



Technical note

The leaching of gold, silver and their alloys in alkaline glycine–peroxide solutions and their adsorption on carbon



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ABSTRACT

The leaching of gold and silver in a pH modified glycine solution in the presence of hydrogen peroxide was investigated. It was found that this lixiviant system can dissolve gold and/or silver under neutral and alkaline conditions at ambient to moderately elevated temperatures (23–60 °C). The gold leaching rate in solutions containing 0.5 M glycine and 1% peroxide at pH 11 after 48 h of leaching was found to be 0.322 $\mu\text{mol}/\text{m}^2 \text{ s}$, which is comparable to gold leaching rate after six days in thiosulphate–EDTA or thiosulphate–oxalate systems in the presence of thiourea (0.22–0.25 $\mu\text{mol}/\text{m}^2 \text{ s}$). In alkaline glycine–hydrogen peroxide solutions, it has been found that gold leach rate from gold–silver (50% Ag) alloy is about 6 times higher than rate from pure gold and the silver leach rate was 0.247 $\mu\text{mol}/\text{m}^2 \text{ s}$. An induction period of 48 h was noted for rolled precious metal foils, after which leaching rapidly accelerated. The reaction rate sensitivity to temperature can be explained by a mechanism that is chemical reaction controlling rather than mass transfer controlling as found for cyanide leaching. Precious metal dissolution rates increased with increased glycine concentration, increased silver content, and increasing pH (up to pH 11). The gold–glycinate complex was found to effectively load on activated carbon up to 13.2 $\text{g}_{\text{Au}}/\text{kg}_{\text{carbon}}$ in 4 h.

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1. Introduction

Many different types of reagent suites have been investigated as potential alternative lixivants for gold and/or silver. Thiosulphate, acidic thiocyanate, halides, thiourea and a range of more exotic organic reagents have been extensively tested (Pangum and Browner, 1996; Jeffrey, 2001; Aylmore, 2005; Heath et al., 2008; Oraby et al., 2010; Yang et al., 2011). However, the oxygenated alkali cyanide reagent suite remains the default choice for the industrial extraction of gold–silver ores. Most recently, thiosulphate based systems have been identified as potential industrial contenders to cyanide based systems (Choi et al., 2013). Yet thiosulphate lixiviant mixtures (e.g. copper ammoniacal (Jeffrey et al., 2003; Senanayake, 2005) or ferric-Tu–EDTA systems (Zhang et al., 2005; Heath et al., 2008)) have many practical complexities and are often not environmentally benign. A calcium thiosulphate system has recently been commissioned by Barrick Goldstrike (Choi et al., 2013) indicating some willingness to consider non-cyanide lixivants for industrial niche applications, such as leaching gold from pregrobbing ores.

The main challenge in developing substitutes for cyanide is that the alternative for gold extraction should be inexpensive; less-toxic and effective to treat gold ore efficiently and economically when cyanide

does not, or it should allow new resources to be processed which were previously uneconomic or not permitted.

In this exploratory research, glycine has been considered as a lixiviant for gold in an alkaline environment, with hydrogen peroxide as an oxidant. Glycine is the simplest α -amino acid with a carboxylic group and an amine group and is manufactured in bulk (in quantities similar in magnitude as cyanide) from acetic acid via chloro-acetic acid.

Amino acids are classified according to their acidity or the number of amino and carboxyl groups into three groups: (1) neutral (one amino and one carboxyl) such as glycine, alanine, valine, leucine (2) acidic (one amino and two carboxyl groups) such as aspartic acids, glutamic acid; (3) basic (two amino and one carboxyl group) such as lysine and histidine (Finar, 1963).

Gold solubility in different amino acid solutions has been studied by Zhang et al. (1997) who studied the environmental geochemistry of gold. Their results indicate that gold is soluble in amino acids and other organic acids and it is most soluble in the basic amino acids such as histidine. From a human health and biochemistry perspective, Brown and Smith (1982) have examined the solubility of gold in solutions of different amino acids in skin secretions in order to assess the ability of these amino acids to dissolve gold and, hence, could be a possible source of allergic reactions in the human skin. Recently, Feng and Van Deventer (2011) have also found that gold extraction in thiosulphate solutions was largely improved by the addition of amino acids (L-valine, glycine, DL-alanine and L-histidine). It has also been noted (Groudev et al., 1995) that during the biological leaching of gold

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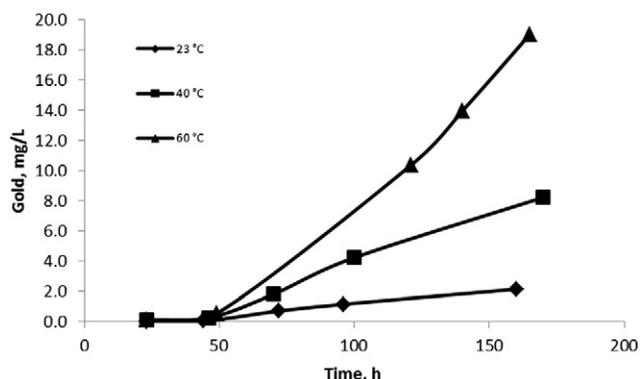


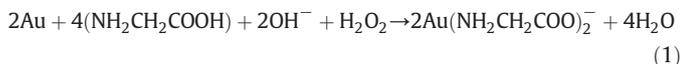
Fig. 1. Effect of temperature on gold dissolution from a pure gold foil: 1 M glycine, 1% H₂O₂, pH 10, 350 rpm, at different temperatures.

from ores, the dissolution of gold was done by occurred via gold-oxidising (peroxides) and a mixture of gold complexing (amino acids) agents secreted by the bacteria. Some of the identified micro-bacterial synthesised amino acids are asparagine, glycine, aspartic, histamine, serine and phenylamine and alanine (Korobushkina et al., 1974). However, none of the authors performed a systematic kinetic study of gold and silver dissolution with single amino acids, or their pre-determined combinations, with suitable oxidants and pH ranges suitable for metallurgical extraction. Nor did any of the researcher's investigate any downstream concentration and recovery processes of gold glycinate and/or silver glycinate from their solutions.

The binding mechanism of gold in solutions containing amino acids involves initially the formation of a hydroxyl gold complex by an ion-exchange mechanism. In alkaline solutions, gold is fixed by reduction of an intermediate species and chelating with gold bound to s-ligands (Bergeron and Harrisony, 1989). Many of the published data have focused on studying the effect of organic matter on gold mobility, transport and chelating during the formation of lateritic gold deposits (Andrade et al., 1991; Colin and Vieillard, 1991; Zang and Fyfe, 1993; Liu et al., 1994).

In alkaline pH, amino acids such as glycine, alanine, valine and phenylalanine complex gold where the amino acid coordination by the metal is effected through the amino and carboxyl groups. However, gold-amino acids bonds in histidine are coordinated through the nitrogen atoms of the amino group (Korobushkina et al., 1983). The mechanism in which gold is combined with amino acid is mainly through coordination with atomic nitrogen in the amino group to form a covalent bonded complex and with oxygen in the carboxyl group to form ionic-bonded complex (Komnitsas, and Pooley, 1990; Jian and Guifu, 1995).

Glycine can exist in aqueous solutions in three different forms, namely $^+H_3NCH_2COOH$ (cation), $^+H_3NCH_2COO^-$ (zwitterion), and $H_2NCH_2COO^-$ (anion). It also forms a strong complex with gold(I) as $Au(H_2NCH_2COO)^{-2}$. The stability constant of gold with glycine at pH 9 is 18.0 which is higher than $AuCl^{2-}$ (9.1), $AuBr^{2-}$ (12.0) and $Au(SCN)^{2-}$ (17.1) and $Au(SO_3)_2^{2-}$ (15.4), (Aylmore, 2005). As an example, the stoichiometry of gold dissolution in alkaline glycine solutions is described in Eq. (1):



The processes of gold dissolution and precipitation depend on pH and E_h conditions of the medium and are more intensive in the presence of peroxides in alkaline media (Korobushkina et al., 1983). Brown and Smith (1982) have found that the presence of hydrogen peroxide greatly enhances the gold and copper dissolution from gold jewellery dissolution.

To progress the understanding of alkaline glycine as a potential lixiviant, the effects of glycine concentration, hydrogen peroxide concentration, precious metal type and composition and temperature on the leaching, and the subsequent adsorption of the gold complexes on granular activated carbon, are evaluated below.

2. Experimental

2.1. Leaching

All experiments were carried out using solutions prepared from analytical grade reagents and Millipore water. Unless specified, all experiments are conducted using magnetic stirrers and Teflon coated magnetic stirrer bars. In a beaker, 400 mL of a solution containing 1 M glycine and 1% hydrogen peroxide was heated to 60 °C and stirred at 350 rpm. The evaporation effect during leaching at 60 °C was controlled by covering the leach beaker. The stirrer and beaker was also placed on an electronic balance to measure the difference of weight due to evaporation frequently during the experiment. Any decrease in the total weight was adjusted by adding distilled water to the leach solution. Rolled gold, silver and gold–silver alloy sheets (10 cm × 1 cm × 0.1 cm) were used in all the experiments and they were made from 99.99% pure gold and silver. Before each experiment, the surface of the each sheet was polished with Struers waterproof silicon carbide paper (FEPA P#2400). Finally, the gold sheet was washed with distilled water and allowed to dry in the presence of air at ambient temperature.

2.2. Adsorption onto activated carbon

To assess the use of activated carbon as a potential upgrading agent to adsorb the gold–glycinate complex, 1.5 g/L of fresh activated carbon was added into the pregnant solutions after leaching. The activated carbon used, had the following characteristics:

- Brand: PICAGOLD G210 AS,
- Size Range: $-2.36 + 2$ mm,
- Ash content of 2%,
- Density of 0.48 g/mL,
- Moisture content 2%

The carbon was washed and air dried at 65 °C prior to use. The adsorption experiments were conducted in 2.5 L Winchester bottles on a bottle roller at room temperature and rotation speed of 150 rpm. In order to evaluate the gold adsorption on carbon, sub-samples were taken at different time intervals and then diluted and stabilised with aqueous sodium cyanide before being analysed using ICP-OES.

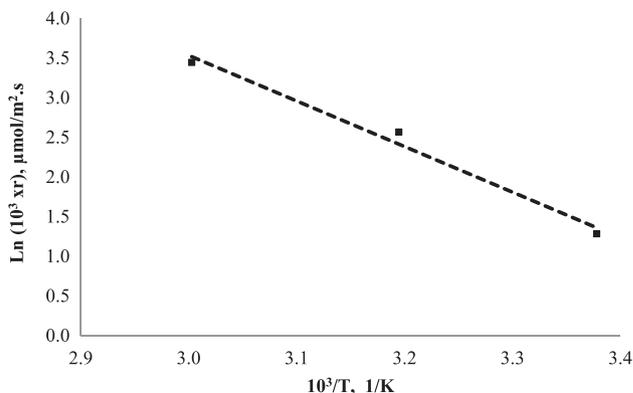


Fig. 2. Arrhenius plot for the rate of gold dissolution as a function of temperature.

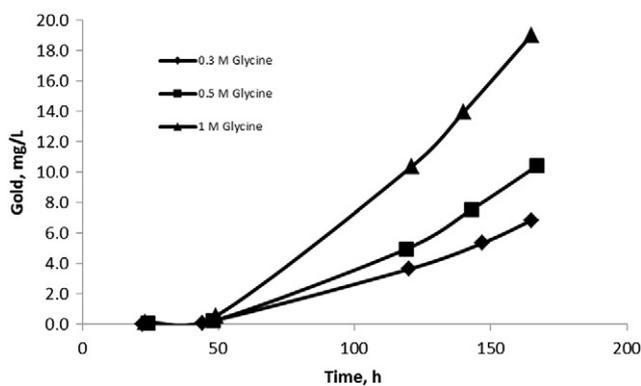


Fig. 3. Effect of glycine concentration on gold dissolution from a pure gold foil: glycine, 1% H_2O_2 , pH 10, 350 rpm, 60 °C.

3. Results and discussions

The effects of temperature, glycine concentration, metal/alloy composition and pH on gold/silver/alloy dissolution are evaluated below.

3.1. Effect of temperature

The gold leach rate as a function of temperature was calculated from the amounts of dissolved gold in the leaching solution to determine the activation energy for gold dissolution in glycine solutions. Fig. 1 shows the effect of temperature on the kinetics of gold leaching. It can be seen that there is an induction time of 48 h and after this time the gold dissolution increases dramatically as the temperature increases. According to the Arrhenius equation shown in Eq. (2):

$$k = Ae^{-E_a/RT} \quad (2)$$

where:

k	is the rate constant ($\text{mol}/\text{m}^2 \text{ s}$);
E_a	is the activation energy (J/mol);
R	is the universal gas constant ($8.314 \text{ J}/\text{K mol}$);
T	is the absolute temperature (K); and
A	is the pre-exponential Arrhenius factor.

In order to determine if the gold–glycine reaction is a chemically controlled or a diffusion controlled reaction, the activity energy for the gold dissolution can be calculated from a plot of $\ln(\text{Rate})$ versus $1/T$ as shown in Fig. 2. The Arrhenius plot was found to be linear and the calculated activation energy was about 50 kJ/mol. According to Power and Ritchie (1975), chemically controlled reactions have an activation energy higher than 25 kJ/mol. The observed activation energy indicates that the gold dissolution in glycine solution is chemically controlled.

3.2. Effect of glycine concentration

The kinetics of gold dissolution in solutions containing different glycine concentrations and 1% hydrogen peroxide at pH 10 and temperature of 60 °C was studied and the results are plotted in Fig. 3. It can be seen from the results shown in Fig. 3 that gold dissolution increases by

Table 1

Gold leach rate from a pure gold foil at different glycine concentrations: glycine, 1% H_2O_2 , pH 10, 350 rpm, 60 °C.

Glycine, M	Au, $10^3 \times \mu\text{mol}/\text{m}^2 \text{ s}$
0.30	11.3
0.50	16.9
1.00	31.3

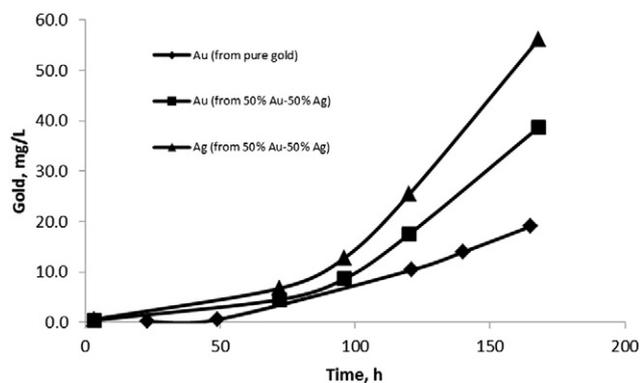


Fig. 4. Effect of silver on gold dissolution from a pure gold and gold/silver foil: 1 M glycine, 1% H_2O_2 , pH 10, 350 rpm, 60 °C.

increasing the glycine concentration up to 1 M. Table 1 also shows the gold leach rate at different glycine concentrations.

The alkaline glycine–peroxide system can be compared to the thiosulphate–ferric EDTA or thiosulphate–ferric oxalate systems. The gold leach rate shown in Table 1 in glycine–peroxide system is significantly higher, or comparable to, the rates of other alternative lixivants such as 0.1 M thiosulphate solutions containing ferric oxalate or ferric EDTA as an oxidant ($2 \times 10^{-3} \mu\text{mol}/\text{m}^2 \text{ s}$) in the absence of thiourea (Heath et al., 2008).

3.3. Effect of silver

To study the effect of silver on gold dissolution, foils with a surface area of 20 cm^2 of pure gold and 50% gold–50% silver have been leached in solutions containing glycine and peroxide. Gold and silver dissolution from pure gold and gold–silver alloy is shown in Fig. 4. It can be seen that the presence of silver enhances gold dissolution and silver dissolves faster than gold in glycine solutions. This observation is consistent with the studies showing that silver enhances the dissolution of gold in the cyanidation (Sun et al., 1996; Jeffrey and Ritchie, 2000; Wadsworth et al., 2000; Wadsworth and Zhu, 2003) and thiosulphate (Liu, 1993; Chandra and Jeffrey, 2004; Oraby and Jeffrey, 2010; Oraby et al., 2012).

Table 2 shows the gold and silver leach rate after 168 h from pure gold and 50% gold–50% silver. It can be seen that gold leach rate from gold–silver alloy is about 6 times higher than rate from pure gold. This indicates that gold from electrum will leach significantly faster in alkaline glycine/glycinate solutions than from pure gold. The leaching of silver from a silver–gold alloy is particularly fast compared to pure gold.

3.4. Effect of pH

The effect of adding hydroxide ions on the gold dissolution is shown in Fig. 5. It can be seen the glycine–peroxide system is very sensitive to the leaching pH and hydroxide ions. From the results shown in Fig. 5, it can be seen that gold dissolution increases significantly by increasing the leaching pH from pH 10 to pH 11. The gold leaching rate was 0.35 ($\mu\text{mol}/\text{m}^2 \text{ s}$) when measured at pH 11 in solutions containing 0.5 M

Table 2

Gold and silver leach rates from pure gold and gold–silver alloy: glycine, 1% H_2O_2 , pH 10, 350 rpm, 60 °C.

Au, Ag source	Au, $10^3 \times \mu\text{mol}/\text{m}^2 \text{ s}$
Gold from (pure gold sheet)	31
Gold (from 50% Au–50% Ag)	185 ^a
Silver (from 50% Au–50% Ag)	247 ^a

^a The leach rate was calculated using the relative surface areas of gold and silver in the alloy.

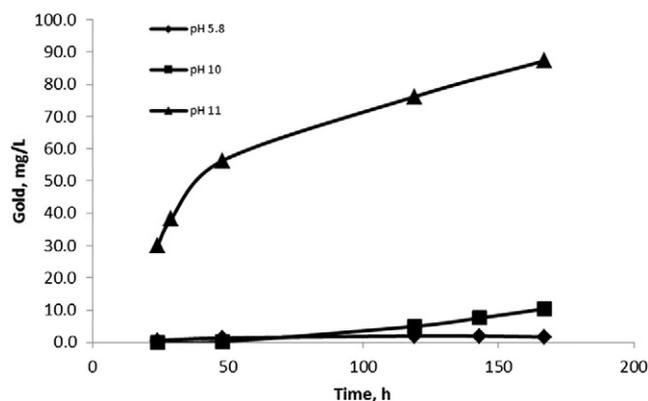
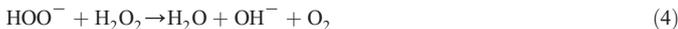


Fig. 5. Effect of leaching solution pH on gold dissolution from a pure gold foil: 0.5 M glycine, 1% H₂O₂, pH, 350 rpm, at 60 °C.

glycine and 1% peroxide at a leaching temperature of 60 °C after only 24 h of leaching.

Increasing gold dissolution by increasing the leaching pH can be explained by peroxide activation by caustic soda at higher pH as shown in Eq. (3). Furthermore, at higher pH, the decomposition of peroxide increases to generate hydroxide ions and oxygen as shown in Eq. (4) or produce hydroxide radicals according to Eq. (5) according to the observations of Suess et al. (1997).



The produced hydroxide radicals enhance the gold dissolution as it was noted by Nowicka et al. (2010) that $\cdot\text{OH}$ radicals in Fenton's reagent quickly dissolve gold from a mechanically polished gold surface.

Table 3 shows the gold leaching rate after different leaching times at pH 5.8, 10 and 11. The data show that gold leach rate at pH 11 is 30 times higher than the rate at pH 10 after 48 h. However, it was found that the leach rate decreases by increasing leaching time at pH 11. The initial gold leaching rate at natural pH of solution (pH ~ 5.8) was similar to the rate at pH 10 up to 100 h and then the rate at pH 10 slightly increases as the leaching time progressed. It can be seen from the gold concentration in the solution (Fig. 5) and gold leach rate (Table 3) that the gold leach rate increases sharply during first 50 h of leaching after which the rate decreases. This may be explained by the passivation of the gold surface by a layer of AuOH adsorbed to the gold surface.

Table 3

Gold leach rate from a pure gold foil at different leaching pH: 0.5 M glycine, 1% H₂O₂, pH, 350 rpm, 60 °C.

Leaching time, h	Au, 10 ³ × μmol/m ² s		
	pH 5.8	pH 10	pH 11
24	8.11	0.59	352
29	8.75	1.30	367
48	5.13	11.47	322
119	4.19	14.34	174
167	3.02	16.93	142

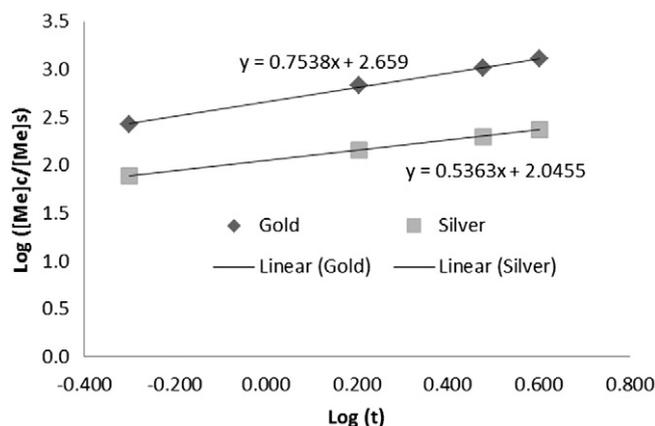


Fig. 6. Plot of $\log (\Delta[\text{Me}]_c / [\text{Me}]_s)$ against $\log (t)$ for 4 h (1 M glycine; pH 10, 25 °C; carbon 1.46 g/L).

3.5. Gold–glycinate complex adsorption on carbon

Carbon adsorption has been extensively used in gold industry to recover gold and silver from cyanide leach solutions. However, one of the thiosulphate system drawbacks, as it is one of the most promising alternatives to cyanide, is that the adsorption of gold–thiosulphate complex on activated carbon is very weak. Therefore, it is appropriate to explore the ability of activated carbon to adsorb gold–glycinate complex from the leach solution particularly from an industrial application perspective.

The kinetics of gold (38.7 mg/L) and silver (56.1 mg/L) adsorption from glycine solutions onto activated carbon has been evaluated by the determination of the carbon activity constant using the Fleming k , n method (Nicol et al., 1984) as shown in Eq. (6). Eq. (6) can be linearized to get the values of k and n as shown in Eq. (7).

$$\Delta[\text{Me}]_c = k \cdot [\text{Me}]_s \cdot t^n \quad (6)$$

$$\frac{\log(\Delta[\text{Me}]_c)}{[\text{Me}]_s} = n \cdot \log(t) + \log(k) \quad (7)$$

Where:

$\Delta[\text{Me}]_c$ change in $[\text{Au}]_c$ or $[\text{Ag}]_c$ on carbon from $t = 0$ to $t = t$ hours;
 $[\text{Me}]_s$ $[\text{Au}$ or $\text{Ag}]$ in solution at $t = t$ hours;
 n an experimental constant; and
 k an empirical adsorption rate constant.

Fig. 6 shows the plot of $\log (\Delta[\text{Me}]_c / [\text{Me}]_s)$ against $(\log t)$ where $[\text{Me}]_c$ refers to metals (Au or Ag). From the data of gold and silver adsorption on carbon in glycine solutions, it is clear to see that, similar to cyanide systems, the carbon adsorbed gold ($k = 460$) significantly faster than silver ($k = 110$). The empirical adsorption rate constant (k) has been calculated from the antilogarithm of the intercept of plots in Fig. 6. The adsorption experiments showed that gold and silver glycinate complexes were found to load on carbon in 4 h up to 13.2 kg_{Au}/t_{carbon} and 3.4 kg_{Ag}/t_{carbon} respectively. Fig. 6 shows the decreasing of gold and silver in the solutions due to metal adsorption on carbon over time.

4. Conclusions

An alkaline glycine hydrogen peroxide leach system was explored in this paper for gold, silver and their alloys and subsequent adsorption onto activated carbon. It was clear that, even though moderate heating was required and observed leach rates were slower than cyanide leaching, glycine based systems may be considered for native gold/silver

leaching for leach modes where leach rates are often determined by bulk and particle diffusion processes, such as in-situ, vat or heap leaching approaches. These leach modes, which are open to the environment, can benefit significantly from environmentally benign, biodegradable, lixivants. The addition of hydroxide ions into glycine–peroxide solution was effective to significantly improve the gold leaching rate. Leaching experiments showed that the gold leach rate increases by increasing the glycine concentration. It was also found that gold dissolves from gold–silver alloy and silver at a faster rate than pure gold. It has been demonstrated that increasing the leaching pH from 10 to 11, increases gold leaching rate from 0.015 to 0.322 $\mu\text{mol}/\text{m}^2 \text{ s}$ respectively after 48 h leaching. It has also been shown that the leaching solution is very sensitive to the leaching temperature and the reaction is a chemically controlled. Activated carbon was shown to effectively adsorb gold and silver from the leach solution, with gold glycinate showing a higher and more rapid loading than that of silver, similar to the adsorption behaviour of their cyanide complexes.

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