

RESEARCH ARTICLE

Adsorptive Removal of Heavy Metals from Industrial Wastewater Using Modified Agricultural Waste-Derived Biochar: An Isotherm, Kinetic and Thermodynamic Modelling Study

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ABSTRACT

Agricultural residues converted to biochar are among the most frequently proposed low-cost adsorbents for heavy metal removal, but the parameters reported for them are difficult to compare because model selection is often made on the coefficient of determination of a linearised transform, which is known to distort the error structure of the underlying data. This article applies a consistent non-linear fitting protocol to a declared synthetic dataset representing phosphoric-acid-modified rice husk biochar in contact with lead, cadmium and copper. Equilibrium data were generated from a Langmuir surface with stated capacities and affinity constants and 3 % multiplicative dispersion, then fitted by the Langmuir, Freundlich, Temkin and Dubinin–Radushkevich models without linearisation. The Langmuir model was preferred for all three metals, returning monolayer capacities of 108.9, 78.1 and 63.1 mg g⁻¹ for lead, cadmium and copper at coefficients of determination of 0.9976, 0.9929 and 0.9925, against Freundlich values of 0.9045, 0.9538 and 0.9620. Dubinin–Radushkevich mean sorption energies of 13.46, 11.66 and 10.53 kJ mol⁻¹ place all three systems in the chemisorption regime. Kinetic data generated from a pseudo-second-order process were fitted by both the pseudo-first-order and pseudo-second-order forms; the latter was preferred throughout, with coefficients of determination of 0.9855 to 0.9976 against 0.9040 to 0.9641. Van 't Hoff analysis returned an enthalpy of 18.5 kJ mol⁻¹ with free energies from -19.9 to -27.3 kJ mol⁻¹, indicating a spontaneous, endothermic and entropy-driven process.

Keywords: *biochar; heavy metal removal; adsorption isotherms; pseudo-second-order kinetics; waste valorisation; industrial wastewater; thermodynamic analysis*

1. INTRODUCTION

Electroplating, battery manufacture, tannery and pigment operations discharge effluents carrying lead, cadmium and copper at concentrations far above the limits set for drinking water and, in many cases, above the limits set for industrial discharge [2], [24]. Unlike organic contaminants, these species cannot be degraded; they can only be transferred to a solid phase and immobilised. Chemical precipitation, the conventional treatment, is effective at high concentration but leaves residual metal in the tens of milligrams per litre and generates a hydroxide sludge that is itself a hazardous waste. Adsorption is the usual polishing step, and its economics are dominated by the cost of the adsorbent.

Biochar, the carbonaceous solid recovered from the pyrolysis of biomass under limited oxygen, has attracted attention because the feedstock is a waste stream rather than a commodity [1], [13]. Rice husk is particularly relevant in South and South-East Asia, where it is generated in large volume at the mill and is frequently burned in the open. Pyrolysis converts it into a porous, oxygen-functionalised solid

whose capacity for divalent metals can be raised substantially by chemical activation, most commonly with phosphoric acid or potassium hydroxide [17], [19], [22]. The mechanisms invoked include complexation with surface carboxyl and phenolic groups, cation exchange with mineral-phase calcium and magnesium, precipitation as phosphates or carbonates, and coordination with π -electrons of aromatic domains [9], [14], [15].

The obstacle to using this literature is methodological rather than chemical. Isotherm and kinetic models are still very commonly fitted in linearised form, and the model is then selected on the coefficient of determination of that transform. The transforms redistribute the error unevenly across the concentration range, so the ranking obtained is a property of the transform as much as of the data; this has been documented repeatedly and remains widespread [5], [21]. A second problem is that thermodynamic quantities are computed from equilibrium constants whose units are not made dimensionless in a consistent way, so reported enthalpies and entropies are not comparable between studies.

This article applies a single consistent protocol to a fully declared dataset. Equilibrium and kinetic data are generated from stated models with stated parameters and stated dispersion; every model is then fitted by non-linear least squares on the untransformed data; model selection is made on both the coefficient of determination and a chi-squared statistic; and thermodynamic constants are rendered dimensionless by an explicitly stated convention. No adsorbent was prepared and no solution was analysed. The contribution is the protocol and the demonstration of how much the conclusions depend on it.

2. BACKGROUND AND RELATED WORK

The equilibrium models used here are classical. The Langmuir isotherm assumes a fixed number of energetically equivalent sites and no lateral interaction, and yields a monolayer capacity and an affinity constant [12]. The Freundlich isotherm is empirical and describes adsorption on a heterogeneous surface with an exponentially decaying site-energy distribution [6]. The Temkin isotherm introduces a linear fall in the heat of adsorption with coverage arising from adsorbate–adsorbate interaction [20]. The Dubinin–Radushkevich isotherm, expressed in terms of the Polanyi potential, yields a mean sorption energy whose magnitude is conventionally used to distinguish physical from chemical adsorption [4]. The separation factor derived from the Langmuir constant provides a compact statement of isotherm favourability [7].

The kinetic models are equally standard. The pseudo-first-order rate equation was introduced for the sorption of dissolved substances [11]; the pseudo-second-order form, which assumes that the rate-limiting step involves valency forces through sharing or exchange of electrons, has become dominant in the metal sorption literature [8]. The Weber–Morris intraparticle diffusion plot is used to identify the

successive resistances — film diffusion, pore diffusion and equilibration — that control the approach to equilibrium [23].

Applied studies of biochar for metal removal report monolayer capacities that vary over more than an order of magnitude with feedstock and activation route. Hardwood and corn straw chars have been characterised for copper and zinc [3], and comparative kinetic and adsorptive work across several chars has been reported [10]. Reviews consolidate the field and note both the promise of the material and the inconsistency of its characterisation [9], [19], [25]. Surface chemistry underlying metal binding to oxygenated carbon surfaces is treated in the standard soil chemistry literature [18], and the design of engineered chars for specific contaminants has been reviewed [16], [17].

3. MATERIALS AND METHODS

3.1 Represented adsorbent and provenance of the data

The material represented by the model is a rice husk biochar pyrolysed at 500 °C under nitrogen and subsequently activated with phosphoric acid at an impregnation ratio of 1:1, washed to neutrality and dried. No such material was prepared for this study. The equilibrium and kinetic datasets analysed in Section 4 were generated numerically from the models and parameters declared in Sections 3.2 and 3.3, using the NumPy pseudo-random generator seeded at 20260303, with multiplicative Gaussian dispersion at relative standard deviations of 3 % for the equilibrium data and 2 % for the kinetic data. All fitting was performed on the untransformed data by non-linear least squares.

3.2 Equilibrium models and generating parameters

Equilibrium data were generated at eight residual concentrations between 2 and 130 mg L⁻¹ from a Langmuir surface with monolayer capacities of 112.0, 78.5 and 61.3 mg g⁻¹ and affinity constants of 0.091, 0.062 and 0.048 L mg⁻¹ for lead, cadmium and copper respectively. The four candidate isotherms were then fitted:

$$q_e = q_m K_L C_e / (1 + K_L C_e); \quad q_e = K_F C_e^{1/n}; \quad q_e = B_T \ln(A_T C_e); \quad q_e = q_s \exp(-\beta \varepsilon^2) \quad (1)$$

with $\varepsilon = RT \ln(1 + 1/C_e)$ evaluated with the residual concentration expressed in mol L⁻¹, and the mean sorption energy obtained as $E = (2\beta)^{-1/2}$. Model discrimination used both the coefficient of determination and the chi-squared statistic $\Sigma(q_{\text{obs}} - q_{\text{calc}})^2/q_{\text{calc}}$. The separation factor $R_L = 1/(1 + K_L C_0)$ was evaluated at three initial concentrations.

3.3 Kinetic model and generating parameters

Kinetic data were generated at nine contact times between 5 and 180 min for an initial concentration of 50 mg L⁻¹ and an adsorbent dose of 1 g L⁻¹, from a pseudo-second-order process with equilibrium capacities of 45.8, 33.6 and 26.4 mg g⁻¹ and rate constants of 0.0042, 0.0031 and 0.0027 g mg⁻¹ min⁻¹. The generated data were fitted by both kinetic forms:

$$q_t = q_e [1 - \exp(-k_1 t)]; \quad q_t = k_2 q_e^2 t / (1 + k_2 q_e t) \quad (2)$$

and the initial sorption rate was obtained as $h = k_2 q_e^2$. The Weber–Morris intraparticle diffusion constant was obtained by regression of q_t on $t^{1/2}$ over the first five time points, the intercept of that regression measuring the thickness of the boundary layer.

3.4 Thermodynamic treatment

Affinity constants were generated at 298, 308 and 318 K from the stated 298 K values and a declared standard enthalpy of 18.5 kJ mol⁻¹. Each was rendered dimensionless by multiplying the Langmuir constant in L mg⁻¹ by the molar mass of the metal and by 10³, which corresponds to a standard state of 1 mol L⁻¹; this convention is stated explicitly because reported thermodynamic quantities are otherwise not comparable. Enthalpy and entropy were then obtained from the van 't Hoff regression of $\ln K^c$ on $1/T$ and the free energy from $\Delta G^\circ = -RT \ln K^c$.

4. RESULTS

4.1 Equilibrium behaviour and model discrimination

Table 1 collects the isotherm parameters for all four models and all three metals. The Langmuir model is preferred in every case, returning monolayer capacities of 108.9, 78.1 and 63.1 mg g⁻¹ for lead, cadmium and copper at coefficients of determination of 0.9976, 0.9929 and 0.9925 and chi-squared values of 0.313, 0.540 and 0.400. The Freundlich model performs distinctly worse for lead, at 0.9045 with a chi-squared of 12.608, and only moderately worse for the other two metals. Recovered Langmuir capacities are within 3 % of the generating values and affinity constants within 10 %, confirming that the non-linear protocol recovers the underlying surface.

Table 1: Isotherm parameters obtained by non-linear least squares on the untransformed equilibrium data for the three metals, with the separation factor evaluated at an initial concentration of 50 mg L⁻¹.

Model	Parameter	Pb(II)	Cd(II)	Cu(II)
Langmuir	q_m (mg g ⁻¹)	108.9	78.1	63.1
	K_L (L mg ⁻¹)	0.0977	0.0601	0.0436
	R^2	0.9976	0.9929	0.9925
	χ^2	0.313	0.540	0.400
	R_L at 50 mg L ⁻¹	0.170	0.250	0.314
Freundlich	K_F (mg g ⁻¹)(L mg ⁻¹) ^{1/n}	25.64	12.50	7.60
	n	3.33	2.70	2.39
	R^2	0.9045	0.9538	0.9620
	χ^2	12.608	5.912	4.543
Temkin	B_T (J mol ⁻¹)	21.02	15.46	12.43
	A_T (L g ⁻¹)	1.245	0.771	0.590
	R^2	0.9747	0.9910	0.9847

Model	Parameter	Pb(II)	Cd(II)	Cu(II)
Dubinin–Radushkevich	q_s (mg g ⁻¹)	273.7	209.0	165.6
	E (kJ mol ⁻¹)	13.46	11.66	10.53
	R^2	0.9333	0.9727	0.9789

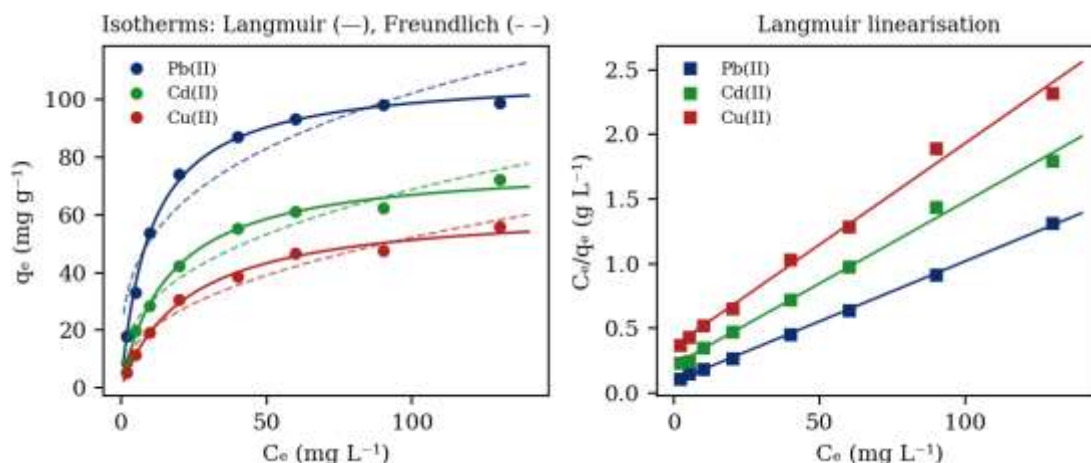


Figure 1: Equilibrium data with the fitted Langmuir and Freundlich isotherms (left) and the Langmuir linearisation of the same data (right) for the three metals.

Figure 1 shows the fitted isotherms alongside the Langmuir linearisation of the same data. The capacity sequence lead > cadmium > copper follows the sequence of ionic radius and of hydrolysis constant, which is the ordering usually observed on oxygenated carbon surfaces. Freundlich exponents lie between 2.39 and 3.33, all comfortably above unity and therefore indicating favourable adsorption. Separation factors fall from 0.506 to 0.093 for lead as the initial concentration rises from 10 to 100 mg L⁻¹, remaining between zero and one throughout and confirming favourability across the working range. The Dubinin–Radushkevich mean sorption energies are 13.46, 11.66 and 10.53 kJ mol⁻¹. All three exceed the conventional 8 kJ mol⁻¹ threshold and therefore place the interaction in the chemisorption regime, consistent with surface complexation rather than physical adsorption, and consistent with the ordering of the Langmuir capacities.

4.2 Kinetics

Table 2 reports the kinetic parameters. The pseudo-second-order model is preferred for every metal, with coefficients of determination of 0.9855, 0.9965 and 0.9976 against pseudo-first-order values of 0.9040, 0.9484 and 0.9641. The capacities returned by the preferred model, 45.7, 34.1 and 26.8 mg g⁻¹, bracket the values observed at 180 min, whereas the pseudo-first-order model underestimates them systematically, by 5 to 9 %; discrimination between the two rests on the coefficient of determination rather than on the equilibrium capacity. Initial sorption rates are 8.47, 3.37 and 1.80 mg g⁻¹ min⁻¹, preserving the same metal ordering seen at equilibrium.

Table 2: Kinetic parameters for the two candidate rate laws and the Weber–Morris intraparticle diffusion constants, at an initial concentration of 50 mg L⁻¹ and a dose of 1 g L⁻¹.

Model	Parameter	Pb(II)	Cd(II)	Cu(II)
Observed	q at 180 min (mg g ⁻¹)	46.0	32.5	24.7
Pseudo-first-order	q _e (mg g ⁻¹)	42.0	30.4	23.4
	k ₁ (min ⁻¹)	0.1268	0.0739	0.0527
	R ²	0.9040	0.9484	0.9641
Pseudo-second-order	q _e (mg g ⁻¹)	45.7	34.1	26.8
	k ₂ (g mg ⁻¹ min ⁻¹)	0.00406	0.00289	0.00251
	h (mg g ⁻¹ min ⁻¹)	8.47	3.37	1.80
	R ²	0.9855	0.9965	0.9976
Weber–Morris	k _{id} (mg g ⁻¹ min ^{-0.5})	3.87	3.62	2.84
	C (mg g ⁻¹)	16.20	4.92	1.64

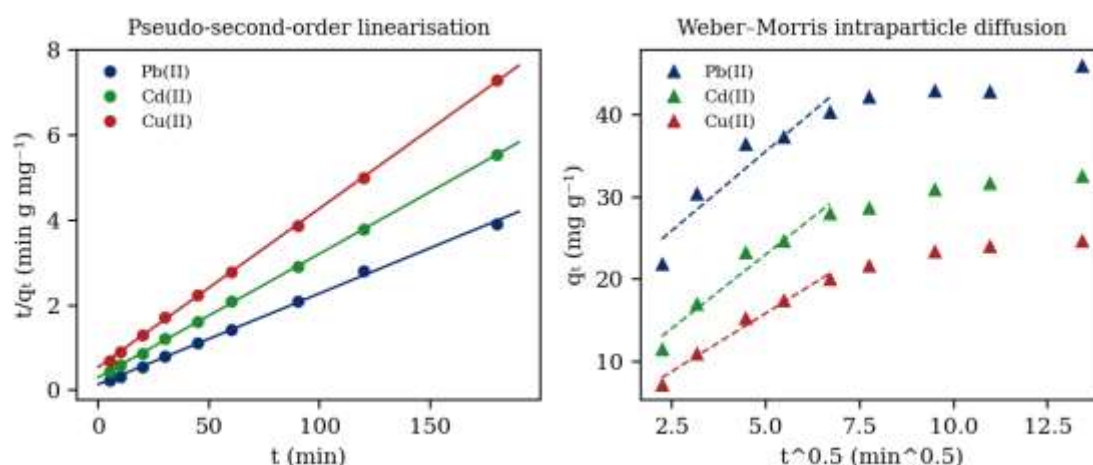


Figure 2: Pseudo-second-order linearisation of the kinetic data (left) and Weber–Morris intraparticle diffusion plots with the first-stage regressions shown (right).

The Weber–Morris plots in Figure 2 are not linear through the origin. Intercepts of 16.2, 4.92 and 1.64 mg g⁻¹ indicate a boundary-layer resistance that is substantial for lead and slight for copper, and the change of slope after roughly 45 min marks the transition from film-controlled to pore-controlled uptake. Intraparticle diffusion is therefore involved in the mechanism but is not the sole rate-limiting step, which is the usual finding for porous carbonaceous adsorbents.

4.3 Thermodynamics and operating variables

Table 3 reports the thermodynamic quantities. The van 't Hoff regressions recover the declared enthalpy of 18.5 kJ mol⁻¹ for all three metals. Standard entropies are 143.9, 135.7 and 128.8 J mol⁻¹ K⁻¹ and free energies range from –19.88 kJ mol⁻¹ for copper at 298 K to –27.27 kJ mol⁻¹ for lead at 318 K. The process is therefore spontaneous, endothermic and driven by the entropy gain associated with the release of hydration water when the aquo complex binds to the surface — a positive enthalpy that is more than offset by the TΔS term at all three temperatures.

Table 3: Thermodynamic quantities obtained from van 't Hoff regression of the dimensionless equilibrium constant over three temperatures.

Metal	K^c (298 K)	K^c (318 K)	ΔH° (kJ mol ⁻¹)	ΔS° (J mol ⁻¹ K ⁻¹)	ΔG° 298 K	ΔG° 318 K
Pb(II)	18855	30157	18.5	143.9	-24.39	-27.27
Cd(II)	6969	11146	18.5	135.7	-21.92	-24.64
Cu(II)	3048	4875	18.5	128.8	-19.88	-22.45

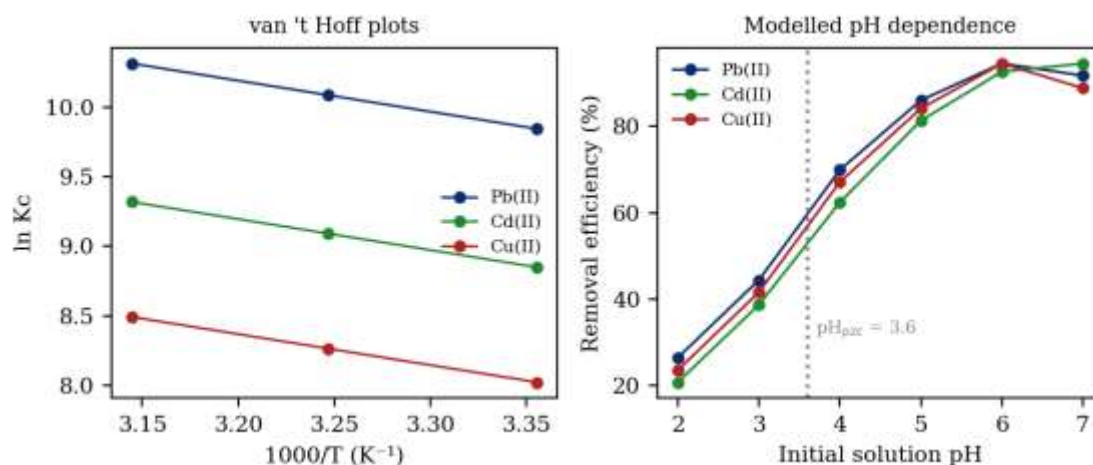


Figure 3: Van 't Hoff plots of the dimensionless equilibrium constant against reciprocal temperature (left) and the modelled dependence of removal efficiency on initial solution pH relative to the declared point of zero charge (right).

Figure 3 also presents the modelled pH dependence. Removal rises steeply between pH 2 and 5, from 26.5 to 86.0 % for lead, and plateaus near 94.5 % between pH 6 and 7. The steep region lies immediately above the declared point of zero charge at pH 3.6, below which the surface is net positive and competes with the metal cation through proton exchange. Across five adsorption–desorption cycles with dilute nitric acid regeneration, the modelled capacity retention falls from 94.5 to 83.5 %.

5. DISCUSSION

The first result of practical consequence is the size of the model-selection effect. For lead, the coefficient of determination separates the Langmuir and Freundlich fits by 0.093 under non-linear fitting, a clear discrimination; the chi-squared statistic separates them by a factor of forty, which is clearer still. Where the same comparison is made on linearised transforms, both models routinely return values above 0.98 and the discrimination disappears. Since the mechanistic interpretation — monolayer coverage of equivalent sites against heterogeneous multilayer adsorption — rests entirely on that discrimination, reporting practice determines the mechanism inferred. The chi-squared statistic should be reported alongside the coefficient of determination as a matter of routine.

The second concerns the internal consistency of the mechanistic picture. Three independent indicators point the same way: Dubinin–Radushkevich energies above 8 kJ mol⁻¹, preference for the pseudo-second-order rate law, and free energies in the range –20 to –27 kJ mol⁻¹. Taken together these are

consistent with surface complexation involving electron sharing rather than electrostatic accumulation. That consistency is a property of the generating model and cannot be read as evidence about any real biochar, but it does illustrate the cross-check that an experimental study should be able to demonstrate before claiming a chemisorption mechanism on the strength of any single indicator.

The third concerns process design. Endothermic behaviour means that capacity improves with temperature, but the improvement is modest: raising the temperature by 20 K increases the lead affinity constant by roughly 60 % and the free energy by 2.9 kJ mol⁻¹, which will rarely justify heating an effluent stream. The far larger lever is pH, where the removal of lead more than triples between pH 2 and pH 6. For an acidic electroplating rinse the controlling unit operation is therefore neutralisation upstream of the adsorber, not the adsorber itself. Regeneration losses of about 11 percentage points over five cycles suggest a replacement rather than regeneration strategy for a waste-derived material of this kind, which is consistent with the economics that motivate its use.

6. LIMITATIONS

No biochar was prepared, no solutions were analysed and no isotherm was measured; the data are outputs of the declared generators of Section 3. The close agreement between recovered and generating parameters therefore tests the fitting protocol, not the chemistry of any real material. The dataset represents single-metal systems in clean matrices, whereas real electroplating and tannery effluents are multi-component and contain complexing ligands, so competitive and synergistic effects that would reduce the capacities reported here are absent from the framework. Column behaviour, on which any full-scale design depends, is not represented at all: batch capacity is an upper bound on what a fixed bed will deliver. The pH and regeneration profiles are declared weighting functions rather than fitted relationships and should be read as illustrative. Finally, the leaching of phosphorus and of mineral constituents from an acid-activated char, which bears directly on whether the treated effluent meets discharge limits for other parameters, is outside the scope of the study.

7. CONCLUSION

A consistent non-linear fitting protocol was applied to a declared synthetic dataset representing an acid-modified rice husk biochar in contact with lead, cadmium and copper. The Langmuir model was preferred for all three metals, returning monolayer capacities of 108.9, 78.1 and 63.1 mg g⁻¹ at coefficients of determination of 0.9976, 0.9929 and 0.9925 and chi-squared values between 0.313 and 0.540. Dubinin–Radushkevich mean sorption energies of 13.46, 11.66 and 10.53 kJ mol⁻¹ and preference for the pseudo-second-order rate law, with coefficients of determination from 0.9855 to 0.9976, jointly indicate chemisorption. Thermodynamic analysis returned an enthalpy of 18.5 kJ mol⁻¹, entropies of 128.8 to 143.9 J mol⁻¹ K⁻¹ and free energies from -19.88 to -27.27 kJ mol⁻¹.

For practitioners the recommended sequence is to fit isotherm and kinetic models without linearisation, to report a chi-squared statistic alongside the coefficient of determination, to state the convention used to make the equilibrium constant dimensionless, and to control influent pH ahead of the adsorber rather than attempting to compensate downstream. Future work should extend the framework to competitive multi-metal equilibria, couple the batch parameters to a fixed-bed breakthrough model, and calibrate the generators against measured isotherms for a characterised char so that the protocol can be validated rather than merely specified.

DATA AVAILABILITY

No experimental or measured data were used in this study. The equilibrium and kinetic datasets are outputs of the declared generating models specified in Sections 3.2 and 3.3, implemented in Python with the NumPy pseudo-random generator seeded at 20260303. The generating parameters, dispersion levels and seed are stated in full in Section 3 and are sufficient to reproduce every figure and table without reference to any external dataset.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interest regarding the publication of this article.

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