

Received June 12, 2014; reviewed; accepted July 20, 2014

DISSOLUTION OF GOLD WITH CYANIDE REPLACING REAGENTS

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Abstract: Cyanide is used in the leaching phase of hydrometallurgical recovery of gold. The toxicity and environmental risks have created a need for safer alternatives. There is a vast amount of information about gold extraction and a selection has been collected and formalized to be used in a decision support tool. The tool Auric Advisor uses case-based reasoning (CBR) to provide process alternatives for the user queries. The aim of this paper is to study leaching of gold with cyanide replacing alternatives such as ammonium thiosulfate, chloride-hypochlorite, thiourea and thiocyanate to provide new cases for the tool. The behaviour of gold was examined with electrochemical tests and weight loss measurements using quartz crystal microbalance (QCM) were used to determine the dissolution rate. Based on literature, the dissolution rate determined for cyanide solution was $2.5 \text{ mg}\cdot\text{cm}^{-2}\cdot\text{h}^{-1}$. Based on polarisation experiments and potential measurements, the ammonium thiosulfate system with cupric ion as oxidant was estimated to dissolve gold slower than cyanide. In the QCM tests the chloride-hypochlorite alternative showed highest dissolution rate of $8.6 \text{ mg}\cdot\text{cm}^{-2}\cdot\text{h}^{-1}$ at the redox potential of 900 mV vs. SHE. The thiocyanate system with ferric ion dissolved gold at a rate of $3.4 \text{ mg}\cdot\text{cm}^{-2}\cdot\text{h}^{-1}$ at the redox potential of 620 mV vs. SHE. Thiourea system with ferric ion showed the dissolution rate of $1.65 \text{ mg}\cdot\text{cm}^{-2}\cdot\text{h}^{-1}$ at the redox potential of 450 mV vs. SHE. The results of the rapid tests were comparable with literature and were included in the case base of the Auric Advisor decision-support tool.

Keywords: *gold leaching, ammonium thiosulfate, chloride, thiourea, thiocyanate*

Introduction

The most common hydrometallurgical methods of primary raw material utilization in the production of gold are based on the use of cyanide (Hilson, 2006). In this method a weak sodium cyanide solution (NaCN) of 100-500 ppm is used. Since the Baia Mare disaster in Romania in January 2000, the use of cyanide has been resisted. Germany passed a decree in 2002 prohibiting mines from using cyanide in leaching processes. The Czech Senate and Czech Parliament made decisions 2000-2002 to forbid cyanide leaching in the Czech Republic. Hungarian Parliament voted against the use of cyanide at mine sites in 2009 (Laitos, 2012, Eurostat, 2010). In 2010, the European

Union rejected a proposal to ban all cyanide use, but set stringent cyanide limits for tailings ponds in Directive 2006/21/EC. In the U.S. several states have restricted use of cyanide. Many provinces in Argentina have banned the use of cyanide in mining operations. Costa Rica banned cyanide in 2010 (Laitos, 2012).

The gold producers have been looking for alternative solutions to cyanide. Some of the alternative methods are thiourea, thiocyanate, thiosulphate, agglomerates of oil and coal, and halides (Hilson, 2006). Operating windows for cyanide leaching and alternative chemistries are shown in Fig. 1. The most actively studied chemistries are thiosulfate, thiourea, halides and α -hydroxynitriles (Aylmore, 2005, Hilson, 2006, Syed, 2012).

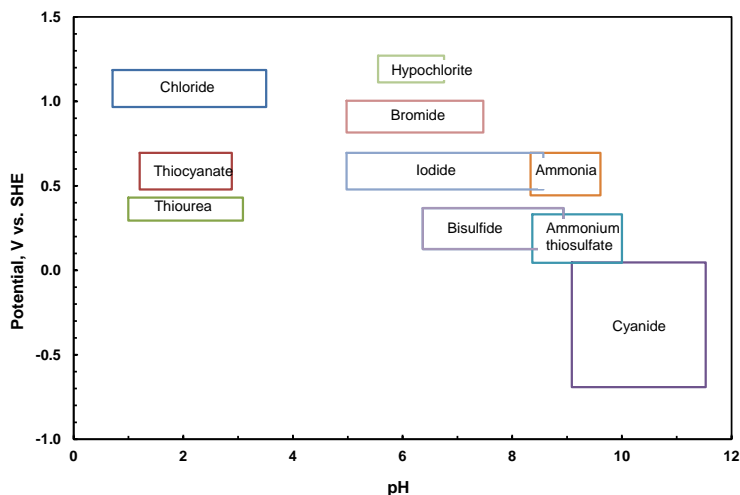
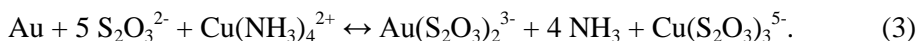


Fig. 1. Potential-pH diagram showing typical operating windows for gold lixiviants (after Aylmore, 2005)

In leaching of gold a complexing ligand and oxidant are needed. Dissolution of gold in cyanide solution is shown in Eq. 1. At low cyanide concentrations, the dissolution rate is a function of cyanide concentration. At high cyanide concentrations the dissolution rate of reaction



is a function of oxygen concentration (Deschenes, 2005). Dissolution of gold in alkaline thiosulfate solution is shown in Eq. 2. Reaction 2 is slow unless Cu^{2+} , that works as an oxidant, catalyses it (Eq. 3) (Feng, 2011). Ammonia is also needed to stabilize the solution

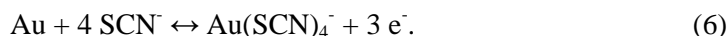
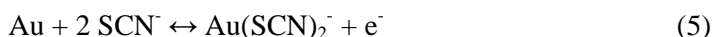


Dissolution in thiourea is shown as:



The reaction is sensitive to pH and redox potential. Thiourea is intrinsically unstable and decomposes rapidly to substances that are unable to leach gold (Murthy, 1996; Aylmore, 2005)

Thiocyanate can dissolve gold as either Au(I) (Eq. 5) or Au(III) (Eq. 6) complexes:



The ferric ion can be used as the oxidant and it can also oxidize thiocyanate to thiocyanogen and/or trithiocyanate (Li, 2012a). Dissolution of gold in a chloride system is:

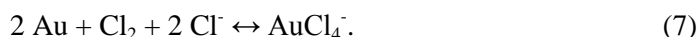


Table 1. Dissolution rates of gold in selected cyanide and alternative lixiviant solutions

Cyanide-based system	Rate / mg·cm ⁻² ·h ⁻¹	Rate / mol·m ⁻² ·s ⁻¹	Reference
0.26 mM O ₂ , 5 mM CN ⁻	0.07	0.1·10 ⁻⁵	Senanayake (2004)
O ₂ , 77 mM CN ⁻ , pH 10.5	0.85	1.2·10 ⁻⁵	Senanayake (2004)
Aerated cyanide, 41 mM NaCN	2.5	3.5·10 ⁻⁵	Nam (2008)
Thiosulfate-based system			
0.7 mM Cu ²⁺ , 0.1 M (NH ₄) ₂ S ₂ O ₃ , 0.5 M NH ₃	0.09	0.13·10 ⁻⁵	Feng (2011)
10 mM Cu ²⁺ , 100 mM S ₂ O ₃ ²⁻ , 0.4 M NH ₃	2.7	3.8·10 ⁻⁵	Senanayake (2004)
Thiourea-based system			
Sat. O ₂ , 5 mM SC(NH ₂) ₂ , 200 mM Na ₂ S ₂ O ₃	0.03	0.04·10 ⁻⁵	Senanayake (2004)
5 mM Fe ³⁺ , 26.3 mM SC(NH ₂) ₂ , 0.1 M H ₂ SO ₄	1.7	2.4·10 ⁻⁵	Senanayake (2004)
Thiocyanate-based system			
50 mM SCN ⁻ , pH = 2	0.73-2.2	1.0·10 ⁻⁵ -3.1·10 ⁻⁵	Li (2012b)
Halide-based system			
27 mM NaOCl, 85 mM NaCl, pH 6	0.5	0.7·10 ⁻⁵	Senanayake (2004)
0.8 mM HOCl, 1700 mM NaCl, pH 3.5	1.0	1.4·10 ⁻⁵	Senanayake (2004)
135 mM NaOCl, 850 mM NaCl, pH 6	10	14·10 ⁻⁵	Senanayake (2004)
190 mM OCl ⁻ , 1.7 M NaCl, pH 6	13	18.3·10 ⁻⁵	Nam (2008)

A gold complex AuCl_2^- forms first and it is rapidly oxidized to AuCl_4^- (Aylmore, 2005; Nam, 2008). A residual amount of oxidant is required to maintain a high solution potential to avoid precipitation of metallic gold from the solution (Aylmore, 2005). Some reported dissolution rates of gold in the cyanide solution and in alternative lixivants are listed in Table 1. Using the same basic lixiviant, the reported dissolution rates can vary up to two orders of magnitude depending on the concentrations of complex forming ligand and oxidant.

In this work we have studied alternative chemistries to cyanide in leaching of gold. The target was to develop a rapid test method for determination of dissolution rates for primary and secondary raw materials that do not require pre-treatment, such as a free-milling ore. The verified experimental test results can then be included in the case base of decision support tool Auric Advisor for process development (Sauer, 2013). The Auric Advisor tool takes a query from the user using selected attributes related to process chemistry, reaction rate etc., and retrieves the most similar cases as a result.

Materials and methods

The experiments included measurement of polarization curves for gold in different media, determination of the redox potential to achieve wanted dissolution rates and measurement of gold dissolution rate with a quartz crystal microbalance (QCM) (Jeffrey, 2005; Zelinsky, 2012). The electrochemical measurements were conducted using a rotating disc electrode (RDE) type PARC RDE0005 at 300 rpm and an ACM Instruments Gill AC potentiostat. The electrochemical measurements were conducted in a three-electrode cell, where a Luggin capillary and KCl bridge connected the cell to the reference electrode. The reference electrode used in the experiments was saturated Ag/AgCl electrode, type Radiometer REF 201. The counter electrode used in the experiments was platinum wire. Polarization curves were measured using 100 mV/min sweep rate. The tests to determine oxidant concentration to achieve wanted redox potential were done by addition of the oxidant and measuring redox potential with a Mettler Toledo combination redox electrode ($E_h = 0.207$ V) using the potentiostat as data logger. All the potential values are reported versus saturated Ag/AgCl ($E_h = 0.197$ V). The dissolution rate tests were done using the SRS 200 quartz crystal microbalance and measuring continuously the weight loss of the gold plated the QCM crystal in a solution with controlled redox potential. The weight loss tests were done in one litre reactor with continuous laminar flow $300 \text{ cm}^3/\text{min}$ to the QCM surface (Von Bonsdorff, 2007).

The test solutions were based on ammonia - thiosulfate, chloride - hypochlorite, thiourea and thiocyanate. The test solutions are shown in Table 2. The pH of the thiosulfate solution was adjusted by ammonia, pH of thiourea and thiocyanate solutions with sulphuric acid and pH of chloride solution with hydrochloric acid. The test solutions were purged with air.

Table 2. Compositions of the test solutions

Solution	Ligand and stabiliser, M	Oxidant	Temperature, °C	pH
Ammonia – thiosulfate	0.1-1, Na ₂ S ₂ O ₃ 1-4, NH ₃	Cu ²⁺	25-40	10-11
Thiourea	0.13, SC(NH ₂) ₂ 36·10 ⁻³ , Na ₂ SO ₃	Fe ³⁺	25-40	1-2
Thiocyanate	0.05-0.2, NaSCN 5·10 ⁻³ , SC(NH ₂) ₂	Fe ³⁺	25-40	1-2
Sodium chloride	3, NaCl	NaClO	25-40	1-3

Results and discussion

Polarization curves

Potentiodynamic polarization curves were measured to determine the overall dissolution behavior of gold in the test solutions. The measurements were done in air-purged solutions without other oxidants. The dissolution rate of gold at different potentials was estimated by using the Faradays law. Figure 2 shows polarization curves in an ammonia – thiosulfate solution. The open circuit potential varied between -200 and 0 mV and dissolution current densities at open circuit potential were in the order of 0.1-6 $\mu\text{A}\cdot\text{cm}^{-2}$. The polarization curves showed passivation at potentials above 200 mV. Figure 3 shows the polarization curves in the thiourea solution. The open circuit potential varied between -100 and 0 mV and the current densities at the open the circuit potential were in the order of 0.7-6 $\mu\text{A}\cdot\text{cm}^{-2}$. The current density increased continuously when the potential was increased and approached limiting

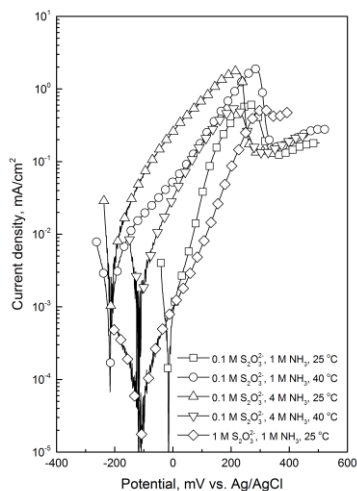


Fig. 2. Polarization curves of gold in ammonia – thiosulfate solution with NH₃ concentration 1 and 4 M and 25 and 40 °C

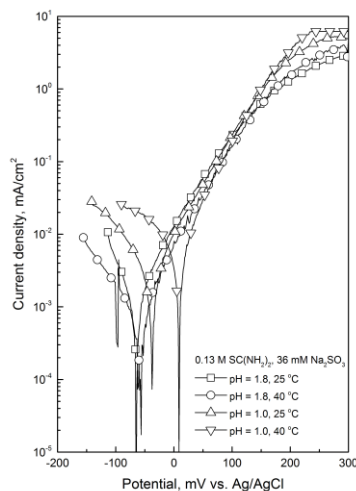


Fig. 3. Polarization curves of gold in thiourea solution with pH = 1.0 and 1.8 and temperatures 25 and 40 °C

current density $2\text{--}6\text{ mA}\cdot\text{cm}^{-2}$ at the potentials above 200 mV. Figure 4 shows polarization curves in the thiocyanate solution. The open circuit potential was 50–100 mV and the current densities at the open circuit potential were $0.1\text{--}0.4\text{ }\mu\text{A}\cdot\text{cm}^{-2}$. The current density increased continuously when the potential was increased and approached limiting current density in the order of $1\text{ mA}\cdot\text{cm}^{-2}$ at the potentials above 500 mV. Figure 5 shows the polarization curves in the chloride solution. The open circuit potential varied strongly with pH being 100–200 mV at pH = 3, 350–450 mV at pH = 2 and 500–600 mV at pH = 1. The current densities at the open circuit potential were $0.2\text{--}3\text{ }\mu\text{A}\cdot\text{cm}^{-2}$. Rapid dissolution started at the potentials above 600–700 mV and the current density increased more rapidly at low pH values.

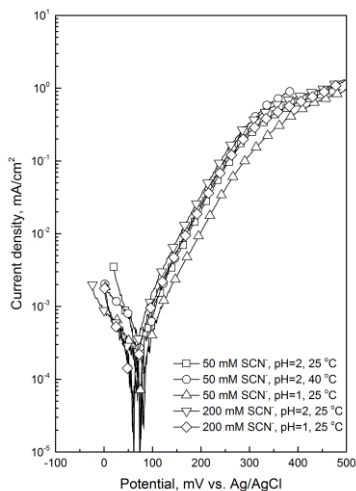


Fig. 4. Polarization curves of gold in 50 and 200 mM thiocyanate solution at pH = 1 and 2 and temperatures 25 and 40 °C

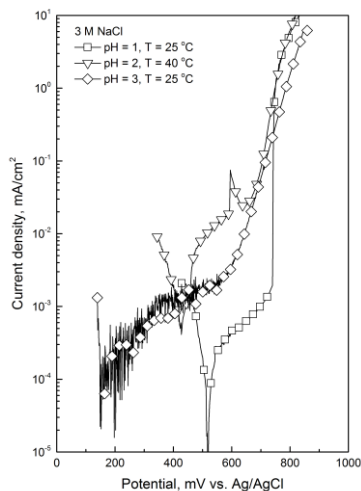


Fig. 5. Polarization curves of gold in 200 g/dm³ sodium chloride solution

Redox potential measurements

The maximum dissolution rate of gold in cyanide solutions equal to $2.5\text{ mg}\cdot\text{cm}^{-2}\cdot\text{h}^{-1}$ corresponds to the current density $1\text{ mA}\cdot\text{cm}^{-2}$. This was used to determine conditions for QCM reaction rate measurements. The tests at different oxidant concentrations were done to determine the redox potential that exceeds the gold dissolution rate $1\text{ mA}\cdot\text{cm}^{-2}$. Based on the polarization curves, the potential was 150–250 mV in the ammonia–thiosulfate solution, 60–100 mV in the thiourea solution, 400–500 mV in the thiocyanate solution, and 750–800 mV in the chloride solution. In the ammonia–thiosulfate solution passivation was noticed at the potentials over 200 mV and this may prevent reaching the wanted dissolution rate.

The control of redox potential was easy when using ferric sulfate in the thiourea and thiocyanate solutions and when using sodium hypochlorite in the sodium chloride solution. The redox potential increased with increasing oxidant concentration

(Table 3) and they remained constant for several minutes. The effect of cupric ions on the redox potential in the ammonia–thiosulfate solution was fluctuating and not easy to control (Fig. 6).

Table 3. Redox potentials in thiourea, thiocyanate and sodium chloride solutions

Oxidant	E_{redox} , mV in 0.13 M $\text{SC}(\text{NH}_2)_2$, 36 mM $\text{Na}_2\text{S}_2\text{O}_3$, pH = 1.0, T = 25 °C	E_{redox} , mV in 0.2 M NaSCN , 5 mM $\text{SC}(\text{NH}_2)_2$, pH = 2.0, T = 25 °C	E_{redox} , mV in 3 M NaCl T = 25 °C
10 g dm^{-3} Fe^{3+}	555±5		
2.5 g dm^{-3} Fe^{3+}	545±5		
1.3 g dm^{-3} Fe^{3+}	505±5		
1.0 g dm^{-3} Fe^{3+}		650±5	
0.5 g dm^{-3} Fe^{3+}		610±5	
0.25 g dm^{-3} Fe^{3+}		570±5	
25 g dm^{-3} OCl^- , pH = 2			1320±10
3.5 g dm^{-3} OCl^- , pH = 2			1220±10
3.5 g dm^{-3} OCl^- , pH = 3			1170±10

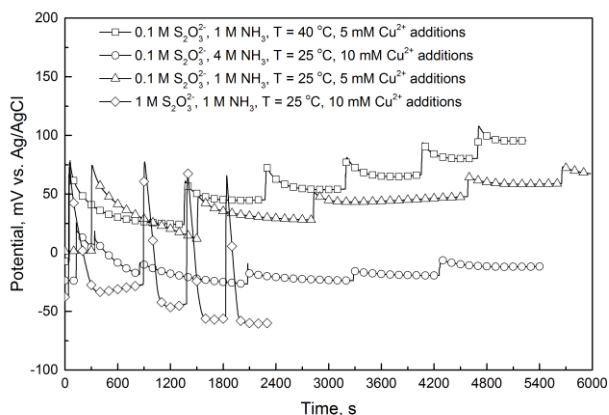


Fig. 6. Effect of Cu(I) ion on redox potential in ammonia – thiosulfate solution

Weight loss tests

Weight loss tests with gold plated QCM crystal were done in three solutions:

- 0.13 M $\text{SC}(\text{NH}_2)_2$, 36 mM $\text{Na}_2\text{S}_2\text{O}_3$, 2.2 g· dm^{-3} Fe^{3+} , pH = 1.0, T = 25 °C
- 0.2 M NaSCN , 5 mM $\text{SC}(\text{NH}_2)_2$, 0.4 g· dm^{-3} Fe^{3+} , pH = 2.0, T = 25 °C
- 3 M NaCl , 3.5 g· dm^{-3} OCl^- , pH = 3.0, T = 25 °C.

The redox potential was 250±15 mV in the thiourea solution, 420±10 mV in the thiocyanate solution, and 700±100 mV in the chloride solution. The ammonia–thiosulfate solution was excluded from this test series because of difficulties in controlling the redox potential.

Figure 7 shows the weight losses during the QCM measurements. The quartz crystal lost weight as gold was dissolved and the weight change stopped when only the chromium layer was in contact with the solution. The dissolution rate was determined from the decreasing slope. Dissolution rate was $1.66 \pm 0.08 \text{ mg} \cdot \text{cm}^{-2} \cdot \text{h}^{-1}$ in the thiourea solution, $3.40 \pm 0.04 \text{ mg} \cdot \text{cm}^{-2} \cdot \text{h}^{-1}$ in the thiocyanate solution, and $8.60 \pm 0.23 \text{ mg} \cdot \text{cm}^{-2} \cdot \text{h}^{-1}$ in the chloride solution. The dissolution rates are in the high end of the rates shown in Table 1.

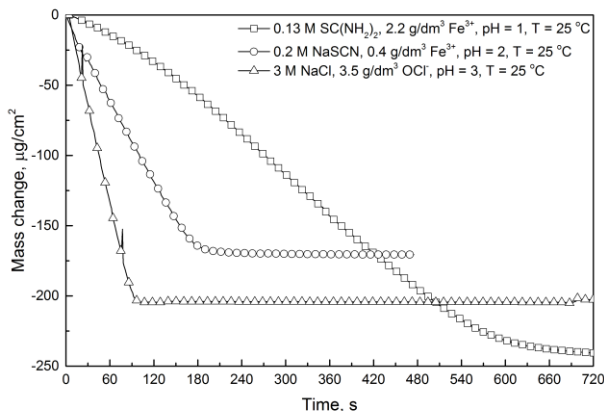


Fig. 7. Weight loss in QCM tests

Decision-support tool

A decision-support tool for development of gold extraction processes was developed using case-based reasoning (Sauer, 2013). The case-based reasoning is similar to human problem solving based on an experience. It operates by taking a problem (query) and searching for similar problems and their solutions (cases) from the case base. The tool called Auric Advisor operates on two levels. The first level searches for cases that describe existing processes most similar to the currently studied raw material. The process development follows the idea that similar raw materials can be treated using similar processes. The second level searches for cases that describe single unit processes or modifications of the unit processes. Literature and laboratory experiments are the sources for the cases (Rintala, 2012). The tests reported in this work provided new cases for the second level case base of the Auric Advisor tool.

Knowledge formalisation is the process of encoding knowledge in a case-based reasoning system. The target is to find balance between necessary grade of detail of knowledge and a lean and minimalistic knowledge model for efficient calculations. Knowledge formalisation begins with identification of the relevant entities in a domain, as well as their relationship with each other. Then, characteristics and important attributes are established for each entity and the form of case representation

is determined. The important attributes in leaching are presented in Table 4. These attributes and their values form in the case base. The most important outcomes of leaching are the dissolution rate, gold recovery and leaching time. The attribute values in Table 4 are taken from Table 1, and the results presented in this paper are included. The lixiviant chemistry has high weight, as basic chemistries cannot be mixed. The ligand concentration is known to affect the dissolution rate, but ligand, stabilisator and oxidant concentrations have to be matched. The oxidant and its concentration can also be coded as the redox potential. Temperature and pH will have a certain variation range, where their effect can be assumed to be monotonous. The missing information, like in Table 4, can be modeled using the regression analysis of similar cases.

Table 4. Case base consists of these attributes and their values for different systems (CN cyanide, TS thiosulfate, TU thiourea, TCN thiocyanate, CL chloride. New results in bold)

System	Ligand and concentration, mM		Stabilisator and concentration, mM		Oxidant and concentration, mM		T, °C	pH	Rate, mg cm ⁻² h ⁻¹
CN	CN ⁻	5			O ₂	0.26	20		0.07
CN	CN ⁻	77			O ₂		25	10.5	0.85
CN	NaCN	41			Aerated				2.5
TS	(NH ₄) ₂ S ₂ O ₃	100	NH ₃	500	Cu ²⁺	0.7	25		0.09
TS	S ₂ O ₃ ²⁻	100	NH ₃	400	Cu ²⁺	10	20		2.7
TU	SC(NH ₂) ₂	5	Na ₂ S ₂ O ₃	200	Sat. O ₂		30		0.03
TU	SC(NH ₂) ₂	26.3	H ₂ SO ₄	100	Fe ³⁺	5	25		1.7
TU	SC(NH ₂) ₂	130	Na ₂ S ₂ O ₃	36	Fe ³⁺	40	25	1.0	1.66
TCN	SCN ⁻	50					amb.	2	0.73-2.2
TCN	NaSCN	200	SC(NH ₂) ₂	5	Fe ³⁺	7	25	2	3.4
CL	NaCl	85			NaOCl	27	20	6	0.5
CL	NaCl	1700			HOCl	0.8	20	3.5	1.0
CL	NaCl	850			NaOCl	135	20	6	10
CL	NaCl	1700			OCl ⁻	190	20	6	13
CL	NaCl	3000			OCl ⁻	70	25	3	8.6

Information in the case base such as Table 4 is used by following way. The wanted outcome of the process step is defined. A vector containing the outcome attributes and their values is formed. The Auric Advisor tool will then select from the case base process alternatives that have similar outcome vectors as the wanted target. When the suitable cases are not found, the decision support tool can use models to indicate possible changes in attribute values to provide an outcome vector that is closer to the target.

Conclusions

The most studied alternative lixiviants for cyanide in gold extraction are the thiosulfate, thiourea, thiocyanate and halide solutions. The dissolution rates in these systems can vary up to two orders of magnitude depending on the lixiviant and oxidant concentrations. For selection of suitable leaching chemistry a decision-support tool has been developed.

In this work dissolution rates of gold have been measured in thiosulfate, thiourea, thiocyanate and halide solutions using the rotating disc electrode (RDE) and quartz crystal microbalance (QCM) methods. The dissolution rates at the open circuit potential in air saturated solutions without other oxidants were in the order of 0.1 to 6 $\mu\text{A}\cdot\text{cm}^{-2}$, and several orders of magnitude lower than what is needed in realistic processes. Oxidant levels to reach the current density equal to 1 $\text{mA}\cdot\text{cm}^{-2}$ were determined. The QCM tests in the determined solutions showed the rate of 1.66 ± 0.08 $\text{mg}\cdot\text{cm}^{-2}\cdot\text{h}^{-1}$ in the thiourea solution, 3.40 ± 0.04 $\text{mg}\cdot\text{cm}^{-2}\cdot\text{h}^{-1}$ in the thiocyanate solution, and 8.60 ± 0.23 $\text{mg}\cdot\text{cm}^{-2}\cdot\text{h}^{-1}$ in the chloride solution.

Acknowledgements

This research was carried out as part of the Finnish Metals and Engineering Competence Cluster (FIMECC)'s ELEMET program. One of the authors (M.K.) is thankful to the K.H. Renlund's Foundation for financial support.

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