Microporous activated carbon developed from KOH activated biomass waste: surface mechanistic study of methylene blue dye adsorption

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ABSTRACT

In this work, sugarcane bagasse waste (SBW) was used as a lignocellulosic precursor to develop a high-surface-area activated carbon (AC) by thermal treatment of the SBW impregnated with KOH. This SBW activated carbon (SBWAC) was characterized by crystallinity, porosity, surface morphology and functional groups availability. The SBWAC exhibited Type I isotherm which corresponds to microporosity with high specific surface area of 709.3 m²/g and 6.6 nm of mean pore diameter. Further application of SBWAC as an adsorbent for methylene blue (MB) dye removal demonstrated that the adsorption process closely followed the pseudo-second order kinetic and Freundlich isotherm models. Conversely, a thermodynamic study revealed the endothermic nature and spontaneity of MB dye adsorption on SBWAC with high acquired adsorption capacity (136.5 mg/g). The MB dye adsorption onto SBWAC possibly involved electrostatic interaction, H-bonding and π–π interaction. This work demonstrates SBW as a potential lignocellulosic precursor to produce high-surface-area AC that can potentially remove more cationic dyes from the aqueous environment.

Key words: adsorption mechanism, activated carbon, KOH activation, methylene blue, sugarcane bagasse waste

HIGHLIGHTS

- Microporous activated carbon was prepared from sugarcane bagasse via KOH activation.
- The activated carbon was used for adsorption of methylene blue dye.
- The adsorption capacity for methylene blue dye was 136.5 mg/g.
- The adsorption thermodynamic indicates spontaneous and endothermic process.

1. INTRODUCTION

Porous materials have received considerable interest for application in many fields such as energy storage, biochemical engineering, solar cell, and environmental abatement. Depending on the application, a material can be tuned into a desired degree of porosity (micropore, mesopore, macropore) using different types of activation methods, conditions, and raw materials (Medhat et al. 2021). For this purpose, prospective raw materials will normally be treated physically or chemically or by a combination of both methods to produce a porous network on their surface. In general, carbon-based materials and their derivatives can be processed into materials with desired porosity for different applications (Rashid et al. 2018; Bardhan et al. 2020). Some notable carbon materials are diamond, graphite, coke, fullerenes, carbynes and their derivatives such as carbon nanotube, graphene, carbon black and activated carbon (AC) out of which AC has gained the most attention within the carbon derivatives group as indicated by its high production and usage frequency every year (Malek & Laiman 2018; Rashid et al. 2019).

AC has become the preferred form of carbonaceous materials due to its excellent features of a substantially large surface area that usually relates to adsorption ability, porosity, favorable thermal stability, and mechanical strength as well as possessing...
abundant useful functional groups for various applications (Illingworth et al. 2019). However, the commercially available AC is largely produced from non-renewable raw materials namely petroleum coke, lignite and coal which are limited resources and costly precursors (Jawad & Abdulhameed 2020). Thus, the economical challenge in AC production triggered the global search for low-cost, renewable, and environmentally friendly precursors. Amongst those precursors, lignocellulosic biomasses emerge as promising green precursors in AC preparation for being renewable, vastly available, ecologically suitable, and costly effective (Supong et al. 2019). In recent years, many studies have focused on utilizing various biomasses to be the alternative precursors for preparation of AC such as corn fiber and soy hulls (Herde et al. 2020), tea waste (Inal & Aktas 2020), switchgrass (Yumak et al. 2020), peanut shell (Wang et al. 2020), cotton waste (Sartova et al. 2019), pine pollen (Wan et al. 2019), and bamboo chip (Jawad & Abdulhameed 2020). The use of raw materials from agricultural and industrial waste has also recovered its economic value, as most waste is normally ignored and disposed of (Bahrudin et al. 2020). For instance, sugarcane (Saccharum officinarum L.) is harvested yearly for the sugar and ethanol industries that have become the major export products for many developing countries (Arencibia 2000). In Malaysia, aside from being produced largely as refined sugar, the juice of sugarcane is often sold by street vendors or night marketers as nutritious and refreshing drink. The sugarcane bagasse waste (SBW) from the sugar industries and street vendors is largely disposed or burned off which can cause further environmental issues. Considering this, sugarcane and its by-products have been heavily researched as raw materials in biofuel, fertilizer, livestock feed and paper production (Chinnadurai 2017). Adding to its wide application spectrum, the SBW has also been converted to ACs as a highly effective adsorbent for dye removal from wastewater via adsorption (Gardare et al. 2015). This is undeniable since adsorption is usually associated with carbonaceous materials as the process requires an adsorbent with high surface area and porosity for high uptake of the target adsorbate which can be found in ACs. That relation has led to a recent strategy of producing ACs from biomass waste with minimal cost for application in environmental cleanup.

In general, physical and chemical activations are frequently applied during the preparation of lignocellulosic biomass-based AC (Njoku et al. 2014). Generally, chemically synthesized activated carbon exhibits a higher specific surface area and higher production amount at lower operational conditions (treatment time and reaction temperature) than the one activated by physical treatment (Brito et al. 2018). In addition, chemical treatment can replace the -OH and phenolic groups of a lignocellulosic material with negatively charged groups of the activating agents used, thus increasing the adsorption favorability (Guarín et al. 2018). During the chemical activation procedure, the raw material was crushed and milled before being impregnated with selected chemical activators like sodium hydroxide (NaOH), phosphoric acid (H₃PO₄), potassium hydroxide (KOH), and zinc chloride (ZnCl₂). The impregnated ACs that undergo heat treatment process are then washed with copious amount of water before drying and use in the desired form (Abdel-Ghani et al. 2016). Among these chemical activation agents, KOH is preferable as it can produce ACs with high specific surface area and form oxygen-based functional groups (Araga et al. 2017; Astuti et al. 2019). AC treated by KOH has been successfully produced from various lignocellulosic based biomasses with large specific surface area and substantial distribution of pores that made them attractive especially as adsorbents for water treatment application (Rashid et al. 2016; Chomiak et al. 2017; Spessato et al. 2019).

Therefore, the current study aims to convert SBW into high-surface-area porous AC or known as SBWAC throughout this study. The ability of SBWAC in the removal of cationic dyes such as methylene blue (MB) dye which has been used as color agent in paper, textile, leather, plastics, pharmaceuticals, and cosmetics-based industries was investigated. The adsorption operational factors, namely, adsorbent dosage, pH of the solution, initial MB concentration and time were optimized based on the dye removal efficiency and adsorption capacity. The adsorption properties involving kinetics, isotherms and thermodynamic were also evaluated based on a series of initial MB dye concentrations, whereas the MB dye adsorption on the SBWAC surface was mechanically discussed according to the related characterizations.

2. MATERIALS AND METHODS

2.1. Materials

The SBW was acquired from a local wet market in Shah Alam, Selangor, Malaysia. Potassium hydroxide (KOH) and probe pollutant, MB dye (MW: 319.86 g/mol; \( \lambda_{\text{max}} = 661 \) nm) were procured from R&M Chemicals. Deionized water was used in preparation of all reagents and solutions in this study.
2.2. Preparation of SBWAC

The SBW was significantly subjected to deionized water to ensure it was free from any dust or attached particles and fully sanitized before drying in an oven at 80 °C overnight. The dried SBW was crushed and sieved into size particles of 250–500 μm. During the impregnation, 1 g of SBW powder was mixed with 2 g of KOH in which the optimum mixing ratio had been predetermined initially. The KOH impregnated SBW sample was left to dry overnight in the oven at 110 °C. The next day, the sample was carbonized in a tubular reactor for 60 min at 700 °C under purified nitrogen (99.99%) and 1 atm of pressure. After cooling down, the SBWAC was washed with acidic solution (HCl, 3M) and then with hot distilled water to remove excess KOH until the pH of water became neutral. Then, the SBWAC was left in the oven for another night at a temperature of 110 °C. Finally, the SBWAC sample was sieved to produce a fine powder with particle size of similar or less than 250 μm for further applications.

2.3. Characterization

The porosity and specific surface area of SBWAC was estimated by surface area analyzer (Micromeritics ASAP 2060 analyzer) at analysis time and temperature of 8 h and 250 °C, respectively. The amorphous/crystalline structure of SBWAC was examined by powder X-ray diffraction (XRD) using an X’PERT PRO diffractometer. The point of zero charge (pH_{pzc}) of the SBWAC was measured as per the reported method by Dalvand et al. (2016). The morphologies of SBWAC and SBWAC after adsorption was analyzed using scanning electron microscopy (SEM) of model Zeiss Supra 40 VP. The investigation of related functional groups of KOH-treated AC before and after MB uptake was performed by a Fourier transform infrared (FTIR) spectrophotometer model Spectrum RX I from Perkin-Elmer.

2.4. Adsorption of MB on SBWAC

The adsorption conditions were optimized consecutively by means of adsorbent dosage ranging from 0.02 g to 0.12 g, various initial concentrations ranging from 50 to 250 mg/L and adjusted pH solutions from 2 to 12. Each optimization step used a set of 250 ml Erlenmeyer flasks containing 100 ml of MB solution, then the flasks were stirred in a water bath shaker at 110 strokes/min. The final absorbances of MB dye solution were measured at 661 nm by UV-Vis spectroscopy of model HACH DR 2800 for which the concentrations were estimated from the build calibration curve. The isotherm of equilibrium was estimated using optimum conditions at 303 K based on initial concentrations of MB dye in the range of 50 to 250 mg/L and contact time from 0 to 720 min. The dye removal percentage (DR %), and adsorption capacity (q_e (mg/g)) of SBWAC toward MB dye at equilibrium were calculated by Equations (1) and (2), respectively:

\[
DR \% = \left(\frac{C_o - C_e}{C_o}\right) \times 100
\]

\[
q_e = \frac{(C_o - C_e)V}{W}
\]

Here, \(C_o\) (mg/L) and \(C_e\) (mg/L) represent the initial and equilibrium MB dye concentrations, respectively whereas \(V\) (L) and \(W\) (g) represent the volume of dye solution and dosage of SBWAC, respectively.

3. RESULTS AND DISCUSSION

3.1. Characteristics of SBWAC

Properties of ACs produced are highly dependent on the type of activation process and raw materials used. The quality of as-produced ACs is usually characterized based on the specific surface area, pore volume and pore size distribution values (Bergna et al. 2018). Figure 1 shows the N2 adsorption-desorption isotherm plot of SBWAC and the corresponding textural properties of SBWAC are tabulated in Table 1. The plot shows Type I isotherm with hysteresis loop of Type 4 according to IUPAC classification (Sing 1985). The adsorption trend of SBWAC shows a sharp increase at the low relative pressure range due to the micropore filling effect whereas at high pressure, the trend of the plot shows multilayer sorption and capillary condensation which suggests the presence of mixed micro- and mesopores within the sample (Qi et al. 2017). The capillary condensation occurred at a relative pressure (P/P_o) over 0.7 which further indicates the presence of narrow slit-like mesopores in the structure of SBWAC (Lv et al. 2015). According to Brunauer–Emmett–Teller (BET) analysis, the SBWAC possesses specific surface area of 709.3 m²/g, pore volume of 0.31 cm³/g while the mean pore diameter was calculated to
be 6.6 nm. The results show that the KOH treatment on the raw sugarcane bagasse had successfully produced AC with large specific surface area. The pores of SBWAC are categorized as mesoporous as per IUPAC classification (Sing 1985). Furthermore, Type I isotherm is usually associated with microporosity which is evidenced by high area covered by micropores (623.1 m²/g) that is about 87.8% of overall specific surface area. This indicates that SBWAC consists of a mixture of micropores and mesopores which further validates the earlier discussion on isotherm and hysteresis loop type of SBWAC.

The XRD analysis pattern of the SBWAC in the 2θ range of 0° to 90° is given in Figure 2. The XRD pattern of SBWAC exhibits distinct peaks at 2θ of around 23° and 44° of graphitic crystallite of carbon-based material corresponding to the

| Property                        | Value  |
|--------------------------------|--------|
| Specific surface area (BET) (m²/g) | 709.3  |
| Micropore surface area (m²/g)    | 623.1  |
| Total pore volume (cm³/g)        | 0.31   |
| Mean pore diameter (nm)          | 6.6    |
| V_m (cm³/g)                      | 0.25   |
(002) and (100) planes, respectively (Xie et al. 2014). The broad diffraction peaks with no significant single sharp peak indicates the amorphous nature of SBWAC and confirms the porous structure of SBWAC (Li et al. 2017). The diffraction peak of SBWAC was not so intense/sharp compared to other ACs-related works indicating that the crystallite size was small. Since the crystallinity is low, the specific surface area is large because of widening or internal structure disorder of the whole wall of AC which consists of graphite crystallite supporting the previously discussed BET result (Xie et al. 2014; Sawant et al. 2017).

3.2. Adsorption study

3.2.1. Effect of SBWAC dosage

The adsorbent dosage is essentially important in an adsorption system since it can influence the removal efficiency of target pollutant and determine the overall cost if used in large scale application. The impact of SWBAC dose on MB dye removal was observed at adsorbent dose from 0.02 g to 0.12 g while maintaining the other key parameters constant as described in Section 2.4. As per Figure 3(a), the MB dye removal efficiency raised from 10.5% to 83.2% in parallel with the increase of

Figure 3 | (a) Effect of adsorbent dosage on MB dye removal, (b) effect of solution pH on the adsorption of MB dye, and (c) pH<sub>pzc</sub> of SBWAC.
the adsorbent dosage from 0.02 g to 0.08 g. It was apparent that additional increase in the SBWAC dosage above 0.08 g did not show any significant dye removal. The increment on the amount of dye removed is related to the increasing number of active adsorption sites which is associated with the SBWAC dosage. However, when passing over maximum dosage, the removal efficiency was maintained, as the available adsorption sites might be excessive to accommodate the fixed amount of MB dye molecules in the treated solution. In addition, the high SBWAC dosage could also induce aggregation that led to fewer functional adsorption sites available for MB uptake. As the highest MB dye removal of 83.2% was achieved at 0.08 g, thus, 0.08 g of SBWAC dosage was adopted for the forthcoming applications.

3.2.2. Effect of solution pH

pH of solution is one of the influential factors in the adsorption process due to its role in the change of adsorbent surface charge and degree of ionization of adsorbate molecules. The influence of pH on MB dye removal was explored in several pH solutions (2–12) using the previously optimized SBWAC dosage while keeping the other parameters constant. In order to understand the MB uptake in any pH solution, it is necessary to find the point in which the surface net charges are neutral termed as point of zero charges (pH\text{pzc}). In this regard, the pH\text{pzc} of the SBWAC was recorded at 6.8 as shown in Figure 3(b). This finding indicates that the SBWAC surface acquired positive charges at pH below 6.8 while the surface was negatively charged above pH 6.8. Beside the pH\text{pzc} of SBWAC, the acid dissociation constant (pK\text{a}) of MB dye also needs to be considered as well. It was reported that the pK\text{a} value of MB dye is estimated to be 3.8 which makes the dye molecules exist mostly in cationic salt in solution as they ionize and become positively charged radicals (Sousa et al. 2019).

At acidic pH region, the surface charge of SBWAC is dominated by positive charge which induces the MB electrostatic repulsion. As presented in Figure 3(c), the highest capacity of 48.1 mg/g was found at pH 8 of MB solution that is in the basic region. At this pH, the surface of SBWAC is changed into negative charges that attract the MB dye cations via electrostatic attraction. Better dye removal is achieved as the result of the interaction between the negatively charged functional groups on SBWAC surface with the cation MB dye molecules as given in Equation (3). Thus, a solution of pH 8 was adopted as an optimum solution pH for next investigations:

\[
\text{SBWAC} - \text{O}^- + ^+\text{MB} \leftrightarrow \text{SBWAC} - \text{O}^- \ldots \ldots ^+\text{MB} \quad (3)
\]

3.2.3. Effect of contact time and initial MB concentration

The influence of contact time on MB dye adsorption on the surface of SBWAC at various MB dye initial concentrations (50, 100, 150, 200, and 250 mg/L) was investigated using the previously optimized parameters. The plots of adsorption capacities of SBWAC, \( q_t \) (mg/g) versus time (min) at various MB dye concentrations is exhibited in Figure 4(a). It can be noticed that the adsorption capacity of SBWAC increased from 60.1 to 142.2 mg/g with increasing MB dye concentration from 50 to 250 mg/L. Rapid increment can be seen at the early stage of the adsorption process of all concentrations due to the availability and vacant adsorption sites of SBWAC for MB dye uptake. As the time increased, the curve became gradually plateaud indicating the adsorption slowed down before reaching the state of adsorbent–adsorbate equilibrium. The equilibrium for SBWAC–MB interaction at concentration 50, 100, 150, 200, 200 mg/L was achieved at 200, 300, 500, 550 and 650 min, respectively. More time was needed to attain equilibrium for the MB dye with higher concentrations because of the concentration gradient that increases the dye diffusion into adsorption sites surface and internal pores of the adsorbent (Jawad & Abdulhameed 2020).

3.3. Kinetic modeling

To design an adsorption system, the kinetic investigation that controls an adsorption system as well as the rate deciding phase is crucial. Two kinetic models, pseudo-first order (PFO) and pseudo-second order (PSO) were applied to the experimental data to elucidate the adsorption mechanism and possible rate control measures, such as mass transport and chemical reaction processes of MB dye adsorption on SBWAC. It has been discussed recently that the linear form of kinetic models is arguable due to its incoherent data fitting and statistical bias (Bahrudin et al. 2019). Thus, the non-linear forms of PFO (Lagergren 1898) and PSO (Ho & McKay 1998) kinetics were applied to the experimental data as shown in Equations (4) and (5), respectively. The most applicable model used to describe the adsorption was chosen based on the best coefficient of
The determination ($R^2$) value as obtained using Equation (6):

$$q_t = q_e(1 - \exp^{-k_1t})$$  \hspace{1cm} (4)

$$q_t = \frac{q_e^2k_2t}{1 + q_ek_2t}$$  \hspace{1cm} (5)

$$R^2 = 1 - \frac{\sum_{n=1}^{n} (q_{e, \text{exp}} - q_{e, \text{cal}})^2}{\sum_{n=1}^{n} (q_{e, \text{exp}} - q_{e, \text{exp}})^2}$$  \hspace{1cm} (6)

where, $q_e$ (mg/g) and $q_t$ (mg/g) signify the quantity of MB dye uptake by SBWAC at equilibrium and at time ($t$), respectively. Conversely, $k_1$ (1/min), and $k_2$ (g/mg min) are the rate constants for the PFO and PSO models, respectively. The plots of PFO and PSO are shown in Figure 4(a) while the extracted parameters and their $R^2$ values are given in Table 2. It was found from Table 2 that the MB uptake by SBWAC obeyed the PSO model because of the higher $R^2$ values, which is related to the
closeness of agreement between the calculated $q_e (q_{e,cal})$ and experimental $q_e (q_{e,exp})$ values as compared with the one of PFO model. This result denotes that the MB uptake by SBWAC preferably occurred via chemical interaction.

### 3.4. Isotherm modeling

For the most efficient use of an adsorbent, a thorough understanding of the essence of the interaction between the adsorbate and the adsorbent is needed. Three renowned isotherms (Langmuir, Freundlich, and Temkin) were adopted to describe MB dye–SBWAC interaction by means of adsorbent affinity and adsorption potential of SBWAC towards MB dye. Langmuir model specifically describes the homogeneous and monolayer adsorption on fixed adsorption sites (Kireç et al. 2021). Freundlich model assumes the heterogeneous multilayer adsorption on different surface energies (Erol et al. 2019a, 2019b). Meanwhile, the Temkin isotherm model describes that the heat of adsorption decreases with the adsorption coverage due to the adsorbent–adsorbate interactions (Bahrudin et al. 2019). The non-linear expressions of Langmuir (Langmuir 1918), Freundlich (Freundlich 1906), and Temkin (Temkin 1940) isotherms are given in Equations (7)–(9), respectively as follows:

$$q_e = \frac{q_{max} K_a C_e}{1 + K_a C_e}$$  \hspace{1cm} (7)

$$q_e = K_f C_e^{1/n}$$  \hspace{1cm} (8)

$$q_e = \frac{RT}{b_T} \ln(K_T C_e)$$  \hspace{1cm} (9)

where $q_e$ (mg/g) and $C_e$ (mg/L) are the quantity and concentration of MB dye adsorbed at equilibrium, respectively. Meanwhile, $q_{max}$ (mg/g) is MB dye uptake (maximum capacity), $K_a$ is Langmuir constant (L/mg), $K_f$ [mg/g (L/mg)$^1/n$] and $n$ are the Freundlich constants. For the Temkin model, $K_T$ refers to the Temkin constant (L/mg), $b_T$ is the heat of adsorption (J/mol) and $R$ is the gas constant (8.314 J/mol K).

The plots of the non-linear isotherms are presented in Figure 4(b), while the values of corresponding isotherm parameters are tabulated in Table 3. Based on the $R^2$ values of isotherm models in Table 3, the MB adsorption onto the SBWAC surface

### Table 2 | PFO and PSO kinetic parameters for MB dye adsorption by SBWAC

| $C_e$ (mg/L) | $q_{e,exp}$ (mg/g) | $q_{e,cal}$ (mg/g) | $k_1$ (1/min) | $R^2$ |
|-------------|------------------|------------------|--------------|--------|
| 50          | 60.1             | 59.0             | 0.050        | 0.96   |
| 100         | 83.4             | 79.9             | 0.035        | 0.89   |
| 150         | 118.3            | 107.4            | 0.018        | 0.83   |
| 200         | 122.9            | 111.9            | 0.036        | 0.80   |
| 250         | 142.2            | 125.3            | 0.033        | 0.78   |

| $C_e$ (mg/L) | $q_{e,exp}$ (mg/g) | $q_{e,cal}$ (mg/g) | $k_2$ (10$^{-3}$ g/mg min) | $R^2$ |
|-------------|------------------|------------------|----------------------------|--------|
| 50          | 61.5             | 61.5             | 0.146                      | 0.99   |
| 100         | 84.3             | 84.3             | 0.071                      | 0.96   |
| 150         | 118.4            | 118.4            | 0.021                      | 0.95   |
| 200         | 118.5            | 118.5            | 0.050                      | 0.94   |
| 250         | 138.4            | 138.4            | 0.039                      | 0.94   |

### Table 3 | Parameters of isotherm models for MB dye adsorption by SBWAC at 303 K

| Adsorption isotherm | Parameter | Value |
|---------------------|-----------|-------|
| Langmuir            | $q_{max}$ (mg/g) | 136.5 |
|                     | $K_a$ (L/mg)    | 16.1  |
|                     | $R^2$           | 0.89  |
| Freundlich          | $K_f$ (mg/g) [L/mg]$^{1/n}$ | 38.6  |
|                     | $n$             | 3.8   |
|                     | $R^2$           | 0.97  |
| Temkin              | $K_T$ (L/mg)    | 0.87  |
|                     | $b_T$ (J/mol)   | 108.3 |
|                     | $R^2$           | 0.94  |
obeyed the Freundlich isotherm, indicating the uptake of MB on the SBWAC was multilayer on the heterogeneous active sites and binding energies (Jawad & Abdulhameed 2020). Besides $R^2$, the calculated $n$ can be used to deduce the favorability of the adsorption which is categorized as follows; $n$ value equals to one is unity, more than one means the adsorption is favorable at high concentrations, and less than one indicates the adsorption is unfavorable. For this study, $n$ is more than one which shows the favorability of SBWAC adsorption towards MB dye at high temperature as shown through the calculated $q_{\text{max}}$ of 136.5 mg/g at 303 K. The $q_{\text{max}}$ of SBWAC is then compared with the adsorption capacities of ACs as prepared by KOH activation as listed in Table 4. The recorded data (Table 4) clarified that the SBWAC is a highly effective adsorbent due to its high specific surface area and adsorption capacity for MB dye removal from aquatic environment.

### 3.5. Adsorption thermodynamics

Thermodynamic functions of MB dye adsorption by SBWAC that include Gibb’s free energy ($\Delta G^\circ$), enthalpy ($\Delta H^\circ$), and entropy ($\Delta S^\circ$) are obtained using the following Equations (10)–(12).

\[
\Delta G^\circ = -RT \ln K_d \tag{10}
\]

\[
K_d = \frac{q_e}{C_e} \tag{11}
\]

\[
\ln K_d = \frac{\Delta S^\circ}{R} - \frac{\Delta H^\circ}{RT} \tag{12}
\]

The values of thermodynamic parameters were obtained from the intercept and slope of the van’t Hoff plot of $\ln K_d$ against $1/T$, respectively as depicted in Figure 5. The thermodynamic parameters are presented in Table 5. The increasing $K_d$ with an increase in reaction temperature postulates that the adsorption capacity of SBWAC towards MB dye also increased. The spontaneity of MB dye uptake is confirmed due to the negative value of the $\Delta G^\circ$ (Alacabey et al. 2020). Moreover, the positive value of the $\Delta H^\circ$ indicates the endothermic nature of the adsorption process. The positive value of $\Delta S^\circ$ further suggests increment in the randomness at solid-solution interface because of water displacement by MB dye during the uptake. The increasing $K_d$ value with rising temperature and endothermic nature of SBWAC-MB reaction advocate that the adsorption can be done at high temperature. The study reveals that the adsorption of MB dye was favorable and became more spontaneous at elevated temperatures as the pores of AC expanded with additional heat as well as increment of the mobility rate of MB dye onto SBWAC pores, thus increased the adsorption capacity (Gedam et al. 2019). Some related studies using MB dye as adsorbate also showed similar thermodynamic characteristics (Rashid et al. 2016).

### 3.6. Characteristics of SBWAC before and after MB adsorption

The morphological structure and involved functional adsorption groups of the SBWAC were investigated by SEM and FTIR analyses, respectively. Figure 6(a) and 6(b) refer to the morphological structures of SBWAC before and after uptake of MB, respectively. Figure 6(a) shows the porous surface of SBWAC with plenty of regular and visible pores well distributed over

### Table 4 | Comparison of adsorption capacities of MB by different activated carbon materials prepared by KOH activation

| Activated carbons          | $S_{\text{BET}}$ (m$^2$/g) | $q_{\text{max}}$ (mg/g) | References                      |
|----------------------------|-----------------------------|------------------------|---------------------------------|
| Sugarcane bagasse waste    | 709.3                       | 136.5                  | This study                      |
| Malaysian Selantik coal    | 1,094.3                     | 491.7                  | Jawad et al. (2019)             |
| Polyethylene terephthalate bottles | 353.307                   | 404.089                | Djahed et al. (2016)            |
| Crofton weed               | 3,918                       | 387.60                 | Cheng et al. (2016)             |
| Pentace species sawdust    | 914.15                      | 357.14                 | Khasri et al. (2018)            |
| India shrub wood           | 1,024                       | 257.73                 | Perasaheb et al. (2016)         |
| Jatropha seed husks        | 1,262                       | 250                    | Qaid et al. (2016)              |
| Karanj fruit hulls         | 828.30                      | 239.4                  | Islam et al. (2017)             |
| Paper mill sludge          | 1,002.0                     | 152.0                  | Kang et al. (2006)              |
| Wood                      | 167.68                      | 59.92                  | Danish et al. (2018)            |
SBWAC surface creating a sponge-like porous structure. This type of porosity is highly preferred in adsorption processes of macromolecules such as MB dye for a successful uptake. Large and well developed pores were obtained as the result of pore development during pyrolysis that promotes the diffusion of KOH into the cavity, induces the reaction with carbon and eventually creates more pores. The corresponding interaction of KOH and carbon layers specifically graphene is based on the following reaction (Omri & Benzina 2012):

\[6\text{KOH} + 2\text{C} \leftrightarrow 2\text{K} + 3\text{H}_2 + 2\text{K}_2\text{CO}_3\]  

(13)

Figure 6(b) shows the morphology of SBWAC after loading the MB molecules on its surface. It can be clearly seen that some of the external pore walls of SBWAC had collapsed which gave the pore its porosity indicating the MB dye molecules had passed through the SBWAC surface and accommodated the pores during the adsorption. However, there was no drastic or significant change in the internal pores that can be seen after adsorption, showing that the produced pores were quite rigid and could potentially induce more dye uptake onto SBWAC for subsequent cycle.

Figure 7(a) and 7(b) display FTIR spectra of SBWAC before and after MB adsorption, respectively. The difference between the functional groups before and after MB dye adsorption can be used to deduce the corresponding adsorption mechanism. As seen in Figure 7(a), the FTIR spectrum of as-prepared SBWAC displays typical characteristic peaks of ACs which are the vibrations of –OH groups at 3,648.35 cm\(^{-1}\) to 3,837.36 cm\(^{-1}\), stretching of C≡C at 2,268.13 cm\(^{-1}\) and vibrations of C=O bond at 1,655.89 cm\(^{-1}\) (Erol 2016; Erol & Uzun 2017). Meanwhile, the peaks at 1,530.95 cm\(^{-1}\) and 1,427.9 cm\(^{-1}\) correspond to the stretching vibrations of C=C bonds of aromatic ring while the peak at 1,186.84 cm\(^{-1}\) relates to the vibrations of C-O-C bonds. On the other hand, the peak at 1,043.78 cm\(^{-1}\) corresponds to the stretching vibrations of the C-O bonds of ethers, alcohols, phenols, and esters, whereas the peak that belongs to the C-H derivatives is observed at 803.28 cm\(^{-1}\) (Anis et al. 2021; Borghesi et al. 2021). After the MB adsorption, the intensity of the peaks of oxygen-containing functional groups like –OH, C=O and C≡O became less intense, as seen in Figure 7(b), evidencing their involvement in MB dye adsorption. The
FTIR analysis has confirmed that the carbonyl and other oxygen-containing functional groups in SBWAC molecular structure were the functional groups responsible for MB dye uptake.

3.7. Mechanism of MB dye uptake

As generally known, the adsorption properties of a carbon material are strongly dependent on the surface area as well as the surface chemistry. From the morphological image and specific surface area analysis of SBWAC, there were plenty of well-developed pores distributed on the adsorbent surface that produced a large surface area which essentially favors the adsorption. Even though the surface of SBWAC was largely dominated by a micropores network, the pore diameter was on an average 6.6 nm that could easily allow the diffusion of MB dye molecules with molecular size of 0.84 nm into the internal pores of SBWAC (Guarin et al. 2018). The mechanism of MB dye uptake on the SBWAC surface is proposed based on the previously observed effect of pH and FTIR analyses, as the presence of various chemical species on the surface of AC shows that there is more than one interaction existing during the process. Several probable interactions between MB and SBWAC surface are demonstrated in Figure 8. Based on the pH solution study, electrostatic interactions might occur between

Figure 6 | SEM images at x6,000 magnification power of (a) SBWAC, and (b) SBWAC after MB adsorption.
the MB dye cations and negatively charged sites (carbonyl and oxygen-containing functional groups) which are available on the SBWAC surface especially seen in neutral and alkaline solutions. Hydrogen bonding is an additional interaction that can exist between free H of oxygen-containing functional groups on the SBWAC surface with nitrogen in MB dye structure as observed in the FTIR result after MB dye uptake. Lastly, π-π interaction comes from interacting the hexagonal skeleton of SBWAC with MB aromatic rings as the electron-donating functional groups (−OH) of SBWAC might elevate the π-donating strength of the aromatic ring (Li et al. 2019; Surip et al. 2020).

4. CONCLUSION

Microporous dominated SBWAC was successfully manufactured via thermal pyrolysis of SBW in the presence of KOH and employed as an adsorbent for the removal of aqueous MB dye. Optimal conditions for MB dye adsorption on SBWAC surface
were found to be in basic dye solution (pH 8) with adsorbent dosage of 0.08 g/L at 303 K. The kinetics and equilibrium isotherms showed that MB dye removal involved chemical and multilayer adsorption on heterogeneous surface energies of SBWAC with a measured $q_{\text{max}}$ value of 136.5 mg/g at 303 K. It was found that electrostatic interaction, $\pi$-$\pi$ interaction, and H-bonding were possibly involved in the mechanism of MB adsorption on the SBWAC. The achieved data demonstrated that the feasibility of using SBW as a low-cost and high-potential lignocellulosic precursor for the synthesis of highly porous activated carbon with a high preference for adsorption of cationic dyes, in addition to possible potential application towards other types of organic water pollutants such as pharmaceutical compounds, pesticide, phenolic compounds, and inorganic metal ions.

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**CONFLICT OF INTEREST FORM**

The authors declare that there is no conflict of interest in this work.

**DATA AVAILABILITY STATEMENT**

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

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