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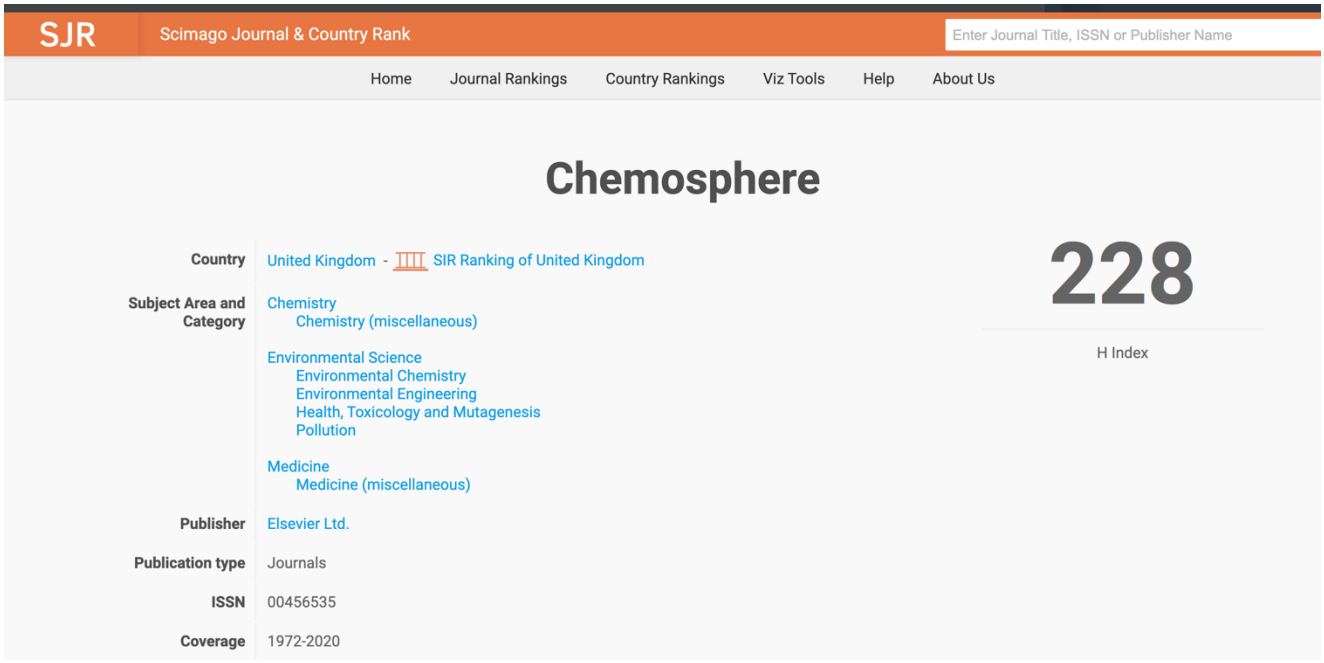

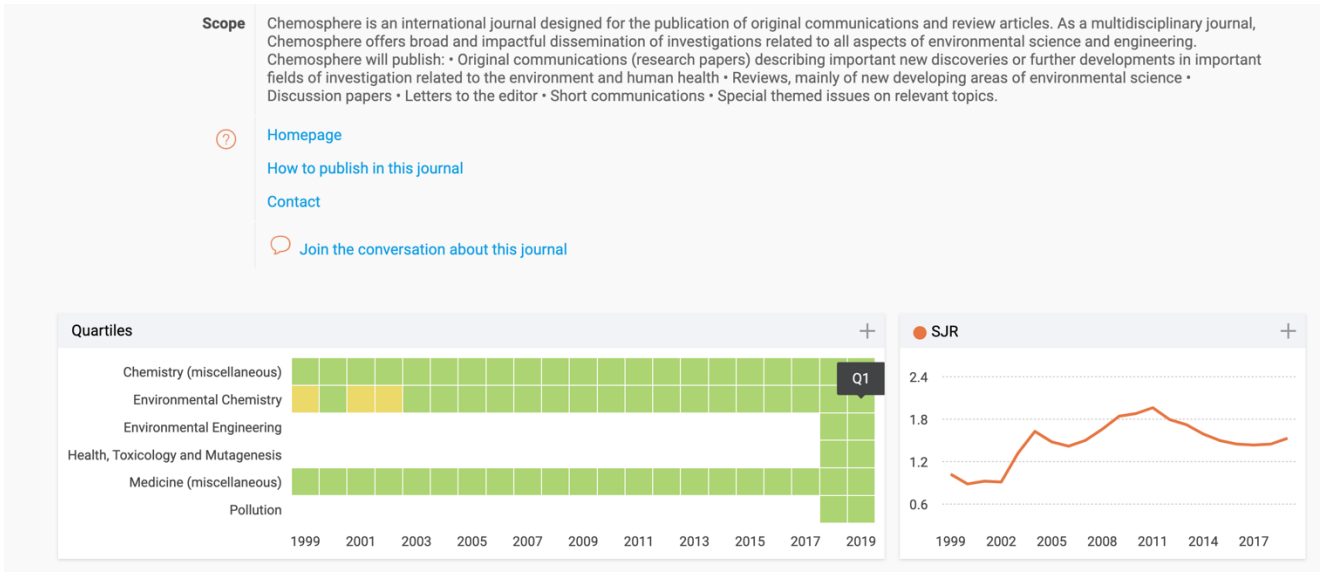

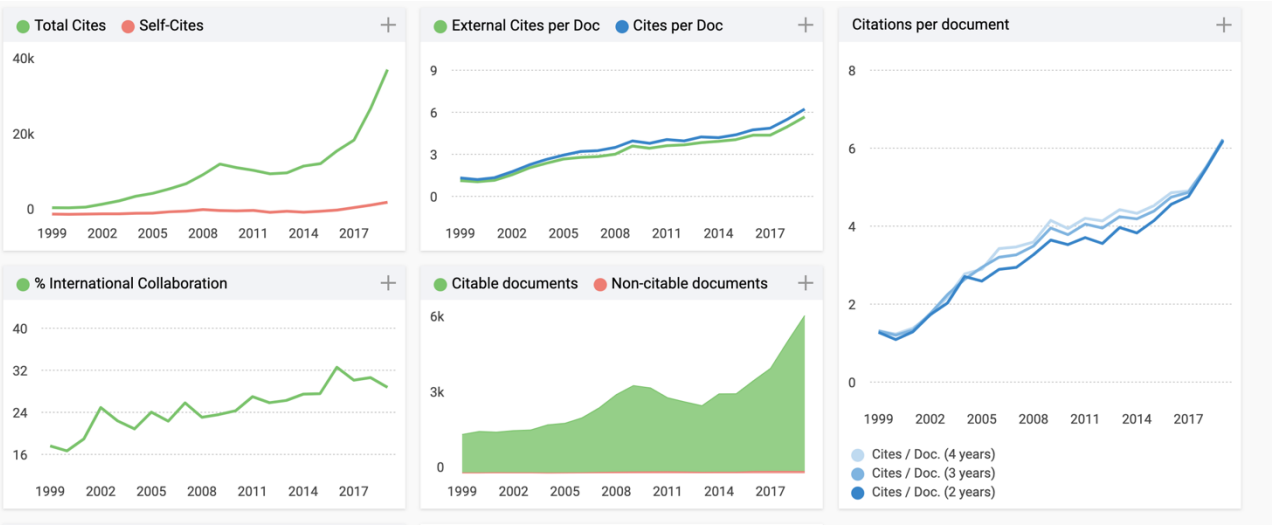

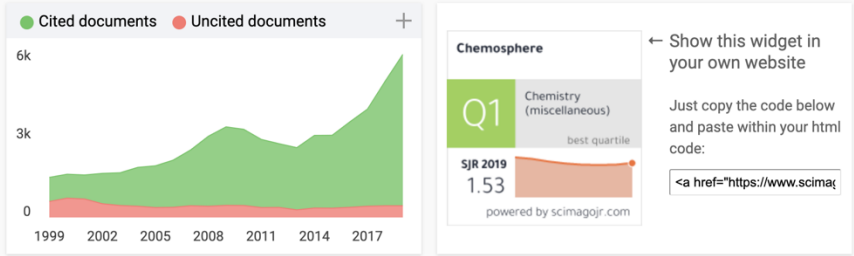

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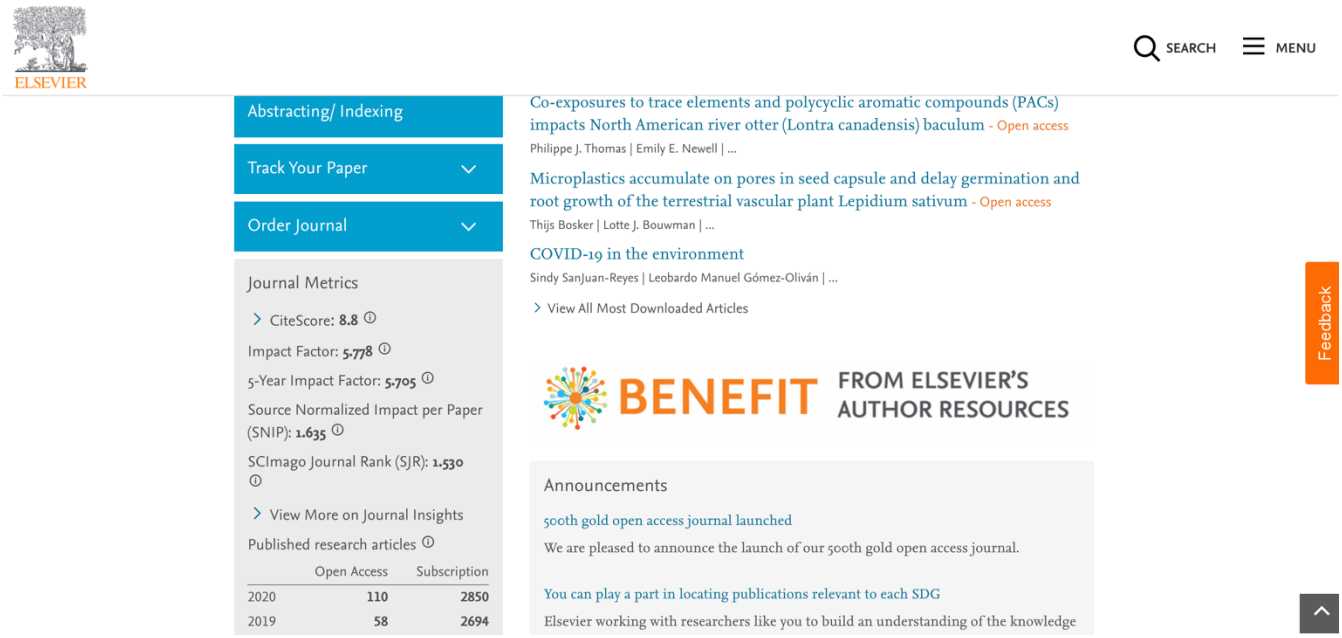

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Preparation of biochar from biomass waste soybean dreg using a one-pot method for methylene blue adsorption from water

Abstract:
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Article Type: Research paper

Section/Category: Treatment and Remediation

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*Title Page

Preparation of biochar from biomass waste soybean dreg using a one-pot method for methylene blue adsorption from water

Zhiwei Ying[a], He Li[a,*], Xinqi Liu[a,*], Chi Zhang[a], Jian Zhang[a], Guofu Yi[a]

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Highlights

A novel biochar was prepared from soybean dreg with $KHCO_3$ using a one-pot method.

The prepared adsorbent has a high specific surface area and large pore volume.

The maximum adsorption capacity for methylene blue at 318 K reached 1273.51 mg g^{-1}.

The adsorption data were well fitted with the pseudo-second-order kinetic model.

The adsorption behavior was suitable for the Langmuir isotherm.

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Abstract: Soybean dreg is a by-product of bean products production, with a large consumption in China. Low utilization value leads to random discarding, which is one of the important sources of urban pollution. In this work, porous biochar was synthesized using a one-pot method and potassium bicarbonate ($KHCO_3$) with low-cost soybean dreg (SD) powder as the carbon precursor to investigating the adsorption of methylene blue (MB). The prepared samples were characterized with scanning electron microscopy (SEM), transmission electron microscopy (TEM), elemental analysis (EA), Brunner-Emmet-Teller (BET), X-ray diffraction (XRD), Raman spectroscopy (Raman), Fourier transform infrared spectroscopy (FTIR), and X-ray photoelectron spectroscopy (XPS). The obtained SDB-K-3 showed a high specific surface area of 1620 m^2 g^{-1}, a large pore volume of 0.7509 cm^3 g^{-1}, and an average pore diameter of 1.859 nm. The results indicated that the maximum adsorption capacity of SDB-K-3 to MB could reach 1273.51 mg g^{-1} at 318 K. The kinetic data were most consistent with the pseudo-second-order model and the adsorption behavior was more suitable for the Langmuir isotherm equation. All standard Gibbs free energy change (ΔG) values were negative, while the standard enthalpy change (ΔH) values and the standard entropy change (ΔS) values of SDB-K-3 were positive, indicating that the adsorption was a feasible, spontaneous, and endothermic process in nature. This study demonstrated that the porous biochar adsorbent can be prepared from soybean dreg by high value utilization and it could hold significant potential for dye wastewater treatment in the future.

Keywords: soybean dreg, porous biochar, methylene blue, adsorption

1. Introduction

The increasing demand for dyes in different industries exacerbates the discharge of dye wastewater (Corso et al., 2012), as well as their toxic effect on the environment and organisms, inducing significant concern from society as a whole (Nethaji et al., 2010). For example, the discharge of dye wastewater into forests or fields will directly damage the soil productivity, and the presence of dyes in water can affect light penetration and photosynthesis of aquatic plants (Vilar et al., 2007; Tang et al., 2018). Some organic dyes and their products display mutagenicity or carcinogenicity toward humans (Liu et al., 2012), which may cause different degrees of damage to some organs, including kidney, brain, reproductive system and liver, etc (Kadirvelu et al., 2003; Dincer et al., 2007; Shen et al., 2009). In the dye industry, the cationic dye MB is an aromatic heterocyclic compound that can burn the eyes of people and animals, and may stimulate the gastrointestinal tract (Senthilkumaar et al., 2005), causing symptoms such as dyspnea, vomiting, diarrhea, insanity, and methemoglobin after ingestion (Tan et al., 2008a; Tan et al., 2008b). Therefore, the effective removal of dye from wastewater and prevent it from polluting the environment is a significant challenge for industrial production that requires an urgent solution.

In the early stage of dye wastewater treatment, only simple balancing and precipitation treatment methods were adopted. Currently, conventional dye removal methods that include biological, chemical, and physical approaches are used for the treatment of dye wastewater (Katheresan et al., 2018), such as biodegradation (Banerjee

and Chattopadhyaya, 2017), membrane separation (Ciardelli et al., 2000), coagulation and flocculation (Thongchai and Somkid, 1986), photocatalytic degradation (Dong et al., 2010), and adsorption (Wang et al., 2011). Among the above removal methods, adsorption has become one of the preferred technologies for the treatment of dye wastewater because of its low cost, good adsorption and convenient operation (Liu et al., 2012). In the current study, a large number of biomass waste products, such as pineapple crown leaves (Astuti et al., 2019), wood chips (Khattri and Singh, 2009), bamboo (Li et al., 2020), walnut shells (Wang and Liu, 2017), bagasse (Juan et al., 2002) and orange peel (Arami et al., 2005) are used as raw ingredients in preparing carbon materials that can absorb dye wastewater.

Of all types of biomass waste products, most of them have little or no significant economic value, and the follow-up treatment is also a big problem, especially for the waste with large output such as soybean dregs. China has 5,000 years of soybean cultivation history, which is a major oil-bearing crop, as well as one of the important food crops and essential in the human diet (Zhu et al., 2011). In recent years, China produced more than 80,000 tons of SD waste annually. SD is a by-product of the production of soybean products, and since only a small portion of the residue is directed at poultry and livestock feed, great majority of it is wasted or carelessly discarded in the fields (Zhu et al., 2012). Processing the considerable amounts of SD produced every year has become a significant challenge in the soybean industry. Furthermore, SD contains about 50 dietary fiber, 25 protein, and 10 lipids and other nutrients (Li et al., 2012), which can satisfy the standards of high-value utilization and environmental

protection of biomass waste. Moreover, the abundance, low cost, and high fiber content of SD make it an excellent raw ingredient source for the carbon materials.

Currently, the conventional methods of biochar preparation are mainly pyrrolysis and hydrogen carbonization (Kumar et al., 2017). The two methods mainly have two steps: (1) precarbonization of biomass waste; (2) activation of pre-carbonized samples (physical or chemical activation). Comparatively, chemical activation is chosen by more researchers to treat samples, because satisfactory pore structure materials can be obtained, but at the same time, more energy and wastewater are consumed in the production process. The most common activators in chemical activation are KOH, $ZnCl_2$, H_3PO_4, K_2CO_3 and $KHCO_3$ (Ioannidou and Zabaniotou, 2007; Deng et al., 2015). Considering the cost of production and environmental sustainability, the one-pot synthesis is an ideal choice.

In this study, porous biochar is prepared using a one-pot method with SD as the carbon precursor and $KHCO_3$ as an activator for the adsorption of MB. The structure and composition of the material were characterized by SEM, TEM, EA, BET, XRD, Raman, FTIR, and XPS. The adsorption mechanism is discussed via the study of kinetic and thermodynamic models, and the analytical rate of the material is evaluated.

2. Materials and methods

2.1. Materials and reagents

SD was obtained from the Shandong Yuxin Biotechnology Co., Ltd., Shandong Province. The SD was first dried in a vacuum oven (GZX-9030MBE, Shanghai Boxun Industrial Co., Ltd. Medical Equipment Factor) at 105℃ for 48 h to obtain the starting

material. All chemicals, including $KHCO_3$, MB, and hydrochloric acid (HCl, 37 wt%) were of analytical grade, purchased from the Sinopharm Chemical Reagent Co., Ltd. and used as received.

2.2. Preparation of SDB-K-X

The SD was mixed with $KHCO_3$ (SD/$KHCO_3$ weight ratio=1:2, 1:3, 1:4, and 1:5) and ground evenly, after which it was activated at 800°C at a heating rate of 10 °C min^{-1} in a N_2 (99.999) atmosphere for 90 min. After the temperature had cooled to room temperature, the subsequent material was placed in a 5 wt% HCl solution and stirred continuously for 24 h, after which it was filtered and separated. The residue was washed with DI water until a neutral pH was reached, and dried at 105°C for 24 h. The activated material that was obtained was labed as SDB-K-X X=2, 3, 4, 5 referring to the SD/$KHCO_3$ weight ratio=1:2, 1:3, 1:4, and 1:5, respectively and finally preserved in a desiccator until further use.

2.3. Characterization of SDB-K-X

The morphology of SD and SDB-K-X was performed using SEM (JEOL JSM-6700F, Japan), while the hole wall microstructure was performed with TEM (JEOL JEM-2100F, Japan) operated at 200 kV. The EA (C, H, O, N, and S) of the samples were acquired with an elemental analyzer (Elementar Vario EL III, Germany). The nitrogen sorption isotherms of the samples were carried out at 77 K using a nitrogen adsorption apparatus (BET, Quantachrome AUTOSORB IQ, USA). The crystallinity of the samples was acquired using XRD (Rigaku Ultima IV, Japan), while the Raman spectra were obtained using Raman (Bruker Optics SENTERRA, Germany, excitation-beam

wavelength: 532 nm). The organic structure of SDB-K-X was characterized by FTIR (SHIMADZU Type 2000, Japan) recorded in a wavenumber range of 4000-400 cm^{-1}. The surface element composition and chemical state of the samples were obtained with spectra recorded using XPS (Thermo Scientific Escalab 250Xi, USA).

2.4. Adsorption experiments

All batches were tested in a Water-bathing Constant Temperature Vibrator (WE-3, Tianjin Ounuo Instrument Co., LTD, China). Then, 0.025 g adsorbent was added to a 25 mL MB aqueous solution of 1000 mg L^{-1}, 1500 mg L^{-1}, and 2000 mg L^{-1}, and oscillated at 150 rpm for 2 h at 298 K. Furthermore, to investigate the MB adsorption kinetics, 0.025 g of SDB-K-3 was added to 25 mL MB aqueous solution at concentrations of 1000 mg L^{-1}, 1500 mg L^{-1}, and 2000 mg L^{-1}, respectively, at a temperature of 298 K and a speed of 150 rpm. At a preset time point (from 0 min to 600 min), a small amount of the sample was removed with a pipette gun for determination. The MB of the isothermal adsorption experiments at three different temperatures (298 K, 308 K, and 318 K), and 0.025 g SDB-K-3 were added into 25 mL of 250, 500, 750, 1000, 1250, 1500, 1750, and 2000 mg L^{-1} MB, respectively. The adsorption was performed for 2 h at 150 rpm, after which the suspension was centrifuged at 4000 rpm for 10 min. Then, the supernatant was filtered through a 0.22 μm filter to measure its adsorption value (UV-vis, Agilent Cary-60, USA) at the maximum value of 664 nm for MB. The adsorption capacity Q (mg g^{-1}) and removal efficiency R (%) of MB on the adsorbent were separately calculated with the following formulation:

$$Q(mg/g) = \frac{(C_0\text{-}C)V}{m} \quad (1)$$

$$R(\%) = \frac{C_0 - C}{C_0} \times 100 \quad (2)$$

where Q (mg g^{-1}) is the amount of MB adsorbed on the adsorbent, C_0 (mg L^{-1}) and C (mg L^{-1}) are the initial concentration and the equilibrium concentration of MB, respectively, V (L) is the volume of the MB solution, m (g) is the mass of the adsorbent used, and R (%) is the removal efficiency of the dye.

3. Results and discussion

3.1. Characterization

3.1.1. SEM and TEM

SEM and TEM characterized the porous structure. According to the SEM image (Fig. 1a, 1b), the surface of the SD appeared as an irregular fold layered structure, which was relatively smooth and dense with no apparent pores (Hu et al., 2018). However, after high-temperature activation with $KHCO_3$, an abundance of pore structures was evident on the surface of the SDB-K-X, showing a 3D framework with randomly opened pores, indicating that $KHCO_3$ played a positive role in the pore formation of biochar. The generation and release of gas during the high-temperature pyrolysis activation reaction caused the SD skeleton to form abundant pores. In addition, metal potassium, reduced by carbon, was converted into gaseous potassium when the activation temperature reached 800 , which infiltrated the inner structure of the biochar, forming a larger specific surface area and abundant hierarchically porous structures. The possible activation reactions between SD and $KHCO_3$ are as follows (eq 3-7). SDB-K-3 (Fig. 1b) showed the most uniform pore structure, while the pores of SDB-K-2, SDB-K-4, and SDB-K-5 contained blocky impurities, which could be

177 attributed to the different proportions of SD and activators, resulting in an incomplete
178 reaction or skeleton collapse and pore blockage.

179 $$2KHCO_3 \rightarrow K_2CO_3 + CO_2 + H_2O \quad (3)$$

180 $$K_2CO_3 \rightarrow K_2O + CO_2 \quad (4)$$

181 $$K_2CO_3 + 2C \rightarrow 2K + 3CO \quad (5)$$

182 $$K_2O + C \rightarrow 2K + CO \quad (6)$$

183 $$CO_2 + C \rightarrow 2CO \quad (7)$$

184 According to the pore structure analysis with SEM, the low-resolution microscopic
185 image (inset of Fig. 1c) revealed the thick carbonaceous sheets of the material.
186 Furthermore, the high-resolution TEM image (Fig.1c) of SDB-K-3 demonstrated its
187 internal graphitic structure. The pores, indicated by white arrows in Fig. 1d were
188 distributed throughout SDB-K-3, showing distinct lattice fringes with a distance of
189 0.246 nm, corresponding to the graphite (101) plane. The structure consisted of graphite
190 particles embedded in disordered carbon, which denoted the precursors of graphene and
191 heated sufficiently for the particles to form large sheets of graphene.

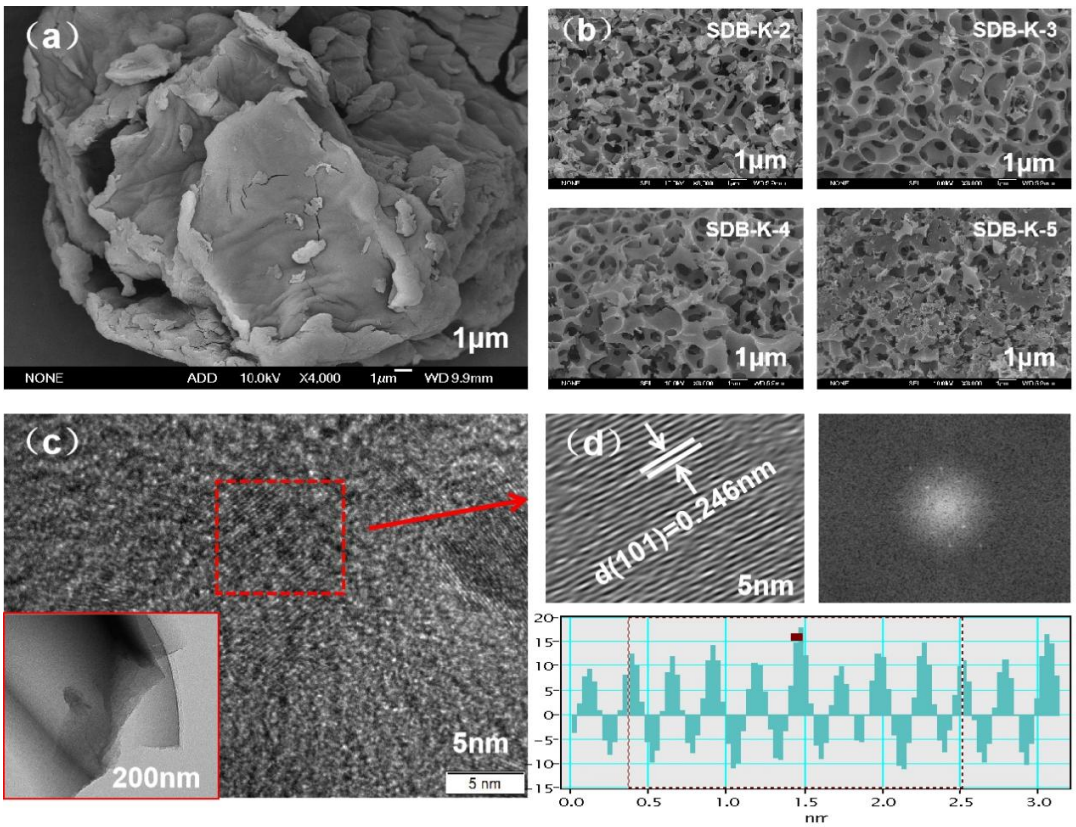

Fig. 1. SEM images of SD (a) and SDB-K-X (b). The TEM image (c) and the high-resolution TEM image (d) of SDB-K-3.

3.1.2. EA and BET

The C, O, H, N, and S content of SD and SDB-K-X were measured using an elemental analyzer. The results in Table S1 showed that, after high-temperature activation with $KHCO_3$, the biochar yield of the samples exceeded 15%, among which SDB-K-3 reached the highest yield of up to 15.61%. The C content of SDB-K-X increased from 41.14% to over 66%, while the O, H, N, and S content all decreased. The O content of SDB-K-X decreased from 46.63% to about 26% and was probably due to the influence of different $KHCO_3$ ratios and the formation of gas during the reaction.

The porous structure of SD and SDB-K-X was characterized by nitrogen sorption isothermal analysis. As illustrated in Fig. 2a, SDB-K-X exhibited a typical type I adsorption-desorption isotherm, indicating the microporous structure mainly existsed.

Fig. 2b showed that the pores of SDB-K-X were evenly distributed, and mainly concentrated at 0.75-1.75 nm. With the interaction between substances and the release of gas products in the reaction process, the framework of SDB-K-X was etched to produce a large number of micropores, thus forming a developed layered porous structure (Norouzi et al., 2018). The summary in Table 1 indicates that as the ratio of SD to $KHCO_3$ increased, all the SDB-K-X samples displayed extremely high specific surface areas, exceeding 1500 $m^2 g^{-1}$. When the mass ratio of SD to $KHCO_3$ was 1:3, the subsequent SDB-K-3 exhibited the maximum specific surface area and pore volume of up to 1620 $m^2 g^{-1}$ and 0.7509 $cm^3 g^{-1}$. As such, the specific surface area first increased and then decreased with the SD to $KHCO_3$ ration became higher, and could be attributed to the degree in which $KHCO_3$ responded to SD. The analysis was consistent with the SEM results. In general, materials with a more extensive specific surface area contain more active sites, and a considerable number of micropores are used as the active adsorption sites, which is conducive to the removal of dyes (Parshetti et al., 2014).

Table 1. Specific surface area, pore volume, and average pore size of SDB-K-X.

Sample	BET ($m^2 g^{-1}$	Vtot ($cm^3 g^{-1}$	Average pore diameter (nm)
SDB-K-2	1572	0.6786	1.727
SDB-K-3	1620	0.7509	1.859
SDB-K-4	1510	0.7375	1.953
SDB-K-5	1425	0.6376	1.790

3.1.3. XRD and Raman

The SDB-K-X were subjected to XRD analysis to characterize the crystalline structures. The SDB-K-X samples all displayed typically disordered amorphous carbon, as suggested by the low-intensity and broadened peaks in the XRD patterns (Fig. 2c). There were two main diffraction peaks around 24° and 43°, corresponding to the (002)

and (101) plane diffraction (PDF#41-1487), respectively (Zhang et al., 2019).

The Raman spectra (Fig. 2d) further confirmed the formation of a graphitized SD structure. Two distinct bands were located at around 1341 cm^{-1} (D band) and 1585 cm^{-1} (G band), providing information regarding the disorder and crystallinity of sp^2 carbon materials (Ferrari, 2007). The I_D/I_G value is usually used to represent the degree of graphitization. All the SDB-K-X samples contained graphitized structures at different degrees, among which the I_D/I_G of SDB-K-3 was the smallest (0.96), indicating that the graphitization degree of SDB-K-3 was relatively high and the structural defects were fewer. This result can be attributed to activators at different quality levels affecting the graphite structure, leading to the formation of a defective texture.

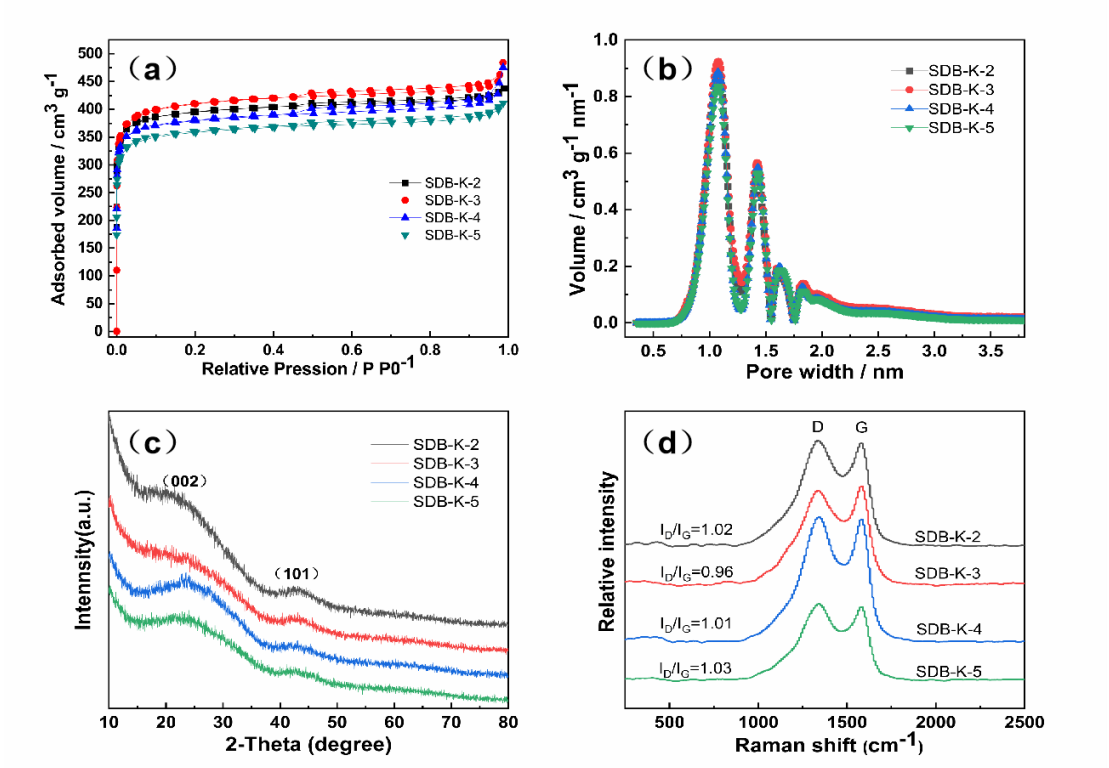

Fig. 2. The N_2 adsorption-desorption isotherms (a), and pore size distribution (b) of SDB-K-X. The XRD image (c) and the Raman image (d) of SDB-K-X.

3.1.4. FTIR and XPS

The FTIR spectra of SDB-K-X are illustrated in Fig. 3a, indicating that functional groups exist on the surface of biochar, which is different from SD. This may be caused by the volatilization of water and organic matter, as well as the interaction between functional groups during high-temperature activation. The band at 3411 cm^{-1} was ascribed to -OH stretching vibration (Wang and Liu, 2017), while the peak at 2933 cm^{-1} was attributed to C-H stretching vibration. Carboxyl C=O stretching vibrations and C=C peaks were identified at 1710 and 1587 cm^{-1}, the peak at 1095 cm^{-1} was ascribed to the C-OH stretching vibration (Yu et al., 2019). Therefore, the samples contained abundant functional groups, which is conducive to improving the adsorption performance of organic dyes.

Based on the structural characterization analysis of SDB-K-X, XPS was used to evaluate the elemental composition and surface groups of SDB-K-3, which primarily contained C and O, as well as a small amount of N. These results corresponded well with the EA findings (Fig. 3b) after the peak-differentiating-imitating analysis to identify the high-resolution spectra of C1s and O1s. As shown in Fig. 3c, and Fig. 3d, the C 1s spectrum was deconvoluted into four peak components at 284.7 eV, 285.6 eV, 287.2 eV, and 289.0 eV, which were attributed to C-C/C=C (67.8), C-O (21.5), C=O (5.2) and O-C=O (5.5) groups, respectively (Choudhary et al., 2013). The O1s peaks confirmed the presence of C=O (21.0), O-(C, H) (52.1), and O-C=O (26.9) at 531.5 eV, 532.6 eV, and 533.8 eV (Datsyuk et al., 2008), respectively. These results were consistent with those of the FTIR characterization, indicating that the SDB-K-3 surface contained a considerable number of oxygen-containing functional groups.

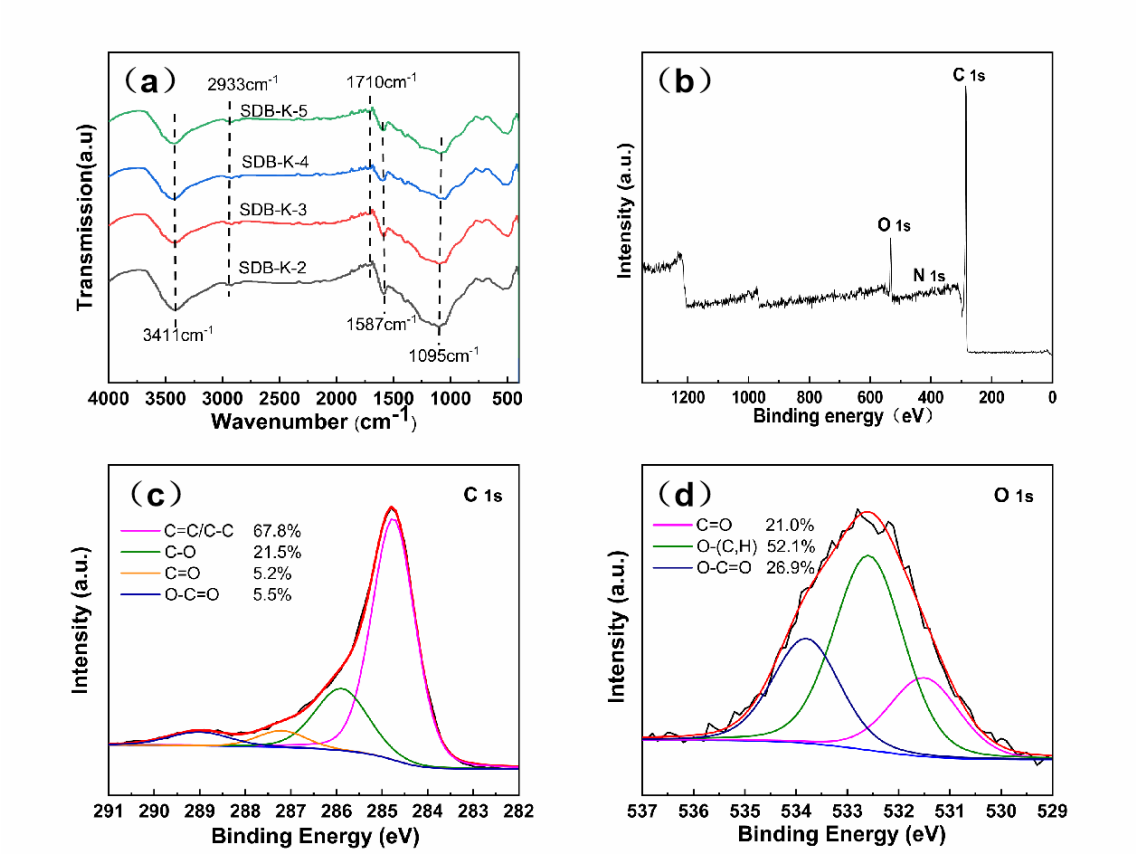

Fig. 3. FT-IR spectra (a) of SDB-K-3, XPS survey (b), high-resolution spectra of C1s (c), and high-resolution spectra of O1s (d) for SDB-K-3.

3.2. The adsorption process

Combined with the structural characterization of biochar, SDB-K-3 was selected as the best adsorbent for the removal of MB, which was attributed to its highest specific surface area, suitable pore volume, and abundant oxygen-containing functional groups. The adsorption of SDB-K-3 at different initial concentrations (1000 mg L^{-1}, 1500 mg L^{-1}, and 2000 mg L^{-1}) was investigated, indicating that the adsorption capacity increased from 999.54 mg g^{-1} to 1175.20 mg g^{-1}. The adsorption kinetics, adsorption equilibrium, and adsorption thermodynamics of SDB-K-3 for MB are further illustrated in the following sections.

3.2.1. Adsorption kinetics

To investigate the effect of the sorption behavior mechanism of SDB-K-3 on MB, kinetic adsorption data were simulated using the pseudo-first-order and pseudo-second-order models. The equations of the two models are expressed as follows (Luo et al., 2013; Liu et al., 2015):

$$\ln(q_e - q_t) = \ln q_e - k_1 t \quad \text{(pseudo-first-order)} \qquad (8)$$

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \quad \text{(pseudo-first-order)} \qquad (9)$$

where q_t and q_e (mg g^{-1}) are the amounts of MB adsorbed at time t and equilibrium, respectively, while k_1 (per min) and k_2 (g mg^{-1} per min) is the pseudo-first-order and pseudo-second-order model rate constants of adsorption, respectively.

Fig. 4a shows that the adsorption efficiency increased with the extension of adsorption time, while the adsorption process reached a balance, and the adsorption sites were saturation, maintaining an equilibrium in the adsorption capacity (Yu et al., 2019). As the concentration increased, the mass transfer driving force rose, the interaction between SDB-K-3 and MB improved, and the adsorption capacity increased until reaching the adsorption saturation. The maximum absorption Q (mg g^{-1}) amounts of 1000 mg L^{-1}, 1500 mg L^{-1}, and 2000 mg L^{-1} MB were 996.29 mg g^{-1}, 1130.00 mg g^{-1}, and 1179.26 mg g^{-1}, and the removal efficiency R (%) were 99.63 , 75.33 and 58.96 , respectively. The adsorption efficiency of SDB-K-3 to MB increased rapidly within 0.5 h and reached the adsorption equilibrium within 2 h. The rapid adsorption process may be ascribed to the existence of effective binding sites, as well as the formation of a hierarchical porous structure with a high SDB-K-3 surface area (Wang et al., 2016).

297 The model regarding the relevant kinetic parameters for MB adsorption is provided
298 in Table 2, while Fig. 4b and Fig. 4c illustrate the kinetic plots. Therefore, the
299 pseudo-second-order model displayed higher R^2 values (>0.99998) in the three MB
300 concentrations than the pseudo-first-order model. Notably, the pseudo-second-order
301 model can describe the adsorption process better than the pseudo-first-order model,
302 meaning that the chemical effect was also involved in the adsorption of MB on
303 SDB-K-3 (Yu et al., 2019). This result can be ascribed to the high surface area and
304 surface abundance of functional groups contributing to the diffusion of organic dye
305 molecules from the water to the surface of the adsorbent. Furthermore, the calculated
306 values of the adsorption amounts at equilibriums of 1000 mg L^{-1}, 1500 mg L^{-1}, and 2000
307 mg L^{-1} MB onto SDB-6-K from the pseudo-second-order model were 1000.00 mg g^{-1},
308 1131.31 mg g^{-1}, and 1179.96 mg g^{-1}, respectively, which were approximately the same
309 as those from the experimental results (996.29 mg g^{-1}, 1130.00 mg g^{-1}, and 1179.26 mg
310 g^{-1}). These findings suggested that the pseudo-second-order kinetic model was more
311 suitable for describing the adsorption behavior of MB onto SDB-K-3.

312 **Table 2**. Kinetic model parameters for the adsorption of MB on SDB-K-3.

Sample	C_0 mg L^{-1}	q_e exp mg g^{-1}	Pseudo-first-order			Pseudo-second-order		
			q_e,cal ($mg \cdot g^{-1}$)	K_1 (min^{-1})	R^2	q_e,cal ($mg \cdot g^{-1}$)	K_2 ($g \cdot mg^{-1} \cdot min^{-1}$)	R^2
SBD-K-3	1000	996.29	18.98	0.00779	0.85332	1000.00	0.00224	1
	1500	1130.00	34.83	0.01065	0.77671	1131.31	0.00120	1
	2000	1179.26	57.35	0.00679	0.70865	1179.96	0.00062	0.99998

313 *3.2.2. Adsorption equilibrium*

314 The adsorption isotherm is used to describe the interaction between adsorbate and
315 adsorbent, indicating the distribution of adsorption molecules between the liquid and

solid phase when the adsorption process reaches an equilibrium state. To analyze the adsorption mechanism of the MB on the surface of SDB-K-3, the adsorption data were fitted using the Langmuir (Langmuir, 1918) and Freundlich (Freundlich, 1907) isotherm models, respectively. The Langmuir adsorption isotherm assumes that adsorption occurs in a single-layer at a specific homogeneous site inside the adsorbent. The Freundlich isotherm is an empirical equation suggested for adsorption systems on the heterogeneous surface. The linear form of the Langmuir and Freundlich isotherm equations can be written as:

$$\frac{C_e}{q_e} = \frac{C_e}{q_m} + \frac{1}{k_L q_m} \quad (10)$$

$$\ln q_e = \ln k_F + \frac{1}{n_F} \ln C_e \quad (11)$$

where C_e (mg g^{-1}) and q_e (mg g^{-1}) are the concentrations of MB in the solution and the adsorbent at the adsorption equilibrium, respectively. q_m (mg g^{-1}) is the MB monolayer adsorption capacity, k_L is the adsorption constants, which are related to the free energy of adsorption. n_F and k_F are Freundlich adsorption constants related to adsorption capacity and sorption intensity, respectively.

The adsorption isotherms of SDB-K-3 for MB at different initial dye concentrations and temperatures are shown in Fig. 4d. By increasing the initial concentration of MB at a specific temperature, q_e gradually increased, and then approached saturation adsorption. With the increase of temperature, the saturation adsorption capacity (qm) increases accordingly, confirming the active influence of temperature on adsorption capacity. Therefore, it can be inferred that the adsorption of SDB-K-3 on MB is an endothermic process.

The curves simulated in Fig. 4e, indicate that the curves of the Langmuir model are more suitable for fitting the adsorption data than those of the Freundlich isotherm model (Fig. 4f). Therefore, the k_1 and q_e values were calculated from the plots provided in Table 3, indicating that the correlation coefficients (R^2 >0.999) of the Langmuir model for all initial MB concentrations were more significant than those found when using the Freundlich model. The results indicated the adsorption process of SDB-K-3 on MB occurred in a single-layer.

Table 3. Isotherm model parameters for the adsorption of MB on SDB-K-3.

Sample	T/K	Langmuir			Freundlich		
		q_m(mg·g^{-1})	K_L(L·mg^{-1})	R^2	n	K_F	R^2
SBD-K-3	298	1179.80	0.1477	0.99954	44.35	1001.83	0.96722
	308	1235.49	0.2891	0.99977	34.08	1022.17	0.97325
	318	1271.95	0.3657	0.99983	31.89	1045.62	0.93420

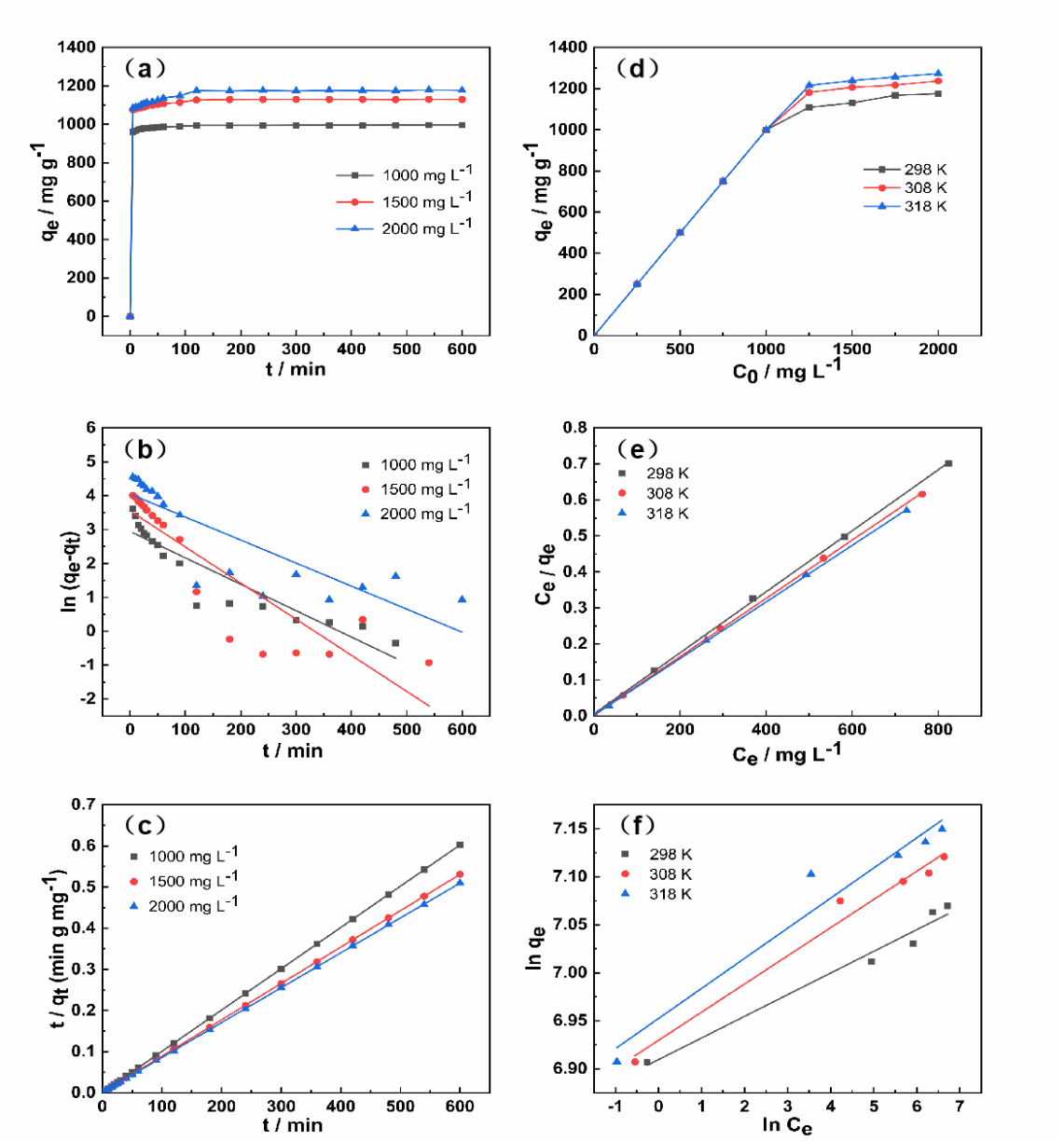

Fig. 4. The effect of the contact time on MB adsorption by SDB-K-3 (a). Kinetic fitting curves for the MB adsorption by SDB-K-3, pseudo-first-order(b), and pseudo-second-order (c). Conditions: T = 298 K; m=0.025 g; V=25L; C0=1000 mg L^{-1}, 1500 mg L^{-1}, and 2000 mg L^{-1}. The effect of the initial concentration and temperature of the dye on the adsorption performance (d). Linearized Langmuir isotherms for 2000 mg L^{-1} MB adsorption by SDB-K-3 (e) and linearized Freundlich isotherms for 2000 mg L^{-1} MB adsorption by SDB-K-3 (f). Conditions: T = 298 K, 308 K, and 318 K; m=0.025 g.

3.2.3. Thermodynamic analysis

To estimate the type of adsorption for SDB-K-3 on MB, the thermodynamic adsorption parameters, such as the ΔG ($kJ \cdot mol^{-1}$), the ΔH ($kJ \cdot mol^{-1}$), and the ΔS ($J \cdot K^{-1} \cdot mol^{-1}$) were calculated. The thermodynamic parameters can be obtained with the

following equations (Manasi et al., 2014):

$$\ln K_c = \frac{-\Delta H}{RT} + \frac{\Delta S}{R} \quad (12)$$

$$\Delta G = -RT\ln K_C \quad (13)$$

$$K_C = \frac{q_e}{C_e} \quad (14)$$

where R is the gas constant (8.314 $Jmol^{-1}$ K^{-1}); K_c is the thermodynamic equilibrium constant, and T (K) is the sorption temperature.

The ΔH and ΔS values can be calculated from the slope and intercept of the linear plot of ln Kc and 1/T. The obtained thermodynamic parameters are shown in Fig. S1 and Table S2. All acquired ΔG values were negative at all temperatures, indicating that the adsorption of MB on SDB-K-3 was spontaneous and feasible, suggesting a physisorption process (Zhang et al., 2020). The positive ΔH values implied the endothermic nature of the adsorption interactions. Furthermore, the positive ΔS value illustrated the adsorbent and the adsorption properties of some structural changes, while indicating the randomness of solid-liquid interface in the process of adsorption was increased (Yao et al., 2011).

3.3. Adsorption performance comparison with other adsorbents

The comparison of the maximum adsorption capacities of MB using various previously reported adsorbents is summarized in Table S3, showing that the adsorption capacity of SDB-K-3 for MB was higher than those of other adsorbents, such as garlic peel (Hameed and Ahmad, 2009), banana peel (Liu et al., 2014), seaweed (Ahmed et al., 2019), coconut shells (Islam et al., 2017b), wood (Danish et al., 2018), rattan (Islam et al., 2017a) and wheat straw (Li et al., 2016), which are considered naturally available

and cost-efficient. Different adsorbents exhibit different adsorption properties for MB, which may be caused by the variances in raw material composition and preparation methods, resulting in the composition and pore structure of biochar. Consequently, considering its low cost and comparable adsorption capacity, SDB-K-3 is assumed to be a promising adsorption material to remove MB from water.

4. Conclusions

In this study, the porous biochar prepared from SD shows a highly specific BET surface area (1620 m^2 g^{-1}), large pore volume (0.7509 cm^3 g^{-1}), and rich oxygen-containing hydrophobic groups. As a cost-effective dye adsorbent, it has significant application potential. SDB-K-3 exhibits excellent adsorption capacity for MB, with a maximum capability of 1273.51 mg g^{-1} for MB at 318K. Combined with the detailed characterization of SDB-K-3, this superior result can be ascribed to the 3D framework structure and active adsorption site, suggesting that the combination of chemical adsorption and physical adsorption, π-π interaction, hydrogen bonding, and electrostatic interaction may be possible adsorption mechanisms. These results demonstrate that the excellent adsorption performance of SDB-K-3 to MB is a possible option for practical industrial wastewater treatment. Further investigation should focus on optimizing the preparation method, reducing production costs, and realizing industrial applications.

Declaration of competing interest

The authors declare that they have no known competing fifinancial interests or personal relationships that could have appeared to inflfluence the work reported in this paper.

CRediT authorship contribution statement

Zhiwei Ying: Data curation and writing-original draft. **He Li:** Writing-review & editing. **Xinqi Liu:** Supervision and project administration. **Chi Zhang:** Software and Methodology. **Jian Zhang:** Formal analysis. **Guofu Yi:** Investigation.

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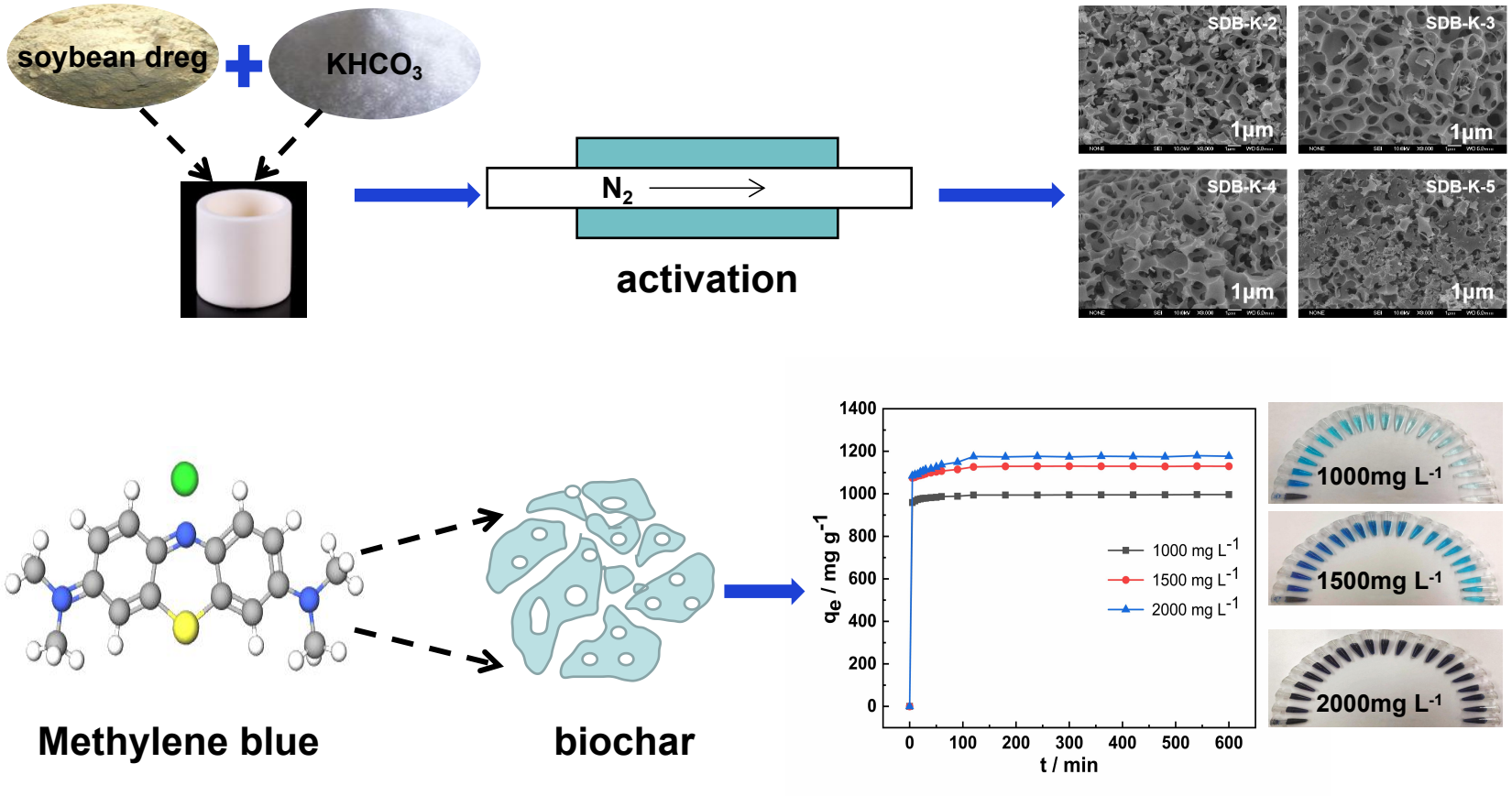
Graphical Abstract



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CHEM73030, Thank you for agreeing to review

1 message

Chemosphere <eesserver@eesmail.elsevier.com> Sun, May 17, 2020 at 8:29 AM
Reply-To: Chemosphere <chem-eo@elsevier.com>
To: widi_astuti@mail.unnes.ac.id, wiwied.unnes@gmail.com

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Dear Dr. Astuti,

Thank you for agreeing to review the manuscript "Preparation of biochar from biomass waste soybean dreg using a one-pot method for methylene blue adsorption from water" by Zhiwei Ying; He Li; Xinqi Liu; Chi Zhang; Jian Zhang; Guofu Yi for Chemosphere

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The manuscript reference is CHEM73030.

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In order to endeavor to offer the author a prompt review I would request that you return a timely report to me. If possible, I would appreciate receiving your review within the coming 21 days, however, if you require an extension of the due date, please contact the Journal Manager, Miss Pallavi Das at chem-eo@elsevier.com.

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Pallavi Das, Journal Manager
on behalf of Yeomin Yoon, Co-Editor in Chief
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3. Bukti reminder to review

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Widi Astuti <widi_astuti@mail.unnes.ac.id>

Reminder to Review for CHEM73030

1 message

Chemosphere <eesserver@eesmail.elsevier.com> Thu, Jun 4, 2020 at 7:19 AM
Reply-To: Chemosphere <chem-eo@elsevier.com>
To: widi_astuti@mail.unnes.ac.id, wiwied.unnes@gmail.com

*** Automated mail sent on behalf of Pallavi Das ***

Re: Preparation of biochar from biomass waste soybean dreg using a one-pot method for methylene blue adsorption from water
by Zhiwei Ying; He Li; Xinqi Liu; Chi Zhang; Jian Zhang; Guofu Yi
Submitted to Chemosphere

Dear Dr. Astuti,

You agreed to review Manuscript Number CHEM73030 on 17 May 2020 and your completed review is due by 07 Jun 2020.

Therefore I would be grateful if you would submit your review as soon as possible at

https://ees.elsevier.com/chem/.
Your username is: widi_astuti@mail.unnes.ac.id

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Best regards

Pallavi Das
Journal Manager
Chemosphere

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4. Bukti thank you for your review

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Widi Astuti <widi_astuti@mail.unnes.ac.id>

CHEM73030: Thank you for your review

1 message

Chemosphere <eesserver@eesmail.elsevier.com> Sat, Jun 6, 2020 at 6:56 PM
Reply-To: Chemosphere <chem-eo@elsevier.com>
To: widi_astuti@mail.unnes.ac.id, wiwied.unnes@gmail.com

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Re.: "Preparation of biochar from biomass waste soybean dreg using a one-pot method for methylene blue adsorption from water" (Dr. He Li)

Dear Dr. Astuti,

Thank you for your review of this manuscript.

You can access your review comments and the decision letter (when available) by logging on to:

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Kind regards,

Pallavi(On behalf of the handling Editor, Dr. Yeomin Yoon)
Chemosphere

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