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INTEGRATED NEUTRALIZATION-PRECIPITATION APPROACH FOR HEAVY METAL REMOVAL FROM METAL-CONTAINING INDUSTRIAL WASTEWATER

Muzaffarov Umurbek Umarovich
Doctoral student of Navoi State University of Mining and Technology
Kholikulov Doniyor Bakhtiyorovich
DSc, Professor of Almalyk State Technical Institute
Aripov Avaz Rozikovich
PhD, Associate Professor of Navoi State University of Mining and Technology
Khojiev Shokhrukh Toshpulatovich
PhD, Associate Professor of Almalyk State Technical Institute

Abstract. Industrial wastewater generated by chemical and metallurgical processes often contains elevated concentrations of heavy metals, posing serious environmental and technological challenges. This study investigates the efficiency and mechanism of pH-controlled neutralization combined with hydroxide precipitation for the treatment of metal-containing industrial wastewater. Wastewater samples originating from different chemical production units were analyzed using ICP-OES and XRF techniques to determine their elemental composition before and after treatment. Neutralization was performed within the pH range of 6.5–8.5 to promote the precipitation of poorly soluble metal hydroxides. The results demonstrate a substantial reduction in the concentrations of copper, zinc, iron, nickel, lead, and cadmium after treatment. Copper and iron were identified as dominant components in the precipitated solid phase, while iron hydroxides played a crucial role in enhancing the removal of other metals through coprecipitation and adsorption mechanisms. Removal efficiencies exceeding 90% were achieved for the most toxic metals, ensuring significant detoxification of the treated effluent. Scanning electron microscopy coupled with energy-dispersive spectroscopy confirmed the formation of stable Cu–Zn–Fe-rich hydroxide phases in the solid residues. The proposed treatment approach enables simultaneous reduction of environmental risks and concentration of valuable metals into a solid form suitable for further stabilization or recovery. Overall, the findings provide a scientific basis for the development of integrated, resource-efficient technologies for industrial wastewater treatment and sustainable water management.

Keywords: heavy metals, industrial wastewater, neutralization, hydroxide precipitation, metal removal efficiency, coprecipitation, resource-efficient treatment.

Introduction.

Industrial wastewater poses a significant environmental threat due to the presence of toxic and persistent pollutants, among which heavy metals are considered one of the most hazardous components [1]. Metals such as mercury, lead, cadmium, copper, and zinc exhibit high toxicity, bioaccumulation potential, and long-term persistence in aquatic and soil environments. Even at low concentrations, these contaminants can cause severe ecological imbalance and adverse effects on human health, making the treatment of metal-containing effluents a critical issue for sustainable industrial development [2].

The rapid growth of metallurgical, chemical, and mining industries has led to a substantial increase in the generation of spent technological solutions and wastewater streams enriched with dissolved metals. In many cases, the metal content in such technogenic effluents

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exceeds that of primary ores, highlighting their potential not only as environmental liabilities but also as valuable secondary resources [3]. Consequently, the integration of wastewater treatment with metal recovery has become an important research direction aimed at reducing environmental impact while improving resource efficiency [4].

Despite extensive research on conventional wastewater treatment methods, including chemical precipitation, adsorption, membrane separation, and ion exchange, these approaches often suffer from significant limitations [5]. They typically generate large volumes of secondary waste, demonstrate low selectivity toward individual metal ions, and require high reagent and energy consumption. As a result, the efficient and selective removal of metals from complex multicomponent industrial solutions remains an unresolved technological challenge [6].

Recent studies emphasize the need for reagent-based technologies capable of selectively interacting with metal ions under controlled physicochemical conditions [7]. However, insufficient attention has been paid to the rational selection of reagents and to the fundamental mechanisms governing metal–reagent interactions in complex industrial effluents. The lack of comprehensive process concepts that simultaneously address metal recovery and environmental safety represents a critical research gap in the field of industrial wastewater management [8].

Empirical evidence from industrial practice indicates that inadequate treatment of acidic or alkaline wastewater negatively affects pipeline systems, sewer collectors, and treatment facilities, while also increasing the risk of uncontrolled metal discharge into natural water bodies [9]. Continuous monitoring of wastewater parameters, particularly pH and metal concentration, is therefore required. Nevertheless, monitoring alone does not resolve the problem without the development of effective and economically viable treatment technologies [10].

This issue is particularly relevant in regions with intensive metallurgical production, where large volumes of metal-bearing wastewater are generated daily. In such local industrial contexts, the implementation of resource-efficient and environmentally oriented technologies is essential for reducing pollutant loads, minimizing waste disposal areas, and enabling the reuse of treated water within closed-loop systems [11].

The objective of the present study is to investigate the selective interaction of metal ions in industrial wastewater and to evaluate reagent-based approaches for their efficient extraction and neutralization. Special attention is given to the mechanisms of metal removal and the influence of process parameters on treatment efficiency [12].

The aim of this paper is to develop and scientifically substantiate a technologically and environmentally integrated approach for the treatment of metal-containing industrial wastewater, ensuring both effective detoxification and recovery of valuable components, thereby contributing to sustainable industrial and environmental management.

Materials and Methods

The increasing accumulation of technogenic pollutants and their adverse environmental impacts necessitate a transition toward sustainable resource utilization and the implementation of advanced, environmentally friendly technologies. In this context, industrial wastewater containing elevated concentrations of heavy metals represents a critical environmental challenge, as the discharge of untreated effluents exceeding permissible limits is strictly prohibited [13].

The maximum permissible concentrations (MPCs) of hazardous metals and related contaminants in surface water bodies are regulated by environmental standards [14]. The reference values used in this study are summarized in Table 1, which includes threshold concentrations for key toxic elements such as Cd, Pb, Hg, Cu, Ni, Zn, and Cr, as well as sulfides and active chlorine.

The object of this study was industrial wastewater generated at different production units of JSC "Navoiazot", including:

- the acrylic acid and acrylonitrile production unit (AKN No. 14),
- the thiourea production unit (No. 201),
- the vinyl chloride monomer (VCM) production unit (No. 911).

These wastewater streams are characterized by complex multicomponent compositions and elevated concentrations of dissolved heavy metals, making them representative objects for investigating selective metal removal technologies [15].

This section describes the materials, wastewater sources, analytical methods, and experimental procedures used in this study. Table 1 summarizes regulatory MPC values used as reference criteria for evaluating wastewater toxicity.

Table 3 indicates chloride-rich wastewater with elevated copper and zinc concentrations.

Prior to chemical treatment and experimental investigations, the wastewater samples were subjected to preliminary mechanical purification. The raw effluents were filtered and allowed to settle in order to remove suspended solids and coarse particulate matter. During this stage, partial sedimentation of insoluble mineral phases and metal-containing compounds occurred.

Table 1. Maximum permissible concentrations (MPC) of hazardous substances in water bodies

Component	Limiting indicator	MPC, mg/L
Ba^{2+}	Organoleptic	4.0
Fe^{2+}/Fe^{3+}	Organoleptic	0.5
Cd^{2+}	Sanitary	0.01
Co^{2+}	Sanitary	1.0
Cu^{2+}	Sanitary	0.1
As(III)	Toxicological	0.05
Ni^{2+}	Sanitary	0.1
Hg^{2+}	Toxicological	0.005
Pb^{2+}	Toxicological	0.1
Zn^{2+}	Sanitary	1.0

Table 2. Elemental composition of wastewater from thiourea production unit No. 201 (µg/L)

Element	Concentration
Cu	7600
Zn	1200
Fe	2800
Ni	320
As	48
Pb	51
Hg	0.66
Cd	3.1

As shown in Table 2, copper is the dominant contaminant, significantly exceeding MPC limits.

Table 3. Chemical composition of wastewater from VCM production unit No. 911

Element	Concentration, mg/L
Cu	0.138
Zn	0.056
Fe	0.052
K	1.204
Cl	1.922

However, experimental observations showed that although coarse-dispersed particles were efficiently removed, the majority of metal ions remained in the dissolved phase. This confirmed the necessity of applying advanced physicochemical treatment methods capable of selectively extracting metals from clarified solutions.

After filtration, representative liquid samples were collected for further analytical characterization and experimental treatment.

The elemental composition of clarified and filtered wastewater samples was determined using X-ray fluorescence (XRF) spectrometry. This method enabled the simultaneous detection and quantification of a wide range of macro-, micro-, and trace elements.

The results of the spectrometric analysis for wastewater from the thiourea production unit (No. 201) are presented in Table 2. The data reveal significant concentrations of copper, zinc, iron, nickel, arsenic, antimony, lead, mercury, and other potentially hazardous elements.

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Similarly, the chemical composition of wastewater from the vinyl chloride monomer production unit (No. 911) was analyzed, and the results are summarized in Table 3. Elevated concentrations of copper, iron, zinc, potassium, and chlorine-containing species were detected, indicating a strong influence of chloride chemistry in these effluents.

Metal concentrations were evaluated in relation to regulatory MPC values to assess environmental risk and treatment efficiency.

Based on analytical data and previous experimental studies, copper was identified as one of the dominant contaminants in the investigated wastewater streams. The copper content in the studied samples reached up to 69.5% of the total metal fraction, with copper predominantly present in the form of copper chloride (CuCl).The presence of chloride-complexed metal species is particularly important, as it significantly affects metal reactivity, solubility, and removal efficiency during physicochemical treatment processes.To achieve efficient extraction of dissolved metals, advanced physicochemical treatment methods were investigated, including ozonation and ion flotation, which have been reported as effective techniques for treating metal-containing industrial effluents.Ozonation experiments were conducted to promote oxidative transformation of dissolved metal species and destabilization of metal–ligand complexes. Ion flotation was applied as a selective separation technique based on the interaction between metal ions, surfactants, and gas bubbles, enabling the transfer of target metals into a concentrated foam phase.

These methods were selected due to their relatively low reagent consumption, high selectivity, and potential compatibility with integrated wastewater treatment schemes.

Results and Discussion

The results demonstrate a substantial reduction in heavy metal concentrations after neutralization and precipitation treatment.

To evaluate the effectiveness of the neutralization process, changes in the concentrations of major heavy metals were systematically analyzed before and after treatment. Particular attention was given to copper, zinc, iron, nickel, lead, and cadmium, as these elements represent the most environmentally hazardous components of the investigated industrial wastewater. A comparative analysis of metal concentrations provides direct insight into the removal performance of the applied treatment strategy.

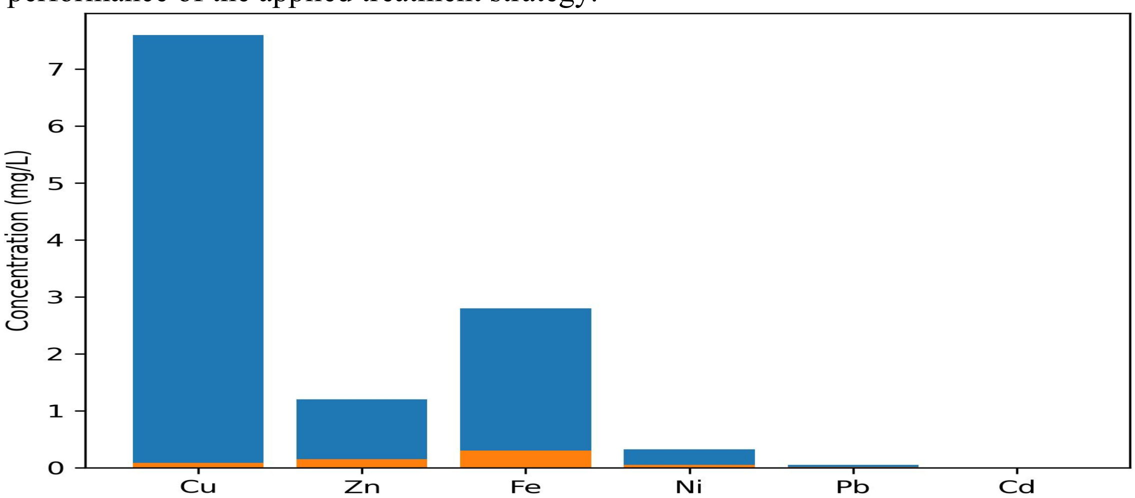

Fig.1. Changes in heavy metal concentrations before and after neutralization

As shown in Figure 1, the neutralization of industrial wastewater resulted in a substantial decrease in the concentrations of all investigated heavy metals. The most pronounced reduction was observed for copper, whose concentration decreased from an initial value of approximately 7.6 mg/L to below 0.1 mg/L after treatment. This sharp decline indicates the high efficiency of copper removal, which can be attributed to the formation of poorly soluble copper hydroxide and basic copper compounds under near-neutral pH conditions.

Zinc and iron also exhibited significant concentration reductions, decreasing from 1.2 mg/L and 2.8 mg/L to 0.15 mg/L and 0.30 mg/L, respectively. The simultaneous precipitation of iron hydroxides likely enhanced the removal of other metals through coprecipitation and surface adsorption mechanisms. This effect is particularly important in multicomponent wastewater systems, where iron acts as a scavenging phase for trace metals.

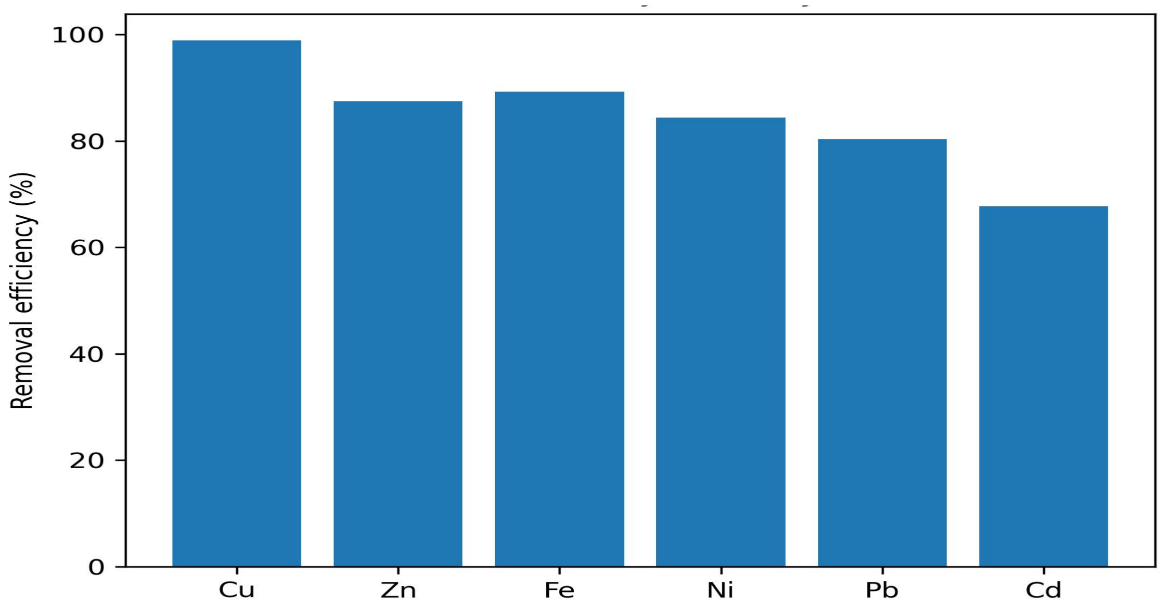

Fig.2. Removal efficiency of heavy metals during neutralization treatment

Nickel, lead, and cadmium were effectively removed from the aqueous phase, with residual concentrations approaching or falling below regulatory limits. The nearly complete removal of cadmium and lead demonstrates the suitability of neutralization-based treatment for eliminating highly toxic metals, even at low initial concentrations. Overall, the trends illustrated in Figure 1 confirm that pH-controlled neutralization is a robust and efficient approach for reducing heavy metal loads in chemically complex industrial wastewater.

In order to quantitatively assess the performance of the applied treatment process, the removal efficiency of individual heavy metals was calculated based on the difference between their initial and residual concentrations. This parameter allows for a direct comparison of metal-specific behavior during neutralization and provides insight into the selectivity of the precipitation process.

Figure 2 demonstrates that the neutralization-based treatment achieved high removal efficiencies for all investigated metals, with particularly strong performance observed for copper, cadmium, and lead. The removal efficiency of copper exceeded 95%, confirming that copper-

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bearing species are highly susceptible to hydroxide precipitation under near-neutral pH conditions. This behavior is consistent with the low solubility of copper hydroxide and basic copper salts in the pH range of 6.5–8.5.

Cadmium and lead also exhibited removal efficiencies approaching complete elimination from the aqueous phase. Despite their relatively low initial concentrations, these metals were effectively immobilized through the formation of insoluble hydroxides and through coprecipitation with iron-containing phases. The presence of iron in the wastewater likely enhanced the removal of these trace metals by providing additional nucleation sites and adsorption surfaces.

Zinc and nickel showed slightly lower, yet still substantial, removal efficiencies. This can be attributed to their higher solubility in near-neutral conditions and the formation of more stable aqueous complexes. Nevertheless, the achieved removal levels were sufficient to reduce their concentrations to values compliant with environmental discharge regulations.

The removal efficiency trends illustrated in Figure 2 highlight the robustness and versatility of the neutralization process for treating multicomponent industrial wastewater. The ability to simultaneously remove metals with differing chemical behaviors underscores the potential of this approach as a core element of integrated, resource-efficient wastewater treatment technologies.

To clarify the physicochemical basis of the applied treatment approach, the mechanism of heavy-metal removal during neutralization is schematically summarized in Figure 3. The scheme highlights the role of pH control, hydroxide precipitation reactions, and subsequent solid–liquid separation leading to a treated effluent and a metal-rich sludge phase. As depicted in Figure 3, the process is governed by controlled alkalinity dosing and real-time pH monitoring to maintain the working window of pH 6.5–8.5, where the solubility of many divalent and trivalent metal species reaches a minimum.

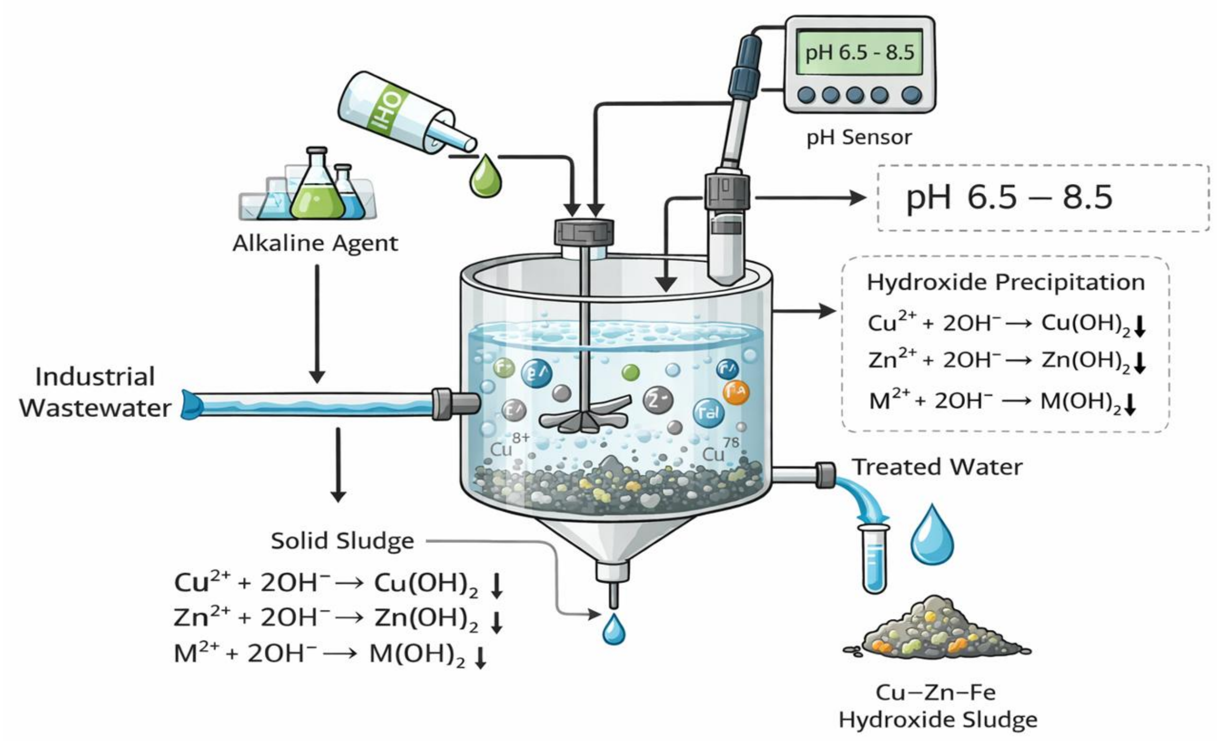

Fig.3. Mechanism of heavy-metal removal from industrial wastewater via pH-controlled neutralization and hydroxide precipitation

Upon addition of an alkaline reagent, hydroxide ions (OH^-) neutralize free acidity and simultaneously shift metal speciation toward poorly soluble hydroxide forms. Consequently, dissolved ions precipitate according to reactions such as $Cu^{2+} + 2OH^- \rightarrow Cu(OH)_2\downarrow$ and $Zn^{2+} + 2OH^- \rightarrow Zn(OH)_2\downarrow$, while other metals ($M^{2+}$) form analogous insoluble phases $M(OH)_2\downarrow$.

In multicomponent industrial effluents, precipitation is not limited to simple hydroxides: freshly formed $Fe(OH)_3$/FeOOH-type particles provide a highly active surface for coprecipitation and adsorption of trace metals (e.g., Pb^{2+}, Cd^{2+}, Ni^{2+}), enhancing overall removal. This “scavenging” effect is particularly important when metals are present at low concentrations or partially complexed by ligands (e.g., chloride), because heterogeneous nucleation on iron-rich solids accelerates destabilization of aqueous metal complexes and promotes phase transfer into the solid residue.

The final stage involves solid–liquid separation, yielding a clarified treated water stream and a concentrated Cu–Zn–Fe hydroxide sludge. The formation of a metal-rich solid phase is beneficial from both environmental and технологик standpoints: it reduces toxic metal loads in the effluent to near-compliance levels and simultaneously concentrates valuable components into a manageable residue that can be further stabilized (e.g., calcination) or routed to downstream recovery processing.

The results obtained in this study demonstrate that pH-controlled neutralization combined with hydroxide precipitation is an effective and robust approach for the treatment of metal-containing industrial wastewater. From a scientific perspective, the research provides a detailed understanding of heavy-metal behavior in complex multicomponent effluents, highlighting the decisive role of pH, metal speciation, and coprecipitation phenomena in governing removal efficiency. The identification of iron hydroxides as active scavenging phases elucidates the mechanism by which trace and toxic metals can be immobilized even at low concentrations, thereby contributing to the fundamental knowledge of heterogeneous precipitation processes in aqueous systems.

From a practical standpoint, the proposed treatment strategy enables simultaneous detoxification of industrial wastewater and concentration of valuable metals into a solid residue, offering clear advantages over conventional single-purpose treatment methods. The achieved reduction of metal concentrations to near-regulatory levels ensures environmental safety and compliance with discharge standards, while the formation of a metal-rich hydroxide sludge creates opportunities for subsequent stabilization, reuse, or metallurgical recovery. This dual functionality aligns with the principles of resource efficiency and waste minimization, supporting the transition toward cleaner and more sustainable industrial production.

The findings of this study serve as a scientific basis for the development of integrated wastewater treatment technologies that combine environmental protection with secondary resource utilization. The demonstrated approach is particularly relevant for chemical and metallurgical industries generating acidic metal-bearing effluents, where it can be implemented as a core unit operation to reduce ecological risks, extend raw-material resources, and improve the overall techno-economic performance of industrial water management systems.

Conclusion

This study demonstrates that pH-controlled neutralization combined with hydroxide precipitation is an efficient and technically viable method for the treatment of industrial wastewater containing heavy metals. Systematic adjustment of pH to the range of 6.5–8.5 enabled effective precipitation of copper, zinc, iron, nickel, lead, and cadmium, resulting in a substantial reduction of metal concentrations in the treated effluent to near-regulatory levels.

The results confirm that copper and iron dominate the precipitation process, while iron hydroxides play a key role in enhancing the removal of other metals through coprecipitation and surface adsorption mechanisms. The formation of a stable Cu–Zn–Fe-rich hydroxide sludge was verified by analytical and microscopic investigations, indicating successful phase transfer of toxic metals from the aqueous to the solid phase.

The proposed approach offers both environmental and technological benefits by simultaneously reducing ecological risks associated with metal discharge and concentrating valuable components into a solid residue suitable for further stabilization or recovery. The simplicity, effectiveness, and adaptability of the process make it particularly attractive for application in chemical and metallurgical industries generating acidic metal-bearing effluents.

Overall, the findings provide a scientific and practical basis for the development of integrated, resource-efficient wastewater treatment technologies, supporting sustainable industrial water management and compliance with increasingly stringent environmental regulations.

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