

**STUDY ON SURFACTANTS FOR PASSIVATION OF NATURALLY OCCURRING  
CARBONACEOUS MATTER IN GOLD BEARING ORES**

\*Stamen S. Dimov and Brian Hart

*Surface Science Western, Western University  
999 Collip Circle  
London, ON, N6A 5B7, Canada  
(\*Corresponding author: sdimov@uwo.ca)*

**ABSTRACT**

The naturally occurring carbonaceous material (CM) in gold bearing ores often exhibits strong-preg-robbing properties. It can adversely affect the process of gold recovery during the cyanidation due to its ability to adsorb, or preg-rob gold from the cyanide leach solution.

Treatments of carbonaceous ores with chemical surfactants as blinding agents to limit preg-robbing have shown promising results but at the same time have demonstrated that, if not properly used, could have a strong detrimental effect on the gold recovery process. High dosages of surfactants used as passivating agents for carbonaceous matter could lead to unwanted partial passivation of the activated carbon added during the CIL process and severely impact the gold recovery. Optimal dosage for various surfactants would strongly depend on the ore mineralogy and the parameters of the pulp (temperature, pH, surface area and maturity of the carbonaceous material, etc).

This paper describes an experimental study on the effectiveness of various commercially available blinding agents used to minimize the preg-robbing capacity of naturally occurring carbonaceous matter. The scope of the study was to: i) establish the optimum addition of blanking agent (surfactant) to minimize the preg-robbing capacity of the CM in the ore, and ii) establish maximum threshold amounts of blanking agents to be used before it negatively impacts the preg-robbing capacity of the activated carbon used in the CIL process. The study procedure involves standardized doping tests to evaluate the maximum preg-robbing capacity of treated CM, coupled with surface analysis with ToF-SIMS to monitor the presence and the degree of loading of these chemical surfactants on both the naturally occurring CM and the added activated carbon.

**KEYWORDS**

Gold recovery, carbonaceous matter, preg-robbing, blanking agents

## INTRODUCTION

Naturally occurring carbonaceous material (CM) in gold bearing ores often exhibits strong-preg-robbing properties. Thus, it can adversely affect the process of gold recovery during cyanidation due to its ability to adsorb, or preg-rob gold from the cyanide leach solution. The preg-robbing properties of this naturally occurring carbonaceous matter can vary widely between ores, and within a single ore body (Dunne et al.,2013;Helm et al.,2009;Miller et al.,2005). The degree of preg-robbing related to the CM within an ore is defined by several parameters: total organic carbon (TOC) content, surface area, maturity (i.e., crystallinity) of the CM and the presence of various functional groups on the surface of the CM. Preg-robbing gold ores continue to be a significant challenge because of the lack of a single or universal solutions.

Research has been directed towards addressing the following issues related to this problem: i) developing predictive tools for characterization and assessment of the preg-robbing behavior of CM in the ore (Helm et al., 2009; Hart et al., 2011), ii) minimizing the losses related to preg-robbing of CM during the gold recovery process (Dunne et al., 2013; Miller et al., 2005), and iii) direct quantitative determination of the surface gold preg-robbed on CM as part of gold deportment balances studies (Dimov et al., 2003; Dimov et al., 2009).

Various approaches have been employed to minimize gold losses related to preg-robbing on CM. They include partial removal of the carbonaceous material by flotation separation, oxidation (chlorination, roasting and bio-oxidation), passivation using various blanking agents or strong competing adsorbents such as activated carbon or resins (Dunne et al., 2013; Zhou et al., 2013). The degree of success using these various techniques depends strongly on the ore mineralogy and the gold extraction process. Certain chemical compounds can passivate the carbonaceous matter by selective adsorption or wetting of its surface. Treatments of carbonaceous ores with chemical surfactants (blinking reagents) have shown promising results but at the same time have demonstrated that, if not properly used, could have a strong detrimental effect on the gold recovery process. Thus, high dosages of surfactants used as passivating agents for carbonaceous matter could lead to unwanted partial passivation (fouling) of the activated carbon added during the CIL process and severely impact the gold recovery. Optimal dosage for various surfactants would strongly depend on the ore mineralogy and the parameters of the pulp (temperature, pH, space charge, surface area and maturity of the carbonaceous material, etc).

This paper describes an experimental study on the effectiveness of various commercially available blanking agents used to minimize the preg-robbing capacity of naturally occurring carbonaceous matter. The scope of the study was to establish: i) the optimum addition of blanking agent (surfactant) to minimize the preg-robbing capacity of the CM in the ore, and ii) the maximum threshold amounts of blanking agents before they negatively impact the preg-robbing capacity of the activated carbon in the CIL process. The study procedure involves standardized doping tests for evaluation of the maximum preg-robbing capacity of treated CM and parallel surface analysis by ToF-SIMS to directly monitor the loading of the chemical surfactants on both the naturally occurring CM and the added activated carbon. The ToF-SIMS (Time of Flight Secondary Ion Mass Spectrometry) technology provides non-destructive elemental and molecular surface analysis with detection limits in the low ppm or ppb range [Dimov et al., 2009].

## EXPERIMENTAL

The study was carried out on a single flotation concentrate sample representing carbonaceous gold bearing ore. Most of the gold contained in the sample (approx. 97%) was present as liberated, visible gold, while the remainder is refractory gold contained in sulphides. The flotation concentrate contained 1.20 wt% TOC that has shown significant preg-robbing properties. Direct cyanidation in a CIL circuit was used to recover the gold. The overall gold extraction was ca. 82%. A gold deportment study carried out on the CIL residue sample showed that 67% of the total gold losses are related to preg-robbing.

Three different commercially available blanking reagents were evaluated in this study:

- Cytec Reagent S-11023 (Cytec Industries Inc.)
- Calfoam SLS-30 (Quadra Chemicals Ltd) and
- Armak 1019 (Akzo Nobel Surface Chemistry, LLC.).

The study procedure involves separate steps of conditioning of the naturally occurring CM and the activated carbon used in the CIL process, followed by ToF-SIMS surface analysis of the conditioned samples and corresponding doping tests. The conditioning and the pulp parameters were chosen to be similar to the standard parameters used for direct cyanidation and CIL process and are summarized in Table 1. In order to be able to independently monitor the effect of the blanking reagents on the CM in the ore sample and on the added activated carbon during the CIL step, the conditioning with the blanking agent is done in two consecutive steps:

Step 1 Pulp only conditioning with blanking agent. After 24 hours of residence time (equal to the standard pulp aeration time before the cyanidation phase), the pulp was filtered, and the wet, conditioned sample was subjected to a doping test with a controlled concentration of  $Au(CN)_2$  in solution. A small amount of the conditioned pulp sample was taken for ToF-SIMS surface analysis prior to the doping test.

Step 2 Conditioning of the activated carbon. The filtered solution from step 1, containing any residual blanking agent, was used for conditioning of the activated carbon for another 24 hours. The conditioned activated carbon was subjected to similar ToF-SIMS surface analysis and a doping test.

For each individual blanking agent the experiments were repeated at various dosage concentrations covering several orders of magnitude range. The effect of the blanking agent on the preg-robbing properties of the naturally occurring CM and on the activated carbon was evaluated by comparing the results from doping tests on the as received, not conditioned and the corresponding conditioned samples.

A possible leaching of free gold has to be taken into account in the estimates for preg-robbing capacity of CM in these samples. Thus, control samples to evaluate possible leaching of free gold were included in the tests. These tests were done exactly under the same conditions as the doping tests, but without  $Au(CN)_2$ . The tests were performed only with the same proportion of NaCN in solution as in the doping tests. The assayed gold values from these control samples show whether free gold has been leached from the solid samples. If the data showed substantial leaching of free gold, then the values for total preg-robbing capacities are corrected by adding the leached gold values. The control samples showed that an insignificant amount of free gold was leached during the doping tests.

Table 1. Experimental conditions

Pulp sample		Activated carbon	
Pulp density	50% solids by weight	Concentration	12 g/L
pH	8.5	pH	10.5
Conditioning with blanking agent	24 hours on a shaking table	Conditioning with blanking agent	24 hours on a shaking table
Doping test	Doping concentration: Au=100ppm Agitation time: 30min	Doping test	Doping concentration: Au=100ppm Agitation time: 30min

### Reagent Cytec S-11023

The test work was done using dosage concentrations ranging from 0.1 kg/t to 5.0 kg/t. These dosage levels provide a blanking effect on the naturally occurring carbonaceous matter in the sample. However, there is also evidence of partial passivation (fouling) of the activated carbon conditioned in the residual solution from this reagent at higher dosage concentrations. The results from the doping tests on the ore conditioned with the blanking reagent Cytec S-11023 are presented in Figure 1a. The data show a blanking effect over the entire range of dosage concentrations leading to significant suppression in the

preg-robbing capacity of the naturally occurring carbon in the sample.

The corresponding data for activated carbon conditioned in residual solutions from conditioning/doping tests for the reagent Cytec S-11023 in the dosage range 0.5 kg/t - 5 kg/t are presented on Figure 1b. There is a definite trend of decline in the assayed preg-robbed gold on conditioned activated carbon samples at higher dosages of the blanking reagent, which can be attributed to the fouling of the activated carbon by residual masking reagent present at higher dosages.

These observations are corroborated by the ToF-SIMS surface data on reagents loading (Figure 2a,b). The data for the conditioned ore sample (Figure 2a) shows a consistent surface loading across the dosage range. The data suggest that there may be some degree of adsorption saturation in the testing concentration range. The data on residual conditioned activated carbon samples (Figure 2b) show a significant increase in the loading levels of Cytec S-11023, which corresponds to the original higher dosage levels in ore conditioning tests. The data are in agreement with the results of the doping tests (Figure 1b), which show a decrease in the Au(CN)<sub>2</sub> adsorption capacity for the conditioned activated carbon at high reagent dosages, i.e. the presence of a fouling effect at higher dosage levels.

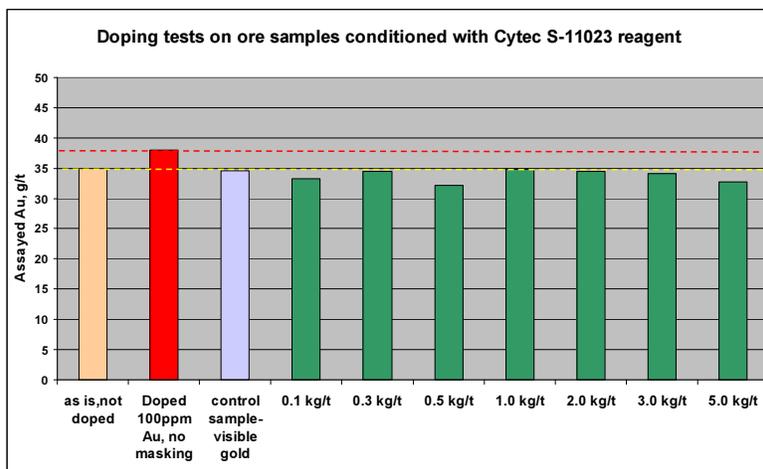


Figure 1a - Results from doping tests on ore samples conditioned with the masking reagent Cytec S-11023.

The tests were carried out over seven dosage concentrations covering the range of 0.1 kg/t to 5.0 kg/t (green colour bars). The graph includes comparative data on assayed gold concentrations of the “as received” ore samples (brown colour bar), doped not conditioned ore sample (red colour bar) and control ore sample for monitoring of possible leaching of visible gold in the samples during the doping tests (gray colour bar). The Au values for “as is, not doped”, “doped with 100 ppm Au, no masking” and “control sample for leached visible gold” are the average values determined from all tests.

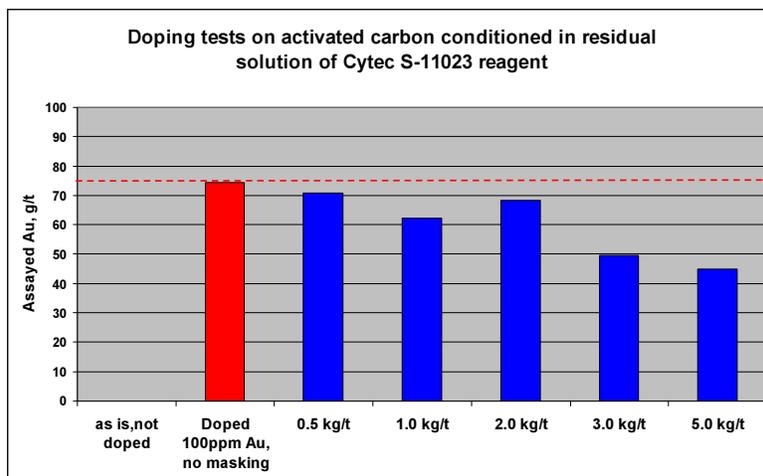


Figure 1b - Results from doping tests on activated carbon samples that have been conditioned in residual solutions retrieved from initial conditioning tests on ore samples at various dosage concentrations of Cytec S-11023 reagent; see Figure 1a. The diagram also includes comparative data on assayed gold concentrations in an “as received not doped” activated carbon sample (assayed value 0.01 g/t) and doped not conditioned activated carbon (red colour bar). The data show a trend of decreasing adsorption capacity of the activated carbon (fouling effect) at higher dosage concentrations.

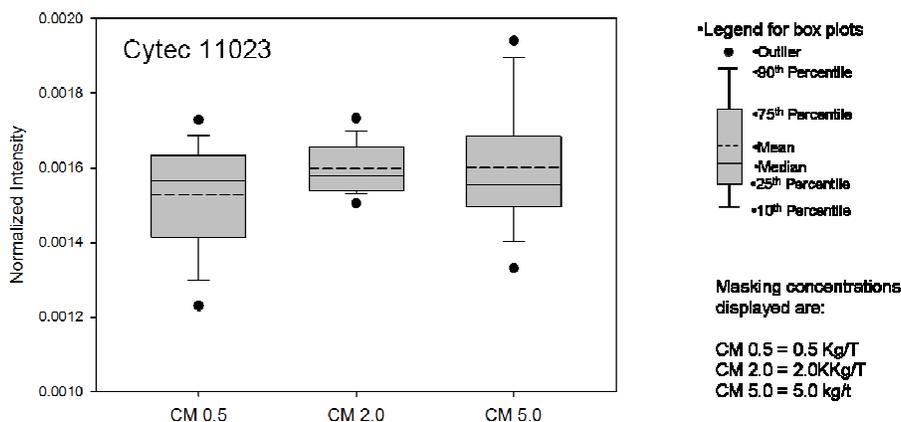


Figure 2a - ToF-SIMS surface data on blanking agent loadings measured on the ore sample after blanking tests for 24 hours with the reagent Cytec S-11023. The data represent the normalized intensities of the detected characteristic peaks in the TOF-SIMS spectra at various concentrations of the blanking agent

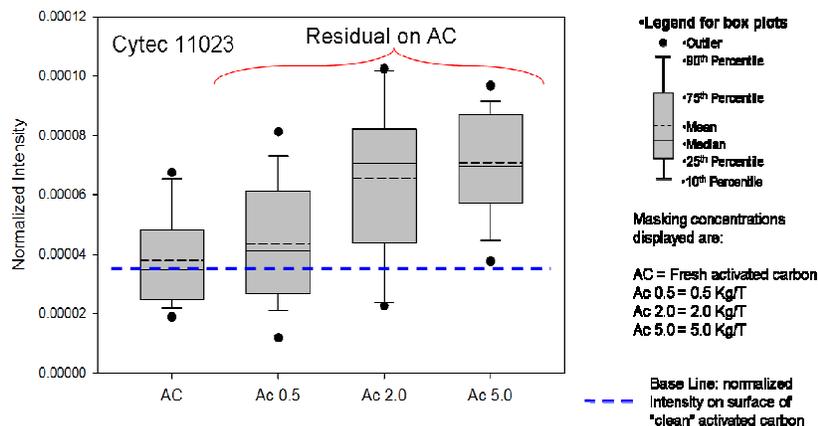


Figure 2b - ToF-SIMS surface data on blanking agent loadings measured on activated carbon conditioned for 24 hours in residual solution from masking tests on the ore sample with the reagent Cytec S-11023.

### Reagent SLS 30

The results from the doping tests on ore samples conditioned with the masking reagent SLS 30 (Sodium Lauryl Sulphate) are shown in Figure 3a. The tests were carried out over dosage concentrations that range from 0.001 kg/t to 3.0 kg/t. The data show a blanking effect on the naturally occurring carbonaceous matter over the whole range of dosage concentrations.

The corresponding data for activated carbon conditioned in residual solutions from conditioning/doping tests for the reagent SLS 30 in the dosage range 0.001 kg/t – 1.0 kg/t are presented on Figure 3b. The data show a definite trend of decline in the assayed preg-robbed gold on conditioned activated carbon samples at higher dosages of the masking reagent, indicative of fouling of the activated carbon by residual masking reagent present at higher dosages.

The ToF-SIMS surface analysis data confirmed the presence of significant SLS 30 on the surface of the ore samples (Figure 4a). The ToF-SIMS data on loading levels of SLS30 on conditioned activated carbon at these conditions confirmed high loading levels of the reagent (Figure 4b). The data indicate that for dosage levels above 0.05 kg/t there will be a significant loading on the surface of the activated carbon and hence, a fouling effect.

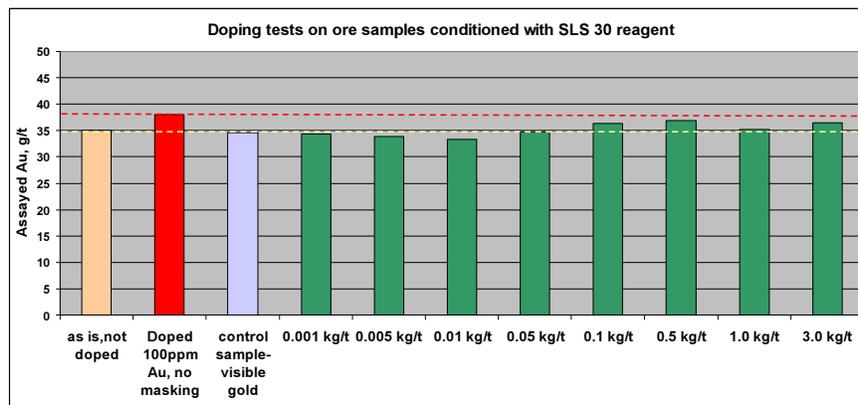


Figure 3a - Results from doping tests on ore samples conditioned with the masking reagent SLS 30 (Sodium Lauryl Sulphate). The tests were carried out over dosage concentrations covering the range 0.001 kg/t - 3.0 kg/t (green color bars).

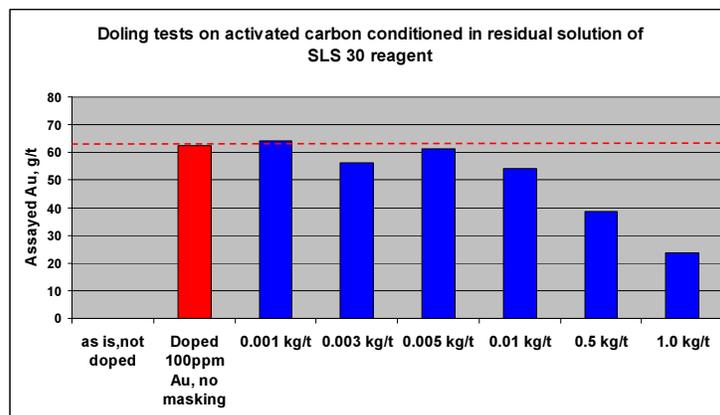


Figure 3b - Results from doping tests on activated carbon samples that have been conditioned in residual solutions retrieved from initial conditioning tests on ore samples at various dosage concentrations of SLS 30 reagent. The diagram also includes comparative data on assayed gold concentrations in an “as received not doped” activated carbon sample (assayed value 0.01 g/t) and doped not conditioned activated carbon (red colour bar). The data show a trend of decreasing adsorption capacity of the activated carbon (fouling effect) at higher dosage concentrations.

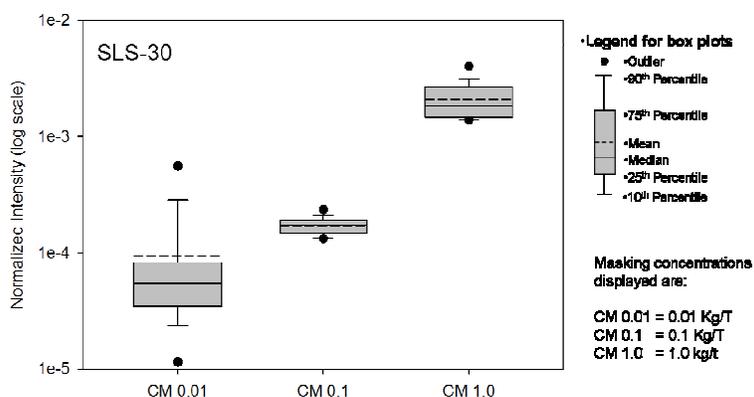


Figure 4a - ToF-SIMS surface data on blanking agent loadings measured on the ore sample after blanking tests for 24 hours with the reagent SLS 30.

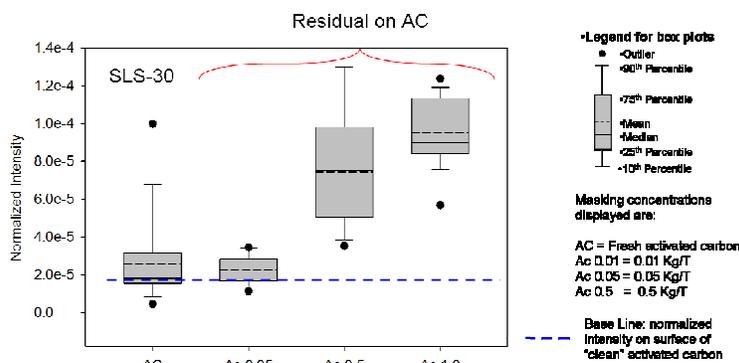


Figure 4b - ToF-SIMS surface data on blanking agent loadings measured on activated carbon conditioned for 24 hours in residual solution from masking tests on the ore sample with the reagent SLS 30.

## Reagent Armak 1019

The conditioning tests were carried out on 9 dosage concentration points covering the range 0.01 kg/t - 5.0 kg/t. The experimental data show significant suppression in the preg-robbing capacity of the naturally occurring carbon in the ore samples for these dosages (Figure 5a). In contrast to the results on the previous blanking agents, the data on Armak 1019 indicate that excessive use of this reagent will lead to a rapid increase in the preg-robbing capacity of the ore sample. The initial test work on ore samples with Armak 1019 at dosage concentrations higher than 0.5 kg/t unexpectedly showed a dramatic increase in the preg-robbing capacity of the conditioned ore samples (Figure 5a). Repeated tests confirmed the same effect.

The corresponding data for activated carbon conditioned in residual solutions from conditioning tests on the ore samples for the reagent Armak 1019 in the dosage range 0.01 kg/t - 0.5 kg/t are presented in Figure 5b. The random fluctuations in the assayed Au values for treated activated carbon at various dosages of Armak 1019 are believed to be related to variations of the effective BET surface area and not to fouling of the activated carbon because a definite trend of decline in the assayed preg-robbled gold was not observed in this reagent concentration range.

A possible cause for the extreme “preg-robbing” effect by Armak 1019 at high dosage levels is related to the chemical structure of the reagent. Armak 1019 is alkyl quaternary surfactant with a positive charge from surfactant head groups at all pH conditions. Continuous adsorption of the surfactant onto component surfaces within the ore could potentially reverse the surface charge of the ore from negative to positive, resulting in an electrostatic attraction of  $\text{Au}(\text{CN})^-$  and in a decrease in Au recovery.

These observations are corroborated by the ToF-SIMS surface data on reagent loadings (Figure 6a,b). The data on conditioned ore samples (Figure 6a) shows increased loading corresponding to an increase in concentration for all dosages. Note that there is a rapid increase in the loading of this reagent on the ore sample at 0.5 kg/t. This is in good correlation with the data from the doping tests (Figure 5a). An increase in the pre-robbing capacity of the ore sample due to a reverse in the surface charge of the sample from negative to positive is observed at the 0.5 kg/t - 1.0 kg/t concentration. The ToF-SIMS data on the activated carbon conditioned in residual solutions of Armak 1019 (Figure 6b) show trace concentrations of the reagent on the surface of the activated carbon grains, but not an increase in residual solution concentration. This is consistent with the data from the doping test on conditioned activated carbon (Figure 5b) indicating no fouling effect on the carbon surfaces. The random fluctuations in the assayed Au values for treated activated carbon at various dosages of Armak 1019 are believed to be related to variations in the effective BET surface area.

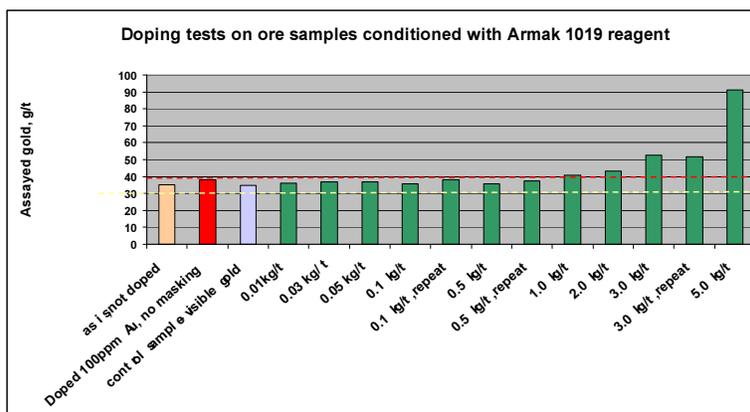


Figure 5a - Results from doping tests on ore samples conditioned with the masking reagent Armak 1019. The tests were carried out over 11 dosage concentrations covering the range 0.01 kg/t - 5.0 kg/t (green colour bars)

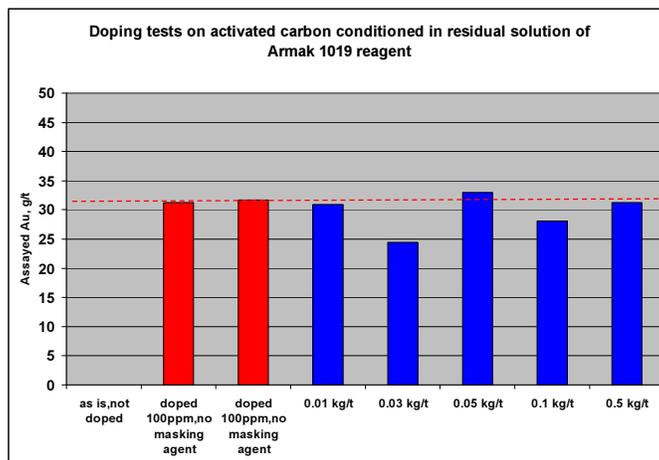


Figure 5b - Results from doping tests on activated carbon samples that have been conditioned in residual solutions retrieved from initial conditioning tests on ore samples at various dosage concentrations of Armak 1019 (see Figure 5a). The diagram also includes comparative data on assayed gold concentrations in “as received” activated carbon samples (0.01g/t) and doped not conditioned activated carbon (red colour bars).

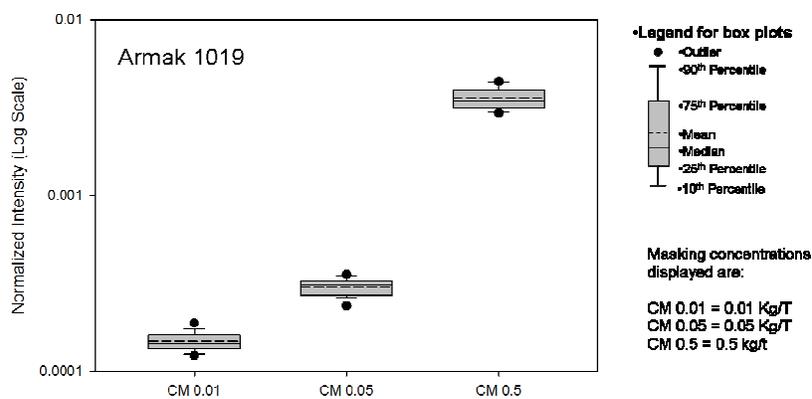


Figure 6a - ToF-SIMS surface data on blanking agent loadings measured on the ore sample after blanking tests for 24 hours with the reagent Armak 1019.

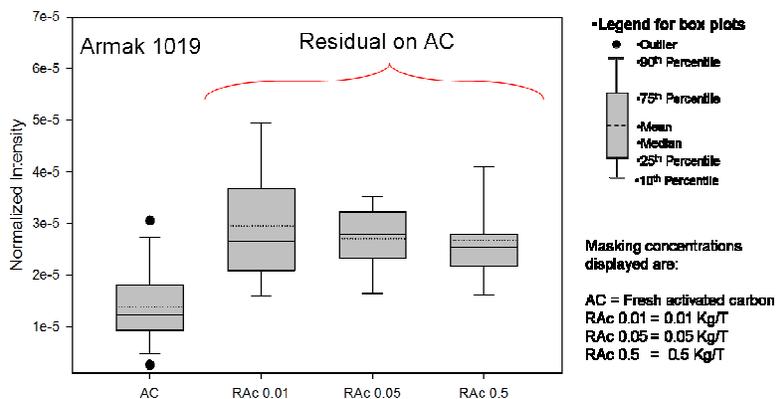


Figure 6b - ToF-SIMS surface data on blanking agent loadings measured on activated carbon conditioned for 24 hours in residual solution from masking tests on the ore sample with the reagent Armak 1019.

## DISCUSSION

The standard procedure used for this study was aimed at duplicating, as closely as possible, direct cyanidation CIL plant operating conditions: pulp densities, pH, and residence time for conditioning tests. There are two ways to introduce the blanking agents into the system: (i) during the grinding stages or, (ii) directly into the pulp “pre-aeration” tank, which provides a 24-hour conditioning window. This study addresses the second conditioning option, namely adding blanking agents into the “pre-aeration tank” and conditioning of the pulp for 24 hours prior to the CIL process.

Every precaution was taken to insure the best possible reproducibility of the required experimental conditions for various tests including sample quantities, reagent concentrations and testing sequences. The reagent effectiveness evaluation is based on the comparative analysis between the assayed preg-robbed gold on doped conditioned samples and doped, not processed reference samples. It also incorporates reference samples for monitoring of possible leaching of visible gold present in the ore samples during the doping tests and the use of quality control reference samples with certified gold content to ensure accuracy and precision of assay results.

Additional factors that may affect the reproducibility of the test data and increase the statistical deviation might be related to the inherent nature of the ore samples. They can include a high content of visible gold, and the relatively narrow dynamic range of the maximum preg-robbed gold levels defined by the naturally occurring CM in the feed sample used in this study. In contrast to the ore samples, the “as received not processed” activated carbon samples had only a trace amount of gold (0.01 g/t). This eliminates the problem with the initial fluctuation of the baseline gold content and ensures a high dynamic range of assayed Au content in conditioned/doped activated carbon samples. In this study, the variability in the data of assayed preg-robbed gold on activated carbon samples from various conditioning/doping tests is related mainly to the small sample quantities, the presence of fine carbon generated during the conditioning step, and the variations in the corresponding BET surface areas. Part of this generated carbon was removed during the filtering/washing of the conditioned samples. This resulted in some changes in the effective BET surface area of the activated carbon subjected to doping tests. This effect was more pronounced for the conditioned activated carbon samples and, evidently, it was a probable cause for rather systematic lower values of assayed preg-robbed gold on conditioned activated carbon samples than those of the “as received not-processed” reference activated carbon samples.

It is important to make the distinction between random variations of the preg-robbed Au content on conditioned activated carbon samples, and systematic trends in the preg-robbed Au content over a large dynamic range of reagent dosage concentrations. While the first phenomenon is most probably related to variations of the effective BET surface area, the second one is a result of the fouling effect on the activated carbon by the residual blanking reagents used at higher/excessive concentrations in the conditioning tests for the feed samples. The findings from the conditioning/doping test work were corroborated by the ToF-SIMS surface data.

The introduction of direct monitoring of the reagents loading by the ToF-SIMS surface analysis provided an important independent confirmation of effectiveness and shortcomings of the conditioning process for the various masking agents.

## CONCLUSIONS

Carbonaceous materials of natural organic carbon have adsorption properties similar to that of commercial activated carbon. The adsorption of surfactants on carbonaceous material/activated carbon is attributed to various mechanisms such as electrostatic interactions, hydrophobic chain-chain interactions, dispersive interaction and precipitation. Surfactant adsorption can be greatly affected by the mineralogy of the ore sample, the temperature and the pH of the solution. In addition, the adsorption is further affected by the inherent characteristics of the carbonaceous material. Parameters such as surface area, porosity of the

carbon, pore volume, surface charge and presence of various functional groups all affect the carbonaceous materials capacity for surfactant adsorption.

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#### REFERENCES

- S.S. Dimov, S.L. Chryssoulis and R.N. Sodhi. (2003) "Speciation of Surface Gold in Pressure Oxidized Carbonaceous Gold Ores by TOF-SIMS and TOF-LIMS", Applied Surface Science, Vol. 203-204, 644-647
- S.S. Dimov, B. Hart and A. Chattopadhyay.(2009) "Speciation and quantification of surface gold in carbonaceous matter from AC POX stream products by TOF-SIMS", COM 2009
- R.Dunne, W.P.Staunton and K. Afewu.(2013), "A historical review of treatment of preg-robbing gold ores- what has worked and changed", World Gold Conference, Brisbane QLD 2013, 99-110.
- B. Hart, S.S.Dimov.R. Mermillod-Blondin and J.Fournier. (2011), "Procedure for characterization of carbonaceous matter in an ore sample with estimation towards its preg-robbing capacity", World Gold Conference, Montreal 2011, 35-50.
- M. Helm, J. Vaughan, W.P. Staunton and J. Avraamides. (2009), "An investigation of carbonaceous component of preg-robbing gold ores"., World Gold Conference, 2009, The Southern African Institute of Mining and Metallurgy, 2009,139-144
- J.D.Miller,R.Y.Wan and X.Diaz. (2005), "Preg-robbing ores", Development in Mineral processing, Vol.15,Elsevier 2005, 937-972.
- Q.Zhou, G.Barett, J.Jiang and Y.Choi. (2013), "Surfactant for gold recovery improvement with preg-robbing gold ores", World Gold Conference, Brisbane QLD 2013, 1-6.