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Research Paper

Sustainable Utilization of K-Humate Coal Residue into Bead Adsorbents for Cu(II) Removal

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Abstract
The increasing discharge of heavy metals, particularly copper (Cu), into soil and water systems poses significant environmental and health risks due to their toxicity, persistence, and potential for bioaccumulation. In this study, coal residue, a solid byproduct derived from potassium humate (K-humate) production, was sustainably converted into composite chitosan beads and evaluated as an adsorbent for the removal of Cu(II) from aqueous solutions. The coal residue was pretreated with HCl, sieved, and incorporated into chitosan gel, followed by crosslinking with sodium tripolyphosphate to form stable beads. Batch adsorption experiments were performed to investigate the effects of pH (4–7), adsorbent dosage (0.1–0.4 g in 25 mL solution), and temperature (30–50 °C) on Cu(II) removal efficiency at an initial concentration of 67.46 mg L^{-1}. The results consistently demonstrated high removal efficiencies (>92%) under all tested conditions, with optimal performance observed at a slightly acidic pH (4), higher adsorbent dosages, and elevated temperatures, indicating an endothermic adsorption process. The coal–chitosan composite beads showed stable performance across varying conditions, highlighting the synergistic role of chitosan as a matrix for residue immobilization and improved reusability. This work not only presents a low-cost and sustainable route for valorizing coal residue but also introduces an effective adsorbent for Cu(II) remediation in wastewater treatment applications.

Keywords *Chitosan beads, Coal residue valorization, Copper removal (Cu(II)), Sustainable wastewater treatment*

INTRODUCTION

The production of potassium humate (K-Humate) from coal generally involves a series of physicochemical steps, including grinding, oxidation, alkaline extraction, evaporation, and drying (Timotius et al., 2025). In the initial stage, coal is finely ground to increase its surface area, followed by an oxidation process using hydrogen peroxide (H2O2) (Kusumastuti et al., 2025) to break down the coal structure and increase the number of hydroxyl and carboxyl functional groups in the coal (Li & Yuan, 2021). The oxidized coal is then subjected to extraction with potassium hydroxide (KOH) (Cheng et al., 2019), producing a liquid rich in humic substances that is further concentrated through evaporation and subsequently dried to obtain K-Humate as the final product (Timotius et al., 2025). However, during the extraction stage, a solid byproduct known as coal residue is generated. This residue still contains significant amounts of unreacted carbon, rich in active functional groups such as carboxyl and hydroxyl groups, which are currently underutilized or burned as fuel. The presence of this residue highlights the need for sustainable valorization strategies, particularly its potential application as a precursor for adsorbent materials.

To address the challenge of underutilized coal residue from K-Humate production, this study proposes a sustainable conversion of this residue into chitosan-based bead adsorbents. Chitosan, a biopolymer derived from chitin, is widely recognized for its biodegradability, biocompatibility, and high affinity toward metal ions due to its amino and hydroxyl functional groups (Timotius et al., 2022). However, pure chitosan beads often suffer from limitations such as low mechanical strength and limited surface area (Vakili et al., 2019). Incorporating coal residue into chitosan beads not only enhances the structural stability and surface properties of the adsorbent but also provides a

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practical valorization pathway for this industrial byproduct. The resulting composite beads are expected to exhibit improved adsorption performance, particularly for divalent heavy metal ions such as Cu(II), making them suitable for wastewater treatment applications while simultaneously reducing solid waste from the K-Humate production process.

Heavy metal contamination in soil and irrigation water represents a serious environmental concern in the agricultural sector, primarily because elements such as copper (Cu), cadmium (Cd), and lead (Pb) can accumulate in crops and subsequently enter the food chain (Ramlan et al., 2022). Agricultural areas located near sources of heavy metal emissions, such as mining activities, industrial effluents, or waste disposal sites, are particularly vulnerable to this form of pollution. Among these metals, copper (Cu) poses unique risks. While copper (Cu) is an essential micronutrient for plants, excessive concentrations beyond permissible limits in soils can exert toxic effects on both crops and humans. Copper is characterized by relatively low mobility under normal environmental conditions, and its concentration in plants is generally within the range of 1–50 ppm (Gonzaga et al., 2018). However, when soil Cu levels exceed this threshold, accumulation within plant tissues can occur, leading to physiological stress, impaired photosynthesis, and reduced crop productivity (Mir et al., 2021). More critically, crops contaminated with elevated levels of Cu may enter the human diet, where long-term exposure can cause adverse health effects, including liver and kidney damage, gastrointestinal disturbances, and oxidative stress (Niknejad et al., 2023). Therefore, developing efficient and low-cost adsorbent materials for removing Cu from contaminated water and soils is of significant importance in safeguarding agricultural sustainability and food safety.

Despite the growing interest in sustainable utilization of coal derivatives, limited attention has been given to the valorization of coal residue generated during K-Humate production (Timotius et al., 2025). Most studies on K-Humate have focused primarily on optimizing humic acid extraction or evaluating the agricultural benefits of the final product. At the same time, the solid byproduct is often overlooked and treated as waste or as an alternative fuel with low calorific value. This residue, however, still contains a significant amount of active site (carboxyl and hydroxyl functional groups) and inorganic components (potassium) that could serve as an active phase in adsorbent synthesis. Although coal-based materials have been investigated in other contexts, such as activated carbon production, there is a lack of research exploring the direct integration of coal residue into biopolymer matrices, particularly chitosan, to produce low-cost, eco-friendly adsorbents. Addressing this gap could not only reduce the environmental burden of coal residue disposal but also contribute to the development of efficient materials for removing heavy metals from aqueous systems.

LITERATURE REVIEW

Copper

Copper is widely utilized due to its high electrical and thermal conductivity (Pisk et al., 2025), ductility (Deshmukh et al., 2022), and corrosion resistance (Ekerenam et al., 2025), with the most significant demand arising from the power and electronics sectors (Rötzer & Schmidt, 2020). In the environment, copper originates from both natural and anthropogenic sources. Natural inputs include geological weathering, volcanic emissions, sea spray, and forest fires (Chávez et al., 2021). However, anthropogenic activities dominate in contaminated watersheds, particularly mining and mineral processing (Izydorczyk et al., 2021), electronics recycling (Quinto et al., 2025), as well as urban runoff from landfill areas (Adu & Aneke, 2025). Biologically, copper is an essential micronutrient that serves as a cofactor in numerous redox enzymes; however, excess Cu(II) is toxic (Collins, 2021). In aquatic ecosystems, elevated dissolved copper is harmful to fish and invertebrates, with toxicity surpassing that of Cadmium (Cd), Arsenic (As), and Lead (Pb) (Cui et al.,

2021). It may reduce biodiversity and disrupt the ecological balance of aquatic ecosystems (Cui et al., 2024). Copper is an essential micronutrient for plants; however, excessive concentrations beyond the permissible limits in soils can exert toxic effects on both crops and humans (Mir et al., 2021).

Removal of copper from wastewater can be achieved through various methods, including coagulation, ion exchange, membrane separation, electrochemical processes, and adsorption (Liu et al., 2023). Coagulation/flocculation (e.g., with polymers) aggregates colloidal/organically complexed Cu but shares sludge drawbacks and pH sensitivity (Huang et al., 2016). Ion exchange (using chelating resins) offers high selectivity and low effluent Cu levels at ppb–ppm concentrations, but resins are more costly and prone to fouling (Virolainen et al., 2021). Membrane processes deliver excellent removal (Kanagaraj et al., 2020), but require high energy, concentrate management, and membrane replacement costs. Electrochemical methods (electrocoagulation, electrodeposition) minimize chemicals and can recover Cu metal, yet demand reliable power and careful control to avoid passivation (Liu et al., 2023). Adsorption has become a leading "polishing" or primary treatment for low–to moderate Cu(II) levels due to its operational simplicity, minimal sludge generation, and potential for sorbent regeneration (Khan et al., 2021).

Adsorption

Adsorption is a surface phenomenon in which molecules, ions, or atoms from a fluid phase (gas or liquid) accumulate on the surface of a solid or, less commonly, a liquid, forming a thin layer at the interface (Ray & Das, 2020). The process can occur through physical adsorption (physisorption), driven by weak van der Waals forces, or chemical adsorption (chemisorption), which involves stronger chemical bonds, such as ionic or covalent interactions (Atif et al., 2022). Adsorption has numerous applications in environmental remediation (e.g., removing heavy metals from wastewater), catalysis, gas separation, energy storage, and sensor technology. It is considered an efficient and eco-friendly method due to its high selectivity, cost-effectiveness, and potential for regeneration of the adsorbent material (Li & Wang, 2025).

Adsorption aligns with circular-economy goals when low-cost, carbon-rich, or bio-based sorbents are used (agro-residues, biochar, coal-derived residues, zeolites, clays, biopolymers) (Almeida-Naranjo et al., 2025). Kinetics are described in several models, including pseudo-first-order, pseudo-second-order, chemisorption, film/intraparticle diffusion, and others (Wirawan et al., 2022). Isotherms commonly fit Langmuir (monolayer, finite sites) or Freundlich (heterogeneous surface) models (Tran et al., 2021). Practical performance depends on pH (typically optimal near 5–6 to balance site deprotonation without $Cu(OH)_2$ precipitation), ionic strength, competing cations, and natural organic matter (Saif et al., 2015).

Oxidized carbonaceous materials, including activated carbon, biochar, graphene oxide, and coal-derived residues, have garnered significant attention as adsorbents due to their surface chemistry and structural versatility (Zhou et al., 2024). The oxidation process introduces oxygen-containing functional groups, primarily carboxyl (–COOH), hydroxyl (–OH), and carbonyl (C=O), onto the carbon framework, thereby enhancing hydrophilicity and providing strong binding sites for metal ions (Derylo-Marczewska et al., 2025). These groups facilitate complexation, ion exchange, and electrostatic attraction with Cu(II), improving adsorption affinity compared to non-oxidized carbons.

Chitosan (deacetylated chitin) is a renewable, biodegradable biopolymer enriched in primary amines ($–NH_2$) and hydroxyls (–OH) (Timotius et al., 2022). It has strong chelation sites for Cu(II) (Chen et al., 2020). Chitosan's metal affinity, low toxicity, and filmability make it a premier "green" sorbent (Ali et al., 2020). As a "green" source, the utilization of chitosan is usually incorporated with other materials, such as carbon-based materials, into beads (Kumar et al., 2022).

RESEARCH METHOD

Materials

The chemicals used in this study included hydrochloric acid (HCl, 37%) for the pretreatment and neutralization of coal residue, chitosan as the primary biopolymer for bead formation, and glacial acetic acid (100% solution) as the solvent for dissolving chitosan into a viscous gel. Sodium tripolyphosphate (NaTPP) was employed as a crosslinking agent to stabilize the composite beads, while distilled water was used throughout the process for rinsing and solution preparation. The Cu(II) ion was obtained from CuSO4 · 5H2O. All chemicals were used without further treatment.

Beads Synthesis

The coal residue was first pretreated by soaking it in a 0.1 N HCl solution to neutralize its pH to approximately 7. The neutralized residue was then dried in an oven and subsequently sieved using a 200-mesh screen to obtain fine carbon powder. Separately, 2 g of chitosan was dissolved in 100 mL of 1% acetic acid solution under continuous stirring with a magnetic stirrer at 30 °C until a viscous gel was formed. An equal amount (2 g) of the sieved carbon powder was then added to the chitosan gel, and the mixture was stirred at room temperature until a homogeneous composite gel was obtained. This composite gel was carefully added to a beaker containing 100 mL of a 10% sodium tripolyphosphate (NaTPP) solution, where it was allowed to stand until stable beads formed. The resulting beads were filtered, rinsed with distilled water to remove residual reagents, and finally dried in an oven at 70 °C for approximately 24 hours to yield well-formed chitosan–coal residue composite beads.

Cu(II) Adsorption

The adsorption performance of the chitosan–coal residue beads was assessed in batch experiments using a Cu(II) solution with an initial concentration of 67.4634 mg L^{-1} as the model contaminant. A working volume of 25 mL was used, and the pH was adjusted to 4, 5, 6, or 7 using dilute HCl to evaluate the influence of acidity on adsorption. To investigate the effect of adsorbent dosage, bead masses of 0.1 g, 0.3 g, and 0.4 g were introduced into the solutions. The experiments were conducted at controlled temperatures of 30, 40, and 50 °C, with contact times varied up to 300 minutes. At predetermined intervals, approximately 10 mL of solution was withdrawn and analyzed for Cu(II) concentration using atomic absorption spectroscopy (AAS). Adsorption efficiency was expressed as both percentage removal and uptake capacity, based on the difference between initial and residual Cu(II) concentrations.

Removal Calculation

The extent of Cu(II) removal was determined from the decrease in solution concentration after adsorption. The percentage removal was calculated according to Equation 1:

$$Removal\% = \frac{C_o - C_e}{C_o} \times 100\% \quad (1)$$

where C_o (mg L^{-1}) is the initial Cu(II) concentration and C_e (mg L^{-1}) is the concentration at equilibrium.

FINDINGS AND DISCUSSION

Effect of pH

The effect of solution pH on Cu(II) removal by coal residue (in powder) and coal–chitosan

beads is shown in Figure 1. Both adsorbents exhibited high removal efficiencies across the tested pH range (4–7), with values exceeding 95%, indicating their strong affinity toward Cu(II) ions. For coal residue, the maximum removal efficiency (99.2%) was observed at pH 5, after which the efficiency decreased sharply to 95.1% at pH 7. In contrast, the coal–chitosan beads demonstrated a slightly lower but more stable removal performance, maintaining removal above 97% across the entire range, with the highest removal (98.7%) observed at pH 4.

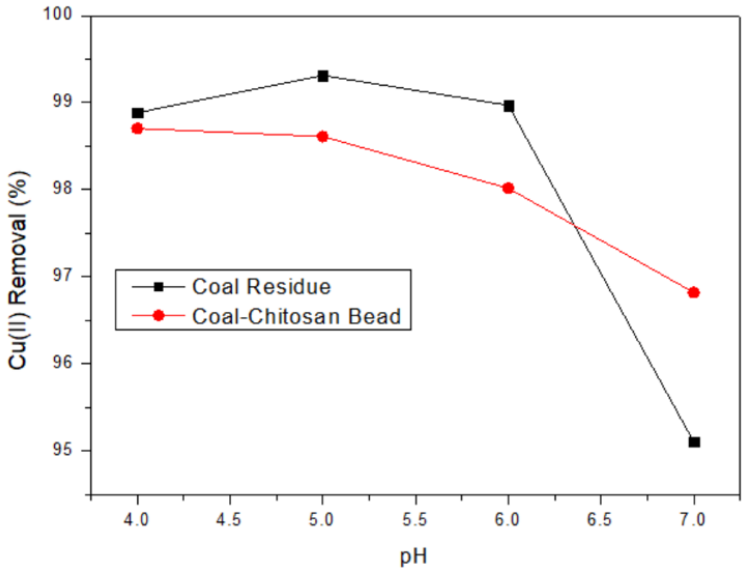

Figure 1. Effect of pH on Cu(II) Removal Efficiency Using Coal Residue and Coal–Chitosan Bead Adsorbents

The interplay between metal ion speciation and surface charge of the adsorbents can explain the observed trends. At lower pH values, competition between protons (H^+), -RNH3+, and Cu(II) ions for adsorption sites typically suppresses metal uptake. However, in this study, both coal residue and chitosan–coal beads maintained high efficiency even at pH 4, suggesting the presence of abundant active sites with strong affinity toward Cu(II). This result was similar to other results (Saif et al., 2015), which adsorb several metal ions (Cu(II), Ni(II), and Cr(IV)). An optimum pH for Cu(II) adsorption was found to be around pH 4-5. As the pH increased to near-neutral values, a decline in removal efficiency was observed. Interestingly, the coal residue alone showed slightly higher removal efficiency than the coal–chitosan beads at acidic pH, likely due to the direct availability of carbonaceous and mineral active sites. However, the incorporation of coal residue into chitosan beads provided a more controlled and stable performance across the entire pH range, highlighting the role of the chitosan matrix in stabilizing the adsorption process. The results indicate that both adsorbents are highly effective for Cu(II) removal, with optimal performance occurring at slightly acidic conditions (pH 4).

Effect of Temperature

The effect of temperature on Cu(II) removal efficiency is presented in Figure 2. The results indicate a clear positive correlation between temperature and adsorption performance, with removal efficiency increasing from 93.8% at 30 °C to 99.0% at 50 °C. This trend suggests that the adsorption of Cu(II) onto the chitosan–coal residue beads is an endothermic process (Wirawan et al., 2022), where higher temperatures enhance the diffusion of Cu(II) ions from the bulk solution to the adsorbent surface, thereby increasing the accessibility of active binding sites. Elevated

temperatures may also increase the pore size, leading to enhanced interactions and coordination between the adsorbent surface and Cu(II) ions (Demirbas et al., 2009).

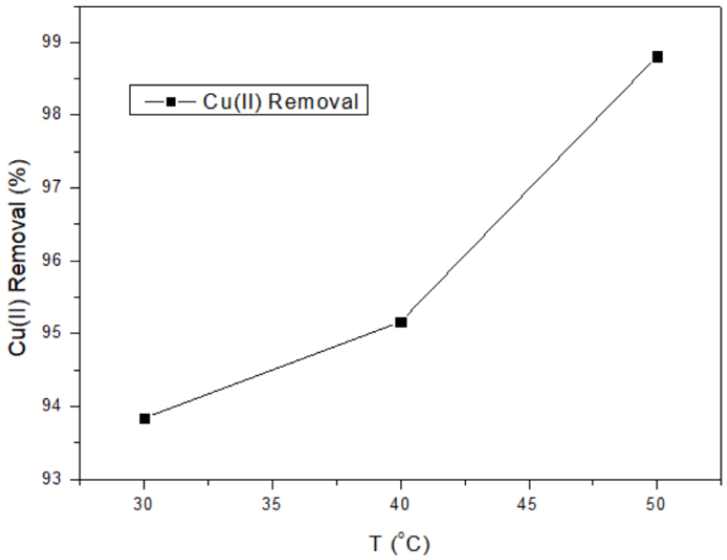

Figure 2. Effect of Temperature On Cu(II) Removal Efficiency Using Coal–Chitosan Bead Adsorbents

The observed improvement in Cu(II) removal with temperature implies that chemisorption likely plays a significant role, since such processes are typically favored at higher thermal conditions. Furthermore, the increase in adsorption efficiency may also indicate a reduction in boundary layer resistance and faster intraparticle diffusion at elevated temperatures. Overall, these results confirm that the adsorption system benefits from moderately higher operational temperatures, although practical applications should balance the energy costs of heating with the enhanced removal efficiency.

Effect of Adsorbent Dose

The influence of adsorbent dose on Cu(II) removal efficiency is illustrated in Figure 3. An increase in bead dosage from 4 g L^{-1} to 16 g L^{-1} significantly enhanced Cu(II) removal from 92.3% to 99.0%. This trend can be attributed to the greater number of available adsorption sites and surface functional groups provided by the higher adsorbent mass (Wirawan et al., 2022), which increases the probability of Cu(II) ions being adsorbed from the solution. The improved performance at higher dosages also reflects the reduction of mass-transfer resistance due to the larger adsorbent surface area, thereby facilitating faster attainment of equilibrium. However, while percentage removal increased with bead dose, it is important to note that adsorption capacity expressed as qe (mg g^{-1}) typically decreases at higher dosages because the same amount of Cu(II) is distributed across a larger adsorbent mass (Demirbas et al., 2009). This phenomenon, often referred to as the "adsorbent dose effect," suggests that beyond a certain point, additional adsorbent does not significantly improve metal uptake per unit mass. Therefore, while higher dosages ensure maximum contaminant removal, optimization is necessary to balance treatment efficiency and material utilization.

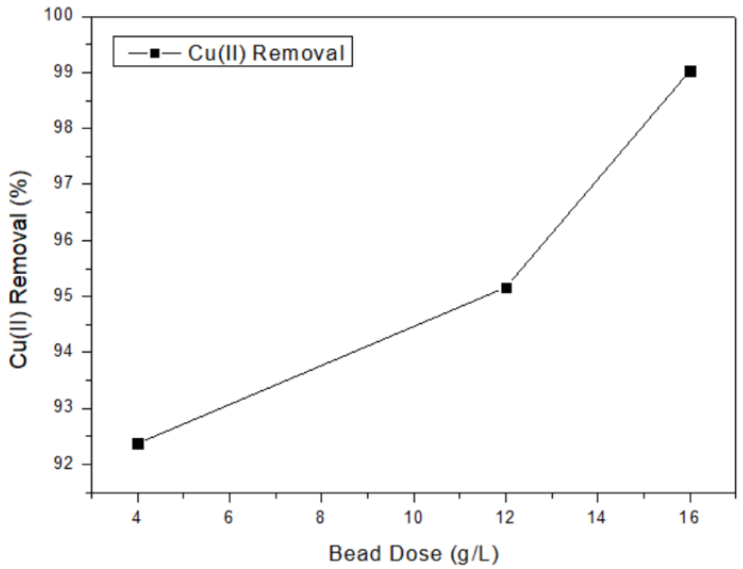

Figure 3. Effect of Bead Dosage on Cu(II) Removal Efficiency

CONCLUSIONS

This study demonstrated the sustainable conversion of coal residue, a byproduct of K-humate production, into chitosan–coal composite beads for the removal of Cu(II) from aqueous solution. The prepared beads exhibited high adsorption performance, with removal efficiencies exceeding 90% under all tested conditions. The adsorption behavior was strongly influenced by pH, temperature, and adsorbent dosage. Optimal removal occurred at slightly acidic conditions (pH 5–6), consistent with the known adsorption characteristics of copper on carbonaceous materials. The process was endothermic, as indicated by enhanced removal at elevated temperatures. Higher bead dosages provided greater removal efficiency due to the increased availability of active sites. These results highlight the dual benefit of waste valorization and effective heavy metal remediation, positioning coal residue–chitosan beads as a promising low-cost adsorbent for wastewater treatment applications.

LIMITATIONS & FURTHER RESEARCH

While the findings confirm the potential of coal residue–chitosan beads for Cu(II) removal, several areas merit further investigation. First, adsorption kinetics and isotherm modeling should be explored in detail to clarify the dominant adsorption mechanisms and capacity limits. Second, thermodynamic parameters such as $\Delta H°$, $\Delta S°$, and $\Delta G°$ should be evaluated to better understand the spontaneity and feasibility of the process. Third, studies on regeneration and reusability are needed to assess the long-term stability and economic viability of the adsorbent. Finally, future work should expand to multi-metal systems (e.g., Pb^{2+}, Cd^{2+}, Zn^{2+}) and real wastewater samples to validate the performance of the beads under practical conditions. Scaling up the synthesis process and integrating it into existing wastewater treatment systems also represent important directions toward industrial application.

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