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Hydrochar Derived from Invasive *Pontederia crassipes*: *In Situ* Modification and Application for Methylene Blue Adsorption

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In this study, hydrochar was prepared from the invasive aquatic plant *Pontederia crassipes* through hydrothermal carbonization and modified *in situ* using zirconium oxychloride ($ZrOCl_2$), poly(acrylic acid) (PAAC), sulphuric acid (H_2SO_4) and phosphoric acid (H_2PO_4). The synthesized materials were characterized by Fourier-transform infrared spectroscopy (FTIR), scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDX). Their adsorption performance was evaluated for the removal of methylene blue (MB) from aqueous solutions using batch experiments and the adsorption data were analyzed using isotherm and kinetic models. The experimental adsorption capacity of the unmodified hydrochar was 30.96 mg g^{-1}. Modification with $ZrOCl_2$, PAAC, H_2SO_4 and H_3PO_4 resulted in adsorption capacities of 39.21, 30.31, 42.20 and 42.31 mg g^{-1}, respectively. The H_2SO_4- and H_3PO_4-modified hydrochars exhibited the highest adsorption capacities among the prepared materials. The adsorption data were reported to follow the Langmuir isotherm and pseudo-second-order kinetic models.

Keywords: *Pontederia crassipes*, Agrowaste, Hydrochar, Dye adsorption, Water pollution, Methylene blue.

INTRODUCTION

Water pollution from industrial and domestic effluents remains a major environmental concern, threatening aquatic ecosystems and the availability of safe drinking water [1-4]. In India, the growth of textile, leather, paper and other industries has increased the production and use of synthetic dyes [5,6]. Dye-containing effluents can harm aquatic life and pose health risks including skin irritation, allergies and potential mutagenic or carcinogenic effects. Their complex aromatic structures often hinder degradation, while even low concentrations can colour water and reduce light penetration, disrupting photosynthesis in aquatic plants [7,8].

Several methods have been investigated for dye removal from wastewater, including catalytic degradation [9], solid-state reactions [10], chemical precipitation [11] and thermal decomposition [12]. Limitations such as high operating costs, energy demands and complex procedures have encouraged the search for simpler and more economical alternatives [13-18]. In this context, adsorption has gained attention as a practical approach, particularly through the use of agricultural waste as a low-cost adsorbent for removing dyes from contaminated water [19]. Hydrothermal carbonization (HTC) converts biomass into carbon-rich hydrochars, with rice husk [20], wheat straw [21], corncob [22] and sugarcane bagasse [23] among the feedstocks investigated. Surface functional groups retained or modified during HTC can influence dye adsorption and reduce the need for additional functionalization [24,25]. HTC processes wet biomass at approximately 180-250 ºC, potentially avoiding energy-intensive drying [26,27]. The properties and adsorption performance of hydrochar depend on feedstock composition, reaction temperature, residence time and the activating agents used [28-30].

Pontederia crassipes, commonly known as water hyacinth, is a rapidly growing invasive aquatic plant that can disrupt freshwater ecosystems. Dense growth restricts light penetration, competes with native aquatic vegetation and impedes water flow. The accumulation and decomposition of dead biomass

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can further contribute to the deterioration of water quality and the gradual infilling of water bodies [31,32]. Its widespread availability creates an opportunity to convert unwanted plant biomass into useful materials. Using this feedstock for hydrochar production may provide a practical means of managing invasive biomass while generating an adsorbent for wastewater treatment. The value of this approach depends on the properties of the resulting material and its demonstrated ability to remove pollutants under relevant treatment conditions.

The present study investigates the preparation and characterization of zirconium modified hydrochar derived from *P. crassipes* through hydrothermal carbonization and examines the influence of different activating agents on its physicochemical properties and adsorption performance towards methylene blue. Batch adsorption experiments were conducted to evaluate the effects of contact time and initial dye concentration on dye removal. Methylene blue dye was selected as a model pollutant owing to its widespread use in textile dyeing and its relevance to dye containing industrial wastewater [33]. The study examines the relationship between hydrochar preparation, material characteristics and adsorption behaviour, with the aim of assessing the suitability of *P. crassipes* derived hydrochar as a low-cost adsorbent for aqueous dye removal.

EXPERIMENTAL

Pontederia crassipes plant was collected from the local neighbourhood and all the chemicals were procured from standard suppliers. Zirconium oxychloride octahydrate ($ZrOCl_2 \cdot 8H_2O$; CAS No. 13520-92-8) was purchased from Spectrochem Pvt. Ltd., Mumbai, India. Polyacrylic acid (PAAC; CAS No. 9003-04-7) was obtained from Sigma-Aldrich, USA, while sulphuric acid (H_2SO_4; CAS No. 7664-93-9) was supplied by Avantor Performance Materials India Ltd., Thane, India. Orthophosphoric acid (H_3PO_4; CAS No. 7664-38-2) was procured from Central Drug House (P) Ltd., New Delhi, India. Methylene blue powder (*m.w.* 319.96 g mol^{-1}) was obtained from S.D. Fine-Chem Ltd., India. The chemicals were used without further purification and distilled water was used throughout the experiments.

Characterization: The synthesized hydrochar was characterized using FE-SEM coupled with EDX (Zeiss Supra 55, Oxford X-MAX 20) to examine its surface morphology and elemental composition. Surface functional groups were identified by FTIR spectroscopy (Bruker ALPHA II) over 4000-400 cm^{-1}. Methylene blue concentration was measured at 664 nm using a UV-visible spectrophotometer (Hitachi UH5300) to evaluate dye adsorption.

Preparation of stock dye solution: A stock solution of methylene blue (MB) at 300 mg L^{-1} was prepared by dissolving a weighed amount of dye in distilled water and storing it in an Amber glass bottle in the dark to minimise light-induced degradation. Working solutions with concentrations of 10, 20, 30, 50, 75, 100, 150 and 200 mg L^{-1} were prepared by diluting the stock solution with distilled water. The UV-Vis absorbance measurements of the working solutions were used to construct a calibration curve.

Synthesis of hydrochar: The plant material was dried at 80 ºC for 24 h and ground into a fine powder. For the preparation of unmodified hydrochar, 10 g of powdered plant material was transferred into an 80 mL Teflon-lined autoclave and 35 mL of distilled water was added. The autoclave was sealed and heated at 220 ºC for 12 h. After the reaction, the autoclave was allowed to cool to room temperature. The resulting solid product was separated by filtration and washed several times with distilled water until the filtrate became transparent to remove water-soluble organic impurities. The recovered solid was then dried at 60 ºC for 6 h. The resulting unmodified hydrochar, designated as HC-I, had a yield of 5.107 g.

For the preparation of zirconium-modified hydrochar (HC-II), 2.0 g of $ZrOCl_2$ was added to 35 mL of distilled water containing 10 g of powdered plant (PC) material. The mixture was then subjected to the same hydrothermal treatment described above. Similarly, PAAC-modified hydrochar (HC-III) was prepared by adding 1.0 g of PAAC to 35 mL of distilled water, followed by the addition of 10 g of powdered PC. For the preparation of sulphuric acid-modified (HC-IV) and phosphoric acid-modified (HC-V) hydrochars, 5 mL of H_2SO_4 and 5 mL of H_3PO_4, respectively, were each mixed with 30 mL of distilled water containing 10 g of powdered plant. Each mixture was subsequently subjected to hydrothermal treatment at 220 ºC for 12 h under the same conditions used for HC-I. After treatment, the products were recovered by filtration, washed with distilled water and dried at 60 ºC for 6 h, following the procedure described above. The yields of HC-II, HC-III, HC-IV and HC-V were 5.673, 4.435, 3.379 and 3.103 g, respectively. All the 5 hydrochars were used in subsequent experiments to investigate their adsorption per-formance toward methylene blue dye.

Dye adsorption studies: Batch adsorption experiments were conducted to evaluate the removal of methylene blue dye from aqueous solutions using the prepared hydrochars. In each experiment, 5 mL of the dye solution was transferred into a 15 mL centrifuge tube and 20 mg of adsorbent was added. The mixture was agitated for a specified contact time under controlled experimental conditions. The effects of adsorbent dosage, contact time and initial dye concentration were investigated by varying the relevant parameters while keeping the other experimental conditions constant.

Adsorption isotherm studies were performed by varying the initial MB concentration while maintaining a constant adsorbent dosage and temperature. The suspensions were agitated until adsorption equilibrium was reached. The equilibrium data were analyzed using the Langmuir and Freundlich isotherm models. The Langmuir model assumes monolayer adsorption on a homogeneous surface, whereas the Freundlich model describes adsorption on heterogeneous surfaces [34].

Adsorption kinetics were investigated at a fixed initial dye concentration and adsorbent dosage. Samples were collected at contact times of 5, 10, 15, 30, 60, 120, 240 and 360 min to evaluate the time-dependent adsorption behaviour. The experimental data were fitted to the pseudo-first-order and pseudo-second-order kinetic models to characterize the adsorption rate and kinetics. After the designated contact time, the suspensions were centrifuged at 3500 rpm for 10 min to separate the

adsorbent from the aqueous phase. A 3 mL aliquot of the clear supernatant was collected and the residual MB concentration was determined using a UV-Vis spectrophotometer at the maximum absorption wavelength (λ_{max}) of 664 nm.

RESULTS AND DISCUSSION

The adsorbent materials were prepared by hydrothermal carbonization (HTC) of the plant-derived precursor (PC) followed by modification using different activating agents. These modifications were intended to improve the surface properties of the resulting hydrochars, particularly their porosity, surface functional groups and availability of active adsorption sites, thereby enhancing their potential for methylene blue dye removal. The effects of different activation treatments on the adsorption performance of the hydrochars were investigated and compared with those of the unmodified material.

FTIR: The surface functional groups of the prepared hydrochars were investigated by FTIR spectroscopy over the wavenumber range of 4000-400 cm^{-1}. All five hydrochar samples exhibited a broad absorption band in the 3600-3400 cm^{-1} region, attributed mainly to O–H stretching vibrations (Fig. 1). The lower intensity of this band in HC-IV and HC-V, prepared using H_2SO_4 and H_3PO_4, respectively, suggests a reduction in the abundance of hydroxyl groups or changes in their chemical environment during hydrothermal treatment. This difference may be associated with acid-catalyzed dehydration and condensation reactions that promote structural rearrangement and carbonization of the plant-derived precursor. Such processes can contribute to the formation of more condensed carbon structures, although FTIR data alone cannot establish the extent of aromatization or carbonization [35-37].

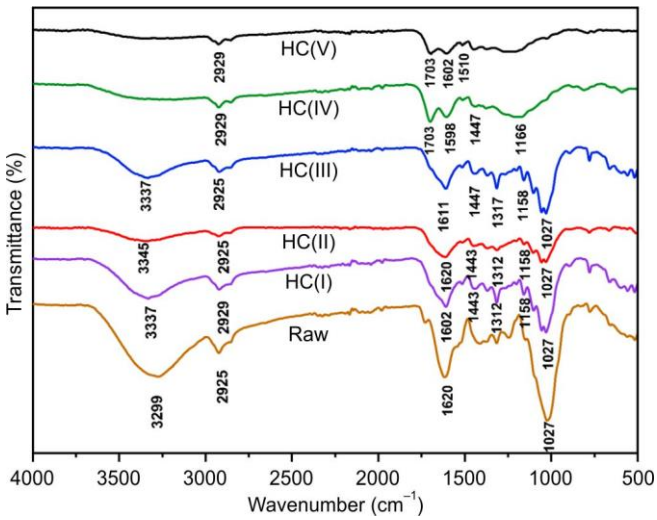


Fig. 1. FTIR spectra of raw and modified hydrochars (HC)

Absorption bands in the 2950-2900 cm^{-1} region were observed in all the 5 hydrochars and were assigned to C–H stretching vibrations associated with aliphatic groups [38]. The bands in HC-IV and HC-V exhibited greater intensity than those in the other samples. This difference may reflect variations in the relative abundance or chemical environment of aliphatic C–H groups following acid treatment. The observed intensity changes should be interpreted cautiously, as they may also be influenced by sample preparation, spectral normalization and differences in band overlap.

The absorption bands within the 1794-1603 cm^{-1} region were assigned to carbonyl (C=O) stretching vibrations associated with carbonyl- and carboxylic acid groups [39]. The precise assignment of individual bands requires consideration of their exact positions and the possible contribution of overlapping vibrations. Bands in the 1500-1400 cm^{-1} region may be associated with aromatic C=C vibrations and other skeletal vibrations, while absorption in the 1380-1350 cm^{-1} region may arise from C–H bending and related vibrations. Bands around 1300-1200 cm^{-1} may be attributed to C–O stretching vibrations of oxygen functional groups such as phenolic, alcoholic and carboxylic groups.

A distinct absorption band at 1172 cm^{-1} was observed for HC-IV (Fig. 1). This band may be associated with S–O stretching vibrations of sulphur species introduced during H_2SO_4 treatment [40]. Bands in the 1027-1022 cm^{-1} region were observed in all modified hydrochars and were assigned primarily to C–O stretching vibrations of oxygenated groups [38]. The unmodified sample did not exhibit a clearly distinguishable band in this region. Absorption bands between 1000 and 1091 cm^{-1} may additionally arise from C–O–C stretching vibrations. The fingerprint region below 1000 cm^{-1} contains several overlapping vibrational modes, which make definitive assignments difficult to characterize [39].

The comparatively lower intensity of the O–H stretching bands in HC-IV and HC-V suggests that sulphuric acid and phosphoric acid treatment altered the surface chemistry of the hydrochars, potentially through dehydration and condensation reactions. These changes may influence the availability and nature of adsorption sites for methylene blue.

SEM and EDX: The surface morphology and elemental composition of the prepared hydrochars were examined using SEM and EDX, respectively. The SEM images of all five samples (HC-I–HC-V) revealed heterogeneous surface morphologies characterized by irregular surfaces and varying degrees of roughness (Fig. 2). These morphological features indicate differences in surface texture among the hydrochars, which may influence the accessibility of adsorption sites during methylene blue removal.

The EDX spectrum of HC-II confirmed the presence of carbon and zirconium, with carbon and zirconium accounting for 96.31 and 3.69 wt.%, respectively. Their corresponding atomic percentages were 99.50 and 0.50 at.%, respectively, supporting the incorporation of zirconium-mediated species into the hydrochar matrix. The EDX analysis of HC-V revealed carbon and oxygen as the major detected elements. Carbon accounted for 91.39 at.% and 88.85 wt.%, whereas oxygen contributed 8.61 at.% and 11.15 wt.%. The high carbon content of HC-V may be associated with the carbonization of the plant-derived precursor during hydrothermal treatment in the presence of phosphoric acid. Similar effects of acid treatment on carbon-rich materials have been reported in the literature [41,42].

Isotherms studies: Adsorption isotherms were used to describe the relationship between the equilibrium concentra-

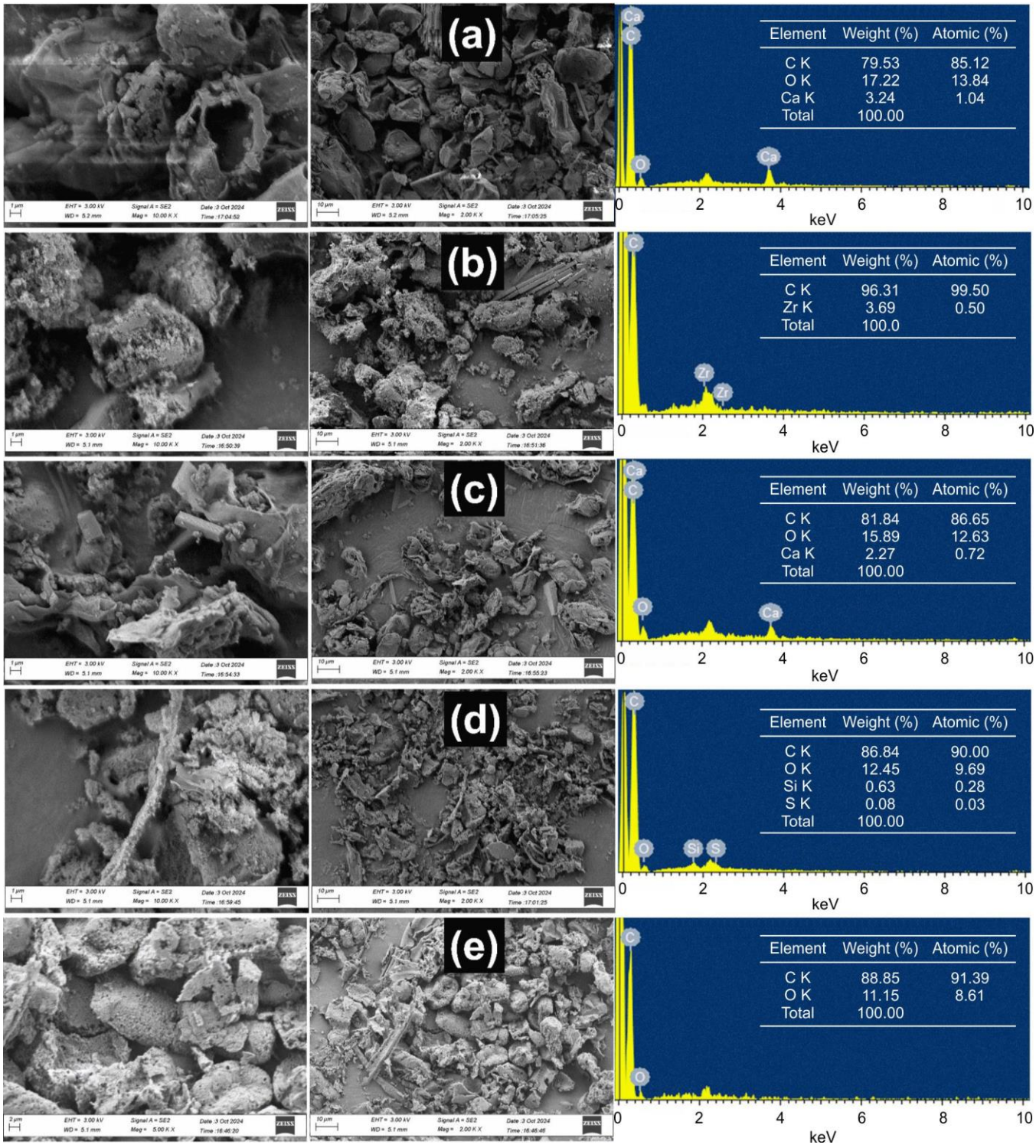


Fig. 2. SEM and EDX images of hydrochars (a) HC-I, (b) HC-II, (c) HC-III, (d) HC-IV, and (e) HC-V

tion of a dye in solution and the amount adsorbed onto the adsorbent at a constant temperature. In this study, the experimental equilibrium data were analyzed using the Langmuir and Freundlich isotherm models. The Langmuir model assumes monolayer adsorption at a finite number of equivalent and energetically uniform adsorption sites, with no interaction between adsorbed molecules. The Freundlich model is an empirical equation used to describe adsorption on heterogeneous surfaces and can account for non-uniform adsorption energies. Accordingly, the experimental data were fitted to the Langmuir (eqn. 1) and Freundlich (eqn. 2) isotherm equations to evaluate the adsorption behaviour of methylene blue on the prepared hydrochars.:

$$\frac{1}{Q_e} = \frac{1}{Q_m} + \left(\frac{1}{K_L Q_m}\right)\left(\frac{1}{C_e}\right) \quad (1)$$

$$\ln Q_e = \ln K_F + \frac{1}{n}\ln C_e \quad (2)$$

where K_L and K_F represent the Langmuir and Freundlich constants, respectively, Q_e (mg g^{-1}) is the adsorption capacity at equilibrium. The equilibrium concentration of dye solution is C_e (mg L^{-1}), whereas the theoretical adsorption capacity for monolayer adsorption is represented by Q_m. The Freundlich constant (1/n) provides insight into the adsorption intensity and favourability of the adsorption process. Table-1 presents the Langmuir and Freundlich isotherm parameters, along with their corresponding coefficients of determination (R^2). The R^2 values obtained for the Freundlich model were 0.9999 for HC-V, 0.9997 for HC-II, 0.9995 for HC-III, 0.9999 for HC-IV and 0.9996 for HC-I. These values indicate an excellent correlation between the experimental data and the Freundlich model. The corresponding 1/n values were 0.9765, 0.9463, 0.9005, 0.9791 and 0.8933 for HC-V, HC-II, HC-III, HC-IV and HC-I, respectively. All values were below 1, which indicate favourable adsorption of methylene blue dye molecules onto the prepared hydrochars.

Kinetics studies: The adsorption kinetics of the prepared functionalized hydrochars were investigated to understand the rate and temporal behaviour of MB uptake from aqueous solutions. The experimental data were analyzed using the pseudo-first-order (eqn. 3) and pseudo-second-order (eqn. 4) kinetic models. These models were used to evaluate the adsorption rate constants and assess the agreement between the predicted and experimental adsorption capacities. The kinetic parameters obtained from both models were compared to determine, which model best described the adsorption behaviour of the prepared hydrochars:

$$\log(Q_e - Q_t) = \log Q_t - \frac{K_1}{2.303}t \quad (3)$$

$$\frac{t}{Q_t} = \frac{1}{K_2 Q_e^2} + \frac{t}{Q_e} \quad (4)$$

The adsorption capacities of the hydrochars at equilibrium (Q_e) and at time (t) Q_t were expressed in mg g^{-1}. The rate constants k_1 and k_2 correspond to the pseudo-first-order and pseudo-second-order kinetic models, with units of min^{-1} and g mg^{-1} min^{-1}, respectively. The kinetic parameters were determined from the linear plots derived from eqns. 3 and 4. For the pseudo-first-order model, a plot of $\log(Q_e - Q_t)$ against time (t) was used to determine the rate constant (k_1) from the slope and the theoretical equilibrium adsorption capacity from the intercept. For the pseudo-second-order model, a plot of t/Q_t against t was used to calculate the equilibrium adsorption capacity (Q_e) from the reciprocal of the slope and the rate constant (k_2) from the slope and intercept.

The adsorption kinetics of the prepared hydrochars were evaluated using pseudo-first-order and pseudo-second-order models. The pseudo-first-order model yielded coefficients of determination (R^2) of 0.4823, 0.6366, 0.9555, 0.6079 and 0.7388 for HC-I, HC-II, HC-III, HC-IV and HC-V, respectively. In comparison, the pseudo-second-order model provided substantially higher R^2 values of 0.9997, 0.9996, 0.9997, 0.9966 and 0.9980, respectively (Table-2). These results indicate that the pseudo-second-order model better describes the kinetic data for all five hydrochars. The corresponding calculated equilibrium adsorption capacities were 26.67, 33.44, 30.77, 42.02 and 43.29 mg g^{-1}, respectively. The experimental equilibrium capacities listed in Table-2 were 30.99, 39.21, 30.31, 42.21 and 42.32 mg g^{-1} for the same samples. The adsorption performance varied with the modification of the hydrochar surface. The acid-treated materials, HC-IV and HC-V, exhibited the highest experimental equilibrium adsorption capacities, whereas HC-II showed an improvement over the unmodified hydrochar, HC-I. HC-III did not show a comparable improvement under the investigated conditions. The enhanced MB uptake by HC-IV and HC-V may be associated with changes in surface functional groups and pore accessibility following acid treatment. The oxygenated groups, such as hydroxyl and carboxyl groups, may contribute to interactions

TABLE-1
LANGMUIR AND FREUNDLICH ISOTHERM CONSTANTS AND CORRELATION COEFFICIENTS FOR ADSORPTION OF DYES ON DIFFERENT HYDROCHARS

	Langmuir constants				Freundlich constants		
	Q_m	K_L	R_L	R^2	K_F	1/n	R^2
HC-I	181.8182	0.001397	0.781638	0.9996	0.346248	0.8933	0.9901
HC-II	263.1579	0.000958	0.839194	0.9997	0.291009	0.9463	0.9981
HC-III	232.5581	0.001035	0.828538	0.9995	0.324523	0.9005	0.9919
HC-IV	1428.571	0.000175	0.966099	0.9999	0.267215	0.9791	0.9995
HC-V	1111.110	0.000226	0.956812	0.9999	0.269200	0.9765	0.9994

TABLE-2
PSEUDO-FIRST-ORDER AND PSEUDO-SECOND-ORDER CONSTANTS AND CORRELATION COEFFICIENTS FOR ADSORPTION OF DYE WITH DIFFERENT ACTIVATIONS OF HYDROCHARS

	Pseudo-first-order kinetics				Pseudo-second-order kinetics		
	Q_e (mg g^{-1}) (experimental)	Q_e (mg g^{-1})	k_1 (min^{-1})	R^2	Q_e (mg g^{-1})	k_2 (g mg^{-1} min)	R^2
HC-I	30.99056	2.070638	0.004836	0.4823	26.66667	0.008611	0.9997
HC-II	39.2146	5.884093	0.004606	0.6366	33.44482	0.005321	0.9996
HC-III	30.31215	0.967486	0.018424	0.9555	30.76923	0.005744	0.9997
HC-IV	42.20516	2.901415	0.006218	0.6079	42.01681	0.002783	0.9966
HC-V	42.31592	0.80383	0.026715	0.7388	43.29004	0.00356	0.998

TABLE-3
COMPARISON OF METHYLENE BLUE ADSORPTION CAPACITY OF *P. crassipes*-DERIVED HYDROCHARS WITH OTHER REPORTED HYDROCHAR-DERIVED ADSORBENTS

Adsorbent	Isotherm	Kinetics	Maximum adsorption capacity, Q_m (mg/g)	Ref.
Pontederia crassipes (HC-IV)	Langmuir	Pseudo-second order	1428.57	This work
Pontederia crassipes (HC-V)	Langmuir	Pseudo-second order	1111.11	This work
Walnut shell powder	Langmuir	Pseudo-second order	36.63	[49]
Coffee husk	Langmuir	Pseudo-second order	418.78	[50]
Tectona grandis sawdust	Langmuir	Pseudo-second order	172.41	[51]
Citrus peel	Langmuir	Pseudo-second order	185.83	[52]
Banana peel waste	Langmuir	Pseudo-second order	991	[53]

with cationic MB molecules through electrostatic attraction and hydrogen bonding [43-46]. The FTIR results, particularly the changes in the O–H stretching bands, also support alterations in the surface chemistry of the acid-treated materials.

The differences between HC-IV and HC-V may reflect the distinct effects of sulphuric acid and phosphoric acid on the hydrochar structure and surface chemistry. Sulphuric acid can promote dehydration and condensation reactions, while phosphoric acid may influence pore development and the formation of phosphorus-functionalized surface groups [47,48]. However, the slight higher experimental equilibrium capacity of HC-V (42.32 mg g^{-1}) compared with HC-IV (42.21 mg g^{-1}) does not support a claim that sulphuric acid activation produces superior adsorption performance. Although the EDX results confirmed zirconium in HC-II and carbon and oxygen as the major detected elements in HC-V, these measurements do not establish the presence of specific sulphonic acid or phosphate groups.

Comparative studies: Table-3 compares the methylene blue adsorption performance of the *Pontederia crassipes*-derived hydrochars with that of other biomass-based adsorbents reported in the literature. The Langmuir maximum adsorption capacities (Q_m) calculated for HC-IV and HC-V were 1428.57 and 1111.11 mg g^{-1}, respectively. These fitted values are higher than those reported for walnut shell powder (36.63 mg g^{-1}), *Tectona grandis* sawdust (172.41 mg g^{-1}), citrus peel (185.83 mg g^{-1}), coffee husk (418.78 mg g^{-1}) and banana peel waste (991 mg g^{-1}). Nevertheless, the calculated (Q_m) values differ substantially from the experimentally determined equilibrium adsorption capacities of approximately 42.21 mg g^{-1} for HC-IV and 42.32 mg g^{-1} for HC-V.

Conclusion

This study demonstrates the potential of *Pontederia crassipes*, an invasive aquatic plant, as a precursor for hydrochar production and methylene blue removal from aqueous solutions. The unmodified hydrochar exhibited an experimental adsorption capacity of 30.96 mg g^{-1}, while modification with $ZrOCl_2$, PAAC, H_2SO_4 and H_3PO_4 resulted in capacities of 39.21, 30.31, 42.20 and 42.31 mg g^{-1}, respectively. Among the prepared materials, the $H_2SO_4^-$ and $H_2PO_4^-$ modified hydrochars exhibited the highest adsorption capacities, which indicate that acid modification can improve methylene blue adsorption under the conditions investigated.

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CONFLICT OF INTEREST

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

DECLARATION OF AI-ASSISTED TECHNOLOGIES

During the preparation of this manuscript, the authors used an AI-assisted tool(s) to improve the language and no data, results or scientific conclusions were generated by AI. All analyses, interpretations and conclusions are the authors' own. The authors reviewed and edited the content and take full responsibility for the published work.

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