



Adsorptive Removal of Heavy Metal Ions from Industrial Wastewater Using Activated Carbon

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Peer Review Information

Submission: 14 Jan 2017

Revision: 01 Feb 2017

Acceptance: 16 March 2017

Keywords

Activated carbon, Heavy metals, Industrial wastewater, Adsorption, Water treatment.

Abstract

Heavy-metal contamination of industrial wastewater is persistent because metallic ions are non-biodegradable and may accumulate in aquatic and biological systems. This methodology paper presents a reproducible batch-adsorption framework for removing Pb(II), Cd(II), Cu(II), and Ni(II) from industrial wastewater using activated carbon. The approach combines wastewater characterization, adsorbent preparation and physicochemical characterization, controlled adsorption experiments, residual metal analysis, and mathematical interpretation using removal efficiency, adsorption capacity, kinetic models, and Langmuir and Freundlich isotherms. Experimental variables are pH, contact time, activated-carbon dosage, and initial metal concentration. Metal concentrations are quantified by atomic absorption spectrophotometry (AAS) or inductively coupled plasma optical emission spectrometry (ICP-OES). Literature shows that activated carbons made from agricultural, municipal, and tyre-derived precursors can provide high metal uptake because of their porous structure and oxygen-containing surface groups. For demonstration, an illustrative data set shows removal efficiency of 94% Pb(II), 85% Cd(II), 90% Cu(II), and 80% Ni(II) at selected optimum conditions. These numerical values are included to demonstrate analysis and should be replaced by actual laboratory measurements in an experimental submission. The proposed protocol offers a low-cost and scalable basis for evaluating activated carbon as a polishing step for metal-bearing industrial effluents.

Introduction

Industrial operations such as electroplating, mining, metal finishing, battery manufacture, pigment production, tanning, and alloy processing can discharge wastewater containing lead, cadmium, chromium, copper, nickel, and zinc. Unlike many organic contaminants, these metals cannot be destroyed by conventional biological degradation and may remain in sediments or enter the food chain. Fu and Wang (2011) reviewed the principal treatment options for metal-bearing wastewater and identified adsorption, ion exchange, membrane filtration,

precipitation, coagulation-flocculation, flotation, and electrochemical processes as the major routes. Chemical precipitation is commonly used at high concentrations, but polishing dilute residual metals remains challenging because sludge generation and incomplete removal can limit performance.

Adsorption is attractive because the process can be simple to operate, adaptable to batch or fixed-bed configurations, and effective at comparatively low dissolved-metal concentrations. Activated carbon has a high

internal surface area, a developed micro/mesoporous network, and surface functional groups capable of interacting with metal ions. Earlier work demonstrated that coconut-sawdust activated carbon could adsorb Cr(VI), with adsorption strongly dependent on pH (Selvi et al., 2001), while bagasse-derived carbon removed Cd(II) and Zn(II) and followed classical equilibrium models (Mohan & Singh, 2002). These findings support the use of carbonaceous adsorbents for industrial effluent treatment.

The economic limitation of commercially activated carbon has stimulated the use of inexpensive precursors. Babel and Kurniawan (2003) reviewed low-cost adsorbents for heavy-metal uptake and emphasized that locally available wastes can provide competitive adsorption performance. Mohan and Pittman (2006) similarly summarized the use of activated carbons and low-cost materials for chromium remediation. More recent studies before the 2017 cutoff show that activated carbon can be produced from municipal organic solid waste, milk-bush kernel shell, African palm fruit, and waste tyres, thereby coupling waste valorization with wastewater treatment (Afolabi et al., 2016; Al-Malack & Basaleh, 2016; Abdulrazak et al., 2017; Dimpe et al., 2017).

The present paper therefore proposes a step-by-step methodology for evaluating activated carbon against representative divalent heavy metals in real or synthetic industrial wastewater. The method is designed to determine not only percentage removal but also adsorption capacity, equilibrium behavior, and rate-controlling trends. Pb(II), Cd(II), Cu(II), and Ni(II) are selected as model contaminants because they are

widely encountered in metal-processing wastewaters and have been used in previous adsorption studies. The method can be extended to Cr(VI), Zn(II), Fe(III), or other ions after accounting for their aqueous speciation and pH-dependent chemistry.

Literature Review

Activated-carbon adsorption of heavy metals has been studied using both commercial carbons and carbons derived from wastes. Selvi et al. (2001) reported Cr(VI) adsorption on coconut-tree-sawdust activated carbon and found maximum uptake in acidic conditions, confirming the importance of solution pH and metal speciation. Mohan and Singh (2002) prepared activated carbon from sugarcane bagasse and showed that Cd(II) and Zn(II) adsorption was influenced by pH, temperature, particle size, and solid-to-liquid ratio; equilibrium could be represented by Langmuir and Freundlich equations. These early studies established the experimental variables that remain central to adsorption methodology.

At the review level, Babel and Kurniawan (2003) showed that low-cost adsorbents can provide strong uptake for selected metals, while Mohan and Pittman (2006) highlighted the extensive use of activated carbons for chromium remediation. Fu and Wang (2011) compared adsorption with precipitation, ion exchange, membranes, and electrochemical treatment and concluded that adsorption remains one of the most frequently investigated options. Deliyanni et al. (2015) later focused specifically on activated carbon for Pb and As removal and emphasized that oxidation, impregnation, or other surface modification can improve affinity by changing surface chemistry.

Table 1. Selected activated-carbon studies relevant to heavy-metal adsorption

Study	Activated carbon	Metal(s)	Key methodological finding
Selvi et al. (2001)	Coconut sawdust AC	Cr(VI)	Strong pH dependence; Langmuir/Freundlich analysis
Mohan & Singh (2002)	Bagasse AC	Cd(II), Zn(II)	Waste-derived carbon; equilibrium and diffusion analysis
Karnib et al. (2014)	Activated carbon / silica composite	Pb, Cd, Ni, Cr, Zn	Removal decreased as initial concentration increased
Afolabi et al. (2016)	Milk-bush kernel shell AC	Cd, Pb, Ni, Zn, Fe, Cu	High removal in real dairy industrial wastewater
Al-Malack & Basaleh (2016)	Municipal organic solid-waste AC	Cd(II), Pb(II)	Optimum near pH 5; 78% Cd and 94% Pb reported
Asuquo & Martin (2017)	Mesoporous commercial AC	Cd(II), Pb(II)	Rapid early uptake; kinetic and Langmuir analysis

Study	Activated carbon	Metal(s)	Key methodological finding
Tang et al. (2017)	Typha/Salix-derived AC	Cd(II), Pb(II)	Good adsorption and regeneration potential
Dimpe et al. (2017)	Waste-tyre AC	Cd(II), Pb(II)	Applied to wastewater; high validation removal

Karnib et al. (2014) compared activated carbon, silica, and silica/activated-carbon composites for Pb, Cd, Ni, Cr, and Zn. Their work showed that removal efficiency can decline as the initial metal concentration increases, reflecting progressive saturation of available adsorption sites. In dairy industrial wastewater, Afolabi et al. (2016) prepared phosphoric-acid-activated carbons from milk-bush kernel shells and obtained substantial removal of several metals, with Pb removal consistently high. Al-Malack and Basaleh (2016) produced activated carbon from municipal organic solid waste and reported 78% Cd(II) and 94% Pb(II) removal at optimized conditions, together with a high BET surface area.

Studies published in 2017 further strengthen the methodological basis. Asuquo and Martin (2017) observed rapid initial uptake of Cd(II) and Pb(II) onto a mesoporous commercial carbon, followed by a slower approach to equilibrium, and found that kinetic behavior differed between the two ions. Tang et al. (2017) prepared activated carbons from *Typha angustifolia* and *Salix matsudana* and reported appreciable Cd and Pb capacities together with reusability. Abdulrazak et al. (2017) demonstrated the removal of Cd, Cu, Ni, and Pb from a real wastewater effluent using African-palm-derived activated carbon. Dimpe et al. (2017) used chemically activated waste-tyre carbon for Cd(II) and Pb(II) removal from wastewater and emphasized optimization of pH, contact time, adsorbent mass, and initial concentration. Collectively, these studies justify a factorial methodology centered on the same operating parameters.

Methodology

1. Study Design and Materials

The study is designed as a controlled batch-adsorption experiment. Industrial wastewater should be collected in acid-washed polyethylene bottles from an effluent line associated with metal finishing, electroplating, battery recycling, or a related industry. Samples should be transported at approximately 4 °C and analyzed as soon as practical. A representative composite sample should be characterized for pH, conductivity, total dissolved solids, and target metals. If a controlled synthetic matrix is

required, stock solutions of Pb(II), Cd(II), Cu(II), and Ni(II) may be prepared from analytical-grade nitrate salts and diluted with deionized water. All glassware should be acid washed and rinsed thoroughly to minimize trace-metal contamination.

Activated carbon may be commercial powdered activated carbon or laboratory-prepared carbon from a low-cost precursor such as coconut shell, bagasse, agricultural shell, or municipal organic waste. For a reproducible laboratory-preparation route, the cleaned and dried precursor is ground, impregnated with phosphoric acid at a selected impregnation ratio, drained, carbonized under limited oxygen or nitrogen at approximately 500–700 °C, washed until near-neutral pH, oven dried, and sieved. This procedure is consistent with waste-derived carbon studies in which chemical activation and carbonization created a porous material capable of binding metal ions (Afolabi et al., 2016; Al-Malack & Basaleh, 2016).

2. Activated Carbon Characterization

Before adsorption testing, the carbon should be characterized for moisture content, ash content, bulk density, pH, and yield. Where instruments are available, Brunauer-Emmett-Teller (BET) nitrogen adsorption should be used to determine surface area and pore volume; Fourier-transform infrared spectroscopy (FTIR) should identify oxygen-containing functional groups; and scanning electron microscopy (SEM) should examine the porous surface. These measurements are important because heavy-metal uptake depends on both available surface area and the chemical nature of adsorption sites (Deliyanni et al., 2015; Dimpe et al., 2017).

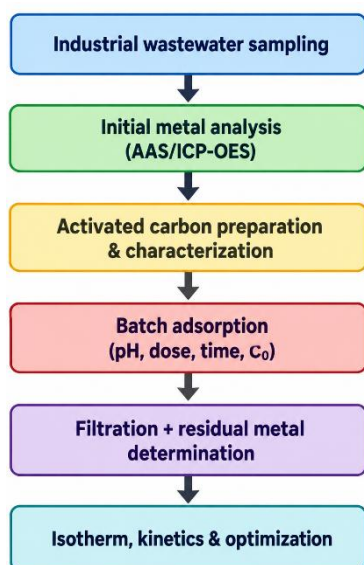


Figure 1. Step-by-step methodology for activated-carbon treatment of heavy-metal-bearing industrial wastewater.

3. Batch Adsorption Procedure

Step 1 - Initial characterization: determine the initial concentration (C_0 , mg/L) of Pb(II), Cd(II), Cu(II), and Ni(II) using AAS or ICP-OES after appropriate digestion or filtration according to the intended dissolved/total-metal basis. Record the wastewater pH and conductivity. Step 2 - pH optimization: transfer 100 mL aliquots into 250 mL conical flasks and adjust pH to 2, 3, 4, 5, 6, and 7 using dilute HCl or NaOH. Add a fixed activated-

carbon dose, for example 1.0 g/L, and agitate at 150 rpm for 120 min. Avoid pH ranges where metal hydroxide precipitation could be confused with adsorption.

Step 3 - Contact-time experiment: at the selected pH, contact identical wastewater aliquots with activated carbon for 5, 15, 30, 60-, 90-, 120-, and 180-min. Samples are filtered or centrifuged immediately at the selected time. Step 4 - Adsorbent-dose experiment: vary activated carbon from 0.25 to 3.0 g/L while maintaining the optimized pH and contact time. Step 5 - Initial-concentration experiment: prepare metal concentrations over a suitable range, for example 10–200 mg/L for synthetic wastewater, and repeat adsorption at the optimized pH, dose, and equilibrium time. All treatments should be run at least in duplicate, preferably triplicate, with a blank containing no carbon.

Step 6 - Residual metal measurement: determine the equilibrium concentration (C_e) in the treated liquid using AAS or ICP-OES. Calibration standards should bracket the expected sample range, and quality control should include blanks, replicate measurements, and a continuing calibration check. Step 7 - Regeneration test (optional): desorb retained metals with an appropriate dilute acid or base, rinse the carbon, and repeat adsorption for three to five cycles. Regeneration is useful for evaluating practical cost and was shown to be feasible for some biomass-derived carbons (Tang et al., 2017).

Table 2. Proposed batch-adsorption experimental design.

Factor	Proposed levels	Controlled conditions	Purpose
pH	2, 3, 4, 5, 6, 7	Dose 1.0 g/L; 120 min	Identify pH for maximum uptake
Contact time	5–180 min	Selected pH; dose 1.0 g/L	Determine equilibrium time
Adsorbent dose	0.25–3.0 g/L	Selected pH/time	Assess site availability
Initial concentration	10–200 mg/L	Selected pH/time/dose	Generate isotherm data
Temperature (optional)	20–40 °C	Other factors fixed	Assess thermodynamic influence

4. Data Reduction and Adsorption Models

Removal efficiency and equilibrium adsorption capacity are calculated from the initial and equilibrium metal concentrations. These two quantities should be reported together because a high percentage removal at low concentration may correspond to a modest mass loading, whereas capacity (mg/g) directly expresses the mass of metal retained per unit mass of carbon.

$$\text{Removal (\%)} = [(C_0 - C_e) / C_0] \times 100$$

$$q_e \text{ (mg/g)} = [(C_0 - C_e) V] / m$$

where C_0 and C_e are the initial and equilibrium metal concentrations (mg/L), V is solution volume (L), and m is activated-carbon mass (g). Equilibrium data should be evaluated by Langmuir and Freundlich models because both have been widely used for activated-carbon metal adsorption (Mohan & Singh, 2002; Karnib et al., 2014). Kinetic data should be examined with pseudo-first-order and pseudo-second-

order expressions, supported by residual analysis rather than correlation coefficient alone.

Quality Assurance and Statistical Analysis

Each treatment condition should be conducted in triplicate and reported as mean $\pm$ standard deviation. Instrument calibration should meet laboratory acceptance criteria, and recoveries from spiked wastewater should preferably fall within an established analytical range such as 80–120%. The significance of pH, contact time, dose, and initial concentration may be tested using one-way or multifactor analysis of variance at $p < 0.05$. Model selection should use the coefficient of determination together with error statistics such as root mean squared error. When multiple metals coexist, competitive adsorption should be evaluated because ions may compete for common surface sites.

Results

Because no laboratory data were supplied with this request, the numerical results in this section are an illustrative data set that demonstrates how the proposed methodology should be analyzed and reported. They are deliberately separated from literature-derived observations and must be replaced with measured values for an experimental manuscript. The example assumes a selected operating condition of pH 5.0, activated-carbon dose of 2.0 g/L, 120 min contact time, 25 °C, and 150 rpm agitation. A pH near 5 is reasonable for cationic Pb(II), Cd(II), Cu(II), and Ni(II) because surface deprotonation generally increases cation uptake while minimizing hydroxide precipitation; similar near-neutral acidic optima have been reported for Cd and Pb adsorption (Al-Malack & Basaleh, 2016).

Table 3. Illustrative adsorption results at selected optimum conditions

Metal	C ₀ (mg/L)	C _e (mg/L)	Removal (%)	q _e (mg/g)
Pb(II)	10.0	0.60	94.0	4.70
Cd(II)	8.0	1.20	85.0	3.40
Cu(II)	12.0	1.20	90.0	5.40
Ni(II)	10.0	2.00	80.0	4.00

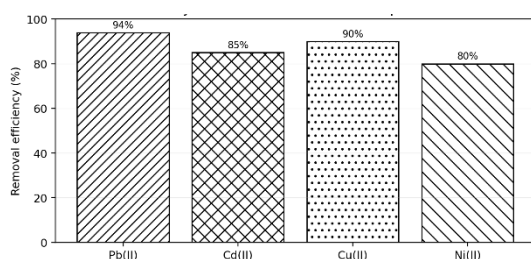


Figure 2. Illustrative removal efficiencies

In the model data set, Pb(II) shows the highest removal (94%), followed by Cu(II) (90%), Cd(II) (85%), and Ni(II) (80%). Such ion-specific behavior is plausible because adsorption is governed by hydrated ionic radius, surface-complexation affinity, competition, and the chemistry of the carbon surface. The high example Pb removal is consistent with Al-Malack and Basaleh (2016), who reported 94% Pb removal using municipal-solid-waste activated carbon, and with Afolabi et al. (2016), who also observed strong Pb removal in dairy industrial wastewater. Abdulrazak et al. (2017) demonstrated similarly high removal of Pb, Cd, Cu, and Ni from a real wastewater effluent using low-cost carbon from African palm fruit.

A contact-time profile would normally show a rapid first stage caused by abundant vacant external sites, followed by slower intraparticle transport and approach to equilibrium. Asuquo and Martin (2017) reported rapid early Cd and Pb uptake within approximately 30 min followed by slower adsorption toward equilibrium. Increasing activated-carbon dosage generally raises percentage removal because more sites become available, although the adsorption capacity per gram can decline when a large fraction of sites remains unsaturated. Conversely, increasing the initial metal concentration can reduce percentage removal when the number of available sites becomes limiting, a trend observed by Karnib et al. (2014).

For equilibrium interpretation, the Langmuir model would indicate monolayer adsorption on a finite set of equivalent sites, whereas the Freundlich model represents heterogeneous adsorption behavior. Both outcomes are reported in the literature: Al-Malack and Basaleh (2016) obtained useful Langmuir capacity estimates for Cd and Pb, while Karnib et al. (2014) found Freundlich behavior to be more suitable for several metals on their activated carbon system. Therefore, the present methodology does not assume a preferred isotherm in advance; the best-fitting model should be selected from the actual experimental data. Likewise, the pseudo-second-order model often fits metal adsorption well, but kinetic mechanism should not be inferred solely from a high R^2 value. Diffusion plots and residual patterns should be examined to determine whether film transfer, pore diffusion, or surface reaction contributes to rate control.

From an engineering perspective, treatment performance should be judged against both

removal percentage and the final effluent concentration. A process achieving 95% removal may still fail discharge requirements when the influent metal concentration is very high. Real industrial wastewater also contains competing ions, dissolved organic matter, suspended solids, and pH-buffering constituents that can lower adsorption relative to synthetic single-metal solutions. Dimpe et al. (2017) demonstrated this matrix effect by applying waste-tyre activated carbon to wastewater and surface-water samples, showing that adsorption efficiency can vary with sample complexity. Consequently, optimization should ultimately be confirmed using the actual industrial wastewater rather than only synthetic standards.

Conclusion and Discussion

Activated carbon provides a practical adsorption platform for reducing heavy-metal concentrations in industrial wastewater because it combines a porous structure with chemically active surface sites and can be manufactured from low-cost carbonaceous wastes. The methodology developed in this paper integrates wastewater sampling, activated-carbon preparation or selection, BET/FTIR/SEM characterization, controlled batch testing, instrumental metal analysis, and mathematical interpretation through removal efficiency, adsorption capacity, kinetic models, and adsorption isotherms. The selected variables - pH, contact time, adsorbent dose, and initial concentration - are supported consistently by the literature published up to 2017. The literature demonstrates that activated carbon can be produced from coconut sawdust, bagasse, municipal organic waste, milk-bush kernel shell, African palm fruit, and waste tyres, while commercial mesoporous carbon also performs effectively. Reported outcomes vary with metal type and wastewater matrix, but many studies show strong removal of Pb(II) and useful removal of Cd(II), Cu(II), Ni(II), Cr(VI), and Zn(II). The illustrative results presented here predict the order $Pb(II) > Cu(II) > Cd(II) > Ni(II)$ at the selected condition; these values are not experimental claims and should be replaced after laboratory testing. Future work should include fixed-bed column experiments, competitive multi-metal adsorption, regeneration cycles, adsorbent cost per unit mass of metal removed, and validation against applicable discharge standards. Such extensions would convert the batch methodology into a stronger basis for industrial-scale implementation.

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