



Nitric-acid-activated biochars from agricultural wastes for methylene blue removal: characterization and pseudo-second-order adsorption kinetics

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ABSTRACT

The discharge of synthetic dyes such as methylene blue (MB) into water bodies poses a serious environmental and health concern, driving the search for low-cost, sustainable adsorbents. In this work, five biochars were synthesized from agricultural biomass wastes pineapple leaves (PL), pineapple peels (PP), sunflower seed hulls (SH), macadamia nut shells (MS) and flamboyant pods (FP) through nitric-acid activation followed by pyrolysis at 500 °C, and were evaluated for MB removal from aqueous solution. The materials were characterized by X-ray diffraction (XRD), Fourier-transform infrared spectroscopy (FT-IR), scanning electron microscopy (SEM) and N₂ adsorption-desorption analysis. XRD confirmed an amorphous, turbostratic carbon structure, while FT-IR revealed abundant oxygen- and nitrogen-containing functional groups generated by acid activation. SEM showed precursor-dependent morphologies, with FP exhibiting an open, interconnected macroporous network. N₂ isotherms were of type IV, and the BET surface areas increased in the order SH < PL < PP < MS < FP. Batch experiments showed that MB removal reached equilibrium within 120 min, with FP reaching 92%. FP, combining the highest surface area, largest pore volume and richest surface functionality, was identified as the optimum adsorbent. Under the identical, low-severity activation conditions used here the FP biochar reaches a Langmuir monolayer capacity of 2.43 mg.g⁻¹. The value of the present work therefore lies in the systematic comparison of five agricultural residues under a common protocol, and in identifying flamboyant pods as the most promising precursor for further activation, rather than in the absolute capacity of the material as prepared.

Introduction

The rapid expansion of the agricultural sector generates millions of tonnes of lignocellulosic residues every year fruit peels, seed hulls, nut shells, leaves and crop straws, most of which are burned or landfilled, creating an additional environmental burden. Converting these

abundant, renewable and low-cost wastes into value-added carbon materials, particularly biochar and activated carbon, has therefore attracted considerable interest as a strategy that simultaneously addresses solid-waste management and water pollution. A wide variety of residues have been successfully valorized in this way, including pineapple waste [1], rice husk [2], and sawdust

[3], while biomass can even be co-processed with plastic waste to yield functional carbons [4]. Pyrolysis of such feedstocks produces a porous, carbon-rich solid whose surface can be further enriched in oxygen-containing functional groups by chemical activation, for example with nitric acid, making it an attractive and sustainable adsorbent for water treatment.

Among the many pollutants released into natural waters, synthetic dyes are of particular concern because of their massive consumption in the textile, leather, paper, food and printing industries and their frequent discharge in poorly treated effluents [5]. Methylene blue, a cationic thiazine dye, is one of the most widely used and most intensively studied of these colourants. Even at low concentrations its intense colour reduces light penetration and disrupts aquatic photosynthesis, while the molecule itself is toxic, stable and poorly biodegradable. Exposure to MB can cause harmful effects in humans, including eye and skin irritation, nausea, vomiting, increased heart rate and, in severe cases, methemoglobinemia. The persistence and toxicity of MB make its removal from wastewater before discharge an important environmental priority, and substantial effort has been devoted to degrading or capturing it, for instance through photocatalysis [6] and adsorption.

Several technologies have been developed for dye removal [7], including adsorption [8], photocatalytic degradation, coagulation–flocculation, membrane filtration and biological treatment. Among these, adsorption remains the most widely applied owing to its simplicity, low cost, high efficiency and ease of operation. A range of conventional adsorbents has been employed, the most prominent being activated carbon, which offers a high surface area and strong affinity for organic pollutants. Other materials, such as chitosan and chitosan-based nanocomposites [5], clays, zeolites, agricultural by-products and, increasingly, biochar and engineered biochar composites [9], have also been explored. Nevertheless, commercial activated carbon is relatively expensive and difficult to regenerate, which motivates the search for cheaper alternatives. In this context, biochars derived from agricultural wastes and activated with HNO_3 [3] represent a promising, low-cost and environmentally friendly option, which provides the rationale for the present study.

Experimental

Materials and Chemicals

Agricultural biomass wastes comprising pineapple leaves (PL), pineapple peels (PP), sunflower seed hulls (SH), macadamia nut shells (MS), and flamboyant pods

(FP) were collected and utilized as precursor materials for biochar synthesis.

All the chemicals were used as received without further purification. Nitric acid (HNO_3 , 65%) was purchased from Xilong Scientific Co., Ltd. Methylene blue (MB) was obtained from HiMedia Laboratories Pvt. Ltd..

Methods

Five biochar samples derived from PL, PP, SH, MS, and FP were synthesized using the following procedure under identical experimental conditions. The collected biomass was thoroughly washed to remove impurities, subsequently sun dried for two days, and then pulverized into a fine powder. The activation ratio of 1 g biomass : 1 mL concentrated HNO_3 : 6 mL distilled water was adapted from [1], where it was reported to introduce oxygenated surface functionality on lignocellulosic char without excessive loss of yield. The mixture was continuously stirred for 24 hours to effectively achieve surface activation of the sample. After activation, the biomass was washed repeatedly with distilled water until a neutral pH ($\text{pH} = 7$) was achieved to remove residual acid. The material was then centrifuged and dried at 150 °C for 3 hours. The activated biomass was subsequently pyrolyzed under a nitrogen (N_2) atmosphere at 500 °C for 1 hour to produce biochar.

The adsorption of MB onto the synthesized five types of biochar was evaluated as a function of contact time at specific intervals spanning 0, 20, 60, 80, 100, and 120 minutes. For each experiment, a fixed mass of 0.45 g of the respective biochar was introduced into a round bottom flask containing 50 mL of the MB solution (10 mg/L). To eliminate any potential interference from ambient light and prevent photolytic reactions, the entire system was completely isolated from light exposure throughout the procedure. Following the adsorption process, the spent adsorbent was separated from the aqueous phase by filtering the suspension through a 0.22 μm mixed fiber membrane syringe filter supplied by Labfil, PRC, thereby yielding a clear filtrate for subsequent spectrophotometric analysis.

The effects of adsorbent dosage and of initial dye concentration were then examined on the FP biochar. For the dosage series, 0.05–0.45 g of dried FP biochar (nine masses in 0.05 g increments) was added to 50 mL of a 10 mg.L^{-1} MB solution. For the isotherm, a fixed mass of 0.45 g of FP biochar was added to 50 mL of MB solution at initial concentrations of 10, 20, 30, 40, 50, 60, 70, 80, 90 and 100 mg.L^{-1} .

The absorption spectra of MB solutions across various concentrations were recorded over the wavelength range of 300 to 800 nm utilizing UV-Vis spectro-

photometry to identify the maximum absorption wavelength $\lambda_{\max}$ = 664 nm. Following the determination of $\lambda_{\max}$, the absorbance values of the initial calibration standards were measured at this specific wavelength to construct a linear calibration curve. The variations in MB concentration during the subsequent adsorption experiments were quantitatively calculated based on the solution absorbance monitored via a Phoenix Instrument SP-V1000 UV-Vis spectrophotometer. X-ray diffraction (XRD) patterns were recorded using a Bruker D8 ADVANCE diffractometer with CuK α radiation (40 kV, 25 mA, 0.1541 nm) across a 2θ range of 5–80° with a step size of 0.02 degrees. Morphological characterization was performed via scanning electron microscopy using a Hitachi S-4800 system. The specific surface area, pore size, and pore volume of the biochar were determined using Quantachrome Instruments software (version 11.0).

Results and discussion

The crystalline structure and phase orientation of the synthesized biochar, all five samples were characterized via XRD, with the corresponding profiles illustrated in Fig 1a. All samples display essentially the same profile, dominated by a broad, low-intensity diffraction hump centred at 2θ = 22–24°, together with a weaker, very broad feature in the $2\theta \approx 43^\circ$ region. These two humps correspond, respectively, to the (002) and (100) reflections of graphitic carbon, while the absence of any sharp, well-defined peaks indicates that the materials are essentially amorphous, consisting of randomly oriented, turbostratic aromatic carbon sheets rather than an ordered graphitic lattice. The broadening of the (002) band reflects a low degree of structural ordering and a small stacking height of the aromatic layers, which is typical of biochars pyrolysed at moderate temperature (500 °C). This disordered, defect-rich carbon framework is favourable for adsorption because it generates abundant edge sites and structural imperfections that can act as anchoring points for dye molecules.

The FT-IR spectra in Fig 1b confirm that the HNO₃ activation generated a surface rich in oxygen- and nitrogen-containing functional groups for all five biochars. A broad band in the 3200–3500 cm⁻¹ region is assigned to O–H stretching of hydroxyl and carboxyl groups and adsorbed water. The weak features observed between about 1850 and 2000 cm⁻¹ are attributed to overtone and combination bands of aromatic out-of-plane C–H bending, as reported for activated carbon prepared from the same precursor, the band near 1600 cm⁻¹, together with the weaker features

extending down to 1400 cm⁻¹, is assigned to aromatic (aryl) C=C skeletal stretching, and the shoulder around 1700 cm⁻¹ to C=O stretching of the carboxyl and carbonyl groups introduced by nitric acid oxidation [10]. Carbons oxidised with HNO₃ are reported to have low points of zero charge ($\text{pH}_{\text{PZC}} \approx 2.0\text{--}3.4$) as a result of these strongly acidic groups, so at the near-neutral pH of the MB solutions used here the surface would be expected to be negatively charged and therefore favourable to the uptake of the MB cation [11]. We emphasise that the point of zero charge and the surface charge were not measured in the present work; the relationship between surface chemistry and uptake proposed here is an expectation drawn from the literature and remains to be verified experimentally.

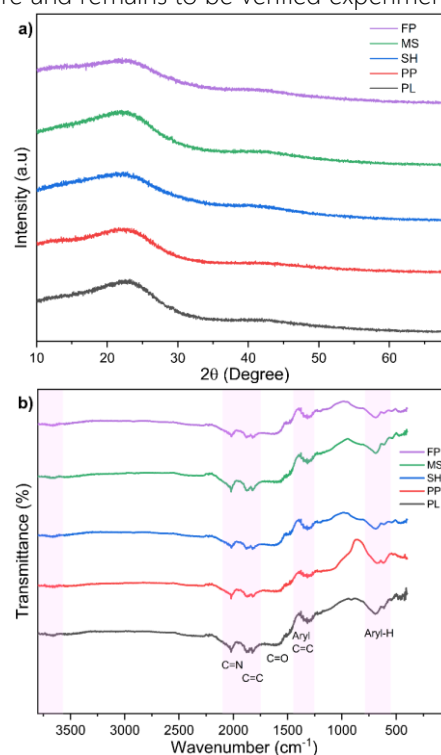


Fig 1: XRD patterns (a) and FT-IR spectra (b) of biochar samples

The surface morphology and porous structure of the synthesized biochars, all five samples were elucidated via SEM, as displayed in the Fig 2. The SEM micrographs reveal distinct morphological variations intrinsically linked to the biological origins of the agricultural precursors. For the pineapple leaves biochar (Fig 2a), the surface exhibits a highly ordered and periodic array of protrusion networks. A highly analogous structural arrangement is observed in the pineapple peels biochar (Fig 2b), which displays a prominent and continuous pattern of nodular protrusions across the carbonaceous framework, originating from the epidermal tissue architecture of the precursor.

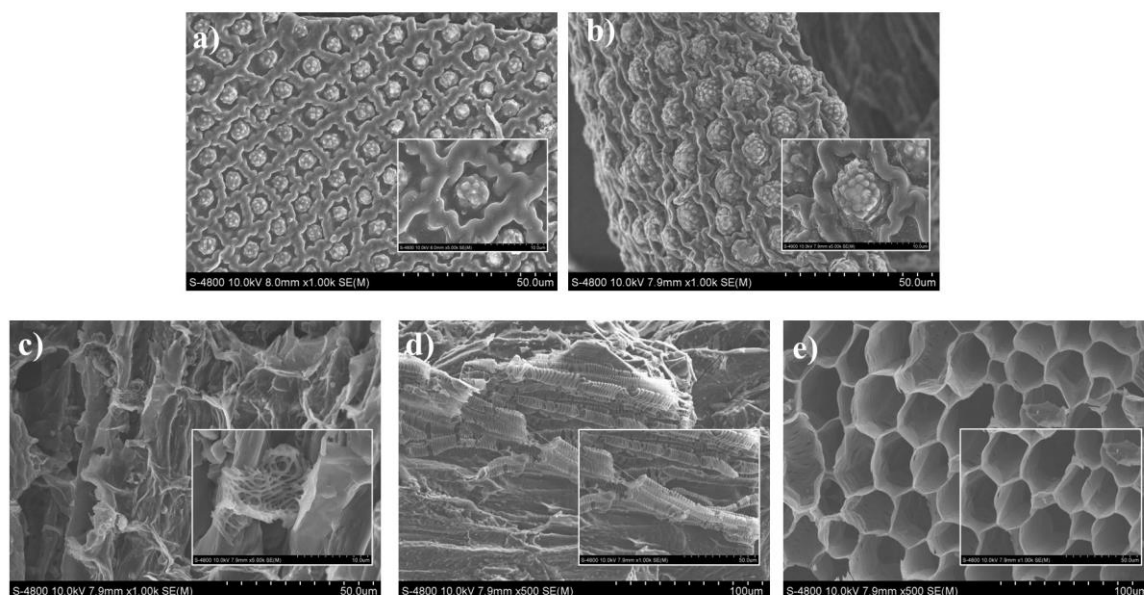


Fig 2: SEM micrographs of the biochars derived from a) PL b) PP c) SH d) MS e) FP. Accelerating voltage 10 kV; magnifications and scale bars are indicated on each panel.

In stark contrast, the biochars derived from hulls, shells, and pods exhibit predominantly fractured and interconnected porous networks rather than surface protrusions. The SH (Fig 2c) presents a highly irregular and rugged topography characterized by fractured carbon layers interspersed with disordered microchannels and localized vascular bundles. For the MS (Fig 2d), the morphology is dominated by elongated, parallel groove tracks and micro-fibrillar channels, reflecting the dense, lignified fibrous conduits typical of hard endocarp tissues. Meanwhile, the FP (Fig 2e) displays a well-developed and highly symmetrical hexagonal honeycomb-like porous infrastructure. This structural network consists of uniformly distributed macro-pores separated by thin, smooth carbonaceous walls, directly derived from the cellular lumen morphology of the plant pod. The diverse structural attributes, ranging from the ordered epidermal protrusions in PL and PP, through the fractured, poorly interconnected layers of SH and the dense, lignified fibrous channels of MS, to the well-developed honeycomb configuration in FP, significantly influence the available surface area and transport pathways, thereby rendering these biochars promising candidates for targeted adsorption applications.

The porous texture of the biochars was probed by N_2 adsorption–desorption measurements at 77 K (Fig 3). All samples exhibit type-IV isotherms with hysteresis loops, indicating the coexistence of mesopores together with macropores and interparticle voids; the steep uptake near saturation reflects condensation in large

meso/macropores. The total adsorbed volume follows the order $FP \gg PP > MS > PL > SH$, in agreement with the BET surface areas, which increase in the same sequence: SH ($6.29 \text{ m}^2/\text{g}$) < PL ($13.86 \text{ m}^2/\text{g}$) < PP ($25.34 \text{ m}^2/\text{g}$) < MS ($49.27 \text{ m}^2/\text{g}$) < FP ($121.57 \text{ m}^2/\text{g}$).

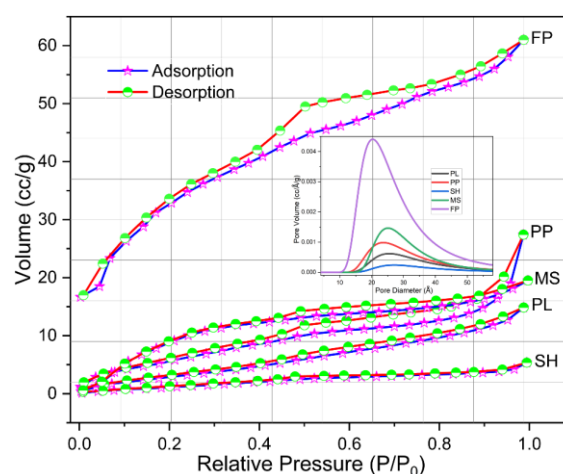


Fig 3: N_2 adsorption–desorption isotherms of the biochars and inset DA pore size distribution

The two rankings coincide except for the PP/MS pair: PP takes up a larger total volume than MS although its BET area is only half as large, indicating that the porosity of PP is dominated by wide meso- and macropores that contribute volume but comparatively little specific surface, whereas MS contains a greater fraction of narrow pores. The inset pore-size distributions are centred in the small-mesopore range (2–3 nm), and the FP sample shows by far the largest pore volume. The combination of the highest BET surface area, the largest

pore volume and the open macroporous morphology gives FP the greatest number of accessible adsorption sites, explaining why it removes MB most effectively. Conversely, the very low surface area of SH limits the number of available sites and accounts for its lowest removal efficiency

The removal of MB as a function of contact time is presented in Fig 4a. For every biochars the uptake was very rapid during the first 20 min, owing to the large number of vacant active sites and the steep concentration gradient between the bulk solution and the surface, after which it increased only gradually and reached equilibrium at about 100–120 min. The equilibrium removal efficiency decreased in the order FP > PP > MS > PL > SH.

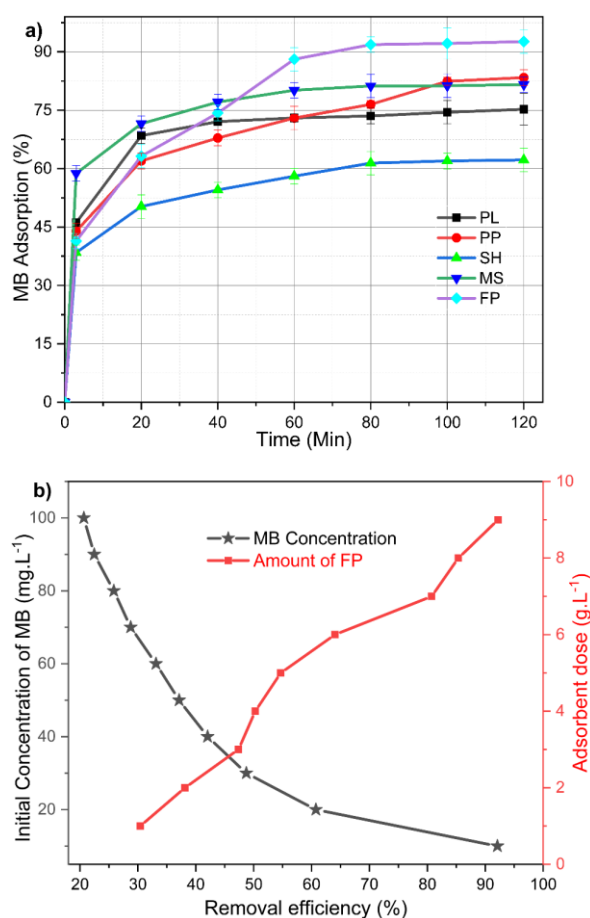


Fig 4: (a) MB removal versus contact time for the five biochars; (b) effect of MB concentration and adsorbent dose on the removal efficiency of the optimum FP biochar.

This ranking correlates strongly with the textural and chemical properties described above: FP, the most porous and the most heavily functionalised of the five materials, and therefore offers both the most accessible and the most chemically active sites for the cationic dye. The influence of the initial dye concentration on the

methylene blue (MB) adsorption efficiency of the optimized FP biochar was investigated across a concentration range of 10 to 100 mg/L, with the corresponding profile depicted in Fig 4b. The experimental results demonstrate a clear and progressive decline in the adsorption efficiency from approximately 92% down to 21% as the initial MB concentration increases. This downward trend is theoretically governed by the relationship between the fixed number of active confinement sites on the adsorbent framework and the total quantity of adsorbate molecules present in the aqueous phase. Beside, the effect of the adsorbent dose on MB uptake by the FP biochar is shown that Increasing the dose from 1 to 9 g.L⁻¹ raised the removal efficiency from about 30 % to about 92 %, because more adsorption sites become available, but lowered the specific uptake from about 3.0 to about 1.0 mg.g⁻¹, since the fixed amount of dye is distributed over an increasing mass of solid. The same inverse relationship has been reported for activated carbon from the same precursor [12]. A dose of 9 g.L⁻¹ was retained for the comparative kinetic runs because it gave the clearest discrimination between the five biochars; the specific uptakes reported in Table 1 are therefore lower bounds set by this choice. The corresponding equilibrium uptakes were calculated as $q_e = (C_0 - C_e)V/m$ where C_0 and C_e (mg.L⁻¹) are the initial and equilibrium MB concentrations, V (L) is the solution volume and m (g) is the mass of biochar. The removal efficiency was calculated as:

$$R (\%) = 100 (C_0 - C_e)/C_0.$$

To identify the rate-controlling mechanism, the time-resolved data were fitted to the PFO and PSO models, whose linearized forms are given in Eqs. (1) and (2):

$$\ln(q_e - q_t) = \ln q_e - k_1 t. \quad (1)$$

$$t/q_t = 1/(k_2 q_e^2) + (1/q_e)t. \quad (2)$$

where q_e and q_t (mg.g⁻¹) are the uptakes at equilibrium and at time t (min), k_1 (min⁻¹) is the PFO rate constant and k_2 (g.mg⁻¹.min⁻¹) is the PSO rate constant. The fitted lines are shown in Fig 5a (PFO) and Fig 5b (PSO), and the resulting parameters are collected in Table 1. Taken together, the following observations show that the PSO equation provides the better empirical description of the uptake curves.

The PSO plots of t/q_t versus t (Fig 5b) are almost perfectly straight over the entire 0–120 min interval, giving correlation coefficients of $R^2 = 0.991$ – 0.999 for all five biochars. The PFO plots (Fig 5a) are appreciably more scattered, with lower R^2 values of 0.862–0.976 that deteriorate as equilibrium is approached. The fact that the PSO model fits every sample, not just one across the

whole time range is a first indication that the PSO form describes the data better than the PFO form.

Table 1: PFO and PSO kinetic parameters for MB adsorption onto the five biochars.

Biochar	$q_{e,exp}$ $mg.g^{-1}$	PFO			PSO		
		$q_{e,cal}$ $mg.g^{-1}$	k_1 min^{-1}	R^2	$q_{e,cal}$ $mg.g^{-1}$	k_2 $g.mg^{-1}.min^{-1}$	R^2
PL	0.83	0.20	0.032	0.923	0.84	0.855	0.999
PP	0.92	0.54	0.032	0.862	0.95	0.167	0.991
SH	0.68	0.37	0.045	0.947	0.88	0.242	0.997
MS	0.89	0.27	0.047	0.976	0.92	0.623	0.999
FP	1.01	0.85	0.050	0.955	1.08	0.141	0.991

A more stringent test than R^2 is the consistency between the model-derived and the measured equilibrium uptake. The PSO-calculated capacities ($q_{e,cal} = 0.84$ – $1.08 mg.g^{-1}$) agree with the experimental values ($q_{e,exp} = 0.68$ – $1.01 mg.g^{-1}$) to within 1–7 % for PL, PP, MS and the optimum FP biochar (1.08 vs $1.01 mg.g^{-1}$, a deviation of 7 %); the agreement is poorer only for SH (+29 %), the sample for which the uptake was smallest and the relative experimental scatter therefore largest. In sharp contrast, the PFO model grossly underestimates the uptake, with $q_{e,cal}$ of only 0.20 – $0.85 mg.g^{-1}$, in several cases less than one-third of the experimental value. The closer agreement obtained with the PSO model, together with the systematic underestimation of the uptake by the PFO model, indicates that the PSO equation provides the better empirical description of the uptake curves over the time range studied. It should be emphasised, however, that a good fit to the PSO equation is a description of the approach to equilibrium and is not, by itself, evidence for a particular mechanism. The PSO model is known to fit a wide range of systems irrespective of whether the interaction is chemical or physical, and the linearised t/q_t form used in Fig 5b is known to yield inflated correlation coefficients because t appears on both axes [13]. The kinetic results are therefore consistent with, but do not establish, an interaction between MB^+ and the oxygenated surface groups generated by nitric acid treatment. Determination of the point of zero charge, zeta potential measurements as a function of pH, and FT-IR and XPS analysis of the biochars before and after adsorption are required to identify the mechanism and are the subject of ongoing work.

The equilibrium adsorption behaviour of the optimum FP biochar was investigated by varying the initial MB concentration at fixed adsorbent dose, and the resulting isotherm is presented in Fig 5c. The equilibrium uptake q_e rose steeply at low equilibrium concentrations and then increased progressively more slowly, levelling off at

a plateau of $2.3 mg.g^{-1}$ at high C_e . This shape is characteristic of a favourable, monolayer-type adsorption process in which a finite number of surface sites become progressively occupied as the dye concentration increases.

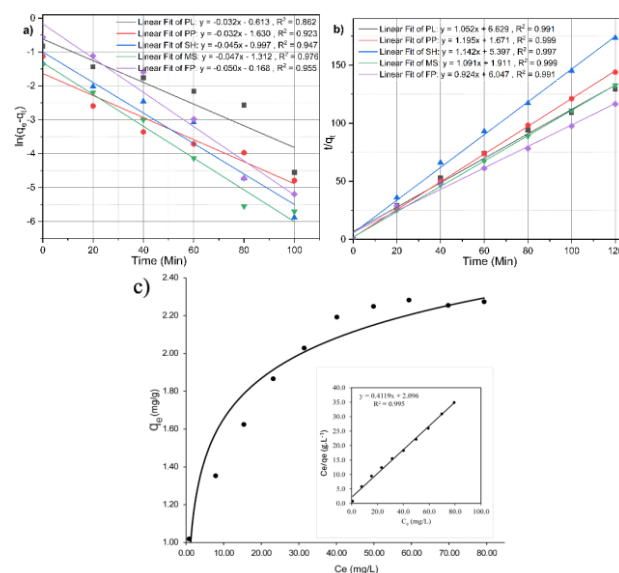


Fig 5: Kinetics of MB removal fitted to the (a) PFO; (b) PSO models; and (c) adsorption isotherm of MB onto the FP biochar; inset: linearized Langmuir plot of C_e/q_e versus C_e .

The experimental data were analysed using the Langmuir isotherm, which assumes monolayer adsorption onto an energetically homogeneous surface containing a fixed number of identical, independent active sites. Its linearized form is given in Eq. (3):

$$C_e/q_e = (1/q_m)C_e + 1/(K_L q_m) \quad (3)$$

where q_m ($mg.g^{-1}$) is the maximum monolayer adsorption capacity and K_L ($L.mg^{-1}$) is the Langmuir constant related to the affinity between the adsorbate and the surface. As shown in the inset of Fig 5c, the plot of C_e/q_e against C_e was highly linear, $y = 0.4119x + 2.096$, with a correlation coefficient of $R^2 = 0.995$. From the slope and intercept, the maximum monolayer capacity was calculated as $q_m = 2.43 mg.g^{-1}$ and the Langmuir constant as $K_L = 0.197 L.mg^{-1}$. The calculated q_m is in excellent agreement with the experimental plateau, confirming the suitability of the Langmuir model. In absolute terms, however, a monolayer capacity of $2.43 mg.g^{-1}$ is modest by comparison with the values usually reported for MB, and two factors account for this: (i) the activation route is mild. HNO_3 is an oxidant, not a porogen: it grafts carboxyl, carbonyl and phenolic functionality onto the carbon skeleton but develops very little new porosity, and the grafted oxygen groups can partially block existing micropores. The materials that

dominate the MB literature are prepared with true activating agents under much more severe conditions. (ii) Even relative to its own surface area the material is under-utilised, and we now quantify this. Taking the widely used projected area of a flat-lying MB molecule, 1.30 nm^2 [12], and $M = 319.85 \text{ g.mol}^{-1}$, a complete monolayer spread over $121.57 \text{ m}^2.\text{g}^{-1}$ would correspond to $\approx 50 \text{ mg.g}^{-1}$; the more conservative value obtained from the molecular dimensions of $1.43 \times 0.61 \times 0.40 \text{ nm}$ ($\approx 0.87 \text{ nm}^2$, giving 74 mg.g^{-1}) leads to the same conclusion. The measured 2.43 mg.g^{-1} is therefore only 3–5 % of the geometric monolayer. It should also be noted that the experiments were run at the unadjusted solution pH, without optimisation of pH, temperature or ionic strength.

The quality of the Langmuir fit indicates that, over the concentration range studied, MB uptake is well described as monolayer coverage of a finite set of surface sites with negligible lateral interaction between the adsorbed dye molecules. This equilibrium picture is fully consistent with the kinetic results: the finite set of surface sites implied by the Langmuir model is consistent with the site-limited form of the PSO rate law, although neither fit by itself demonstrates that the uptake is chemisorption-controlled.

Conclusion

In this study, five biochars were prepared from PL, PP, SH, MS and FP by nitric acid activation followed by pyrolysis at 500°C , and were compared for the removal of MB under identical conditions. The precursor governed both the porous texture and the uptake, and flamboyant pods gave the best-performing material. Uptake was well described by the PSO rate equation and by the Langmuir isotherm, although these fits describe the data and do not by themselves establish the adsorption mechanism. The monolayer capacity obtained under the mild activation conditions used here is modest compared with chemically activated carbons prepared from the same precursor at higher severity, so the material is best regarded as a starting point rather than as a finished adsorbent. Its well-developed macroporosity and abundant surface functionality make FP biochar an attractive precursor for further activation and as a support for metal nanocatalysts, which is the direction of our ongoing work.

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