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Cyanide Amenability Studies of Gold Ores-The Use of Design of Experiment (DOE)

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Abstract: *In this research, Design of Experiment (DOE) was employed to investigate the influence of operational parameters (e.g. leaching time, particle size and cyanide concentration) on gold cyanidation process of three pit samples. The gold content of the pit samples were 13.13 g/t, 3.28 g/t and 7.83 g/t respectively for Prestea underground ore (Pit A), Beta North Oxide ore (Pit B) and Mampong Transition ore (Pit C). Design of Experiment (DOE) was employed to design leaching (bottle roll) test, model and evaluate the influence of particle size, cyanide concentration and leaching time on the cyanidation of the three pit samples. The particle size, cyanide concentration and leaching time were varied from* d_{80} *of 150 to 75 µm, 120 to 500 ppm and 3 to 24 hrs respectively. Leaching of the samples (in the absence of activated carbon) for 24 hrs gave recoveries below 88%. Thus, recoveries for Pit A, Pit B and Pit C were 87%, 75% and 51% respectively. Simulation of the results using DOE at 95% confidence level to predict the relationship between the gold recoveries and influential parameters showed that gold recovery correlates positively with cyanide concentration and residence time. In terms of particle size, gold recovery was observed to increases marginally with decreasing particle size from 106 to 75 µm. A confirmatory test performed showed that not much difference in Au recovery is observed upon fine grinding (i.e. from 80% passing 106 µm to 80% passing 75 µm) and Carbon in leach (CIL) of the three Pit samples significantly improved Au recoveries. The findings from the study suggest that DOE could be used for cyanide amenability studies.*

1. INTRODUCTION

Gold is found in very low concentrations in the ores from which it is mined. Generally, to recover the gold requires separation of it from the associated minerals in the ore, which could be achieved by selectively dissolving the gold in a suitable lixiviant. Worldwide, majority of gold is extracted from it ores using cyanide as a lixiviant. This is because of its relatively low cost, advancement of general understanding on it usage compared to other lixiviants, and the fact that it has greater efficacy for gold dissolution than other metals (Marsden and House, 2006).

Gold cyanidation process, like any other process, is affected by several parameters such as pH, temperature, slurry density, particle size, oxygen, cyanide concentration, agitation and the presence of other minerals in the ore (Marsden and House, 2006, Heath and Rumball, 1998, Ellis and Senanayake, 2004, Aghamirian and Yen, 2005, Lorenzen and Van Deventer, 1992, Cathro, 1963, Perky et al., 1999, Dai and Jeffrey, 2006). The optimization of cyanidation operation of gold ores is a complex process since numerous characteristics at the same time influence the task, with some of them being clashing in nature. Therefore, an appropriate choice of the cyanidation process with relevant properties is significant to maximize the percent recovery with minimal operating cost (Kondos et al., 1995, Baral et al., 2014, Dowd and Onur, 1993).

Generally, prior to cyanidation of gold ores, cyanide amenability studies is performed on the ore to determine the optimum conditions for the operational parameters (e.g. leaching time, cyanide strength, particles size etc.) to maximize gold recovery and gold grade. The traditional way of identifying the optimal operational conditions is to perform one-factor-at-a-time experiment (OFAT). Thus, varying the parameters one at a time and keeping the others constant until its best setting is found. However, the use of OFAT experimental approach can be very time consuming, costive and difficult to determine the effects that are caused by several factors acting in combination (Antony et al., 2003). Design of Experiments (DOE) is an alternative answer to the above challenge. It allows the researcher/investigator to study a large number of variables in a small number of experimental runs. DOE uses statistical experimental methods to develop the best factor and conditions to optimize a process or a design. The statistical analysis of the data can be performed quickly using specialized software analysis packages such as Minitab, Design expert, JMP etc (Telford, 2007, Shina, 2002, Anderson and Whitcomb, 2016).

This paper seeks to perform cyanide amenability studies using Design of Experiment (DOE) to optimize operational parameters such as particle size, cyanide concentration and leaching time of three (3) gold ore samples obtained from a mining company in Ghana; Prestea Underground pit ore (PUG, A), Beta North pit (B) ore and Mampong Transition pit (C).

2. EXPERIMENTAL

2.1. Ore and Sample Preparation

Three pit samples (Prestea underground sample, Pit A), Beta North Oxide sample, Pit B) and Mampong Transition sample, Pit C) used in this study were supplied by Bogoso/Prestea Limited (GSBPL). The pit samples were dried, crushed and ground to 80% passing 150, 106 and 75 µm. For each grind size, 10 kg (which was subdivided into 500 g each) was prepared and bagged. Representative samples of each pit were taken for chemical analysis. The total carbon, organic carbon, carbonates and total sulphur content for each pit samples were determined using Leco$^{\text{TM (SC-144DR)}}$.

2.2. Bottle Roll Test

The design of experiment (DOE) was used to design an organized and controlled series of bottle roll test work (Table 1). The operational parameters varied during the DOE design were particle size (150, 106 and 75 µm), cyanide concentration (500, 310 and 120 ppm) and leaching time (3, 13.5 and 24 h). For each bottle roll test, a sample mass of 500 g was weighed and pulped to 45% solids. Lime was used as the pH modifier to raise the pH to 10.5 for each leaching test. After each leaching test, samples were taken, filtered and samples taken for free cyanide and Au analysis. The residues were thoroughly washed with water, dried, sampled and then fire assayed to determine the Au content.

2.3. Carbon-In-Leach Test (Cil)

The carbon-in-leach (CIL) test was done to confirm the results obtained from the bottle roll test after modelling with DOE. The CIL test was carried out for 24 h at a pH of 10.5, pulp density of 45% solid, cyanide concentration of 230 ppm, particle sizes of 106 and 75 µm and fresh activated carbon addition of 15 g for all the three pit samples. After each leach test, the activated carbon was screened off and the pulp filtered. Gold and cyanide concentrations were determined on the filtrate and the residue thorough washing, dried and submitted for fire assay.

Table1. *Bottle Roll Test Runs Obtained from DOE*

TEST NUMBER.	ORE	CN(ppm)	PARTICLE SIZE(µm)	LEACHING TIME(hrs)
Test 1	PITA	120	150	24
Test 2	PIT B	500	75	24
Test 3	PIT B	120	150	3
Test 4	PIT A	500	150	24
Test 5	PIT C	120	75	24
Test 6	PIT B	120	75	3
Test 7	PIT B	500	75	24
Test 8	PIT A	120	75	24
Test 9	PIT B	500	75	3
Test 10	PIT C	120	75	24
Test 11	PIT B	120	150	3
Test 12	PIT A	500	75	3
Test 13	PIT A	120	75	3
Test 14	PIT C	500	75	3
Test 15	PIT C	500	150	24
Test 16	PIT A	500	150	3
Test 17	PIT B	310	106	13.5
Test 18	PIT A	310	106	13.5
Test 19	PIT C	500	150	3
Test 20	PIT B	500	150	24
Test 21	PIT B	120	150	24
Test 22	PIT C	120	150	3

2.4. Determination of Free Cyanide Concentration

To determine the free cyanide in solution after each leaching test, 10 ml of filtered solution was pipetted into a 250 mL conical flask. 2 to 3 drops of rhodamine indicator was added and the colour noted (yellow). The 10 ml solution in the conical flask was diluted to 100 ml with 0.01 M dilute sodium hydroxide (NaOH) and afterwards, titrated against silver nitrate of concentration 0.0096 M until a colour change from yellow to light orange was observed. The end-point value on the burette was noted and the concentration of free cyanide is determined;

$$[\mathrm{CN}^-] = \frac{\mathrm{Vol.\ of\ AgNO_3} \times [\mathrm{AgNO_3}]}{\mathrm{Vol.of\ cyanide\ solution}} \tag{1}$$

2.5. Determination of Gold Concentration in Filtrate

To determine the concentration of gold in the filtrate, 10 ml of the filtrate was pipetted into a separation funnel and 10 ml of Diisobutyl ketone (DIBK) added. The mixture was agitated with an electronic shaker for 20 minutes and allowed to stand for separation. The organic layer was then drained into a test tube and analysed using Atomic Absorption Spectroscopy (AAS).

2.6. Determination of Gold Concentration on Loaded Carbon

Screened off carbon from the pulp was washed with distilled water and dried at temperature of 120 °C. 1 g of the dried carbon was weighed into a crucible and ashed in a laboratory industrial furnace at temperatures of 1100 °C to1300 °C. The ashed carbon was transferred into a beaker and 30 ml of aqua regia (10 mL of 50% HNO_3 and 20 mL of 50% HCL) was added and heated on a hot plate under a fume chamber. The digested solution was diluted to 100 ml with distilled water, extracted into an organic solvent (DIBK) and sent for AAS analysis.

3. RESULTS AND DISCUSSION

3.1. Chemical Analysis of Pit Samples

Table 2 shows the organic carbon, total carbon, carbonates, total sulphur, copper and gold content of the pit samples. Pit sample A had the highest head assay for gold (13.13 g/t) followed by pit sample C (7.83 g/t) and pit sample B (3.28 g/t). Pit sample C has the highest copper content (432.95 g/t), which may lead to higher cyanide consumption rate compared to the other pit samples. The organic carbon content was between 0.17% and 0.59%, which suggest that the samples have the potential to preg-rob. The organic carbon content of the pit samples was in the order of Pit A> Pit C>Pit B. The total sulphur content for the pit samples was in the ranged of 0.05% and 0.62%. The carbonate components for the samples were also high for Pit sample A and C with Pit B having the lowest.

Table2. *Head assay and sulphur/ carbon speciation*

Sample	Total Sulphur, %	Total Carbon, %	Organic Carbon, %	Carbonates, %	Gold, g/t	Copper, g/t
Pit A	0.55	1.93	0.58	0.41	13.13	86.01
Pit B	0.05	0.59	0.18	0.06	3.28	69.46
Pit C	0.62	0.82	0.43	0.28	7.83	432.95

3.2. Leaching in the Absence of Activated Carbon

Figure 1 show the Au recovery for the different leaching test conditions designed with DOE for all the pit samples. The pit samples were all subjected to leaching test at varying particle sizes (d_{80} of 150 to 75 µm), cyanide strength (120 to 500 ppm) and residence time (3 to 24 h) to determine the optimum operational values of the aforementioned parameters that yields maximum Au recovery. Au recovery lower than 90% was achieved for all the test condition. Pit A gave the highest recovery (86.1%) followed by pit B (83.8%) and pit C (46.1%), all at a residence time of 24 h.

Lower recoveries (<88%) observed for the pit samples (especially for Pit C) can be related to the preg-robbing nature as well as the presence of cyanicides (e.g. copper, sulphur) in the samples. The copper and organic content for pit samples A, B and C, were 86.1 g/t and 0.58%, 69.46 g/t and 0.18%, and 432.95 g/t and 0.43% respectively. Copper minerals dissolve to varying degrees in alkaline cyanide solutions. The dissolution of copper during leaching of gold ores is generally undesirable because it can consume cyanide and dissolved oxygen available in the pulp, leading to retard gold dissolution rates and reduced free cyanide ions in solution for the complexing of the dissolved gold

(Marsden and House, 2006). Moreover, it must be mentioned that the influence of copper minerals on cyanide leaching of gold differ depending on type of copper minerals present in the ore (Jiang et al., 2001). The higher Au recovery observed for pit sample A, the pit sample with the highest organic carbon content, suggest that the carbonaceous materials in the ore have low gold adsorption capacity. Studies by Helm et al (2009) showed that the extent to which the overall gold recovery decreases depends on the type of carbonaceous material, the degree of maturation and the quantity available. Pit sample A is an underground ore hence, the carbonaceous material present in the ore may be resulting from wood chips formed by the degradation of the timber used for roof supports in underground mines, and/or roots of vegetation cleared for surface mining. The preg-robbing nature of such materials is known to be low(Amanya et al., 2017).

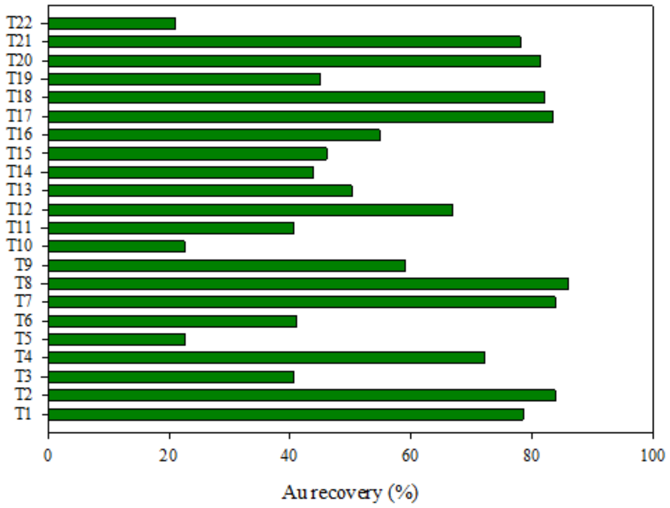

Figure1. *Au recovery for the various leaching test*

3.3. Modelling of Leaching Data

The objective was to determine the effects of three variables (CN dosage, particle size and leaching time) on the leachability of three gold ore samples. All the three factors were varied over two levels; a low and high level. The statistical software package JMP was used to perform the regression modeling. The result of the regression fit to the recovery data is presented in Table 3. The analysis of variance (ANOVA) of the linear regression fit is presented in Table 4. Both tables (3 and 4) were analysed to determine if the variation in the factor levels affect the leachability of the three gold ores. Results of the analysis of variance clearly show that the regression model is significant at $p<.0001$. The ratio of the mean square regression to the mean square error is large (12.3). The R^2 and R^2 (adjusted) values are 79.4% and 72.9% respectively, which suggest that the model could explain about 72.9% of the variability in the gold recoveries for the gold ores understudied.

Table3. *Regression model parameter estimates*

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	56.485	2.498	22.61	<0.0001
Ore[Pit A]	13.638	3.549	3.84	0.0014
Ore[Pit B]	9.301	3.344	2.78	0.0134
Ore [Pit C]	-22.938	3.696	-6.21	<0.0001
CN(120,500)	7.782	2.584	3.01	0.0083
PS(75,150)	-0.092	2.584	-0.04	0.9720
LT(3,24)	9.598	2.584	3.71	0.0019

Table4. *Analysis of Variance for the recovery regression model*

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	5	8212.801	1642.56	12.3024
Error	16	2136.243	133.52	**Prob > F**
C. Total	21	10349.044		<.0001

Figure 2 and Table 5 show the recovery prediction profile as a function of cyanide concentration (CN, ppm), particle size (PS, µm) and leaching time (LT, hrs) and the parameter effect test for the pit samples. The solid black lines show all the possible what-if scenarios if that input were to change and all other inputs held constant. The blue lines on the other hand represent the 95% confidence band. The steepness of the black line for any of the input parameters signifies its importance or effect on gold recovery for the different gold ores. A general trend of increase in Au recovery with increasing cyanide strength, leaching time and decreasing particle size is observed for all the pit samples (Fig. 2). The positive steep slope observed between Au recovery and cyanide and leaching time suggest that the leaching of the three pit samples are greatly controlled by cyanide strength and leaching time. Thus, the recovery increases with increasing cyanide concentration and leaching time. The negative slight slope observed in the case of particle size suggests that the leachability of the three pit samples are marginally influenced by particle size under the conditions of study. Between grind size of 75 and 106 µm the black line remains unchanged suggesting the possibility of achieving the same Au recovery at those grind sizes. Comparatively, Pit A gave the highest Au recovery followed by Pit B and C, respectively. Maximum Au recoveries of about 87%, 75% and 51% was obtained after 24 hrs of leaching time at 75 µm and cyanide strength of 500 ppm CN for pit samples A, B and C respectively. The data in Figure 2 and Table 5 suggest that it easier to leach pit sample A than that of B and C and reaffirm the claim that leaching of gold ores are greatly influenced by the ore type/mineralogy, cyanide dosage, particle size and leaching time.

Table5. *The parameter effect Tests*

Source	Nparm	DF	Sum of Squares	F Ratio	Prob > F
Ore	2	2	5159.0088	19.3199	<.0001
CN(120,500)	1	1	1211.1905	9.0716	0.0083
PS(75,150)	1	1	0.1693	0.0013	0.9720
LT(3,24)	1	1	1842.4321	13.7994	0.0019

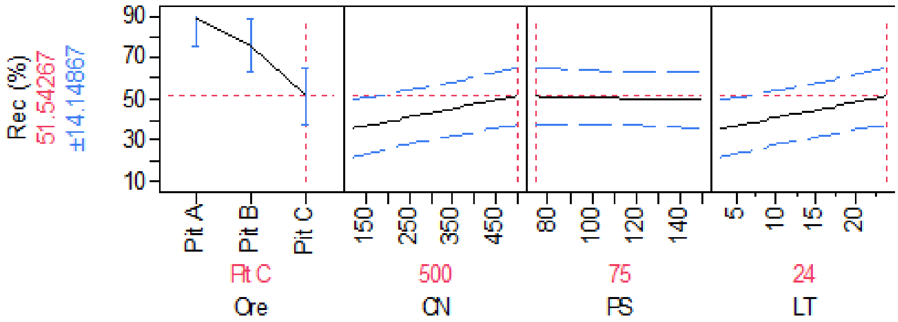

Figure2. *Au recovery as a function of cyanide strength, particle size and leaching for pit samples A, B and C*

The cyanide consumption rates determined after each leaching test (Fig. 1) were also modelled to ascertain the trends in cyanide consumption rate for each pit sample. Figure 3 show the result for the cyanide consumption rate for each pit sample. The data suggest higher cyanide consumption rate for pit sample C compared to pit samples A and B. The higher cyanide consumption rate observed for Pit sample C can be related to its high copper content. The latter also contributed to the low Au recovery observed for Pit sample C since there will be less CN in the system for gold complexation.

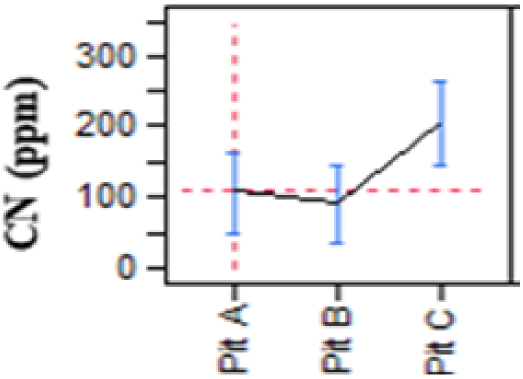

Figure3. *Cyanide consumption rate for the Pit samples*

3.4. Leaching in the Presence of Activated Carbon

From Figure 2 and Table 1, it was clear that the poor Au recovery observed for the pit samples were due to the preg-robbing nature as well as the presence of cyanicides. In addition, the data in Figure 2 also showed that particle size had marginally influenced for all the Pit samples under the conditions of study for the 75 and 106 µm grind size. To confirm all this hypothesis, carbon in leach test was performed on all the pit samples at 75 and 106 µm grind size. The outcome of the test is presented in Table 6. The data clearly confirms the hypothesis that particle size had marginal effect on Au recovery and that the poor Au recoveries were largely due to the preg-robbing nature of the pit samples. A significant improvement in Au recovery for all the pit samples was observed when leaching was performed in the presence of activated carbon. The observed improvement in Au recovery can be attributed to the higher gold adsorption capacity of the activated carbon compared to the carbonaceous material (Amanya et al., 2017). These results support and explain why carbon in leach is the preferred choice for the leaching of ores containing carbonaceous materials. In terms of the grind size, approximately the same Au recovery were obtained for the 75 and 106 µm grind size, affirming the data in Figure 2.

Table6. *Au recovery for the carbon in leach test for all the pit samples*

Sample	Au in sol^{n}. (ppm)		Recovery (%)		Standard Deviation
	75 µm	106 µm	75 µm	106 µm	
Pit A	0.014	0.013	97.20	96.3	0.64
Pit B	0.012	0.015	92.0	91.7	0.21
Pit C	0.014	0.014	55.4	54.3	0.78

Figure 4 show the corresponding results for the CN consumption analysis of the CIL. The data show higher CN consumption for Pit sample C followed by Pit A and Pit B, which also reaffirm the result obtained in Fig 3. Comparatively, higher CN consumption was observed for the grind size of 75 µm than the 106 µm. This is because, as we grind finer the surface area of the mineral of interest and other gangue minerals increases, causing the dissolution of more of the mineral of interest and other constituent of the ore which eventually leads to higher CN consumption since the cyanide will form complexes with the Au and other liberated gangue minerals.

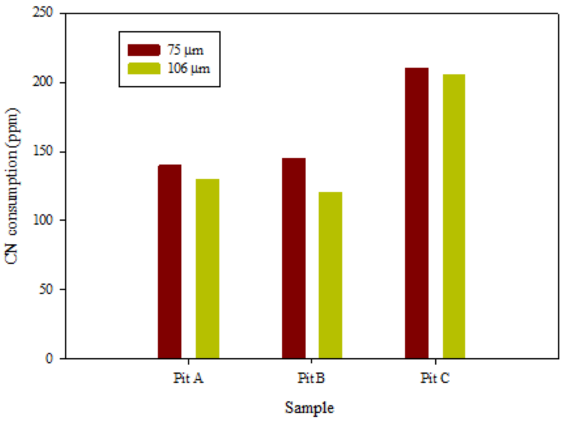

Figure4. *Cyanide consumption rate of CIL test*

4. CONCLUSION

The aim of this study was to perform a cyanide amenability studies using Design of Experiment (DOE) for three (3) gold ore samples. Assay of the pit samples used for the study showed that Prestea underground sample (Pit A), Beta North Oxide sample (Pit B) and Mampong Transition sample (Pit C) contained 13.13 g/t, 3.28 g/t and 7.83 g/t of gold respectively. Results obtained from the various DOE leaching test designed showed that Au recoveries for all pit samples were low for the test where leaching was done in the absence of activated carbon. The latter is due to the preg-robbing nature of the samples. Pit A recorded the highest recovery followed by Pit B and C. Comparatively, Pit C had the highest cyanide consumption rate due to the high copper content in the ore.

Modeling of Au recoveries for the various DOE leaching test revealed that cyanide concentration and leaching time were the main parameters that affected the leachability of the three pit samples. The Au recovery for all Pit samples correlate positively with the leaching time and cyanide concentration. Particle size however, correlated negatively to the Au recovery and had slight influence on the leaching of the three pit samples. Not much difference in Au recovery was observed upon fine grinding (i.e. from 80% passing 106 µm to 80% passing 75 µm). Carbon in leach (CIL) of the three Pit samples significantly improved Au recoveries.

The findings from the study show that DOE accurately predicted the correlations between Au recovery and leaching operational parameters such as leaching time, cyanide concentration and particle size while conducting few numbers of experiments than the conventional approach of varying individual parameter as a function of each other. Additionally, the findings also suggest that DOE could be used for cyanide amenability studies.

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