The Beneficiation of Oxidised PGMs from High-Chromite Ore tailings using Flotation

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***Abstract*—Flotation is a very crucial downstream processing stage in the Platinum Group Metals (PGMs) value chain, as it affects the preceding downstream processes should economic recoveries not be met. This process takes advantage of separating minerals based on their hydrophobicity or hydrophilicity. The flotation of oxidised PGMs has been an area of concern over the years, as conventional methods have failed to achieve economic recoveries. Early efforts to process oxidized PGM ores using conventional flotation methods yielded poor recoveries, typically below 50%, making commercial exploitation challenging. Thus, the effect of co-collectors in the flotation of oxidised PGM ore was investigated by assessing their impact on flotation performance, recovery, and selectivity. The experimental setup was designed using Response Surface Methodology (RSM). Due to the nature of the ore source, the sample was first preconcentrated using the shaking table to reduce the chromite content before flotation. The parameters under study for the flotation process were the dosage of the collector, the depressant, and the effect of time. Based on the optimal conditions determined through RSM, a kinetic study was conducted under those conditions to investigate the flotation rate mechanisms of oxidised PGMs, which are known to exhibit both fast and slow-floating particle behavior. The optimal conditions according to RSM were found to be at pH 8 and dosages of 300 g/t,80 g/t for both SIBX and Senkol 1238, respectively. Under these conditions, a maximum grade of 3.9058 g/t and a recovery of 48.43% for 3PGE+Au was achieved. The flotation kinetics study indicated that all 3PGE+Au components followed the modified Kelsall model.**

***Keywords*—Flotation, Kinetics, Oxidised PGMs, Collector**.

I. INTRODUCTION

Africa is home to some of the world's richest PGM deposits, situated in the southern part of Africa, particularly in South Africa and Zimbabwe. These deposits are distributed across the Main Sulfide Zone (MSZ) of the Great Dyke in Zimbabwe and the Bushveld Igneous Complex (BIC) in South Africa [1]. The BIC, which accounts for 77.78% of PGMs in the world, hosts 3 main reefs, namely the Merensky reef, Plat reef, and Upper Group 2(UG2) [2]. The Merensky Reef has been heavily exploited over the years due to its relatively high PGM grades. Historically, the PGMs from the UG2 chromite value chain were considered uneconomic to process due to their presence in low concentrations [3]. However, due to the fast depletion of high-grade PGM deposits and technological advancements over the years, this has induced the need to recover these PGMs from chromite tailings. Due to prolonged exposure to the water and oxygen, these tailings undergo oxidation and a change in their surface properties [4].

Traditional flotation methods using xanthate-based collectors have shown limited effectiveness in treating oxidized PGM ores, primarily due to surface oxidation, the formation of metal hydroxides, and weak interactions between collectors and mineral surfaces [5]-[6]. The inefficiency of these conventional sulfide collectors results in inconsistent recoveries, often below 50% depending on the degree of oxidation. Despite various efforts to enhance PGMs flotation from oxidized ores, no commercially viable process has been developed, as previous attempts have failed to achieve economically sustainable recoveries [7]. Thus, the scope of the current study seeks to bridge the knowledge gap by evaluating the effect of co-collectors in the flotation of oxidised PGMs from a high chromite source, as well as to study the rate mechanisms that govern the flotation process.

II. METHODOLOGY

A. *Materials and analytical equipment*

The received chromite tailings sample was sourced from the western limb of the Bushveld complex. The reagents used to conduct the study were supplied by AECI Mining, South Africa.

The X-ray fluorescence (XRF) analysis was employed to determine the elemental composition of the ore sample. Then X-ray diffraction (XRD) was used to identify and characterize the mineral phases within the sample, to better understand the mineralogical composition. The SEM-EDS was also employed to study the surface morphology of the oxidised PGM's ore and to better understand the degree of liberation. Due to the limitations of XRF to quantify precious metals, fire assay analysis was conducted to quantify the concentration of PGEs using the Atomic Absorption Spectroscopy (AAS) technique.

B. *Flotation experiments*

Based nature of the ore tailings sample, which is characterized by high chromite, the sample was first pretreated with shaking to reduce the chromite content, and then the middling and tailings of the shaking table were reduced to 80% passing 75 μm, which served as the feed to the flotation process. The flotation experiments were designed using Response Surface Methodology (RSM) to determine the optimum operating conditions for oxidized PGMs flotation. The experimental design based on RSM variables resulted in a total

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of 15 experimental runs, with the factors under investigation summarized in Table I.

TABLE I: FACTORS UNDER STUDY

Factor Name	Coded Symbol	Low (-1)	Mid (0)	High (+1)
Senkol 1238	A	40 g/t	60 g/t	80 g/t
pH	B	8	9	10
SIBX	C	300 g/t	450 g/t	600 g/t

The flotation tests were done in batch stages, wherein Senfroth 150 and Sendep 30D were kept constant at dosages of 40g/t and 400 g/t in all the tests, with a pulp density of 20% solids, which was also held constant throughout. The ore sample was milled for 45 minutes at 80% passing 75 µm, then transferred into a 1.0-L flotation cell using water and filled to the correct volume for the flotation process. The slurry was stirred using a top-driven impeller at a constant speed, set to 1200 rpm. After each flotation stage, the concentrates and tailings were taken to the oven drier to dry overnight at 50℃, then taken for characterization. The reagent dosages, functions, as well as the conditioning time to allow the reagents to stabilize in the flotation system are well summarized in Table II.

TABLE II: SUMMARY OF FLOTATION REAGENT SCHEMES

Reagent(s)	Reagent Dosage(s)	Conditioning Time(min)	Function(s)
SIBX	300-600 g/t	3	Collector (1)
Senkol 1238	40-60 g/t	3	Collector (2)
Sendep 30D	150-450 g/t	3	Depressant
Senfroth 150	40 g/t	2	Frother
$CuSO_4$	30-60 g/t	3	Activator
CaO / H_2SO_4	1-4 drops	-	pH modifiers

C. *Flotation kinetics*

Due to the inherent heterogeneous nature of the PGM’s ore, which is characterized by both fast-floating and slow-floating particles, a kinetic study was also conducted. To conduct the kinetic flotation study, 4 kinetic models were adopted and fitted using MATLAB, as highlighted in Table III.

TABLE III: KINETICS MODELS FOLLOWED

Model Name	Model	Kinetic Parameters
Classical first-order model	$R = R\infty(1 - e^{-kt})$	R∞and k
Klimpel	$R = R\infty(1 - \frac{1}{k_{max}t}(1 - e^{-k_{max}t}))$	R∞and k□ax
Kelsall	$R = R_{fast}(1 - e^{-k_{fast}t}) + R_{slow}(1 - e^{-k_{slow}t})$	100%=Rfast + Rslow
Modified Kelsall	$R = R_{fast}(1 - e^{-k_{fast}t}) + R_{slow}(1 - e^{-k_{slow}t})$	R∞ =Rfast + Rslow

Where **R∞**-maximum recovery, **K_{fast}**-Flotation rate constant for fast-floating particles, **K_{slow}**-Flotation rate constant for fast-floating particles, **R_{fast}**-Recovery of fast-floating particles, **R_{slow}**-Recovery of fast-floating particles, **t** – flotation time.

Fig. 1 depicts how the flotation kinetics study was conducted. R_1, R_2, R_3, and R_4 are the recoveries of the concentrate recovered at times 1, 4, 9 and 17 minutes, respectively. RT is the final tailings that remained after the final concentrate R_4 at time 17 minutes was recovered.

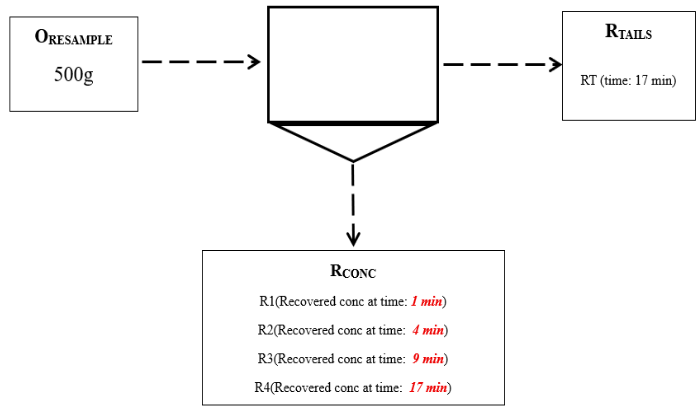

Fig. 1. Magnetization as a function of applied field.

III. RESULTS AND DISCUSSION

A. *Particle size distribution (PSD) of the as-received sample*

To characterize the head sample in terms of size, the particle size distribution was plotted to determine the F80, as highlighted in Fig. 2. It can be observed from Fig. 2 that 80% of the head sample passed the screen size 350 µm, as highlighted by the red interpolation.

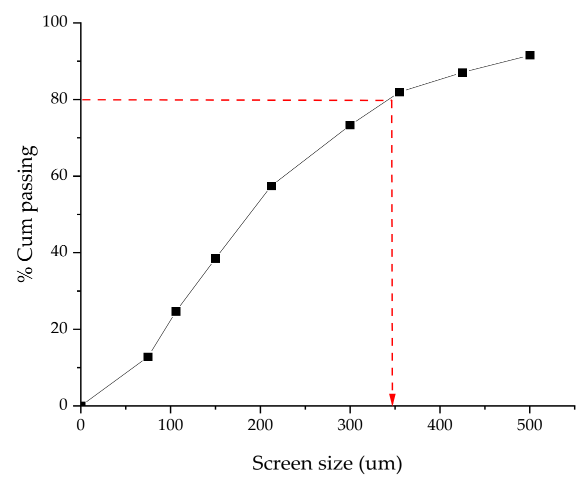

Fig. 2. PSD of the as-received sample.

B. *Characterisation of the as-received sample*

To account for the composition of the representative head sample, XRF analysis was conducted to determine its chemical composition, while XRD was used to determine the present mineral phases in the ore sample. Due to the limitations of XRF to quantify precious metals, fire assay analysis was also conducted to quantify the concentration of precious metals using the Atomic Absorption Spectroscopy (AAS) technique.

According to Table IV, the Head sample contained 28.15% Iron (Fe), 11.03 %Chromium (Cr), 9.61% Silicon (Si), 0.053% Copper (Cu), and 0.520 % Nickel (Ni). The major elements in the ore sample are Fe, Cr, and Si, with Cu and Ni as the minor elements. According to [2], elevated Fe and Cr contents indicate the presence of chromite, while the occurrence of Cu and Ni suggests the presence of PGMs, which are closely associated with base metals.

TABLE IV: XRF RESULTS OF THE AS-RECEIVED SAMPLE

Elements	**Fe**	**Cr**	**Si**	**Cu**	**Ni**
Wt%	28.15	11.03	9.61	0.053	0.52

Further analysis was done to quantify the presence of precious metal. According to Table V, the major elements present were 3PGE + Au. Platinum (Pt) has been quantified as the major PGE with rhodium (Rh) as the minor element. As highlighted in the literature, the presence of low Pt: Pd signifies the occurrence of oxidation. According to [8], the Pt: Pd ratios increase from 1.7 in pristine ores to greater than 2.3 in oxidized ores due to the mobilization of palladium (Pd). Based on the obtained results, the ratio of Pt: Pd is ≈ 4.3, which signifies that the ore sample has undergone oxidation due to the mobilization of Pd.

TABLE V: HEAD GRADE ANALYSIS USING AAS

Au(g/t)	Pd(g/t)	Pt(g/t)	Rh(g/t)	3PGE+Au
0.02	0.54	2.32	0.29	3.17

Confirmatory analysis was carried out to complement the bulk chemical results, with XRD employed for qualitative phase identification. According to Fig. 3, XRD reveals the presence of chromite, quartz, chalcopyrite, and pentlandite. The presence of these mineral phases is well justified by XRF chemical analysis, which highlighted Fe/Cr and Si as the major elements. Trace amounts of Cu and Ni detected by XRF indicate the presence of PGMs, which are typically associated with chalcopyrite and pentlandite based on their geological formation, and were further confirmed by XRD analysis [4]. Early researchers noted that the presence of phyllosilicates (such as talc, augite, and clinochlore) serves as an indicator of hydrothermal alteration in magmatic Ni-Cu-PGM ores. Their occurrence as gangue minerals often implies poor flotation response of PGMs in the affected samples [2].

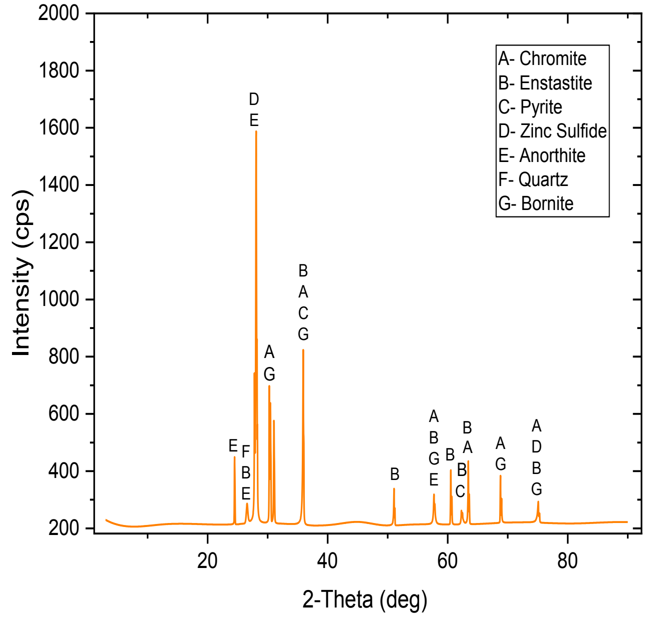

Fig. 3. XRD pattern of the as-received sample.

C. Flotation optimization results

As highlighted in the methodology, the RSM approach was used as the optimization tool to analyze closely the parameters that govern the flotation process. The optimization process begins with an analysis of the factors influencing the response variables using a Pareto chart generated from the RSM, as shown in Fig. 4.

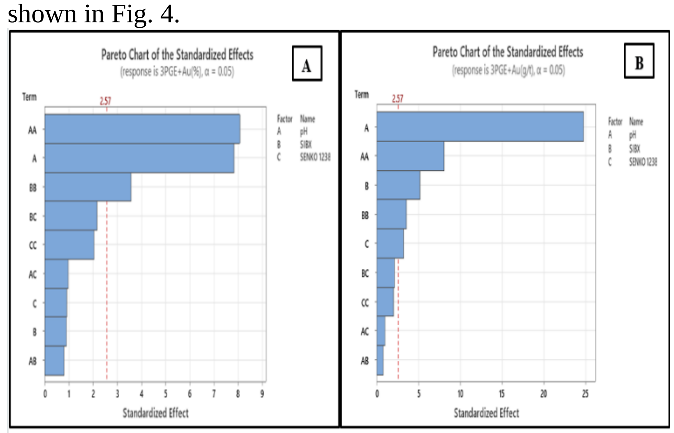

Fig. 4. Pareto charts for PGEs & Au (recovery and grade)

Fig. 4 presents two Pareto charts of the responses under study, namely 3PGE+Au %recovery and 3PGE+Au grade. The red dotted lines at a P-value of 2.57 serve to distinguish between interactions that have a significant and non-significant influence on the responses under study. All the interactions above the red dotted line are deemed to have a significant influence on the responses, and those below have less significant influence on the responses.

According to the response for 3PGE+Au% recovery, it can be observed that pH and SIBX are the major parameters that influence the response, as shown by interactions (AA) followed by (A) and (BB), respectively, from Pareto chart A. For 3PGE+Au grade, pH, Senkol 1238, and SIBX interactions are the major parameters that influence the response, as shown by interactions (A), (AA), (B), (BB), and (C), respectively.

Further analyses were made to closely study the interactions between Kerosene and Oleic acid on the responses under study, as shown in Fig. 5. The contour plots in Figures C and D represent the 2D interactions, whereas the surface plots in Figures E and F represent the 3D interactions of the variables on the response.

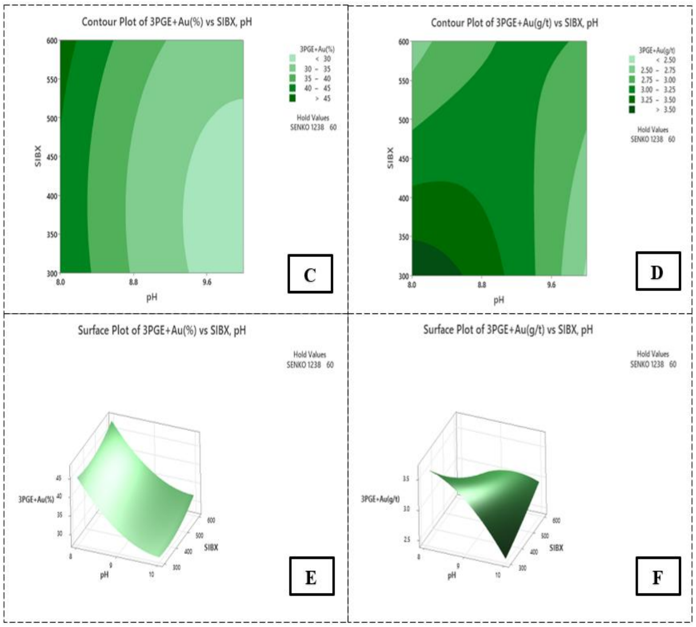

Fig. 5. Contour plots and surface plots for each response.

The surface plots and the contour plots in Fig. 5 were used to further explain the interactions of the parameters, considering the SIBX and pH against Senkol 1238 as key parameters based on the scope of the study. According to the contour plot of the % 3PGE+Au recovery, it can be seen that to maximise the recovery to > 45%, the SIBX dosage should be between 500 to 600 g/t at pH 8 to 8.2. Similarly, for 3PGE+Au grade, to get a grade >3.5, the pH needs to be between 8 and 8.6 at SIBX dosages (300 -350 g/t).

Fig. 6 depicts the conditions where maximum %3PGE+AU recovery and 3PGE+Au grade can be achieved based on the set parameters.

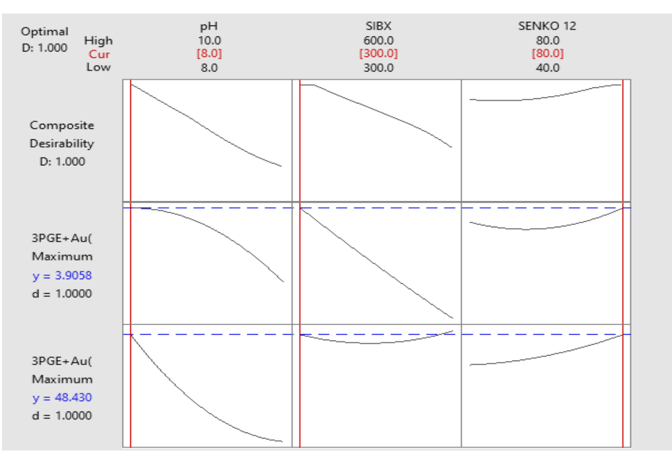

Fig. 6. The desirability responses.

Through the regression models generated, it was observed that based on the set parameters, the maximum 3PGE+Au grade that can be achieved is 3.9058 at %3PGE+Au recovery of 48.430% as shown in Fig. 6. These responses can be achieved at pH 8, dosage (80 g/t) for Senkol 1238 and (300 g/t) SIBX. As noted in the literature review, at pH levels 8 - 9, the PGMs particles exhibit high recoveries. According to [9], this phenomenon can be attributed to the pH-dependent modulation of surface charge and collector-mineral interactions.

D. *Flotation kinetics*

To account for the rate mechanisms that govern the flotation process, 4 kinetic models were tested . The models were fitted to the experimental results using MATLAB. Table VI highlights the kinetic results that were generated following the optimal conditions from RSM. The table highlights the recovery percentages of the 4 elements of interest, R1, R2, R3, and R4 are the recoveries of the concentrate generated from flotation times 1,4,9, and 17 minutes, respectively.

TABLE VI: FLOTATION KINETICS ON THE OPTIMAL CONDITIONS FROM RSM.

Time	Optimal Conditions	pH 8, SIBX 300 (g/t), Senkol 1238 (80 g/t)				
(mins)	Cumulative recovery	%Pt	%Pd	%Rh	%Au	%3PGE+Au
1	R1	44,69	40,22	33,55	0,915	42,63
4	R1 + R2	53,94	49,29	41,63	8,165	51,73
9	R1 + R2 + R3	57,29	51,5	42,74	9,275	53,84
17	R1 + R2 + R3 + R4	59,64	53,86	43,854	10,51	55,95

According to Table VI, it can be noted that platinum had a relatively high cumulative recovery for all the flotation times. This observation is consistent with findings published by [10], who reported that platinum exhibited the fastest floatability among the PGEs studied, reflected by the highest ultimate recovery values and strong kinetic fits, which confirms its tendency to maintain high recoveries throughout flotation tests.

The model was selected on the basis of the coefficient of determination(R^2) as well as the maximum recovery. Among the evaluated models, the modified Kelsall model stood out, with an R2 value which closer to unity, as highlighted in Table VII in the appendix. The Kelsall model had a higher coefficient of determination for the 3PGEs. However, a deviating trend can be observed for Au, which shows that the flotation kinetics is defined by both the Classical and Kelsall models. Further evaluations were carried out to identify the most appropriate kinetic model describing gold flotation, with the Root Mean Square Error (RMSE) being used as a measure of model accuracy. The Kelsall model had a <RSME compared to the classical model, thus it can be concluded that the modified Kelsall model provided the best fit to the experimental data and is better suited to explain the rate mechanisms governing the flotation of PGEs+Au.

Fig. 7 complements the data presented in Table VII in the appendix. *Plots G, H, I, and J present the fitting plots for Platinum (Pt), Palladium (Pd), Rhodium (Rh), and Gold (Au), respectively.*

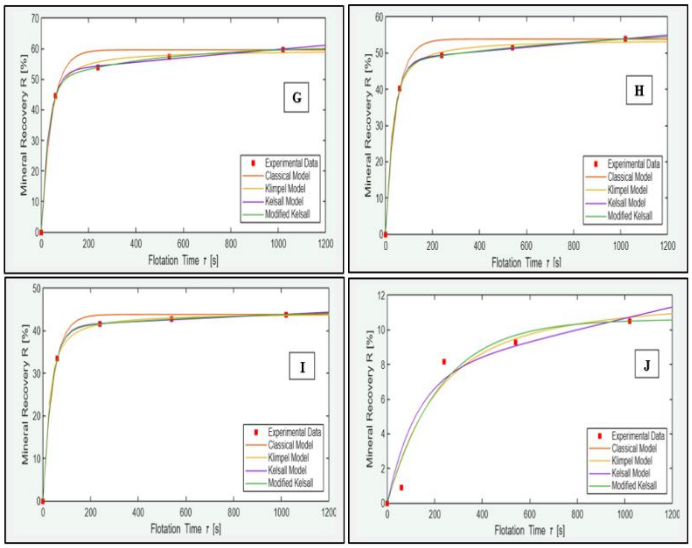

Fig. 6. 3 PGEs +Au Recovery data fit results using 4 models.

IV. Conclusion

The study provided the basis for the flotation of oxidized PGMs, which is one area of study that is still in an embryonic stage. The study evaluated the effect of co-collectors in the flotation of oxidised PGMs from a high chromite source, as well as the rate mechanisms that govern the flotation process. Mineralogical analysis confirmed the presence of PGMs closely associated with high chromite content. Flotation optimisation results indicated that pH and SIBX dosages were the most significant factors affecting both grade and recovery, as illustrated by the Pareto chart. The optimal conditions according to RSM were found to be at pH 8 and dosages of 300 g/t,80 g/t for both SIBX and Senkol 1238, respectively. Under these conditions, a maximum grade of 3.9058 g/t and a recovery of 48.43% for 3PGE+Au can be achieved. The flotation kinetics study indicated that all 3PGE+Au components followed the modified Kelsall model.

Appendix

TABLE VII: KINETIC MODEL EVALUATION

Modified Kelsall model	**R^2**	**R∞**	**K_{fast}/min**	**K_{slow}/min**	**RMSE**
Pt	1	0,60915	2,117	0,13059	0,00132
Pd	1	0,57606	1,8448	0,061079	0,00106
Rh	1	0,45429	1,7156	0,06688	0,0061
Au	0,98149	0,10615	0,5	0,27082	**0,0046**
3PGE+Au	1	0,58676	1,8909	0,071706	0,00188
Classical model	**R^2**	**R∞**	**K/min**		**RMSE**
Pt	0,99608	0,5964	1,3592	-	0,0010
Pd	0,99688	0,5386	1,3516	-	0,00548
Rh	0,99896	0,43854	1,4375	-	0,00255
Au	0,98149	0,10615	0,27082	-	**0,0052**
3PGE+Au	0,99755	0,55953	1,4182	-	0,00980
Klimpel model	**R^2**	**R∞**	**K_{fast}/min**		**RMSE**
Pt	0,98411	0,5964	3,7706	-	0,0089
Pd	0,98963	0,5386	3,7689	-	0,0061
Rh	0,99879	0,44268	4,0592	-	0,00541
Au	0,97008	0,1215	0,50153	-	0,00445
3PGE+Au	0,99226	0,55953	4,0581	-	0,00631
Kelsall model	**R^2**	**phi (∂)**	**K_{fast}/min**	**K_{slow}/min**	**RMSE**
Pt	0,99985	0,47471	1,8432	0,0099636	0,00112
Pd	0,99998	0,51984	1,7712	0,0071427	0,00771
Rh	0,99999	0,58907	1,6727	0,0028807	0,00356
Au	0,9744	0,92666	0,5	0,0021999	0,00447
3PGE+Au	0,99997	0,4941	1,8075	0,0069073	0,00118

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