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Bio-based activated carbon from husk- and wood-based biomass: comparison of carbon activation methods on organic pollutants removal

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ABSTRACT

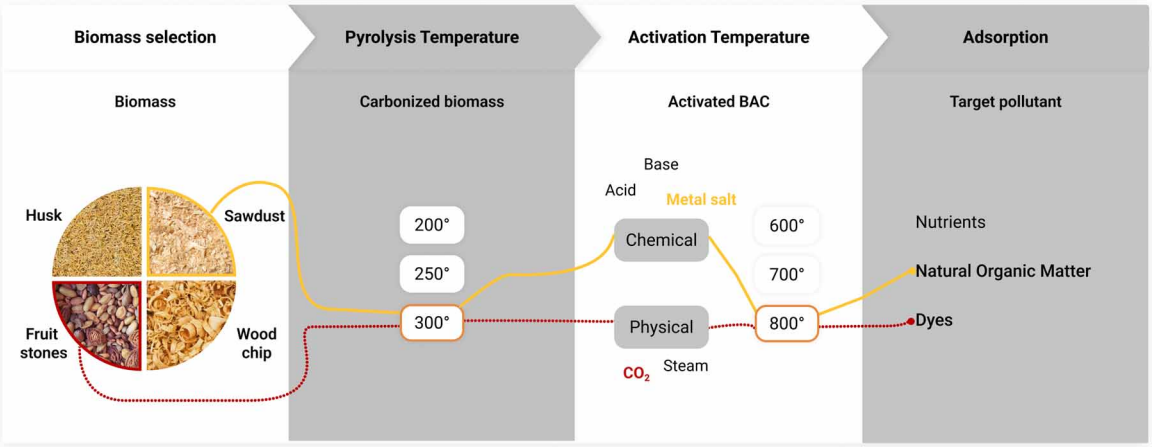
This study aimed to investigate the effect of different activations on the properties of bio-based activated carbons (BACs) for water treatment. BACs were produced via pyrolysis by the carbonization stage and were followed by four different activation procedures. Chemical activation included the introduction of metal oxides or alkali on the structure of the sawdust-derived BACs, resulting in iron-activated carbon (BAC-Fe), copper-activated carbon (BAC-Cu), and sodium-activated carbon (BAC-Na). The physical activation was conducted in a CO_2 environment with the usage of two types of locally available biomasses, resulting in husk-activated carbon (HAC) and wood-activated carbon (WAC). Depending on the activation, BACs can be developed with high porosity, active sites, and different additional functionalities such as antimicrobial and magnetic. The adsorption of natural organic matter (NOM) with chemically activated BACs yielded high removal percentages (97, 87, and 80% for BAC-Fe, BAC-Cu, and BAC-Na, respectively). The physically activated BACs demonstrated high adsorption capacities for dye – 278 mg/g for WAC and 213 mg/g for HAC. This outlines a wide range of BAC production possibilities with advanced functionalities.

Key words: activation, adsorption, bio-based activated carbon, dye, natural organic matter, water treatment

HIGHLIGHTS

- Various production parameters and methods allow the modification of final properties of bio-based activated carbons.
- Physical activation provides sufficient surface area for pollutants such as dye; it is cheap and environmental friendly.
- Chemical activation allows the implementation of functional groups on carbon surfaces to improve adsorbent selectivity and reach specific properties.

GRAPHICAL ABSTRACT



INTRODUCTION

When problematic pollutants, such as nutrients, natural organic matter (NOM), or dyes appear in surface water, it requires additional methods of treatment to decrease their harmful impacts on human health and the environment (Rasheed *et al.* 2019). NOM has a direct influence on the water treatment process quality by contributing to membrane fouling and competing with the removal of other pollutants (Matilainen *et al.* 2010). An additional step, such as adsorption, is often performed as a tertiary treatment for the remediation of such pollution. The adsorption process is widely used for more sufficient drinking water purification. Conventional adsorption is usually performed with activated carbon (AC). However, AC cannot be considered an environmentally friendly material. Conventional AC is usually made of coal, whose extraction and transportation to the end-users have a significant carbon footprint (Kim *et al.* 2019). Thus, bio-based activated carbon (BAC), also called biochars, which can be produced from locally available biomass, is increasingly getting popular as a substitute for conventional AC (Wu & Wu 2019).

Besides local availability and the renewable origin of their raw material, BACs have become popular as adsorbents thanks to their adsorptive surface functionality and high porosity. An underexplored advantage of BACs is, however, the adjustability of their properties during production. The production conditions, activation method, or biomass type affect the final properties of BACs. Thus, by controlling the pyrolysis temperature and activation method, it is possible to create specific carbons for improved removal of target pollutants. We call this process tailoring (Yazdani *et al.* 2019; Tomin *et al.* 2021; Tomin & Yazdani 2022). BAC can be manufactured from numerous types of biomasses, such as algae, rice husk, sludge, etc. For example, recent studies display the effective removal of antibiotics (Fan *et al.* 2020) or heavy metals (Deng *et al.* 2020) by BACs, that are produced from local biomass. Nevertheless, the most prevalent biomass sources are forestry and agricultural by-products (Tripathi *et al.* 2016). The production method also can vary. BACs may result from the thermal treatment of raw material in an inert gas atmosphere (pyrolysis), analogous to AC, or hydrothermal carbonization. The most frequent methods for BACs production are fast, slow, and microwave pyrolysis (Sakhiya *et al.* 2020). The temperature of the production method is one of the key factors, which determines BAC's properties. The pyrolysis can be performed at low (200–300 °C) or high temperatures (600–800 °C) and the quality of the produced carbon will be different (Kambo & Dutta 2015). Following carbonization, the production process may include an activation stage, which increases the surface area significantly (Leng *et al.* 2021). Activation can be either chemical or physical.

During chemical activation, BAC reacts with the chemical activating agent, which can be acid, alkali, or salt, under heat treatment. It improves the BAC's properties by modifying its surface and introducing functional groups in its structure. The developed functional groups facilitate specific interactions on the adsorbent surface, e.g. π–π interactions, polar/electrostatic interactions, etc. Such interactions improve the adsorption efficiency of pollutants from water (Gwenzi *et al.* 2017). Chemical activation has its advantages over a physical one, such as lower temperature during production, greater carbon yield, additional activator-related characteristics, qualitative and quantitative micro-porosity, etc. Nevertheless, for big-scale production, it might be economically or environmentally unfeasible. Many chemical activators are costly or harmful to the environment and human health. Thus, if the produced carbons are not stable, or not rinsed carefully, the leakage of the chemical activator from the carbon might cause secondary pollution of water. Physical activation increases the porosity of the carbon due to high temperature in an oxidative environment. The prevalent activation agents used for a such method are CO_2 or steam. Compared to the chemical one, physical activation requires less time for production and does not implicate chemicals, which makes it cheaper and poses fewer environmental risks (Sakhiya *et al.* 2020). To the best of our knowledge, a comparative study of different activation methods for carbon synthesis from forestry and agricultural waste that is used for organic pollutant removal is still missing from the literature.

Previously we reported highly porous biochars from two different biomass – pinecone (Yazdani *et al.* 2019) and softwood (Tomin *et al.* 2021) via chemical activation. The research strengthened our hypothesis that the production conditions play a key role in the development of porous structure and affinity of biochars for specific pollutants, e.g. NOM or phosphate. This means that the right production procedure enables an enhanced removal of the target pollutant by the BAC from water. The illustration of this hypothesis is given in Figure 1. As a possible example scenario, if we follow the yellow production line in Figure 1, take sawdust as a raw biomass, pyrolyze it at 300 °C during the carbonization stage and apply chemical activation at 800 °C with metal salt as an activator, at the end we would have an efficient, selective carbonous adsorbent for NOM. In case we follow the red line

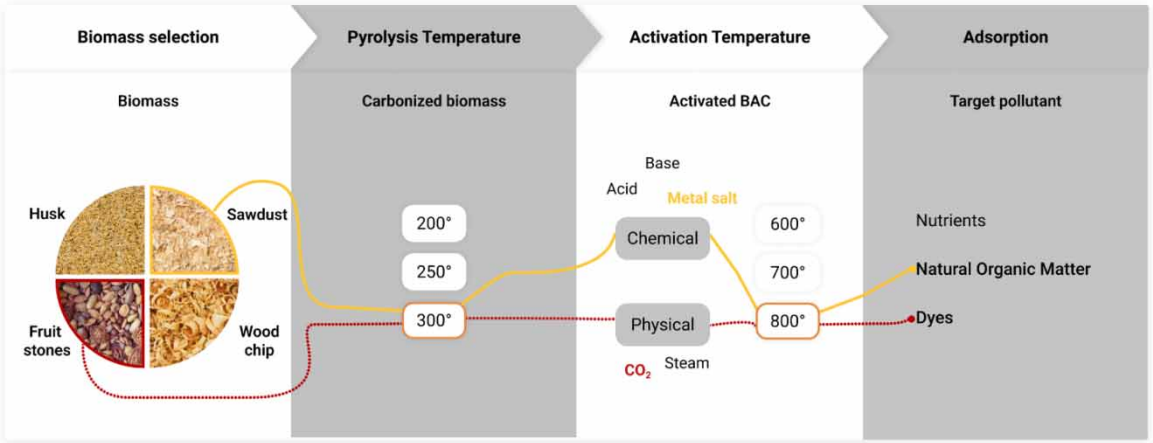


Figure 1 | The illustration of the biochar tailoring hypothesis, the yellow and red lines represent possible scenarios of production parameters selection and outcomes. Please refer to the online version of this paper to see this figure in colour: http://dx.doi.org/10.2166/wpt.2023.019.

(in Figure 1), the parameters of production differ, and the final product would be more efficient for dyes. Many studies report about biochar production and efficiency for specific pollutants. However, the systematic understanding of the effects of biomass type and activation conditions on resulting adsorptive properties still calls for further research. The deeper understanding on how final properties of BAC differ depending on chosen activation method is on big importance. This enables a more deliberated choice on selecting an appropriate activation method based on the specific need. Additionally, it might help to save money and decrease environmental risks during production. Despite that, the comparative studies of biochars, produced with physical and chemical activation are still missing from the literature.

In this study, we aim to show how BAC's properties can be adjusted by different activation methods and how the quality of the biochar may differ by changing one of the production conditions. Three different chemically activated BACs, including metal salt and sodium hydroxide activation, were produced and tested for NOM removal. Two physically activated carbons were produced from different local raw biomass sources and tested for dye removal. Thereby we intend to gain deeper knowledge on the process of tailoring via (1) producing BACs from locally available forest-based (sawdust) and waste-based (husk) biomass; (2) performing chemical modification and comparing metal-based activators to alkali-based activator; (3) exploring the performance of the activated BACs for the removal of NOM or textile dye.

MATERIALS AND METHODS

BAC production

Three types of BACs were produced via two-step pyrolysis with chemical activation according to a process, which is fully described and reported in our previous research (Tomin *et al.* 2021; Tomin & Yazdani 2022). The spruce was used as common and accessible wood biomass. The biomass sawdust was first carbonized at low-temperature pyrolysis (300 °C) for an hour under a nitrogen atmosphere in Naber N60/HR furnace. The carbonized sawdust was then modified with different chemicals: $FeCl_3{\cdot}6H_2O$ (Merck), $CuCl_2{\cdot}2H_2O$ (Sigma Aldrich), or NaOH (Supelco). The modification was performed by mixing the chemical with carbon with a 1/2 ratio of carbon/activator for 2 h at room temperature. Finally, it was re-pyrolyzed in the same furnace under a nitrogen atmosphere at high-temperature pyrolysis (800 °C). To ensure the removal of excessive chemicals from BAC, after production, all products were rinsed with 0.1 M HCl followed by reverse osmosis water until a neutral pH was obtained. After rinsing, the carbons were dried at 105 °C overnight. The products were named iron-activated carbon (BAC-Fe), copper-activated carbon (BAC-Cu), and sodium-activated carbon (BAC-Na).

Two types of BACs were produced via the first step of carbonization followed by physical activation using a tube furnace (NBD-O1200-50IC). The biomasses were wood sawdust and food waste – husk consisting of peach sones, cherry stones, and walnut shell blend. Firstly, the carbonization of biomass was carried out at 300 °C for 1 h under a nitrogen atmosphere. Then, physical activation was performed by heating the carbons

to 800 °C and pyrolyzing them for 2 h in a CO_2 environment. Obtained BACs were named according to the used biomass, as husk-activated carbon (HAC) and wood-activated carbon (WAC).

BAC characterization

The scanning electron microscopy was performed on the Zeiss Sigma VP analytical field emission scanning electron microscope, using a 10-μA probe current and 10 kV of acceleration voltage, to explore the surface morphology of the samples. To prepare the sample, BAC was fixed on a metal stub with a carbon tape. The coating was not performed.

Before Brunauer, Emmett, and Teller (BET) specific surface area/porosity measurement was performed, samples were dried overnight and then put in a Micromeritics FlowPrep 060 sample preparation system for degasification under 200 °C for 2 h with flowing N_2 gas. The BET measurement was done in the Micromeritics TriStar II 3020 automated gas adsorption analyzer.

Batch experiments and analytical measurement

The adsorption batch experiments were conducted to study the target pollutant removal by the BACs. The experiments were conducted on a shaker at 160–180 rpm and at room temperature for 3 h with no pH adjustment. Then, the solutions were filtered through 0.45-μm syringe filters for further measurement. All batch experiments were conducted in two replicates. To determine the concentration of NOM and dye, the UV absorbance measurement was performed, using a UV-VIS spectrophotometer (Shimadzu UV-1201), for NOM at 254 nm wavelength and for the dye at 507 nm wavelength, respectively. The absorbance was converted to concentration using the calibration curves.

The concentration of NOM was measured and represented with a collective parameter COD, as originally NOM is a complex network of different molecules, the exact composition of which cannot be specified (Worch 2012). The NOM removal experiments were conducted on the lake water, collected from Lake Päijänne in Asikkala, Finland. The collected lake water had a very low concentration of NOM (CODMn = 6.5 mg/l). Thus, to model the pollution, the solution for the NOM adsorption experiment was prepared by adding a certain amount of humic acid sodium salt powder (Alfa Aeser) to the lake water. Different doses of chemically activated BAC within the 0.1–2 g/l range were used for the NOM adsorption in a 50 ml volume of the model solution.

For the dye removal experiment, reverse osmosis water (RO water) was used as in this case the decolorization can be observed more clearly. The stock solution of Direct Red 23 dye (Sigma Aldrich) with 1,000 mg/l concentration was prepared by weighing an accurate amount of dye powder and dissolving it in RO water. The removal efficiency was tested with two physically activated BACs in the dye solutions in the 20–150 mg/l concentration range.

As previous research showed the antibacterial performance of copper-consistent materials (Fanta *et al.* 2019), chemically activated BACs were tested for such effect. The test was executed on *Escherichia coli* bacteria by a pour plate method in compliance with SFS-EN ISO 6222 and SFS 3016 standards. The m-Endo Agar LES (Sigma–Aldrich) was prepared and poured into the plates. The model wastewater samples were collected from the wastewater treatment plant and diluted in 1:1,000 dilutions in a sterile saline solution. Diluted solutions were filtered and filters were put in agar plates. Plates were put in the oven at 37 °C for 24 h and after this, the grown colonies were counted. The experiment was done in three repetitions.

RESULTS AND DISCUSSION

NOM removal with chemically activated BAC

Figure 2(a) displays the adsorption of NOM with different doses (g/l) of produced BACs. As seen, all three BACs showed good NOM adsorption abilities. BAC-Cu reaches its best performance (87%) already at 1 g/l, while BAC-Fe and BAC-Na show the best results (97 and 80% respectively) at 2 g/l. In the absence of competing ions, the BAC-Fe shows a considerable removal of NOM (97%). Nevertheless, BAC-Fe can show selective adsorption for phosphorus, which often appears in water with NOM (Omoike & vanLoon 1999). Therefore, its performance may decrease significantly in a binary solution of NOM and phosphorus (Tomin *et al.* 2021). At the same time, BAC-Cu requires less material for sufficient adsorption performance, even though it has slightly less adsorption ability than BAC-Fe at 2 g/l. This is an indication of monolayer adsorption which is a typical characteristic of chemically modified adsorbents (Tomin & Yazdani 2022). BAC-Na showed good results (84% removal with 3 g/l BAC), which is promising as it provides a better feasibility prospect for big-scale production. The porous

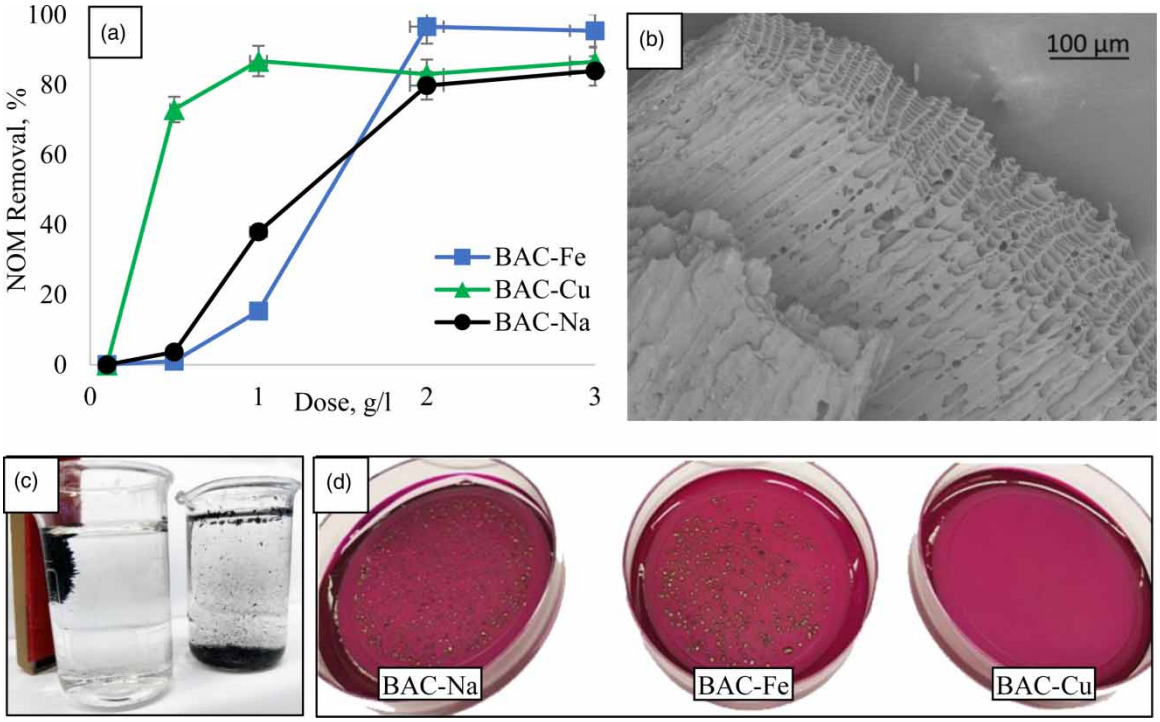


Figure 2 | (a) The adsorption of NOM (initial COD = 16 mg/l); (b) SEM image of BAC-Na; (c) the magnetic properties of BAC-Fe; (d) the *Escherichia coli* inactivation study on BAC-Na, BAC-Fe, and BAC-Cu.

structure of BAC-Na, which can be found in Figure 2(b), reveals honeycomb-like pore patterns. This differs from the surface of metal-activated BACs (Tomin *et al.* 2021) which were less porous but contained factionalized metal oxide active sites.

Table 1 compiles the BET surface area, pore volume, and pore size for produced BACs. BAC-Na showed the highest surface area as 519 m^2/g, while BAC-Fe and BAC-Cu indicated 101 and 98 m^2/g surface area values, respectively. In a recent study, the eucalyptus-derived alkali-activated biochar also showed a high surface area of 459 m^2/g (Phinyothanmakorn *et al.* 2022). The decreased surface area of metal-activated BACs indicates that pores were filled up with metal oxide crystals (Tomin *et al.* 2021). However, as we can see from NOM removal results, surface area might not play a key role in the adsorption of the target pollutants. The effective adsorption of NOM by metal-activated BACs shows that impregnated active functional sites in the chemically activated BAC favor adsorption even better than a high surface area. Surprisingly, the mean pore size of BAC-Fe appeared considerably higher than for other BACs.

As mentioned earlier, chemical activation can result in additional characteristics. For instance. BAC-Fe has magnetic properties (Figure 2(c)), which facilitates the removal of carbon powder from water (Tomin & Yazdani 2022). The antibacterial test showed a significant antimicrobial effect on *E. coli* bacteria for BAC-Cu (Figure 2(d)), while other BACs had no effect. This suggests that the main reason for the bacteria's deactivation is the type of chemical used in the activation. Presumably, copper sites of the BAC-Cu were able to interact with negatively

Table 1 | BET surface area, pore size, and pore volume parameters for the obtained BACs

Sample	Surface area, m^2/g	Pore volume, cm^3/g	Pore size, nm
BAC-Fe	101.3	0.28	12.2
BAC-Cu	98.4	0.11	4.3
BAC-Na	519.3	0.29	2.3
WAC	568.6	0.35	2.6
HAC	415.1	0.27	2.5

charged compounds on the bacterial cell wall, consequently destabilizing, damaging the cell wall, and inhibiting bacterial growth (Guan *et al.* 2021).

Dye removal with physically activated BAC

The removal of different dye concentrations with WAC, HAC, wooden biomass (WB), and husk biomass (HB) shows that the raw biomass materials are incapable of adsorption, as they show no adsorption capacity (Figure 3(a)). The physically activated BACs display a high 283 and 211 mg/g adsorption capacities for WAC and HAC, respectively. These results comply with the obtained high surface area of BACs: 568 and 415 m^2/g for WAC and HAC, respectively. For such pollutants as dyes, the high surface area is the main factor of adsorption, which can be obtained via physical activation. The decolorization of dye with WAC is shown in Figure 3(c). HAC shows great performance, slightly lower than WAC, demonstrating that food waste such as fruit stones can be reused for BAC production and thus contribute to a circular economy. HAC has higher adsorption capacity and surface area values in comparison to biochars produced with different activation methods and used for dye removal (Table 2).

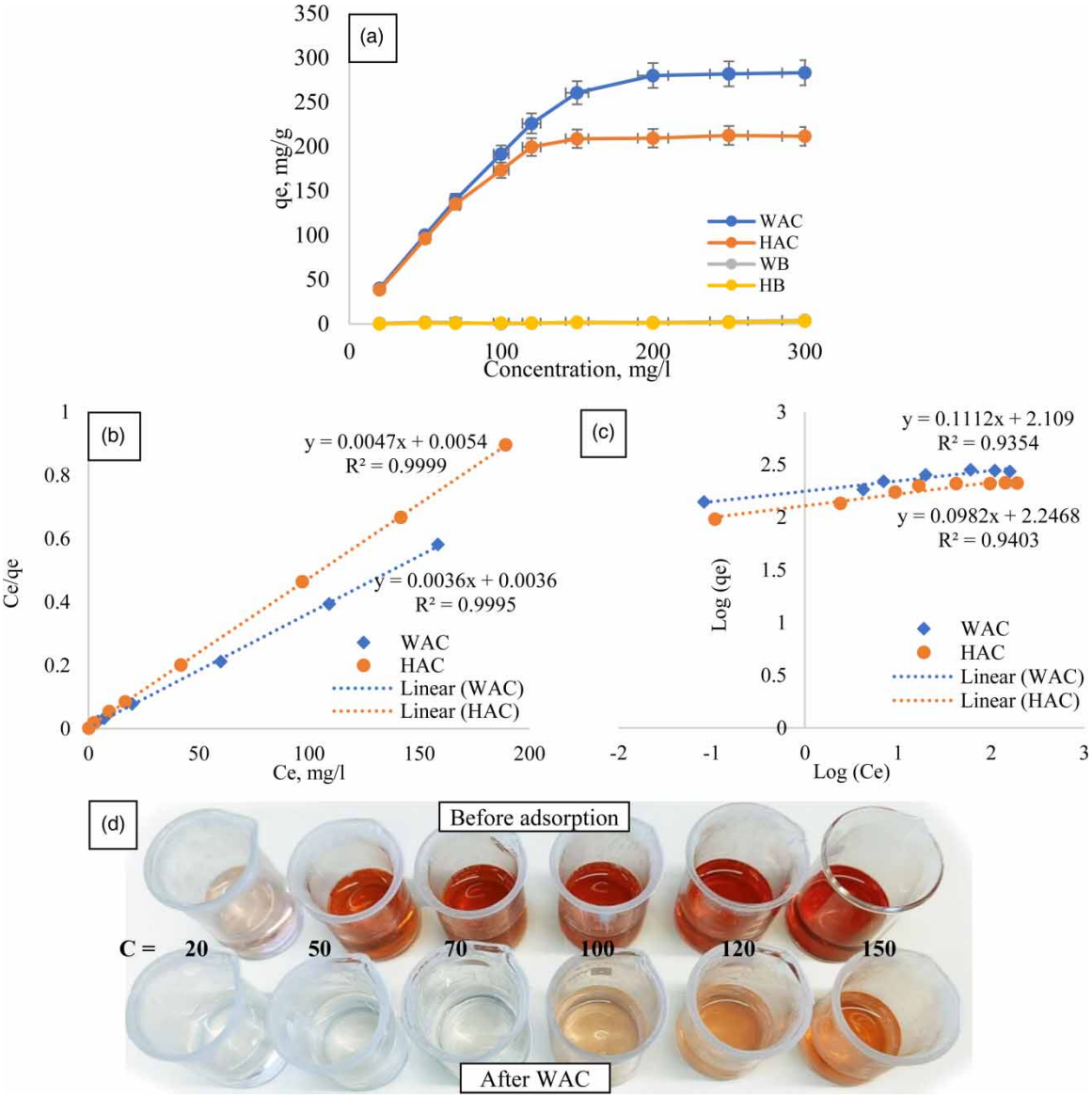


Figure 3 | (a) The adsorption capacity of BACs and biomass (dosage 2 g/l, contact time 3 h, no pH adjustment, room temperature); (b) linear Langmuir isotherm; (c) linear Freundlich isotherm; and (d) imaging of the dye removal process.

Table 2 | The comparison of different adsorbents based on activation methods and final properties

Biomass	Activation method	Surface area (m^2/g)	Pollutant	Removal (%)	q_L (mg/g)	Reference
Sawdust	Chemical, NaOH	519.3	NOM	84	–	Present work
Eucalyptus	Chemical, KOH	459	NOM	73.4	–	Phinyothanmakorn *et al.* (2022)
Sawdust	Chemical, $FeCl_3$	98.9	NOM	96	–	Tomin *et al.* (2021)
Fruit stones	Physical, CO_2	415.1	Direct red dye	–	212.5	Present work
Lychee seeds	Chemical, KOH	154	Methylene blue dye	–	124.5	Li *et al.* (2019)
Switchgrass	Only pyrolysis	255	Congo Red dye	–	22.6	Park *et al.* (2019)

The Langmuir (Equation (1)) and Freundlich (Equation (2)) models were employed to understand the nature of dye adsorption on the physically activated BACs.

$$\frac{C_e}{q_e} = \frac{C_e}{q_L} + \frac{1}{(q_L K_L)} \tag{1}$$

where q_L and K_L are the Langmuir monolayer capacity and the equilibrium constant, respectively.

$$logq_e = logK_F + \frac{1}{nlogC_e} \tag{2}$$

where K_F is the Freundlich thermodynamic constant ((mg/g)·(l/mg)$^{1/n}$) and $1/n$ is the Freundlich intensity factor.

The model was studied under nine dye concentrations at room temperature and neutral pH (6.5). The results are plotted in Figures 3(b) and 3(c), and the calculated values are presented in Table 3. As seen from R^2 values the adsorption onto BACs followed the Langmuir model better for both adsorbents. According to the Langmuir model, a monolayer coverage of the dye occurs on the solid surface with homogeneous adsorptive groups. The considerably high monolayer capacity (q_L) of the BACs corresponds well with the adsorption capacity values in Figure 3(a). The monolayer coverage (278 and 213 mg/g) is considerably higher than the values reported in the literature for other dyes (124.5 mg/g) (Li *et al.* 2019). A Langmuir monolayer coverage of phosphate was also reported for wood-derived magnetic carbon (Tomin & Yazdani 2022).

Table 3 | Langmuir and Freundlich model parameters of BACs

	Langmuir			Freundlich		
	q_L, mg/g	K_L	R^2	K_F	1/*n*	R^2
WAC	277.77	1	0.99	8.24	0.11	0.93
HAC	212.76	0.87	0.99	9.46	0.098	0.94

CONCLUSIONS

In this paper, different physical and chemical activation methods were used to produce BACs. The adsorption affinity of chemically activated BACs activated with iron oxide (BAC-Fe), copper oxide (BAC-Cu), and sodium hydroxide (BAC-Na) was tested on NOM adsorption, resulting in a high removal of NOM with metal-activated BACs (87% for BAC-Cu and 97% for BAC-Fe) and slightly less effective BAC-Na (80%). BAC-Cu also showed a noticeable antibacterial effect on *E. coli* bacteria, while BAC-Fe was magnetic, and BAC-Na was highly porous. The physically activated BACs were produced from locally available waste biomasses: sawdust (forestry waste) and fruit stones with walnut shells (food sector waste). The physically activated BACs demonstrated high adsorption capacities for dye – 278 mg/l for WAC and 213 mg/l for HAC, following Langmuir isotherm ($R^2 = 0.99$). This points toward a wide range of BAC production methods to create carbons from renewable sources and with desired properties.

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DATA AVAILABILITY STATEMENT

All relevant data are included in the paper or its Supplementary Information.

CONFLICT OF INTEREST

The authors declare there is no conflict.

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