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SELECTIVE REMOVAL OF HEAVY METALS FROM COMPLEX INDUSTRIAL EFFLUENTS BY pH-REGULATED HYDROXIDE PRECIPITATION

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Abstract. Industrial wastewater from chemical and metallurgical processes contains high levels of heavy metals, creating serious environmental challenges. This study evaluates pH-controlled neutralization combined with hydroxide precipitation for treating such wastewater. Samples from various production units were analyzed using ICP-OES and XRF before and after treatment. Neutralization at pH 6.5–8.5 enabled the formation of poorly soluble metal hydroxides. Results showed significant reductions in Cu, Zn, Fe, Ni, Pb, and Cd concentrations. Copper and iron dominated the solid phase, while iron hydroxides enhanced removal via coprecipitation and adsorption. Removal efficiencies above 90% were achieved for the most toxic metals. SEM-EDS analysis confirmed stable Cu–Zn–Fe hydroxide phases in residues. The method reduces environmental risks and concentrates valuable metals into a recoverable solid form, supporting resource-efficient wastewater treatment and sustainable water management.

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

Introduction

Maintaining the purity of soil, water, and air in cities, towns, villages, and industrial facilities is essential for their improved appearance and the sanitary and epidemiological well-being of the population. The concentration of harmful substances entering a water body with wastewater is not constant. It changes due to wastewater dilution and various chemical, physicochemical, and biological processes of interaction, release, transformation, and decomposition of these substances [1].

Wastewater in chemical production is generated from spent solutions, rinse water, cooling system fluids, and also from equipment and room cleaning. It has a complex composition, including toxic organic compounds, heavy metals, acids, and alkalis. The main hazard is the high content of dissolved salts and the difficulty of treatment. The main sources and types of wastewater are process water (spent solutions from reactors, mother liquors, and product wash water), auxiliary water (cooling water, water from condensers, vacuum pumps, and gas cleaning), and operational wastewater (equipment, floor, and pipeline cleaning) [2].

Industrial wastewater containing heavy metals poses serious environmental and health risks due to toxicity, persistence, and bioaccumulation. Rapid growth of metallurgical and chemical industries has increased the volume of metal-rich effluents, which can also serve as valuable secondary resources. Conventional treatment methods, such as precipitation, adsorption, and membrane processes, often face limitations including low selectivity, high cost, and secondary waste generation [3-6].

Recent research highlights the need for reagent-based approaches capable of selective metal removal under controlled conditions, yet fundamental interaction mechanisms and optimal reagent selection remain insufficiently studied. Inadequate treatment can damage infrastructure and increase environmental pollution, making effective technologies essential, especially in regions with intensive industrial activity [7-9].

The problem of neutralizing industrial process solutions and extracting valuable components from them, followed by their use as secondary raw materials, is a pressing issue. Currently, increasing attention is being paid to technologies that allow for the effective extraction of metal ions from industrial solutions and the creation of closed-loop water recycling systems. Experiments to study the effect of the initial pH of the medium on the intensity of industrial wastewater treatment have shown that the oxidation of impurities in both ozone-air mixtures and atmospheric oxygen occurs intensively for the first 15 minutes in almost all cases, after which the oxidation rate decreases. During the experiments, the ozone-air mixture was fed into the unit at a flow rate of 2.5 g/l. The initial pH values of the medium were within the range of 2-5. The temperature of the process solutions was constantly maintained at 200C. The results of experimental studies show that the maximum extraction of metals into the sediment is observed at the beginning of the process. The degree of metal purification is 96-98 % [10-12].

This study aims to investigate selective metal ion interactions and evaluate reagent-based methods for efficient wastewater treatment. The work focuses on improving metal removal efficiency and developing an integrated approach that ensures both detoxification and recovery of valuable metals, contributing to sustainable industrial and environmental management [13-16].

MATERIALS AND METHODS

Industrial wastewater with high heavy metal content represents a serious environmental issue, requiring sustainable and efficient treatment technologies. Maximum permissible concentrations (MPCs) for toxic elements such as Cd, Pb, Hg, Cu, Ni, Zn, and Cr are regulated by environmental standards and were used as reference criteria in this study [17].

Wastewater samples were collected from different production units of JSC “Navoiazot”, including acrylic acid–acrylonitrile, thiourea, and vinyl chloride production. These effluents are characterized by complex composition and high concentrations of dissolved metals [18-20].

Before experiments, samples underwent preliminary mechanical treatment, including filtration and settling to remove suspended solids and coarse particles. Partial sedimentation of insoluble mineral and metal compounds also occurred at this stage, preparing the wastewater for further chemical analysis and treatment (table 1).

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

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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

Experimental results showed that while coarse particles were removed, most metal ions remained dissolved, requiring advanced physicochemical treatment methods. Filtered samples were analyzed using XRF, revealing high concentrations of Cu, Zn, Fe, Ni, As, Sb, Pb, Hg, and other hazardous elements. Wastewater from the vinyl chloride unit also showed elevated Cu, Fe, Zn, K, and chloride species, indicating strong chloride chemistry influence.

Copper was identified as a dominant contaminant, reaching up to 69.5% of total metals, mainly as CuCl. The presence of chloride complexes significantly affects metal behavior and treatment efficiency.

To enhance metal removal, ozonation and ion flotation were applied. Ozonation promoted oxidation and destabilization of metal complexes, while ion flotation enabled selective separation of metals into a foam phase. These methods were chosen for their high efficiency, selectivity, and low reagent consumption, supporting integrated wastewater treatment approaches.

Results and Discussion

Results show a significant reduction in heavy metal concentrations after neutralization and precipitation. The effectiveness of treatment was evaluated by comparing metal levels before and after processing. Key elements analyzed included Cu, Zn, Fe, Ni, Pb, and Cd, as the most hazardous components. This comparison clearly demonstrates the efficiency of the applied method in removing heavy metals from industrial wastewater.

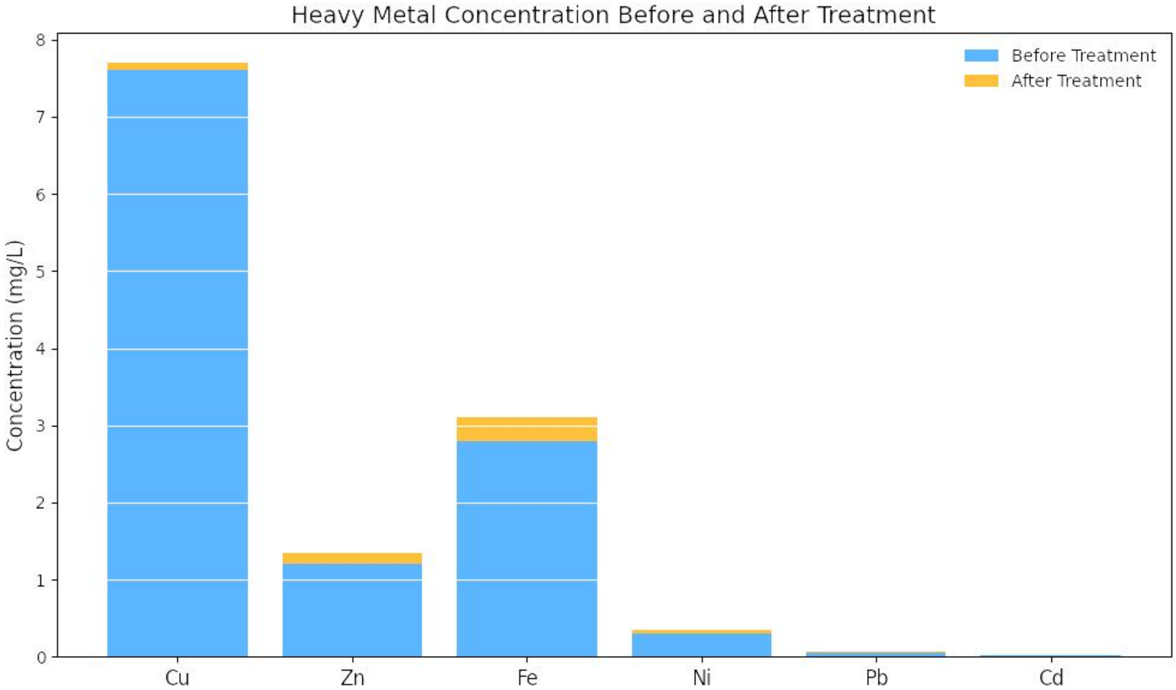

Fig.1. Changes in heavy metal concentrations before and after neutralization

Figure 1 shows that neutralization significantly reduced all heavy metal concentrations. Copper decreased most notably, from about 7.6 mg/L to below 0.1 mg/L, indicating efficient removal due to formation of insoluble hydroxides.

Zinc and iron concentrations also dropped markedly (from 1.2 and 2.8 mg/L to 0.15 and 0.30 mg/L). Iron hydroxide formation enhanced removal of other metals through coprecipitation and adsorption, playing a key role in multicomponent systems.

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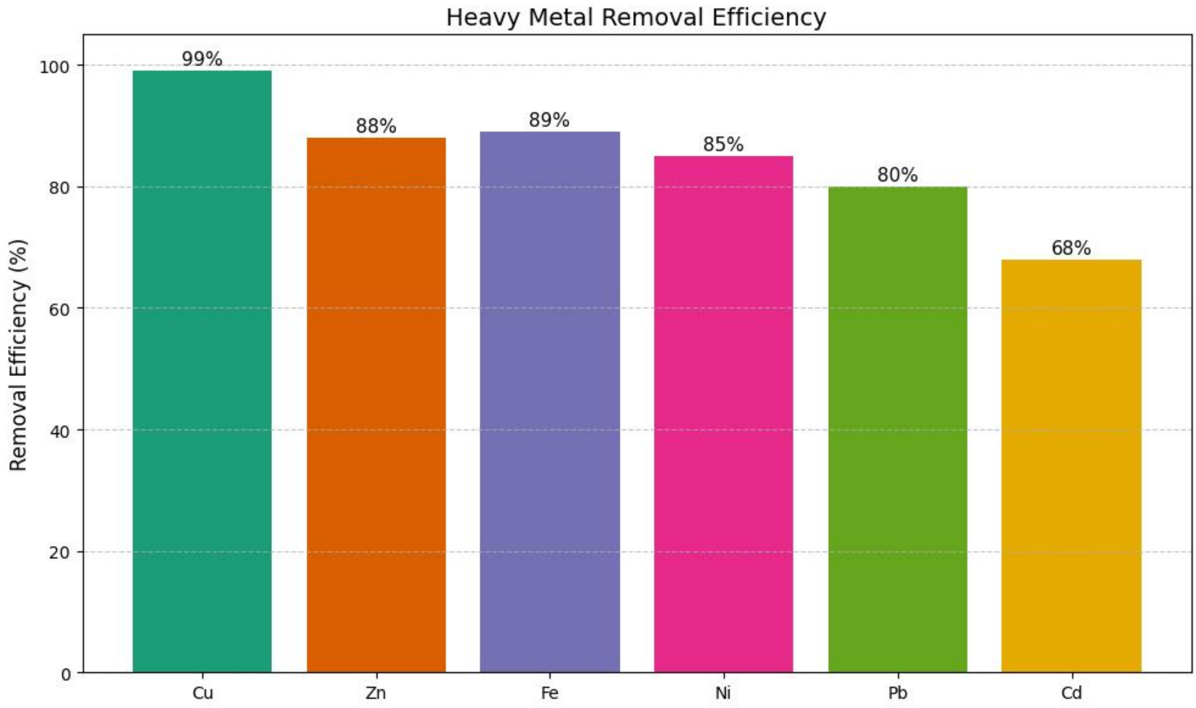

Fig.2. Removal efficiency of heavy metals during neutralization treatment

Nickel, lead, and cadmium were effectively removed, with residual concentrations meeting or approaching regulatory limits. Nearly complete removal of Cd and Pb confirms the efficiency of pH-controlled neutralization for toxic metals.

Removal efficiency was calculated from initial and final concentrations, showing high performance for all metals, especially Cu (>95%), Cd, and Pb due to low solubility of their hydroxides. Iron presence enhanced removal via coprecipitation and adsorption.

Zn and Ni showed slightly lower removal due to higher solubility, yet still reached acceptable levels. Overall, results confirm that neutralization is a robust and versatile method for treating multicomponent wastewater. The process mechanism involves pH control (6.5–8.5), hydroxide formation, and solid–liquid separation, producing treated water and metal-rich sludge.

According to the proposed technological scheme, after the 1st stage of precipitation, the main non-ferrous metal precipitate is separated by filtration, washed and dried to obtain a ready-made copper and zinc concentrate. Since the filtrate solution remaining from the filtration is not completely clean, in order to reduce the negative impact on the environment and to maximize the precipitation of metals, precipitation is carried out in the 2nd stage using ozonation technology with the participation of oxidants. The ozonation process is carried out by changing the pH value back to an acidic environment, from which the precipitate and residual solution formed by filtration are removed, then the final 3rd stage is the precipitation of non-ferrous metals using the cementation method with the participation of iron scrap.

Upon addition of an alkaline reagent, hydroxide ions neutralize acidity and convert dissolved metals into insoluble hydroxides (e.g., $Cu(OH)_2$, $Zn(OH)_2$). In multicomponent systems, $Fe(OH)_3$ particles act as active surfaces for coprecipitation and adsorption, enhancing the removal of trace metals such as Pb, Cd, and Ni.

The process results in clarified water and a Cu–Zn–Fe-rich sludge, reducing environmental risk while concentrating valuable metals.

The findings confirm that pH-controlled neutralization with hydroxide precipitation is an effective and robust treatment method. It highlights the key role of pH and iron hydroxides in metal removal. Practically, this approach enables both wastewater detoxification and metal recovery, supporting resource-efficient and sustainable industrial technologies.

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Conclusion

This study confirms that pH-controlled neutralization with hydroxide precipitation is an effective and practical method for treating metal-containing industrial wastewater. Adjusting pH to 6.5–8.5 enabled efficient removal of Cu, Zn, Fe, Ni, Pb, and Cd, reducing concentrations to near-regulatory levels.

Copper and iron dominated the process, while iron hydroxides enhanced removal through coprecipitation and adsorption. Formation of stable Cu–Zn–Fe sludge confirmed successful transfer of metals into the solid phase.

The method reduces environmental risks and concentrates valuable metals for further recovery. Its simplicity and efficiency make it suitable for chemical and metallurgical industries, providing a basis for sustainable and resource-efficient wastewater treatment technologies.

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