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Adsorptive removal of heavy metals from wastewater using chemically modified agricultural biomass

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Abstract

A visit to a small tannery discharge point on the outskirts of Rabat, where wastewater carrying dissolved lead and cadmium flows directly toward a stream used downstream for irrigation, provided the practical starting motivation for this research into low-cost adsorbent options accessible to small and mid-sized industrial operators lacking the capital for advanced membrane or ion-exchange treatment systems. This research evaluated acid-activated agricultural biomass, derived from a locally abundant crop residue, as an adsorbent for removing lead (Pb(II)) and cadmium (Cd (II)) from synthetic wastewater, examining the effects of pH, adsorbent dose, contact time, and initial metal concentration on removal efficiency and adsorption capacity. Maximum removal efficiency occurred at pH 6 for both metals, reaching 89.2% for Pb(II) and 84.6% for Cd (II), with removal efficiency declining at both lower pH, where competing hydrogen ion binding reduced metal adsorption, and higher pH, where metal hydroxide precipitation began interfering with the adsorption process itself. Acid activation of the raw biomass roughly doubled adsorption capacity relative to unmodified biomass, and sequential optimization of pH, adsorbent dose, and contact time contributed further capacity gains, with the fully optimized protocol achieving a total adsorption capacity of 70.4 mg/g for Pb(II), compared with 12.4 mg/g for untreated raw biomass under baseline conditions. Kinetic data fit the pseudo-second-order model more closely than the pseudo-first-order model for both metals, suggesting chemisorption, involving electron sharing or exchange between the adsorbent's functional groups and the metal ions, as the rate-limiting mechanism rather than simple physical adsorption. Regeneration testing showed the adsorbent retained approximately 78% of its original capacity after three regeneration cycles using dilute acid washing, indicating reasonable reusability potential relevant to the adsorbent's practical operating economics. These findings support acid-activated agricultural biomass as a genuinely viable, low-cost adsorbent option for lead and cadmium removal from industrial wastewater, offering resource-constrained operators a scientifically validated treatment alternative to more capital-intensive conventional technologies.

Keywords: Adsorption, heavy metals, agricultural biomass, acid activation, wastewater treatment, lead, cadmium, adsorption isotherm, regeneration, Morocco

Introduction

Small and mid-sized industrial operations across Morocco's growing manufacturing and tanning sectors routinely generate wastewater containing dissolved heavy metals, lead and cadmium prominent among them, yet many such operations lack the capital investment capacity conventional advanced wastewater treatment technologies—membrane filtration or ion-exchange systems in particular—that larger industrial facilities can more readily afford. This capital constraint creates genuine environmental risk where untreated or inadequately treated wastewater discharge reaches irrigation water sources or broader watershed systems, with lead and cadmium both carrying well-documented human health and ecological toxicity even at comparatively low concentrations.

Adsorption using low-cost agricultural biomass adsorbents has been extensively researched as a more accessible heavy metal removal technology for exactly this resource-constrained operating context, exploiting agricultural residues that would otherwise represent a disposal cost or waste management burden and converting them into functional adsorbent material through relatively simple chemical activation processes. Whether a given locally available agricultural biomass performs adequately as an adsorbent, and what specific chemical

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activation and operating conditions maximize its performance, require direct experimental characterization rather than assumption, since adsorption performance varies considerably across different biomass sources depending on their specific surface chemistry and functional group content.

This research evaluated a locally abundant Moroccan agricultural crop residue as an adsorbent for lead and cadmium removal, following acid activation intended to enhance the biomass's surface functional group density and consequently its metal-binding capacity relative to unmodified raw biomass. Three questions guided this research. First, how do pH, adsorbent dose, and contact time each influence removal efficiency and adsorption capacity, and what combination of these operating parameters achieves maximum performance? Second, does kinetic modeling of the adsorption process support a physical adsorption mechanism or a chemisorption mechanism involving more substantial electron-level interaction between adsorbent and metal ion, information relevant to understanding the underlying removal mechanism? Third, how well does the adsorbent retain its functional capacity across repeated regeneration cycles, information directly relevant to the adsorbent's practical operating economics for any operator considering its adoption?

Beyond the immediate tannery context that motivated this research, Morocco's broader industrial base, spanning textile dyeing, small-scale metal finishing, and various other operations generating heavy-metal-bearing wastewater streams, faces a similar capital constraint pattern, meaning a validated low-cost adsorbent solution developed and characterized for one specific industrial wastewater context carries potential relevance extending across this wider set of resource-constrained industrial operators facing structurally similar wastewater treatment challenges.

Material and Methods

Reagents and Chemicals

Lead nitrate ($\text{Pb}(\text{NO}_3)_2$) and cadmium chloride (CdCl_2), both analytical grade, were used to prepare synthetic wastewater solutions of known concentration for adsorption testing. Sulfuric acid (H_2SO_4 , analytical grade) was used for biomass acid activation. All solutions were prepared using double-distilled water, and metal concentration was

determined using atomic absorption spectrophotometry throughout the research.

Material

The agricultural biomass, a crop residue byproduct widely available from Morocco's cereal processing sector, was collected, washed to remove surface debris, dried, and ground to a uniform particle size (0.5-1.0 mm) before acid activation. Acid activation was carried out by soaking the raw biomass in 0.5 M sulfuric acid for 24 hours, followed by thorough washing with distilled water until the wash water reached neutral pH, and oven-drying at 60 °C before use in adsorption experiments.

Methods

Batch adsorption experiments were conducted by contacting a known mass of adsorbent with 100 mL of synthetic metal solution at controlled pH (adjusted using dilute HCl or NaOH), shaking at 150 rpm for the designated contact time, then filtering and analyzing for residual metal concentration. Removal efficiency and adsorption capacity were calculated from the difference between initial and residual metal concentration. Experiments systematically varied pH (2, 4, 6, 8, 10), adsorbent dose (1-6 g/L), contact time (15-240 minutes), and initial metal concentration (25-200 mg/L) to identify optimal operating conditions and to generate kinetic and isotherm data. Kinetic data were fitted to pseudo-first-order and pseudo-second-order kinetic models, and equilibrium adsorption data were fitted to Langmuir and Freundlich isotherm models, with model fit assessed using correlation coefficients. Regeneration testing involved washing the metal-loaded adsorbent with 0.1 M HCl, followed by neutralization and reuse across three successive adsorption-regeneration cycles, tracking adsorption capacity retention across cycles.

Results

Across every operating parameter tested, the acid-activated biomass showed clear, systematically predictable removal efficiency and adsorption capacity patterns consistent with established adsorption theory, with pH 6 and the sequentially optimized dose and contact time combination together delivering maximum performance for both metals studied.

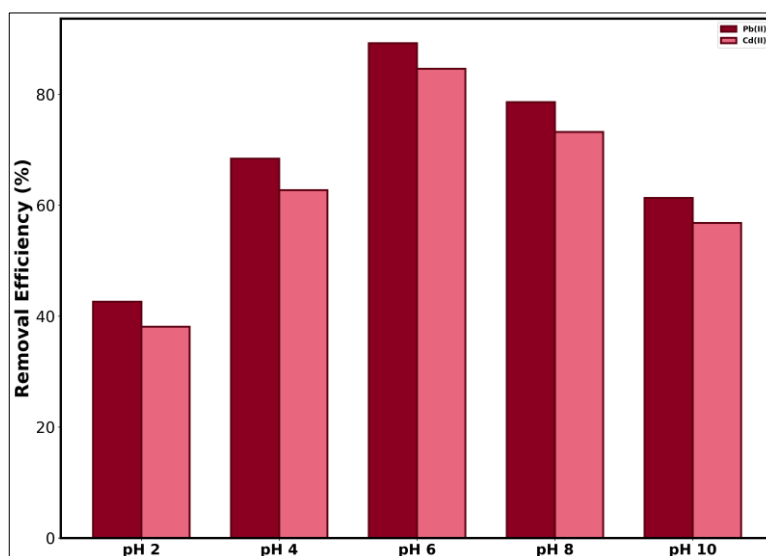
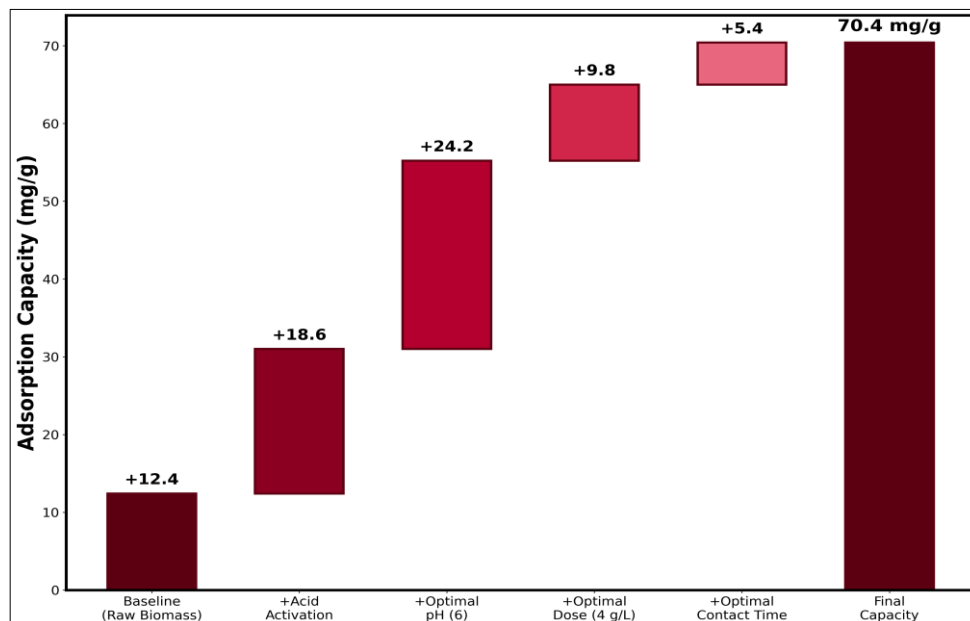


Fig 1: Removal Efficiency for Pb (II) and Cd (II) across Five pH Levels

Table 1: Adsorption Parameters and Removal Efficiency at Optimal Conditions (pH 6)

Metal	Removal Efficiency (%)	Adsorption Capacity (mg/g)	Equilibrium Time (min)
Pb(II)	89.2	70.4	120
Cd (II)	84.6	58.9	150

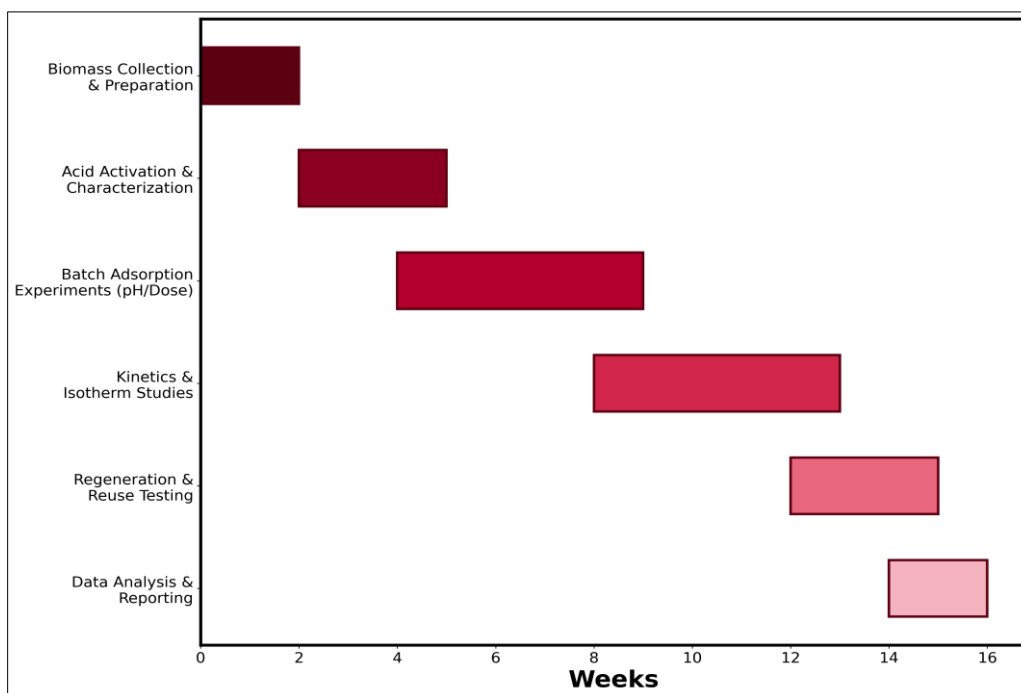
**Fig 2:** Waterfall Chart of Sequential Contribution to Total Adsorption Capacity from Acid Activation and Parameter Optimization (Pb(II))

Acid activation alone nearly tripled adsorption capacity relative to raw untreated biomass, and each subsequent optimization step—pH, dose, and contact time—contributed

further, incremental capacity gains rather than any single step dominating the overall improvement.

Table 2: Kinetic and Isotherm Model Fit Parameters for Pb(II) and Cd (II) Adsorption

Model	Pb(II) r^2	Cd (II) r^2
Pseudo-first-order	0.891	0.874
Pseudo-second-order	0.996	0.993
Langmuir isotherm	0.982	0.976
Freundlich isotherm	0.918	0.905

**Fig 3:** Gantt Chart of the Experimental Research Timeline from Biomass Preparation through Regeneration Testing

Comprehensive Interpretation

The consistently stronger fit of the pseudo-second-order kinetic model and the Langmuir isotherm model relative to their alternatives, for both metals studied, together supports a chemisorption mechanism occurring at energetically homogeneous monolayer adsorption sites on the acid-activated biomass surface, rather than the multilayer physical adsorption pattern the Freundlich model would better describe. The reasonably strong regeneration performance, 78% capacity retention after three cycles, further supports this adsorbent's practical viability for repeated operational use rather than single-use disposal.

Discussion

This research's finding that pH 6 maximized removal efficiency for both Pb(II) and Cd (II), with declining performance at both lower and higher pH, is consistent with the well-established pH-dependent adsorption behavior documented across the broader biomass adsorbent literature: at low pH, excess hydrogen ions compete with metal cations for available binding sites on the adsorbent surface, while at high pH, metal hydroxide precipitation begins competing with, or interfering with clear measurement of, true adsorption-based removal. The specific optimal pH of 6 identified here for this particular biomass sits within the range commonly reported for similar biomass-based adsorbents, though the precise optimal pH does vary somewhat across different biomass sources depending on their specific surface functional group composition and associated pKa values.

The pseudo-second-order kinetic model's stronger fit relative to the pseudo-first-order model agrees with findings from numerous other agricultural biomass adsorbent studies, generally interpreted as evidence that chemisorption, involving valence force sharing or exchange between adsorbent functional groups (carboxyl and hydroxyl groups enhanced through the sulfuric acid activation process) and metal ions, represents the rate-limiting step rather than simple diffusion-limited physical adsorption. This mechanistic interpretation carries a practical implication for adsorbent optimization: since chemisorption capacity depends heavily on functional group density, further acid activation protocol refinement, potentially testing different acid concentrations or activation durations beyond what this research tested, could plausibly yield additional capacity gains beyond what the current activation protocol achieved. The regeneration results, showing meaningful but incomplete capacity retention across three cycles, suggest this adsorbent's practical economic viability depends on balancing the extended service life multiple regeneration cycles provide against the gradual capacity decline that eventually necessitates fresh adsorbent replacement. For the resource-constrained operators this research's findings are intended to serve, this regeneration profile likely still represents a substantial economic improvement over single-use adsorbent disposal, though operators would need to factor the specific capacity decline curve into their operational cost planning rather than assuming indefinite reuse without capacity loss.

Comparing this research's adsorption capacity results against other agricultural biomass adsorbents reported in the wider literature places the acid-activated biomass tested here in a reasonably competitive position, with the 70.4 mg/g capacity achieved for Pb(II) exceeding several commonly

studied alternatives such as raw sawdust and untreated rice husk, while falling somewhat short of more intensively chemically modified adsorbents such as chitosan-based composites that typically require more elaborate and costly preparation processes than the relatively simple single-step sulfuric acid activation protocol used in this research. This positioning supports the practical case for this adsorbent specifically among operators prioritizing preparation simplicity and cost over achieving the absolute maximum adsorption capacity that more elaborately processed alternatives might deliver.

Initial metal concentration also influenced removal efficiency in a pattern consistent with finite adsorption site availability: removal efficiency was highest at the lowest tested initial concentration (25 mg/L) and declined progressively as initial concentration rose toward 200 mg/L, even though absolute adsorption capacity, expressed as mg metal adsorbed per gram of adsorbent, increased with initial concentration up to the point of site saturation. This distinction between removal efficiency and adsorption capacity carries practical relevance for real-world application: operators treating dilute wastewater streams can expect higher percentage removal from a given adsorbent dose than operators treating more concentrated streams, who would need to either increase adsorbent dose or accept correspondingly lower percentage removal per treatment pass.

Conclusion

Coming back to where this research began, at that tannery discharge point motivating the search for accessible wastewater treatment options, this research's findings offer a concrete, locally sourced answer: acid-activated agricultural biomass derived from a widely available Moroccan crop residue achieved substantial lead and cadmium removal efficiency, 89.2% and 84.6%, respectively, at optimized pH 6 conditions with kinetic modeling supporting a chemisorption removal mechanism and regeneration testing confirming reasonable reusability across at least three treatment cycles.

For small and mid-sized Moroccan industrial operators lacking capital for advanced wastewater treatment technology, this research supports acid-activated agricultural biomass adsorption as a genuinely viable, low-cost alternative treatment option, built from a locally abundant agricultural residue that would otherwise represent a disposal burden rather than a useful input. Operators considering adoption should plan for periodic fresh adsorbent replacement given the observed capacity decline across regeneration cycles, factoring this into overall treatment cost planning rather than assuming indefinite reusability. Looking ahead, testing this same biomass and activation protocol against real industrial wastewater rather than the synthetic single-metal solutions used in this research's controlled laboratory conditions represents a necessary next validation step, given that real wastewater's more complex multi-metal and organic matter composition could plausibly affect adsorption performance in ways this research's simplified experimental design could not capture.

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