The Effects of Pre-treatments and Low-temperature Pyrolysis on Surface Properties of Biochar from Sunflower Straw as Adsorption Material

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Carbon adsorbent materials that were prepared from sunflower straw by a combination of pre-treatment and low-temperature pyrolysis showed better adsorption compared with untreated carbon. Four different pre-treatment agents (steam, alkali (KOH), phosphoric (H_3PO_4), and salt ($ZnCl_2$)) were analyzed with respect to their effects on the maximum surface area and the micropore area. Samples were measured by thermogravimetric analysis (TGA), X-ray powder diffraction (XRD), scanning electron microscopy (SEM), surface area analysis, and pore size analysis. The surface area, pore volume, and N_2-adsorption capacity of the samples were closely correlated with the pre-treating agent. A biochar with a maximum surface area of 877.6 m^2/g and a micropore area of 792.8 m^2/g was prepared with phosphoric acid (H_3PO_4) as the pre-treatment agent at a temperature of 400 °C. The main result of the one-stage pre-treatment procedure was the number of micropores. The two-stage, low-temperature pyrolysis procedure focused on the volume of the pores. Carbonized sunflower straw, with pretreated and low-temperature pyrolysis procedures, was judged to be a highly effective and economic method to prepare carbon adsorbents.

Keywords: Low-temperature pyrolysis; Pre-treated method; Biochar; Sunflower straw; Adsorption

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INTRODUCTION

With the acceleration of the urbanization process and the development of industrialization, the use of sunflower straw and other agricultural waste as feed for fuel applications has been continuously reduced (Stigka *et al*. 2014). However, agricultural waste production continues to grow. Sunflower straw is one of the main sources of agricultural waste. The total production of sunflower seeds is estimated to be 175.8 million tons for September 2016, which could generate a total of 527.4 million tons of sunflower straw calculated according to the ratio of sunflower straw and sunflower production in China (Bi *et al*. 2009; Bao 2014; Mucri *et al*. 2016). The disposal of agricultural waste has been a crucial concern for the last two decades in light of environmental pollution and global climate changes (Fu *et al.* 2016).

Recently, cheap and environmentally friendly adsorbents using agricultural wastes have become a popular research topic (Njoku *et al.* 2014; Yahya *et al*. 2015; Adebisi *et al*. 2016; Narimane *et al*. 2016). In general, activated carbon adsorbent is prepared through carbonization and then activated with two steps, with the activation

temperature being higher than the carbonization temperature (> 500 °C) (Liou 2010; Dinesh *et al*. 2014; Eda and Canan 2015). However, it is difficult to achieve continuous production due to the complex preparation process, and a high temperature leads to a longer production time. One alternative is to find new methods with both an environmental and continuous simplified process to produce carbons (Huang *et al*. 2015). Porous carbon from tomato waste (TWNC) was obtained by activating the tomato waste (TW) with salt ($ZnCl_2$) (TW/$ZnCl_2$ weight ratio of 1:1), followed by pyrolysis of the impregnated sample at 500 °C for 1 h under a nitrogen atmosphere of 99.99% and a flow rate of 100 mL/min (Güzel *et al*. 2014). Activated carbon from fox nutshell has been prepared in a one-step chemical activation process and then pyrolyzed at temperatures ranging from 500 °C to 700 °C (Kumar and Jena 2015). Compared with the activating followed by carbonizing, the process of carbonizing followed by activating is a simpler process according to the preceding studies. However, to prevent toxic gases from being produced when $ZnCl_2$ is heated over 280 °C, other regulatory reagents are required in pyrolysis, and the time is also increased due to the higher temperature. (Li *et al.* 2015) has shown that the bio-based carbon, prepared at low temperatures, has a higher adsorption capacity for organic pollutants than that prepared at high temperatures.

In the present work, biochars were prepared from sunflower straw through a combination of pre-treatment steps using different treatment agents (*i.e.*, steam, alkali (KOH), phosphoric (H_3PO_4), and salt ($ZnCl_2$)). The biochars were subsequently pyrolyzed at a low temperature in a simple and continuous process. The physical and chemical characteristics of the bio-based carbons were examined by thermogravimetric analysis (TGA), X-ray powder diffraction (XRD), scanning electron microscopy (SEM), and N_2 adsorption analysis. The surface area and pore characteristics were measured using a surface area and pore size analyzer. The focus was the application of the process using a combination of pre-treatment methods with a low-temperature pyrolysis procedure.

EXPERIMENTAL

Materials

Pretreatment of sunflower straw

Sunflower straw, a by-product of agricultural crop sunflower, was harvested from Yuling, China, dried at 80 °C for 72 h in a blast oven (DHC-9053A, DAOHAN Industrial Co., Ltd., Shanghai, China), and crushed by a powder machine. About 100 g of the dry and crushed sunflower straw (SS) was equally divided into four parts, which were treated by steam explosion, wet impregnation with KOH (Jiuyi Chemical Reagent Co., Ltd., Shanghai, China) using a KOH SS ratio of 2.25 wt.%, $ZnCl_2$ (Jiuyi chemical reagent Co., Ltd., Shanghai, China) using a 3 mol/L $ZnCl_2$ solution, and H_3PO_4 (Nanjing Chemical Reagent Co., Ltd., Nanjing, China) using a H_3PO_4 SS ratio of 4 wt.%. The mixtures were stirred with a magnetic stirring apparatus for 2 h. The resulting slurries were dried at 105 °C for 72 h. The dry solids were sealed in a vacuum bag for carbonization.

Preparation of bio-based carbon from sunflower straw

Biochar samples of about 25 g from each pre-treatment were pyrolyzed in a vacuum furnace (VHS-234H-1600, Shenyang Jayu Vacuum Technology Co., Ltd., Shenyang, China) at 320 °C (10 °C/min^{-1}) for 25 min, into a N_2 flux (10 L/min^{-1}) at 300

°C (10 °C/min^{-1}) for 25 min, 400 °C (10 °C/min^{-1}) for 25 min, and 200 °C (10 °C/min^{-1}) for 25 min. The products resulting from steam-, KOH-, H_3PO_4-, and $ZnCl_2$-treated biochar were named SSBS, SSBA, SSBP, and SSBZ, respectively; the yield for all samples was more than 30%. After cooling, the SSBA, SSBP, and SSBZ were washed with 0.10 M HCl (Nanjing Senking Chemical Co., Ltd., Nanjing, China) and ultra-pure water several times until the pH was between 6 and 7. The products were dried at 105 °C for 48 h. SSBS was dried at 105 °C for 48 h. All samples were sealed in vacuum bags.

Methods

The four types of carbon were characterized by several techniques. The proximate analysis of the samples was conducted by TGA coupled with a differential thermal analyzer (DTA) (STA449F3, NETZSCH, Selbe, German), as recommended by Rashidi *et al.* (2012) and Chowdhury *et al*. (2016a). In the TGA analysis, 5 to 10 mg of each powder sample was sealed into a ceramic crucible (Φ8×5) and heated under a 5 mL /min N_2 flow at 1300 °C with a heating rate of 10 °C/min. Also, the phase identification of the samples was analyzed by XRD (X'Pert PRO, Almelo, Holland) (Chand *et al*. 2008). The morphology of the samples was characterized by scanning electron microscopy (SEM) (ZEISSEVO18, German Zeiss, Oberkochen, Germany) (Chand *et al*. 2009). The surface area and pore sizes in the pyrolyzed biochar were determined by the multipoint N_2 adsorption-desorption method at -196.15 °C using a surface area and pore size analyzer (JW-BK132F, Beijing Micro High-Bo Ltd., Beijing, China) (Li *et al*. 2013). Surface functional groups were identified by FTIR analysis (Nicolet iS-10, Thermo Fisher Scientific, Waltham, USA). The samples were dried and crushed with KBr. The sample mixed with KBr was pressed to form transparent sheets. Spectra were measured in the range between 400 and 4000 cm^{-1}.

RESULTS AND DISCUSSION

Proximate Analysis

Proximate analysis defines carbonaceous materials by their moisture content, volatile substance, ash content, and formed carbon. The formed carbon content was calculated using Eq. 1,

$$Q = \frac{m_2}{m_1} \times 100\,\% \quad (1)$$

where m_1 and m_2 were the mass of sunflower straw before and carbonization (g), respectively, and Q was the biochar yield of sunflower straw (%).

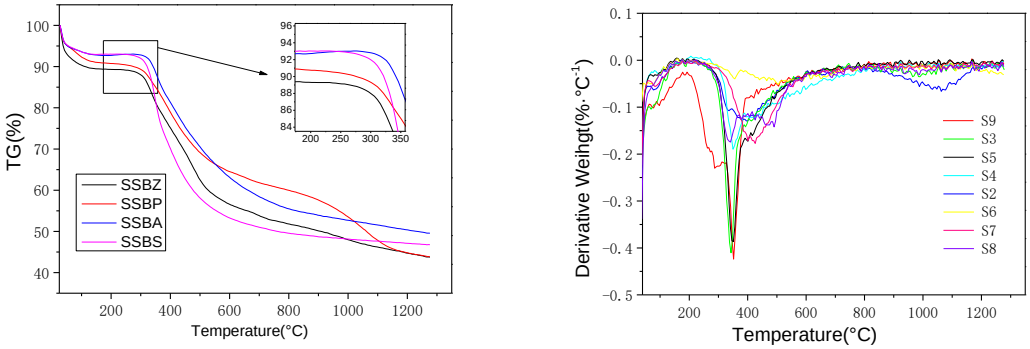
The proximate analysis was obtained by testing the weight loss as a function of temperature. The TG and DTG curves are shown in Fig. 1. The process was divided into two stages. The initial stage of the TG graph shows the moisture and volatile substance of the four types of carbons based on sunflower straw. The moisture content reflects the water retained by physical bonds only, and intrinsic and extrinsic moisture are the two basic kinds of moisture available. The extrinsic moisture is affected by the weather conditions, yet the intrinsic moisture is the moisture content of the material itself (Cox *et al*. 2002). The second stage of the curves included the loss of the volatile substances and organics. The biomass composition (*i.e.*, cellulose, hemicelluloses, and lignin) were reduced in this stage. While hemicelluloses and cellulose start to degrade at 220 °C and 300 °C, respectively, lignin degrades gradually in a wider temperature range (Pazó *et al*.

2010). Sma *et al*. (2011) reported that the temperature ranges for the degradation of hemicellulose, cellulose, and lignin are 250 °C to 320 °C, 320 °C to 380 °C, and room temperature to 900 °C, respectively. In contrast, the ash contents were the rest of the inorganic materials that remained after the combustion process, and the formed biochar was calculated by the difference in mass before and after carbonization.

The biochars from the sunflower straw had pronounced effects on the development of the volatile substance, ash substances, and formed biochar, as shown in Fig. 1. When the temperature increased to 100 °C, there were noticeable weight losses; the $ZnCl_2$- and H_3PO_4-treated samples lost more weight, indicating that they had higher moisture contents. As the temperature reached 200 °C, the biochar contents were expected to increase and the volatile matters to decrease due to pyrolysis. However, a large area of decomposition occurred between 280 °C and 300 °C. When the temperature increased further, the volatile substances were gradually released as gases. The weight remained stable after 700 °C, which indicated that the volatile substances and organics were removed (Chowdhury *et al*. 2016b). These results coincide with those reported by Lua and Guo (1998), who proposed that the proximate analysis of the activated carbon could be determined by the nature of the feedstock and operating conditions. In general, the results acquired in this study were comparable with previous reports.

X-ray Diffraction (XRD) Analysis

The XRD patterns of the biochars from sunflower straw using different methods are shown in Fig. 2. The XRD patterns of sunflowers treated with steam, KOH, H_3PO_4, and $ZnCl_2$ before and after pyrolysis (SSS, SSA, SSP, SSZ, SSBS, SSBA, SSBP, and SSBZ) exhibited broad peaks at about 2θ =25°, which corresponded to the graphite [002] crystal face and reacted graphitization degree. The biochar structure was transformed to a more orderly form after being activated. Meanwhile, SiO_2 and other impurities were contained in the samples according to pdf card analysis. And the sharp peak of SSBZ in XRD around 35° depicted impurity SiO_2 crystal [110] crystal face according to pdf card analysis (Liou and Wu 2009). In addition, peak crystal was not obvious in SSBA, SSBP, and SSBZ because KOH, H_3PO_4, and $ZnCl_2$ reacted with the raw materials and impurities at high temperature to form easily soluble compounds, most of which were removed after being pickled. This suggests that the crystalline structures of the prepared biochar changed after treatment with KOH and H_3PO_4. The crystalline index of the samples are given in Table 1 (Chowdhury *et al*. 2016c).



a.TG curves b. DTG curves

Fig. 1. Proximate analysis of the biochars from sunflower straw

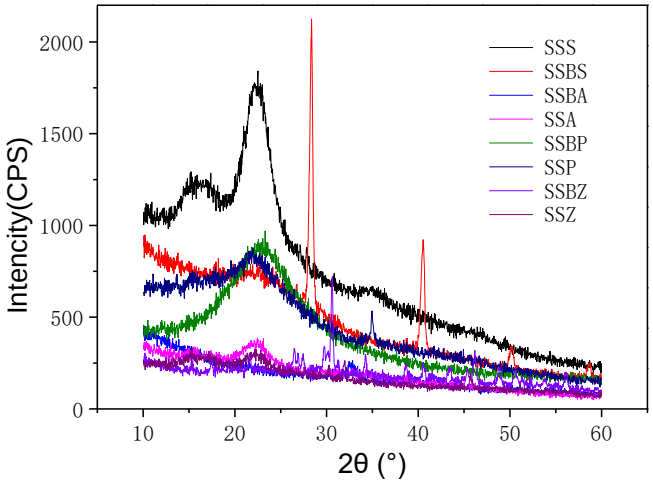


Fig. 2. X-ray diffraction patterns of the biochars from sunflower straw

Surface Morphologies

The biochars pre-treated with H_3PO_4, KOH, and $ZnCl_2$ were more effective in creating well-developed pores on the surface.

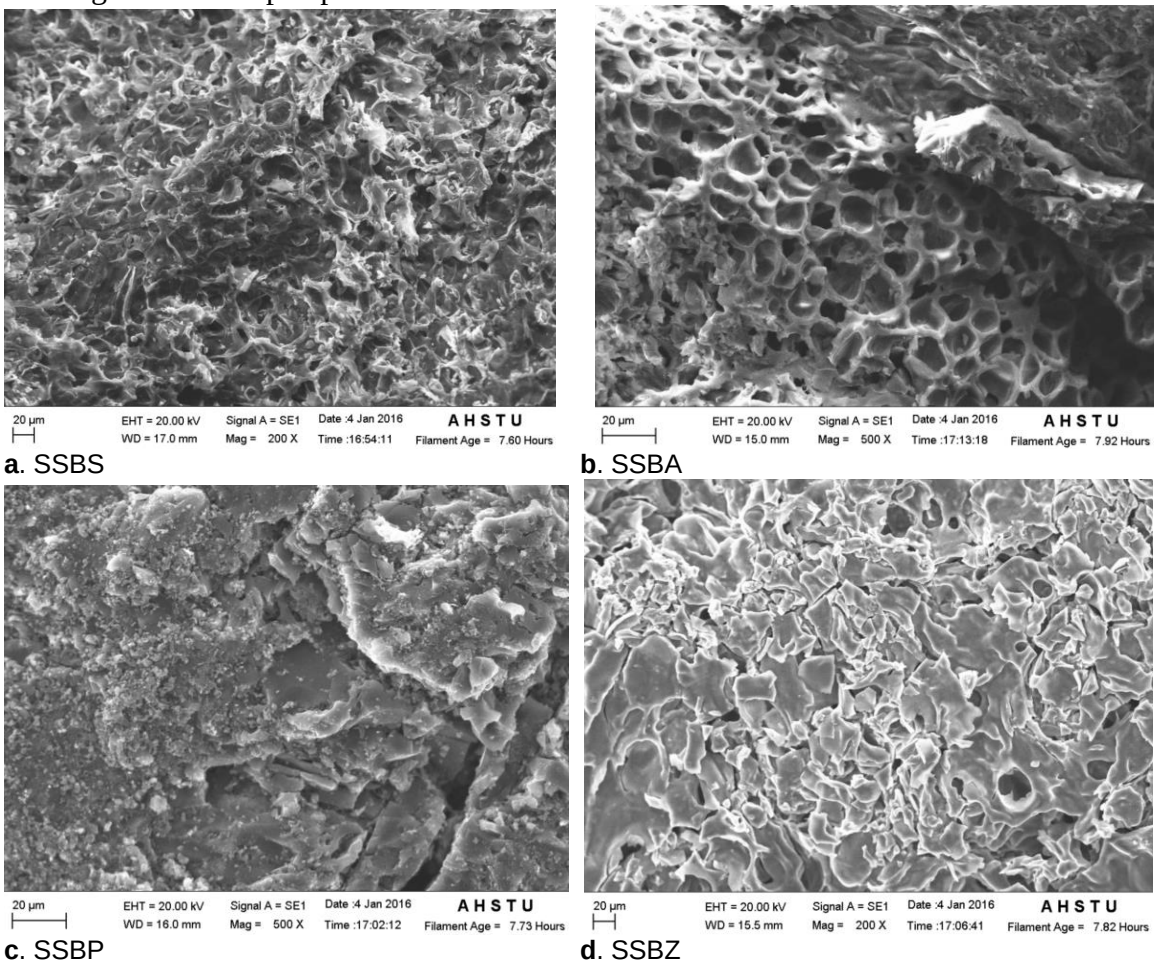


Fig. 3. The SEM morphologies of biomass carbon/activated carbon with four methods

Table 1. Crystallinity Index of all Types of Carbon with Starting Precursors

Type	SSS	SSA	SSP	SSZ	SSBS	SSBA	SSBP	SSBZ
Crystallinity index	13.99	16.87	18.24	15.08	19.23	25.42	29.25	26.38

In general, there were new pores formed because of the interaction between carbon and the activating agent (*i.e.*, H_3PO_4, KOH, and $ZnCl_2$) during the chemical activation. The SEM images of the samples displayed in Fig. 3 show the differences in surface morphology. As shown in Fig. 3a, the surface of the biochar based on sunflower straw without treatment was relatively smooth and had some pores on the surface. However, there were more pores or cracks in the three samples pre-treated with KOH, H_3PO_4, and $ZnCl_2$ (Figs. 3b, c, and d, respectively), which had a larger surface area because of high porosity.

Fourier Transform Infrared Spectroscopy (FTIR) Analysis

To determine the change of functional groups, the FTIR spectra of sunflowers with different treatments before and after pyrolysis are shown in Fig. 4. The main absorption peaks of samples appeared in the wavenumber region between 500 and 2000 cm^{-1}. Generally, the C=O stretching vibration in the carboxyl group was at 1706 cm^{-1}. The characteristic peak interval of benzene ring or aromatic was at 1450 to 1610 cm^{-1}. Familiarly, the absorption peaks that are representatives of C = O stretching vibrations, C = C stretching and-OH out-of-plane bending vibration of phenols, ethers and alcohols appeared at 1102 to 1252 cm^{-1}. The absorption peak of the aromatic compound C-H was at 793 cm^{-1}. This indicated that the samples with different treatments before and after pyrolysis had hydroxyl, aromatic, and some oxygen-containing functional groups, but the absorption intensity was different significantly. The results were similar to activated carbon (Chowdhury *et al*. 2015).

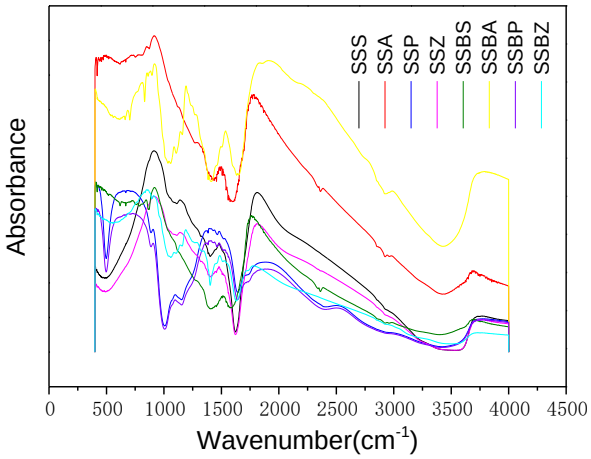


Fig. 4. FTIR spectra of all types of sample including the starting material

Surface Area and Pore Size Analysis

The N_2 adsorption-desorption isotherms and the pore size distribution of the four kinds of carbons based on sunflower straw are shown in Fig. 5. Adsorption-desorption data were obtained over a relative pressure (P/P_0) range from 10^{-4} to 0.95. The surface

area (S_{BET}) of the four samples based on sunflower straw was calculated by the Brunauer-Emmett-Teller (BET) equation, with the P/P_0 ranging from 0.006 to 0.10, and the micropore area (S_{mic}) was calculated by the T-plot method. The micropore volume (V_{mic}) was determined by the Horvath-Kawazoe (HK) method. The total pore volume (V_t) was obtained from the amount of N_2 adsorbed at a relative pressure (Ceng 2005).

As shown in Fig. 5, the adsorption isotherm curve was slightly biased towards the Y-axis at relatively low pressures where adsorption occurs within micropores, which indicated that there many micropores present. The adsorption volume for N_2 of biochars with the KOH, H_3PO_4, and $ZnCl_2$ treatments was higher. However, the adsorption volume for N_2 of carbon without treatment was far less than the other three samples. The physical performances of the samples are shown in Table 2. The BET surface area and micropore area of the biochars based on sunflower straw with the KOH, H_3PO_4, and $ZnCl_2$ treatments were higher; SSBP had the maximum values at 877.56 m^2/g and 792.77 m^2/g, respectively. However, the SSBS was also up to 49.616 m^2/g and 33.424 m^2/g. Thus, the treated samples were reasonably better for adsorption, and the one without treatment also acted as an absorbent according to other studies on straws (Li *et al*. 2015). The adsorption-desorption comparison of the four samples showed that the adsorption curve and desorption curve nearly overlapped (Figs. 5). As shown in Fig. 5 and Table 2, there were micropores (< 2 nm) and mesopores (2 nm to 50 nm) in the four samples, but the micropore volume (V_{mic}) occupied about 78% of the total pore volume. Smaller particle sizes of porous carbon result in greater rates of diffusion and adsorption (Yang *et al.* 2015), which further revealed that the samples were effective adsorbents.

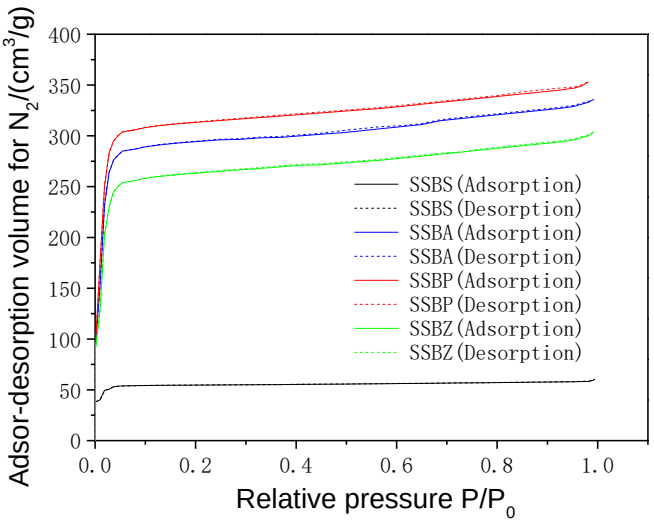


Fig. 5. Adsorption-desorption curves for N_2 of the biochars from sunflower straw

Table 2. The BET Surface Area and Pore Distribution of Samples

Type	SBET (m^2/g)[a]	S_{mic} (m^2/g)[b]	V_t (cm^3/g)[c]	V_{mic} (cm^3/g)[d]	D_P (nm)[e]
SSBS	49.616	33.424	0.1008	0.0965	0.49
SSBA	764.362	698.36	0.4840	0.4607	0.51
SSBP	877.56	792.77	0.5491	0.5013	0.52
SSBZ	642.37	587.88	0.3976	0.3620	0.49

a. BET surface area, b. Micropore area, c. Total pore volume, d. Micropore volume, and e. BJH desorption average pore diameter

CONCLUSIONS

1. The pre-treatment methods and the low-temperature pyrolysis procedure that was used to carbonize sunflower straw were found to be feasible and beneficial to the recycling of agricultural wastes.
2. The surface area and micropore area were mainly determined by the pre-treatment agent. A biochar with a maximum surface area of 877.6 m^2/g and micropore area of 792.8 m^2/g was prepared by using phosphoric acid (H_3PO_4) as the pre-treatment agent at the temperature of 400 °C. The SSBS using the steam pre-treatment only had a surface area of 49.6 m^2/g and a micropore area of 33.4 m^2/g.
3. The methods combined a pre-treatment step with a low-temperature pyrolysis procedure, which simplified the preparation of carbon adsorbents.
4. Based on this work, it was concluded that the carbon derived from sunflower straw has an enormous potential as an environmentally friendly, commercial, and effective adsorbent. However, there is a more simplified process for the bio-based pre-treated agent, which should be further investigated.

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