

# Impact of Recirculating Mining Process Water on the Flotation Performance of a Sedimentary Phosphate Ore

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**Abstract** – Water is one of the scarce resources in the world and the mining industry like many other sectors is expected to discretionally utilise this resource sustainably. It was on the premise of adding to the body of knowledge related to water sustainability and flotation performance that an investigation was prompted to evaluate the impact of recirculating mining process water on the flotation performance of a sedimentary phosphate ore. A series of flotation locked cycle tests were conducted with the objective to simulate recycling process water, which by default modifies the water chemistry and influences flotation performance. The laboratory flotation results showed that recycling process water in the laboratory scale did not show significantly noticeable influence on  $P_2O_5$  grade and recovery performance. A saleable product of 33.27%  $P_2O_5$  grade at reasonable average recovery of 65% was achievable. Water analyses revealed that there was an increase in total dissolved solids [452 to 846mg/L], electrical conductivity [662  $\mu\text{m}/\text{cm}$  to 1025  $\mu\text{m}/\text{cm}$ ],  $\text{CaCO}_3$  [146 to 304mg/L], sodium [57.7 to 76.6mg/L], chloride [100.7 to 109.7mg/L] and magnesium [10.4 to 12.6mg/L] after circulating the process water for 15 cycles. The results achieved however confirmed the importance of water quality and understanding process water chemistry in flotation processes. The compounding salts and inorganic elements in the water necessitate the need for water clarifiers for process water prior to being recycled to the flotation process.

**Keywords:** Sedimentary phosphate, Water quality, Flotation, Process water, Water chemistry.

## 1. Introduction

Water chemistry in flotation processes continues to be a challenge although there has been previous studies and details in literature [1, 2]. The present work focuses on the Sedimentary phosphate ore fines circuit (-212+20 $\mu\text{m}$ ) flotation feed samples, where bench-scale simulation of recycled process water was carried out to evaluate the influence on flotation performance and water quality. One of the major categories of a flotation system is the chemistry component which includes amongst others collectors, depressants, frothers and pH adjusters which are interrelated [1]. There are various parameters that influence the quality of water and they include amongst others, total suspended solids (TSS), total dissolved solids (TDS), electrical conductivity (EC), Iron (Fe) content and typical contaminants such as amongst others Magnesium (Mg), Sulphate ( $\text{SO}_4$ ), Calcium (Ca), Potassium (K) and Sodium (Na). All these parameters are critical and each or combined have a direct influence on the quality of water [2].

The use of strong inorganic acids causes accumulation of ions in the process water, especially  $\text{Ca}^{2+}$  and  $\text{PO}_4^{2-}$  which leads to problems in apatite recovery and in the environment [3]. The influence of water quality on the flotation performance of complex sulphide ores showed that the influence of process water on lead flotation depends on its composition and concentrations of constituents. It was established amongst others that the addition level of recycled water (during the grinding or at the start of flotation) has no significant effect on the flotation of the complex sulphide ores [4].

In this study, a comprehensive water analysis of recirculated process water and its impact on Apatite recovery from a phosphate sedimentary rock is presented.

## 2. Method

The sample used in this study was sourced from sedimentary phosphate mine in Western Cape, South Africa. A series of laboratory flotation tests were conducted in the laboratory using Denver D12 flotation continually simulating recycling process water. For each cycle completed, a sub-sample of recycled process water was collected and sent for chemical analysis.

The flotation tests were conducted continuously on a bench-scale and the products for each test were filtered, dried, weighed and submitted for X-Ray fluorescence (XRF) chemical analysis. A total of 15 locked cycles were conducted and process water was simulated by decanting and recycling water streams. Fig. 1 shows the sample preparation process flow prior to flotation tests.

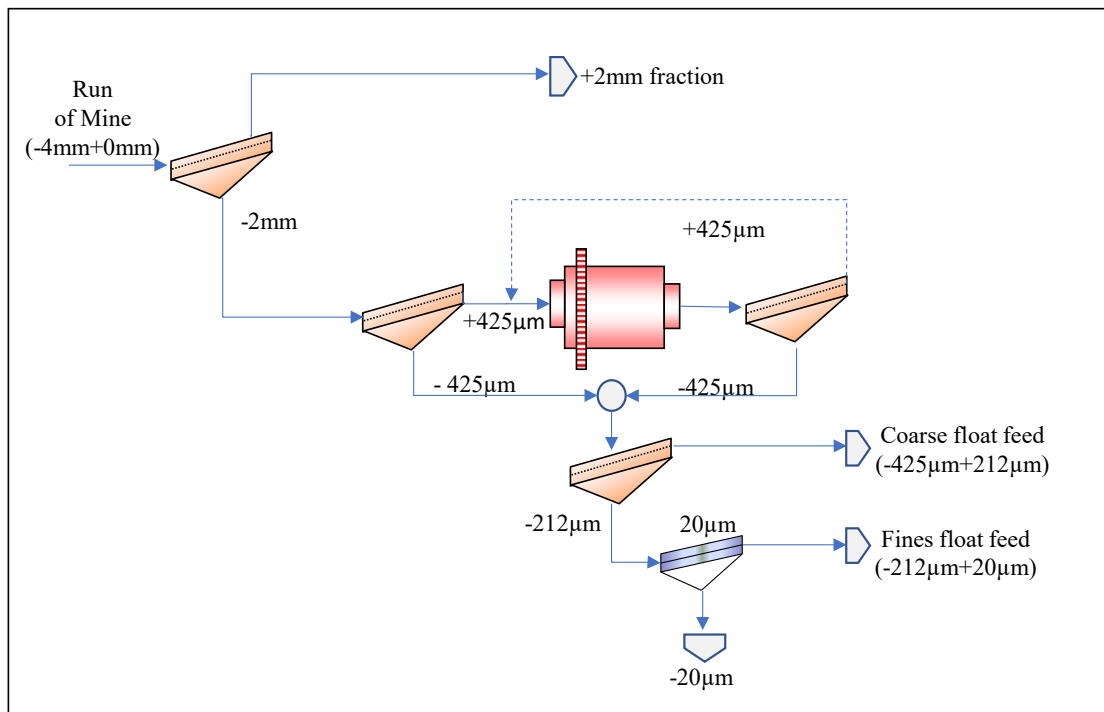


Fig. 1: Sample preparation (Milling and screening)

From Fig. 1, sample preparation involved dry screening Run of Mine (ROM) at 2mm screen and further screening the -2mm fraction at 425µm. The +425µm fraction was milled using laboratory ball mill (D 0,265m, L0,305m) in closed circuit to produce 100%-425µm which was wet screened at 212µm to produce fines (-212µm) and coarse (-425µm+212µm). The stream of focus for the test work was the fines circuit where the -212µm was further deslimed at 20µm to remove ultra-fines that have proven to be a challenge in flotation processes. For the purpose of the bench-scale test work, approximately 11kg bulk sample (-212+20µm) was produced and split homogeneously using a rotary splitter into 700 grams representative samples for further laboratory test work.

Laboratory flotation tests were conducted using a Denver D12 flotation machine as shown in Fig. 2.

Fig. 2 shows that the fines ( $-212\mu\text{m}+20\mu\text{m}$ ) from milling and screening were used for 15x locked cycle flotation test. All the exiting water streams were collected, combined and reused to simulate recycling process water. The process involved scrubbing the float feed sample in the presence of Sodium Silicate at 50-60% solids for 3min, with the aim to enhance flotation by increasing flotation rates of phosphates when compared to silicates and therefore improving selectivity and yielding faster flotation rates. The scrubbing action is expected to aid in removing impurities and with separating clay coatings from phosphate mineral surface.

The resulting pulp was deslimed using a  $20\mu\text{m}$  screen. Desliming oversize fraction was transferred into a Denver D12 for direct flotation. Anionic collector (herein referred to as Fatty Acid (FA) was added and conditioned at approximately 60% solids and floated until froth collapsed and dissipates. The Anionic collector is expected to attach to the mineral surface and with the introduction of airflow produce bubbles loaded with the positively charged mineral surface apatite. The FA concentrate was de-oiled with sulphuric acid ( $\text{H}_2\text{SO}_4$ ) and the pH was shifted from acidic to alkaline medium (7.5-8.5) prior to reverse flotation for every cycle.

Cationic collector (herein referred to as Amine) was conditioned for 30 seconds at approximately 50% solids and with introduction of airflow, reverse flotation was effected and silica was collected over the cell-lip. For reverse flotation, Amine collector attached to the negatively charged minerals surfaces (silicates) whilst the negatively charged mineral surface (apatite) stayed in the liquid phase due to the bubbles failing adhere to its surfaces, thus being hydrophilic and herein referred to as 'reverse-conc'. The Fatty acid and Amine chemicals used were from Arrmaz (USA).

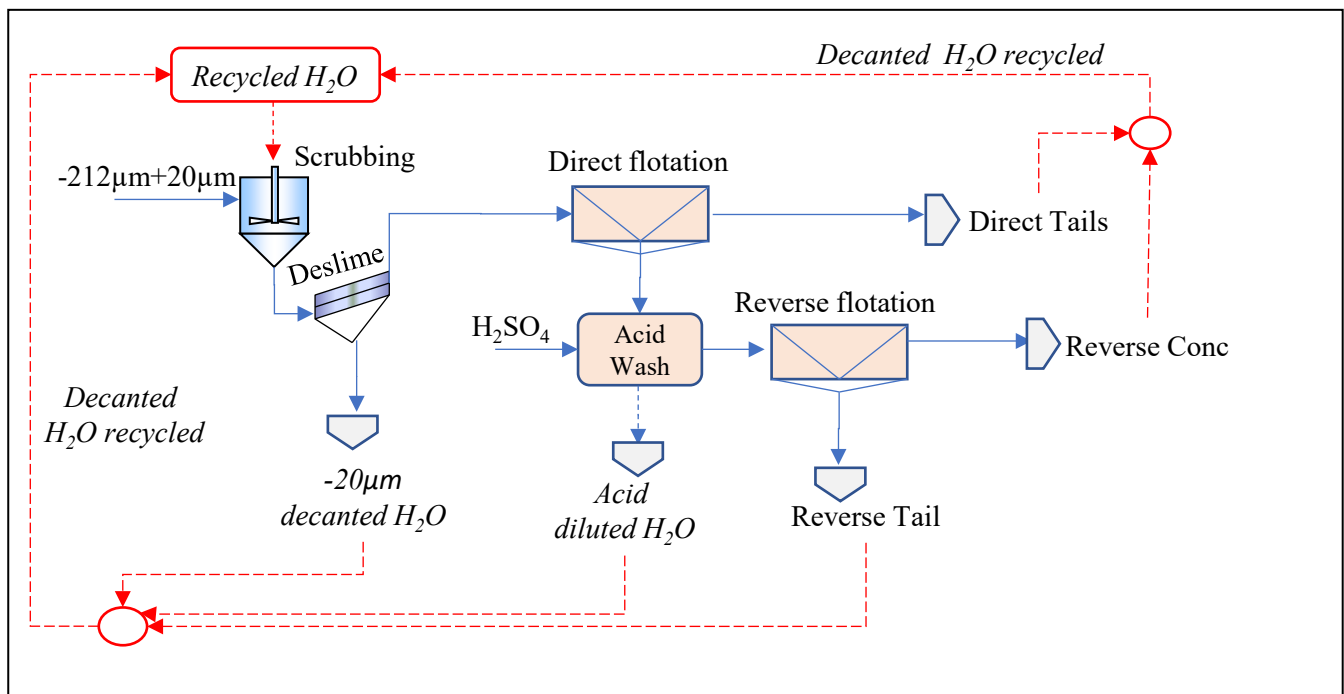


Fig. 2: Laboratory flotation test work process flow

For each cycle completed, a sub-sample of recycled process water was collected and sent for chemical analysis. The flotation tests were conducted continuously on a bench-scale and the products for each test were filtered, dried, weighed and submitted for major phosphate minerals analysis using XRF press pellet method.

### 3. Results

#### 3.1 Bench Flotation Results

The flotation cycles were grouped to minimised inconsistencies and laboratory errors. During the flotation tests, no variables were altered, reagents dosages were kept consistent for all the flotation cycles. The major elements in the head grade, direct and reverse flotation concentrates are summarised in Tables 1, 2 and 3.

Table 1: Head grade analysis of flotation feed sample (-212 $\mu$ m+20 $\mu$ m)

Flotation Cycles	Head grade (%)					
	P <sub>2</sub> O <sub>5</sub>	SiO <sub>2</sub>	MgO	CaO	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>
Cycle 1 - 3	7.69	77.5	0.09	10.96	0.86	0.57
Cycle 4 – 6	8.25	76.05	0.13	11.67	0.88	0.61
Cycle 7 – 9	8.07	76.34	0.12	11.42	0.85	0.62
Cycle 10 - 12	8.08	76.62	0.10	11.40	0.91	0.61
Cycle 13 - 15	8.04	76.75	0.11	11.42	0.86	0.58

Table 2: Direct flotation concentrate

	Mass pull	Grade %		Recovery %		
	wt %	P <sub>2</sub> O <sub>5</sub>	SiO <sub>2</sub>	P <sub>2</sub> O <sub>5</sub>	SiO <sub>2</sub>	CaO/P <sub>2</sub> O <sub>5</sub>
Cycle 1 - 3	22.34	23.27	36.47	67.61	10.51	1.38
Cycle 4 – 6	34.64	20.60	43.76	86.50	19.93	1.38
Cycle 7 – 9	36.55	19.45	46.41	88.05	22.22	1.38
Cycle 10 - 12	25.38	22.90	37.43	71.90	12.40	1.38
Cycle 13 - 15	25.43	22.68	37.89	66.14	11.57	1.38

Table 3: Reverse flotation concentrate

	Mass pull	Grade %		Recovery %		
	wt %	P <sub>2</sub> O <sub>5</sub>	SiO <sub>2</sub>	P <sub>2</sub> O <sub>5</sub>	SiO <sub>2</sub>	CaO/P <sub>2</sub> O <sub>5</sub>
Cycle 1 - 3	10.98	35.20	5.93	50.27	0.84	1.38
Cycle 4 – 6	20.6	32.15	14.01	80.30	3.8	1.38
Cycle 7 – 9	21.89	29.23	20.84	79.26	5.97	1.37
Cycle 10 - 12	14.72	34.32	7.93	62.50	1.52	1.37
Cycle 13 - 15	12.25	35.47	5.29	54.09	0.84	1.37

The overall average head grade was 8.02% P<sub>2</sub>O<sub>5</sub> with 76.65% SiO<sub>2</sub> and CaO/P<sub>2</sub>O<sub>5</sub> ratio of 1.42. Direct flotation enriched the phosphate grade from 8.02% to an average of 21.78% P<sub>2</sub>O<sub>5</sub> and rejected 60% of the silica content. Reverse flotation tests on the enriched direct flotation concentrate (21.78% P<sub>2</sub>O<sub>5</sub>) produced a final average concentrate grade of 33.27% P<sub>2</sub>O<sub>5</sub> at a reasonable average 65.28% P<sub>2</sub>O<sub>5</sub> recovery.

The CaO/P<sub>2</sub>O<sub>5</sub> ratio of averaged 1.37 was achieved which is reasonably accepted to industry specification of 1.6 for downstream processing. A high CaO in phosphate concentrate may increase the consumption of sulfuric acid during the production of phosphoric acid [5].

The flotation results achieved during this bench-scale tests can be considered satisfactorily given that there was no optimisation carried out. It can be said that with further optimisation of reagents and primary objective of achieving 32%P<sub>2</sub>O<sub>5</sub> grade, P<sub>2</sub>O<sub>5</sub>% recoveries above 85% could be achieved on this specific feed sample.

### 3.2 Simulated Processing Water Analysis

The impact of circulating processing water on total dissolved solids and water hardness was investigated and the results are shown in Fig. 3. Fig. 3 shows that total dissolved solids (TDS) compounded as the flotation cycles increased from 452mg/l to 864mg/l after 15 flotation cycles. A similar observation was evident with recycled water hardness ( $\text{CaCO}_3$ ) increasing from 146mg/l to 304mg/l. The persistent increase of TDS and the total hardness of process water could lead to operational challenges related to ineffectiveness of mineral and particle interactions, surface wettability and other factors that influences flotation response. The reagents effectiveness could be significantly compromised which will lead to poor mineral surface absorption, selectivity and ultimately loss of mineral recovery.

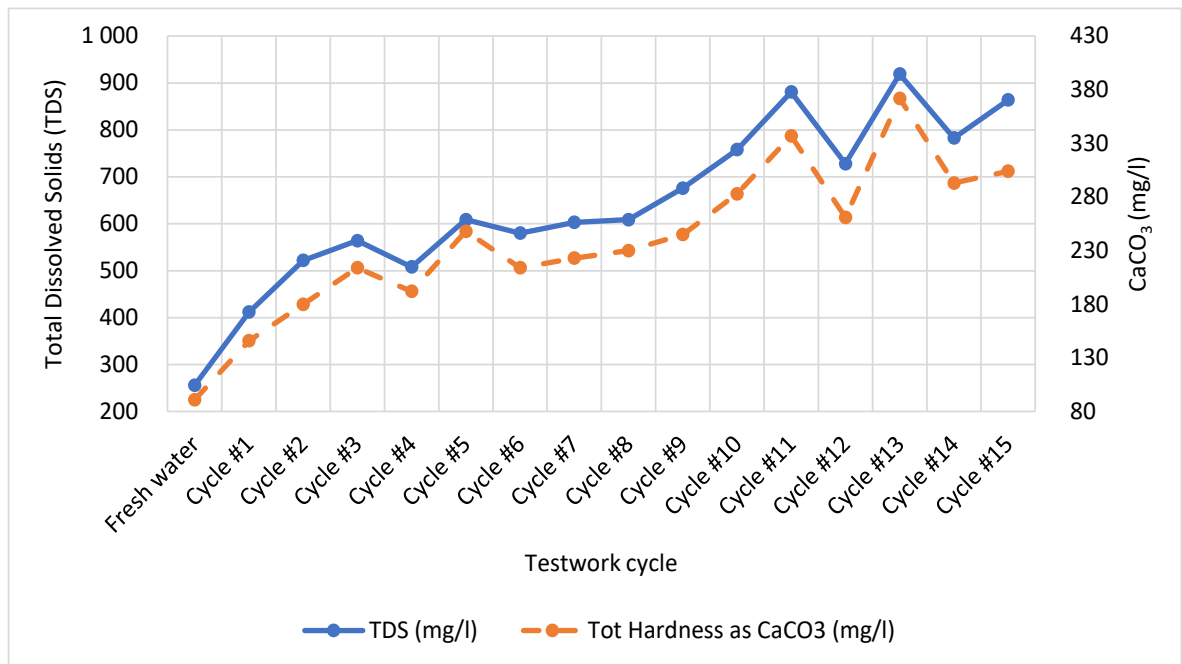


Fig. 3: Total dissolved solids (TDS) and water hardness per cycle.

The effect of circulating processing water on pH and electrical conductivity (EC) was investigated and the results are illustrated in Fig 4. Fig. 4 shows that the pH gradually decreased from 7.45 to 6.37 as the process water was recycled, indicating that the process water shifted from alkaline to acidic medium. [6] found that it should be easy to separate quartz from minerals, using reverse flotation in acidic circuit, or by direct flotation in neutral or alkaline circuits. Electrical conductivity (EC) consistently increased from 662  $\mu\text{m}/\text{cm}$  to 1025  $\mu\text{m}/\text{cm}$  as the process water was continually recycled. A positive linear correlation of EC and TDS can be observed from the data herein analysed.

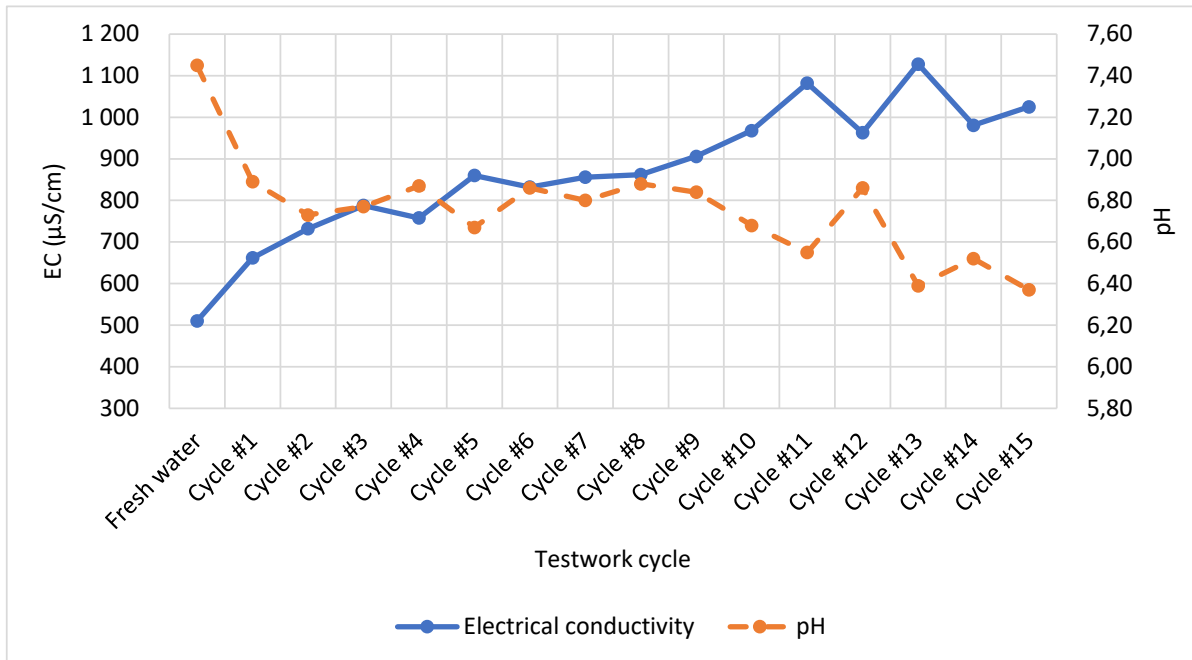


Fig. 4: Electrical conductivity (EC) and pH measurement of simulated process water per cycle.

Process water was analysed for Sodium and Chloride content after circulation and the results are shown in Fig 5. Fig. 5 shows a similar build-up trend of Sodium (57.7 – 76.6 mg/L) and Chloride (100.7 – 109.7mg/L) ions as the process water was recycled from cycle 1 to 15. A positive linear correlation of Sodium and/or Chloride ions with TDS is quite evident from the data. TDS is a significant measure of water quality, the elevated Sodium and Chloride ions in the processing water would be expected to negatively impact the interactions of chemicals and mineral surface properties.

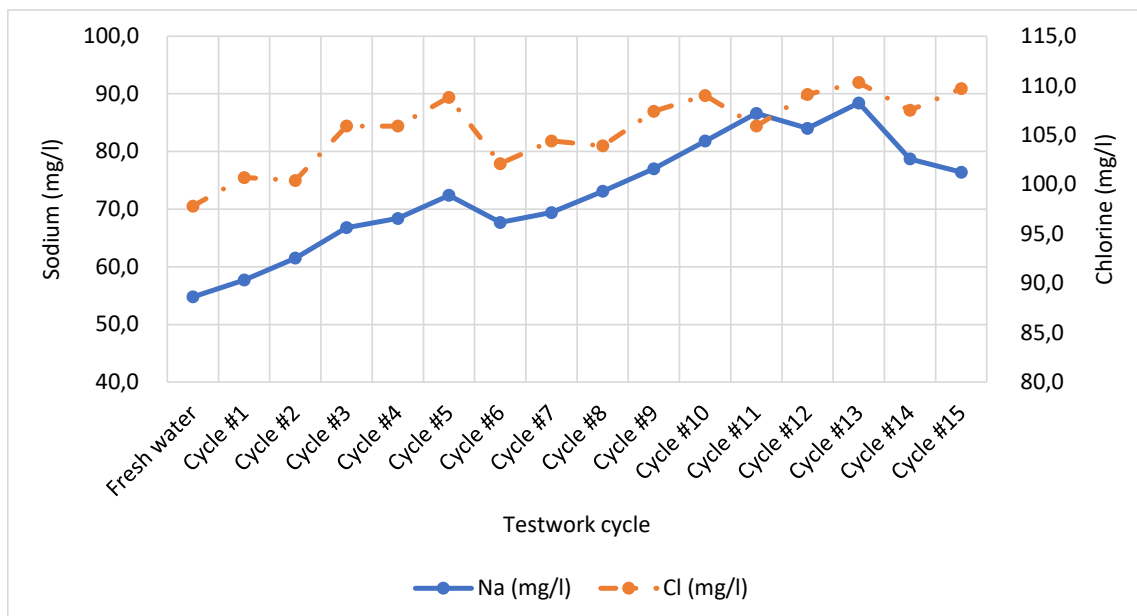


Fig. 5: Sodium and Chlorine content of simulated process water per cycles

Fig.6 shows that Magnesium and Calcium increased from 10.4-12,6mg/L and 40.8- 100.6mg/L respectively.  $\text{Ca}^{2+}$  and  $\text{Mg}^{2+}$  are sometimes troublesome for flotation and they are controlled by addition of soda ash to the pulp prior to anionic flotation [6].

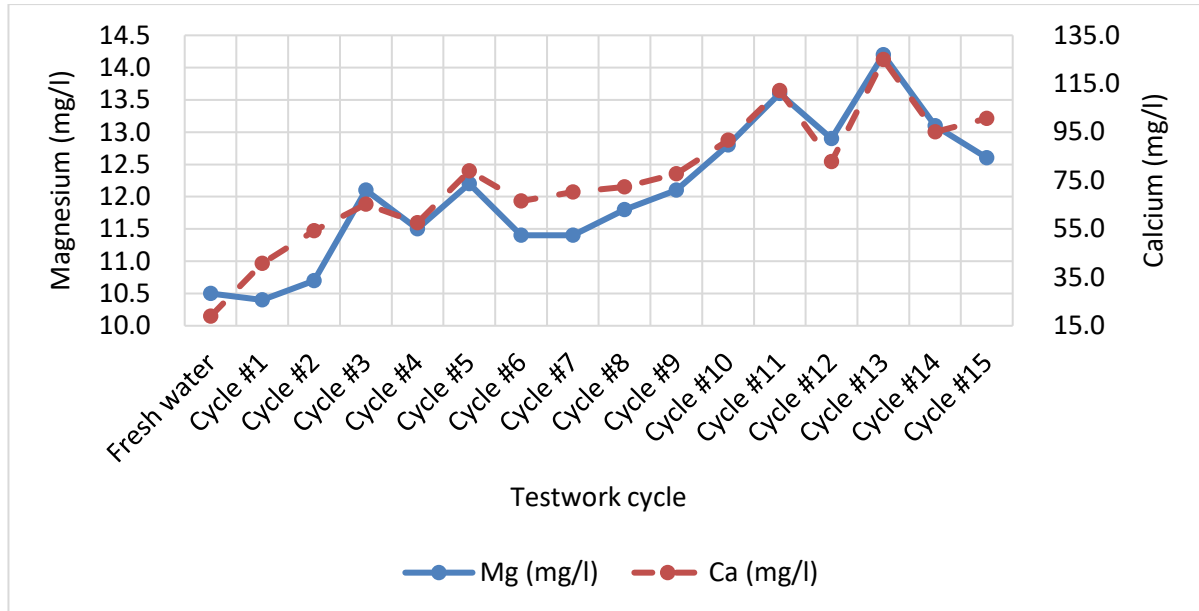


Fig. 6: Magnesium and calcium content of simulated process water per cycles

#### 4. Conclusion

The laboratory flotation tests conducted on the sedimentary phosphate ore showed that recycling process water on a bench-scale could produce satisfactorily results with caution. The flotation results herein achieved should however be considered along with the complexities associated with flotation processes. The accumulation of the inorganic salts when the process water is recirculated cannot be overlooked because they have been proven to be detrimental to flotation performance.

The bench-scale herein conducted showed that circulating the process water compromises the water quality by altering the process water chemistry. The incremental increase of Ca, Mg, Na, and Cl ions together with the intensified build-up of total dissolved solids, unquantified reagent residuals and higher electrical conductivity in the process water were evident. In a pilot scale and operational scale this effect would necessitate an intervention such as water clarifiers to remove and manage process water salinity and inorganics build-up prior to recirculating process water to flotation processes.

The tests were not optimised and it is recommended that further tests be considered for optimisation purposes of critical variables such as amongst others reagents dosages, flotation stages and reagents conditioning.

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