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Innovative Flotation Reagents: Locked Cycle Flotation Testing by Florrea Specialty Reagents for the Challenging Gold Ores in Sarawak

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Abstract. Refractory gold ores, particularly those containing sulfide, pose significant challenges. Flotation is a promising alternative to enhance the recovery of gold from these types of ore. This study investigates gold ore samples from Sarawak, Malaysia. The primary objectives were to assess the concentrate recovery and grade, and identify the effect of the recycle stream through a locked-cycle flotation test. It optimized the flotation process to enhance gold grade and recovery. The flotation process was carried out using mechanical flotation cells, along with generic and Florrea reagents, which included depressants (CMC, Florrea D140-7), activators (CuSO4, Florrea 621), collectors (PAX, Florrea X2), and frothers (MIBC, Florrea 530X). The flotation products were analyzed for Au, SxS, Fe, and Sb content using fire assay and atomic absorption spectroscopy (AAS) methods. Results from the Locked Cycle Test indicated that utilizing the Florrea Baseline yielded the best Au/S recovery, with +28.50% for Au and +18.66% for S, resulting in concentrate grades of 56.65 ppm Au and 37.35% S.

1 Introduction

Gold is one of the metals classified as a precious metal, which refers to elements that are considered rare and chemically non-reactive [1]. Among precious metals, gold is the most found and widely used in the electronics industry. This is due to its excellent electrical conductivity, making it a vital component in nearly all modern electronics, including smartphones and computers.

Gold ores are generally classified by metallurgists into two main categories: free-milling ores and refractory ores [2]. Free-milling ores are typically of the oxide type, can be liberated through milling, and are relatively easy to process, resulting in high gold recovery rates (greater than 90%). In contrast, refractory ores contain fine gold particles that are often encapsulated within sulfide minerals, such as pyrite (FeS_2). As a result, gold in this type of ore cannot be recovered solely through liberation and requires pre-treatment [3].

To process this type of refractory ore, flotation concentration is one of the alternative methods that can be used to enhance gold recovery. This is because the fine particle size of gold minerals makes gravity concentration ineffective, whereas flotation is more suitable for fine particles, typically around 200 mesh or 75 µm [4].

In the development of mineral processing circuits, a variety of laboratory- or pilot-scale tests are conducted to collect data essential for optimizing circuit design and operational parameters. One widely adopted method is the locked cycle test, which is designed to simulate the behavior of a continuous process through a series of repetitive batch tests [5]. This testing approach offers a more realistic indication of how reagent would perform and recovery can be obtained in actual plant conditions [6].

A locked cycle test consists of a sequence of batch tests in which the intermediate products (middlings) generated in each cycle (n) are recycled into the subsequent cycle (n+1). The procedure begins with a complete batch test in the initial cycle, followed by a series of iterative tests where material from the previous cycle is incorporated at the appropriate stage of the next. This repetition continues for a predetermined number of cycles. At the end of each cycle, final products, namely, the concentrate and tailings are filtered and removed from further processing, thereby simulating steady-state conditions in a continuous circuit [7].

In order to generate reliable data from locked cycle tests, achieving a steady state is essential. Despite their various advantages, several limitations have been identified through experimental studies. These tests are often time-intensive, as reaching a steady state typically requires at least five cycles even in simple circuits, and generally six or more cycles are needed. Additionally, as the volume of recycled middlings increases, attaining a steady state becomes increasingly challenging [5].

In this study, we will analyze the recovery and concentrate grade of gold, as well as identify the effects of the recycle stream on gold ore from the Sarawak region of Malaysia using locked cycle flotation tests.

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This test will also be conducted using three different reagent scheme variations. The first test will use generic reagents, while the second and third tests will utilize specialized reagents from Florrea. The difference lies in the second test scheme, which will be conducted without the use of a depressant, whereas the third test will include the use of a depressant.

2 Methodology

2.1 Materials

The samples used in this study is sulphide ore from Bau, Serawak, Malaysia. The samples were sulphide ores containing gold (arsenic pyrite, arsenopyrite, and gangue). Ore grades were Au: 3.55 ppm and S: 2.7%. The chemical used in the experiment is activators (CuSO4, Florrea 621), depressants (CMC, Florrea D140-7), collectors (PAX, Florrea X2) and frothers (MIBC, Florrea 530X). Distilled water was used to dilute solid chemical. Meanwhile, tap water was used as make-up water for slurry preparation.

2.2 Sample characterization

The experimental procedure encompassed locked cycle, mechanical flotation, and column flotation. Samples, weighing 1100 grams per pack, were prepared using a jaw crusher, roll crusher, coning and quartering, and a rotary splitter. The prepared sample was then ready for flotation, with each pack added to the grinding to achieve a target P80 of 106 µm. After that, the slurry from grinding ready for flotation test.

2.2.1 *Locked Cycle Experiment*

The locked cycle test can be performed by circulating the mechanical flotation product for 4 cycles. Locked cycle flotation test consists of a rougher-scavenger circuit and a cleaner circuit. Scavenger concentrate is recycled back to its previous stage, so that rougher-scavenger product comes only from rougher concentrate which is then feed to the cleaner circuit. While the tail of each stage is a feed of its next stage and tail of scavenger 3 is become final tail of each cycle. Then the first stage cleaner concentrate will feed the next stage cleaner until the final concentrate is obtained each cycle and the tail of the cleaner circuit will be recycled back to the previous stage. During flotation, pH, Eh, water flow, and agitation speed are checked to ensure that each test is comparable. The detailed flowsheet for the locked cycle test can be seen in Fig. 1.

2.2.2 *Open Cycle Experiment*

Similar to locked cycle, open cycle consists of rougher- scavenger circuit and cleaner circuit. In open cycle flotation, the rougher-scavenger concentrate will feed the cleaner circuit. Unlike the locked cycle, the next cleaner stage feed uses the tail of the previous cleaner stage. During flotation, pH, Eh, water flow, and agitation speed are checked to ensure that each test is comparable. The detailed flowsheet for the locked cycle test can be seen in Fig. 2.

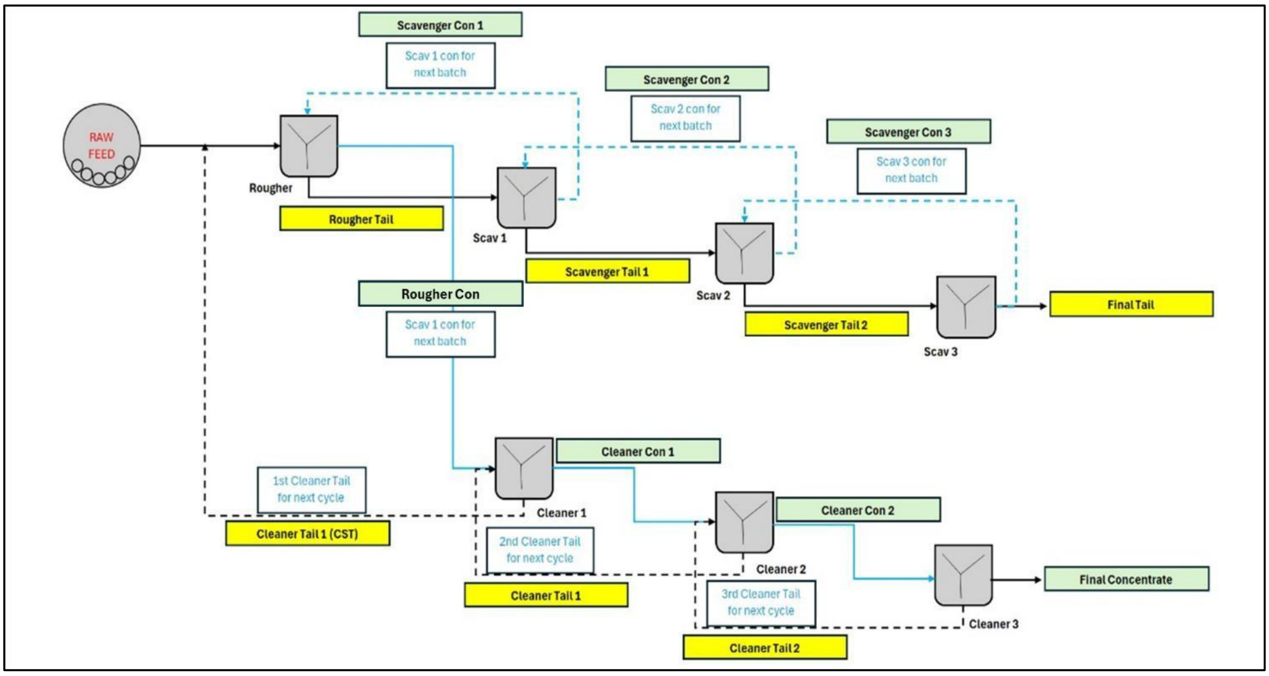

Fig. 1. Locked Cycle Test Flowsheet

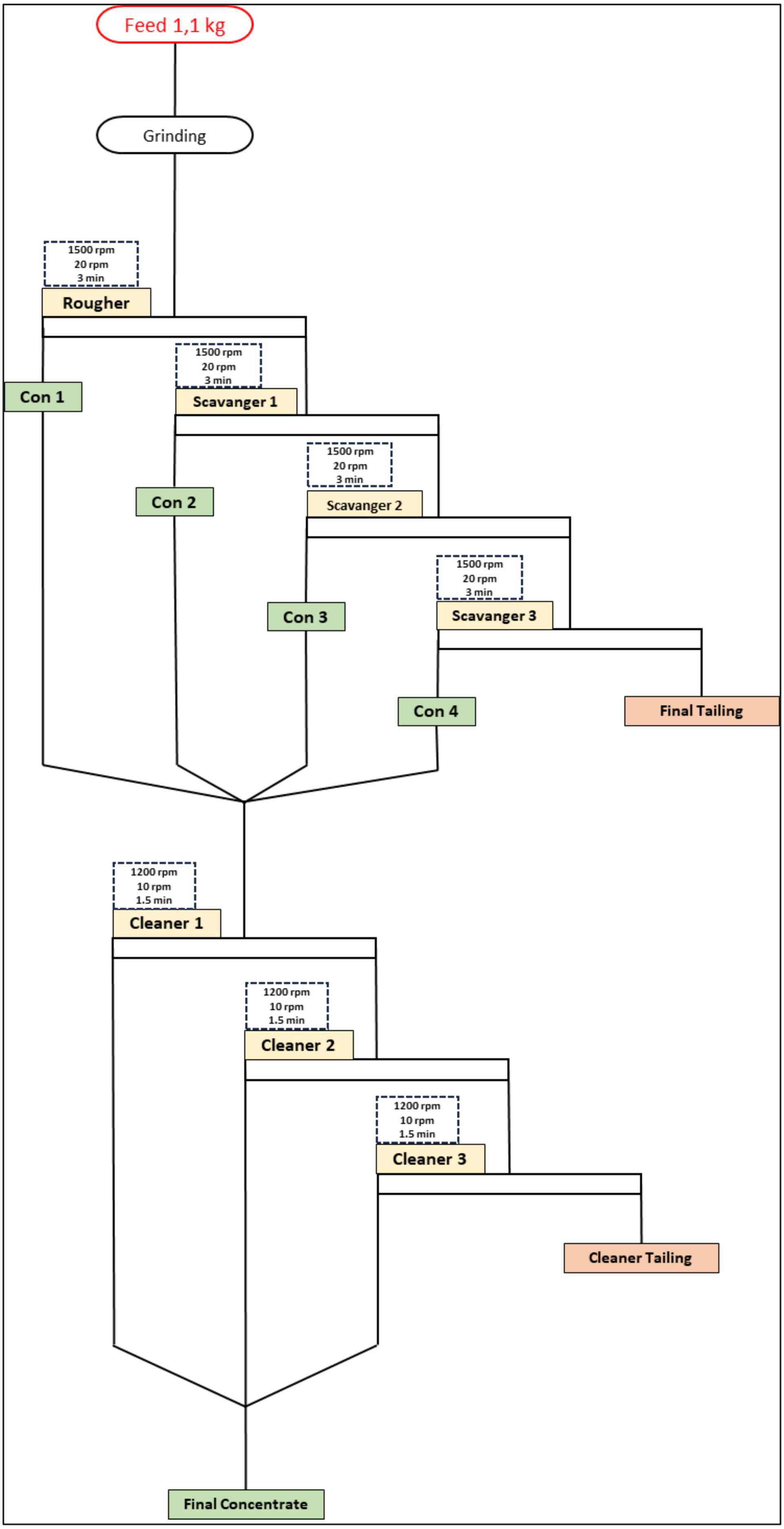

Fig. 2. Open Cycle Test Flowsheet

3 Results and discussion

3.1 Mass pull result

Based on the results obtained, the mass pull in the locked cycle test shows a decrease compared to the open cycle test, both the mass pull in the rougher-scavenger circuit and the mass pull in the cleaner circuit. To find the raw data of mass pull, the following calculations are performed.

$$Mass\ Pull\ = \frac{Concentrate\ Mass\ (gr)}{Feed\ Mass\ (gr)} \times 100\%$$

Fig. 3 shows the mass pull locked cycle and open cycle test graphs.

Based on circuit performance in locked cycle test, the circulating load for the first test or T1 (using generic reagents) up to 4.7% mass pull. Then the second test or T2 (using Florrea reagents without depressant), up to 16.5% mass pull. Last the third test or T3 (using Florrea reagents with depressant), up to 16.5% mass pull.

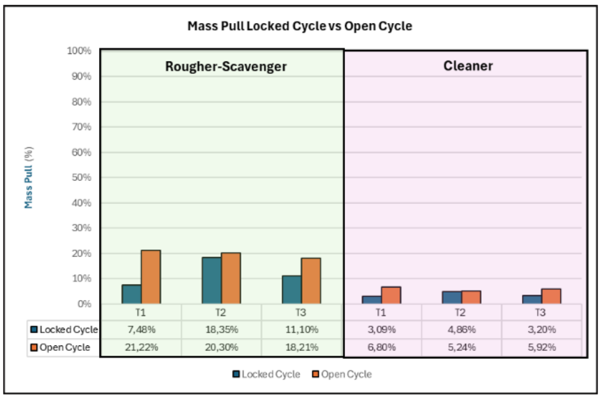

Fig. 3. Mass Pull Locked Cycle vs Open Cycle

3.2 Au recovery and grade result

The data obtained (by measurement) will be subjected to a metallurgical balance for the four cycles that have been declared stable. To find the raw data of recovery, the following calculations are performed.

$$Recovery = \frac{Concentrate\ Mass(gr) \times Metal\ Grade}{Feed\ Mass\ (gr) \times Metal\ Grade} \times 100\%$$

3.2.1 Locked Cycle and Open Cycle Test using Generic Reagents

In the first test (T1), which used generic reagents, the results showed that Au recovery in the open cycle (70.18%) was higher than in the locked cycle (51.00%), with a difference of 19.18%.

Contrast to the Au recovery, the Au grade in the cleaner concentrate has a trend that shows an increase in Au grade in the locked cycle test. The Au grade increase of 21.29 ppm, with the acquisition by the open cycle test of 35.80 ppm and the locked cycle test of 57.09 ppm.

Based on circuit performance in locked cycle test, overall, Au recovery is lower than rougher-scavenger 51.0% vs 59.8%. It is reflecting the effect of cleaner tail (CST) in the locked cycle flotation test as a result of concentrate grade enrichment through cleaner flotation, this recycle effect is not captured in rougher-scavenger flotation test. From 59.8% Au recovery in rougher- scavenger, almost 13.5% Au is recovered from scavenger stages. Fig. 4 shows the circuit performance locked cycle for the first test.

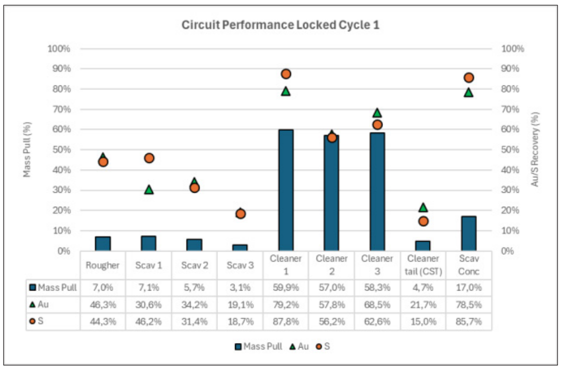

Fig. 4. Circuit Performance Locked Cycle 1

In rougher-scavenger circuit, Au was predominantly collected at rougher stage with recovery 46.3%, and Au grade is 32.1 g/t. Scavenger stages were only collecting the remain small amount of Au in rougher tail. High Au recovery at rougher stage contributed by recycling of scavenger concentrate which is increase the probability of sulphide minerals to floating and being recovered.

However, Au concentrate grade in rougher-scavenger is relativity high 20.76 g/t. Strategy of recycling back scavenger concentrate slurry to its previous stage has a positive effect on grade, but not for concentrate recovery.

3.2.2 Locked Cycle and Open Cycle Test using Florrea Reagents without Depressant

Then for the second test (T2), using Florrea reagents without depressant, Au recovery reached 67.68% in the open cycle test and 79.50% in the locked cycle test.

The Au grade obtained in the open cycle test was 43.80 ppm and the locked cycle test was 56.65 ppm, so that the increase in the locked cycle test was 12.85 ppm.

Based on circuit performance in locked cycle test, overall, Au recovery is lower than rougher-scavenger 79.5% vs 84.7%. It reflects the effect of cleaner tail (CST) in the locked cycle flotation test as a result of concentrate grade enrichment through cleaner flotation, this recycle effect is not captured in rougher-scavenger flotation test. From 87.47% Au recovery in rougher-scavenger, almost 19.7% Au recovered from scavenger stages. Fig. 5 shows the circuit perfomance locked cycle for the second test.

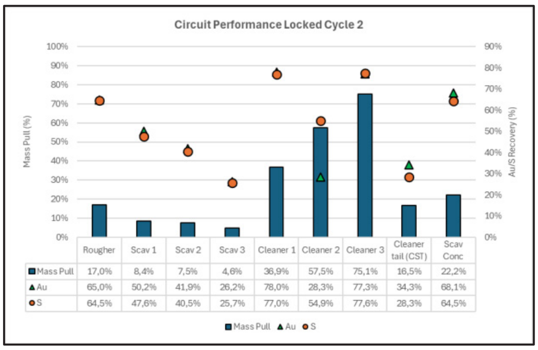

Fig. 5. Circuit Performance Locked Cycle 2

In rougher-scavenger circuit, Au was predominantly collected at rougher stage with recovery 65%, and Au grade is 18.4 g/t. Scavenger stages were only collecting the remain small amount of Au in rougher tail. High Au recovery at rougher stage contributed by recycling of scavenger concentrate which is increase the probability of sulphide minerals to floating and being recovered.

However, Au concentrate grade in rougher-scavenger is relativity high 14.44 g/t. Strategy of recycling back scavenger concentrate slurry to its previous stage has a positive effect on grade, but not for concentrate recovery.

3.2.3 Locked Cycle and Open Cycle Test using Florrea Reagents with Depressant

In the third test (T3), which used Florrea reagents with depressant, the Au recovery between the two cycles was not significantly different, with 70.87% for the open cycle and 71.73% for the locked cycle, where the Au recovery from the locked cycle test was slightly higher than the open cycle.

The Au grade increase and obtained in the locked cycle test of 36.65 ppm, with the acquisition of Au grade of 77.45 ppm and in the open cycle test of 40.80 ppm.

Based on circuit performance in locked cycle test, overall Au recovery is lower than rougher-scavenger 71.7% vs 81.9%. It reflects the effect of cleaner tail (CST) in the locked cycle flotation test as a result of concentrate grade enrichment through cleaner flotation, this recycle effect is not captured in rougher-scavenger flotation test. From 81.9% Au recovery in rougher-scavenger, almost 22.6% Au recovered from scavenger stages. Fig. 6 shows the circuit perfomance locked cycle for the third test.

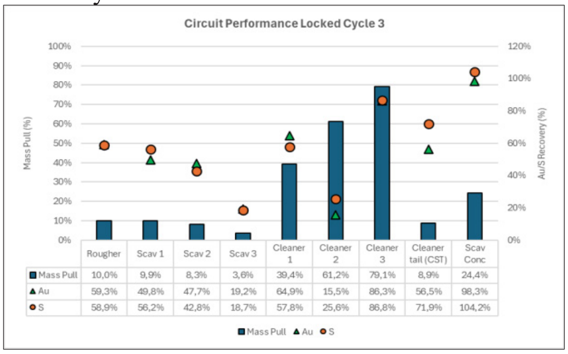

Fig. 6. Circuit Performance Locked Cycle 3

In rougher-scavenger circuit, Au was predominantly collected at rougher stage with recovery 59.3%, and Au grade is 36.7 g/t. Scavenger stages were only collecting the remain small amount of Au in rougher tail. High Au recovery at rougher stage contributed by recycling of scavenger concentrate which is increase the probability of sulphide minerals to floating and being recovered.

However, Au concentrate grade in rougher-scavenger is relativity high 21.47 g/t. Strategy of recycling back scavenger concentrate slurry to its previous stage has a positive effect on grade, but not for concentrate recovery.

The all Au recovery (Open cycle and locked cycle test) shows in Fig. 7 and the all Au grade (Open cycle and locked cycle test) shows in Fig. 8.

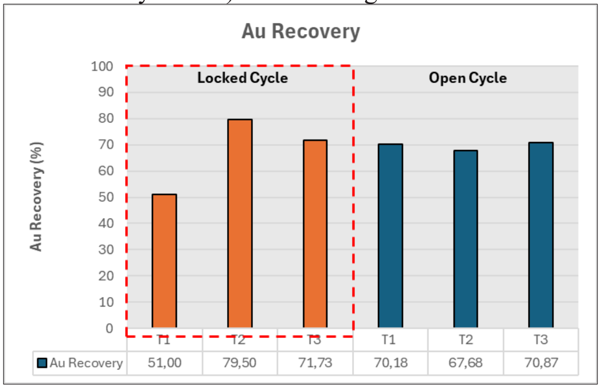

Fig. 7. Au Recovery for Open and Lock Cycle

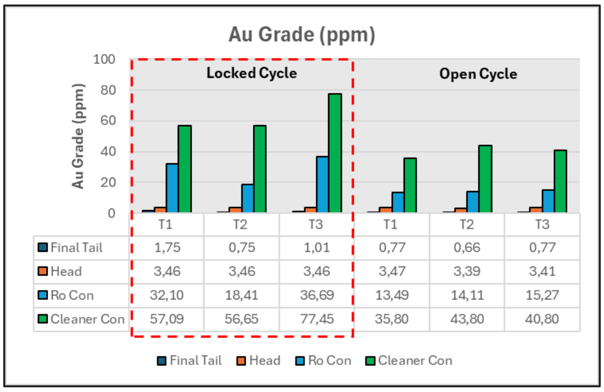

Fig. 8. Au Grade for Open and Lock Cycle

3.3 S recovery and grade result

In the first test (T1), the results showed that S recovery in the open cycle (73.55%) was higher than in the locked cycle (59.85%), with a difference of 13.70%.

Then for the second test (T2), S recovery reached 65.32% in the open cycle test and 78.51% in the locked cycle test.

In the third test (T3), the S recovery for the open cycle 68.38% and 71.7% for the locked cycle, where the S recovery from the locked cycle test was slightly higher than the open cycle. The graph of S recovery can be seen in Fig. 9.

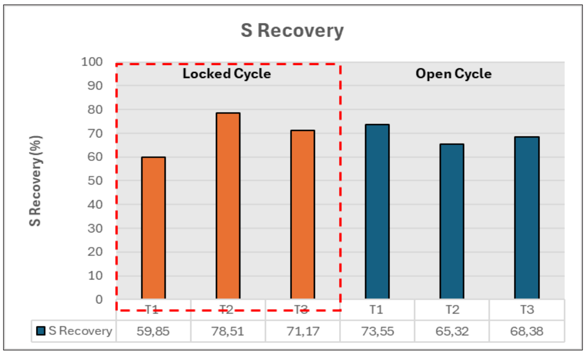

Fig. 9. S Recovery for Open and Lock Cycle

Similar with Au grade, the S grade in the cleaner concentrate has a trend that shows an increase in S grade in the locked cycle test.

In the first test (T1) there was an increase of 17.43 ppm, with the acquisition by the open cycle test of 27.30 ppm and the locked cycle test of 44.73 ppm.

Then in the second test (T2), the S grade obtained in the open cycle test was 29.60 ppm and the locked cycle test was 37.35 ppm, so that the increase in the locked cycle test was 7.75 ppm.

Furthermore, in the third test (T3) an increase was obtained in the locked cycle test of 26.60 ppm, with the acquisition of S grade of 51.30 ppm and in the open cycle test of 24.70 ppm. The graph of S grade can be seen in Fig. 10.

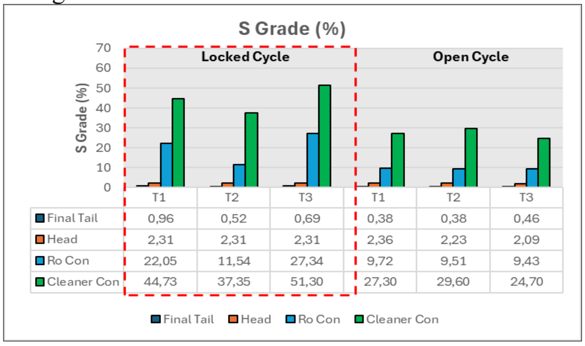

Fig. 10. S Grade for Open and Lock Cycle

3.4 Enrichment ratio

Enrichment Ratio (ER) is one of parameters assessed to determine the flotation circuit performance in capability of upgrading the concentrate grade in each flotation circuit configuration. ER is defined as the ratio of feed grade to concentrate grade in a flotation circuit. The enrichment ratio equation can be seen as follows :

$$ER = \frac{c}{f} \qquad (1)$$

Where c is concentrate grade and f is feed grade.

This ER values are reasonably good in respect to the ore characteristics. However, optimum ER value should be confirmed through a separate lab test program. The optimum ER value is depending on the ore type, processing technology, flotation reagent, as well as the targeted trade- off between recovery and concentrate grade.

Based on enrichment ratio (ER), locked cycle test can significantly increase Au and S grade, as seen from the higher ER compared to open cycle test in the Fig. 11.

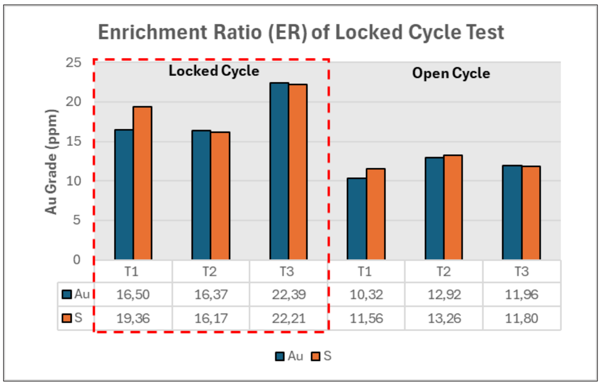

Fig. 11. Enrichment Ratio (ER) of Locked Cycle Test

3.5 The effect of the different reagent on Au recovery and grade in locked cycle test

Basically, the reagents used in tests 1 (T1) and 3 (T3) have the same base components. 621 is a mixture of sulfate with CuSO4 base, X2 is a formulated xanthate, and D140-7 has a similar function to CMC. Meanwhile, test 2 (T2) only uses activator, collector, and frother reagents.

It can be seen from the locked cycle results that testing using Florrea reagent (T1) increases Au recovery and grade compared to using generic reagent (T2). This is possible because Florrea reagent is formulated or modified from the base component (generic reagent) which has a high compatibility with the characteristics of this ore.

Then there is a comparison between the use of depressants and no use of depressants. Wills [4] mentions that depressants are used to increase the selectivity of valuable minerals by preventing impurity minerals from floating during flotation. Therefore, valuable minerals that are not completely liberated from gangue minerals can be depressed during flotation. As a result, the recovery of these valuable minerals will decrease, but the grade of valuable minerals will increase.

It can be seen test 2 (T2) in the locked cycle test, which does not use a depressant, has the highest Au recovery compared to tests that use a depressant. However, the Au grade in test 2 (T2) is quite low when compared to test 3 (T3) and is not better than test 1 (T1). The difference in grades between tests 2 (T2) and 3 (T3) shows that the use of depressants here works well, resulting in a high Au grade. However, when compared to test 1 (T1) and test 2 (T2), the use of depressants in test 1 (T1) did not show good separation of valuable minerals. This can be seen from the insignificant difference in Au grades obtained, which were only slightly different with a considerable difference in recovery between them.

3.6 The effect of the locked cycle on Au recovery and grade

As stated by Philip Keller [6], locked cycle tests can provide the best representation of reagent application in real flotation plants, compared to open cycle or single flotation tests. This is because locked cycle tests are continuous tests involving the recycling of certain products (such as concentrate or tailings from each stage) into the next cycle, in accordance with a pre-designed circuit. Therefore, the recovery and grade of valuable metals obtained from locked cycle tests can closely reflect the recovery and grade expected in the plant.

Based on the results of the locked cycle test, the highest recovery and grade were obtained in the second test (T2) using Florrea reagents without depressant, with Au/S recovery of +28.50% Au and +18.66% S, and grades of 56.65 ppm Au and 37.35% S, compared to Au/S recovery the first test (T1) using generic reagents.

However, the effect of recycling products in locked cycle tests, only have a positive impact on the Au grade, but not necessarily on the Au recovery. This may be due to the repeated recycling process in the locked cycle, which separates valuable minerals from gangue, allowing only high-purity valuable minerals to persist through each recycle. In contrast, valuable minerals with contaminants are less likely to remain and are instead carried to the tailings. This phenomenon explains the observed decrease in recovery and increase in the grade of valuable minerals, as also stated by Robert C. Dunne et al [8]

4 Conclusion

The locked cycle test method is one of the flotation methods that provides the most accurate representation of a real flotation plant, particularly in terms of reagent usage and the recovery and grade of valuable minerals, compared to the open cycle test.

Based on the obtained data, Au/S recovery in the locked cycle test show better results (but not significant) than the open cycle test. However, the Au/S grade in the locked cycle test was higher compared to the open cycle test. This may be due to the repeated recycling of products in the locked cycle test, which enhances the selectivity of valuable minerals being recovered. As a result, valuable minerals associated with contaminants are discarded into the tailings, leading to a decrease in recovery but an increase in Au/S grade.

The results of locked cycle are the most representative of what would be expected in a real flotation plant. Among the three test schemes conducted, the best results were obtained in the second test using Florrea reagents without depressant, with Au/S recovery of +28.50% Au and

+18.66% S, and grades of 56.65 ppm Au and 37.35% S, compared to Au/S recovery the first test using generic reagents.

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