Development of a Sustainable Thiosystem Leaching and Nano-Adsorption Process for Recovery of Platinum Group Metals from Oxidised Ores

Nancy R Mabwe, Willie Nheta and Vusumuzi Sibanda

Abstract**—**Platinum group metals (PGMs) are strategic materials whose recovery from oxidised ores is constrained by complex mineralogy and the limitations of conventional processing. This study developed an integrated hydrometallurgical route combining thiosystem leaching with a bio-derived carbon-nanotube (CNT) adsorbent. An oxidised Great Dyke flotation concentrate grading 50.96 g/t 4E PGMs was treated using mixtures of thiosulphate, thiourea and thiocyanate with Cu (II) catalyst and H_2O_2 oxidant. The highest observed aggregate 4E leaching efficiency was 41.35%. The leach liquor was treated using a potato-peel-derived CNT adsorbent. CNT adsorption reached 99.43% PGM removal at 0.6 g/L, while the non-linear pseudo-first-order model gave $R^2 = 0.995$. After five sorption cycles and thermal regeneration, sorption remained 52 ± 4%. Economic modelling indicated higher mean NPV for the nano-adsorbent system (USD 1.45 million) than activated carbon (USD 0.92 million). The integrated route demonstrates potential for greener PGM recovery, while further scale-up and metal-specific validation are required.

Index Terms**—**Adsorption, Carbon Nanotubes, Oxidised Ore, Platinum Group Metals, Thiosystem Leaching

I. Introduction

Platinum group metals (PGMs)—platinum (Pt), palladium (Pd), rhodium (Rh), ruthenium (Ru), iridium (Ir), and osmium (Os)—are valued for their catalytic activity, corrosion resistance, electrical properties, and high-temperature stability. They are used extensively in automotive catalysts, chemical processing, electronics, jewellery, and emerging hydrogen technologies. Recent reviews emphasise that the increasing strategic importance of PGMs is accompanied by a need for lower-impact recovery routes and improved separation technologies for increasingly complex and dilute resources [1]–[4].

In Zimbabwe, the Great Dyke hosts extensive PGM resources, including near-surface oxidised materials. Oxidation alters mineral surfaces and associations and can reduce the effectiveness of conventional flotation and downstream processing. The flotation concentrate used in this study contained Pt, Pd, Rh and Au as the measured 4E suite, with a combined grade of 50.96 g/t. The challenge is therefore not simply dissolution, but development of an integrated route that can liberate PGMs using less aggressive chemistry and subsequently recover dissolved species from a complex liquor.

Cyanide remains a benchmark lixiviant for precious-metal extraction because of its strong complexing ability and mature downstream recovery technology. However, concerns associated with cyanide toxicity and environmental management have stimulated research into alternative lixiviants. Thiosulphate, thiourea and thiocyanate are among the most studied alternatives. Thiosystem chemistry can provide stable metal complexes, but reagent consumption, degradation, redox control and downstream recovery remain important barriers to wider implementation [1], [5], [6].

Adsorption is attractive for recovering PGMs from dilute leach liquors because adsorbents can combine high surface area, tunable chemistry and regeneration potential. Recent reviews identify solid-phase adsorption as an important route for PGM separation, while also emphasising the need to move from idealised single-metal solutions toward realistic multicomponent liquors [2], [3].

This study therefore investigated an integrated process comprising flotation upgrading, modified thiosystem leaching, adsorption using a potato-peel-derived CNT nano-adsorbent, regeneration, and preliminary economic evaluation. The work specifically sought to demonstrate process compatibility rather than claim industrial-scale optimisation. The conference paper also addresses the three reviewer-specified themes by providing more detail on the PGMs, incorporating recent literature, and quantifying the economic implications of the proposed process.

II. Materials And Methods

A. Feed Characterisation and Flotation

The oxidised PGM ore was sourced from a platinum operation on the Great Dyke, Zimbabwe. Mineralogical and chemical characterisation employed X-ray diffraction (XRD), scanning electron microscopy with energy-dispersive spectroscopy (SEM–EDS), wavelength-dispersive X-ray fluorescence (WDXRF), atomic absorption spectroscopy (AAS), inductively coupled plasma optical emission

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spectroscopy (ICP–OES), and particle-size analysis. The ore was dominated by siliceous and talc–serpentine gangue. Flotation was conducted using a mixed collector system of sodium isobutyl xanthate and a hydroxamate co-collector, carboxymethyl cellulose depressant, copper sulphate activator, and an alcohol-based frother. The target grind was approximately 60% passing 75 μm.

The flotation concentrate was homogenised and analysed for Pt, Pd, Rh and Au. The aggregate 4E grade was calculated from the four measured metals. These four metals were used as the analytical response suite in the subsequent leaching and adsorption work.

TABLE I: Measured 4E Composition Of The Flotation Concentrate

Metal	Concentrate grade (g/t)
Pt	30.28
Pd	15.14
Rh	2.77
Au	2.77
Total 4E	50.96

B. Thiosystem Leaching

Leaching experiments used 100 g of flotation concentrate. The investigated lixiviant system combined sodium thiosulphate, thiourea and thiocyanate. Cu (II) was used as a catalyst and hydrogen peroxide as an oxidant. Reagent combinations were evaluated using a response-surface design. Tests were conducted at ambient temperature unless otherwise specified, with the leaching vessel stirred briefly after reagent addition and then allowed to react for 72 h.

The aggregate 4E leaching efficiency was calculated from the total PGM content measured in the leachate relative to the total 4E PGM content in the feed. Individual Pt, Pd, Rh and Au values are reported as measured leachate grades; they are not interpreted as individual recovery percentages because a consistent metal-specific feed–solution–residue mass balance was not established.

TABLE II: Highest-Response Thiosystem Condition (Mix 12)

Parameter	Mix 12 condition
Flotation concentrate	100 g
Thiosulphate	92 mL of 2 M
Thiourea	Not added in Mix 12
Thiocyanate	184 mL of 4 M
Cu (II) catalyst	<1 mL
H_2 O_2	10 mL
Observed aggregate 4E efficiency	41.35%

C. Nano-Adsorbent Preparation and Adsorption

Potato peel waste was washed, dried and milled before chemical activation. Phosphoric acid and potassium oxalate were used to prepare activated carbon intermediates. CNT formation employed an iron oxalate precursor and melamine followed by controlled pyrolysis at 600 °C and 900 °C. The resulting carbon nanomaterial was washed, dried and characterised using XRD, FTIR, BET surface-area analysis, SEM–EDS, and proximate/ultimate analysis.

Adsorption tests used 20 mg adsorbent dispersed in 100 mL thiosystem leach liquor in a shaking reactor. The variables investigated in response-surface modelling were pH (2–10), contact time (10–180 min), adsorbent dose (0.1–0.8 g/L), and temperature (20–60 °C). Adsorption performance was assessed from the reduction in dissolved aggregate PGM concentration. Langmuir and Freundlich equilibrium models and non-linear pseudo-first-order (PFO) and pseudo-second-order (PSO) kinetic models were fitted to the data.

Regeneration was evaluated using pristine CNTs and CNTs subjected to five sorption cycles followed by thermal regeneration. Acid-assisted desorption used 3 M HNO_3 . A preliminary Monte Carlo economic model compared the nano-adsorbent route with activated carbon and conventional leaching assumptions.

III. Results And Discussion

A. Flotation Upgrading

The flotation stage increased the total measured 4E grade from 2.37 g/t in the ore to 50.96 g/t in the concentrate, corresponding to an enrichment factor of approximately 21.5. The concentrate contained 30.28 g/t Pt, 15.14 g/t Pd, 2.77 g/t Rh and 2.77 g/t Au. The strong enrichment confirms that flotation can provide a suitable feed for downstream hydrometallurgical treatment even though the oxidised ore presents mineralogical and surface-chemistry challenges.

The distribution of the measured 4E metals was dominated by Pt and Pd. This is relevant to downstream recovery because the value of the concentrate is strongly influenced by the relative contribution of the individual PGMs. Nevertheless, the subsequent leaching response is reported here as an aggregate 4E response because metal-specific leaching recoveries were not independently established on a complete mass-balance basis.

B. Thiosystem Leaching Performance

Across the tested reagent combinations, Mix 12 produced the highest experimentally observed aggregate 4E leaching efficiency of 41.35%. The corresponding measured leachate grades were 75.54 g/t Pt, 18.03 g/t Pd, 8.26 g/t Rh and 1.43 g/t Au, giving a reported total leachate grade of 104.26 g/t. These values describe the measured composition of the leachate and should not be interpreted as 41.35% recovery for each individual metal.

TABLE III: Mix 12 Leachate Composition

Mix 12 leachate	Measured value
Pt	75.54 g/t
Pd	18.03 g/t
Rh	8.26 g/t
Au	1.43 g/t
Total 4E leachate grade	104.26 g/t
Aggregate 4E leaching efficiency	41.35%

The combined action of the lixiviants is interpreted cautiously. Literature indicates that thiosulphate can form

stable complexes with precious metals and that Cu (II) can participate in oxidation–reduction cycling [1], [5]. Thiourea can provide rapid complexation under acidic conditions but is susceptible to oxidative degradation, while thiocyanate can form stable complexes with Pt and Pd [1]. In the present study, these chemical roles are proposed explanations rather than directly demonstrated mechanisms because pH, redox potential, solution speciation, and reagent degradation products were not monitored.

Response Surface Methodology identified interaction effects among the three lixiviant components. The model predicted a theoretical optimum aggregate 4E dissolution efficiency of 92.4%; however, this optimum was not independently validated experimentally. The 92.4% value is therefore treated as a model-derived prediction and not as an achieved recovery. This distinction is important for process development because statistical fit within an experimental domain does not substitute for validation at the predicted optimum.

C. Adsorption and Nano-Adsorbent Performance

The potato-peel-derived CNT adsorbent showed substantially stronger aggregate PGM removal than the activated-carbon intermediate in dosage tests. CNT removal reached 99.43% at an adsorbent dosage of 0.6 g/L, leaving a reported residual PGM concentration of approximately 0.59 ppm. At higher dosages, removal declined slightly, consistent with possible particle aggregation and reduced effective surface accessibility.

Response-surface analysis indicated a favorable operating region around pH 5–6, 60 min contact time, approximately 0.6 g/L adsorbent, and 35 °C. These values should be regarded as an optimisation region from the model and experimental trends rather than as a universally optimal condition for all thiosystem liquors, because liquor composition and metal speciation can change adsorption behaviour.

TABLE IV: Non-Linear Adsorption Kinetic Parameters for CNTs

Kinetic model	**k**	**q_e, exp (mg/g)**	**q_e, calc (mg/g)**	**R^2**
PFO, non-linear	0.0415769	172.783	168.321	0.99514
PSO, non-linear	0.000238373	172.783	195.902	0.98683

The non-linear PFO model provided the higher coefficient of determination (R^2 = 0.99514) and a calculated equilibrium capacity closer to the experimental value than the PSO model. This indicates that the observed aggregate adsorption kinetics were more consistently represented by the PFO formulation under the tested conditions. The result is interpreted as a kinetic description rather than proof of a specific molecular adsorption mechanism, consistent with the caution recommended for empirical adsorption models [8].

D. Regeneration and Reusability

Pristine CNTs showed an aggregate PGM sorption level of 51 ± 2%. After five sorption cycles followed by thermal regeneration, the sorption level remained 52 ± 4%. This indicates that the adsorbent retained comparable sorption performance after repeated use. Thermal regeneration alone did not completely desorb the PGMs; subsequent 3 M HNO_3 treatment produced high PGM concentrations in the first desorption fractions, demonstrating the importance of chemical regeneration for restoring the adsorbent.

TABLE V: CNT Regeneration Performance

CNT condition	**PGM sorption**	**First 3 M HNO_3 fraction (µg/L)**	**Third fraction (µg/L)**
Pristine	51 ± 2%	7648.6	80.0
5 cycles + thermal regeneration	52 ± 4%	9025.7	239.4

The regeneration behaviour is consistent with the broader literature identifying adsorption as a promising PGM separation route while noting that regeneration, selectivity, and performance in realistic multicomponent liquors remain important development challenges [2], [3].

E. Economic and Sustainability Implications

A preliminary Monte Carlo economic assessment was used to examine uncertainty in the integrated process. The thiosulfate-based leaching scenario produced a mean NPV of approximately USD 3.8 million compared with approximately USD 2.6 million for the conventional chloride/cyanide comparison scenario. For the downstream adsorption stage, the nano-adsorbent system produced a mean NPV of approximately USD 1.45 million compared with USD 0.92 million for activated carbon.

These results suggest potential economic benefits from combining lower-toxicity lixiviant chemistry with waste-derived nanomaterials. The economic advantage is also linked to valorisation of potato peel waste, reduced dependence on conventional activated carbon, and the potential for adsorbent reuse. However, the economic values are preliminary and depend on laboratory-scale assumptions, recovery inputs, reagent prices, capital factors, and regeneration performance. They should therefore be viewed as scenario-based indicators rather than a bankable feasibility assessment.

The sustainability value of the integrated route lies in process integration: flotation reduces the mass entering leaching; the thiosystem avoids cyanide; potato peel waste is converted into a functional carbon material; and regeneration offers an opportunity for adsorbent reuse. Recent work on sustainable PGM recovery similarly emphasises process integration, green solvents, waste valorisation and improved separation as important components of circular PGM supply chains [3], [4].

F. Integrated Process Significance

The proposed flowsheet links four complementary functions: concentration of PGMs by flotation, dissolution using a modified thiosystem, capture of dissolved PGMs by a bio-derived CNT adsorbent, and regeneration of the adsorbent. The results show that the individual stages are technically compatible at laboratory scale. Importantly, the process should not yet be presented as a fully validated industrial flowsheet. The leaching stage requires independent validation of the RSM-

predicted optimum, and future work should establish metal-specific Pt, Pd, Rh and Au mass balances across the feed, leachate, residue and adsorption products.

Further process development should also include continuous monitoring of pH and redox potential during leaching, direct measurement of lixiviant degradation, evaluation of competing base metals, longer-term regeneration testing, and techno-economic assessment at a realistic pilot scale. These measurements would strengthen the mechanistic basis and determine whether the sustainability advantages observed at laboratory scale persist during scale-up.

IV. Conclusion

An integrated hydrometallurgical route was developed for recovery of PGMs from an oxidised Great Dyke flotation concentrate. Flotation upgraded the feed to 50.96 g/t 4E PGMs, an enrichment factor of approximately 21.5. Modified thiosystem leaching produced a maximum experimentally observed aggregate 4E leaching efficiency of 41.35% for Mix 12. The RSM model predicted a higher theoretical optimum of 92.4%, but this prediction was not experimentally validated and therefore requires further confirmation.

The downstream potato-peel-derived CNT adsorbent achieved 99.43% aggregate PGM removal at 0.6 g/L in dosage tests, and the PFO kinetic model provided the better non-linear fit (R^2 = 0.99514). After five sorption cycles and thermal regeneration, the sorption level remained 52 ± 4%, indicating promising reusability. Economic modelling indicated higher mean NPV for the nano-adsorbent route than activated carbon.

Overall, the study demonstrates the potential of combining non-cyanide thiosystem leaching with a waste-derived nano-adsorbent for greener PGM recovery. The principal next steps are experimental validation of the predicted leaching optimum, metal-specific recovery and mass-balance measurements, controlled pH/redox studies, and pilot-scale techno-economic and environmental assessment.

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