

Optimization of flotation conditions in the beneficiation of PGMs tailings

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Abstract: For several years, mining waste has shown a negative impact on both the environment and human health. The mining industry remains the backbone of the economic growth. Different technologies have been implemented to beneficiate and recover platinum group metals from tailings. The recycling of tailings has been a point of research interest due to their extensive applications. Flotation has been the primary process of upgrading and recovering PGMs. The focus of this study was to optimize flotation conditions in the beneficiation of PGMs for particular small-scale mine tailings. This was done to obtain the most favourable conditions for the small-scale mine tailings to improve operating conditions of specific particle sizes. PGMs tailings obtained from a small-scale mine were characterized using XRD, XRF, SEM/EDS, and ICP – OES to understand the properties of the tailings prior to mineral processing. Flotation batch tests were conducted. The results showed that the chosen particle size was 75 μm , and the favorable reagent dosages were 150 g/Mg and 100 g/Mg for SIBX (collector) and Starch(depressant), respectively. At favourable conditions, the recovery was 65.75% (Pt = 70.38%, Pd = 59.33%, Ru = 34.56%), and the grade was 31.46 g/Mg (Pt = 21.43 g/Mg, Pd = 9.62 g/Mg, Ru = 0.41 g/Mg). It can be concluded that all the flotation parameters are related; lower particle sizes yield high recoveries and better grades due to the exposure of the particle surface to the reagents responsible for the flotation of the PGMs. It was observed that the high collector dosages produce high recoveries with low grades of PGMs. High depressant dosages produce low recoveries with high grades of PGMs. The relationship between the collector and the depressant is of essential importance in the flotation process.

Keywords: PGMs tailings, beneficiation, flotation, particle size, reagent dosages

1. Introduction

The societal demand for minerals is said to be growing, parallel with the growing demand in the mining sector. The mining industry has been working to increase economic growth, whereby the industrial tailings aid immensely in the minerals recovery and its necessity for economic growth. Industrial tailings contribute towards social services and jobs, public services wealth, and the substantial collection of goods that are part of our daily modern life (Ramirez-Llodra et al., 2015; Hudson-Edwards, 2016).

In recent years, it has been shown that the recycling of PGMs has become an important topic, in so doing, sustaining the supply of these metals and transforming waste into valuable renewable resources. Intensified research has been influenced by the increasing demand for potential secondary sources of PGMs, which aims to recover PGMs from waste material. The secondary sources include metal-containing deposits, tailings from ore plants, military equipment, and many more (Nikoloski et al., 2015; Wang et al., 2017).

The platinum group metals consist of six (Ir, Os, Ru, Pt, Pd, and Rh) metals with comparable physical and chemical properties. These metals are rare on the earth's surface and have non – corrosive qualities with more extended uses and regular exposure to the ambience and environment. The PGMs tend to be concentrated in nature and have similar geochemical behaviours. These metals are used in different industries for different applications, such as the petrochemical and chemical industries, electronics, glass, jewellery, automobiles, and medical sciences (Jena et al., 2016).

Technological processes all over the world improve functionality and developments on Earth. Due to the various applications of PGMs, they are found in many secondary sources. The recovery of PGMs from spent materials and tailings is made feasible by their high level of concentration and their high economic value in their secondary sources, in comparison to their primary sources (Matthey, 2013; Suoranta et al., 2015).

Many studies have observed many ways to characterize numerous PGMs in mines and the benefits and limitations of many technologies for recovering PGMs from mines. These technologies include conventional technologies (pyrometallurgical, hydrometallurgical, and bio – metallurgical processes) and developing technologies (electrochemical, supercritical fluid, mechanochemical, and ionic liquids processing) (Wang et al., 2017).

South Africa is said to be the world's leading source of PGMs and contains the world's known resources. Hence the study of the flotation process is of critical importance in the concentration of PGMs. The PGMs are concentrated by froth flotation using xanthate – type collectors and depressants of the polysaccharides family. Reagent dosages play the main role in the maximum recovery of PGMs and the rejection of the maximum volume of gangue (Alvarez-Silva et al., 2014).

Froth flotation is the most used beneficiation method in mineral processing. This is due to its versatility in design parameters that allow for selectivity. The separation of hydrophobic materials from hydrophilic materials is the basic flotation principle. Collectors are added to separate the mineral of interest, which is made hydrophobic. The chemicals used are thermodynamically selective to adsorb to the surface of the mineral particles, which assists the minerals in binding to the air bubbles and floating to the surface of the slurry to be collected. Selective collectors and frothing agents are used to promote the formation of bubbles (Sefako, 2018; Vaccarezza, 2018).

The hydrophobic particles are carried to the froth phase at the top of the flotation cell, where the hydrophilic gangue remains in the pulp and results as tailings. A successful flotation process lies in the thermodynamics of the mineral surfaces, adsorption, and wetting. The main advantage of flotation is its ability to change design parameters for selectively, and the choice of surfactants allows for such selectivity (Sefako, 2018; Vaccarezza, 2018).

The operation parameters can affect the flotation performance since they are non – chemical variables. The temperature depends on the weather, and the mineralogy depends on the mined ore. Feed rate, pulp density, and particle size may be adjusted to meet the operational requirements (Ucurum & Bayat, 2007).

Recent studies show that the re-treatment of low-grade tailings is seen as a way of conserving minerals with the global emergence of the thought of a circular economy, and it can be harnessed with economically efficient techniques (Gibson, et al., 2023).

This paper focuses on the optimisation of particle size and reagent dosages on the grade and recovery from tailings in the flotation process. For the reagent dosage optimization, the collector and depressant were varied, whereas, the frother and activator were kept constant.

2. Materials and methods

2.1. Materials

PGM-containing tailings were collected from a ferrochrome mine for experimental purposes. Reagents such as copper sulphate (CuSO_4), soluble starch, sodium isobutyl xanthate (SIBX), and SOMFROTH were purchased from Sigma Aldrich, Rochelle, and United Scientific. The 2800 μm to 75 μm screening sieves and shaker, rotary ball mill, rotary sample splitter, analytical balance, flotation bay with cell, beakers, sampling pipettes, sample bags, volumetric flasks were utilised from the chemical and metallurgical laboratory at Tshwane University of Technology, Pretoria, South Africa.

2.2. Methods

2.2.1. Research equipment

The collected PGMs tailings were crushed using a rotary ball mill using stainless steel balls of various sizes as grinding media, with an abrasion-resistant manganese steel lining. Crushing/milling was done to obtain 80% screening of the size of a 75 μm sieve as the chosen size exposes the surface area of particles. Milling was done for 2 hours to obtain the required amount of particle size. Screening was

done to determine the particle sizes of the tailings sample, sieves ranging from 2800 μm to 75 μm were used on a sieve shaker to determine the percentage passing of the tailing particles. The milled tailings sample was evenly blended and split into equal representative mass samples using the rotary sample splitter. The samples were divided into equally representative 500 g samples. The obtained split samples were then used in the flotation process. The flotation cell used in the beneficiation of the PGMs is shown in Fig. 1. The required particle size samples were used in the flotation process to evaluate the different parameters that influence the beneficiation of flotation.

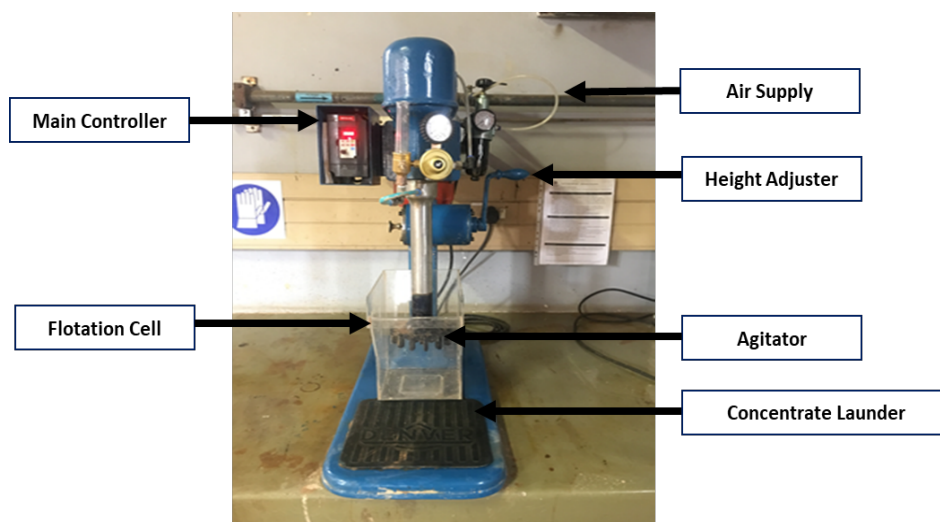


Fig. 1. Experimental flotation bay set – up for the flotation process (Tshwane University of Technology)

2.2.2. Preparation of flotation reagents

The reagents were prepared in bulk a day before usage, and the dosages were varied to obtain the optimum reagent dosages to be used in achieving the high yield of PGMs, the reagents concentrations ranged between 2% and 10%. To prepare the CuSO_4 solution; 15.9 g of solid CuSO_4 crystals were mixed with 1 dm^3 of deionised (DI) water and the solution was allowed to mix before use. For the preparation of the soluble starch solution; 58.06 g of soluble starch powder was added to 200 cm^3 of deionised water to allow proper mixing, then add the solution to 800 cm^3 of hot water to all complete dissolution. For the preparation of the SIBX solution; 17.22 g of SIBX powder was mixed with 1 dm^3 of DI water and allowed to mix before use. The schematic experimental procedures for the PGM tailings are shown in Fig. 2.

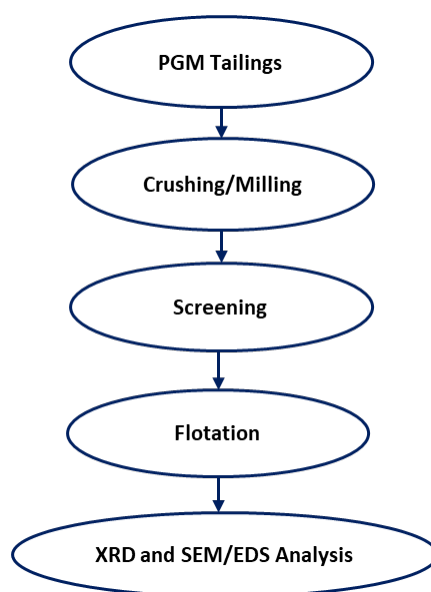


Fig. 2. Schematic experimental procedures for the PGM tailings

2.2.3. Experimental procedure

PGMs tailings were collected from a ferrochrome small-scale mine for the conducted research; the tailings were split and blended to ensure that any sampling done represents the bulk sample. Particle size distribution was done to evaluate the sample particle size. Screening was done to obtain particle sizes of 75 μm , 106 μm , and 212 μm to determine the optimum particle size. Norori-McCormac et al., 2017 reported that the favourable particle size for flotation was between 20 – 150 μm , these findings influenced the selection of the studied particle sizes. Flotation tests of three repetitions were conducted on the three different particle sizes floated separately.

For the flotation process, test runs were conducted with 30% solid with deionised water, the samples were mixed for 5 min. The solid-liquid ratio of 30% was adopted from Wiese 2005 & 2006 and Sekgarametso 2018. 50 g/Mg of CuSO_4 (activator) was added and mixed for 5 min. After 5 min, 50 g/Mg of SIBX (collector) was added and allowed to mix. After 5 min of mixing 50 g/Mg of soluble starch (depressant) was added, and mixing occurred. After 5 min, an addition of a SOM frother (frother) and mixing was allowed for 5 min. After 5 min, the air was introduced until bubble formation; once bubbling started, scrapping was done every 2 min. Air speed was kept at a constant of 4 dm^3/min and the rotational speed of 45 rpm in a 3 dm^3 flotation cell.

Once the optimum particle size was obtained, the remaining tailing sample was milled for to 2 hours to achieve 80% of the accepted size. The screening process determined the milling time, which aimed to achieve 80% passing. The sample was screened after milling to confirm the 80% passing. The tailings sample of the required size was further used to determine the effect of solid-water ratio and the reagent dosage. The reagent dosages varied at different SIBX (collector) and starch (depressant) dosages are presented in Table 1. The variation of reagent dosage was influenced by the method carried out by Sekgametso 2018 and Sefako 2018 (Sekgarametso, 2018; Sefako, 2018)

Table 1. Experimental scheme for optimising reagent dosages for flotation

Experiments	1	2	3	4	5
SIBX (collector) (g/Mt)	50	100	150	200	250
Starch (depressant) (g/Mg)	30	50	100	150	200
CuSO_4 (activator) (g/Mg)	50	50	50	50	50
SOM (frother) (g/Mg)	50	50	50	50	50

XRD (Bruker, D8 Discover, X-ray source Cu, 3 kW (Delhi)) examinations were done to determine the bulk mineralogical composition of the tailings. Due to the amorphous minerals, the obtained XRD results required justification by element analysis. The XRD analysis can only identify crystalline elements. Therefore, additional analysis using the SEM/EDS were conducted to detect the elements present in the ore.

3. Results and discussion

3.1. PGMs tailings mineralogy

The sample was taken for analysis to identify its mineral composition. The XRD was used to determine the mineral composition, and SEM/EDS (Hitachi SU – 70 Schottky Field Emission) was used to observe the sample's microscopic image and elemental composition. Fig. 3 shows the mineral composition of the sample.

The obtained XRD results showed that the feed PGM tailings had a high content of chromite, making it a dominant mineral. Gangue minerals and small traces of 2 – nitrophenyl phenoxyformate and pyroxene were also present. This characterisation test also indicated that the tailings were from chromite-containing ore. The tailings showed characteristics similar to those studied by Dawson (2010) and Sekgarametso (2018), with traces of orthopyroxene and silicate (Dawson, 2010; Sekgarametso, 2018). Due to the phases of the minerals, some minerals could not be identified using the XRD. Therefore, more tests were done using SEM/EDS to identify low-content minerals. The obtained elementary analysis are tabulated in Table 2 and demonstrated in Figs. 4 and 5.

The EDS elemental results were in support of the XRD results, showing that the dominant minerals are chromite and gangue minerals. EDS results showed that the sample had high concentrations of silicon and iron. The feed sample analysis showed similar characteristics as the ore treated by Sefako and Sekgarametso, which showed high concentrations of Silicate (Sefako, 2018; Sekgarametso, 2018). ICP-OES was used to analyse PGMs (Table 2). The identification of PGMs in the feed sample was undetectable using EDS and XRF due to their moderately low content. In most ore and tailings, PGMs range in ppm and usually have lower content than other elements (Sefako, 2018).

3.2. Optimization of flotation parameters

Four parameters were studied to optimise flotation parameters to obtain the favorable conditions for the flotation process in the beneficiation of PGMs from tailings. The investigated parameters were; particle size, reagent dosage, solid-liquid ratio, and flotation retention time.

3.2.1. Effect of particle size on the flotation process

The particle size of the sample plays an important role in flotation because of the ability of the particles to float or settle. Three different particle sizes, 75 μm , 106 μm , and 212 μm , respectively, were investigated to establish the optimum particle size for the flotation process. The conditions were investigated both under observations and analysis. Fig. 6 shows the effect of particle size on mass recoveries.

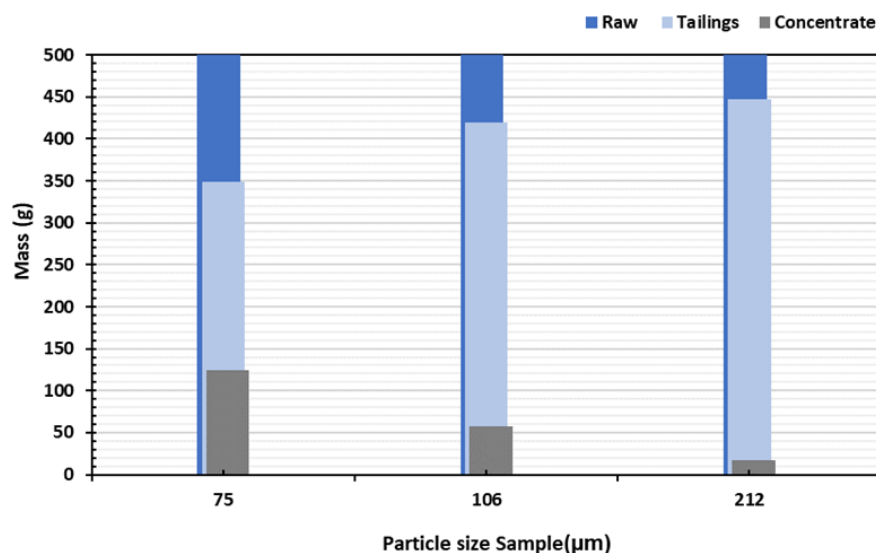


Fig. 6. Mass recovery of the sample at different particle sizes

Fig. 6 shows that at smaller particle sizes, more froth is collected at high masses compared to larger particle sizes. From the obtained results, it is seen that 75 μm is the favourable particle size for the flotation process. This is due to the differences between wanted particles and undesirable particles' surface properties (Norori-McCormac et al., 2017). Studies have shown that the optimum particle size range for froth flotation is comparatively fine, being approximately 20–150 μm (Gaudin et al., 1942; Jameson et al., 2007).

The morphology of the floated samples with different particle sizes of 75 μm , 106 μm , and 212 μm is shown in Fig. 7. The morphology demonstrates the physical relationship of the particles' sizes and crystallinity. The chemistry of the floated concentrates is of great importance. This determines the particle surface properties, which are very important in separating valuable minerals from gangue minerals (Moimane et al., 2016).

3.2.2. Effect of reactant dosage on flotation

The experiments and tests conducted on particle size showed that 75 μm was the optimum size. The tailings sample was milled to 80% passing of 75 μm to investigate the effect of reactant dosage on the flotation of PGM tailings. The reactant dosages were studied at 5 different experimental schemes, where

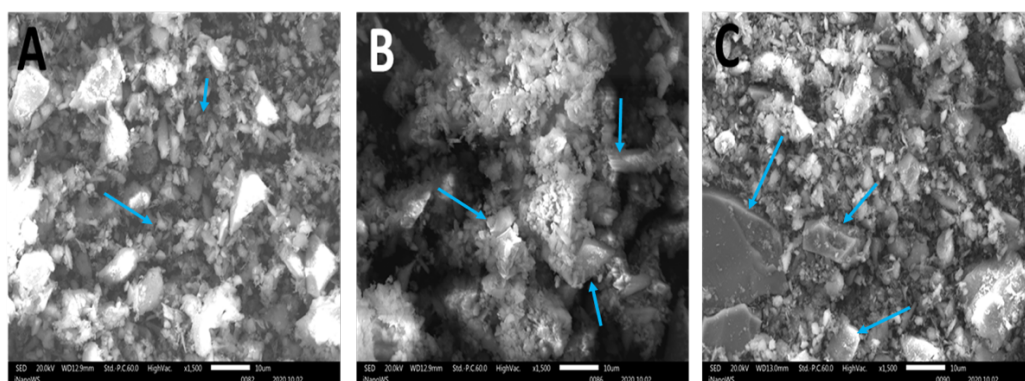


Fig. 7. SEM morphology of floated samples with particle sizes (A) 75 μm , (B) 106 μm , and (C) 212 μm

the dosages of the collector (SIBX) and the depressant (Starch) were varied, and the activator (CuSO_4) a frother (SOM froth) were kept constant. Table 3 shows the investigated dosages. Due to the similarity in the feed samples, the variation of reagent dosages was adopted from Sekgarametso 2018. The combinations of the reagents were selected based on the relationship between the collector and the depressant and to investigate their behaviour at different dosages (medium – medium and high-high).

The collector plays a vital role in the collection of the desired minerals, whereas the depressant aids in suppressing the gangue materials. Figs. 8 – 10 show the effect of reactant dosage on the grade and recovery of PGMs from ferrochrome tailings. The results show the relationship between the collector and the depressant and their effect on the flotation process (Kawatra, 2016).

Table 3. Experimental scheme for optimising reactant dosages for flotation

Experiments	1	2	3	4	5
SIBX (collector) (g/Mg)	50	100	150	200	250
Starch (depressant) (g/Mg)	30	50	100	150	200

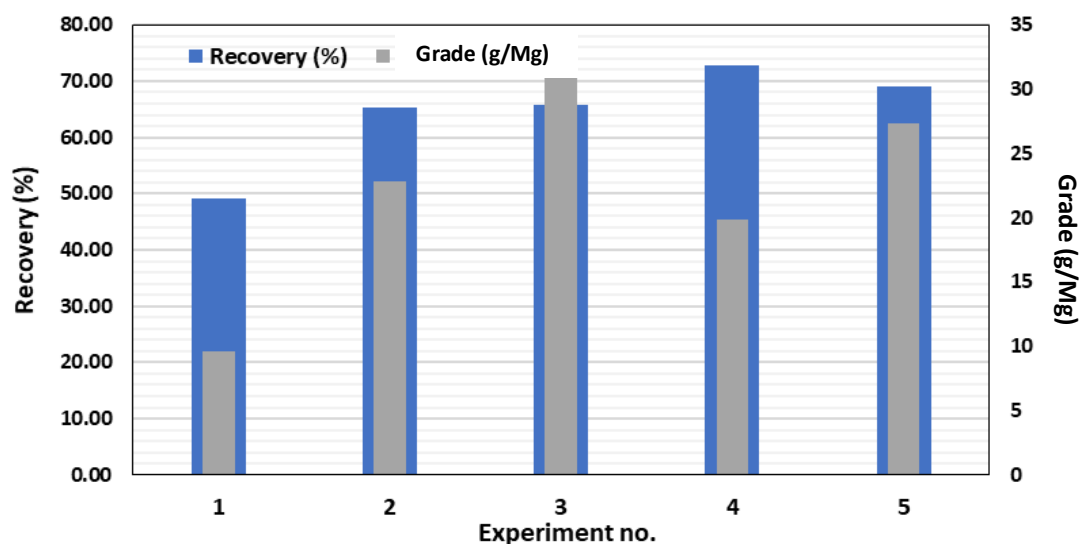


Fig. 8. Effect of collector and depressant dosages on the 3E PGM grade (g/Mg) and recovery(%)

The results obtained, illustrated in Fig. 8 show the effect of reactant dosages on the grade and recovery of PGMs. Following the experimental scheme utilised for the research, Experiment 1, consisting of SIBX (50 g/Mg) and Starch (30 g/Mg), had a grade of 9.64 g/Mg and a recovery of 49.10%. Experiment 2, consisting of SIBX (100 g/Mg) and starch (50 g/Mg), had a grade of 22.87 g/Mg and a recovery of 65.34%. Experiment 3, consisting of SIBX (150 g/Mg) and starch (100 g/Mg), had a grade of 31.46 g/Mg and a recovery of 65.75%. Experiment 4, consisting of SIBX (200 g/Mg) and starch (150

g/Mg), had a grade of 19.86 g/Mg and a recovery of 65.34%. Experiment 5, consisting of SIBX (250 g/Mg) and starch (200 g/Mg), had a grade of 27.3 g/Mg and a recovery of 69.16%.

The grade and recovery of the three PGM are also affected by the gangue minerals present in the tailings. The initial characterisation of the test samples showed high oxides concentrations, usually treated with co – co-collectors in the flotation process to beneficiate the grade of PGMs (Sefako, 2018; Sekgarametso, 2018).

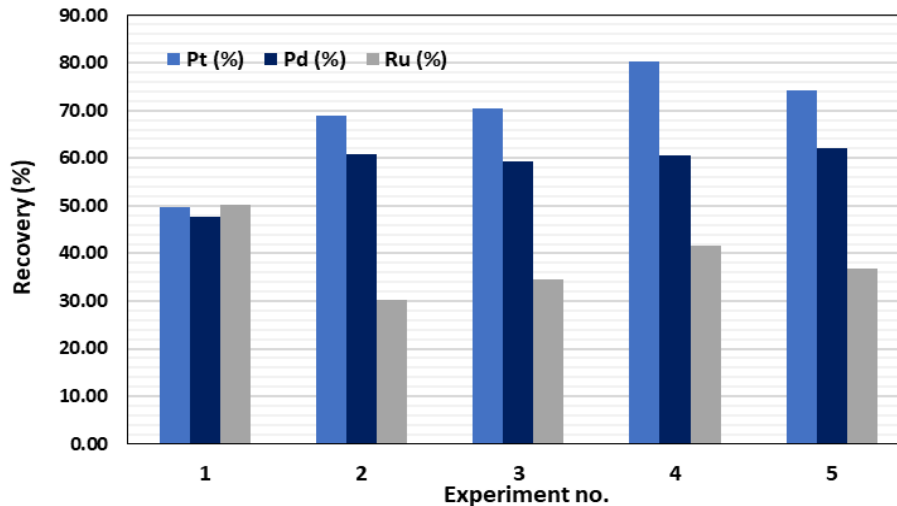


Fig. 9. Effect of collector and depressant dosages on 3E recovery (%)

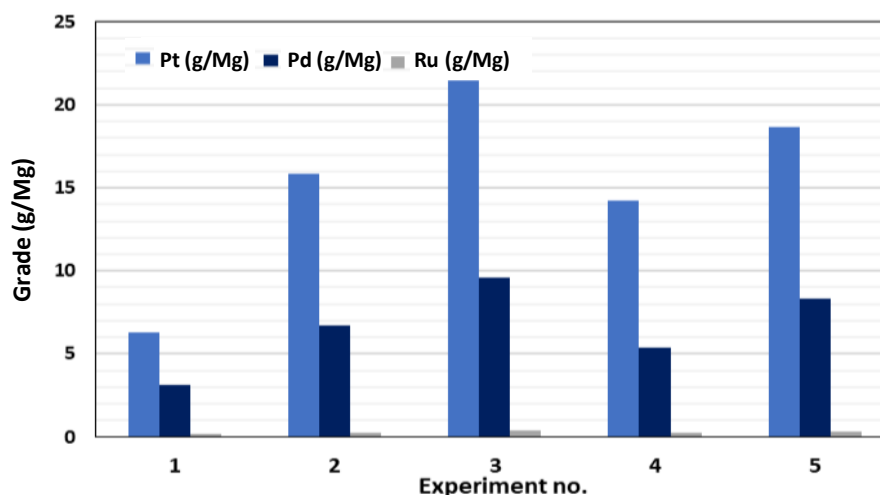


Fig. 10. Effect of collector and depressant dosages on 3E grade (g/Mg)

Figs. 9 and 10 show the grade and recovery of 3E at varied SIBX and starch dosages. Experiment 3, consisting of 150 g/Mg SIBX and 100 g/Mg starch, showed a higher grade of Pt and the three PGM present in the tailings sample. Experiment 3 showed a grade of 21.43 g/Mg with a recovery of 70.38% for Pt. A higher recovery was observed under Experiment 4 for Pt, which consisted of 200 g/Mg SIBX and 150 g/Mg starch, having a recovery of 80.27% with a grade of 14.21 g/Mg. The results support the increased recoveries affecting the grade of the minerals recovered. The high grade recovered is attained by the collector's ability to be adsorbed onto the desired mineral, they essentially form a layer of non – polar hydrophobic hydrocarbons which are bubbled out during flotation (Mpongo & Siame, 2006; Langa et al., 2014; Kawatra, 2011). This means that the bubbles in the froth are too large and sweep the particles past without coming into contact.

The highest grade of 9.62 g/Mg with a recovery of 59.33% was obtained in Experiment 2 for Pd, with reactant dosages of 150 g/Mg SIBX and 100 g/Mg starch. The highest recovery of 62.20% with a grade of 8.32 g/Mg was observed in Experiment 5 for Pd, consisting of reactant dosages of 250 g/Mg SIBX and 200 g/Mg starch. Experiment 1, 2, and 4 for Pd shows how the high recoveries affect the grade of

the Pd present. According to observations of the presented results, the relationship between the collector and depressant is very important. The high flotation recoveries affect the desired minerals' grade (Langa et al., 2014; Mpongo & Siame, 2006).

The amount of Ru present in the tailings was of very low-grade concentration. However, a higher grade of 0.41 g/Mg with a recovery of 34.56% was obtained at 150 g/Mg SIBX and 100 g/Mg starch dosages. The highest recovery of 50.27% with a grade of 0.21 g/Mg was observed at 50 g/Mg SIBX and 30 g/Mg starch for Ru. The effect of recoveries on grade was also observed, as noted by Lang et al. 2014. High recoveries caused by collectors result in low grades (Wiese et al., 2005; Mpongo & Siame, 2006; Langa et al., 2014).

3.2.3. Effect of solid-liquid ratio on the flotation process

The solid-liquid ratio experiments were conducted with a particle size of 75 μm , collector dosages of 150 g/Mg, and depressant dosage of 100 g/Mg. Table 4 and Fig. 11 show the effect of the solid-liquid ratio fed into the flotation cell. The results showed that the lower the solid ratio to water, the lower the amount of solid recovered. Whereas, high solids are recovered at a high solid ratio to water. The reactant dosages also assist the recovery of the solids.

Table 4. Effect of solid-liquid ratio of the flotation process

Solid - liquid Ratio	20:80	30:70	40:60
Feed (g)	500	500	500
Conc. (g)	43.56	101.24	234.62
Tailings (g)	456.44	398.76	265.38

All experiments were conducted at 500 g of sample feed and optimum reactant dosages. The feed ratio of 20:80 resulted in low solid recoveries of 43.56 g due to the large amount of water in the slurry. The highest solid recoveries were at the feed ratio of 40:60 due to the high solid feed and low water content in the slurry. However, high recovery doesn't necessarily present good recovery as it affects the grade of the desired mineral (Jameson et al., 2007). At high solid recoveries, there are high possibilities of recovery of gangue minerals as well, mainly because the amount of water in the slurry is not enough to assist the depressant in suppressing gangue minerals and allowing hydrophobic particles to float (Gupta & Yan, 2016).

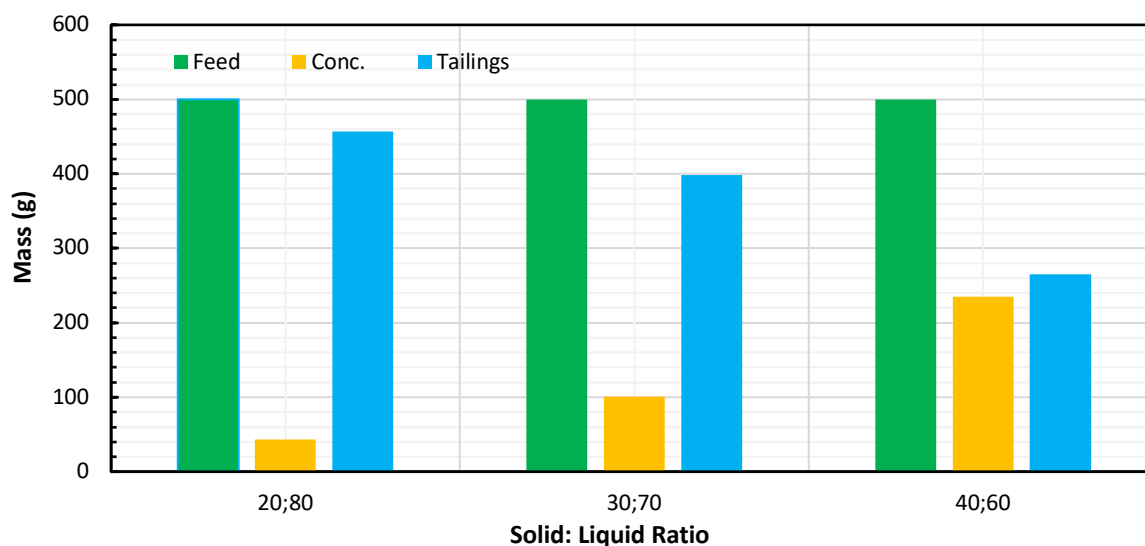


Fig. 11. Effect of solid-liquid ratio on flotation

Fig. 11 shows the mass balance around the flotation process and how the solid - liquid ratio affects the solid recovery and the grade of the desired minerals. The solid-liquid ratio of 30:70 results in a recovered solid concentration of 101. 24 g and 398,76 g tailings retained. The 30% w/w of solid has

shown the optimum flotation results and agrees with previous studies (Wiese et al., 2006; Sekgarametso, 2018).

Table 5 presents Experiments 1 to 3 relating to solid – liquid ratios of 20:80, 30:70, and 40:60, respectively. These experiments were conducted at a fixed particle size, depressant, and collector dosages to observe the effect of solid-liquid ratio on the grade and recovery of the investigated PGMs.

Table 5. Conducted experiment for the solid-liquid ratios

Solid – liquid ratio	20:80	30:70	40:60
Experiments	1	2	3

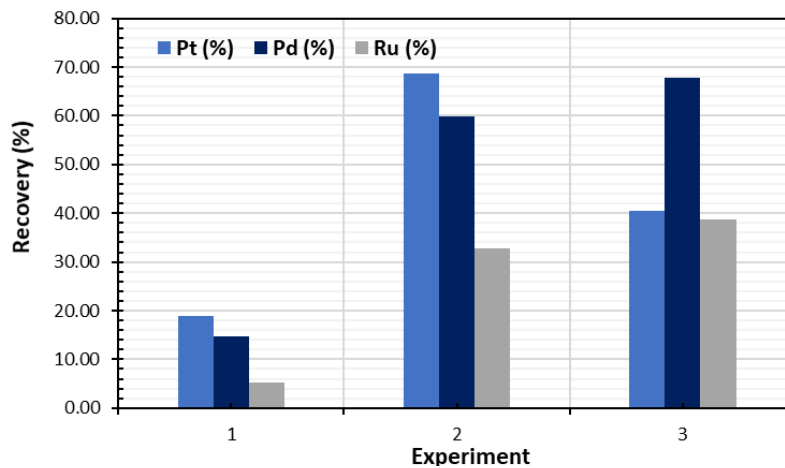


Fig. 12. Effect of solid-liquid ratio on the 3E recovery (%)

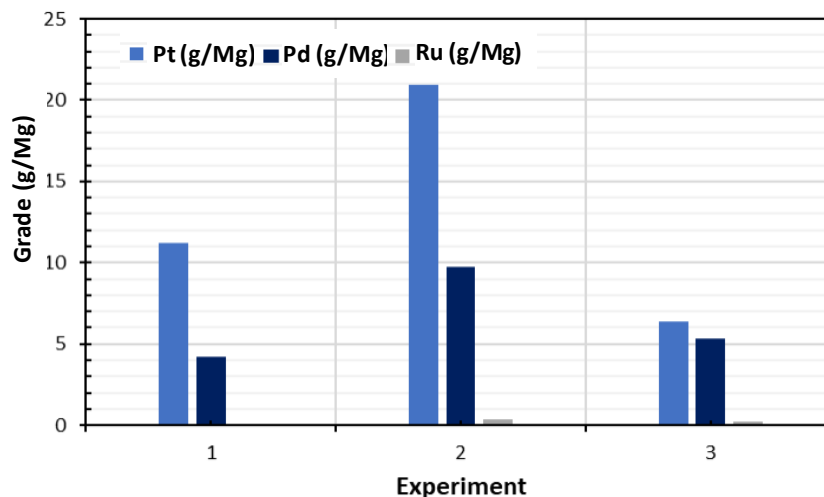


Fig. 13. Effect of solid – liquid ratio on the 3E grade (g/Mg)

Figs. 12 and 13 show the recoveries and grades of the 3E in all three experiments at different ratios (20:80, 30:70 and 40:60). The highest grade for Pt was observed at Experiment 2 (30:70), with a grade of 20.92 g/Mg and a recovery of 19.22 %. The highest recovery for Pt was observed in Experiment 3 with a poor grade, the recovery was 23.83% and a grade of 6.35 g/Mg. This shows that the high solid recovery affects the grade as the gangue mineral manages to be floated due to the low amount of water in the process and the depressant cannot sustain the suppression of these minerals (Gupta & Yan, 2016).

The highest grade for Pd was observed in Experiment 2 (30:70), with a grade of 9.72 g/Mg and a recovery of 86.73%. Both Experiments 2 and 3 for Pd had high recoveries. However, Experiment 3 (40:60) had a high recovery with a low grade. High recoveries do not essentially yield good grades, as seen in Experiment 3, which consisted of a high amount of solid fed (Jameson et al., 2007).

The grade obtained for Ru was 0.39 g/Mg and 47.65% recovery. The presented results show that Experiment 3 for Ru had high recoveries due to the high amount of solid fed and low water content in the slurry. The desired minerals are often affected at high recoveries; hence, high recoveries do not essentially present good grade recovery (Jameson et al., 2007). High recoveries of solids also yield high recoveries of gangue minerals because the low amount of water in the process cannot suppress gangue minerals (Gupta & Yan, 2016).

Table 5 presents the overall grade and recovery of minerals at the different solid-liquid ratios. The highest grade was obtained at the ratio of 30:70, where the grade was 31.03 g/Mg and the recovery 64.86 %. The lowest grade was observed at the ratio of 11.91 g/Mg and the recovery of 49.26%.

Table 6. Overall grade (g/Mg) and recovery (%) of the varied solid-liquid ratios

Experiments		Grade (g/Mg)	Recovery (%)
1	20:80	15.5	17.19
2	30:70	31.03	64.85
3	40:60	11.91	49.26

4. Conclusions

All flotation parameters are related; therefore, they all play a significant role in increasing the grade and recovery of PGMs. The addition of reagents in the flotation process is to separate the gangue minerals from the desired minerals. A balance between operating parameters should be taken into account. This should be done to produce minerals of high-grade concentrations without compromising the recovery (Mberi et al., 2018). Based on the results obtained, the following conclusions can be drawn:

- The optimum particle size for the beneficiation of PGMs from the specific ferrochrome tailings is 75 μm . The smaller particle sizes assist in stabilising the froth formed. The exposed particle surface area makes it easier for the collector to be attached to the desired minerals and forms a hydrophobic layer that can be floated and collected with high recovery.
- The favourable reagent dosage was obtained at the dosages of 150 g/Mg SIBX (collector) and 100 g/Mg starch (depressant), with a grade of 31.46 g/Mg (Pt = 21.43 g/Mg, Pd = 9.62 g/Mg, Ru = 0.41 g/Mg) and a recovery of 65.75% (Pt = 70.38%, Pd = 59.33%, 34.56%). The collector dosage is an essential factor in the flotation process as it determines the efficiency of the process. However, high collector dosages yield high recoveries with low grade. The depressant assists in the separation of minerals by suppressing the gangue minerals and allowing the desired minerals to float.
- The ratio between the solids and liquid fed into the flotation system was evaluated by comparing solid recoveries of 3 different solid-liquid ratios. The favorable solid-liquid ratio was obtained at 30:70, as supported by other authors. Several studies have indicated and recommended the observed optimum solid-liquid ratio. It was also observed that low solid ratios produce fewer solid recoveries, whereas the higher ratios produce more solid. The obtained grade and recovery of the solid – liquid ratio also showed that 30:70 is the best performing for the process.

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