Optimisation of Pulp Chemistry for the Recovery of Base Metals from Mine Tailings using froth flotation

M. D. Fernandez and W. Nheta

Abstract**—** A large deposit of Platinum Group Metals (PGMs) exists in the Bushveld Igneous Complex (BIC), with the majority of the Platinum Group Element (PGE) mineralisation occurring within the Merensky Reef, UG2, Platreef and Middle Group Chromitite reefs, which are also associated with base metals. This investigation focussed on studying and optimising the impact of pulp chemistry on the flotation recovery of base metals from PGM tailings. The parameters investigated included percentage solids, collector dosage, depressant dosage, starch dosage, activator dosage, and pH. The effects of these parameters at different levels on the recovery of Ni and Cu were investigated using the application of the response surface methodology (RSM) through central composite design (CCD).

The PGM plant tailings contained a high chromium content (15.217 %) indicating that it’s a UG2 plant tailings. Major base metal sulphide mineral phases were nickel sulphide, chalcopyrite, pyrrhotite, and pentlandite. The highest Cu and Ni recovery achieved was 74.20% and 27.53% at a grade of 2.75% and 0.51% for Cu and Ni, respectively. The pulp chemistry was affected by collector and activator dosages, solid-liquid ratio and pH.

Keywords**—** Base metals, Flotation, Surface response methodology, Tailings

I. INTRODUCTION

Environmental concerns related to mineral processing plant tailings focus on the potential hazards associated with the pollutants released by the dust, dam seepage, and the possibility of tailings dam failures [1] Also, this problem is intensified by the excavation of lower-grade ores and the weather conditions brought about by climate change [2]. Consequently, strict regulations govern the complete management of tailings facilities. While the conventional mining method had created waste materials that still contained considerable residual value, those waste products are now considered potential secondary resources for extracting valuable minerals and metals [3]. Nevertheless, the specific composition of Platinum Group Metals (PGMs) in the Bushveld Igneous Complex (BIC) is quite complex, requiring suitable beneficiation technologies for enhanced recovery and a reduced impact on the environment [4]. PGMs are found associated with base metal sulphides (BMS) in the BIC.

Flotation is a process that separates valuable minerals from gangue minerals by utilising their differences in surface properties. It is used in concentration of PGMs in the BIC. The process involves the use of chemical reagents at water-solid-gas interfaces to render the mineral surface hydrophobic (water-repellent), leaving the other hydrophilic (water-attracting) [5]. Pulp chemistry is one of the critical aspects that affect flotation. It significantly influences flotation efficiency by influencing mineral interactions with reagents and air bubbles. The composition and physicochemical properties of the flotation pulp are critical determinants of the flotation process.

This project studies the impact of pulp chemistry on the recovery of base metals from PGM tailings. The impact of pH, collector, activator, starch, and depressant dosage on base metal recovery was investigated using Response Surface Methodology (RSM). Furthermore, the study also underscores the adoption of analytical techniques, including XRF, SEM-EDS, and XRD, to study the mineral composition of the tailings. Developing beneficiation strategies as well as practicing environmental stewardship in the mining industry is dependent on this research.

II. METHODOLOGY

A. Materials

The PGM tailings sample used in this project was sourced from a platinum mineral processing plant in Limpopo Province, South Africa. The flotation reagents copper sulphate ($CuSO_4$), Sodium Isobutyl Xanthate (SIBX), and Starch were supplied by BetaChem, South Africa, and Sendep (purity of 98%) and flatanol were sourced from Senmin, South Africa. Sulphuric acid (H_2SO_4) and lime, which were used as pH modifiers, were supplied by Associated Chemical Enterprises, South Africa.

B. Mineralogical characterisation of the PGMs tailings sample

The chemical composition of the PGM tailings was determined using Rigaku ZSX X-ray fluorescence (XRF) analysis equipment. Sample preparation involved 10 grams of the pulverised sample and 5 grams of Sasol wax binder which were thoroughly mixed and then analysed. The mineralogical phases were identified using Rigaku X-ray diffraction (XRD) analysis equipment. The aluminium sample holder contained

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10 grams of the pulverised sample, was scanned at a scanning speed of 0.5°/min with a step of 1A° on the XRD machine with a 2-theta angle ranging from 0° to 162° at a range of 5 to 90°. The surface morphology was analysed using TESCAN scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDS). Sample preparation involved first mounting 10 g of each sample with 15 ml epoxy resin and 2 ml epoxy hardener, then polishing the mounts before analysing.

C. *Design of flotation experiments*

The batch laboratory flotation experiments were designed using Response Surface Methodology (RSM) [6]. The laboratory experiments were optimised using mathematical and statistical methods to ensure they yielded the best base metal recovery and grade. RSM's central composite design (CCD) shows parameters that can be varied to improve the recovery of BMS. Moreover, since it is more accurate, it further assumes biases in range to increase its accuracy. Table 1 indicates the parameters selected for the study and their modalities. A total of 50 runs were conducted to provide the results required to understand the potential effect of the input variables on the responses (grade and recovery of Ni and Cu).

TABLE I: THE EXPERIMENT DESIGN WAS DEVELOPED USING DESIGN EXPERT 13 SOFTWARE FOR FLOTATION.

Factor	Name	Units	Min	Max	Coded Low	Coded High
A	Collector dosage	g/t	-41.63	481.63	-1 ↔ 110.00	+1 ↔ 330.00
B	Activator dosage	g/t	-18.92	218.92	-1 ↔ 50.00	+1 ↔ 150.00
C	Starch dosage	g/t	15.54	134.46	-1 ↔ 50.00	+1 ↔ 100.00
D	SenDep dosage	g/t	-10.6	122.6	-1 ↔ 28.00	+1 ↔ 84.00
E	pH		4.24	13.76	-1 ↔ 7.00	+1 ↔ 11.00

D. *Bench flotation experimental procedure*

All the flotation test work was done using a Denver laboratory machine with a 2.5L flotation cell. A sample of 0.75 kg of PGM tailings was prepared for each flotation run. The milled material was transferred to a flotation cell and followed by distilled water to make up the volume of the cell before the required 1-2% of the reagents. The following reagents were added in different dosages as per DoE created by design expert software: SIBX (collector), Sendep (depressant), copper sulphate (activator), and starch. More water was added, when necessary, to make up the complete volume of the cell. pH was then adjusted to levels 7 - 11 using lime and sulphuric acid. A frother (Flatanol) was added according to the froth stability conditioning with the air valve closed at 1500 rpm agitation. After 2 minutes, the froth was allowed to overflow, and the float was kept at a constant level mark using distilled water from a wash bottle. A sample of the concentrate was collected every 10 seconds for 10 minutes. The collected samples were then oven-dried, weighed, and analysed. The amount of metal Ni and Cu recovered was calculated using equation 1, based on the feed, tailings, and concentrate assays.

$$\% \text{ Metal Recovery} = \left(\frac{C \times c}{F \times f}\right) \times \mathbf{100} \qquad (1)$$

III. RESULTS AND DISCUSSION

A. *Chemical composition of the sample*

The chemical composition of the sample is presented in Table 2. It is observed that the sample contains an appreciable amount of chromium (Cr), which can lead to the conclusion that this is UG2 ore plant tailings. The UG2 chromitite reef in the BIC contains a large amount of chromite that makes up approximately 50–75% of the ore [7]. Goqwane et al. [8] stipulated that chromite is one of the main gangues of UG2 ore. SiO_2 is the main gangue mineral in this sample. The sample shows the presence of BMS (Ni, Fe, and Cu) in low quantities of 0.228, 23.05, and 0.071 % wt., respectively. PGEs are associated with BMS or single grains in the UG2 reef. Therefore, the low quantity of BMS in the tailing sample is due to the recovered BMS in the initial flotation process of the ROM [8].

TABLE II: CHEMICAL COMPOSITION OF THE UG2 PLANT TAILINGS.

Na	**Mg**	**Al**	**Si**	**P**
0.211	9.381	11.015	34.447	0.070
V	**Cr**	**Mn**	**Fe**	**Co**
0.235	15.217	0.282	23.050	0.040
S	**Cl**	**K**	**Ca**	**Ti**
0.067	0.031	0.154	4.895	0.630
Ni	**Cu**	**Zn**	**Sr**	
0.228	0.071	0.051	0.013	

B. *Mineralogical composition of the UG2 plant tailings sample*

The mineralogical composition of the UG2 plant tailings was determined using XRD and the results are shown in Fig. 1.

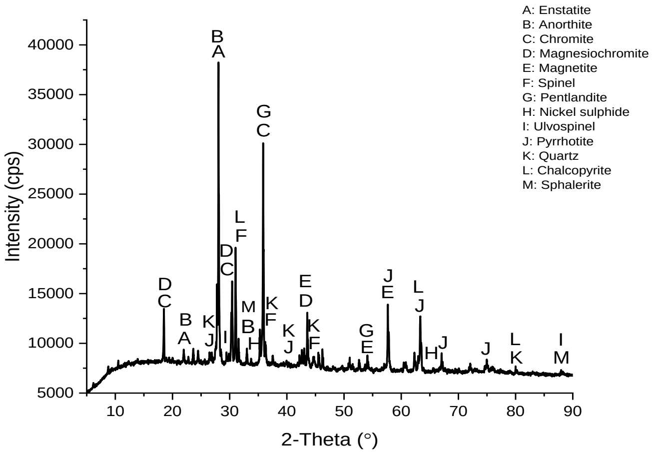

Fig.1. Mineralogical composition of the UG2 plant tailings

The results revealed the most prominent peaks, namely enstatite, anorthite, chromite, magnesiochromite, and magnetite. The main base metal sulphide minerals found were pentlandite, sphalerite and chalcopyrite. The sample was further analysed using SEM-EDS, and the results are shown in Fig. 2. SEM images in Fig. 2 and the mineralogy in Table 3 corroborate with the results observed from XRD, thereby confirming the presence of enstatite as the most abundant phase. Anorthite, chromite, magnesiochromite, and magnetite phases have been observed as relatively abundant phases that comprise the gangue material. XRD and SEM-EDS results show the valuable minerals: nickel sulphide, chalcopyrite, pyrrhotite, and pentlandite.

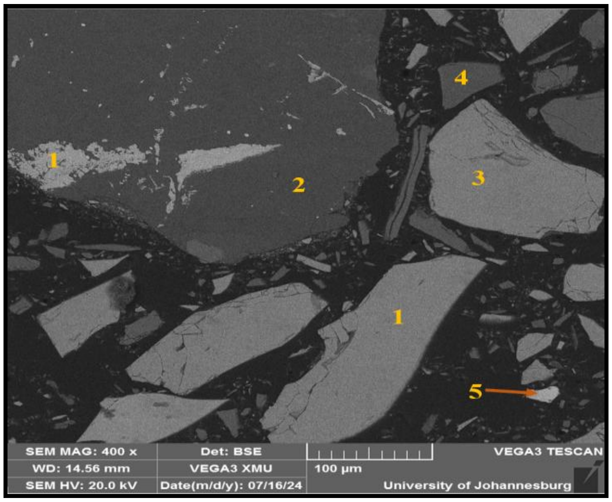

Fig. 2. SEM-EDS micrograph of the as-received sample at 400 Magnifications.

TABLE III: EDS CHARACTERIZATION OF THE AS-RECEIVED SAMPLE.

Spectrum	Mineral Phases
1	Magnetite, Fayalite
2	Quartz, Fayalite
3	Chromite, Magnesiochromite, Magnetite, Ulvospinel, Spinel
4	Anorthite, Enstatite
5	Nikel sulfide, Chalcopyrite, Pyrrhotite, pentlandite

C. *Flotation experimental results.*

Base metals Ni and Cu were monitored for the recovery and grade in the flotation concentrate. The reagent dosages were prepared according to the results of the RSM design of experiments (Table 1). The RSM plots, aiming to envision the relationship between variables and the response (grade and recovery), were plotted, and the results are shown in Fig. 3. This project relied on the RSM-CCD method to optimise the independent variables in the flotation of base metals.

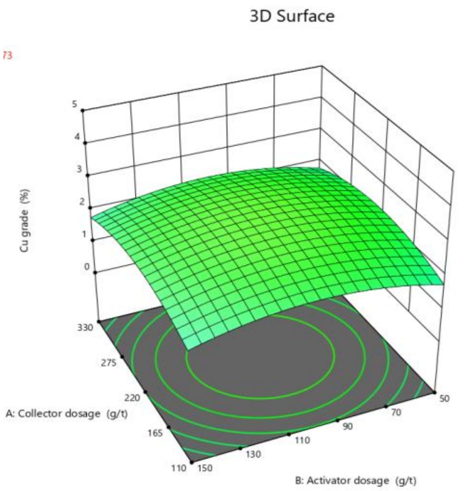

Fig. 3a. Effect of activator and collector dosages on Cu grade

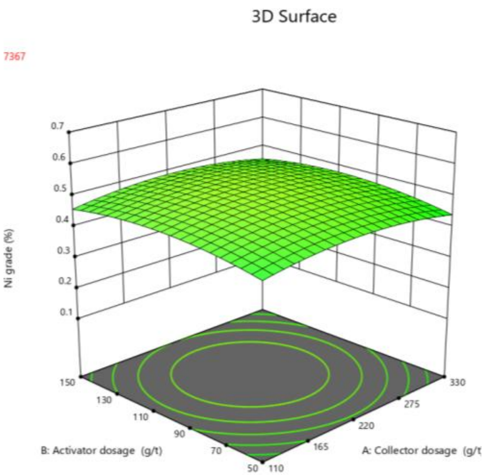

Fig. 3b. Effect of activator and collector dosages on Ni grade

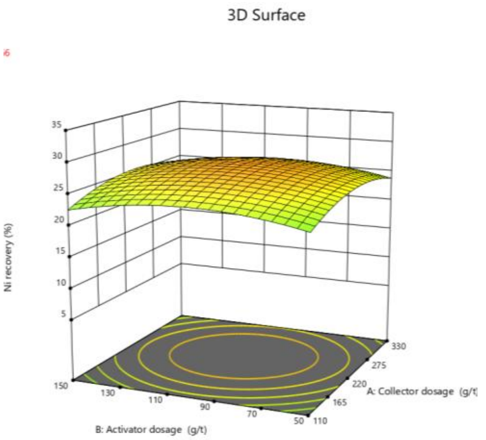

Fig. 3c. Effect of activator and collector dosages on Ni Recovery

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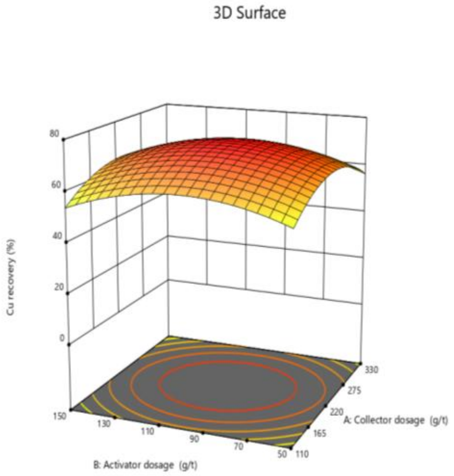

Fig. 3d. Effect of activator and collector dosages on Cu recovery

It is evident from Fig 3a that the maximum Cu grade was obtained at an activator and collector dosages of 50 and 224g/t respectively. Further increase in activator dosages has a negative impact on Cu grade. However, Cu grade increased with an increase in collector dosage. Collectors in flotation processes are crucial in mineral separation and recovery efficiency. The addition of collectors is essential for enabling hydrophobic minerals and recovering them in the froth phase. Therefore, an overdosage of the reagent can activate unwanted material, which reduces the recovery and grade of the valuable metal(s). An insufficient collector negatively impacts the response since valuable metals aren't activated to their best potential, which will result in unwanted minerals' entrainment. Activators play a crucial role in activating mineral surfaces for the uptake of other reagents, enhancing the selectivity and efficiency of the process [9]. It is evident on the surface plots that there's an increased trend within the range of 50–90 g/t, enhancing the recovery and grade of Cu. The highest Cu grade of 2.75% and recovery of 74.20% were obtained.

Similar trends were observed with Ni grade (Fig 3b). The maximum grade of Ni obtained was 0.51% at a recovery of 27.53%. It was obtained at collector/activator dosages of 90 and 220 g/t respectively. It is evident that an overdose or insufficient reagent negatively impacts the responses.

The impact of collector/activator dosages on the recovery of Cu and Ni are shown in Fig. 3c and Fig. 3d respectively.

D. *Effect of pulp density on base metal recovery*

Pulp density is critical in flotation performance, affecting mineral recovery and concentrate grade. The pulp density experiments were conducted with a particle size of 80%, passing 75 µm with the plant baseline flotation dosages collector dosages of 110 g/t, activator dosage of 10g/t, and depressant dosage of 28 g/t. The results are shown in Fig 4. The lowest pulp ratio (10% solids) resulted in moderate recovery, yet it was a lower grade than the other pulp density ratios for Ni and Cu.) Lower pulp density can improve rheological behavior, which improves flotation outcomes, thereby raising nickel and copper recoveries. However, the lowest pulp ratio can result in insufficient particle-bubble interactions, which may result in lowered recoveries and decreased concentrate grade due to a decreased likelihood of collisions between particles and air bubbles. The best pulp densities for Cu and Ni were 30 and 20% solids respectively.

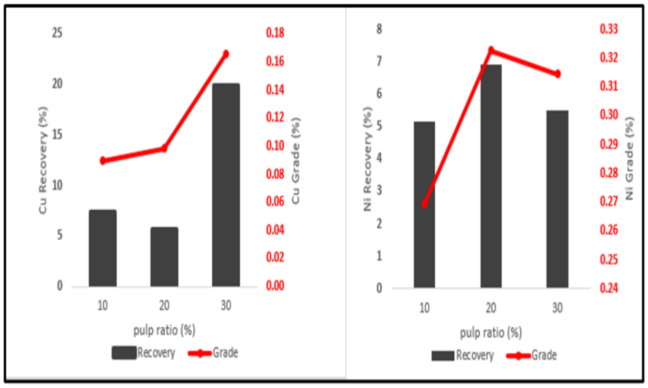

Fig.4. Effect of pulp density on Cu and Ni recovery and grade.

E. *Quadratic models for Ni and Cu recovery and grade*

Based on the RSM-CCD on recovery and grade of Cu and Ni, quadratic models were fitted to the results to predict and optimize the outputs. Four quadratic models are presented by equations.

$$Cu\ grade = 2.75 - 0.4984\ A^2 - 0.4862\ B^2 - 0.4951\ C^2 - 0.4802\ D^2 - 0.4968\ E^2 \qquad (2)$$

$$Cu\ Recovery = 74.0756 - 10.3611\ A^2 - 8.5799\ B^2 - 9.4633\ C^2 - 8.2025\ D^2 - 10.0534\ E^2 \qquad (3)$$

$$Ni\ grade = 0.5133 - 0.0321\ A^2 - 0.0315\ B^2 - 0.0455\ C^2 - 0.0557\ D^2 - 0.0451\ E^2 \qquad (4)$$

$$Ni\ Recovery = 27.5280 - 2.2490\ A^2 - 2.0561\ B^2 - 2.5297\ C^2 - 3.4261\ D^2 - 2.7898\ E^2 \qquad (5)$$

Where A, B, C, D and E are collector, activator, Starch, SenDep dosages, and pH respectively. The grade and recovery of both metals were affected by collector dosages >depressenant dosages > pH.

IV. CONCLUSION

This study aimed to optimize the flotation of base metals using RSM-CCD by improving the pulp chemistry. The PGMs plant tailings contained a high amount of chromium and based on the mineralogy, it is a UG2 ore plant tailings. The pulp chemistry of the tailings was affected by pulp density, pH collector and activator dosages. The RSM models developed were significant and can be used to predict the grade and recovery of Cu and Ni from the UG2 plant tailings. The Ni grade and recovery were very low as compared to Cu. With the further optimization of other flotation parameters, PGM plant tailings can be a secondary source of BMS, and intern PGMs since they are associated in the UG 2 ore.

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