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ADSORPTION OF HEAVY METALS CD(II), PB(II), AND CU(II) FROM WASTEWATER BY USING AGRICULTURE WASTE PRODUCT AS A LOWCOST ADSORBENT: A KINETIC STUDY

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ABSTRACT

This study investigates the adsorption kinetics of Cd(II), Pb(II), and Cu(II) on hydrochars derived from wheat straw, rice straw, and bagasse using both conventional and microwave-assisted hydrothermal treatments. Adsorption experiments were conducted to evaluate the effectiveness of these materials, with kinetic models applied to analyze the mechanism. The pseudo-second-order model demonstrated a better fit for all metals, indicating chemisorption as the primary process. Modified hydrochars, particularly those prepared using microwave methods, exhibited significantly higher adsorption capacities. Among them, m-HRS showed the best performance, adsorbing 137.36 mg/g of Pb(II). These findings highlight the potential of microwave-modified hydrochars for efficient heavy metal removal from aqueous solutions, offering an environmentally friendly solution to water contamination.

1. INTRODUCTION

Heavy metal pollution, particularly from metals such as Pb(II), Cd(II), and Cu(II), has become a significant concern in recent years due to their high mobility and biological accumulation, posing serious environmental and human health risks(1). Heavy metals are introduced into the environment through various human activities, including mining, refining, industrial processes, and agricultural runoff(2, 3). Once these metals enter aquatic systems, they can bioaccumulate in living organisms, causing long-term ecological damage and health hazards for humans. The figure 1. showing heavy metals, their sources, and effects(4-7).

Among the various methods developed for removing heavy metals from contaminated water, adsorption has gained prominence due to its high efficiency, low cost, and minimal secondary pollution (8-10). Activated carbon, chitosan, organic materials, and biochar are widely used as adsorbents (11), but hydrochar, produced through hydrothermal carbonization (HTC), has shown particular promise as an alternative sorbent. Unlike biochar, hydrochar exhibits a high degree of surface aromatization and contains numerous oxygen-containing functional groups, which enhance its adsorption capacity for heavy metals in aqueous environments(11-16) .

Hydrochar is derived from agricultural wastes like wheat straw, rice straw, and bagasse through HTC or microwave-assisted carbonization. These processes yield carbon-rich materials that possess specific physicochemical properties, including porous structures, thermal stability, and high adsorption potential for heavy metals such as Pb(II), Cd(II), and Cu(II). While pyrolytic biochar (pyrochar) has been extensively studied for environmental applications like soil improvement, water treatment, and carbon sequestration(17-19), hydrochar research is still

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in its infancy(20). Despite this, recent studies suggest that hydrochar materials can outperform biochar in terms of heavy metal adsorption, particularly after chemical modifications (21-23).

This research focuses on a comparative analysis of hydrochars produced from wheat straw, rice straw, and bagasse using both conventional hydrothermal carbonization and microwave-assisted methods. The study evaluates key parameters such as yield, elemental composition, surface area, pore volume, and thermal stability, highlighting the advantages of microwave-assisted carbonization, which results in lower surface areas but increased pore volumes and higher thermal stability(24). The investigation also explores the adsorption kinetics and isotherms for the removal of Cd(II), Pb(II), and Cu(II) from aqueous solutions, applying both Langmuir and Freundlich isotherm models to quantify adsorption capacities and mechanisms.

Heavy metal removal from wastewater is critical due to the negative impact of these contaminants on human health and the environment. Lead (Pb) exposure can lead to neurological and developmental disorders, cadmium (Cd) can cause kidney damage and bone demineralization, and copper (Cu) in high concentrations can result in gastrointestinal and liver problems. Addressing these pollutants is essential, especially as industrial and agricultural waste continues to introduce large quantities of heavy metals into natural water bodies(22, 25-29).

By comparing the performance of hydrochar produced by different methods has been discussed in our earlier work(24). This study seeks to provide insights into optimizing biomass-derived adsorbents for heavy metal remediation. The findings are expected to contribute to the development of cost-effective, environmentally friendly solutions for mitigating water pollution and enhancing sustainability in wastewater treatment.

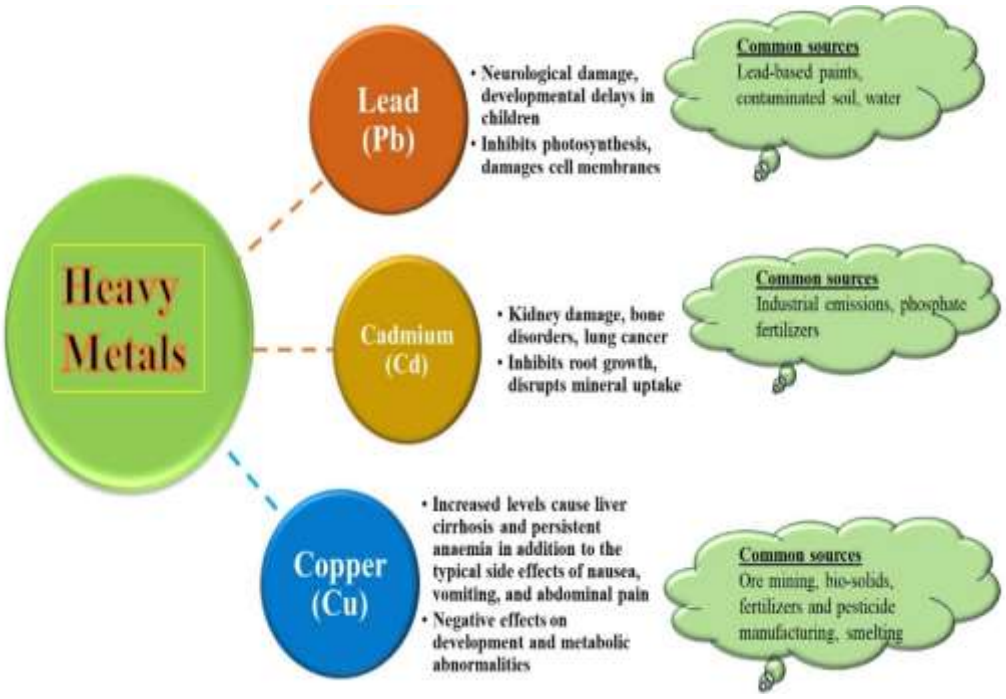

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Figure. 1. The primary sources of heavy metals such as lead (Pb), cadmium (Cd), and copper (Cu) from industrial, agricultural, and natural activities, and their adverse effects on human health and ecosystems.

2. EXPERIMENTAL

Materials and Methods

Materials

Hydrochars were produced from agricultural residues (e.g., rice straw, wheat straw, and bagasse) through hydrothermal carbonization (HTC) and microwave-assisted methods. The residues were sourced locally and dried before processing. Reagents for adsorption experiments, including $Pb(NO_3)_2$, $Cd(NO_3)_2$, and $Cu(NO_3)_2$, were purchased from a certified chemical supplier and used without further purification. Deionized water was used throughout the experiments.

2. **Hydrochar Production:** Agricultural residues (rice straw, wheat straw, and bagasse) were collected and subjected to two methods for hydrochar production: conventional hydrothermal carbonization (HTC) and microwave-assisted hydrothermal carbonization (MHTC).

HTC: 60 g of dried biomass was mixed with 400 mL of DI water and heated in a 500 mL stainless steel autoclave at 300 °C for 5 hours under 1000 psi. The resultant hydrochar was rinsed, dried at 80 °C, ground, and sieved (0.5–1.0 mm) for further use.

MHTC: 1 g of biomass was placed in a glass vial with 5 mL of DI water and heated in a microwave to 200 °C within 20 minutes. The hydrochar was filtered, dried at 105 °C for 3 hours, and stored.

The details of these methods, along with modification using H_2O_2, are discussed in earlier published work(24).

3. Adsorption Experiments

Batch adsorption experiments were performed to evaluate the adsorption of Pb(II), Cd(II), and Cu(II) onto the prepared hydrochars. The initial concentrations of metal solutions were prepared by dissolving respective nitrates in deionized water. Adsorption experiments were conducted by mixing a known amount of hydrochar (typically 0.1 g) with 100 mL of heavy metal solutions at varying concentrations (10–100 mg/L) in 250 mL conical flasks. The pH of the solutions was adjusted to 5.0 ± 0.1 using 0.1 M NaOH or HCl, and the flasks were shaken at 150 rpm in a temperature-controlled shaker at 25°C for 24 hours to reach equilibrium.

3.1. Effect of pH Value

The adsorption of heavy metal ions, including Pb(II), Cd(II), and Cu(II), is significantly influenced by the solution pH, as it affects the electrostatic interactions between the metal ions and the functional groups on the hydrochar surface. Variations in pH can lead to changes in the ionization of functional groups, altering the active sites available for adsorption and thereby affecting the reaction kinetics and equilibrium characteristics of the adsorption process.

To optimize pH for maximum metal ion removal, the effect of pH was investigated over a range of 2 to 10. For this study, 50 ml of metal ion solutions (10 mg/l for each ion) were prepared in separate flasks. The pH of these solutions was adjusted to the desired levels (2, 3, 4, 5, 6, 7, 8, 9, and 10) using 0.1N HCl and 0.1N NaOH added dropwise. The optimum dose of each hydrochar adsorbent was then added to the respective flasks, which were agitated at 170 rpm for a duration of 2 hours. After agitation, the remaining concentrations of Pb(II), Cd(II), and Cu(II) in the filtrate were analyzed to determine the optimal pH value for each adsorbent, at which maximum removal efficiency was achieved(24).

4. Kinetic Studies

Kinetic studies were carried out by withdrawing samples at different time intervals (0, 5, 10, 30, 60, 120, 180, 240, 300, 360, and 420 minutes). The concentration of heavy metals in the

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supernatant was analyzed using Atomic Absorption Spectroscopy (AAS). The adsorption kinetics were modeled using Pseudo-First-Order and Pseudo-Second-Order kinetic models.

In Figure 2, the flowchart provides a comprehensive overview of the experimental approach adopted in this study.

4.1. Application of Adsorption Kinetics

The adsorption kinetics provides insight into the rate at which heavy metal ions, such as Pb(II), Cd(II), and Cu(II), are taken up by the hydrochar adsorbents, which ultimately determines the time required to reach adsorption equilibrium. A thorough understanding of adsorption kinetics is crucial for effectively modeling the adsorption process and identifying the rate-controlling steps involved.

In this study, kinetic parameters were derived from batch adsorption experiments using heavy metal ion concentrations of 10 mg/l for Cd(II), Cu(II), and Pb(II)). The contact time was varied from 10 minutes to 360 minutes, depending on the specific adsorbent used. The percentage removal of each metal ion was calculated for each time interval. The experimental data were analyzed by plotting the removal percentage against time, and the results were subsequently fitted to both pseudo-first-order and pseudo-second-order kinetic models to determine the most accurate representation of the adsorption process. This analysis allows for a better understanding of the adsorption mechanisms and the efficiency of the hydrochar adsorbents in removing heavy metals from solution.

4.2. Characterization of Adsorbent

The hydrochars were characterized using Scanning Electron Microscopy (SEM) for surface morphology, Fourier Transform Infrared Spectroscopy (FTIR) for functional group identification, and Brunauer–Emmett–Teller (BET) analysis for surface area and pore size distribution. Thermogravimetric Analysis (TGA) was performed to assess the thermal stability of the hydrochars.

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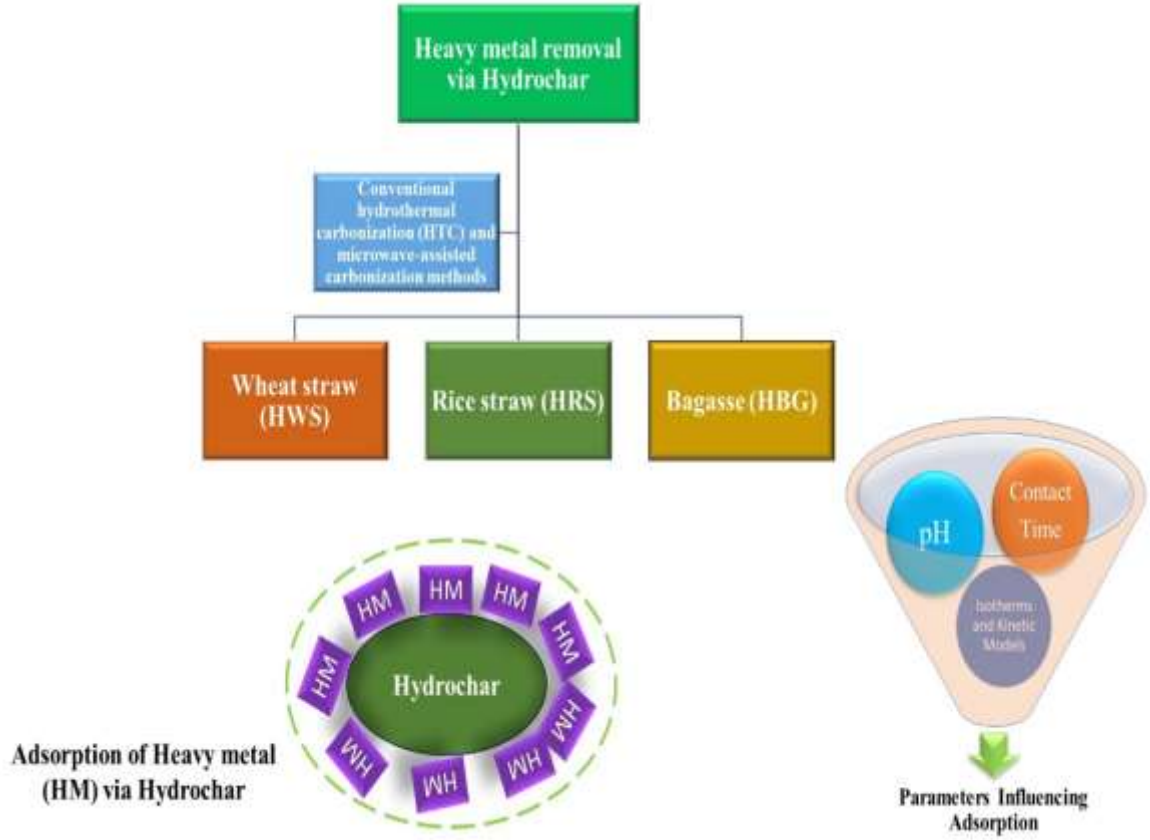

Figure 2. Overview of the research methodology

3. RESULTS AND DISCUSSION

Effect of pH

The adsorption of heavy metals onto hydrochar is highly dependent on the solution's pH, which influences both the surface charge of the adsorbent and the speciation of the metal ions. The adsorption capabilities of hydrochars produced from various agricultural wastes were examined over a pH range of 2 to 7.

The data indicate a consistent trend in the adsorption behavior of Pb^{2+}, Cd^{2+}, and Cu^{2+} across different hydrochars as pH increases. Specifically, the adsorption efficiency for these metal ions decreases at lower pH levels. This can be attributed to the competition between metal ions and H^+ ions for available adsorption sites on the hydrochar surface. At reduced pH values, the hydrochar surface becomes protonated, resulting in a more positive charge that creates electrostatic repulsion between the positively charged hydrochar and the metal ions, thereby diminishing the hydrochar's adsorption capacity.

As the pH rises, the removal of protons from the oxygen-containing functional groups on the hydrochar surface lessens its positive charge. This reduction in positive charge decreases the competition for adsorption sites between H^+ ions and metal ions, thereby enhancing the availability of these sites for metal ion adsorption. Consequently, the hydrochar's adsorption capacity increases with higher pH levels. Maximum adsorption capacities for Pb^{2+}, Cd^{2+}, and Cu^{2+} are recorded at pH levels of 6 and 5, yielding respective values of 139.44 mg/g for Pb^{2+}, 52.92 mg/g for Cd^{2+}, and 31.25 mg/g for Cu^{2+}. These findings are consistent with previous research reporting similar patterns in plant-based hydrochars.

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However, when pH levels exceed 6, there is a noticeable decline in the hydrochar's adsorption capacity. This reduction is partly due to the precipitation of metal ions as hydroxides, with $Pb(OH)_2$ and $Cd(OH)_2$ beginning to precipitate at pH values of 5.5 and 6.5, respectively. Therefore, the maximum pH for effective adsorption in this study is below 7 to prevent precipitation and ensure efficient adsorption.

Additionally, when comparing the adsorption performance of hydrochars produced at different microwave powers, it is evident that m-HRS shows better adsorption capabilities for heavy metals than m-HBG. This suggests that the preparation conditions, such as microwave power, significantly influence the adsorption performance of hydrochars. The superior adsorption ability of m-HRS may be attributed to its larger surface area and specific structural features that enhance heavy metal adsorption(24).

Pseudo-First Order Kinetic Model

The pseudo-first-order kinetic model describes the adsorption rate in relation to the adsorption capacity, represented by the equation:

$$\log (qe-qt) = \log qe - k_1 t/2.303 \quad \ldots\ldots(1)$$

where qe and qt are the amounts of metal adsorbed (mg/g) at equilibrium and at time t, respectively, and k_1 is the adsorption rate constant, which can be determined from the slope of linear plot of log(qe-qt) against t(30).

Pseudo-Second Order Kinetic Model

The pseudo-second-order kinetic model is applied to describe chemisorption processes that involve valence forces and ion exchange. The model is represented by the equation:

$$\frac{1}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} t \quad \ldots\ldots(2)$$

where qt is the amount of solute adsorbed on the adsorbent at time t(mg/g), and qe is the amount at equilibrium (mg/g), and k_2 is the rate constant of the model, which can be determined by plotting t/qt against t. To determine the best fitted kinetic model, it depends on correlation coefficient (R^2) value, the closest to 1 indicate the suitable kinetic model. If kinetic model best fits with pseudo-first-order reaction, it shows that the reaction is tender towards physisorption. Likewise, if the reaction fits well with the pseudo-second order model it shows a tendency towards chemisorption(30).

The adsorption kinetics of Cd(II), Pb(II), and Cu(II) ions onto hydrochars derived from wheat straw (HWS), rice straw (HRS), and bagasse (HBG), including their microwave-modified versions (m-HWS, m-HRS, m-HBG), were thoroughly evaluated using both pseudo-first-order and pseudo-second-order kinetic models. These models were applied to understand the adsorption behavior and to identify the mechanisms governing the adsorption process. The correlation coefficient (R^2) values were used to determine the best-fitting model, providing insights into whether the adsorption follows a physical adsorption mechanism (physisorption) or a chemical adsorption mechanism (chemisorption).

For Cd(II) ions, the experimental data indicated that the pseudo-first-order model offered a reasonable fit for certain hydrochars, particularly for HRS (R^2 = 0.9964) and HBG (R^2 = 0.9910). However, the pseudo-second-order model demonstrated superior fitting, particularly for microwave-modified hydrochars. For instance, the highest R^2 value of 0.9971 was observed for m-HWS, indicating that chemisorption plays a key role in the adsorption of Cd(II) ions. The adsorption capacity of Cd(II) ions was notably higher for microwave-modified hydrochars, with m-HRS exhibiting a capacity of 52.92 mg/g, reflecting the effectiveness of microwave modification in enhancing adsorption properties. These results suggest that microwave treatment improves the surface area and functional group availability of hydrochars, making them more efficient for Cd(II) adsorption (Fig. 3).

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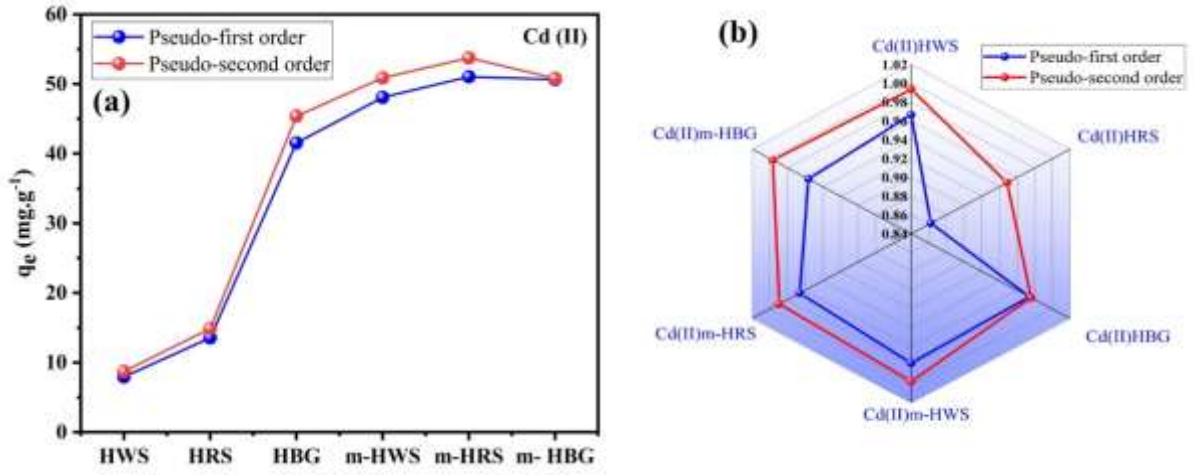

Figure 3: (a) Adsorption capacity of Cd (II) by different Hydrochars at 298 K, (b) Correlation coefficient (R^2) for the Cd (II) in Pseudo 1st order and Pseudo 2nd order

For Pb(II) ions, the pseudo-second-order model consistently provided a better fit across all hydrochars, with R^2 values exceeding 0.98, indicating that chemisorption is the dominant mechanism for Pb(II) ion adsorption. Notably, the microwave-modified hydrochars exhibited significantly higher adsorption capacities compared to their non-modified counterparts. The maximum adsorption capacity was recorded for m-HRS at 137.36 mg/g, followed closely by m-HBG at 131.44 mg/g. This trend underscores the positive impact of microwave-assisted hydrochar preparation, which enhances the adsorption sites available for Pb(II) ions. The pseudo-second-order model's superior fit, along with higher adsorption capacities, suggests that Pb(II) ions interact strongly with the hydrochar surface, possibly through mechanisms involving valence forces and ion exchange (Fig. 4).

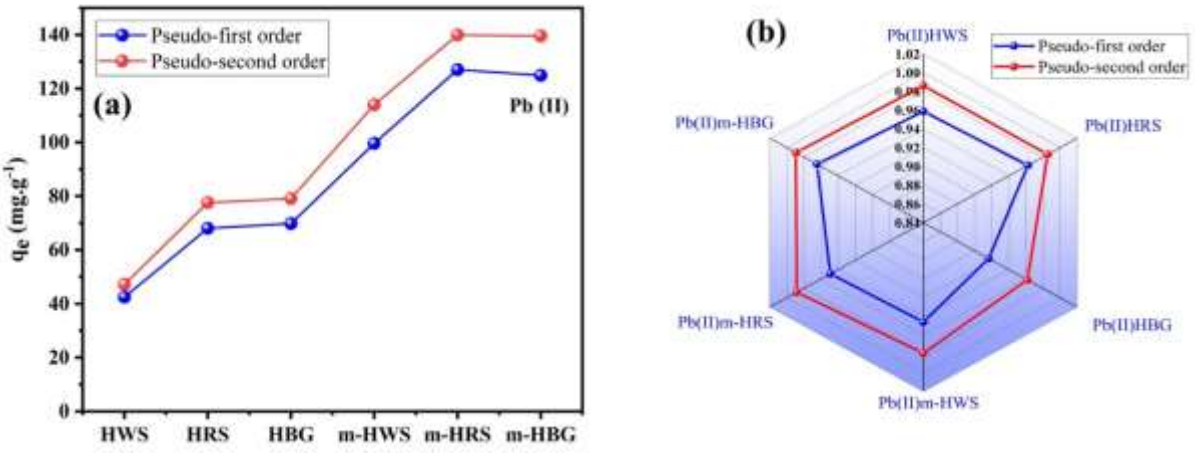

Figure 4: (a) Adsorption capacity of Pb (II) by different Hydrochars at 298 K, (b) Correlation coefficient (R^2) for the Pb (II) in Pseudo 1st order and Pseudo 2nd order

Similarly, for Cu(II) ion, the pseudo-second-order model proved to be a better fit than the pseudo-first-order model, with the highest R^2 value of 0.9828 observed for m-HBG. While the

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overall adsorption capacity for Cu(II) was lower compared to Cd(II) and Pb(II), the microwave-modified hydrochars again outperformed the conventional hydrochars. The highest adsorption capacity for Cu(II) was observed for m-HRS at 30.29 mg/g, followed by m-HBG at 26.61 mg/g. These findings highlight the enhanced adsorption properties of microwave-modified hydrochars, particularly in the adsorption of Cu(II) ions. This can be attributed to the structural improvements introduced during microwave-assisted hydrothermal treatment, which increase the accessibility and availability of adsorption sites on the hydrochar surface (Fig. 5).

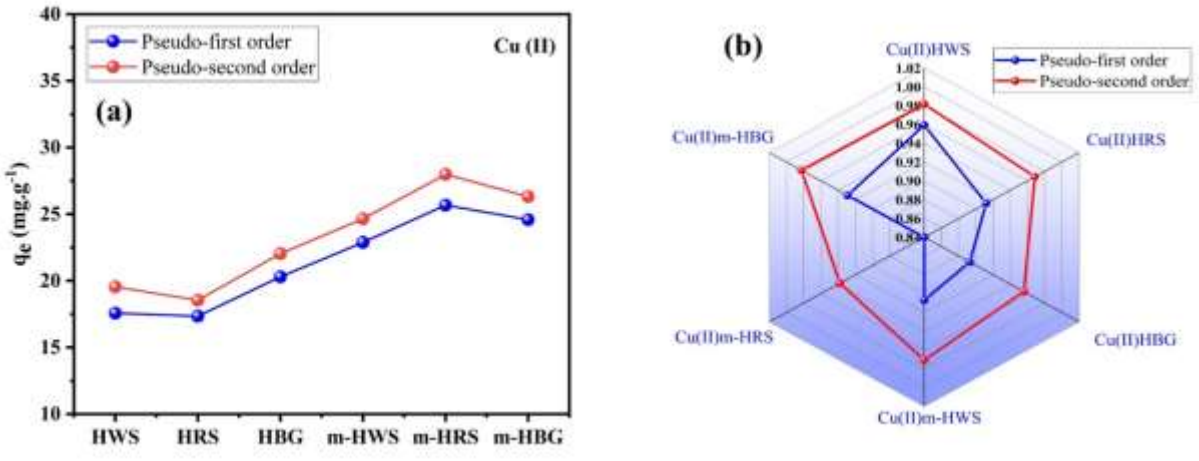

Figure 5: (a) Adsorption capacity of Cu (II) by different Hydrochars at 298 K, (b) Correlation coefficient (R^2) for the Cu (II) in Pseudo 1st order and Pseudo 2nd order

Overall, the data suggest that the pseudo-second-order kinetic model better describes the adsorption processes of Cd(II), Pb(II), and Cu(II) onto hydrochars, indicating that chemisorption is the primary mechanism governing the adsorption of these heavy metal ions. The higher correlation coefficients in the pseudo-second-order model, along with the closeness of the calculated and experimental adsorption capacities (qe), further support the predominance of chemisorption. Additionally, the microwave-modified hydrochars consistently exhibited higher adsorption capacities than their unmodified counterparts, underscoring the effectiveness of microwave treatment in improving the adsorption efficiency of hydrochars for heavy metals.

The enhanced performance of microwave-modified hydrochars can be attributed to several factors, including increased surface area, improved porosity, and the exposure of more functional groups that are responsible for binding metal ions. This makes them highly suitable for applications in water treatment and environmental remediation, particularly for the removal of toxic heavy metals from aqueous solutions. These findings illustrate the potential of hydrochars, especially those subjected to microwave modification, as effective and sustainable adsorbents for heavy metal removal. The superior adsorption capacities observed in this study suggest that these materials could play a crucial role in addressing pollution challenges in industrial wastewater treatment.

Thus, the results emphasize that the pseudo-second-order kinetic model provides a more accurate representation of the adsorption kinetics for Cd(II), Pb(II), and Cu(II) ions, and that microwave-assisted hydrochars have the potential to significantly enhance the adsorption capacities, particularly for metals like Pb(II), where the adsorption capacity reached up to 137.36 mg/g in m-HRS. The data presented in Figures 3, 4, and 5 highlight these trends and confirm the importance of preparation methods

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in optimizing the adsorption properties of hydrochars. The kinetic parameters for Cd(II), Pb(II), and Cu(II) ions adsorption onto different hydrochars, as shown in **Table 1**, provide a deeper understanding of the adsorption process. The experimental qe values (qe, expt.) for each metal ion are compared with the calculated qe values from both the pseudo-first-order and pseudo-second-order models, along with the respective rate constants (k_1 and k_2) and correlation coefficients (R^2).

Table 1. Adsorption kinetics fitting for pseudo-first order and pseudo-second order models

Heavy Metals	Hydrochar	Pseudo-first order				Pseudo-second order		
		q_e (expt.) ($mg.g^{-1}$)	q_e (mg·g1)	k_1 (min-1)	$_R2$	q_e (mg.g-1)	K_2 (g.mg-1.min-1)	$_R2$
Cd(II)	HWS	8.41	7.94	0.9937	0.9666	8.73	0.0010	0.9937
	HRS	14.92	13.54	0.9964	0.8624	14.88	0.0003	0.9486
	HBG	43.29	41.54	0.9910	0.9739	45.39	0.0003	0.9759
	m-HWS	50.42	48.08	0.9791	0.9776	50.89	0.0007	0.9971
	m-HRS	52.92	51.06	0.9799	0.9663	53.76	0.0006	0.9894
	m-HBG	50.67	48.22	0.9723	0.9564	50.82	0.0009	0.9966
Pb(II)	HWS	44.88	42.49	0.9940	0.9587	47.26	0.0001	0.9861
	HRS	69.58	68.02	0.9969	0.9625	77.57	0.0005	0.9857
	HBG	71.36	69.81	0.9972	0.9164	79.13	0.0004	0.9618
	m-HWS	102.36	99.62	0.9971	0.9459	114.02	0.0003	0.9790
	m-HRS	137.36	127.05	0.9919	0.9491	139.92	0.0008	0.9884
	m-HBG	131.44	124.91	0.9947	0.9648	139.54	0.0005	0.9894
Cu(II)	HWS	18.28	17.57	0.9949	0.9593	19.54	0.0003	0.9816
	HRS	18.45	17.34	0.9887	0.9121	18.54	0.0010	0.9687
	HBG	21.58	20.30	0.9935	0.8929	22.04	0.0004	0.9558
	m-HWS	24.63	22.88	0.9887	0.9070	24.65	0.0007	0.9708
	m-HRS	30.29	25.67	0.9877	0.8309	27.98	0.0006	0.9381
	m-HBG	26.61	24.58	0.9854	0.9292	26.32	0.0008	0.9828

CONCLUSION

The study investigated the adsorption kinetics of Cd(II), Pb(II), and Cu(II) onto hydrochars prepared from agricultural residues, including wheat straw, rice straw, and bagasse, using both conventional and microwave-assisted hydrothermal treatments. The adsorption behavior was evaluated using pseudo-first-order and pseudo-second-order kinetic models to assess the mechanisms governing the process. The results revealed that the pseudo-second-order kinetic model consistently provided a better fit across all hydrochars and metal ions, with higher correlation coefficients (R^2 values) and close agreement between the experimental and calculated adsorption capacities (qe). This suggests that chemisorption, involving valence forces and ion exchange, is the predominant mechanism driving the adsorption of Cd(II), Pb(II), and Cu(II) on hydrochars. The microwave-assisted hydrochars demonstrated enhanced adsorption capacities compared to their conventionally produced counterparts, particularly for Pb(II), where the highest capacity (137.36 mg/g) was observed for m-HRS. Additionally, the study underscores the influence of preparation conditions on the adsorption performance of hydrochars. The microwave modification significantly improved surface characteristics, resulting in higher adsorption capacities across all metal ions. Among the hydrochars, m-HRS consistently showed superior adsorption capabilities, demonstrating its potential for efficient removal of heavy metals from aqueous solutions. In conclusion, the findings indicate that hydrochars, especially those modified using microwave treatment, are promising adsorbents for heavy metal removal. The dominance of the pseudo-second-order kinetic model suggests that future adsorption studies should consider chemisorption as a key factor. Furthermore, optimizing preparation methods, such as microwave-assisted hydrothermal carbonization, could enhance the efficacy of hydrochars in environmental remediation applications, particularly in water purification and heavy metal sequestration.

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