

THE IMPACT OF CRUSHED ORE AGEING ON METALLURGICAL PERFORMANCE

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ABSTRACT

Ageing tests on a crushed Cu-porphyry ore were conducted to assess the impact of sample oxidation on flotation performance. The objective of the testwork was to simulate ageing of ore that would be blasted through block caving but not milled for possibly several months.

Ageing was performed in specially designed columns, using site and deionised water to keep the samples moist over the entire ageing period. Samples from each ageing interval (0, 0.5, 1, 2, 4, and 8 months) were examined for physical and metallurgical changes resulting from the ageing process through chemical analyses, size analyses, and batch flotation tests.

INTRODUCTION

The oxidation of sulphide minerals has been well documented as being detrimental to metallurgical performance. Conventional mining operation can often necessitate the stockpiling of sulphidic rocks. Experiments on pyrite-sand mixtures by Leon et al [1] evaluated the level of oxidation through the measurement of sulphate generation. Kelebek and Nanthakumar [2] also conducted extensive testwork on fresh and stockpiled copper-nickel sulphide ores. In this case, the impact of ageing was characterized through variation in fast and slow floating fraction rate constants, ultimate recovery, and the pentlandite-pyrrhotite selectivity index and examined both collectorless and collector-induced flotation.

The objective of the current testwork was to evaluate the impact of underground weathering of a block-caved Cu-porphyry ore on metallurgical performance. The impact of oxidation was measured through chemical analyses and flotation tests on the weathered ore.

EXPERIMENTAL

A Cu-porphyry ore sample, crushed to 100% passing 10 mesh (1.2mm), was split into ten 7-kg test charges,

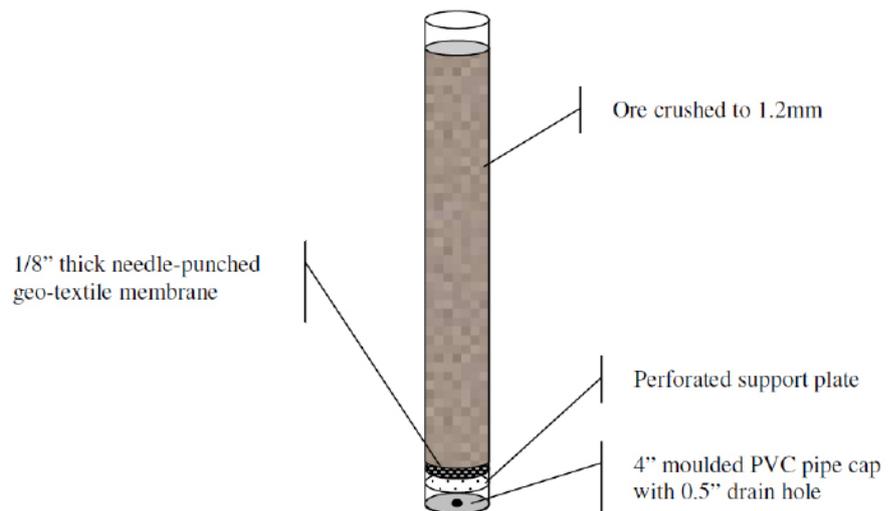


Figure 1 Ageing column schematic

and fifteen 2-kg test charges, and a head sample. The 7-kg charges were placed in specifically designed columns as depicted in Figure 1. The columns were made of 26" L x 4" I.D., schedule 40 PVC pipe with a perforated plate mounted 1" above the bottom to support the sample. The bottom of the columns were capped with a standard 4" moulded PVC pipe cap with a 0.5" hole drilled at its centre to allow any excess water to escape. A

4" diameter circular piece of 1/8" thick needle-punched geo-textile membrane was placed on top of the perforated plate prior to loading the test charge to reduce the risk that fines would escape with any excessive water. The columns were placed in 20L pails (5 per pail), which ensured that the columns remained vertical and collected any excessive moisture.

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The samples were initially moistened with either site or deionised (DI) water by adding water in 250mL aliquots until the first sample discharged liquid. The temperature and humidity were maintained at 15-20°C and 95% RH, respectively. Once the desired relative humidity was obtained, water was added in 250mL aliquots every two weeks. If the humidity was found to drop in one of the pails, a small additional amount of the appropriate water was added to the pail.

A sample was removed from each pail at the following ageing intervals: 0, 0.5, 1, 2, 4, and 8 months. The contents of the columns were split into three 2-kg (dry equivalent) test charges and five smaller sub-samples that were submitted for Cu, Mo, Fe, Zn, Au, S, and Cu speciation assays, size analysis, EDTA extraction tests, shake flask extraction tests, and modified acid-base accounting tests. For each water system tested, one of the 2-kg test charges was used to conduct a batch cleaner test and the remaining test charges were stored in a refrigerator to stop the oxidation process. The batch cleaner test was conducted using the respective treatment water.

SIZE ANALYSES

A detailed size analysis to minus 10 microns was performed on the original AT composite and on each aged sample. The sizing included a conventional Tyler screen analysis to 38 microns followed by a Malvern analysis of the minus 38 microns fraction.

EDTA EXTRACTION TESTS

In order to determine the degree of oxidation, the fresh and aged samples were subjected to an EDTA extraction test. The EDTA extraction test brings into solution all of the metal ions (cations) that are present at the mineral surface as metal oxides through “gentle” chelation using a low concentration of sodium ethylenediaminetetraacetic acid (NaEDTA). The test procedure was based on that detailed by Lascelles and Finch [2]. A 50 gram sample was mixed for 30 minutes in 500 ml of a 0.01 Molar solution of NaEDTA. At the end of the mixing period, the sample was filtered and the filtrate was submitted for an ICP-Scan. The solids were dried and

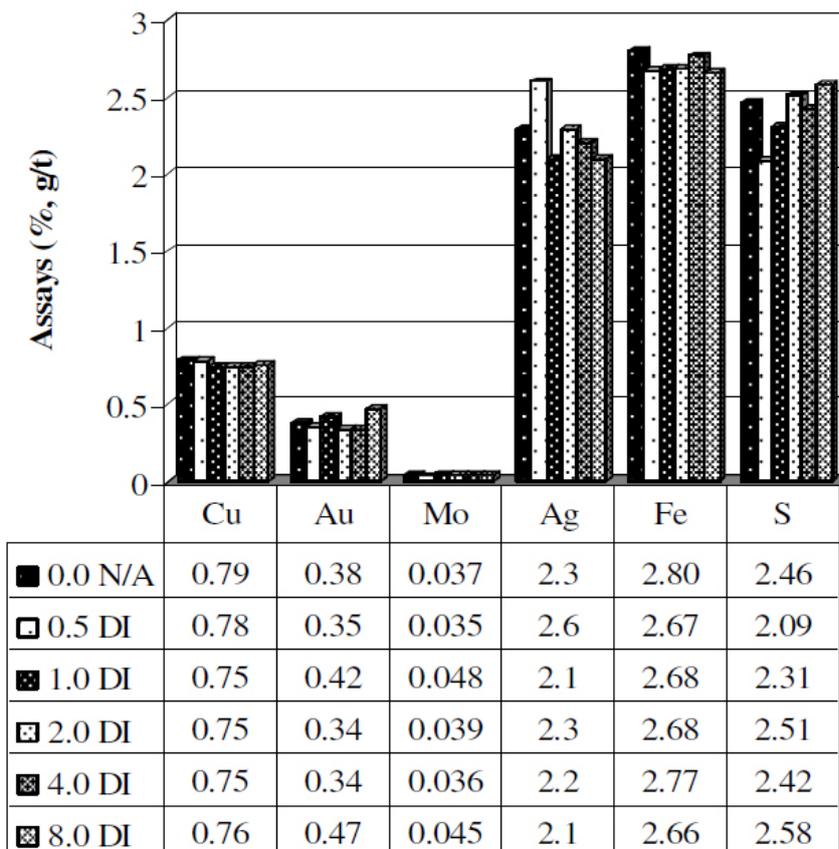


Figure 2 Variability in major head assays with ageing time (DI water)

weighted. The % of EDTA extracted Cu was then calculated taking into account the units of EDTA extracted Cu and the units of Cu in the feed sample.

A number of tests were carried out on a smaller sample mass of only 2 g. In addition, these tests were performed using a pulverised sample rather than the original -10 mesh material. However, this procedural difference did not impact the overall amount of extractable Cu by EDTA.

RESULTS AND DISCUSSION

HEAD ASSAY

The major head assays obtained for each of the ageing intervals are presented in Figure 2 and Figure 3 for the samples aged with DI and site water, respectively. Although the Ag and S assays showed some variance, clear upward or downward trends were observed with ageing period for the six elements analyzed.

Although the overall Cu analyses did not vary with ageing period, the Cu

speciation analyses did show some trends over time. The Cu distributions obtained for each sampled period are presented in Figure 4. The distribution for each of the first three leaching stages in the sequential Cu analyses increased with time, while the distribution of aqua regia leached Cu (A/R) decreased. This suggests that some of the non-leachable chalcopyrite was oxidized to easily leachable Cu-sulphoxy species.

SIZE ANALYSES

The relative deviation of the K_{30} , K_{50} , and K_{30} values presented in Figure 5 suggests only a slight decrease in the size distribution of the sample with ageing time. It is believed that the increase in size distribution at t=2 months is representative of the standard deviation of the test. Note that samples AT 0.5 and AT 1.0 were only screened to 38 microns and that they were not subjected to a Malvern analysis.

It was concluded that the size distribution differences of both samples are likely the result of test-to-test variance rather than a physical change in

the sample as a result of ageing. The following variables have an impact on the results of each size analysis, thus leading to a test-to-test variance:

- Sample Preparation;
- Relatively small sample size (150 grams);
- Sub-sampling of aged sample;
- Drying and de-agglomeration;
- Screen analysis.

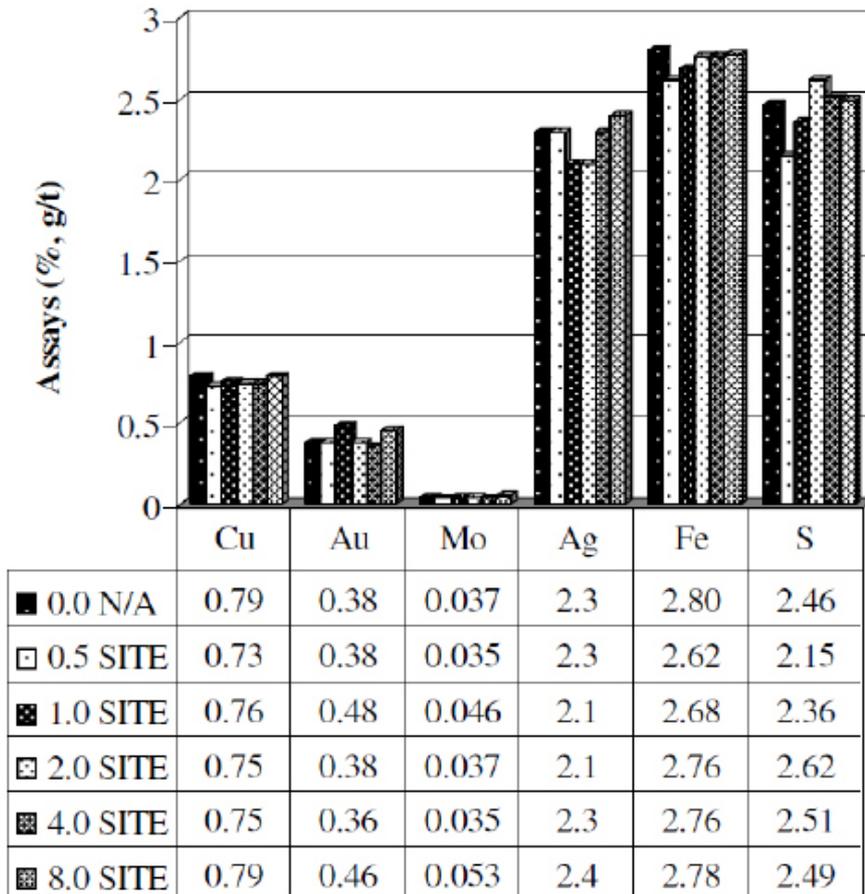


Figure 3 Variability in major head assays with ageing time (site water)

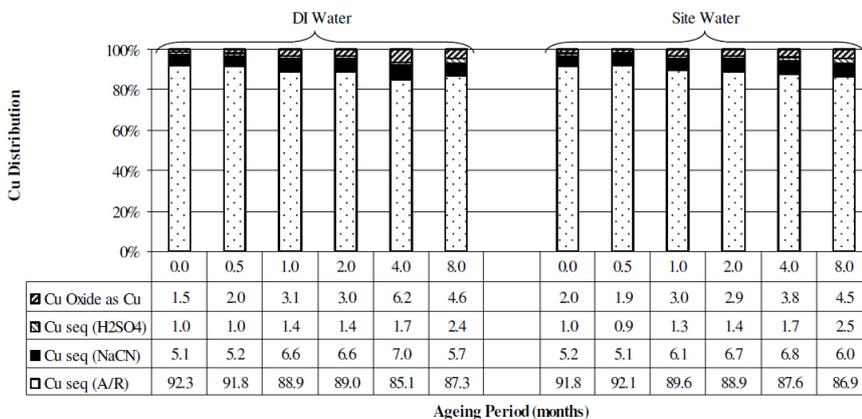


Figure 4 Effect of ageing period on Cu speciation

EDTA EXTRACTION TESTS

The exposure of the sulphide minerals to the environment over the ageing period of 8 months resulted in various surface reactions. While some reactions can be detected as released ions by measuring ion concentrations in the effluent, other reactions cannot be readily discovered, because the ions remain on the mineral surfaces. In order to quantify these reactions, an indirect analysis had to be carried out such as the extraction of ions with the chelating agent ethylenediaminetetraacetic acid (EDTA).

The results of the tests for the baseline at t=0 months and the 5 ageing periods are presented in Figure 6. The graph shows that the amount of extractable Cu gradually increased by 0.34% per month as the ageing proceeded. This was in line with the changes observed for the Cu speciation analyses.

MODIFIED ABA ANALYSIS

All samples were found to have alkaline paste pH values, but a fizz rating of 1 which suggests that the samples do not have a carbonate source of neutralization potential (NP). The bulk NP values were determined to be in the range of 5.2 to 16.5 t CaCO₃/1000 t, while all samples had sulphide contents of 2-3%, which resulted in a significant acid generation potential (AP) values, in the range of 62.0 to 79.5 t CaCO₃/1000 t. Consequently, all Net NP values were negative and all NP/AP ratios were 0.26 or less. It is generally considered that an NP/AP ratio of less than 1 indicates that a sample is acid-generating as it will generate more acidity over time than it can neutralize.

SHAKE FLASK EXTRACTION

Shake Flask Extraction tests were carried out on the original AT composite as well as the DI and site water aged samples. The concentration of most elements did not reveal any trends as the ageing proceeded with the exception of seven elements. Concentrations of Ca, K, Mg, SO₄, S, and Sr gradually increased as the ageing proceeded, while the Na concentrations decreased with time. These trends are illustrated in Figure 7.

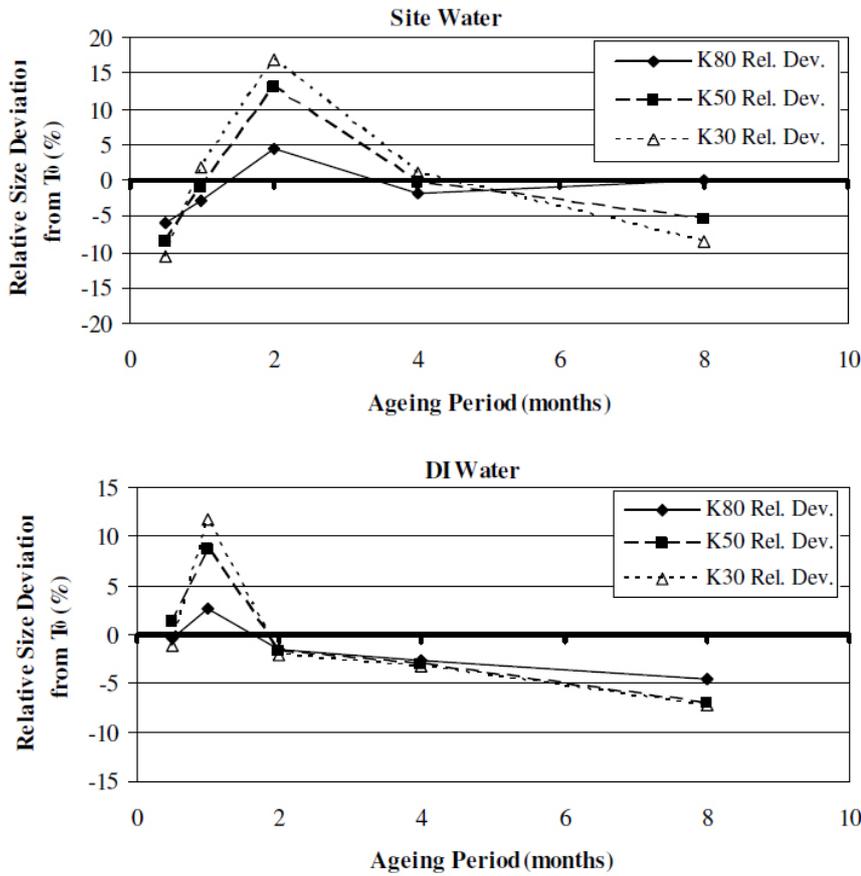


Figure 5 Effect of ageing period on size analyses

FLOTATION TESTS

The Cu, Au, and Mo grade recovery curves are depicted in Figure 8, Figure 9, and Figure 10, respectively. Two trends were observed for the Cu and Mo metallurgy. Firstly, as the ageing time increased, the grade-recovery curves for Cu and Mo deteriorated. Secondly, the Cu and Mo flotation response of the sample that was aged with site water was worse when comparing the DI and site water test results for a given ageing period.

The deterioration of the flotation response was more pronounced for Mo until the last ageing period. In the last test, the Mo grade-recovery curves improved again and outperformed the flotation response of the tests carried out at t=4 months. The ageing process does not appear to have a consistent impact on the flotation response of Au.

For this study, the impact of ageing on the metallurgical performance of the sample, was assessed by comparing the recovery of Cu, Au, and Mo to a cleaner concentrate at a standardized

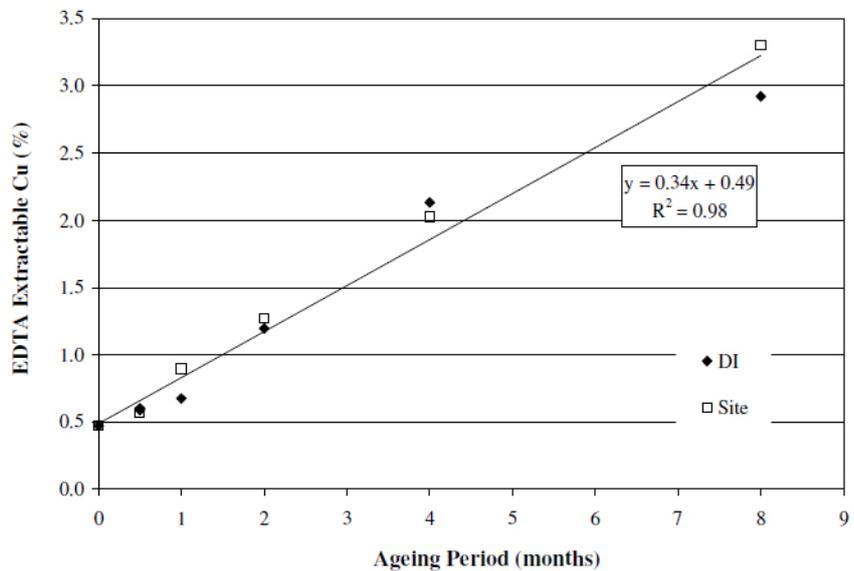


Figure 6 EDTA extractable Cu as a function of the ageing time

concentrate grade. The recovery to a 26% Cu concentrate is plotted against the ageing time in Figure 11. Copper, gold, and molybdenum recoveries to a cleaner concentrate (26%Cu) decreased with sample ageing from 90% for Cu and Mo and 60% for Au to ~80% for Cu and Mo and ~55% for Au.. Additionally, ageing with site water was more detrimental than ageing with DI water.

Trend lines were calculated for the curves in Figure 11. The trendline R2 values for Cu of 0.94 and 0.98 were exceptionally good. The fit of the trend lines for Mo and Au were moderate and poor, respectively. The poor R2 values for Au were somewhat expected due to the possible 'nuggeting' effect of Au with such relatively small samples and the relatively complex flowsheet. The recovery losses for Cu, Mo, and Au averaged 1.5%, 0.7%, and 0.8% per month, respectively (Figure 12).

WATER ANALYSIS

Since the flotation response was noticeably better for the samples that were aged with de-ionized water instead of site water, samples of both water types were submitted for a chemical analysis. Further, a sample of the Lakefield tap water was also submitted at the same time for reference purposes. The chemical analysis data of the three water samples are presented in Table 1.

The ICP scan results that are presented in Table 2 include the ratio of the concentration of each element in the site water and the de-ionized water to highlight the most significant differences between the two water samples. The site water contained concentrations of Ca, Mg, Si, and S that were between 50 and 250 times higher compared to the Lakefield de-ionized water. As a result of these higher ion concentrations in the site water, the conductivity of the site water was approximately 33 times higher than the de-ionized water. This suggests that the oxidation of the sulphide species is enhanced through ionic transfer (i.e. electrochemical reactions involving electron transfer).

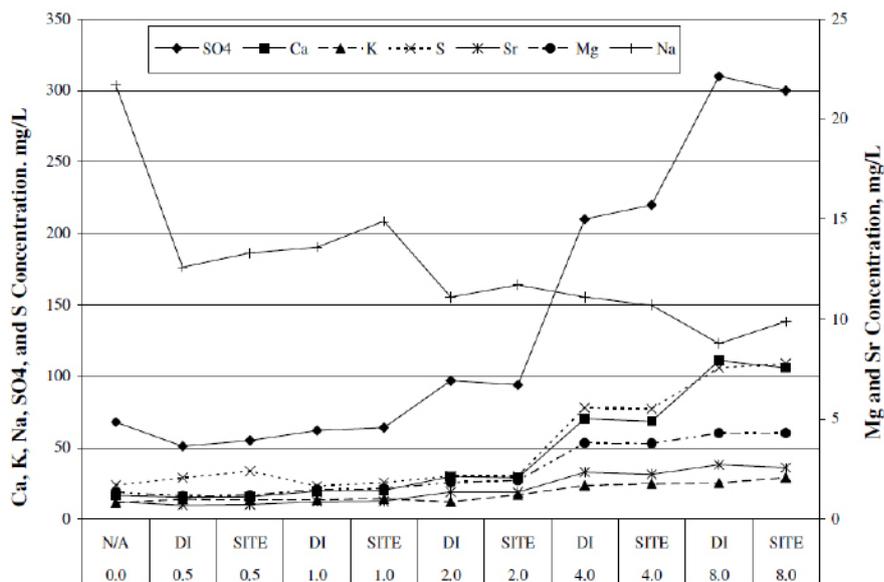


Figure 7 Shake flask test results - concentrations of selected elements

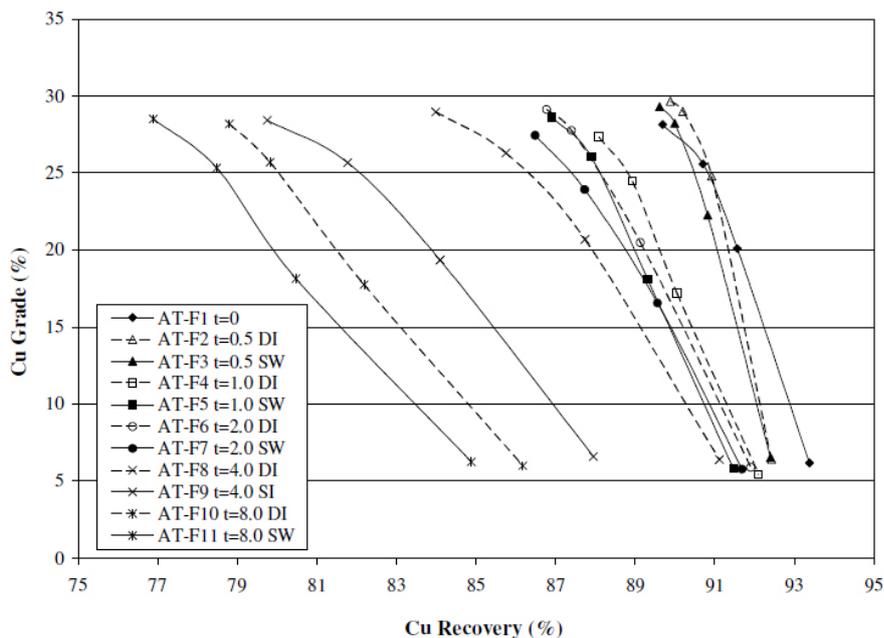


Figure 8 Cu grade-recovery curves for original AT composite and aged samples

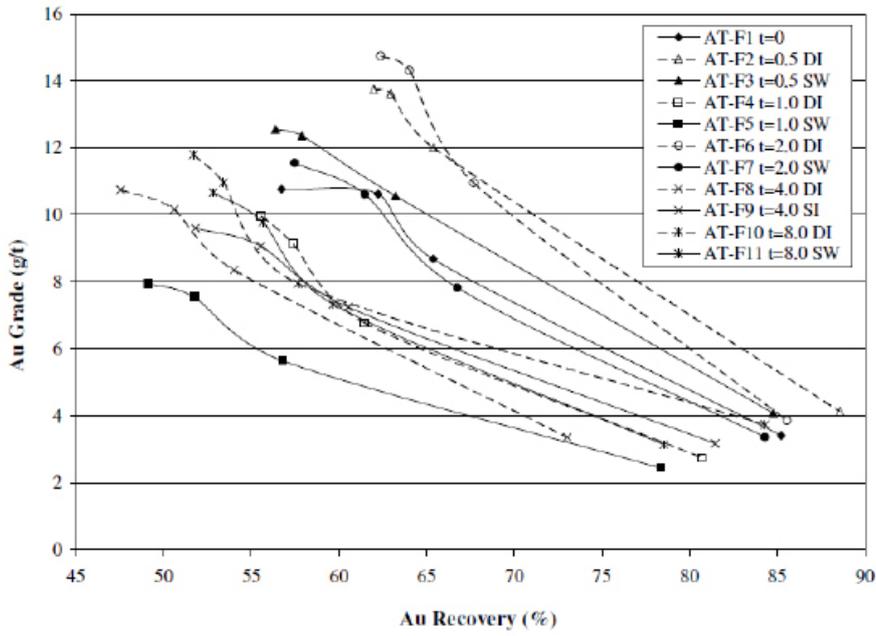


Figure 9 Au grade-recovery curves for original AT composite and aged samples

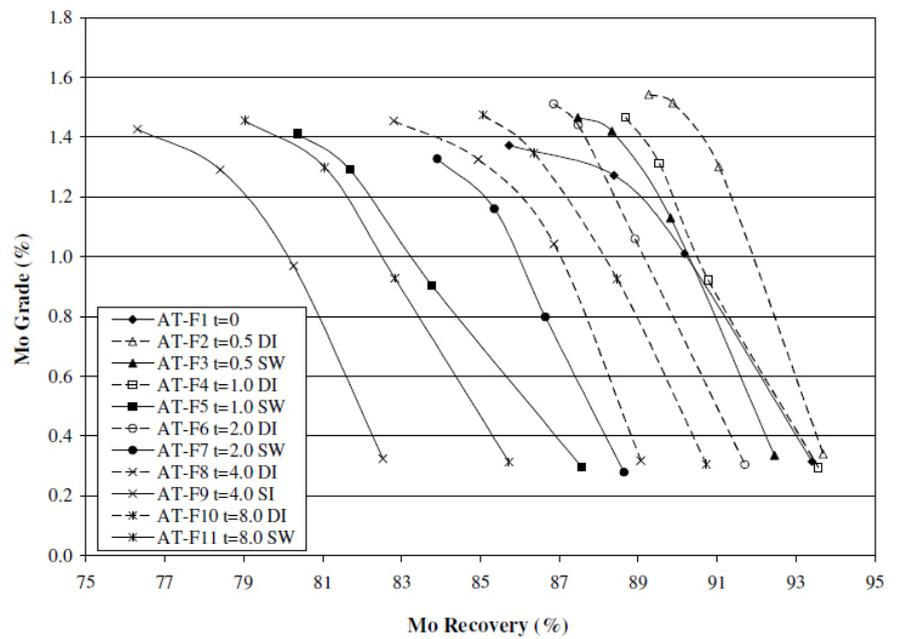


Figure 10 Mo grade-recovery curves for original AT composite and aged samples

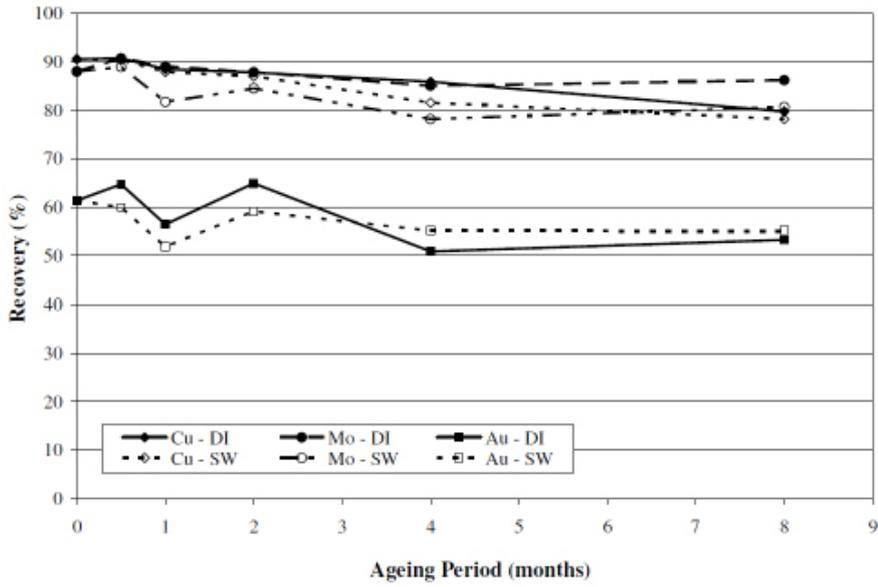


Figure 11 Cu, Au, and Mo recovery (normalized) at 26% Cu concentrate grade

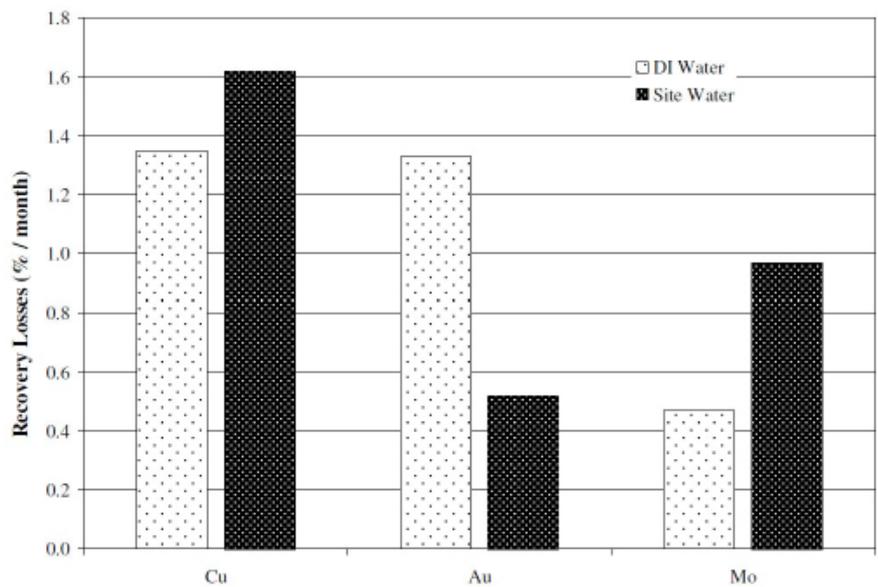


Figure 12 Cu, Mo, and Au losses as a function of ageing time

Table 1 Effluent water analysis results

SAMPLE ID UNITS	TEMPERATURE AT RECEIPT °C	TOT SUSP. SOLIDS mg/L	pH UNITS	CONDUCTIVITY uS/cm	ACIDITY mg/L as CaCO3	ALKALINITY mg/L as CaCO3
Tap Water	20	<3	6.93	67	<2	9
DI Water	20	<2	5.88	3	<2	<2
Site Water	20	2	6.96	68	<2	9
Sample ID	Tot. Dissolved Solids	HCO ₃	Carbonate	Tot. Reactive O	SO ₄	NO ₂
Units	mg/L	mg/L as CaCO ₃	mg/L as CaCO ₃	mg/L	mg/L	as N mg/L
Tap Water	149	<2	<2	<0.03	28	<0.06
DI Water	126	<2	<2	<0.03	<0.5	<0.06
Site Water	51	9	<2	<0.03	15	<0.06
Sample ID	NO ₃	NO ₂ +NO ₃	Cl	F	Fe ²⁺	Hg
Units	as B mg/L	as N mg/L	mg/L as CaCO ₃	mg/L	mg/L	ug/L
Tap Water	< 0.05	<0.06	13	<0.06	<5	<0.1
DI Water	<0.06	<0.06	<0.2	<0.06	<5	<0.1
Site Water	0.09	0.09	0.8	0.06	<5	<0.1

Table 2 Effluent water ICP results

SAMPLE ID	Assays, mg/L								
	Ag	Al	As	Ba	Be	B	Bi	Ca	Cd
Tap Water	0.00001	0.0263	0.0005	0.0265	<0.00002	0.0094	0.00003	30	0.00002
DI Water	<0.00001	0.0023	0.001	0.00054	<0.00002	0.0008	0.00002	0.12	0.00007
Site Water	0.00002	0.0145	0.0007	0.00611	<0.00002	0.0025	0.00017	6.29	0.00006
SW/DI	2	6.3	0.7	11.3	1	3.1	8.5	52.4	0.9
SAMPLE ID	Co	Cr	Fe	K	Li	Mg	Mn	Mo	Na
Tap Water	0.000263	<0.0005	0.03	0.67	<0.002	2.98	0.011	0.00023	15
DI Water	0.000205	<0.0005	0.01	0.03	<0.002	0.008	0.0184	0.00008	0.05
Site Water	0.0006	<0.0005	0.04	0.95	<0.002	1.64	0.0027	0.00034	2.48
SW/DI	0.3	0	4	31.7	1	205	0.1	4.3	49.6
SAMPLE ID	Ni	P	Pb	D	Sb	Se	Si	Sn	Sr
Tap Water	0.0043	0.01	0.00293	11.5	<0.00002	<0.001	1.76	0.00042	0.0985
DI Water	0.0026	<0.01	0.00447	0.07	<0.00002	<0.001	0.02	0.00011	0.00093
Site Water	0.003	<0.01	0.00314	5.51	<0.00002	<0.001	5.17	0.00019	0.0372
SW/DI	1.2	1	0.7	78.7	1	1	258.5	1.7	40
SAMPLE ID	Ti	Th	Tl	U	V	W	Y	Zn	
Tap Water	0.0005	0.0001	0.000003	0.000015	0.00118	<0.00003	0.00002	0.007	
DI Water	0.0002	0.0001	0.000003	0.000006	0.00005	<0.00003	0.00001	0.006	
Site Water	0.0005	0.0001	0.000006	0.000022	0.00022	0.00004	0.00003	0.023	
SW/DI	2.5	1	2	3.7	4.4	1.3	5.7	3.8	

CONCLUSION

The following conclusions can be made about the effect of crushed ore ageing on metallurgical performance of a Cu-porphyry ore:

1. Head assays of the aged samples showed no significant changes in metal value with time except for the composition of the Cu minerals
2. This translated in an increasing trend in EDTA extractable Cu with ageing time
3. There was no noticeable effect of ageing on feed size and modified acid-base accounting
4. Shake flask extraction tests showed an increase in the release of Ca, K, Mg, SO₄, S, and Sr and a decrease in Na release over time
5. Recovery of Cu, Mo, and Au (at a target 26%Cu grade) decreased at a rate of 1.6, 0.7, and 0.8 %/month, respectively, with ageing time
6. The impact on recovery was greater with site water than de-ionized water. This may be due to the significantly higher ion content of the site water

REFERENCES

E. León, A. Rate1, C. Hinz1 and G. Campbell, "Weathering of Sulphide Minerals at Circum- Neutral-pH in Semi-Arid/Arid Environments: Influence of Water Content," SuperSoil 2004: 3rd Australian New Zealand Soils Conference, University of Sydney, Australia, 5-9 December 2004, www.regional.org.au/au/asssi/

S. Kelebek and B. Nanthakumar, "Characterization of stockpile oxidation of pentlandite and pyrrhotite through kinetic analysis of their flotation," Int. J. Miner. Process., 84 (2007), pp 69-80.

D. Lascelles and J.A. Finch, "Quantifying accidental activation .Part I. Cu ion production," Minerals Engineering 15 (2002), pp 567-571

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