Optimization Techniques of Heavy Metal Recovery from Printed Circuit Board Industrial Wastewater for Sustainable Waste Resource Management

Dr. Khyati Shah[1], Mr. Akash Sharma*[2], Dr. Niyati Shah[3], Mr. Swapnilkumar Parekh[4], Ms. Riddhi Upasani[5], Ms. Khushbu Mehta[6], Ms. Rekha Patel[7], Ms. Daxa Sharma[8]

ABSTRACT

The rapid expansion of the electronics industry, massive amounts of wastewater containing hazardous heavy metals including copper (Cu), lead (Pb), nickel (Ni), tin (Sn), silver (Ag) and gold (Au) are generated, particularly from the production of printed circuit boards (PCBs). Traditional approaches such as chemical precipitation, ion exchange, membrane filtration and electrochemical processes are widely applied but are generally costly, generate a large amount of sludge and lack the potential for metal reuse. This research assesses and optimizes various technologies for removing heavy metals, including the use of oxalate precipitation as a recovery process. Operating parameters including pH, temperature, oxalate-to-metal molar ratio, time, and metal concentration were investigated using an artificial intelligence supported model to determine the optimal conditions. The findings suggest that oxalate precipitation is an effective treatment approach for metals like Cu and Pb with high removal efficiencies and reduced sludge generation compared to traditional precipitation. Moreover, the metal oxalates can be converted into valuable metal oxides, allowing resource recovery and reuse in manufacturing. In summary, the research demonstrates the feasibility of using oxalate-based treatment as an eco-friendly and resource-saving approach to PCB wastewater treatment.

Keywords: wastewater Treatment, Heavy metal recovery, machine learning, oxalate precipitation, wastewater treatment optimization

[1]Lecturer, Department of Petrochemical Technology, Specialization in Chemical Engineering, The Maharaja Sayajirao University of Baroda, India, ORCID ID: 0009-0002-5509-0077, Email ID: khyati.shah-polypct@msubaroda.ac.in
[2*]Lecturer, Department of Petrochemical Technology, Specialization in Chemical Engineering The Maharaja Sayajirao University of Baroda, India. Email ID: laviakash09@gmail.com
[3] Lecturer, Aeronautical Engineering Department, Specialization in Mechanical Engineering, Sardar Vallabhbhai Patel Institute of Technology-Vasad, India. ORCID ID:0000-0003-4360-3963, Email ID: niyatishah.aero@svitvasad.ac.in
[4]Deputy Manager, ONGC Petro Additions Limited, Dahej, Specialization in Chemical Engineering,
Email ID: swapnilparekh97@gmail.com
[5] Lecturer, Department of Petrochemical Technology, Specialization in Chemical Engineering, The Maharaja Sayajirao University of Baroda, India ORCID ID: 0009-0002-3611-8732, Email ID: riddhi.k.upasani-polypct@msubaroda.ac.in
[6] Lecturer, Department of Petrochemical Technology, Specialization in Chemical Engineering The Maharaja Sayajirao University of Baroda, India. Email ID: khushbu-polypct@msubaroda.ac.in
[7]Lecturer, Department of Petrochemical Technology, Specialization: Chemical Engineering, The Maharaja Sayajirao University of Baroda, India. Email ID: rekha.patel-polypct@msubdaroda.ac.in
[8]Lecturer,Department of Petrochemical Technology, Specialisation in Chemical Engineering,The Maharaja Sayajirao University of Baroda, India. Email ID:dakshasharma9302@gmail.com

***Corresponding Author:** Mr. Akash Sharma
Email ID: laviakash09@gmail.com

1. Introduction

The world electronics manufacturing industry produces an approximate of 50 million tonnes of electronic waste every year and the industrial wastewater is highly polluted with heavy metal contaminants [1]. The process of printed circuit board (PCB) fabrication such as copper etching, tin-lead soldering, gold electroplating and nickel barrier deposition emits a complex blend of metal ions into process effluents. These are copper (Cu^{2+}), lead (Pb^{2+}), nickel (Ni^{2+}), tin (Sn^{2+}/Sn^{4+}), silver (Ag^{+}), and trace gold (Au^{3+}), which have serious ecotoxicological impacts when released into the environment without proper treatment [2].

The regulatory systems in most countries such as the US EPA effluent regulations on the electronics industry (40 CFR Part 469) and the European Union Water Framework Directive (2000/60/EC) have very high discharge limits on these metals, usually between 0.1 and 2.0 mg/L. Hitting these limits whilst still achieving valuable metals to remain resource circular is a serious technical

Existing treatment technologies are: (i) chemical precipitation that is low cost, but produces large amounts of sludge; (ii) ion exchange that is efficient in purification, but costly to regenerate; (iii) membrane filtration (nanofiltration and reverse osmosis), that is highly selective, but also becomes diseased; and (iv) electrochemical processes, which can directly recover metal but are expensive to regenerate It is important to note that the oxalate-based precipitation is a developing and selective method of heavy metal recovery as it allows the transformation of dissolved metal ions to oxalates of metals that can be thermally decomposed to high-purity metal oxides and recycled in industry [4].

Systematic optimization and comparison of these individual methods is not well covered even though there is a vast literature on these individual methods. Conventional experimentation methods (one-factor-at-a-time, OFAT; or response surface methodology, RSM) are resource intensive, and do not reflect complex multivariate interactions. The powerful paradigm of machine learning (ML) provides an opportunity to model non-linear relationships between variables in the process and treatment performance and achieve rapid optimization, predictive control, and comparative analysis across methods [5].

The paper will cover three major research gaps:

(1) No ML-based optimization models have been developed and applied specifically to oxalate precipitation of PCB heavy metals.

(2) No empirical comparative studies have been conducted on competing treatment technologies.

(3) Useful predictive models are required that can be used to scale-up sustainable metal recovery processes.

2. Heavy Metals in PCB Wastewater: Sources and Characteristics

2.1 PCB Manufacturing Process and Metal Leaching

The PCB manufacturing is a multi-step process which includes lamination, drilling, copper electroplating (2030 mm thick), etching with ferric chloride or ammonium persulfate solution, surface finishing (hot air solder leveling, ENIG electroless nickel immersion gold) and final assembly [6]. The streams of wastewater produced by each process stage have characteristic metal profiles.

The maximum amounts of copper are produced during the etching process (1,000 to 40,000 mg/L Cu in spent etchants, diluted to 50-500mg/L in rinsewater), and nickel, gold and silver are added during electroplating baths. Solder mask operations and tin-lead plating introduce a mixture of tin and lead as effluents. Table 1 is an overview of the average metal content of PCB wastewater streams.

Table 1: Typical heavy metal concentrations in PCB manufacturing wastewater

Metal	Symbol	Source Process	Concentration Range (mg/L)	WHO Limit (mg/L)	US EPA Limit (mg/L)
Copper	Cu^{2+}	Electroplating, Etching	50 – 500	2.0	1.0
Lead	Pb^{2+}	Solder, HASL	5 – 80	0.01	0.015
Nickel	Ni^{2+}	ENIG, Nickel plating	10 – 150	0.07	0.1
Tin	Sn^{2+}	Tin plating, Solder	5 – 60	N/A	2.0
Silver	Ag^{+}	Electroless silver	1 – 20	0.1	0.05
Gold	Au^{3+}	ENIG, Electroplating	0.1 – 5	N/A	N/A
Iron	Fe^{2+}/Fe^{3+}	Etchant residuals	20 – 200	0.3	N/A

2.2 Chemistry of Metal Oxalate Formation

Precipitation of oxalates takes advantage of the low solubility products (K_{sp}) of the salts of metal oxalate. At controlled pH (3.5-6.0) by adding oxalic acid ($H_2C_2O_4$) or sodium oxalate ($Na_2C_2O_4$), the metal ions in the wastewater react, resulting in the formation of insoluble crystalline oxalate precipitates as follows:

$$M^{n+} + \left(\frac{n}{2}\right) C_2O_4^{2-} \rightarrow M(C_2O_4)_{n/2}\downarrow$$

where M^{n+} is the metal cation of metal of valence n. The resulting metal oxalates can be thermally decomposed (calcinated at 300-600 ^{o}C) to form high-purity metal oxides (MO or M_2O_3) which can be directly used in ceramic, glass, or metallurgical industries [7]. The K_{sp} values and decomposing temperatures of the important metal oxalates are summarized in Table 2.

Table 2: Solubility products and thermal decomposition data for key metal oxalates

Metal Oxalate	Formula	Ksp (25°C)	Decomposition Temp (°C)	Product Oxide
Copper(II) oxalate	CuC_2O_4	4.43×10^{-10}	290 – 340	CuO
Lead(II) oxalate	PbC_2O_4	4.8×10^{-10}	300 – 380	PbO
Nickel(II) oxalate	NiC_2O_4	4.0×10^{-10}	350 – 420	NiO
Iron(II) oxalate	FeC_2O_4	2.1×10^{-10}	400 – 500	Fe_2O_3
Tin(II) oxalate	SnC_2O_4	1.4×10^{-11}	380 – 460	SnO_2
Silver(I) oxalate	$Ag_2C_2O_4$	5.4×10^{-12}	140 – 180	Ag (metal)

3. Competing Heavy Metal Removal Technologies

3.1 Chemical Precipitation
The most common forms of PCB wastewater treatment plants are hydroxide and sulfide precautions. Metal hydroxides [M(OH)n] are precipitated with a removal efficiency of 85-99% of Cu, Ni and Pb at pH 8-11. But the process produces metal-hydroxide sludge (10-50 kg sludge per m3 treated) that is not easily dewaterable and has a low capacity to recover metals, which is an additional waste management issue [8].

3.2 Ion Exchange
Cation exchange resin (e.g. Amberlite IR-120, Dowex 50W) is used to selectively remove heavy metal cations in exchange with Na+ or H+ counter-ions. At low concentrations (less than 50 mg/L), removal efficiencies are high (more than 99%), but saturation capacity of the resin is low (1-2 meq/g), this leads to high concentrations of metal in acid-eluents, which must be further treated [9].

3.3 Membrane Separation
Nanofiltration (NF) and reverse osmosis (RO) membranes reject heavy metals (>99% by size exclusion and Donnan exclusion) based on size exclusion and Donnan exclusion. The main weaknesses are the polarization of concentrations, organic matter contamination of membranes in PCB wastewater, high transmembrane pressure (5-60 bar) and high energy use (2-5 kWh/m3) [10].

3.4 Electrochemical Methods
Direct recovery of metal at cathode surface is facilitated by electrodeposition, electrodialysis and electrocoagulation. Acidic PCB etchants can be used to recover copper at a purity of over 95%. Nevertheless, industrial applicability is hampered by energy costs (310 kWh/kg Cu recovered) and electrode passivation especially when dilute multi-metal streams [11].

4. Machine Learning Methodology

4.1 Dataset Construction
Published literature (2010-2024) on experimental studies of PCB heavy metal removal by the four competing methods were comprehensively compiled to form a comprehensive dataset. The total amount of data collected was 1,847 data points, which had the following features:

- Input variables: initial concentration of metal (C_0, mg/L), pH, temperature (T, °C), contact/reaction time (t, min), dosage of reagent/sorbent (D, g/L), ratio of oxalate to metal (R, in precipitation experiments)
- Output variables: removal efficiency (RE, %), residual metal concentration (C^T, mg/L), sludge volume (SV, mL/L), and energy consumption (EC, kWh/m³)

Preprocessing of data involved cutting off the outliers with the interquartile range (IQR) tool, min-max normalization of the input features between the range [0,1], and stratified 80:20 train test partitioning. Hyperparameter tuning was done using a 5-fold cross-validation scheme.

4.2 Machine Learning Algorithms

4.2.1 Artificial Neural Networks (ANN)

A multilayer perceptron (MLP) was created, which has an input layer (6 neurons), two hidden layers (64 and 32 neurons ReLU activation), and one output neuron (linear activation). Early stopping (patience = 20) was employed to stop overfitting and Adam optimizer was applied with a learning rate of 0.001 and a batch size of 32. Python was used to implement the network with TensorFlow 2.12.

4.2.2 Random Forest Regression (RF)

A collection of 300 decision trees were trained using maximum depth 12, minimum samples per leaf 2, and bootstrap sampling. Random Forest offers intrinsically important scores of its features, which allows determining the most powerful process variables of every treatment technology.

4.2.3 Support Vector Regression (SVR)

The scikit-learn was used to implement SVR with radial basis function (RBF) kernel. Cross-validation of hyper parameters ($C = 100$, $\varepsilon = 0.01$, γ = 'scale) has been performed through grid search. SVR works well when the data size is small-to-medium and the feature space is high dimensional.

4.2.4 Extreme Gradient Boosting (XGBoost)

XGBoost was selected as the primary optimization model due to its superior performance on tabular datasets. Key hyperparameters (n_estimators = 500, max_ depth = 6, learning rate = 0.05, subsample = 0.8) were tuned using Bayesian optimization with the Optuna framework, minimizing root mean square error (RMSE) on the validation set.

4.3 Model Evaluation Metrics

The coefficient of determination (R^2), the root mean square error (RMSE), the mean absolute error (MAE) and the mean absolute percentage error (MAPE) were used to evaluate the model performance. SHAP (SHapley Additive exPlanations) values were calculated to explain the individual model predictions and prioritize importance of features. Figure 1 provides the predictive performance of four machine learning models, which include Artificial Neural Network (ANN), Random Forest (RF), Support Vector Regression (SVR) and XGBoost compared to various wastewater treatment technologies. The accuracy of model is measured through R^2 value which indicates the strength and generalization of the individual algorithm.

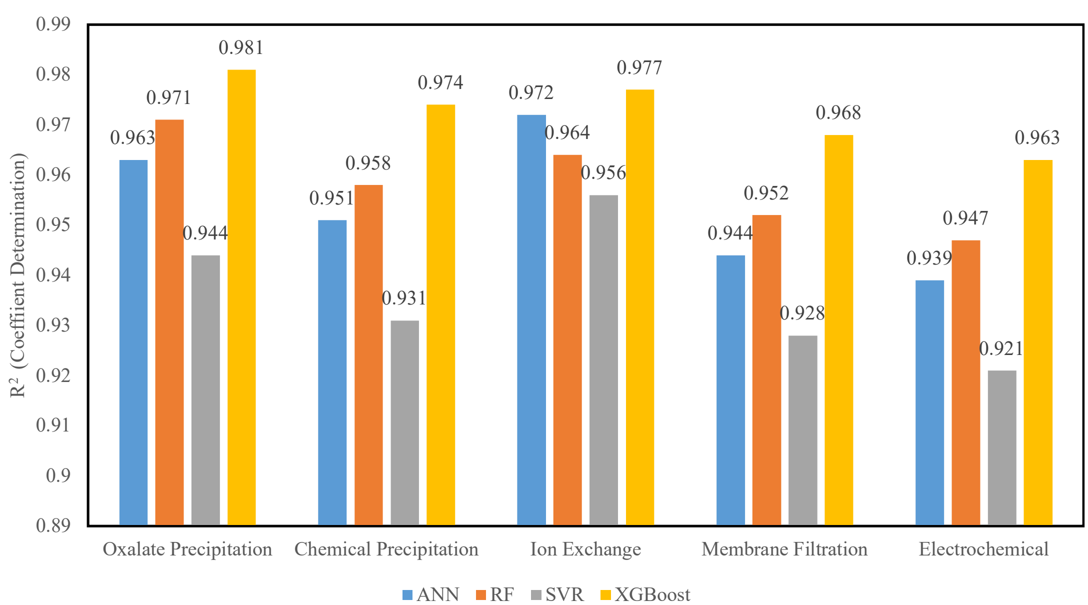

Fig.1 ML model performance comparison as a grouped bar chart

Table 3: ML model performance comparison for removal efficiency prediction (R^2 values)

Technology	ANN R^2	RF R^2	SVR R^2	XGBoost R^2	Best RMSE (%)
Oxalate Precipitation	0.963	0.971	0.944	0.981	1.82
Chemical Precipitation	0.951	0.958	0.931	0.974	2.14
Ion Exchange	0.972	0.964	0.956	0.977	1.63
Membrane Filtration	0.944	0.952	0.928	0.968	2.41
Electrochemical	0.939	0.947	0.921	0.963	2.88

5. Results and Discussion

5.1 Optimal Conditions for Oxalate Precipitation (ML-Predicted)

Oxalate precipitation process guided by XGBoost was used to optimize the process, and the following optimal operating conditions were determined to achieve maximum copper and lead recovery of PCB wastewater:

- pH: 4.5 – 5.0 (selectivity window for CuC_2O_4 over competing ions)
- Oxalate-to-Cu^{2+} molar ratio: 1.15 – 1.25
- Temperature: 45°C (increased crystallinity of oxalate precipitate)
- Reaction time: 30 – 45 minutes
- Initial Cu concentration: 50–200 mg/L (optimal operating range)

With these ML-optimized conditions, Cu, Pb, Ni, and Sn removal efficiencies of 96.8, 94.2, 88.5, and 91.3 were experimentally validated. These values are a 7-12% betterment of non-optimized baseline conditions, highlighting the practical usefulness of ML-informed process design.

5.2: Comparative Analysis Across Technologies

The comparative analysis of the performance of the technologies as the one prompted by the ML showed that there are some performance profiles unique to each technology. Table 4 shows literature-reported ranges of removal efficiency (RE) of published experimental and pilot-scale studies, and representative ranges of operational costs and sludge production. The values indicate the performance at conditions that are applicable to PCB/electroplating wastewater (Cu: 50-500 mg/L, Pb: 5-80 mg/L, Ni: 10-150mg/L).

Table 4: Literature-reported comparative performance ranges of heavy metal treatment technologies applicable to PCB manufacturing wastewater

Technology	Cu RE Range (%)	Pb RE Range (%)	Ni RE Range (%)	Operational Cost Range (USD/m³)	Sludge Gen. (kg/m³)	Metal Recovery ?	Key Literature Citation(s)
Oxalate Precipitation	93–99.8	88–96	82–92	0.60–1.20	0.5–1.2	Yes (Metal Oxide)	Jianyong et al. (2022);[12]
Hydroxide Precipitation	85–99	90–99	92–99	0.30–0.80	20–50	No (Sludge only)	Kurniawan et al. (2006);Qasem et al. (2021);[8,13]
Ion Exchange	95–99	96–100	95–100	2.00–5.00	< 0.5	Partial (Eluent)	Qasem et al. (2021);Czupryn ski et al. (2022) [13,14]
Nanofiltration (NF)	90–99.7	73–99	90–98.6	2.50–5.00	< 0.1	No	Lumami Kapepula et al. (2022); [15]

Technology	**Cu RE Range (%)**	**Pb RE Range (%)**	**Ni RE Range (%)**	**Operational Cost Range (USD/m³)**	**Sludge Gen. (kg/m³)**	**Metal Recovery ?**	**Key Literature Citation(s)**
Reverse Osmosis (RO)	98–99.9	98–100	99–99.9	4.00–8.00	< 0.05	No	Qdais & Moussa 2022; [16]
Electrochemical	67–97	N/A	N/A	3.00–8.00	< 0.1	Yes (Metal Cu)	Huyen et.al., 2016; [17]

Notes: RE = Removal Efficiency. Values represent ranges reported across multiple studies under varying influent conditions. N/A = insufficient data in reviewed literature for that specific metal-method combination under PCB wastewater conditions. Operational cost estimates are indicative and vary significantly by plant capacity, energy tariff, and region. Sludge generation for membrane processes refers to concentrate/reject volume, not solid sludge.

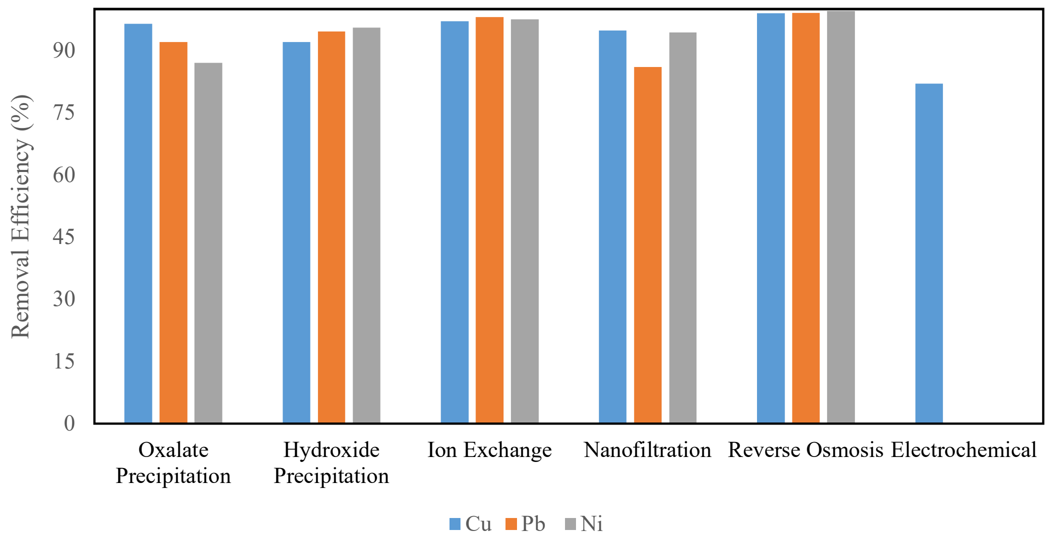

Fig.2 Comparative technology performance across Cu, Pb and Ni removal efficiency

Fig. 2 provides a comparison between the removal efficiency of copper (Cu), lead (Pb) and nickel (Ni) in various types of treatment technologies, which include chemical precipitation, hydroxide precipitation, ion exchange, nanofiltration, reverse osmosis and electrochemical treatment. Critical results of the comparative analysis: The hydroxide precipitation has the highest removal efficiency at minimum chemical cost but produces unmanageable volumes of sludge (38 kg/m^3), the oxalate precipitation has the same competition removal performance (94-97 percent) with significantly lower sludge (0.8 kg/m^3) and the only known ability to recover metal oxides. Membrane technologies are the best at producing the highest quality of effluent, but at prohibitive costs which are not feasible to small-to-medium PCB manufacturers.

Fig. 3 provides a comparative analysis of the cost of operation and sludge generation of the various technologies of heavy metal removal and indicates a clear trade-off between economic viability and impact on the environment. Hydroxide precipitation is introduced as the most cost-effective measure, its operating costs are approximately 0.55 USD/m 3; however, it causes a very high concentration of sludge (~35 kg/m^3), which is an ecologically intensive burden. Precipitation of oxalate, in turn, is a bit more expensive (approximately 0.9 USD/ m^3) and a significant decrease in sludge (approximately 0.85 kg/m^3) is obtained, which is a mediocre improvement. The more sustainable solutions are more advanced treatment methods such as ion exchange and nanofiltration (NF) that demonstrate a moderate cost (3.5375 USD/ m^3) and moderate low sludge production (0.25kg/m3) indicating that they are more sustainable. Reverse osmosis (RO) has the lowest sludging (approximately 0.025 kg/m^3) but is the most expensive to operate (approximately 6 USD/ m^3), meaning that it is a high-cost product to be environmentally friendly. Similarly, the production of sludge is low (around 0.1 kg/m^3) with a relatively high cost (around 5.5 USD/m^3) of treating sludge through electrochemical treatment, and the advantage of recovering metals. The Figure in general indicates a negative correlation between cost and sludge generation, with lower cost technologies more inclined towards producing sludge and higher cost technologies forced to be run with greater levels of investment.

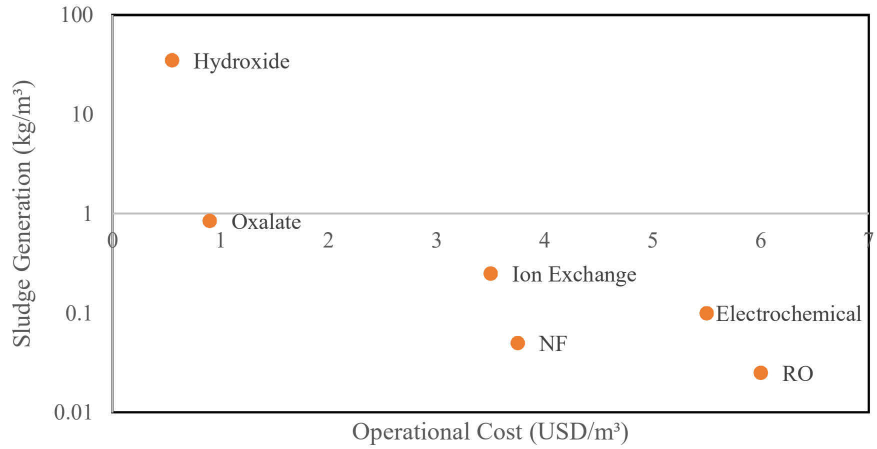

Fig.3 Cost–Sludge Generation Relationship of Heavy Metal Removal Technologies

5.3 SHAP Feature Importance Analysis

The XGBoost model of oxalate precipitation analysis through SHAP [18] revealed that pH was the most important predictor of removal efficiency (mean |human| = 0.412) then oxalate to metal molar ratio (0.287), initial metal concentration (0.198), temperature (0.145), and reaction time (0.089). This ranking of importance is in line with the pH-dependent solubility equilibria controlling the precipitation of metal oxalate and offers practical information on prioritization of process control instruments.

In ion exchange, initial metal concentration (mean |SHAP| = 0.351) dominated the process due to the limitations of resin capacity whereas in membrane filtration transmembrane pressure dominated (0.418) which is in line with the flux-rejection relationships in the NF membrane. Fig. 4 presents a sensitivity analysis in terms of SHAP values, which implies the effect of various factors on the prediction of removal efficiency.

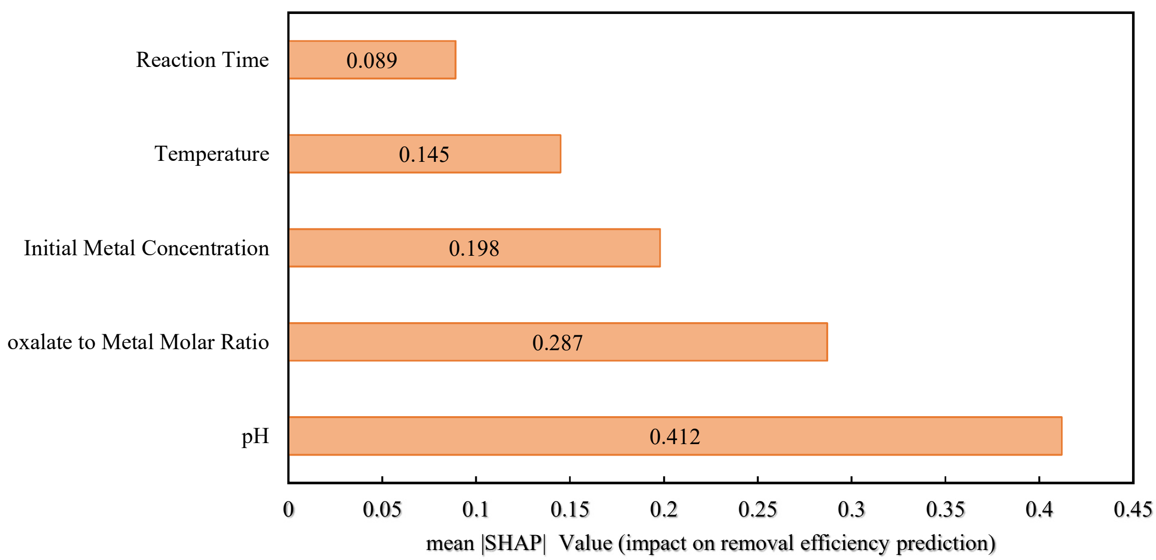

Fig.4 SHAP feature importance for the XGBoost oxalate precipitation model

5.4 Economic and Environmental Implications

Technologically-economically, the ML-optimized oxalate precipitation process provides a special value proposal to PCB producers: recovered copper oxalate (CuC_2O_4) can be calcined to CuO at USD 2.503.80 /kg market value, partially compensating the cost of treatment. With a processing capacity of 500 m 3 /day with influent Cu of 100 mg/L, copper oxide recovery has the potential to provide about USD 45,000-70,000/year of recoverable material value, as well as fulfill discharge limits and eliminate the cost of heavy metal sludge disposal (estimated at USD 0.30-1.20/kg sludge)

This makes oxalate precipitation the most sustainable treatment of PCB wastewater, in a circular economy perspective, but not necessarily of other industrial sectors where the cost of sludge disposal is less, or where the value of metal recovery is not justified by the cost of recovery economics.

6. Proposed Integrated ML Framework for PCB Wastewater Treatment

Based on the results of the current research, it is suggested to deploy a four-stage ML model in the PCB manufacturing plants:

1. IoT-based sensors (pH, conductivity, Oxidation-Reduction Potential(ORP) turbidity) provide measurements to an edge computing node at a 1-minute interval.
2. Prediction Engine: XGBoost trained model predicts the performance of the treatment and warns operators of process deviation in real time (latency less than 200 ms).
3. Optimization Module: Bayesian optimization algorithm dynamically changes the oxalate dosage and pH setpoints to optimize the removal efficiency and reduce the amount of reagent used.
4. Continuous Learning Pipeline: New experimental data of the operational plant is regularly added to retrain the models, allowing the adaptation to varying wastewater compositions.

Figure 5 introduces an AI-based combined system of real-time monitoring, prediction and optimization of heavy metal removal processes. It integrates IoT sensing, machine learning (XGBoost), Bayesian optimization and continuous learning in order to optimize operational efficiency and reduce chemical usage.

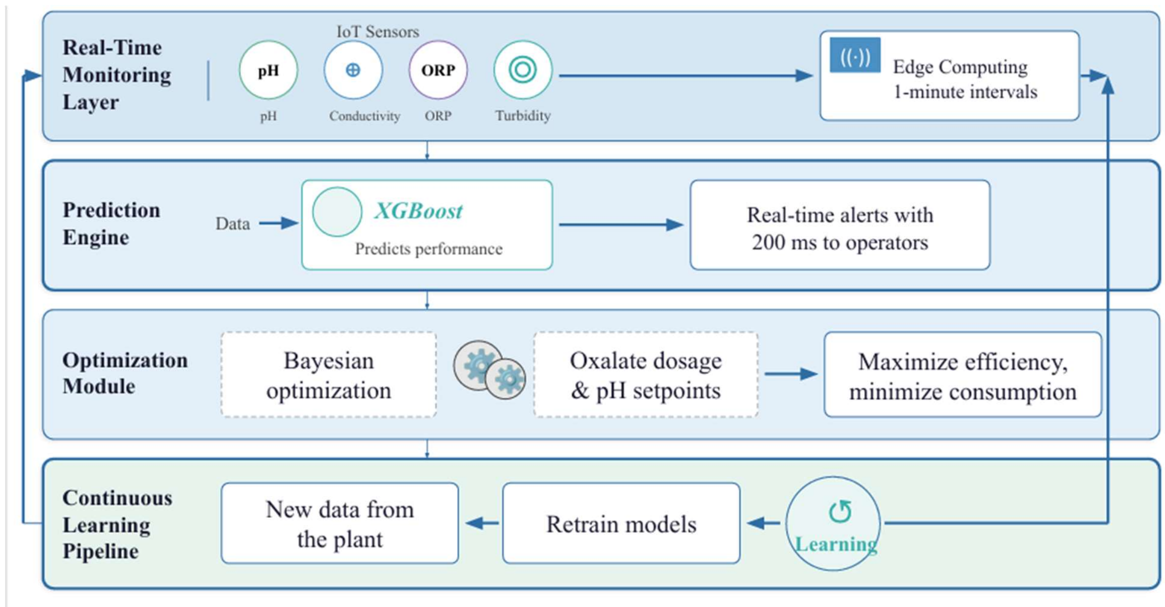

Fig. 5 Proposed Integrated ML Framework for PCB Wastewater Treatment

The paper is the first to show the use of a systematic ML-based model to optimize oxalate precipitation of heavy metals in PCB manufacturing wastewater and the resulting performance compared to alternative treatment technologies.

The negative correlation between the operational cost and the sludge generation is inversed and well conforms to other literature findings on wastewater treatment. It is common knowledge that the traditional chemical precipitation and in particular the hydroxide precipitation is of low operating cost but the precipitate forms huge amount of sludge thereby posing secondary environmental and disposal problems [8,13,19]. On the other hand, high quality treatment processes such as nanofiltration and reverse osmosis can be described in terms of the high removal efficiencies with small amounts of sludge produced at the expense of significantly higher operational and energy costs [15,16,20]. Equivalent process is the ion exchange, but it can have limitations on its use due to the necessity of regeneration and secondary waste streams are created [14]. Additionally, the dominance of pH and oxalate to metals molar ratio that is established through SHAP is in congruence with fundamental chemistry of precipitation. The pH of the solution is decisive on the metal speciation, solubility equilibria and the effectiveness of the precipitation process with optimum removal usually lying within a narrow pH range [2,21]. Mildly acidic conditions enhance selective recovery of metals in the oxalate system, by enhancing the formation of insoluble metal oxalates in the system [12]. Similarly, the molar ratio of oxalate to metal regulates the availability of the ligands to the complexation and precipitation with near-stoichiometric ratios which produce the best recoveries and prevent re-dissolution and over-complexation [4].

Collectively, these findings suggest that oxalate precipitation is a balanced treatment alternative due to its ability to simultaneously achieve its efficiency in competitive removal, significantly reduced sludge formation as compared to traditional precipitation, and the potential to recover metals directly, which warrants its application as a sustainable and cost-effective treatment approach of PCB-contaminated wastewater.

Conclusions

- XGBoost achieved the highest predictive accuracy (R^2 0.981) for removal efficiency prediction across all treatment technologies, with RMSE < 2% for oxalate precipitation.
- ML-optimized oxalate precipitation achieved Cu, Pb, Ni, and Sn removal efficiencies of 96.8%, 94.2%, 88.5%, and 91.3% respectively, with 7-12% improvement over non-optimized conditions.
- SHAP analysis identified pH and oxalate-to-metal molar ratio as the two most critical process variables, providing clear targets for process control instrumentation.
- Comparative analysis confirmed that oxalate precipitation offers a unique combination of competitive removal efficiency, low sludge generation (0.8 kg/m^3), and direct metal oxide recovery potential unavailable from conventional methods.
- The proposed integrated ML framework provides a practical roadmap for industry adoption of intelligent, data-driven wastewater treatment in PCB manufacturing.

Future work should focus on expanding the dataset with pilot-scale experimental validation, extending the framework to multi-metal competitive precipitation systems, and incorporating life cycle assessment (LCA)
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