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Procedure for determination of preg-robbing in gold ores

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Test Method to determine of preg-robbing gold ores

Abstract

A new method utilising standard additions of gold for the initial characterisation of preg-robbing potential for a variety of ores is proposed. The method is compared with the Barrick Gold Mines Incorporated (BGMI online) preg-robbing test currently used in industry and shows good correlation for carbonaceous ores. It is shown that for silicate and sulphide ores the residence time of the BGMI preg-robbing test is too short to allow equilibrium to be achieved and in all cases it was demonstrated that allowances in the BGMI preg-robbing test need to be made for gold leached from the ore.

Keywords: Precious metal ores; Leaching

1. Introduction

Gold losses during processing are a major problem throughout the gold mining industry and if not properly identified, they can lead to significant amounts of gold reporting to tailings. One of a number of phenomena by which these gold losses occur is known as preg-robbing, whereby constituents of the ore adsorb the aurodicyanide complex from solution. These ore constituents can be carbonaceous matter present in the ore or they can be the minerals themselves, such as sulphides or silicates.

The characterisation of the preg-robbing potential for gold ores is of great importance to processing. While a number of methods exist for fast online determination of this phenomenon little work has been performed on the initial characterisation of preg-robbing and its relation to refractoriness.

2. Preg-robbing

The most common and well documented cause of preg-robbing is carbonaceous matter present in the ore. This can be in the form of heavy hydrocarbons, organic acids or natural carbon (Osseo-Asare et al., 1984). Of this material, native carbon is the most important species for preg-robbing. Heavy hydrocarbons do not interact with gold (Osseo-Asare et al., 1984) and although Radtke and Scheiner (1970) suggested that organic acids could adsorb gold, Stenebraten et al. (1999) demonstrated recently by GC-MS that they were unlikely to be a major factor in preg-robbing. Stenebraten et al. (2000) showed for ore from the Carlin trend, Nevada that the preg-robbing characteristics were inversely correlated to the L_c (002) crystallite dimension and directly related to the d-spacing of the carbonaceous material.

The other documented causes of preg-robbing are the minerals themselves, predominantly either sulphides or silicates. A number of investigations into the preg-robbing power of sulphide minerals have been performed. Quach et al. (1993) showed that chalcopyrite and

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pyrrhotite adsorbed gold from oxygen free solutions and Rees and van Deventer (2000) demonstrated a clear cyanide concentration dependence for preg-robbing on pyrite and chalcopyrite with very strong adsorption of gold in cyanide deficient solutions. Hausen and Bucknam (1984) also looked at the adsorption of gold on pyrite but suggested that preg-robbing would only occur if it constituted more than 40% of the ore.

Silicate minerals, especially clays have also been proposed as potential preg-robbers. It has been suggested that the positively charged edge surfaces of clay particles could attract negatively charged colloidal gold (Hausen and Bucknam, 1984). This can be seen in an electron micrograph presented by van Olphen (1977) that shows negative colloidal gold particles attached to positive kaolinite edges, although this was in acid solution and adsorption would be greatly reduced in an alkaline environment. A study by van Vuuren et al. (2000) on shale material from the Beatrix gold mine in South Africa demonstrated that gold was not adsorbed but that decomposition of phyllosilicates caused co-precipitation of gold with $Mg(OH)_2$ and $Fe(OH)_3$ phases. The propensity of gold to adsorb to clay minerals was also investigated by Hausen and Bucknam (1984) who showed that illite, kaolinite, montmorillonite and pyrophyllite did adsorb gold but to a much lesser extent than carbonaceous matter.

The mechanism by which preg-robbing occurs is largely dependent on the constituents in the ore causing the problem. Menne (1992) suggested that preg-robbing mechanisms can be divided into two types. Reversible (Type I) preg-robbing occurs by simple ion exchange of the large aurodicyanide anion. This type of preg-robbing is common to some extent in most ores, but in most cases is reversible in the presence of activated carbon or by washing. Irreversible (Type II) preg-robbing is considered so because of the long time or unusually severe conditions required to redissolve the gold (Menne, 1992).

Type I preg-robbing provides few problems in modern CIP/CIL circuits if the activity of the ore constituents is less than that of activated carbon, however, constituents with a greater activity will compete strongly for adsorption of the aurodicyanide complex. It has been shown for a number of ores, most notably those from Carlin, Nevada that native carbon will compete with activated carbon in this way (Osseo-Asare et al., 1984). The adsorption of gold onto native carbon is thought to follow much the same mechanism as the adsorption to activated carbon in a CIP/CIL circuit (Schmitz et al., 2001a,b) and although the number of active sites is smaller, it has been demonstrated that the adsorption kinetics are up to 4 times faster for native carbon (Hausen and Bucknam, 1984). For sulphide ores (Rees and van Deventer, 2000) suggested that preg-robbing was cyanide concentration dependent. In the presence of free cyanide it was shown that preg-robbing was unlikely to occur and any that did occur was easily reversed by the addition of activated carbon.

Irreversible (Type II) preg-robbing involves precipitation of the gold complex and can occur by a number of separate mechanisms (Menne, 1992). The first mechanism (Type IIa) involves a lack of available cyanide causing the aurodicyanide complex to be stripped of one radical and the resulting auromonocyanide radical to form long chains, which are only vulnerable to attack from the tips making redissolution possible but very slow (Menne, 1992). A similar mechanism was presented by Rees and van Deventer (2000) who showed that aurocyanide was reduced to the surface of chalcopyrite in a cyanide deficient environment. The second mechanism for irreversible preg-robbing (Type IIb) is described by Menne (1992) as co-precipitation of gold with metal cyanides.

3. Preg-robbing analysis on the lab scale

A number of tests exist that are used extensively in industry. The simplest and most widely used method for determination of preg-robbing is the Barrick Gold Mines Incorporated (BGMI) preg-robbing test. This test is described by Schmitz et al. (2001a,b) and involves the addition of 10 ml of 2.0 g/l NaCN solution with 3 ppm gold to 5.0 g of ore. The resulting slurry is then equilibrated for 15 min and the solution analysed for gold. The concentration of gold lost from solution is then compared to the original concentration and a percent preg-robbing (%PR) value is determined. A similar method was proposed by Hausen and Bucknam (1984) that employed a longer residence time and determined a preg-rob value that was simply stated as the concentration of gold lost from solution. These methods were both developed for ores from the Carlin trend but are equally applicable to other preg-robbing ores.

The existing methods for determination of preg-robbing, although widely accepted, have been designed for online examination of changes in ore type. This is applicable to a production setting where relative changes in preg-robbing effects are of highest importance. However, these quick methods are not as applicable for initial characterisation of the preg-robbing potential of an ore and its relation to other ore characteristics because of uncertainty caused by the use of small sample sizes and leaching of gold from the ore within the residence time of the trial. A further development on the existing methods was the use of standard additions (Bader, 1980) to ensure reliability by using multiple assays to determine each preg-rob value. This approach placed more emphasis on the characterisation of preg-robbing as a stage of initial ore characterisation studies.

4. Methods and materials

4.1. Objectives

The method proposed in this study and referred to as the preg-robbing potential (PRP) method addresses the characterisation of preg-robbing by utilising representative sample sizes and a residence time sufficient to ensure equilibrium is attained. By addressing these issues it was possible to reliably determine the preg-robbing potential of an ore, not only in conventional cyanide leaches but also in other hydrometallurgical assay techniques such as aqua regia digestion.

The objective of this study was to develop a method capable of reliably determining preg-robbing in any type of ore and for this reason ores containing each of the main preg-robbing constituents were selected. A silicate ore (Ore 1) and a sulphide ore (Ore 2) from the Pilbara region of Western Australia were chosen to demonstrate the usefulness of the proposed method of showing preg-robbing in any type of ore. A known preg-robbing carbonaceous ore (Ore 3) from the western district of Victoria, Australia was also analysed to indicate how the proposed method compares with existing methods for the characterisation of preg-robbing in carbonaceous ores.

4.2. Sample preparation

The importance of using a sample size representative of the ore body is apparent in all assay work and is especially important for gold analysis. The presence of coarse free gold can result in unreliable assays due to a phenomenon known as the 'nugget effect'. This can be reduced by careful sample preparation but can only be eliminated by the use of larger samples to minimise error.

All samples were received from site as +1 mm crushed ore. Samples were split using a rotary splitter and dry ground to a P80 of 75 μm in a Labtechnics ring mill.

Ore mineralogy was determined by X-ray diffraction (XRD) analysis using a Philips PW 1800 X-ray diffractometer. Copper K radiation, a graphite monochromator and a proportional detector were used to collect the diffractogram. Results of this analysis are given in Table 1. The head grade of each ore is shown in Table 3. For Ores 1 and 2 this was determined by fire assay analysis and for Ore 3, it was determined by aqua regia digestion.

Table 1
Ore Mineralogy determined by XRD analysis

Ore 1	Ore 2	Ore 3
Quartz	Quartz	Quartz
Magnesite	Ankerite	Chlinochlore
Dolomite	Chlorite	Pigeonite
Chlorite	Pyrite	Pyrite
Anorthite	Calcite	
Talc	Muscovite	

4.3. NaCN leaching

Leaches were performed using four 3L baffled reactors and agitated by an overhead stirrer with a Rushton impeller. Two litres of 1.0 g/l NaCN solution at pH greater than 10.5 was added to each reactor. The solutions were respectively spiked with 0 ppm, 1 ppm, 2 ppm and 5 ppm of gold in the form of 1000 ppm AuCl standard solution. Solutions were agitated for 30 min to allow complete conversion of AuCl to $Au(CN)_2^-$ with a 10 ml aliquot taken after this time to ensure equilibrium had been reached. Finally, an optimized sample size of split and ground ore was added to each vessel and the slurry agitated for 24 h to allow complete equilibrium to be attained. After this time solutions were allowed to settle for 1 h and a 20 ml aliquot taken for gold analysis.

Gold analysis was performed by solvent extraction into DIBK/Aliquat 336 and analysed on a Varian SpectraAA 800 Atomic Adsorption Spectrometer (AAS) at a wavelength of 242.795 nm.

Analysis of representative sample sizes is an important factor in any gold study because of the inherent heterogeneity of gold in its ores. Extensive examination of sampling theory has been performed by Pitard and Gy (1989) and nomographs describing the optimum sample size to achieve representivity dependent on gold concentration and particle size have been presented. This sampling theory relies upon an understanding of both the gold concentration and the average gold particle size, however although the concentration is generally known for initial characterisation the average gold grain size is not. For this reason the most representative sample size for each ore was calculated by an empirical method utilising the standard deviation of results over 30×25 g aqua regia digestions and extrapolation of this data to determine the sample size representing an error of 5%. The optimum sample size determined for each ore investigated in this study is shown in Table 3.

4.4. Analysis

Leaching results were graphed and compared to the values expected if preg-robbing was not present. The difference of the slope from unity was taken as the preg-robbing potential (PRP) of the ore. This was calculated by Eq. (1) and is quoted as the percent preg-robbing potential (% PRP).

$$\% \text{ PRP} = (1 - m) \times 100 \tag{1}$$

where % PRP = preg-robbing potential, m = slope.

It is also possible to predict the recoverable grade if preg-robbing did not occur using the PRP method. This is calculated by Eq. (2) and is referred to as the inferred grad

$$x = \left(\frac{c/m}{C}\right) \times 100 \qquad (2)$$

where x = inferred recovery (%), c = recovered grade with preg-robbing (g/t Au), C = head grade (g/t Au), m = slope.

5. Results and discussion

In characterisation of the preg-robbing potential for an ore its behaviour in NaCN leaches is of most interest to industry. Initially the preg-robbing potential (PRP) method was demonstrated using blank samples of metallurgical sand to show that linearity could be achieved and that precipitation of the aurodicyanide complex would not mask preg-robbing. Results of the blank analysis are shown in Fig. 1. The data shows that although there is a small deviation from linearity, the error is less than 5%. This is considered inconsequential.

Also shown in Fig. 1 are the results of a trial with the addition of 1% w/w activated carbon to simulate the effects of a strong preg-robber. It can be seen from this that all the gold was adsorbed to the activated carbon. It would be expected that most preg-robbing ores would fall somewhere between these two extremes.

Initial analysis of each of the three ores being studied was also performed using the BGMI preg-robbing test with no addition of a gold spike. This was performed to demonstrate if leaching was occurring within the residence time allowed for this method. Results of this test are shown in Table 2 and clearly indicate that in all cases a proportion of the gold present in the ore was leached in the first 15 min. This suggests that preg-robbing could be masked or significant leaching occurs in this time.

Table 2
BGMI PR test with no gold spike added

	Ore 1	Ore 2	Ore 3
Gold leached (ppm)	0.36	0.99	0.24
% Recovery	5.45	12.61	15.6

The preg-robbing potential (PRP) method was initially tested using Ore 1. This ore consisted predominantly of silicate and carbonate minerals and hence was not expected to show significant preg-robbing. The results of this analysis can be seen in Fig. 2 and Table 3 and indicate that as expected preg-robbing was not a major factor in the leaching of this ore. Although characterisation of the preg-robbing potential of Ore 1 showed it to be low it should be noted that the recovery value indicated that the ore was highly refractory. Combining these results in the context of initial characterisation of the leaching behaviour for Ore 1 indicated that although preg-robbing was not an issue further characterisation of the refractory behaviour would be required.

The importance of using a representative sample is highlighted by Ore 1. Although the most representative sample size had been determined for Ore 1 (see Table 3) it was found that this was too large for analysis and hence a smaller sample size of 1 kg was used resulting in significant errors. The large sample size required for Ore 1 was caused by a large proportion of gold occurring as coarse free gold. This problem occurs in many ores and although it is generally amenable to gravity recovery, this results in extensive problems in obtaining representative assays.

Analysis of the preg-robbing potential for Ore 1 by the BGMI preg-robbing test can be seen in Table 3 as "PR". It was apparent that significant preg-robbing

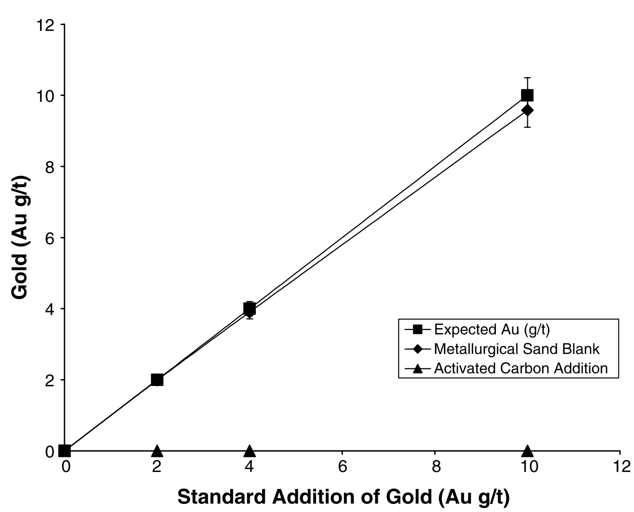

Fig. 1. Preg-robbing potential blank tests. Error bars represent 5% error.

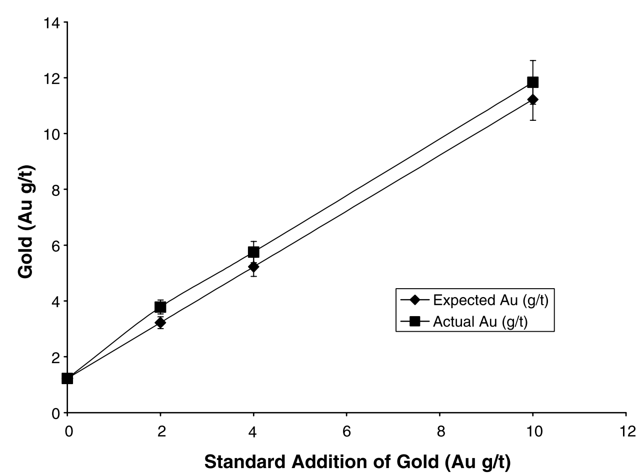

Fig. 2. Ore 1 preg-robbing potential. Error bars represent 6.6% error.

Table 3
Results of preg-robbing tests for Ores 1, 2 and 3

Ore	Head grade (g/t Au)	Optimum sample size (g)	PRP %	Recovery (%)	Inferred recovery (%)	PR (%)	Adjusted PR (%)
Ore 1	6.62	2250	0	22.2	22.2	15.6	27.6
Ore 2	7.85	432	12.03	54.82	62.31	0.1	33.1
Ore 3	1.54	50	29.12	51.95	73.38	15.2	23.2

was suggested using this method, which was in direct contradiction to the results of the PRP method. It is postulated that this was caused by initial weak adsorption or precipitation of aurodicyanide to silicate minerals present in the ore, which was reversed as the system approaches equilibrium. This time was obviously longer than 15 min and hence apparent preg-robbing was seen in the BGMI PR test. Although the length of the PRP method precludes it from use as an online method for determination of preg-robbing this result indicated that care should be taken when applying methods utilizing short residence times to non-carbonaceous ores.

Analysis of Ore 2 by the PRP method was performed as a demonstration of the method using a potentially preg-robbing sulphide ore. Results of this test are shown in Fig. 3 and Table 3. It was apparent that weak preg-robbing was occurring in the ore, which was consistent with preg-robbing by sulphide minerals in the presence of free cyanide (Rees and van Deventer, 2000). The inferred recovery, although higher than the actual recovery was still very low (62.31%) and suggested that the major processing problem for this ore was a refractory component of the gold that must be released before being amenable to cyanidation.

Analysis of Ore 2 using the BGMI preg-robbing method is shown in Table 3. When these results were analysed without accounting for leaching from the ore it was apparent that no preg-robbing was occurring. It can however be seen from Table 2 that significant leaching occurred in the first 15 min for Ore 2 and if this was taken into account it becomes apparent that preg-robbing is significant for this ore. This effect was more pronounced than predicted by the PRP method although once again this could be attributed to initially faster kinetics of adsorption followed by reversal of the preg-robbing as the system comes to equilibrium.

Ore 3 was analysed as an example of how a known preg-robbing carbonaceous ore could be characterised using the PRP method. Results of the analysis for Ore 3 can be seen in Fig. 4 and Table 3. These verified that this ore was a strong preg-robber, as was expected. Although the inferred recovery is better than for Ore 2 the recovery for Ore 3 is still low and suggests that there is a refractory component in this ore as well. This highlighted the applicability of applying the PRP method to show the inter-relationship between preg-robbing and refractoriness during initial characterisation.

When correlated to the BGMI preg-rob method it can be seen from Table 3 that the PRP method compared reasonably well.

The correlation between the proposed PRP method for initial characterisation of preg-robbing and the BGMI method for its online examination has been shown to be good for carbonaceous ores but less applicable for other ore types. While this result was expected it highlighted that care should be taken in the circumstances for which the methods are applied. The PRP method has been designed to concentrate on initial characterisation of the preg-robbing potential and its relation to other leaching characteristics such as the

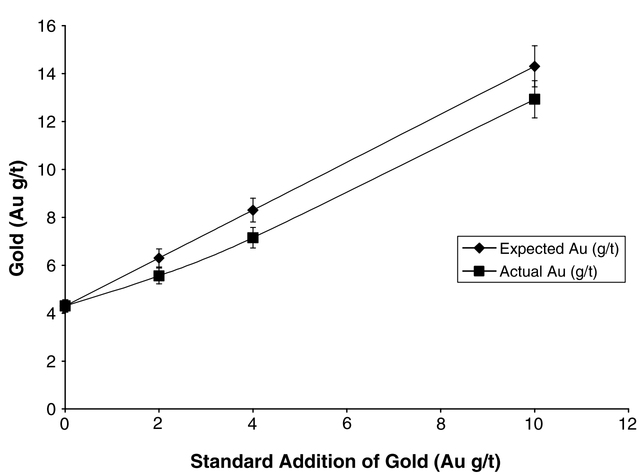

Fig. 3. Preg-robbing potential for Ore 2. Error bars represent 5% error.

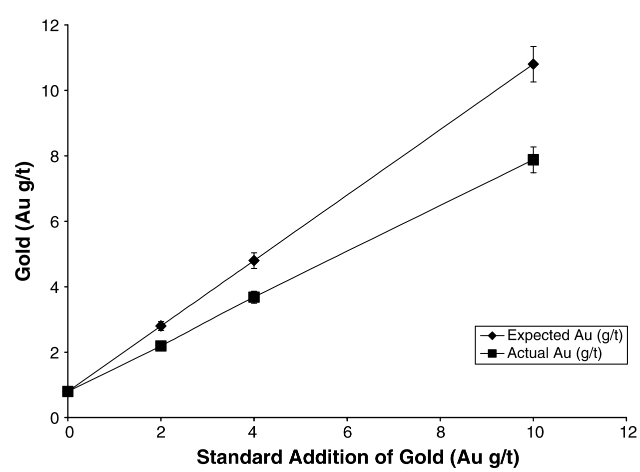

Fig. 4. Preg-robbing potential results for Ore 3. Error bars represent 5% error.

degree of refractoriness but the length of analysis required precludes it from online analysis. The BGMI and other online methods for determination of preg-robbing, although highly applicable in a production setting were shown not to give the same depth of information possible by the PRP method and hence are not as useful for initial ore characterisation.

It was noted that leaching of gold from the ores had a significant effect on the BGMI preg-robbing test, even with the short residence time used. This was more pronounced for ores containing significant fine gold with up to 15% of the gold leached from Ore 3. This suggested that the BGMI preg-robbing test would underestimate the total preg-robbing unless this was accounted for.

It was also shown that for silicate and sulphide ores the BGMI preg-robbing test indicated a higher preg-robbing potential than the PRP method. This suggested that initial preg-robbing was occurring by the type I mechanism in the form of weak adsorption or precipitation. This initial preg-robbing was reported by the BGMI preg-robbing test because of the short residence time but as the system came to equilibrium at longer residence times, it was reversed. It is therefore desirable to utilise longer residence times in characterisation of preg-robbing because it more accurately simulates plant conditions. This is not feasible for online tracking of changes in preg-robbing behaviour in a production setting but during initial characterisation should give a much better overview of the general leaching characteristics of the ore.

Overall, the PRP method was more accurate in the determination of preg-robbing by mineral constituents than the BGMI preg-robbing test for the ore types and preg-robbing potentials tested. As well as allowing for leaching of gold from the ore and giving sufficient time for the system to reach equilibrium, information on the expected recovery if preg-robbing was absent can also be gathered. This can be useful for determining if there is a refractory component of the ore and whether further pre-treatment of the ore is required to get acceptable recovery. This was apparent for all the ores in which the data suggests that even if preg-robbing was eliminated, economic recovery from these ores would still not necessarily be achieved. The inter-relationship between preg-robbing and refractoriness is an important point to consider during initial characterisation of the leaching characteristics of an ore to determine the most appropriate processing route.

6. Conclusions

It has been shown for the ore types analysed that the preg-robbing potential (PRP) method can be used effectively to characterise whether preg-robbing is a significant factor in the leaching of gold ores. The correlation of results obtained by this method with existing methods, such as the BGMI preg-robbing test are accurate for carbonaceous ores but when preg-robbing is caused by other constituents, such as silicates or sulphides, it has been shown that the PRP method is more accurate. This has highlighted the advantages of targeting different characterisation methods for varying situations.

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