Na_2S$  concentration on tellurium leaching was determined using  $Na_2S$  concentrations of 20, 40, 60, 80, and 100 g/L. Figure 5 shows that the tellurium extraction increased rapidly from approximately 56.36% to 79.43% with an increase in  $Na_2S$  concentration from 20 g/L to 100 g/L, indicating that

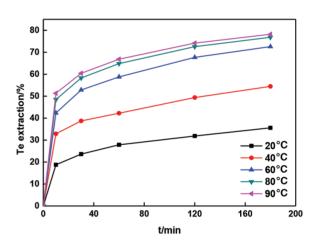


Figure 4—The effect of leaching temperature on tellurium extraction (stirring speed 500 r/min, average particle size 17.77  $\mu m$ , Na<sub>2</sub>S concentration 80 g/L, NaOH concentration 30 g/L)

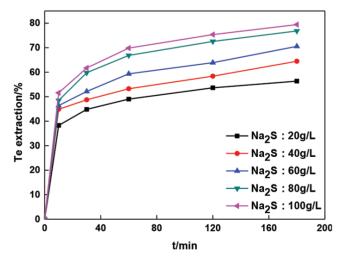


Figure 5—The effect of Na $_2$ S concentration on tellurium extraction (stirring speed 500 r/min, average particle size 17.77  $\mu$ m, NaOH concentration 30 g/L, leaching temperature 80°C)

Na<sub>2</sub>S concentration has a great influence on tellurium leaching. Tellurium is sulphurophilic. An increase in Na<sub>2</sub>S concentration increases the activity of sulphur ions, and then tellurium dissolves in the Na<sub>2</sub>S solution and is transformed to soluble thiotellurate (TeS<sub>4</sub><sup>2-</sup>) (Guo *et al.*, 2017), contributing to the enhanced reaction between tellurium and sodium sulphide and thus improving the leaching rate. Based on these results, 80 g/L was considered to be the optimum Na<sub>2</sub>S concentration.

## The effect of NaOH concentration

The effect of NaOH concentration on the leaching rate of tellurium was studied at NaOH concentrations of 10, 20, 30, 40, and 50 g/L (Figure 6). It can be seen that tellurium extraction increased as NaOH concentration increased, but only slightly, by 4.40–6.33% as the NaOH concentration increased from 10 g/L to 50 g/L. NaOH concentration has no marked influence on tellurium leaching, and 30 g/L was therefore selected as the optimum NaOH concentration.

Based on these results, the optimum leaching conditions were determined as 500 r/min stirring speed, 17.77  $\mu m$  average particle size, 80 g/L Na\_2S, 30 g/L NaOH, 80°C leaching temperature, a L/S ratio of 4:1, and 180 minutes leaching time. Under these optimum conditions, 79.14% of the tellurium was extracted into solution. The result indicates that alkaline sulphide is an effective lixiviant for tellurium dissolution from tellurium-bearing gold ores. In order to further enhance the leaching process, it is necessary to do some kinetic analysis to identify the rate-controlling steps in the leaching process.

## Kinetic analysis

#### Leaching kinetics

Table IV shows the chemical composition of the leach residue. As can be seen by comparing with Table I, the grade changes for

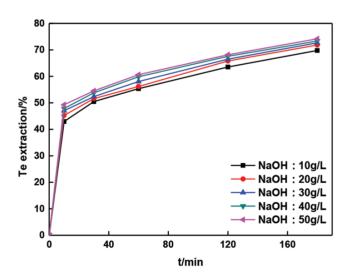


Figure 6—The effect of NaOH concentration on tellurium extraction (stirring speed 500 r/min, average particle size 17.77  $\mu$ m, Na<sub>2</sub>S concentration 80 g/L, leaching temperature 80°C)

Table IV  Chemical composition of leach residue			
Au (g/t)	Ag (g/t)	Te (g/t)	Pb (%)
88.75	92.14	50.84	6.21

gold and silver are very small, indicating that during the direct leaching of tellurium from gold concentrate very little dissolution of gold occurs, and there will therefore be no effect on the subsequent recovery of Au and Ag. Tellurium in gold flotation concentrate occurs mainly in petzite and altaite. During leaching, tellurium is dissolved mainly from petzite and altaite, and gold remains undissolved and locked in the mineral particles in some form. Therefore, the leaching of tellurium from the concentrate is a multiphase liquid-solid reaction. The rate-controlling processes include surface chemical reaction, film diffusion through a fluid film, and film diffusion through the product layer with the shrinking core model (Hua, 2004). A new model, proposed by Dickinson and Heal (1999), suggests that both the interface transfer and diffusion through the product layer or insoluble substance layer can affect the reaction rate. It is also used to explain the rate-controlling steps in the leaching process (Bingöl, Canbazoğlu, and Aydoğan, 2005; Rao et al., 2015). The leaching

Table V Kinetic models	
Rate-controlling step	Leaching rate equation*
Film diffusion through the fluid film	$1-(1-\alpha)^{2/3} = kt$
Film diffusion through the product layer	$1-2/3\alpha-(1-\alpha)^{2/3}=kt$
Surface chemical reaction	$1-(1-\alpha)^{1/3} = kt$
Both interface transfer and diffusion through the product layer	$^{1}/_{3}\ln(1-\alpha)-1+(1-a)^{-1/3}=kt$

 $<sup>^{\</sup>star}$  Here,  $\alpha$  is the leaching rate of tellurium, t is the leaching time (min), and k is the apparent rate constant (min-1)

rate equations for different rate-controlling processes are shown in Table V.

## Rate-controlling processes

Based on the experimental data at different leaching temperatures, the leaching kinetics of tellurium samples are fitted by the kinetic models in Table V. The kinetic model that gives the best-fitting results for the experimental data is defined as the rate-limiting step (Figure 7).

Table VI shows the correlation coefficients (R²) of the fitted data from Figure 7. It can be found that the R² value of  ${}^{1}/_{3}$   $\ln(1-\alpha)-1+(1-\alpha)^{-1/3}$  vs. t is closest to 1.0, indicating that  ${}^{1}/_{3}$   $\ln(1-\alpha)-1+(1-\alpha)^{-1/3}$  is the best model to simulate the leaching kinetics of tellurium. This observation confirms that the kinetics of leaching tellurium with alkaline sulphide solution is controlled by both interface transfer and diffusion across the product layer.

Additionally, the leaching rate is closely related to the activation energy, which can be calculated by the Arrhenius equation ( $\ln k = -E / RT + A$ ). Based on the Arrhenius equation,  $\ln k$  is plotted *versus* 1/T using the values of  $\ln k$  and 1/T in Figure 7d, as shown in Figure 8. The data in Figure 8 is in an approximate straight line. The values of E and E are calculated to be 34.384 kJ/mol and 4.414, respectively. The activation energy of a diffusion-controlled process is typically from 4 to 12 kJ/mol, while that of a chemically controlled process is usually larger than 40 kJ/mol (Abdel-Aa, 2000; Habashi, 1969). Therefore, an activation energy of 34.384 kJ/mol demonstrates the tellurium leaching in alkaline sulphide solution is controlled by both interface transfer and diffusion through the product layer.

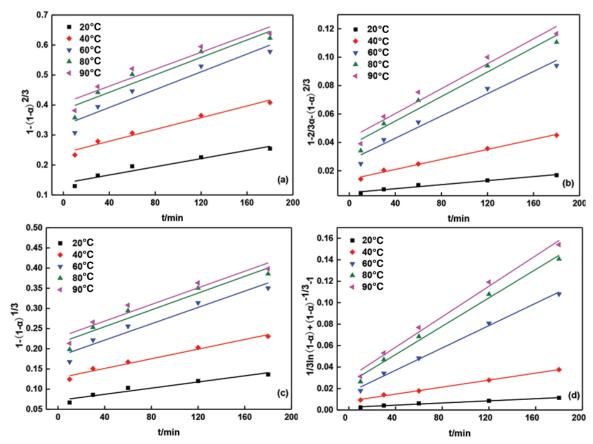


Figure 7-Different kinetic models versus time at different leaching temperatures

Table VI
Correlation coefficient values of regression equations

R <sup>2</sup>			
1- (1-a) <sup>2/3</sup>	1-2/ 3α- (1-α) <sup>2/3</sup>	1- (1-\alpha)1/3	1/3 ln(1-α)- 1+(1-α) <sup>1/3</sup>
0.9145	0.9677	0.9218	0.9797
0.9625	0.9899	0.9700	0.9964
0.9046	0.9628	0.9277	0.9934
0.8887	0.9473	0.9154	0.9891
0.8913	0.9460	0.9176	0.9886
	0.9145 0.9625 0.9046 0.8887	$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	$\begin{array}{c ccccccccccccccccccccccccccccccccccc$

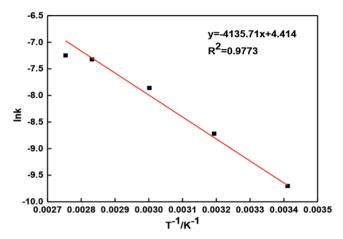


Figure 8-Arrhenius plots for tellurium leaching

## Leaching kinetics equation

The dependence of the rate constant on the stirring speed, average particle size, Na<sub>2</sub>S concentration, NaOH concentration, and leaching temperature may be expressed as

$$k = k_0 C^a D^b F^c G^d \exp(-E/RT)$$
 [1]

where  $k_0$  is the frequency factor, E is the apparent activation energy, R is the gas constant, T is the leaching temperature, C is the stirring speed, D is the average particle size, E is the Na<sub>2</sub>S concentration, E is the NaOH concentration, and E, E, and E, are the constants (Demirkiran and Künkül, 2007).

It can be seen from Figures 8–11 that  $^1/_3 \ln(1-\alpha)-1+(1-\alpha)^{-1/3}$  is related to average particle size, stirring speed, Na<sub>2</sub>S concentration, and NaOH concentration according to the experimental data in Figures 2, 3, 5, and 6.

The slopes of the lines in Figures 9–12 represent the rate constant. The value of  $\ln k$  can be calculated from these k values. The values of a, b, c, and d in Equation [1] are determined by linear regression in Figures 13–16: a = 0.3843, b = -0.5955, c = 1.0064, d = 0.1465. By combining the Arrhenius equation and Equation [1], the following expression is obtained:

$$A = \ln(k_0 C^a D^b F^c G^d)$$
 [2]

Based on Equation [2], the value of  $k_0$  is calculated as 1.1854. The final leaching kinetics equation is

$$1/3\ln(1-\alpha) - 1 + (1-\alpha)^{-1/3} = 1.1854$$

$$C^{0.3843}D^{-0.5955}F^{1.0064}G^{0.1465}\exp\left(\frac{-34384}{RT}\right)t$$
[3]

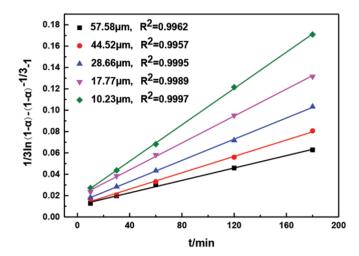


Figure 9—1/3  $\ln(1-\alpha)$  – 1+(1- $\alpha$ )-1/3 versus time at different average particle sizes

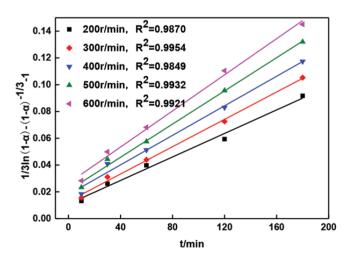


Figure 10–1/3  $\ln(1-\alpha)$  – 1+(1- $\alpha$ )-1/3 versus time at different stirring speeds

## Recommendations

According to the above kinetic analysis, the leaching of tellurium from gold concentrate is controlled by interface transfer and diffusion across the product layer. However, through the condition test, it can be seen that optimizing the leaching conditions has little further effect on the tellurium leaching rate, or it is necessary to increase the tellurium leaching rate by increasing the energy input. Therefore, further studies should be undertaken to improve the tellurium leaching rate.

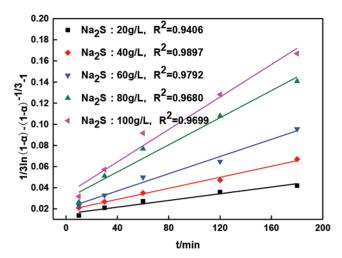


Figure 11 – ½ ln(1– $\alpha$ ) – 1+(1– $\alpha$ )–13 versus time at different Na $_2$ S concentrations

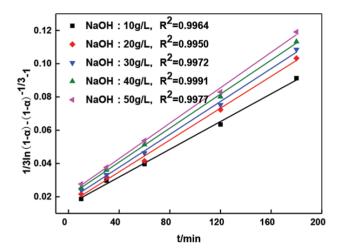


Figure 12–1/3  $\ln(1-\alpha)$  – 1+(1- $\alpha$ )-1/3 versus time at different NaOH concentrations

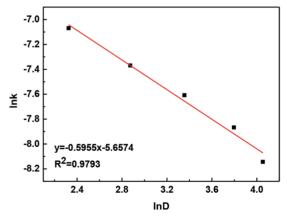


Figure 13-Ink versus InD at different average particle sizes

Future work will include investigation of tellurium minerals from leach residues to find out why they are difficult to leach, and then testing different measures to enhance the leaching process. It is suggested that different oxidants could be employed. The leaching kinetics of tellurium under different conditions is studied by researching the effect of changes in grain parameters, apparent activation energy, and the reaction

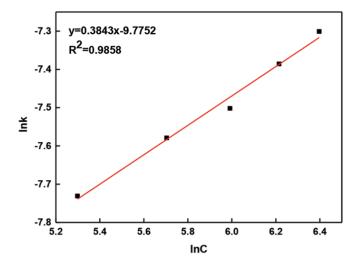


Figure 14-Ink versus InC at different stirring speeds

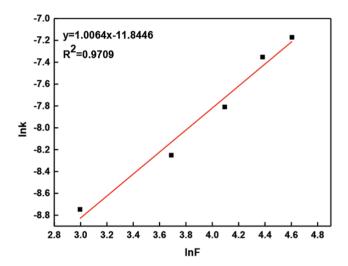


Figure 15-Ink versus InF at different Na<sub>2</sub>S concentrations

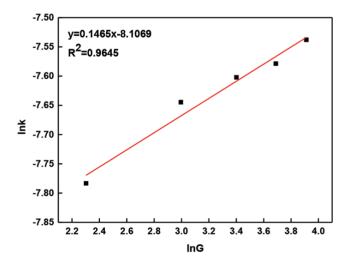


Figure 16-Ink versus InG at different NaOH concentrations

order of tellurium under alkaline sulphide leaching and oxidation leaching. Tellurium leaching may also be enhanced by roasting pretreatment, especially oxidation roasting.

#### Conclusion

In the range of parameters studied, the most suitable conditions for the leaching process of tellurium from gold concentrate were an average particle size of 17.77 um, stirring speed 500 r/min, Na<sub>2</sub>S concentration 80 g/L, NaOH concentration 30 g/L, and leaching temperature 80°C. Under these conditions. a tellurium extraction of 79.14% was obtained. The leaching rate increased with increasing Na<sub>3</sub>S and NaOH concentration. stirring speed, and leaching temperature, and decreasing average particle size. The leaching temperature had the most significant effect on tellurium leaching, followed by Na<sub>2</sub>S concentration, average particle size, stirring speed, and NaOH concentration. The leaching kinetics of tellurium by alkaline sulphide is controlled by interface transfer and diffusion across the product layer. According to a new model and the Arrhenius equation, the leaching kinetics equation was represented as  $1/\sqrt{3} \ln(1-\alpha)$  $1+(1-\alpha)^{-1/3}=1.1854C^{0.3843}D^{-0.5955}F^{1.0064}G^{0.1465}\exp\left(\frac{-34384}{RT}\right)t$  and the activation energy was 34.384 kJ/mol.

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