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REMOVAL OF Cu^{2+} AND Zn^{2+} FROM INDUSTRIAL WASTEWATER BY ADSORPTION STUDY OF OPERATIONAL FACTORS

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ABSTRACT

This research investigates the efficiency of commercial activated carbon (CAC) in eliminating copper (Cu^{2+}) and zinc (Zn^{2+}) ions from petroleum wastewater. The wastewater sample was characterized by an acidic pH, high conductivity, and notable metal contents, with copper at 63.14 mg/L and zinc at 9.38 mg/L, reflecting the complexity of such industrial effluents. Structural and chemical analysis of CAC, carried out using FTIR spectroscopy and N_2 adsorption–desorption, revealed the presence of multiple surface functional groups and a mainly microporous framework with a high specific surface area (1147.48 m^2/g).

Batch adsorption tests were conducted to investigate how operational parameters such as contact duration, agitation rate, solution acidity (pH), and sorbent dose influence the process performance. Kinetic analysis revealed that the pseudo-second-order model provided the closest agreement with the experimental data ($R^2 > 0.96$), indicating that chemisorption is the primary mechanism, whereas intraparticle diffusion contributes only at the later phases of adsorption. The results further highlighted that removal efficiencies were strongly enhanced by higher stirring speeds, increased CAC loading, and alkaline environments, ultimately achieving almost complete elimination of both metals.

In contrast to many studies that rely on synthetic solutions, this investigation validates the practical potential of CAC for real wastewater treatment. The outcomes confirm CAC as an effective, and scalable option for the removal of heavy metals, reinforcing its relevance in sustainable wastewater management strategies.

Keywords: Cu^{2+}; Zn^{2+}; Industrial wastewater; Commercial activated carbon; operational factors.

INTRODUCTION

The presence of heavy metals in water environments is considered a critical issue for both ecological integrity and human health (Faye, 2017; Baba Hamed, 2021; Chadee et al., 2024; Ihsan and Derosya, 2024), mainly as they are bio-accumulative, and not easily degraded. Copper and zinc are of particular significance since they are commonly released into the environment through petroleum refining, electroplating, mining, and chemical production processes (Amjad et al., 2020). Although trace levels of these elements play essential physiological roles, an excessive accumulation of these metals may cause pronounced toxicity, particularly affecting vital organs including the liver, kidneys, and the nervous system.

Traditional technologies applied for metal removal including precipitation (Youcef and Achour, 2005), ion exchange (Aw et al., 2024), membrane processes (Gaouar and Gaouar, 2016; Aroua-Berkat and Aroua, 2022), oxidation and advanced oxidation often encounter practical and economic barriers (Achour and Chabbi, 2017; Ziati et al., 2018). These methods are generally less effective for actual effluents containing diverse pollutants at low concentrations and are further hindered by high operating costs and the generation of additional residues requiring further management (Youcef and Achour, 2014; Falfushynska et al., 2024; Piwowarska et al., 2024).

Adsorption emerged as a promising alternative owing to its simplicity, high removal capacity, cost efficiency, and the possibility of adsorbent regeneration (Achour et al., 2002; Youcef et al., 2014; Kheliel et al., 2015 ; Ouakouak and Youcef, 2016; Khelifi et al., 2016; Iyer et al., 2025). Because of its large specific surface, well-developed microporous structure, and diverse surface functional groups, activated carbon continues to be a commonly used adsorbent material (Ghomri et al., 2013; Larakeb et al., 2014; Masmoudi et al., 2018; Youcef et al., 2022; Chauhan and Dikshit, 2023; Karnwal, 2024). However, most reported studies have been performed with synthetic or model solutions, which do not fully capture the physicochemical complexity of real effluents where multiple contaminants coexist and operating conditions fluctuate (Benalia et al., 2022 ; Qasem et al., 2021; Rajakaruna et al., 2023; Akhtar et al., 2024).

To bridge this gap, the present study explores the use of commercial activated carbon (CAC) for treating petroleum wastewater collected from Hassi Messaoud, Algeria. The effluent is highly saline, strongly acidic, and contains considerable concentrations of copper and zinc. The study aims to:

- Characterize CAC's physicochemical and textural features using advanced analytical.
- Evaluate how process variables including contact duration, agitation rate, solution pH, and commercial activated carbon (CAC) dose influence the removal.
- Apply adsorption kinetic models to elucidate the mechanisms governing Cu^{2+} and Zn^{2+} removal.

The expected findings provide valuable insight for designing effective and context-specific treatment systems. Additionally, integrating CAC adsorption with complementary processes such as electrocoagulation or photocatalysis may further enhance performance and long-term sustainability in treating metal-contaminated wastewater.

MATERIAL AND METHODS

Wastewater samples

The wastewater effluent was obtained from the Baroid Algeria de Services aux Puits (BASP), located in the Hassi Messaoud industrial area. The samples were taken at the discharge point prior to any form of treatment. Analytical characterization revealed an acidic pH (4.18), extremely high electrical conductivity (98.3 mS/cm), and a significant organic load with a COD of 1440 mgO_2/L. Metal quantification indicated high concentrations of copper (63.14 mg/L) and zinc (9.39 mg/L), accompanied by a minor amount of iron (0.495 mg/L). These parameters reflect the hazardous nature of the effluent and highlight the complexity involved in its remediation.

Adsorbent

The commercial activated carbon (CAC) used in this study was obtained from Riedel-de Haen laboratory. Fourier Transform Infrared (FTIR) spectroscopy was employed to analyze the surface functional groups of the adsorbent, using a Shimadzu IR Affinity-1 device. The spectra were recorded based on the KBr pellet method. For sample preparation, commercial activated carbon (CAC) was homogenized with spectroscopic-grade KBr, finely milled to ensure homogeneity, and pressed into transparent disks with a hydraulic press. The resulting pellets were analyzed to identify absorption bands characteristic of functional groups attached to the carbon framework.
The textural properties of CAC were identified through nitrogen adsorption–desorption measurements using a Micromeritics ASAP 2010 V5.00 analyzer. Before testing, the sample was degassed under vacuum at high temperature to eliminate moisture and residual gases.

Adsorption experiments

Adsorption tests were carried out under continuous stirring to evaluate the efficiency of commercial activated carbon (CAC) in eliminating Cu^{2+} and Zn^{2+} from petroleum wastewater. In each test, a predetermined dose of CAC was added to 50 mL of raw effluent. The assays were conducted at 20 °C using an orbital shaker operated at predetermined speeds and contact durations.
After mixing, the suspensions were passed through 0.45 µm membrane filter, and the remaining metal concentrations were measured by atomic absorption spectrophotometry (XplorAA Dual, GBC). The investigation focused on four major operational variables: contact time (0–6 h), stirring speed (100–900 rpm), solution pH (2–12), and CAC dosage (1–10 g/L). Each parameter was evaluated through independent experimental sets, with all runs performed in triplicate to ensure reproducibility.

Kinetic models

The experimental results were analyzed with four widely used kinetic models: pseudo-first-order (PFO), pseudo-second-order (PSO), Elovich, and intraparticle diffusion (IPD). These models were fitted to estimate the kinetic constants and to clarify whether Cu^{2+} and Zn^{2+} uptake by CAC is mainly controlled by surface interactions or by mass transfer within the adsorbent.

According to the pseudo-first-order (PFO) model introduced by Lagergren (1898), the adsorption rate is considered proportional to the availability of unoccupied active sites, as represented in the following equation:

$$q_t = q_e\left(1 - e^{-k_1 t}\right) \tag{1}$$

Herein q_t (mg/g) and q_e (mg/g) denote the adsorption capacities given at time t (min) and at equilibrium, respectively. while k_1 (min^{-1}) represents the PFO model rate constant.

The PSO model, developed by Ho and McKay (1999), assumes that chemisorption governs the adsorption rate, relying on valence interactions through electron exchange or sharing, as expressed in the following equation:

$$q_t = \frac{q_e^2 k_2 t}{1 + q_e k_2 t} \tag{2}$$

Herein k_2 (g/mg.min) denotes the PSO model rate constant.

The Elovich model, proposed by Mclintock (1967), is typically used for adsorption systems with heterogeneous surfaces. It considers that the adsorption rate declines exponentially over time as surface coverage increases, and its mathematical form is presented as follows:

$$q_t = \frac{1}{\beta}\ln\left(1+\alpha\beta t\right) \tag{3}$$

where α (mg/g.min) indicates the initial adsorption rate, while β (mg/g) is related to the surface coverage and the activation energy of chemisorption.

Finally, the IPD model, proposed by Weber and Morris (1963), was applied to explore diffusion-controlled processes, as expressed in the following relationship:

$$q_t = K_{int}\, t^{1/2} + C \tag{4}$$

In this case, K_{int} (mg/g.min$^{1/2}$) represents the intraparticle diffusion rate constant, while C (mg/g) corresponds to the intercept, reflecting the boundary layer impact.

Comparative evaluation of these models allows distinguishing between adsorption dominated by physisorption, chemisorption, or diffusion-limited processes in the removal of each ion (Cu^{2+} and Zn^{2+}) using CAC.

RESULTS AND DISCUSSION

CAC characterization

The chemical functionalities of the CAC were investigated through FTIR spectrum (Fig. 1a). A broad absorption band around 3430 cm^{-1} was assigned to O–H stretching vibrations, originating from surface hydroxyl or phenolic groups as well as adsorbed moisture. A smaller peak near 2920 cm^{-1} was associated with aliphatic C–H stretching. The band observed at 1630 cm^{-1} corresponded to C=O stretching vibrations, indicative of carbonyl or carboxyl functionalities (Pramanik et al., 2025), Vibrations in the 1510–1420 cm^{-1} region were attributed to aromatic C=C bonds. while the signal detected at ~1100 cm^{-1} was linked to C–O stretching modes of alcohols, ethers, or phenolic groups. In addition, a peak at 620 cm^{-1} represented out-of-plane bending of aromatic C–H bonds (Cárdenas-Piñeros et al., 2025).

The presence of these surface groups confirms the chemical heterogeneity of CAC, offering a variety of active sites capable of interacting with metal ions. In particular, hydroxyl, carbonyl, and carboxyl moieties are likely to contribute significantly to adsorption through mechanisms such as ion exchange, surface complexation, and electrostatic attraction.

After analyzing nitrogen adsorption–desorption isotherm (Fig.1b). The results revealed a large surface area, with BET and Langmuir values of 1147.48 and 1429.54 m²/g, respectively. Microporous structures accounted for the majority of this area (840.61 m²/g), while the external surface area was measured at 306.87 m²/g. And the total micropore volume was estimated at 0.34 cm³/g. The average pore diameter was 1.71 nm, this kind of size confirms the microporous nature of the adsorbent (Çeçen, 2024).

Taken together, the FTIR spectra and textural analysis demonstrate that CAC combines a chemically diverse surface with a highly microporous architecture. This synergy provides a large number of binding sites and promotes efficient heavy-metal uptake through both rapid surface interactions and effective diffusion within narrow pores.

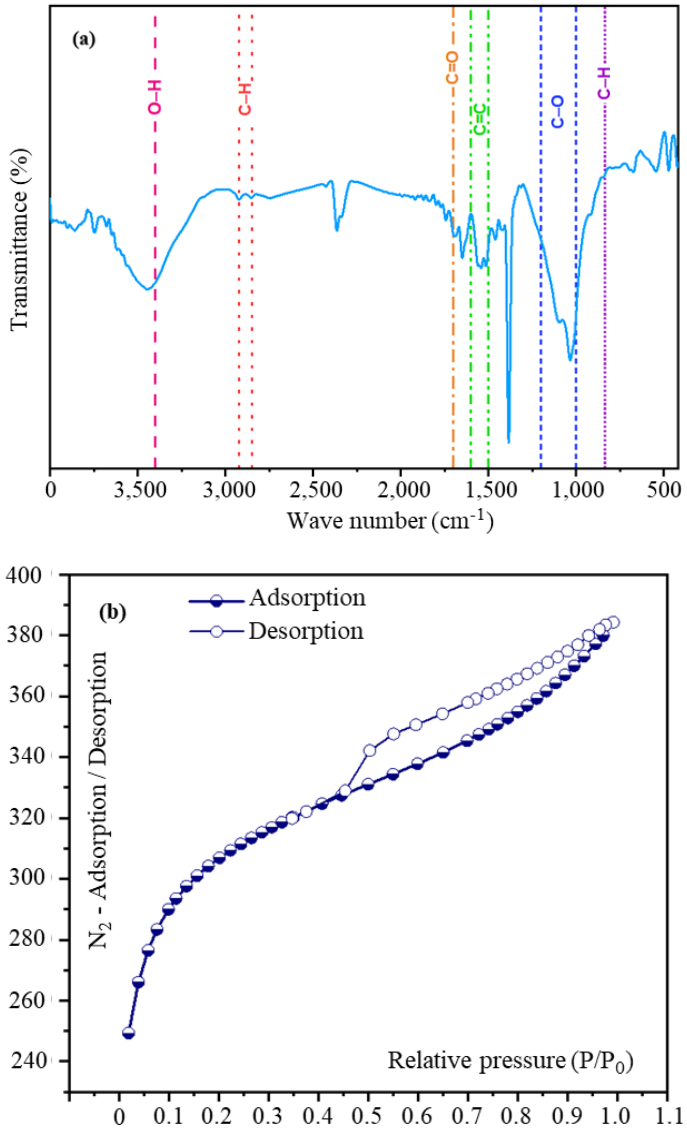

Figure 1: (a) FTIR spectrum, (b) N_2-adsorption/desorption isotherms.

Adsorption kinetics study

As illustrated in Fig. 2a, metal removal rose sharply during the initial 60 min owing to the high availability of active sites on the outer surface of CAC, after which the uptake rate slowed as equilibrium conditions were approached. For both Cu^{2+} and Zn^{2+}, equilibrium was reached after about 240 min. The analysis of application of adsorption kinetic models revealed that the PSO model achieved the best agreement with the experimental results, showing high correlation coefficients ($R^2 > 0.99$) for both Cu^{2+} and Zn^{2+}, whereas the PFO and Elovich models yielded weaker correlations. The strong fit of the PSO model suggests that chemisorption is the dominant pathway, involving electron exchange or sharing between the metal ions and the functional groups present on the surface of CAC (e.g., –OH, –COOH, –C=O) (Youcef et al., 2022; Sharaf, 2015; Maguie et al., 2017; Soudani et al., 2024). The corresponding model parameters are summarized in Table 1.

The PFO model exhibited poor correlation ($R^2 = 0.79$ for Cu and 0.893 for Zn), confirming its inadequacy to represent the adsorption kinetics, as evidenced by the deviations between experimental and calculated q_e values (6.45 vs. 6.15 mg/g for Cu^{2+} and 2.63 vs. 2.50 mg/g for Zn^{2+}). By contrast, the PSO model provided the best agreement with experimental data, with high R^2 values (0.898 for Cu^{2+} and 0.965 for Zn^{2+}) and q_e values nearly identical to experimental ones (6.45 mg/g for Cu^{2+} and 2.63 mg/g for Zn^{2+}), demonstrating that chemisorption was the dominant mechanism.

Furthermore, the intraparticle diffusion model (Fig. 2b) revealed a multilinear behavior, indicating that the adsorption proceeded in two stages: an initial rapid phase corresponding to surface adsorption on external sites, followed by a slower stage attributed to intraparticle diffusion into the micropores of CAC. However, since the plots deviated from the origin, it can be inferred that intraparticle diffusion was not the exclusive controlling factor, and that both film diffusion and surface interactions contributed to the adsorption mechanism (Yalçin et al., 2005; Shi et al., 2023).

These findings highlight that the adsorption of Cu^{2+} and Zn^{2+} onto CAC is a complex process dominated by chemisorption. According to (Nasser et al., 2024; Haerifar and Azizian, 2013), both surface interactions and intraparticle diffusion mechanisms play important roles in chemisorption.

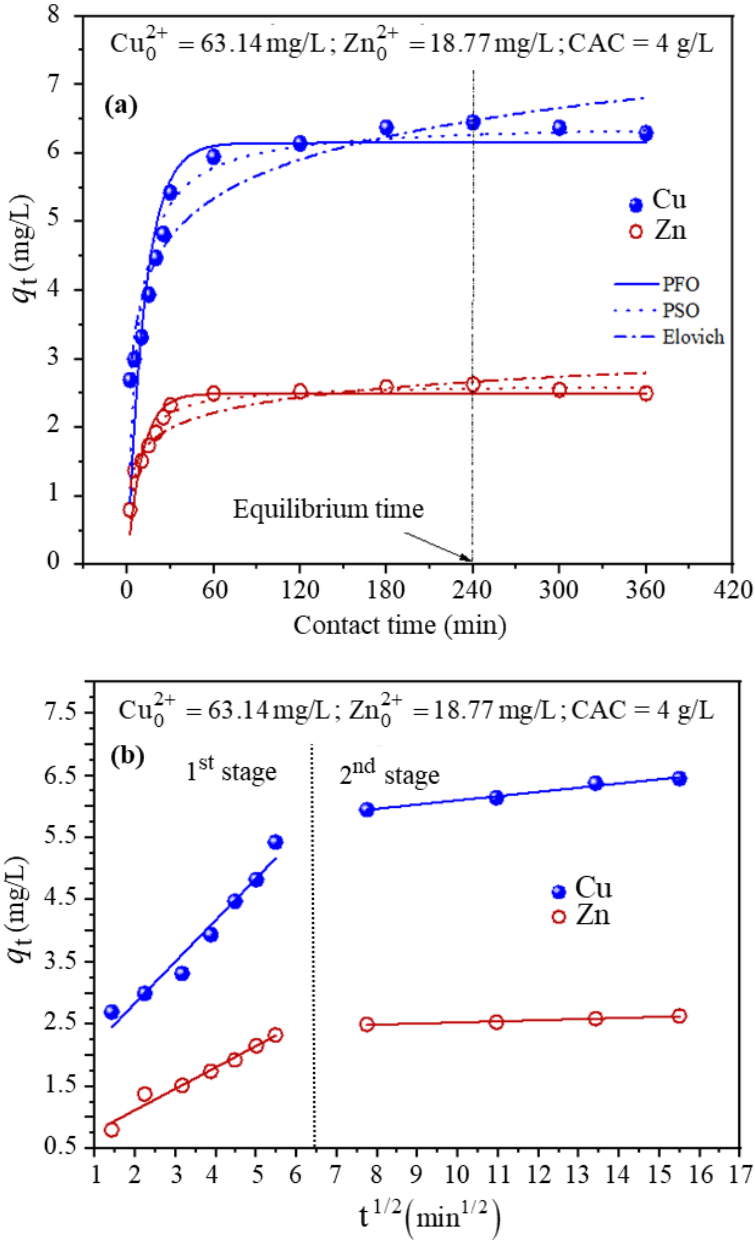

Figure 2: Experimental data, and (a) fitting of the PFO, PSO, and Elovich models, (b) fitting of the intraparticle diffusion model for copper and zinc (adsorbent dose = 4 g/L; agitation speed = 900 rpm).

Table 1: Kinetic adsorption model parameters for copper and zinc onto CAC.

Metal ion	Kinetic model				
	PFO				
	$q_{e,Exp}$ (mg/L)	$q_{e1,Cal}$ (mg/g)	K_1 (L/min)	R^2	χ^2
Cu^{2+}	6.450	6.153	0.081	0.79	0.45
Zn^{2+}	2.632	2.499	0.099	0.893	0.039
	PSO				
	$q_{e,Cal}$ (mg/g)		k_2 (g/mg.min)	R^2	χ^2
Cu^{2+}	6.447		0.023	0.898	0.218
Zn^{2+}	2.630		0.066	0.965	0.013
	Elovich				
	β (mg/g)		α (mg/g.min)	R^2	χ^2
Cu^{2+}	1.22		9.208	0.925	0.16
Zn^{2+}	3.062		4.826	0.876	0.045
	IPD				
	$K_{int,1}$ (mg/g/min$^{1/2}$)	C_1 (mg/g)	R^2	$K_{int,2}$ (mg/g/min$^{1/2}$)	C_2 (mg/g)	R^2
Cu^{2+}	0.669	1.506	0.96	0.068	5.425	0.981
Zn^{2+}	0.433	0.343	0.97	0.018	2.351	0.967

Effect of agitation speed

The impact of stirring rate on the adsorption of Cu^{2+} and Zn^{2+} onto CAC was evaluated within the 100–900 rpm range (Fig. 3). At the lowest agitation speed (100 rpm), the removal performance was relatively poor, mainly due to the presence of a thicker liquid boundary layer that hindered the diffusion of metal ions toward the adsorbent surface (Bašić et al., 2023; Zahoor 2011). As the agitation speed increased, a noticeable enhancement in adsorption efficiency was observed, reaching approximately 41% for Cu^{2+} and 56% for Zn^{2+} at 900 rpm. This improvement can be attributed to more effective external mass transfer, which reduces film resistance around CAC particles and facilitates

the approach of ions to the active sites (Syauqiah et al., 2020; Kuśmierek and Świątkowski, 2015).

Furthermore, higher stirring rates ensured better dispersion of CAC particles in the solution, preventing agglomeration and promoting more homogeneous contact between the solid adsorbent and the liquid phase (Pasaribu and Kurniawati, 2024).

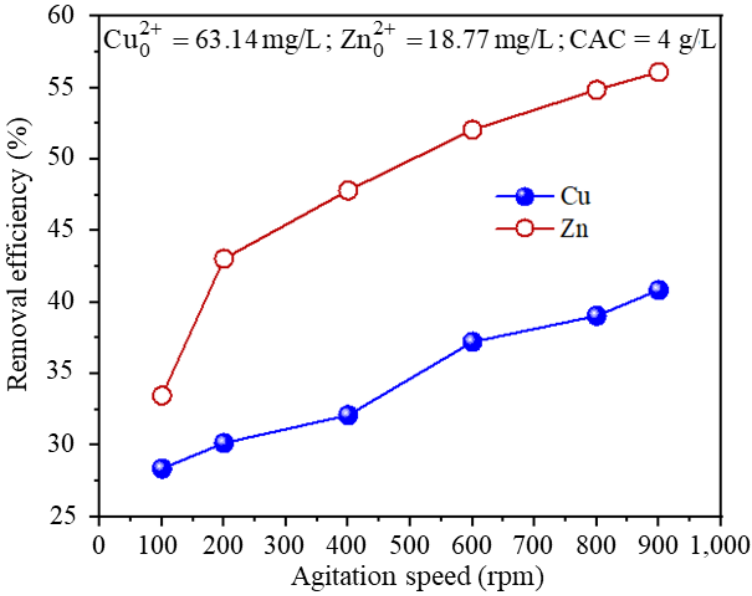

Figure 3: Effect of agitation speed on the removal efficiency of Cu^{2+} and Zn^{2+} from wastewater.

Effect of pH

As illustrated in Fig. 4, the uptake of Cu^{2+} and Zn^{2+} improved steadily as pH increased from 2 to 12. Under strongly acidic conditions (pH 2–3), however, the removal remained very limited below 16% for Cu^{2+} and 24% for Zn^{2+} mainly because the excess H^+ ions competed with the metal cations for negatively charged adsorption sites on the CAC surface.

With increasing pH, this competition diminished, which favored electrostatic attraction and surface complexation between metal ions and –OH, –COOH, –C=O groups on CAC surface. Around neutral conditions (pH 6–7), the adsorption capacity improved markedly, achieving more than 56% removal for Cu^{2+} and 63% for Zn^{2+}. The highest efficiencies were reached under alkaline conditions (pH ≥ 8), approaching complete removal of both ions (Anirudhan et al., 2010; Romero et al., 2004).

The observed enhancement at higher pH can be explained by the increased negative charge on the CAC surface, which promotes stronger electrostatic attraction with cations. Additionally, the precipitation of metal hydroxide species may also contribute to removal (Romero et al., 2004; Youcef et al., 2022 ; Wang et al., 2016).

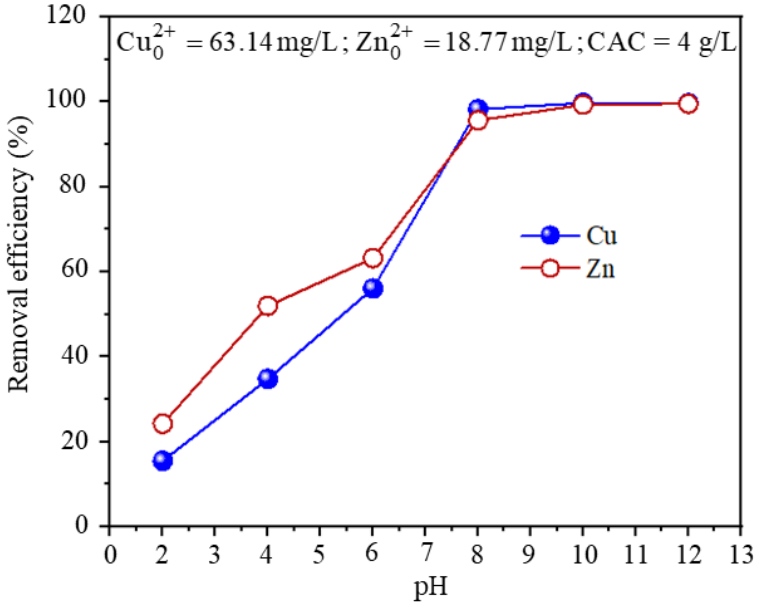

Figure 4: Effect of initial pH on the removal of Cu^{2+} and Zn^{2+} from wastewater

Effect of adsorbent dose

The influence of CAC concentration on Cu^{2+} and Zn^{2+} removal was examined within the range of 1–10 g/L (Fig. 5). Adsorption efficiency increased consistently as the CAC dose rose. At lower concentrations (1–2 g/L), however, the limited number of active sites relative to the initial metal content resulted in poor removal efficiencies, below 21% for Cu^{2+} and 50% for Zn^{2+}.

As the adsorbent amount increased, the density of active sites expanded, facilitating more ion–surface interactions. At 6 g/L, removal efficiencies rose to over 44% for Cu^{2+} and 65% for Zn^{2+}, highlighting a substantial enhancement relative to the lowest doses. Further increases in CAC content produced only marginal gains: at 10 g/L, the efficiencies leveled off at ~84% for Cu^{2+} and ~88% for Zn^{2+}. In this case, (Zhao et al. (2020) and (Pérez et al. (2022) suggested that most ions had already been adsorbed and additional adsorbent contributed little improvement.

Interestingly, recent studies report similar trends for other carbonaceous adsorbents, such as biochar and surface-modified carbons, which combine high efficiency with low cost and sustainability, making them attractive for large-scale wastewater treatment (Chen et al., 2022; Malik et al., 2024; Youcef et al., 2022).

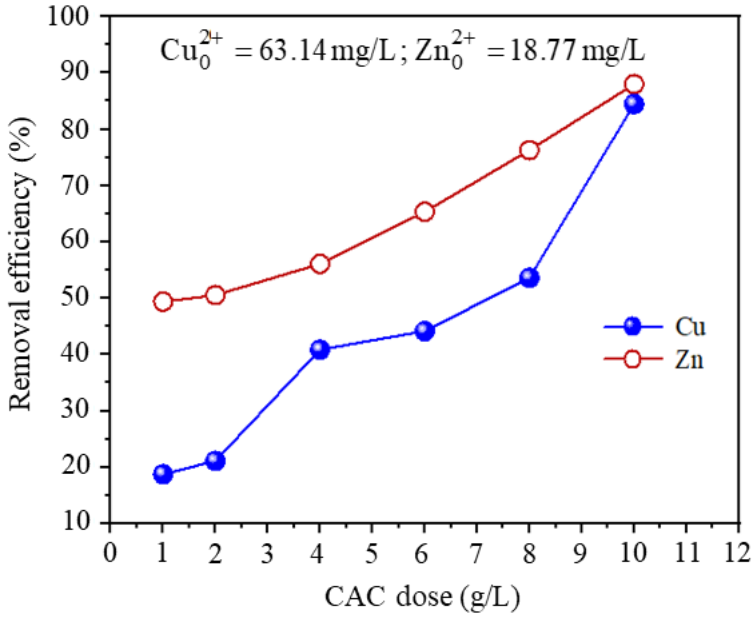

Figure 5: Effect of CAC dosage on the removal efficiency of Cu^{2+} and Zn^{2+} from wastewater.

CONCLUSION

The present study demonstrated the strong capability of commercial activated carbon (CAC) in removing copper and zinc from real industrial effluents. The adsorbent was characterized by a predominantly microporous structure enriched with oxygenated functional groups, which facilitated metal uptake through mechanisms including ion exchange, surface complexation, and chemisorption. Kinetic analyses revealed that the adsorption behavior followed a pseudo-second-order model, with intraparticle diffusion contributing only as a secondary rate-limiting step.

Operational conditions were found to play a decisive role: higher agitation improved external mass transfer, elevated pH promoted electrostatic attraction and complexation, and increasing CAC dosage enhanced removal efficiency until saturation was reached. Under optimized parameters, removal exceeded 80% for both Cu^{2+} and Zn^{2+}, confirming the suitability of CAC for treating acidic wastewater streams.

Beyond confirming CAC's performance, this work provides mechanistic understanding that can aid in the design of more efficient adsorption systems. The findings also underline the potential of integrating CAC into sustainable hybrid treatment strategies for the remediation of heavy-metal-laden industrial discharges.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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