

# Recovery of PGMs from Chromite Tailings and Selective Adsorption

Happy Mphahlele and Elvis Fosso-Kankeu

**Abstract**— The recovery of Platinum Group Metals (PGMs) from chromite tailings presents a promising future of increasing valuable PGMs in South Africa, which holds over 80% of the PGMs. This study aims to investigate the potential of combining roasting, leaching, and selective adsorption methods to efficiently recover PGMs from chromite tailings, which are often considered as waste material. The observation and research work by previous researchers indicated that it is a challenge to recover PGMs from chromite tailings using conventional methods. A pre step of roasting the chromite tailings at 1000 in presence of air was investigated and found to be too high for conversion of metals to their most soluble forms. A hydrometallurgical process for the recovery of PGMs by hydrochloric acid was also investigated. It was found that the sample contained a head grade of 2,05 g/t of Platinum and g/t of palladium.

The acid managed to leach both platinum and palladium on the roasted and unroasted samples. The unroasted sample showed high recoveries of PGMs (Pt 40-61% and Pd 50-75%) than the roasted sample with recoveries of (Pt 30-55% and Pd 40-75%) and was concluded that the roasting temperature was too high and not really effective. Further investigation on different roasting temperatures needs to be done to know the optimal roasting conditions for solubility of PGMs. An adsorption process was also investigated on both activated and raw mesoporous silica. The activated mesoporous silica adsorbent showed higher recoveries of 80-90% for platinum adsorption and 20-40% for palladium as expected. The adsorption on raw adsorbent did not perform well. Additional research work on alternative adsorbent materials with higher selectivity and efficiency and pregnant solution with minimised base metal concentrations still need to be done to further improve and check the effectiveness the adsorption of PGMs.

**Keywords**— Chromite Tailings, Roasting, Adsorption and Leaching.

## I. INTRODUCTION

Platinum mining in south Africa is important due to the country's abundant reserves, which account for more than 80% of global Platinum Group Metals (PGM) deposits. These mineral resources are primarily located at the bushveld igneous complex. The region's rich PGM deposits have made south Africa the leading producer of Platinum (Pt), Palladium (Pd), Rhodium (Rh) and other metals [1]. The growing demand for these metals has driven the need for alternative,

efficient and sustainable recovery methods. Chromite tailings are generated as a byproduct of chromite ore processing, which also represent a promising secondary source of PGMs [2]. However, the valuable platinum metals in the chromite tailings are usually present in very low concentrations and it makes their recovery challenging.

There was speculation and agreement that existed between the PGM and chromite producers that chromite was not to be recovered from UG2 tailings if platinum group metals were not recovered from chromite tailings. This alleged agreement was in place during the time Samancor and BHP Billiton acquired the Mill shell operation. While the alleged agreement could have been the factor, several external factors have contributed to the limited recovery of PGMs from chromite tailings such as [3]: the limited understanding of how PGMs occur in chromite seams which resulted in incorrect recovery methods causing the recovery of PGMs from chromite tailings to be uneconomical. Another factor is that due to the depressed PGM prices with less producers reduced the attractiveness and need of low volume PGM production.

In the late 1990s, three major changes occurred that mitigated the factors that were limiting the recovery of PGMs from chromite tailings such as when the demand for commodities increased worldwide, which resulted in higher PGM prices. This led to a significant increase in PGM productions volumes. Major PGM producers, especially Impala Platinum, began to offer refining services for third-party PGM concentrators, providing a market for low-volume platinum metals production [3]. The third change that occurred involved legislative changes to mineral rights and the emergence of Black Economic Empowerment (BEE) led to the growth of small and medium sized mining firms. These firms were targeting smaller deposits, such as PGM in chromite tailings, as the capital limitations made larger deposits less practical to pursue.

The methods that are being investigated to maximise the recovery of PGMs from chromite tailings include roasting, leaching, and selective adsorption using a suitable adsorbent. There are several reasons why these methods are now being preferred when dealing with byproduct of chromite ore processing such as increasing the demand for PGMs [4]. Recovering PGMs from chromite tailings reduces the quantity of material that needs to be disposed of and it also reduces the environmental impact they can cause.

This study is motivated by economic, environmental and technological considerations. Chromite tailings are generally

Happy Mphahlele, Department of Metallurgy at the University of Johannesburg in South Africa.

Elvis Fosso-Kankeu, Department of Metallurgy at the University of Johannesburg in South Africa

considered as waste products with little to no economic. However, the recovery from the tailings can add a significant economic value and create new revenue streams from materials that were previously considered as waste. As primary ore reserves of PGMs are gradually being depleted, exploring the secondary byproducts like chromite tailings can help diversify the PGM supply [5]. This will reduce the reliance on conventional methods which are often associated with high capital. Therefore, this research is needed for all these reasons mentioned above.

There are several factors that need to be considered when treating the chromite tailings to recover the platinum group metals such as optimum conditions of roasting and leaching while also minimizing consumption of energy and reagents so that they can differ from conventional methods. The process controls such as temperature and contact time should also be evaluated and controlled.

It has been observed by previous researchers that there is a challenge in recovering PGMs from chromite tailings using conventional methods, therefore there is a need to develop new methods and technology that will promote the effective recovery of PGMs from chromite tailings. A more efficient process such as Roasting, leaching, and selective adsorption need to be implemented to extract the PGMs that might be locked within the chromite tailings. Several types of adsorbents have previously been developed to remove metals from solutions [6-13].

The aim of this study is to selectively recover PGMs from chromite tailings through successive leaching and photocatalytic adsorption.

## II. METHODOLOGY

### A. Experimental procedures

#### Characterization

Chromite tailings was collected from the mineral processing lab at the University of Johannesburg, dried and pulverised to a fine powder to prepare the sample for roasting. It 15g of the representative sample was mixed with 5g Sasol wax and placed in an aluminum cup and compressed with a hydraulic press at a pressure of 20 tons for characterization using X-ray fluorescence (XRF) to check the chemical composition of the sample.

Fire assay was used to determine the concentration of precious metals in the sample before it was roasted. The pulverised sample of about 50g was mixed homogeneously with 150g of fire assay flux. The mixed sample was heated in a furnace at a temperature of 1100°C for an hour. The molten material was poured into a mold and allowed to cool and solidify. The metal button was then removed from slag and cleaned using a brush (this was done to remove any slag material attached to it), pulverized, and transferred to a 500 ml glass beaker. 200ml of nitric acid was added to the 500ml glass beaker and heated on the hotplate at maximum temperature until complete dissolution. The glass beaker was allowed to cool and 200ml of hydrochloric acid was added

and heated up at max temperature until half of the 200ml was dissolved. 10ml of hydrogen peroxide was added to the solution while it was still on the hotplate and heated for 30 minutes. The solution was then removed, transferred to the volumetric flask filled with water to the mark and analysed with AAS.

#### Roasting

Half of the raw sample was roasted at 1000°C for 1 hour oxidize the PGMs to their most soluble forms. After applying the optimum roasting conditions, the roasted product containing PGMs was directed to leaching process to further separate the PGMs from chromite tailings.

#### Leaching process

Leaching was carried out on both the roasted and unroasted sample using 2stage leaching process. All leaching experiments were conducted in a beaker, with solid/liquid ratio of 20% and constantly agitated at a speed of 300rpm using a magnetic stirrer. 1M Sulfuric acid was first used to leach base metals present in the sample at room temperature for 2 hours. The remaining solids after filtration were dried and used again for PGM leaching using 2M hydrochloric acid. The PGM leaching experiment was done at different time intervals of 30, 60, 90, 120, 150 minutes at temperatures of 25, 35, 45, 55, 65°C respectively to check the effect of time and temperature. The solution was filtered using filter paper and the Atomic adsorption spectroscopy (AAS) was used to determine the concentration of platinum and palladium in the pregnant solution. The remaining solids were discarded.

#### Selective adsorption

The activated and unactivated adsorbent (mesoporous silica) was received and used for adsorption of Pt & Pd from the pregnant leaching solution of both roasted and unroasted products. To identify the functional groups and bonds present in the adsorbent, Fourier-transform infrared spectroscopy (FTIR) was used. The mesoporous silica was placed in a beaker containing pregnant solution of PGMs using a solid/liquid ratio of 0,06%. The adsorption experiment was carried out at room temperature for a time period of 15, 30, 45, 60, 75 minutes to check the effect of time, providing sufficient time for Pt and Pd to be adsorbed onto the mesoporous silica surface. The mixture was filtered and the remaining solution was characterized using AAS to determine the concentration of the remaining Pt and Pd in the solution.

## III. RESULTS AND DISCUSSION

### A. Characterization

The Chromite tailings sample was analyzed for chemical composition and PGMs composition using XRF and Fire assay respectively and the results are shown in Table I and II.

TABLE I: CHEMICAL CONTENT OF MAIN ELEMENTS IN THE CHROMITE TAILINGS BEFORE ROASTING

Element	Na	Mg	Al	Si	P	S	K	Ca	Ti	V
Wt. %	0,37	4,67	6,02	12,43	0,001	0,06	0,08	2,97	0,44	0,20
Element	Cr	Mn	Fe	Ni	Cu	Zn	Sr	O		
Wt. %	13,41	0,14	19,69	0,16	0,03	0,05	0,01	39,24		

Table I shows high percentage of Cr (13,41%), Si (12,43%), Al (6,02%) and Fe (19,69%). The high content of chromium was expected in a chromite sample and the high iron content could be present in many forms such as magnetite  $Fe_3O_4$  and hematite  $Fe_2O_3$ . The content of silicon and aluminum could present if the form of silicate minerals. The low sulphur content of 0,06% could indicate the presence of sulphide minerals such as pyrite and chalcopyrite but in very low concentrations. It is important to note that might be present as sulphates rather than sulphides.

A low concentration of PGMs can mean that only a small amount of the metals can be extracted, making it challenging to economically extract. Generally, UG2 tailings with PGM grade of less than 3g/t is classified as low grade [14]. Therefore low recoveries should be expected for this type of material. For more accurate, XRD analysis is required.

Figure 2 and 3 shows the FTIR results for raw and activated adsorbent used for the selective adsorption.

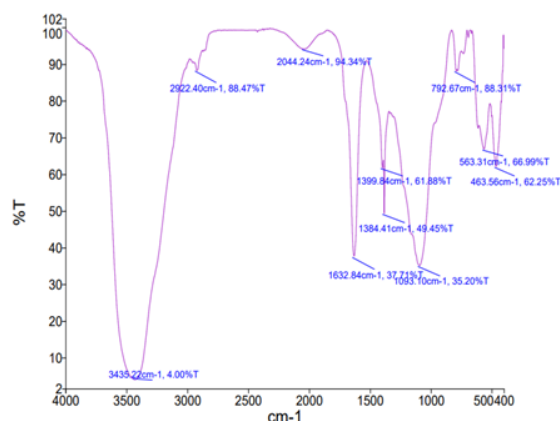


Fig. 1: FTIR spectra of the raw adsorbent

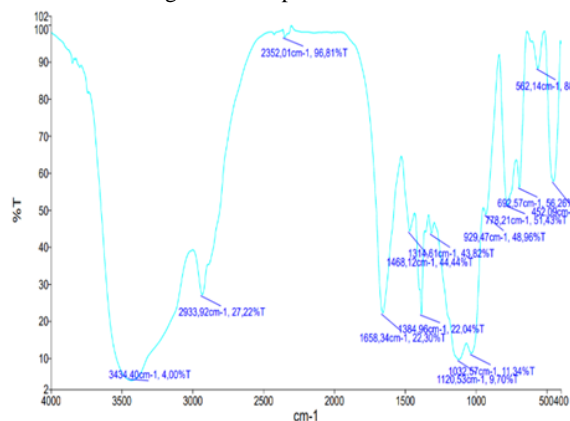


Fig. 2: FTIR spectra of the activated adsorbent

The FTIR graph for activated adsorbent on Figure 3 show more clear peaks compared to the graph of FTIR on raw

adsorbent. More peaks on Figure 3 indicate that some functional groups were modified during the functionalisation process and indicates that the functionalisation process has introduced new functional groups which are detectable and high number of binding sites present in the adsorbent which makes it more effective at binding to PGM ions. Both figures show a broad peak around 3438cm-1, which indicates O-H bonds that can increase the adsorption rate, but the raw adsorbent has more strong peaks in the range of 1500-1700cm-1, indicating C=O and C=C bonds. C-H bonds are found in the range of 2700-3000cm-1, which are less effective for ion adsorption because they are non-polar. There is a presence of COOH bonds in the range of 1700-1800cm-1, which means that there is going to be an interaction between ions in solution and the carboxyl groups, making the adsorbent more effective at binding of PGMs. The activated adsorbent will be more effective at binding PGM ions because it has more functional groups of OH, COOH, and C=O.

### B. Base metals leaching

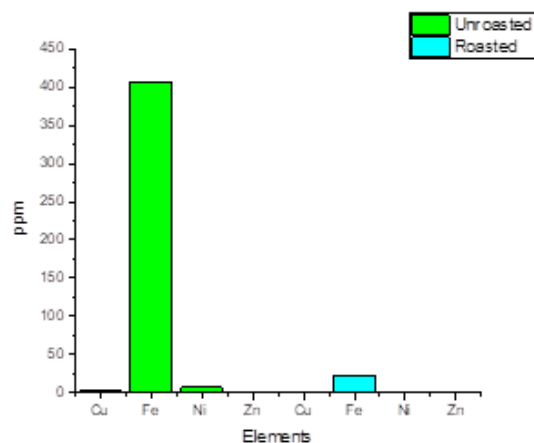


Fig. 3: Base metal leaching for both roasted and unroasted sample

Figure 4 shows the concentrations of leached base metals. The base metals were leached out more on the unroasted sample than the roasted one. This indicates that roasting has formed phases that are less reactive during leaching. This experiment was done because base metal ions can compete with PGM ions for adsorption sites on the adsorbent. Therefore by leaching out base metals first at optimal conditions, adsorption efficiency of PGMs can improve.

C. PGMs leaching

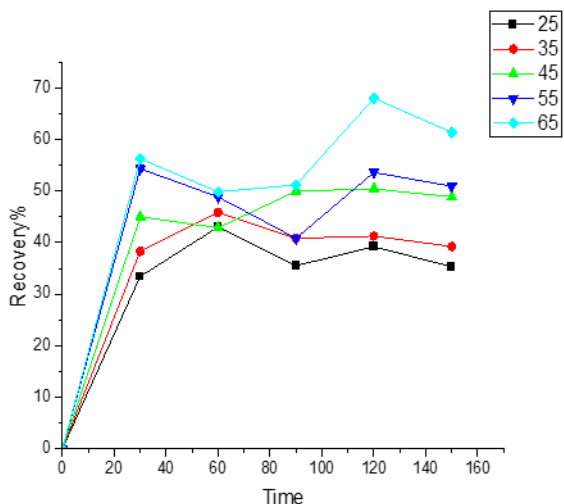


Fig. 4: Pt unroasted recovery graph

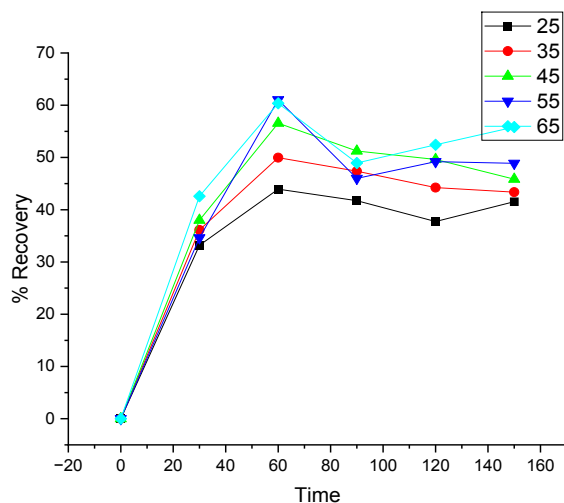


Fig. 5: Pt roasted recovery graph

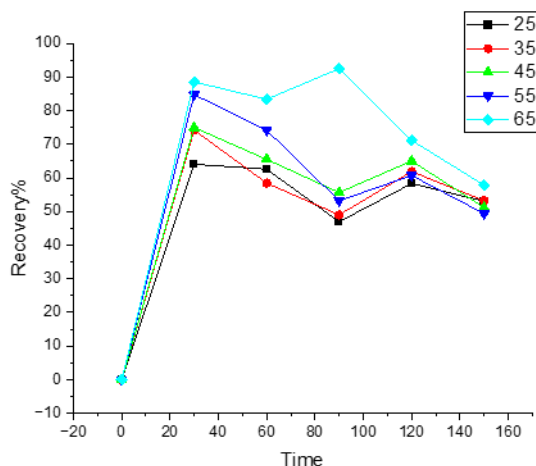


Fig. 6: Pd unroasted recovery graph

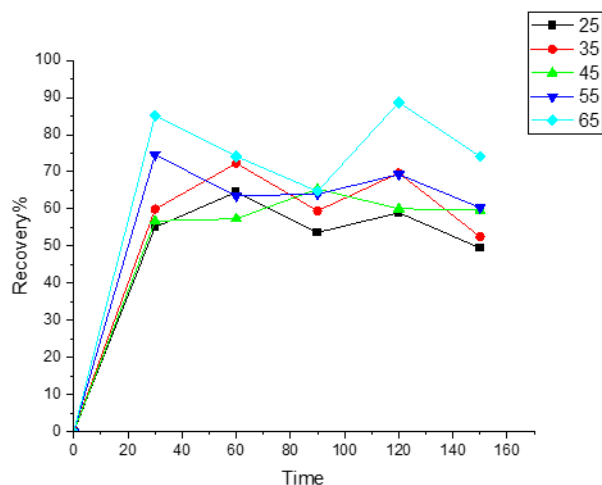


Fig. 7: Pd roasted graph

Highest recoveries were achieved at 65°C for all the PGMs but on figure 6 it was at 55°C and 65°C, which indicates that high temperatures can improve PGM recovery. For platinum, both roasted and unroasted samples behaved almost the same with just over 60% for roasted and over 65% for unroasted. For palladium, both roasted and unroasted also behaved almost the same with just below 90% for roasted and over 90% for unroasted. The roasting temperature used might have been too high and that may be reason why there isn't much difference on the recovery of these PGMs from the samples. The leaching times indicate high recoveries between 90 and 120 minutes for unroasted sample and 60-120minutes for roasted sample. This shows that even though longer leaching times can generally improve recovery, there is an optimal range of leaching times for both roasted and unroasted sample. The use of HCl and other leaching acids were found to effectively leach all PGMs present in the chromite tailings in literature, and for this experiment all PGMs present in sample were leached effectively with just HCL. Figure 11-18 shows the graphs of two models used in Shrinking core model.

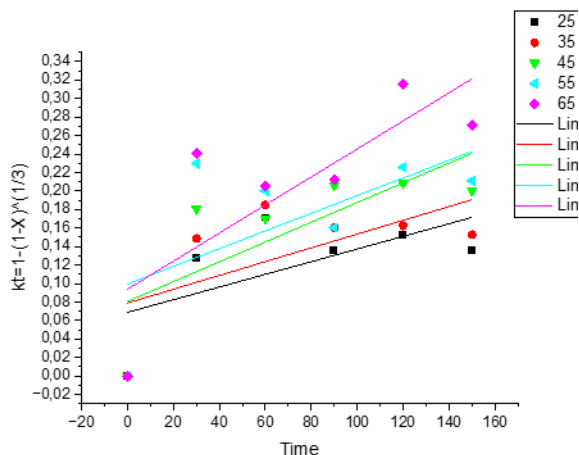


Fig. 8: 1st kinetic eq vs t plot of Pt Roasted

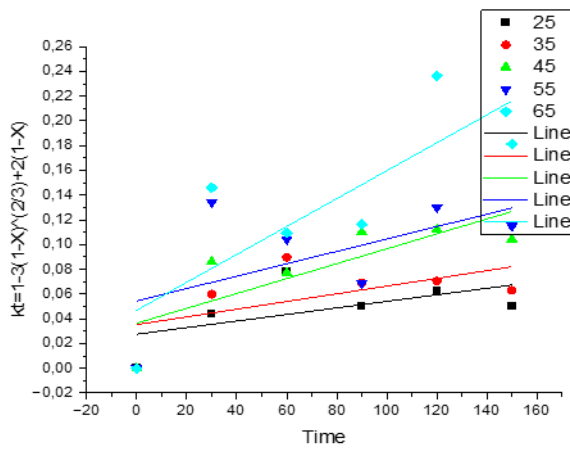


Fig. 9: 2<sup>nd</sup> kinetic eq vs t plot of Pt unroasted

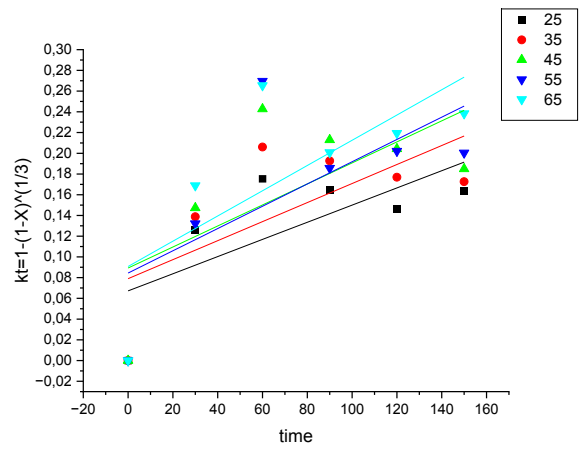


Fig. 121: 1<sup>st</sup> kinetic eq vs t plot of Pt unroasted

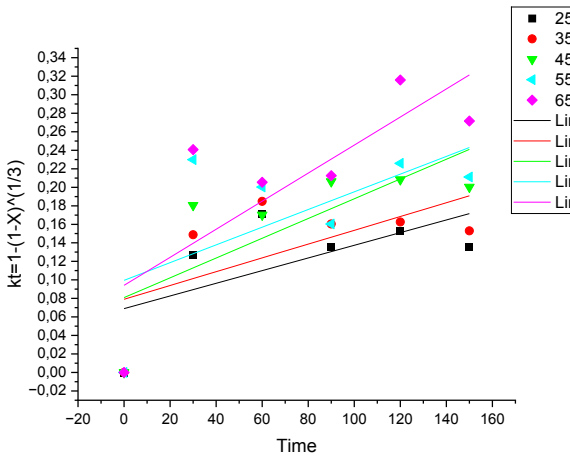


Fig. 10: 1<sup>st</sup> kinetic eq vs t plot of Pt roasted

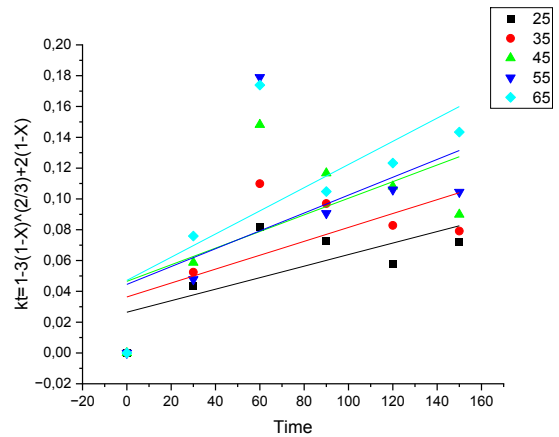


Fig. 13: 2<sup>nd</sup> kinetic eq vs t of Pt unroasted

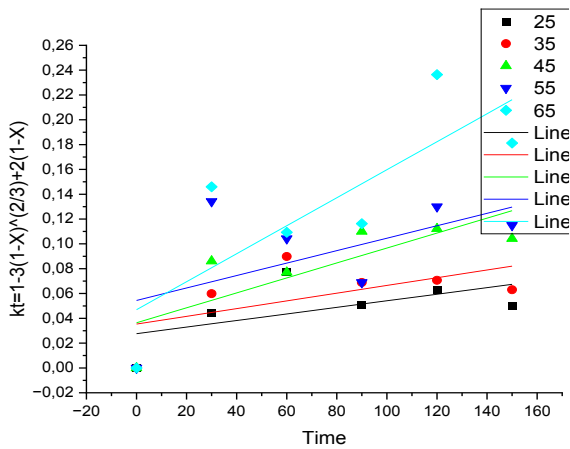


Fig. 11: 2<sup>nd</sup> kinetic eq vs t plot of Pt Roasted

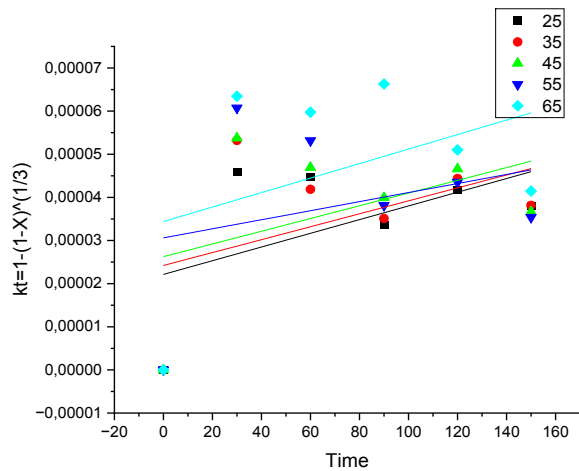


Fig. 142: 1<sup>st</sup> kinetic eq vs t plot of Pd unroasted

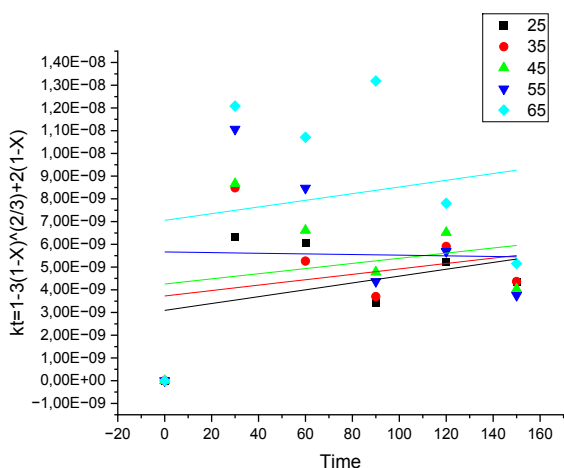


Fig. 15: 2<sup>nd</sup> kinetic eq vs t plot of Pd unroasted

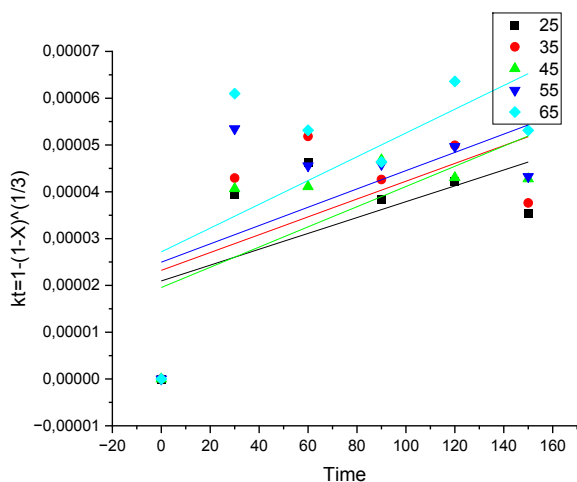


Fig. 16: 1<sup>st</sup> kinetic eq vs t plot of Pd roasted

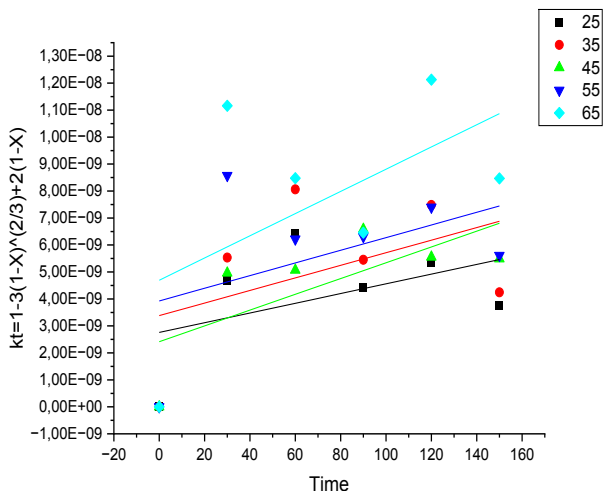


Fig. 17: 2<sup>nd</sup> kinetic eq vs t plot of Pd roasted

The activation energies of all kinetic equations used in Figures 11-18 were calculated using Arrhenius Equation and listed below in Table II. The slopes of the graphs presented in Figures 11-18 were determined and noted down as values of  $k$ .  $\ln(k)$  and  $1000/T$  were determined and used to calculate the activation energies of all conditions.

The study is focused on the leaching kinetics of Pt and Pd on roasted and unroasted samples and the shrinking core

model with constant particle size was applied for these results to help analyse the leaching experiment. To assess the goodness of fit for both order, values of  $R^2$  have to be closer to 1 to indicate a better fit. Even though the values of  $R^2$  are low, Leaching of platinum on roasted sample showed a better fit with  $R^2$  value of 0,60 for first equation and 0,63 for second equation followed by Pt unroasted, Pd roasted and Pd unroasted respectively.

The activation energy was also investigated to analyse the mechanism of the reaction during leaching of both precious metals. The activation energy of a chemically controlled, mixed and diffusion are usually  $>20$ ,  $(4-20)$  and  $<4$  kJ/mol respectively [15]. For Pt unroasted, both values from the two kinetic equations indicate a mixed control reaction. For Pt roasted and Pd roasted, first kinetic equation indicates mixed controlled reaction and second equations indicates a chemically controlled reaction. The Pd unroasted indicates a diffusion controlled reaction for first kinetic equation and chemically controlled for second kinetic equation.

The two different kinetic models used for each condition (roasted and unroasted sample) are:

First Kinetic equation:  $kt=1-(1-X)^{1/3}$

Second Kinetic equation:  $kt=1-3(1-X)^{2/3}+2(1-X)$

The activation energies (kJ/mol) were calculated for both Pt and Pd under different conditions and models. Activation energy is an important factor that can help indicate the minimum energy needed for the leaching reaction to occur. For Pt, as roasting is introduced, the activation energy increases. This can mean that roasted Pt might be more difficult to leach or it might be because of the high roasting temperature. If the sample was roasted at different temperatures below  $1000^{\circ}\text{C}$  it would be more clear as to how the leaching behaves under those conditions. The second kinetic equation shows higher activation energies compared to the first kinetic equation and it indicates that the leaching process may include multiple rate-limiting steps.

Generally, lower activation energies indicate that the PGMs will be easier to leach and based on the calculations, the unroasted sample is easier to leach, while the roasted sample might require more energy to efficiently leach out platinum.

#### D.Adsorption

Figure 19 shows the graph of recovery vs time for adsorption of PGMs on both raw and activated adsorbents.

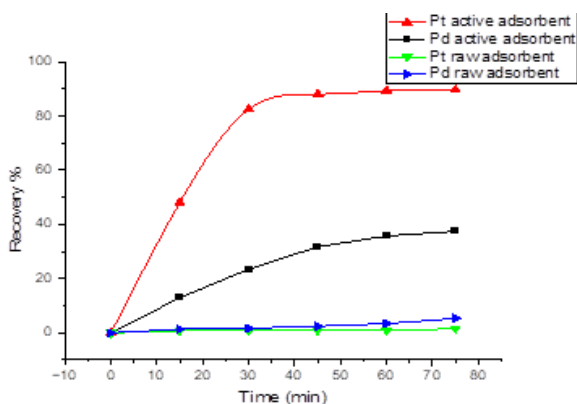


Fig. 18: Adsorption Recovery graph

For platinum recovery, the graph shows a faster response to the adsorption on the activated adsorbent reaching about 90% within 45min. For raw adsorbent, platinum shows almost no recovery. For palladium recovery on the activated adsorbent, there is a moderate increase compared to platinum. The Pd recovery was reaching 40% as time contact time increases. Palladium on raw adsorbent shows very little to no recovery throughout the experiment. For both PGMs with the activated adsorbent, the recovery rate is faster only within the first 30minutes. after 30 minutes it indicates that the adsorbent sites have been occupied. The raw adsorbent is less effective than the activated adsorbent. The mesoporous silica showed highly selective adsorption capacity for noble metal ions such as Pd<sup>2+</sup> and Pt<sup>2+</sup>, while the binding capacity for other metal ions (Ni<sup>2+</sup>, Cu<sup>2+</sup>, etc) as mentioned in the literature.

E. Adsorption kinetics

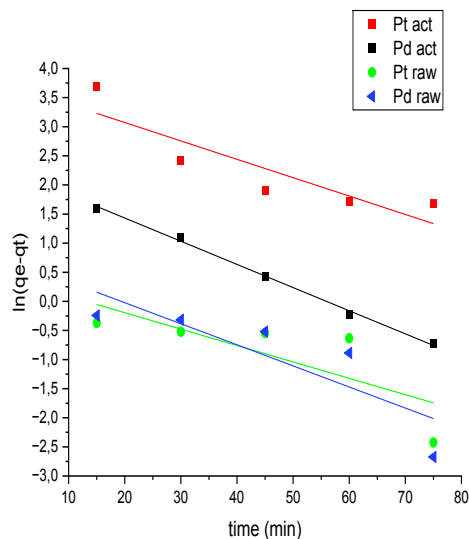


Fig. 19: Pseudo first order

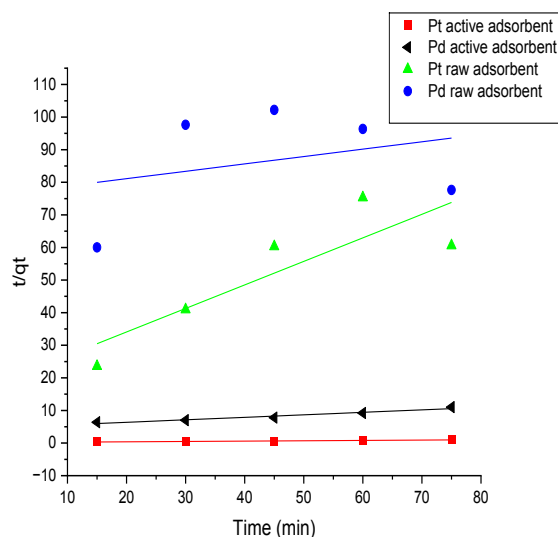


Fig. 20: Pseudo second order

A study of adsorption kinetics helps us analyse the adsorption mechanism, which is an important factor for efficiency of process. And the following equations were to plot graphs of both orders:

Pseudo first order:  $\ln(q_e - q_t) = -k \times t + \ln q_e$

Pseudo second order:  $t/q_t = (1/q_e) \times t + (1/k^2 \times q_e^2)$

The slopes ( $k_1$  &  $k_2$ ) and R squared of  $\ln(q_e - q_t)$  vs  $t$  and  $t/q_t$  vs  $t$  graphs were determined and listed below in Table II.

TABLE III: PSEUDO FIRST AND SECOND ORDER DATA

	Pseudo first order			Pseudo 2nd order	
	q <sub>e</sub>	K <sub>1</sub>	R <sup>2</sup>	K <sub>2</sub>	R <sup>2</sup>
Pt active adsorbent	80,1586	0,03157	0,79169	0,01084	0,97359
Pd active adsorbent	7,303023	0,03981	0,99659	0,07648	0,95413
Pt raw adsorbent	1,326707	0,02816	0,60523	0,7223	0,7268
Pd raw adsorbent	1,034951	0,03621	0,72688	0,22645	0,09262

To assess the goodness of fit for both order, values of R<sup>2</sup> have to be closer to 1 to indicate a better fit. The first order is more relevant for physisorption process and the second order is more relevant for chemisorption process.

Pt and Pd on active adsorbent shows that the second order model fits better than first order, indicating that chemisorption process is more dominant on Pt and on Pd it indicates a mix of chemisorption and physisorption with more dominance of physisorption. Pt on raw adsorbent shows that the second order fits better than the first order and Pd on raw adsorbent shows that the first order fits better than the second order, indicating that physisorption process is more dominant. K<sub>1</sub> values are similar on all adsorbents with a range of 0,028 to 0,0398. K<sub>2</sub> values vary, with Pt on raw adsorbent having the highest value of 0,07223 and Pt on active adsorbent having the lowest value of 0,01084.

IV. CONCLUSION

The investigation into the recovery of PGMs from chromite tailings and selective adsorption has proved that methods such



as roasting, leaching, and selective adsorption can improve the extraction efficiencies. The results indicate that optimizing roasting conditions can improve the solubility of PGMs. Furthermore, the use of selective adsorption with activated mesoporous silica shows promise in separation of PGMs from complex pregnant solutions. This study emphasises the importance of combining advanced recovery techniques such as roasting, leaching and selective adsorption to transform PGMs from tailings into valuable precious metals. As demand for PGMs globally continues to increase, these results offer a solution towards common conventional methods used in the industry as mentioned in the problem statement to further increase profits.

To improve the recovery of PGMs from chromite tailings, it is recommended that future research focuses on optimizing each stage of the recovery process, especially roasting and leaching and characterising the sample using XRD and SEM-EDS to know more about the mineral phases and surface analysis of the sample. Research on the effects of varying temperatures during roasting and reagent concentrations during leaching should be done to identify optimal conditions for PGM recovery. However, additional research work on alternative adsorbent materials with higher selectivity and efficiency and pregnant solution with minimised base metal concentrations still need to be done to further improve and check the effectiveness the adsorption of PGMs.

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