



The major role played by calcium in gold plant circuits

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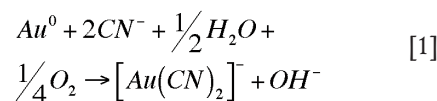
Synopsis

Arguments are presented in favour of a new interpretation of the chemistry pertaining to the dissolution of gold, when this takes place in typical gold plant leach circuits where calcium is present in solution. It is suggested that under these conditions the standard chemistry, as it applies in the absence of calcium, is no longer valid. Thus the concept of very soluble sodium and aurocyanide ionic species in solution is replaced by the presence of weakly soluble calcium aurocyanide, which is a covalent coordination complex that is non-ionic. Supporting evidence for this is provided and the implications that the presence of calcium in gold plant circuits may have for operational efficiency are discussed. In particular, because of the formation of this weakly soluble calcium complex, the addition of lime to the leach circuit is questioned. Arguments for the use of a sodium hydroxide/sodium carbonate/sodium bicarbonate buffer solution as a means of controlling pH and of increasing ionic strength in a so-called 'soft' leach are presented. Such a lime-free system, besides benefiting the leaching of gold, would have many downstream benefits in existing CIP circuits. Furthermore, the direct electrowinning of gold from the leach now becomes potentially feasible.

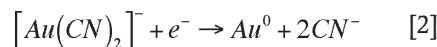
Introduction

Cyanide was first used on the Witwatersrand gold mines around 1890 and for more than 100 years has been the preferred route for gold extraction.

Cyanidation progresses according to the following equation¹:



Thus, one mole of hydroxide ion is produced for every two moles of cyanide ions consumed. The electrowinning of gold from simple solutions is readily carried out according to the equation:



From available thermodynamic data¹ reaction [1] has a highly negative free energy, even for saturated aurocyanide solutions,

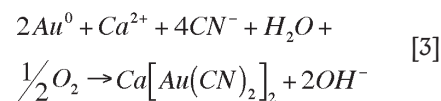
resulting in the formation of the very stable $[\text{Au}(\text{CN})_2]^-$ complex ($K = 10^{58}$).

Potassium aurocyanide is highly soluble in synthetic solutions (55–90 g/l at 25°C)², while sodium aurocyanide is moderately soluble at 5–25 g/l. The solubility of both complexes is temperature- and ionic strength-dependant. However, no data related to the solubility of such complexes in typical gold plant solutions appear to be available.

While Equation [1] may well apply to the cyanidation of gold in controlled conditions, using either potassium or sodium cyanide, the validity of this equation, when related to actual plant conditions, is questioned. In cases where plant conditions pertain, the addition of lime, together with the addition of either sodium or calcium cyanide, is the general practice. Such additions of calcium may well have a significant effect on the subsequent route of gold dissolution and on the solubility of the resulting aurocyanide complex, with further implications for various processes used during the recovery of gold.

The calcium aurocyanide hypothesis

Arguments will be presented in favour of the following gold dissolution reaction under typical plant conditions:



From this equation it may be seen that the end product is a calcium aurocyanide complex, rather than the highly soluble dissociated potassium or sodium aurocyanide complexes

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predicted by Equation [1]. No solubility data for calcium aurocyanide could be found, but in United States Patent 4468303 calcium aurocyanide adsorbed onto activated carbon is described as 'highly insoluble', and in order to be solubilized to recover gold, a prolonged soak in aqueous caustic cyanide is required. However, since gold values of about 5 mg/t Au have been determined in (unfiltered) cyanide leach solution (see Table I), it seems that calcium aurocyanide could be slightly soluble. As will be shown later, gold electrowinning is impossible from gold plant cyanide leach solutions containing calcium, indicating that calcium aurocyanide, if at all soluble, would not be significantly dissociated. Therefore this compound can be considered to be non-ionic and covalent in nature. This interpretation would explain many unexpected features related to gold recovery on existing gold plants.

Supporting historical evidence and implications

When compared with the mechanism of gold adsorption onto activated carbon^{3,4} the evidence for a calcium aurocyanide complex as predicted in Equation [3], is convincing. Thus the adsorption of gold as an insoluble or weakly soluble calcium aurocyanide covalent complex is strongly suggested, rather than the adsorption of an ion-paired calcium complex, as has been proposed⁵. This is because the impossibility of electrowinning gold from typical plant solutions indicates the adsorption of a more stable covalent coordination complex. The limited solubility of this calcium complex, when compared with the higher solubilities of potassium and sodium aurocyanides, would explain the preferential adsorption of calcium aurocyanide onto activated carbon (and possibly wood fibre originating from the milling circuit).

Poor gold adsorption may also be encountered due to the passivation of carbon surfaces with calcium carbonate. The origin of the calcium carbonate results from the catalytic oxidation of free cyanide in the contacting circuit with the formation of ammonia and carbon dioxide. This carbon passivation is very sensitive to the pH of the pulp. While calcium loadings below 20 kg/t carbon may not have a serious effect on the kinetic response, loadings of 40 to 60 kg/t have a detrimental effect on gold adsorption. The

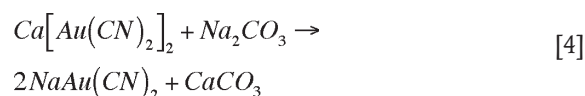
Table I
Absence of electrowinning of gold from leach solution/slurry

Temp. (°C)	Time (min)	Residual gold (mg/t)
Electrowinning from leach solution ^A		
0	0	6.74
20	20	6.72
55	55	6.66
Electrowinning from leach slurry ^B		
27	0	4.77
28	5	4.81
30	15	4.65
32	25	4.75

A: no U/S; C felt / Ni/Cr foam cathode; 1.0 A; flow-through system
B: U/S; Ni/Cr foam cathode; 0.5 A; U/S agitation

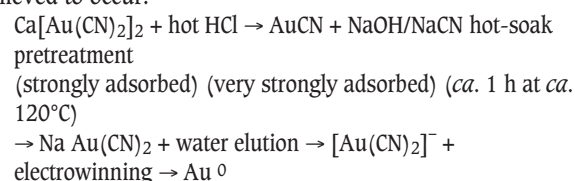
deleterious effect on leaching caused by excessive additions of calcium in the leach circuit⁶ may also be explained by the adsorption of this calcium aurocyanide complex by gangue materials⁷.

While calcium appears to play a significant role in the selective adsorption of gold onto activated carbon, its removal from the loaded carbon prior to elution and the subsequent electrowinning of gold from the pregnant eluate is also very relevant. In earlier elution test work using deionized water as an eluant⁴, carbon pretreatment at 90°C using potassium and sodium carbonates resulted in good gold elution. This was ascribed to the following exchange reaction mechanism:



The better elution recoveries obtained with potassium carbonate implied that potassium aurocyanide was less firmly adsorbed than was sodium aurocyanide. This may be expected when the greater solubility of potassium aurocyanide is considered². In the case of calcium aurocyanide, which is even less soluble than the sodium complex, the adsorption bonding may be expected to be even stronger. Current mine practice at Kloof Gold Mine and elsewhere for eluting gold-loaded activated carbon (14 kg/t Au) involves a hot soak with 2–3 per cent NaOH for up to 1 h at 120°C under pressure, followed by a 3.5 h elution with water at 120°C. This emphasises the extreme measures required to release the gold from the activated carbon. Even under these intense conditions not all the gold is released. Interestingly, a lower concentration of NaOH allows for better release of gold, which implies that there is an equilibrium between gold release and gold re-adsorption, with too high a sodium ion concentration favouring re-adsorption.

When using the original AARL elution procedure^{5*}, calcium is first removed from the activated carbon using a hot acid wash prior to activated carbon pretreatment and subsequent elution with softened water. The following is believed to occur:



When related to elution in particular, the hot acid washing of carbon prior to elution serves several purposes.

- Under acid conditions, the elution system can be brought to operational temperatures using preheated water while not eluting any gold or silver values from the carbon. This preheating of the elution circuit to the required operating temperature ensures the subsequent rapid and efficient elution of gold in a well-defined profile.
- In the case of severe calcium passivation, acid washing has a very beneficial effect on subsequent gold elution.

*A modified procedure at the Kloof gold mine and others utilizes cold HCl and no addition of NaCN

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- It has also been adequately demonstrated⁵ that elution efficiency is very sensitive to water quality, and to the presence of calcium in particular. Hot acid-washed carbon (1.2 kg/t Ca) was found to be less sensitive to water quality than either the cold acid-washed carbon (5.4 kg/t Ca), or nonacid-washed carbon (23 kg/t Ca).

The fact that a hot acid-wash removes more calcium than a cold acid-wash is consistent with the idea that calcium is present on the carbon in two forms, viz., as a readily soluble calcium carbonate and as a very stable calcium aurocyanide complex. As mentioned above, it requires rather severe conditions of high temperature combined with excess sodium hydroxide, in order to release the gold species from the activated carbon. The same is true for the Zadra process⁸ where free cyanide is present as well, and where electro-winning is carried out continuously at temperatures of > 90°C.

In the case of electrowinning, unpublished work by Davidson at the then Anglo American Research Laboratories in 1972, and by Sole, as reported in the present paper, showed that it was impossible to recover gold from pregnant plant solutions. The formation of a stable, non-ionic, covalent, coordinated calcium aurocyanide complex, rather than the dissociated aurocyanide anion predicted in Equation [1], would explain why no electrowinning is possible. This is because the relevant aurocyanide complex must be disassociated and the ions so formed charged, as is the case with sodium aurocyanide, so that reduction at the cathode of the aurocyanide ion to gold may take place under the action of the electric field.

If the formation of the calcium complex in Equation [3] is responsible for the inability to electrowin gold from plant solutions, it must also follow that the presence of calcium in the electrowinning circuit must be kept to a minimum. However, as will be shown later, the presence of excess sodium ions at elevated temperature does allow electrowinning to take place in pregnant leach solutions containing calcium.

Furthermore, as calcium is a well-known catalytic oxidizing agent for the water-gas reaction, hot acid washing prior to thermal regeneration will minimize carbon losses and maintain valuable physical carbon hardness. Thermogravimetric evidence supporting this conclusion was presented in a review by Davidson⁵.

In a lengthy investigation² into the intensive cyanidation of gravity concentrates (6–22 kg/t Au) the solubilities of both potassium and sodium aurocyanides were found to be critical. The reaction variables, including pH, cyanide concentration and time, had very complex compounding effects, resulting in some poor recoveries, which were not adequately explained. While the use of potassium cyanide invariably gave the best leach recoveries, the presence of variable contents of calcium (200–2000 g/t Ca) and even of magnesium (500–2500 g/t Mg), may well have affected overall gold recoveries from such concentrates. The formation of a diffusion barrier by precipitated calcium and/or magnesium complexes on reaction surfaces was suspected at the time.

Recent investigations into gold electrowinning

These studies were carried out on a small scale in the

laboratory of Sole. They were done in parallel with pilot-plant investigations at the Kloof Gold Mine (Gold Fields Ltd.), and provided very useful guidelines and insights into what was happening on a larger scale. In particular, it was found that while electrowinning was possible from eluate solution containing < 10 mg/l Au, in the form of dissociated sodium aurocyanide, it was not possible from leach solutions or slurries of similar gold concentration. However, when excess sodium ions were added to the latter so as to solubilize the calcium aurocyanide present, presumably by a type of cation exchange mechanism (see Reaction [4]), then electrowinning did occur. In the absence of current and sodium ions, but using an activated carbon electrode, excellent gold extractions from leach solution occurred as a consequence of the adsorption of calcium aurocyanide. It was these electrowinning investigations, the details of which follow, that subsequently triggered the re-examination of the role calcium plays in gold plant operations.

In future work it is proposed to investigate cyanide leaching of gold from ore by comparing the efficiencies and kinetics of leaching when using sodium hydroxide or lime, respectively, in order to control pH. In the case of the former an attempt will be made to compare gold recoveries using conventional CIP, where adsorption of calcium aurocyanide is key, with recoveries achieved using direct electrowinning, where the presence of dissociated sodium aurocyanide is essential.

Experimental

A standard 5-litre ultrasonic cleaning bath was filled with water which could be heated by means of a fish-tank heater. A rectangular plastic container fitted with a lid was located in the water. It contained the electrolyte, the stainless steel mesh anodes, and the relevant high surface area (HSA) cathode, thereby constituting the electrochemical cell used in these experiments. Initially mixing and agitation were achieved by the action of ultrasonics, but in later experiments a pump was also added to the system, and the electrolyte recirculated and forced to flow through the HSA electrode. Current was supplied by a small DC rectifier.

Various HSA electrodes were used. Most of the initial work was done with cathode strips of Ni/Cr metal foam, with typical dimensions of ca. 100 × 30 × 5 mm, and with an open pore size of 400 or 600 microns. Later sandwich electrodes consisting of two metal foam sheets, with either carbon felt or activated carbon granules sealed in between them, were used. Finally, an activated carbon adsorption electrode was developed. This consisted of a double sandwich in which two compartments containing more activated carbon were sandwiched between a central and two outer stainless steel meshes, respectively. In the later experiments the electrolyte was forced through the electrodes.

Experiments were conducted on gold leach solutions and slurries typically containing about 5 mg/l Au. Solution samples were taken as a function of time, under the various experimental conditions of each run, and analysed for gold.

Results and discussion

Previous test work on eluate solutions with gold concentrations in the range 7–1100 mg/l Au had shown that both on a small scale and on a pilot-plant scale, very good gold

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extractions (> 98 per cent in 3–4 h) could be achieved by electrowinning at temperatures of *ca.* 50°C and using ultrasonics (U/S). When similar small-scale test-work was carried out on gold leach solutions and slurries, it was found that no electrowinning was possible, as shown in Table I. Similar negative results were obtained in the pilot plant, when leach solutions were treated for 3 hours at 50°C under ultrasonic conditions

Initially it was thought that the absence of electrowinning was due to a pH or low conductivity effect, and so sodium hydroxide or sodium carbonate was added to the leach. Gold extractions of up to 97 per cent were then achieved. The findings are illustrated graphically in Figure 1 and comparing the results with those of Table I, it is evident that even leach slurry (Run 1), is amenable to electrowinning in the presence of sodium hydroxide. Run 2 shows that the same is true for leach solutions under more favourable operational conditions. Run 3 shows that the addition of sodium carbonate has the same effect, the kinetics being somewhat faster because of the higher temperature used.

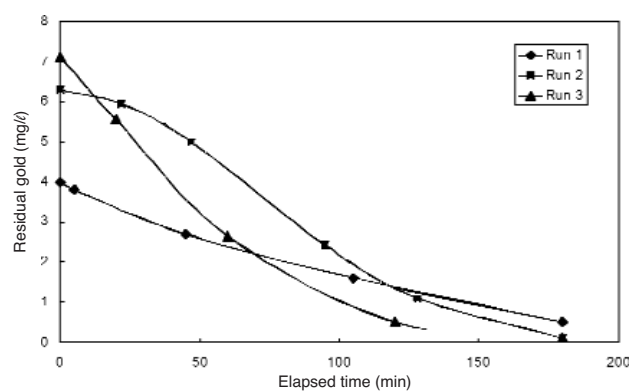
It is therefore clear that the addition of sodium ions to either leach slurry or solution will, under appropriate conditions of pH, temperature and ultrasonics, enable gold electrowinning to take place—in the case of leach solution to the extent of 97 per cent extraction in 3 h. It is therefore now suggested that under such operating conditions there is an exchange of calcium and sodium ions, resulting in the release of $[\text{Au}(\text{CN})_2]^-$ ions from which gold can then be electrodeposited. Thus, the exchange mechanism related to the pretreatment of carbon prior to elution (see Equation [4]) would seem to apply here as well.

These tests were carried out using metal foam cathodes, but later it was decided to explore the phenomenon further using sandwich cathodes of metal foam and either carbon felt (which has a very high specific surface area) or activated carbon of the type currently in use on the mines. In these latter tests the effects of current reversal, or no current at all, were also explored. The results are illustrated in Figure 2. In Run 4 a carbon felt sandwich cathode was used, with current applied from the start of the test. As before, no electrowinning took place, but when sodium carbonate was added after 1 h of operation gold extraction commenced immediately, even at a temperature as low as 35°C. Here the very high surface area of the carbon felt may have contributed to the reaction at lower-than-expected temperature. When compared with runs 5, 6 and 7 where activated carbon was used and gold extraction did take place in the absence of current, it is clear that gold adsorption plays no role at all in the case of carbon felt.

The remaining runs employed activated carbon sandwich cathodes. For Runs 5 and 6 the (forward) current was applied only some 10–15 minutes after the cathode had been submerged in the leach solution, and yet during this time very fast gold extraction occurred. The shape of the extraction curve was not materially affected when current was applied, nor when it was later reversed, clearly indicating that current had little or no effect. However, in Runs 5, 6, and 7 there was a consistent deviation from the norm for a short time when the current was reversed. This could be due to a transient cathodic electrodeposition of gold onto the former anode of stainless steel mesh. Nevertheless, the indications are that

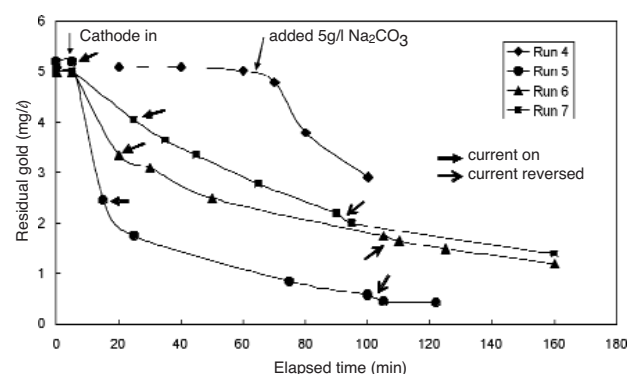
gold extraction was largely due to adsorption of gold by the activated carbon. The faster kinetics of Run 5 may be attributed to the use of fresh activated carbon, whereas in Run 6 the carbon was reused. In Run 7, in which 3 g/l sodium carbonate was added before the start of the run, similar effects may be noted for the role of the applied current. However, the initial kinetics are slower, suggesting that the presence of carbonate ions may have had a poisoning effect on the (outer) surfaces of the activated carbon. In the latter stages of the tests after about 90 minutes, the slopes of the curves were all similar, suggesting again that the main reaction occurring was that of gold adsorption by activated carbon. In practice, however, lime is normally used to raise pH during the cyanide leach operations, and the later addition of sodium hydroxide or carbonate would not appear to be an economic proposition.

The possibility of using a sandwich electrode incorporating stainless steel metal mesh, with additional activated carbon sandwiched inside, was next explored. The results are shown in Figure 3. In the absence of current and added sodium ions, it is evident from Figure 3 that the extraction of



Experimental conditions
Run 1: Slurry, U/S, 8 g/l NaOH added, 39°C, 0.5 A.
Run 2: Solution, U/S, 5 g/l NaOH added, 56°C, 1.0 A.
Run 3: Solution, U/S, 5 g/l Na₂CO₃ added, 60°C, 1.0 A

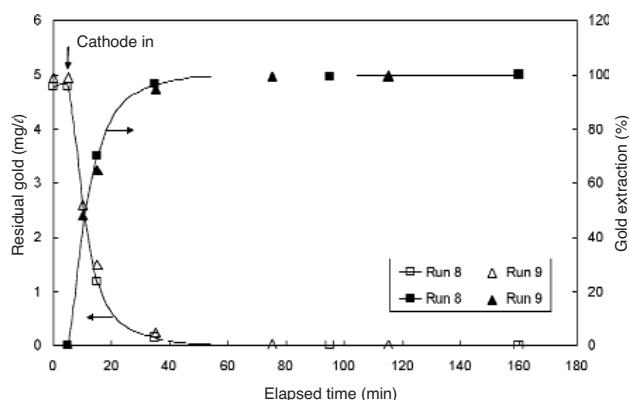
Figure 1—Effects of the addition of sodium ions to leach solutions and slurry on gold electrowinning



Experimental conditions.
Run 4: Carbon felt, intermittent U/S, 35°C, 1.0 A.
Run 5: Activated carbon, intermittent U/S, 30°C, 1.0 A.
Run 6: Activated carbon (flow through), U/S, 30°C, 1.0 A.
Run 7: Activated carbon (flow through), U/S, 3 g/l Na₂CO₃ added, 32°C, 1.0 A.

Figure 2—Effects of sodium ions on gold electrowinning from leach solutions using carbon cathodes

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Experimental conditions
Run 8: No U/S, 29°C
Run 9: Intermittent U/S, 28°C

Figure 3— Gold extraction using a flow-through activated carbon electrode

gold can be due only to gold adsorption on to the activated carbon inside the electrode. In Run 8 no ultrasonics was used and in Run 9 intermittent ultrasonics was employed. The results are almost identical. Thus, at near-ambient temperatures excellent extractions of 98 per cent in 1 h and 99.8 per cent in 3 h could be achieved without current. This suggests that the weakly soluble calcium aurocyanide complex is very quickly and strongly adsorbed on to the activated carbon and that this occurs at near ambient temperature. It was also established that the gold content of the gold-loaded carbon increased from 165 ppm to 522 ppm, while the calcium content increased from 0.113 per cent to 0.377 per cent during the course of these experiments. These results also indicate the overriding effect of adsorption by activated carbon, when compared with electrowinning (or electrodis-solution), in the extraction of gold from such solutions. The increases in gold and calcium contents of the activated carbon during use again support the theory of an adsorbed complex of calcium and gold.

As a consequence of these findings a pilot-plant investigation was initiated at Kloof Gold Mine to examine the possibility of electrowinning gold using a gold-loaded activated carbon anode. The anode typically contained about 7 kg activated carbon. The gold loading was about 14 kg/t Au. The electrolyte was similar to that used in previous gold electroforming tests⁹, and contained 2–3 per cent NaCN, 4–5 per cent NaOH, and about 700 mg/t Au. Preliminary experiments showed that the gold, present in the electrolyte as sodium aurocyanide, was adsorbed onto the carbon in the absence of current, causing the gold loading to increase to about 21 kg/t Au, and leaving the gold concentration in solution at about 60 mg/t Au.

The electrowinning runs which followed showed that, while electrowinning was indeed possible at *ca.* 50°C and using ultrasonics, the cathode efficiency for electro-deposition was low (1–2 per cent). Moreover, the alloy so formed was very high in silver (up to 35 per cent Ag). This suggested that the strength of the bonding of argentocyanide complexes to activated carbon was much lower than that of corresponding aurocyanide complexes. Furthermore, the

initial gold adsorption from solution implied that while anodic dissolution of gold was taking place, there was a simultaneous reverse adsorption process occurring. In view of this, the loaded activated carbon was then pretreated with ethanol or xylene, in order to poison the free surfaces of the carbon with an organic and thereby inhibit re-adsorption. Improvements in both yield and gold content of the alloy resulted. It is also interesting to note that the alloy deposits on the polished stainless steel cathode were of reasonable quality, bearing in mind that the average gold concentration of the electrolyte during electrodeposition was usually below about 50 mg/t Au and that quite high cathode current densities (0.5–1.0 A/dm²) were used.

Clearly, the bonding between activated carbon and both calcium and sodium aurocyanide complexes is very strong and requires highly elevated temperatures, as used in carbon-in-pulp (CIP) elution procedures, in order to release the gold effectively. Thus, despite the improvements noted, electrowinning conditions involving an electric potential of up to 10 V (with pulse plating), a temperature of 50°C, the presence of ultrasonic agitation, and with an excess of cyanide ions present, this was still not sufficient to make the process effective. However, electrowinning by this method¹⁰ remains a possibility at temperatures in excess of 90°C, as is the case for the Zadra process⁸.

In view of the electrowinning evidence presented, it seems pertinent to investigate the use of sodium hydroxide and/or sodium carbonate and/or sodium bicarbonate as alternatives to lime during leach operations. Such sodium compounds would increase the ionic strength of this so-called 'soft' leach¹¹ and provide a means of buffering the operating leach pH at the optimum value of 10.5, thereby improving process control and reducing reagent consumption. By increasing the solubility and the dissociation of the leached reaction products, more efficient leaching of gold, particularly for more complex gold ores, would result. Instrumental control would also be simplified in a sodium-based circuit and safety aspects would be improved.

Under such conditions gold would, in the case of CIP, then be adsorbed onto carbon as the ion-paired sodium aurocyanide complex. Adsorption should be increased as a result of the higher ionic strength, with both the rate of gold adsorption and the equilibrium gold loading increasing. Furthermore, no regular acid washing of the carbon would be required, as calcium carbonate fouling arising from the use of lime would not take place, and so reagent consumption would be further reduced. In addition, carbon pretreatment and elution might well be possible at lower temperatures, because of the absence of the very strongly adsorbed calcium aurocyanide complex. In the absence of calcium more efficient regeneration of carbon would be possible, together with a better retention of the physical hardness of the carbon.

Finally, if the above 'soft' leach with sodium hydroxide and/or sodium carbonate and/or sodium bicarbonate was followed, it has now been shown that the direct electro-winning gold from such solutions and slurries could then be feasible. Such an alternative, if cost-effective, would eliminate the use of activated carbon and all the associated procedures that this step involves. It should be possible to offset the higher cost of sodium-based reagents by recycling them to the grinding/mixer-settler circuit. An electrowinning-

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in-leach process¹² has been suggested, which incorporates a tall leach vessel with slurry recirculation. The majority of the slurry resides as an expanded bed in the bottom of the vessel and the relatively clear leach solution at the top is then re-circulated through a separate electrowinning reactor. Such a reactor could be based on either metal-foam electrowinning cathodes followed by a gold electroforming step^{9,13}, or on fluidized-bed cathodes^{14,15}. Both rely on HSA-type cathodes that are suitable for the extraction of gold at low concentration levels. If the latter type of electrowinning reactor proved effective this would eliminate yet another stage in the overall process of gold recovery, as the product (gold balls) could be sent directly to the refinery for further purification.

While the above views relating to the use of a 'soft' leach are somewhat speculative and would require further investigation, they do emphasize yet again the important role played by calcium in existing gold circuits.

Conclusions

A number of conclusions arise as a consequence of this review and investigation. If the reaction proposed in Equation [3] does take place in gold plant leach circuits, then the previously held view as predicted in Equation [1] is perhaps misleading. As outlined above, the production of a covalent coordination calcium aurocyanide complex with a limited solubility can explain many plant observations, which previously were not well understood. This conclusion is supported by the electrowinning results reported here for the first time. The adsorption behaviour of this specific complex onto activated carbon would also agree with the earlier and present findings relating to the mechanism of gold adsorption.

The second conclusion relates to calcium in the up- and downstream operations of carbon-based circuits. For example, the linear screens used for wood-fibre removal, as well as the CIP inter-stage screens would operate more efficiently in the absence of calcium. Also, the passivation of activated carbon surfaces would be reduced with little or no calcium ions present. At present efficient hot acid washing of loaded carbon prior to elution is strongly advocated, as the presence of calcium in plant eluates, electrolytic circuits, as well as in the regeneration stage, appears to have detrimental effects for these stages.

Thirdly, the need for calcium (as the hydroxide) in the leach circuit is questionable due to the adsorption of gold as calcium aurocyanide onto gangue materials. More efficient leaching should undoubtedly result if the leach circuit were calcium-free.

Finally, the electrowinning findings reported here and their relationship to the presence or absence of calcium aurocyanide in gold plant cyanide leach solutions and slurries, strongly suggest that it may be possible to electrowin gold in sheet or spherical form directly from such solutions or slurries. In this case the CIP and other operational stages in gold extraction would be eliminated and the gold product could be sent directly to a refinery for further purification. If this scenario proves viable the overall process for recovering gold could be revolutionized world wide.

Acknowledgement

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