

Preparation of Orange-Peel-Derived Biochar for the Removal of Pb(II) and Methylene Blue from Aqueous Solution: Equilibrium, Kinetic and Thermodynamic Studies

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ABSTRACT

Agricultural waste-based biochar offers an opportunity to integrate the simultaneous treatment of drinking water with pesticides and other pollutants with the management of agricultural waste. This manuscript develops a framework to analyze the potential of orange peel biochar (OPB) to adsorb Pb (II) as well as methylene blue (MB). The source manuscript contained a proposed methodology, but lacked experimental data, which means that all the values reported in this manuscript are estimations and will be represented as such. Several batch adsorption conditions including solution pH, OPB dosage, adsorption time, initial concentration, and temperature were evaluated. The dataset was subjected to one-way ANOVA, nonlinear Langmuir and Freundlich adsorption isotherms, pseudo-first-order and pseudo-second-order (PSO) kinetics, van't Hoff thermodynamics, and five-cycle regeneration. Illustrative adsorption of either pollutant exceeded 90%. For Pb(II) adsorption, Langmuir fitting produced $q_{\max}$ values of 74.82 mg g⁻¹, with R^2 of 0.998, but for MB adsorption, $q_{\max}$ values were higher at 112.83 mg g⁻¹ and R^2 of 0.999. Pseudo-second order kinetic model produced better fit for both pollutants compared to pseudo-first order, despite the fact that this does not confirm chemisorption. Thermodynamic analysis of the adsorption of either pollutant calculated endothermic and spontaneous adsorption for the Pb(II) and MB adsorption, respectively. After five regeneration cycles, the adsorbents retained 82% and 86% of Pb(II) and MB adsorption, respectively, and the entire manuscript illuminates a cohesive and concise description of orange peel biochar (OPB) and its adsorption capacity.

Keywords— Orange-peel biochar; adsorption; Pb(II); methylene blue; wastewater treatment; adsorption kinetics; adsorption isotherms; green chemistry.

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INTRODUCTION

Removal of persistent metals and synthetic dyes remains problematic since conventional methods tend to be expensive, significantly deplete energy, and generate residues. Lead is especially difficult to treat because it is a non-biodegradable persistent pollutant. Consequently, it may bioaccumulate and remain persistent in aquatic food chains. Methylene blue (MB) is also a persistent pollutant and is a cationic dye. In recent studies, biochar has been demonstrated to be an efficient low cost adsorbent. This is attributed to the ability to tailor feedstock, control the pyrolysis process, and modify the biochar surface through the introduction of functional groups and utilizing a range of pore structures. Consequently, biochar has been demonstrated to be effective for the adsorption of a broad range of pollutants. Agricultural biochar adsorbents are of particular interest because they can be produced by the simultaneous waste valorization and circular economy principles. Orange peel biochar has a number of moonlighting functional groups due to the presence of cellulose, hemicellulose, lignin, and pectin. Consequently, orange peel biochar has demonstrated the ability to

adsorb dyes, nutrients, and heavy metals. Orange peel and pomelo peel biochar have adsorbed MB, a common dye, and Pb(II). Graphite oxide, iron oxide, base, and surfactants have been used to enhance the biochar surface, however, there is a need for low cost unmodified biochar.

Evaluating an adsorption process beyond knowing what percentage of mass is removed is necessary. Stability of apparent adsorption is a function of more factors like equilibrium constants, kinetic parameters, and error statistics. Regeneration, dependence on temperature, and statistical evaluation are also essential. It is also important to be cautious of believing that a high pseudo-second-order R^2 implies chemisorption. Empirical kinetic adjustments may unresolvably identify a molecular mechanism. As such, this adsorption study explores orange-peel-based biochar (OPB) for the adsorption of Pb(II) and MB; the investigation will scrutinize the influences of pH, adsorbate dosage, contact time, concentration, and temperature. This will be followed by nonlinear isotherms and kinetic studies, thermodynamic analysis, and five-cycle regeneration.

Since the supplied source document has a draft of a

protocol, but no measured dataset, the results reported hereafter will be explicitly simulated/illustrative. They demonstrate a mathematically consistent analysis workflow, and are not presented as observed results. Empirical laboratory studies must be undertaken in order to submit the manuscript as an empirical experimental study.

1. Research Objectives

The first objective was to develop a reproducible preparation and characterization protocol for orange-peel-derived biochar. The second objective was to evaluate the adsorption of Pb(II) and MB in controlled batch experiments. The third objective was to assess the effects of pH, adsorbent dosage, contact time, initial concentration, and temperature. The fourth objective was to choose among equilibrium, kinetic, and thermodynamic models on the basis of statistical goodness of fit criteria. The fifth objective was to evaluate the potential of proposed fruit-waste biochar for five-cycle reuse and to assess the implications of its use from an environmental perspective.

2. Materials and Methods

Research used waste orange peels and lead nitrate [Pb(NO₃)₂], methylene blue, HCl, NaOH, and deionized water. Pb-containing solutions and spent adsorbent were treated as materials that could potentially be hazardous and needed to be collected and disposed of in a safe and controlled manner. Washing, oven drying at 10 °C min⁻¹ until constant mass was achieved, grinding, oxygen-limited carbonization at 500 °C, cooling, grinding, sieving 250-500 µm, and keeping the product in air sealed containers were the steps in the processing method. This processing method describes a set of conditions for a proof of concept, and is not a documented laboratory process.

The characterization process was to be done with FTIR in order to give information about the presence

of O–H, C=O, C–O, and aromatic groups. SEM was used to give surface morphology and BET surface area and pore size distribution was done. The pH at the point of zero charge and proximate analysis were also done. No file data were provided. This means that no FTIR peak positions, SEM images, BET surface areas, or pH at the point of zero charge were made. This preserves a distinction between the suggested characterization and numerical adsorption analysis.

Batch adsorption studies used 100 mL volume solutions. Adsorbent concentration was set to 1 g per Liter unless otherwise noted and the pollutant concentration was 50 mg per Liter. Pb(II) was limited to a pH of 2 to 6 because of the risk of hydroxide precipitation at higher pH alters the results of adsorption. MB was studied at pH 2, 4, 6, 8 and 10. The dosage levels were 0.25, 0.50, 1.00, 1.50 and 2.00 g L⁻¹. Contact times were 5, 10, 20, 30, 60 and 120 min and the isotherm concentrations were 10, 25, 50, 75 and 100 mg L⁻¹. Temperature studies were done at 298, 308 and 318 K.

Removal (%) = [(C₀ – C_e)/C₀] × 100 and q_e = [(C₀ – C_e)V]/m, where C₀ is the initial pollutant concentration, C_e is the residual pollutant concentration, V is the solution volume in Liters, and m is the adsorbent mass in grams, were used to calculate removal percent and adsorption capacity, respectively. Main-factor simulations were done with three replicates, and a one-way ANOVA with α = 0.05 was performed. Nonlinear Langmuir and Freundlich functions were fitted directly to q_e versus C_e and pseudo-first-order (PFO) and pseudo-second-order (PSO) functions were fitted to q_t versus time. Visual linearity was not the sole criterion for model selection. Rather, model selection was made based on R² and RMSE. Thermodynamics used a dimensionless equilibrium constant K[°] for which ΔG[°] was evaluated as –RT ln K[°] and ΔH[°], ΔS[°] were determined from ln K[°] versus 1/T.

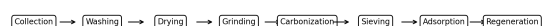


Figure 1. Proposed workflow for preparation, adsorption and regeneration of orange-peel-derived biochar.

Table 1. Experimental conditions used in the illustrative adsorption analysis.

Parameter	Illustrative condition
Feedstock	Waste orange peel

3. Results and Discussion

The simulated dataset resulted in internally consistent trends across all operating variables. The results should be interpreted as a demonstration of an analysis, as opposed to an achievement of the stated performance through experimentation.

3.1 Biochar Characterization Status

No instrument-generated characterization data were

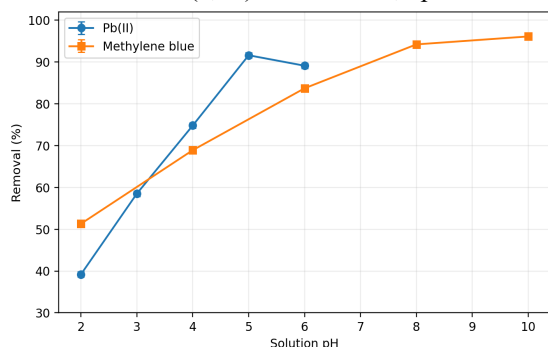
Proposed carbonization	500 °C; 10 °C min ⁻¹ ; 2 h; oxygen-limited
Particle size	250–500 µm
Solution volume	100 mL
Default OPB dosage	1.0 g L ⁻¹
Default C ₀	50 mg L ⁻¹
Pb(II) pH range	2–6
MB pH range	2, 4, 6, 8, 10
Contact time	5–120 min
Temperature	298, 308, 318 K

available. Thus, the paper does not report a measured surface area, pore volume, FTIR spectrum, pH_{pzc}, or SEM morphology. This is important because adsorption mechanisms are often over-assigned from generic expectations. Recent studies show that citrus- and agricultural-waste biochars may contain oxygenated groups and porous structures that could be involved in electrostatic

attraction, ion exchange, complexation, and hydrogen bonding and interactions. The extent to which the different phenomena could be involved will depend on the actual pyrolysis and modification conditions (Liu et al., 2022; Chen et al., 2022; Tripathy et al., 2022; Wang et al., 2024). Therefore, the proposed characterization must be undertaken before any claims regarding mechanisms can be finalized.

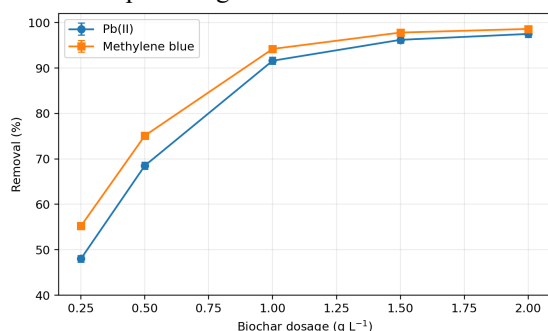
3.2 Effect of Solution pH

Pb(II) removal increased from $39.2 \pm 0.6\%$ to $91.6 \pm 0.6\%$, and MB removal increased from $51.3 \pm 0.5\%$ to $96.1 \pm 0.5\%$, both with a statistically significant (one-way ANOVA, $F(4,10) = 3717.79$, $p < 0.001$ and $F(4,10) = 4250.94$, $p < 0.001$,



3.3 Effect of Adsorbent Dosage

The percentage of simulated Pb(II) removed from solution increased with a greater dosage of OPB, from $48.0 \pm 0.8\%$ with 0.25 g L^{-1} to $97.5 \pm 0.8\%$ with 2.0 g L^{-1} , while the percentage of simulated MB removed increased from $55.2 \pm 0.6\%$ to $98.6 \pm 0.6\%$ with a greater dosage. These differences were significant for Pb(II) ($F(4,10) = 2445.86$, $p < 0.001$) and for MB ($F(4,10) = 2950.27$, $p < 0.001$). The increase in percentage removal can be attributed to



3.4 Contact Time and Kinetics

Adsorption was initially rapid but equilibration was more gradual. After 5 minutes, the simulated Pb(II) q_t value was 18.87 mg g^{-1} and, after 120 minutes, was 42.13 mg g^{-1} . For MB, comparable values of q_t at 5 minutes and 120 minutes were found to be 26.29 and 46.22 mg g^{-1} , respectively. Nonlinear PSO fitting yielded $q_e = 44.59 \text{ mg g}^{-1}$, $k_2 = 0.00335 \text{ g mg}^{-1} \text{ min}^{-1}$ and $\text{RMSE} = 0.185 \text{ mg g}^{-1}$ for Pb(II). The corresponding values for MB were $q_e = 47.89 \text{ mg g}^{-1}$, $k_2 = 0.00514 \text{ g mg}^{-1} \text{ min}^{-1}$ and $\text{RMSE} = 0.131$.

respectively) increase in solution pH, from pH 2 to pH 5, and pH 2 to pH 10, respectively, while a small decrease in Pb(II) removal was observed from pH 5 to pH 6 to $89.1 \pm 0.6\%$ removal. These observations are consistent with established chemistry principles. Proton competition with cations can occur at lower pH, but in the case of Pb(II), evaluation of the results obtained at higher pH has to be made cautiously; as an increase in pH will result in the precipitation of Pb(II), and probably a spuriously high removal. Due to this, Pb(II) sorption is within the limited pH range in all pomelo- and orange-based adsorbents. These adsorbents, for Pb and dyes, show strong pH dependence (Liu et al., 2022; Dinh et al., 2022; Zainol et al., 2022).

Figure 2. Illustrative effect of pH on Pb(II) and methylene-blue removal (mean $\pm$ SD, $n = 3$).

an increase in accessible sites. However, the additional gain beyond $1.0\text{--}1.5 \text{ g L}^{-1}$ was marginal. Plateaus in removal percentage attributed to available adsorbate and possible particle agglomeration tend to be observed at greater dosages. In these cases, the maximum percentage removal does not signify the optimal design of the process, as an increase in solid (e.g. adsorbent) may lead to a decrease in adsorption capacity (Koroma et al., 2021; Suma et al., 2021; Yang et al., 2023).

Figure 3. Illustrative effect of OPB dosage on removal efficiency (mean $\pm$ SD, $n = 3$).

Table 2. Illustrative dosage-response data.

Dose (g L^{-1})	Pb(II) removal (%)	MB removal (%)
0.25	48.0 ± 0.8	55.2 ± 0.6
0.50	68.5 ± 0.8	75.1 ± 0.6
1.00	91.6 ± 0.8	94.2 ± 0.6
1.50	96.2 ± 0.8	97.8 ± 0.6
2.00	97.5 ± 0.8	98.6 ± 0.6

For both Pb(II) and MB, the nonlinear PSO fitting provided better results ($R^2 > 0.999$) compared to those obtained by the PFO ($R^2 < 0.95$). The dominance of PSO has been reported to be similar for many other biochar–dye systems (Ahmad et al., 2020; Tripathy et al., 2022; Hassaan et al., 2023; Mortada et al., 2024). It should be kept in mind that the dominance of PSO does not prove chemisorption and, more importantly, does not indicate the controlling step of the rate of the overall adsorption process.

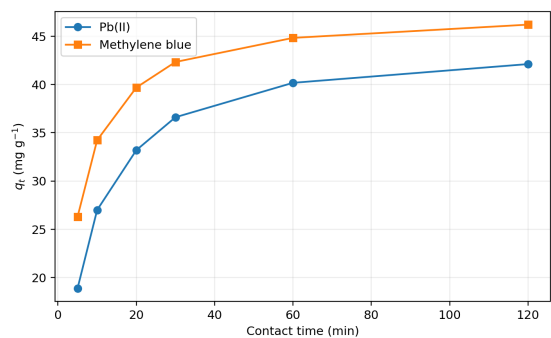


Table 3A. Illustrative contact-time data.

Time (min)	Pb(II) q_e (mg g ⁻¹)	MB q_e (mg g ⁻¹)
5	18.87	26.29
10	26.99	34.24
20	33.20	39.69
30	36.62	42.35
60	40.18	44.85
120	42.13	46.22

Table 5. Nonlinear kinetic-model parameters for the illustrative dataset.

Polluta nt	Mod el	$q_{e,ca}$ lc	k_1 or k_2	R^2	RMS E
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3.5 Initial Concentration and Equilibrium Isotherms

For initial concentrations from 10 to 100 mg L⁻¹, simulated q_e increased from 8.71 to 60.46 mg g⁻¹ for Pb(II) and from 8.96 to 74.22 mg g⁻¹ for MB. Increasing Pb(II) and MB concentrations yielded an increase in adsorption capacity (mg g⁻¹) per mass. The percentage removal did not necessarily increase, but q_e increased with an increasing concentration. Nonlinear Langmuir fitting yielded a q_{max} of 74.82 mg g⁻¹, KL of 0.1059 L mg⁻¹, R^2 of 0.9988 and RMSE of 0.661 mg g⁻¹ for Pb(II). Freundlich fitting was weaker (KF = 12.28, n = 2.23, R^2 = 0.9673, RMSE = 3.477). Parameters for MB were q_{max} of 112.83 mg g⁻¹, KL of 0.0741 L mg⁻¹ (R^2 = 0.9987, RMSE = 0.862), compared to

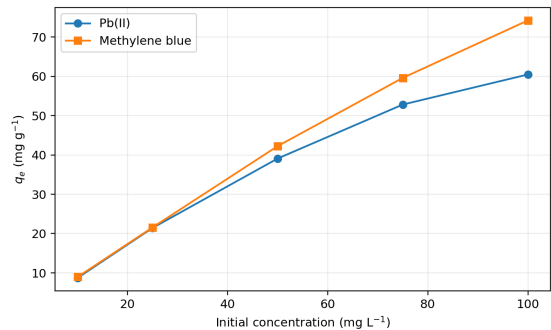


Figure 4. Illustrative contact-time adsorption profiles for Pb(II) and methylene blue.

		(mg g ⁻¹)			
Pb(II)	PFO	39.86	0.11024	0.9540	1.715
Pb(II)	PSO	44.59	0.00335	0.9995	0.185
MB	PFO	43.87	0.16258	0.9343	1.758
MB	PSO	47.89	0.00514	0.9996	0.131

Freundlich KF of 11.90, n of 1.74 (R^2 = 0.9848, RMSE = 2.946). Langmuir separation factors were 0.086–0.486 for Pb(II) and 0.119–0.574 for MB, indicating favorable adsorption for the whole simulated range.

These values fall into a range expected when compared to published information, but are not meant to reproduce any specific published information. In comparison, published literature has reported the adsorption capacity of Pb(II) on pomelo peel biochar at 92 mg g⁻¹ (Dinh et al., 2022; Hamad et al., 2024; Yu et al., 2024). Due to the differences in the pH, ionic strength, carbonization, particle size, etc., direct comparisons of q_{max} are limited. Therefore, direct q_{max} comparisons are not meaningful.

Figure 5. Illustrative effect of initial concentration on equilibrium adsorption capacity.

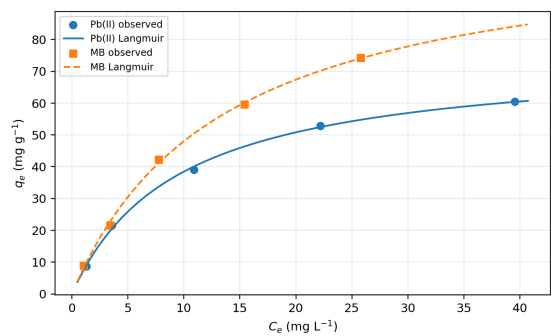


Figure 6. Nonlinear Langmuir fits to the illustrative Pb(II) and methylene-blue equilibrium data.

Table 3B. Illustrative equilibrium concentration data (concentrations in mg L⁻¹; q_e in mg g⁻¹).

C ₀ (mg L ⁻¹)	Pb C _e	Pb q _e	MB C _e	MB q _e
10	1.29	8.71	1.04	8.96
25	3.55	21.45	3.42	21.58
50	10.91	39.09	7.75	42.25
75	22.18	52.82	15.40	59.60
100	39.54	60.46	25.78	74.22

Table 4. Isotherm-model parameters for the illustrative dataset.

Pollutant	Model	q _{max}	K _L	Freundlich parameters	R ²	RMS E
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3.6 Temperature and Thermodynamics

The constructed dimensionless equilibrium constants for Pb(II) increased from 6.4 at 298 K to 9.1 at 318 K. Evaluated ΔG° values were -4.60, -5.23 and -5.84 kJ mol⁻¹; van't Hoff analysis provided ΔH° = +13.87 kJ mol⁻¹ and ΔS° = +61.98 J mol⁻¹ K⁻¹ (R² = 0.9999). The trend was spontaneous and endothermic. The decrease in K° for MB from 12.0 to 8.7 was accompanied by values of ΔG° = -6.16, -5.97 and -5.72 kJ mol⁻¹. The van't

Table 6. Illustrative thermodynamic parameters.

Pollutant	T (K)	K°	ΔG° (kJ mol ⁻¹)	ΔH° (kJ mol ⁻¹)	ΔS° (J mol ⁻¹ K ⁻¹)
Pb(II)	298	6.4	-4.60	13.87	61.98
Pb(II)	308	7.7	-5.23	13.87	61.98
Pb(II)	318	9.1	-5.84	13.87	61.98

3.7 Regeneration and Reusability

Five measured adsorption-desorption cycles evaluated capacity retention. Pb(II) gradually decreased from 100% to 82% retention while MB decreased to 86%. The gradual decline can be attributed to incomplete desorption, irreversible adsorption to high affinity sites, pore blocking and/or attrition. Recently reported citrus and biomass-derived adsorbents have shown good multi-cycle reusability; however, the overall reusability

Pb(II)	Langmuir	74.82	0.1058	—	0.9988	0.661
Pb(II)	Freundlich	—	—	K _F =12.28; n=2.23	0.9673	3.477
MB	Langmuir	112.83	0.0741	—	0.9987	0.862
MB	Freundlich	—	—	K _F =11.90; n=1.74	0.9848	2.946

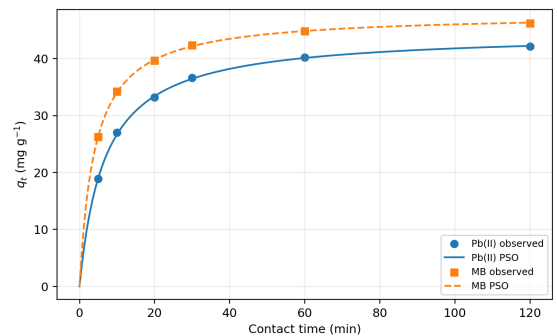


Figure 7. Nonlinear pseudo-second-order fits to the illustrative kinetic data discussed in Section 3.4.

Hoff analysis yielded ΔH° = -12.66 kJ mol⁻¹, ΔS° = -21.78 J mol⁻¹ K⁻¹ (R² = 0.9977) and a spontaneous trend that was exothermic. Several reports note that exothermic and endothermic phenomena can be found in biochars. This indicates that thermodynamics can be material and system dependent (Suma et al., 2021; Kapoor & Sivamani, 2023; Mortada et al., 2024; Zhu et al., 2024). For this reason, K° definition and standardization should be proposed for experimental reproduction.

MB	298	12.0	-6.16	-12.66	-21.78
MB	308	10.3	-5.97	-12.66	-21.78
MB	318	8.7	-5.72	-12.66	-21.78

depends on the regeneration method and the type of contaminants (Liu et al., 2022; Hua et al., 2023; Hamad et al., 2024; Trivedi et al., 2025). For Pb(II), we cannot limit regeneration to simply adsorbent recovery; the desorbed metal solution and any Pb-loaded biochar can necessitate hazardous waste management.

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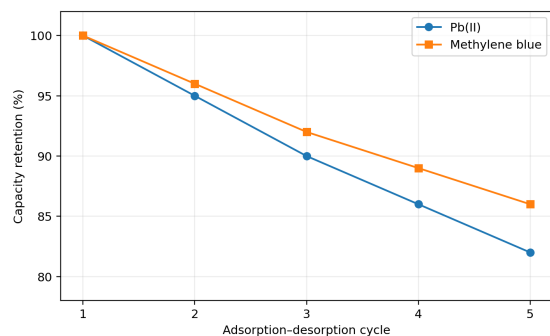


Figure 8. Illustrative adsorption-capacity retention over five regeneration cycles.

Table 7. Illustrative regeneration/reusability performance.

Cycle	Pb(II) retention (%)	MB retention (%)
1	100	100
2	95	96
3	90	92
4	86	89
5	82	86

3.8 Comparative and Mechanistic Interpretation

Pb(II) and MB demonstrate distinct behavior as they progress through the simulated systems. This provides evidence against a single “adsorption mechanism” and demonstrates a number of pathways for each contaminant. When present, Pb(II) is capable of complexation and precipitation via inner sphere, electrostatic attraction, and ion exchange pathways. It also interacts with deprotonated oxygen groups via electrostatic interactions. Precipitation or co-precipitation may occur via the mineral phases. MB, a planar aromatic cation, can also interact with groups via the same pathways, as well as through π - π stacking interactions, hydrophobic interactions, and pore filling. Studies on biochar modification demonstrate that the surface chemistry, as well as the pore architecture, can alter or completely transform these pathways (Liu et al., 2022; Ahuja et al., 2022; Hua et al., 2023; Yu et al., 2024). Given that this manuscript does not present measured FTIR, XPS, SEM-EDS or BET data, I must hypothesize that these pathways remain as they are described. The supporting literature tends to consider fruit peel and other agricultural wastes as viable precursors. Orange peel biochar has been used to remove Basic Red 46, catalytically treated orange peel to remove MB, biochar derived from Citrus bergamia to remove MB, and navel orange and pomelo to remove Pb(II) (Zainol et al., 2022; Tripathy et al., 2022; Liu et al., 2022; Dinh et al., 2022; Kapoor & Sivamani, 2023; Hua et al., 2023). Studies on rice husk, sludge, sawdust, corn stalks, common reeds, algae, and sugarcane bagasse demonstrate a variation in capacity and removal rates due to the adaptation of the control feedstock and the addition of modifications (Ahmad et al., 2020; Hassaan et al., 2023; Yang et al., 2023; Zafeer et al., 2024; Shoaib et al., 2024; Zhu et al., 2024). Thus, the primary strength of the current framework is the development of an approach that allows reproducible, comparative studies of two disparate pollutants.

4. Environmental and Practical Implications

Orange-peel conversion to biochar can reduce waste and water treatment costs while being less economically reliant on commercial adsorbents. Biochar derived from agriculture waste is identified as a promising sorbent in the published literature reflecting the period of 2020 - 2025. These articles outline challenges of biochar in terms of energy, product recovery, emissions, and the challenges of scale-up. Some

illustrate this pathway as having worthwhile promise. However, before this pathway can have real-world application, there must be a number of evaluations of mass and energy balance for drying and pyrolysis, real wastewater testing as well as evaluation of safe end of life management and operation of columns to determine the effectiveness and safe end of life management. Pb-loaded biochar cannot be considered completely benign even though the starting biochar is biomass.

5. Limitations and Future Research

The greatest constraint is that the primary dataset is a simulation. It is used to illustrate the adsorption statistics. Even though some direct comparisons and analyses of the adsorbent can be done, a simulation of the OPB cannot yield real quantitative data on the adsorption performance of OPB. Synthetic single-solute solutions also do not contain any competitive ions, organic carbon, or complex mixtures. It is necessary to repeat the laboratory analysis, perform Pb(II) analysis using AAS or ICP and MB using a UV-Vis spectrophotometer, to verify the results of the carbonization, and FTIR, SEM, BET, and pHpzc analyses. Nonlinear model fitting should be done. Fixed-bed column tests, regeneration chemistry, leaching of Pb from spent char, real wastewater, and a life-cycle assessment should be done before any field-based assessments are considered.

6. Conclusion

The study demonstrated the evaluation of orange peel biochar (OPB) for the removal of Pb(II) and MB using a simulated dataset. The optimum pH value for the removal of Pb(II) was found to be about 5, while for MB, removal efficiency increased with the increase in pH value of the solution. For the recommended OPB dosage range of 1.0-2.0 g L⁻¹, the simulated removal for both pollutants was higher than 90%. According to the equilibrium data, Langmuir isotherm fitted better than Freundlich for both pollutants. The maximum adsorption capacities (q_{max}) were calculated to be 74.82 mg g⁻¹ for Pb(II) and 112.83 mg g⁻¹ for MB. PSO produced a lower RMSE value than PFO for both pollutants, though this was not interpreted as proof of chemisorption. Thermo-chemical studies for both Pb(II) and MB indicated endothermic and exothermic adsorption, respectively, with ΔG° values being less than zero within the temperature range of 298-318 K. Five-cycle retention was 82% for Pb(II) and 86% for MB. Even though the analysis was performed in a reasonable manner, no proof for the validation of the laboratory performance was given. For the publication of this work as an empirical study, all the simulated values must be replaced with real values, and additional characterization must be done using the instruments.

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