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Effect of Microwave Heating Conditions on the Preparation of High Surface Area Activated Carbon from Waste Bamboo

Abstract: The present study reports the effect of microwave power and microwave heating time on activated carbon adsorption ability. The waste bamboo was used to preparing high surface area activated carbon via microwave heating. The bamboo was carbonized for 2 h at 600°C to be used as the raw material. According to the results, microwave power and microwave heating time had a significant impact on the activating effect. The optimal KOH/C ratio of 4 was identified when microwave power and microwave heating time were 700 W and 15 min, respectively. Under the optimal conditions, surface area was estimated to be 3441 m²/g with pore volume of 2.093 ml/g and the significant proportion of activated

carbon was microporous (62.3%). The results of Fourier transform infrared spectroscopy (FTIR) were illustrated that activated carbon surface had abundant functional groups. Additionally the pore structure is characterized using Scanning Electron Microscope (SEM).

Keywords: high surface area activated carbon, microwave heating, waste bamboo

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Introduction

Activated carbon is a material that has abundant pore structure and high adsorption ability. Activated carbon which is widely used in many fields such as environmental, chemical, metallurgy, food processing and pharmaceutical has become an indispensable product in national economic development [1]. Activated carbon is produced from different raw carbon resources like wood, sawdust, coconut shell, almond shell and feces of silkworms [2–5]. Bamboo is a green construction material that has a lot of advantages such as capability of being reused or recycled, sustainability and environmental friendliness. Bamboo has been used in lots of fields such as construction, clothing, household appliances and entertainment materials [6–8]. However, waste bamboo materials are often dumped or burnt as wastes, which causes some serious pollution problems. Activated carbon which is prepared by waste bamboo has a broad prospect in our society. Moreover, bamboo-based activated carbon has been used for dyes, ammonia and so on [9, 10].

The activation process of activated carbon is mostly divided into two categories: physical activation process and chemical activation process [11, 12]. In physical activation process, carbon dioxide [13] and steam [14] are often used to make carbonized material mild oxidation. In chemical activation process, there are some activating agents widely used such as KOH [15–19], ZnCl_2 [20], K_2CO_3 [21, 22] and H_3PO_4 [23].

A conventional heating method is widely adopted in the preparation of activated carbon; however, it has some disadvantages such as long heating time and high energy consumption [24, 25]. Microwave heating is a new technique for the production of materials and it is widely used in the preparation and regeneration of activated carbon. Microwave heating has been proved beyond doubt to possess qualities such as fast heating, energy efficient, easy to control, small thermal inertia and selective heating [26–30].

The present work attempts to utilize microwave heating to prepare activated carbon from waste bamboo. The effects of microwave power and microwave heating time to activated carbon using KOH to be the activating agent have been studied. The resultant products are characterized by using the pore structure, pore size distribution, Fourier transform infrared spectroscopy (FTIR) analysis and Scanning Electron Microscope (SEM) analysis.

Experimental

Materials

Waste bamboo is selected as the raw material for activated carbon. The main reagents utilized in the experiments are KOH (AR) and hydrochloric acid (AR).

Methods

Waste bamboo removed of impurities was put into the crucible and heated to a carbonization temperature of 600°C at a heating rate of $10^\circ\text{C}/\text{min}$ and held for 2 h in an inert atmosphere. The carbonized material is put out from muffle furnace and crushed more than $600\text{--}1,000\ \mu\text{m}$ after it is cooling to room temperature in the muffle furnace. The carbonized material was put into the bottle after drying enough. The carbonized material was mixed with KOH/C mass ratio of 4 uniformly and put into the microwave furnace with crucible with heating at N_2 atmosphere. High-temperature material after microwave heating was transferred to distilled water for the formation of pores. The activated carbon was washed with 1:1 volume of hydrochloric acid ($0.1\ \text{mol/l}$), followed by rinsing with distilled water with pH value more than 6–7. Finally, samples were filtered, dried at 110°C for 4 h to be high surface area activated carbon.

Microwave heating apparatus was developed by Yunnan Provincial Key Laboratory of Intensification Metallurgy. The microwave frequency was 2,450 MHz. The maximum microwave output power was 700 W and power was continuously adjustable.

Characterization of activated carbon

The nitrogen adsorption isotherm of activated carbon is measured by automatic physical and chemical adsorption instrument (Autosorb-1-C, Quantachrome) at 77 K. Specific surface area of activated carbon is calculated by BET method and total pore volume is determined by nitrogen adsorption isotherm until a relative pressure of p/p_0 of 0.98. T-plot method is used to calculate micropore volume and external specific surface area. The pore size distribution is determined by the nitrogen adsorption isotherm generated utilizing non-localization density functional theory (NLDFT). FTIR spectra are generated in the range of $4,000\text{--}400\ \text{cm}^{-1}$ by using Nicolet 8700 spectrophotometer. The transmission spectra of samples are prepared by mixing with KBr crystals and pressed into a pellet.

Results and discussion

Effect of microwave power

Pore structure analysis

Effect of microwave power to activated carbon adsorption ability, yield and pore size distribution has been studied at optimal conditions. The nitrogen adsorption isotherms of the activated carbon prepared with different activating agents are shown in Figure 1. The condition of microwave power experiment is microwave heating time of 15 min. The pore structural parameters deduced from the isotherms are summarized in Table 1. Pore size distribution of activated carbon characterized through NLDFT is shown in Figure 2.

Figure 1: Nitrogen adsorption isotherm of the activated carbon with different microwave power.

As can be seen from Figure 1, the isotherms based on the International Union of Pure Applied Chemistry (IUPAC) [31] classification can be categorized to be type I isotherm. It is illustrated that main pore of activated carbon is micropore and nitrogen adsorption increases with relative pressure increase. Adsorption

Figure 2: Pore size distribution chart for different microwave power.

ability reaches more than 60% of saturated adsorption ability at a p/p_0 of 0.1. Adsorption ability increases with relative pressure increases beyond a p/p_0 of 0.1; however, there is a slow increasing trend of the isotherm and the adsorption isotherm is almost horizontal. Adsorption ability increases shapely and has tailing phenomenon until a p/p_0 of 1, which indicates that activated carbon contains a certain amount of mesopores and macropores.

Table 1 shows pore structure parameters such as BET surface area and total pore volume. With microwave power increasing, BET surface area, total pore volume, micropore surface area, external surface area, micropore volume and mesopore volume increase. At the same time, average pore size decreases with power increase. The micropore contents of activated carbon exceeded 60% at different power, which means activated carbon is micropore style.

Pore size distribution of activated carbon with the different microwave power is presented in Figure 2. According to IUPAC classification, pore size distribution of activated carbon is concentrated in the micropore field. With microwave power increasing, pore volume has a significant increase. Pore size distribution is widely spread.

Table 1: Details of pore structure of activated carbon with different microwave power.

Microwave power (W)	S_{BET} (m ² /g)	V_{tot} (ml/g)	D (nm)	V_{mic} (ml/g)	S_{mic} (m ² /g)	V_{mes} (ml/g)	S_{external} (m ² /g)	$V_{\text{mic}}/V_{\text{tot}}$ (%)
400	1,208	0.792	2.624	0.498	908	0.295	300.1	62.85
500	1,793	1.096	2.445	0.720	1,427	0.376	365.3	65.71
600	2,347	1.430	2.438	1.006	1,914	0.424	433.1	70.35
700	3,441	2.093	2.434	1.304	2,591	0.789	850.1	62.30

Figure 3: FTIR spectra of activated carbon at different microwave power from waste bamboo.

FTIR analysis

Figure 3 is the FTIR analysis of the activated carbon at different microwave power. As can be seen from Figure 3, FTIR spectra of activated carbon at different microwave power are similar, with the major peaks observed at 3,435, 1,705–1,630, 1,085 and 913–780 cm^{-1} . The band at around 3,435 cm^{-1} can be assigned to the –OH stretching vibration mode of hydroxyl functional groups. The band at 1,705 cm^{-1} indicates that there may exist C=O functional groups and the band at around 1,630 cm^{-1} can be assigned to C=C symmetrical stretching of pyrone and C=O of carboxylic groups and the band at 1,090 cm^{-1} is assigned to be C–O stretching vibration mode. The absorption peak at 1,210 cm^{-1} disappears at 700 W and the adsorption peak at 913–780 cm^{-1} is weakened, which obviously means C–H outer surface rocking vibration absorption is weak and carbon network structure is large.

Activation yield analysis

Figure 4 illustrates the effects of microwave power on the activation yield of activated carbon. It can be seen from Figure 4 that activation yield of activated carbon decreases with microwave power increase. With the microwave power increase, the material temperature increases and the reaction rate accelerates. At the same time, the ignition loss degree of activated carbon is larger. This is why activation yield decreases. When microwave power is 700 W, yield is 39.82%. Similar yields have been reported in literatures for other precursors, namely for holm-oak sawdust (25.5%) and rock-rose (20%) [32].

Figure 4: Effect of microwave power on the activation yield of activated carbon.

Effect of microwave heating time

Pore structure analysis

Figure 5 shows the nitrogen adsorption isotherms of the activated carbon prepared with different microwave heating times. Table 2 shows the details of pore structure while Figure 6 shows pore size distribution of activated carbon. The condition of experiment is microwave power of 700 W.

It can be seen from Figure 5 that the type of isotherms are close to type I isotherms as referred to IUPAC classification. When relative pressure reaches 0.1, adsorption is about 60% of saturation adsorption. With the relative pressure continuing to increase, adsorption increases

Figure 5: Nitrogen adsorption isotherm of the activated carbon with different microwave heating times.

Table 2: Details of pore structure of activated carbon with different microwave heating times.

Heating time (min)	S_{BET} (m^2/g)	V_{tot} (ml/g)	D (nm)	V_{mic} (ml/g)	S_{mic} (m^2/g)	V_{mes} (ml/g)	S_{external} (m^2/g)
10	2,569	1.687	2.627	1.079	2,050	0.608	518.5
15	3,441	2.093	2.434	1.304	2,591	0.789	850.1
20	3,003	1.813	2.415	1.331	2,543	0.482	459.3
25	2,896	1.796	2.480	1.275	2,422	0.521	474.6
30	2,659	1.694	2.548	1.041	2,032	0.653	626.8

Figure 6: Pore size distribution chart for different microwave heating times.

slowly and the isotherm is seen as an upwardly convex shape. Adsorption ability increases quickly with relative pressure increase close to a p/p_0 of 1.

It is shown in Table 2 that BET surface area, total pore volume, micropore surface area, external surface area, micropore volume and mesopore volume increase quickly when microwave heating time is less than 15 min. When microwave heating time reaches around 15 min, BET surface area, total pore volume and mesopore reach a maximum value. The micropore contents of activated carbon are beyond 60% at different heating times so that activated carbon could be seen in micropore style. Hence, microwave heating time has a significant effect to activated carbon.

Figure 6 indicates that pore size distribution is concentrated in micropore field according to IUPAC classification. The mesopore distribution around 20–40 Å is abundant. Pore volume becomes very small when pore size is beyond 40 Å.

FTIR analysis

Figure 7 shows the FTIR analysis of the activated carbon at different microwave heating times. It can be seen from

Figure 7 that FTIR spectra of activated carbon at different microwave heating times are similar, with the major peaks observed at 3,438, 1,630, 1,300–1,000 and 917–620 cm^{-1} . The band at around 3,438 cm^{-1} can be assigned to the –OH stretching vibration mode of hydroxyl functional groups. The band at around 1,630 cm^{-1} can be assigned to C=C symmetrical stretching of pyrone and C=O of carboxylic groups and the band at 1,200–1,000 cm^{-1} is assigned to the C–O stretching vibration mode. The adsorption peaks within 917 cm^{-1} are weak. When the microwave heating time becomes shorter, the C–H outer surface rocking vibration absorption is weaker.

Figure 7: FTIR spectra of activated carbon at different microwave heating times from waste bamboo.

Activation yield analysis

Figure 8 illustrates the effects of microwave heating time on the activation yield of activated carbon. As can be seen from Figure 8, activation yield of activated carbon decreases when microwave heating time increases. When the heating time becomes longer and longer, the activating effect is better and ignition loss degree of activated carbon is larger. Similar yields have been reported in

Figure 8: Effect of microwave heating time on the activation yield of activated carbon.

literatures for other precursors, namely for crofton (18.03%) [33].

Research analysis

Table 3 compares the surface area of various activated carbons prepared using KOH activating agent utilizing different precursors as reported in the literature. Although it is a small sample, from the large number of activated carbon reported in the literature, the high surface area of the activated carbon prepared in the present work as compared with the literature is clearly evident. A surface as high as $3,441 \text{ m}^2/\text{g}$ is very rarely reported in the literature, which could form the basis for additional work on exploring the combination of bamboo precursor with the KOH activating agent.

SEM analysis

Figure 9 shows the SEM analysis of activated carbon. As can be seen from Figure 9, it is a picture of activated

Figure 9: SEM image of activated carbon.

carbon particle. The activated carbon attests a significant development of pore structure and the impurities on the surface of the activated carbon are less.

Conclusions

Effect of microwave heating time and microwave power to bamboo-based activated carbon was discussed. The optimal conditions were microwave heating time of 15 min and microwave power of 700 W with KOH/C ratio of 4. At the optimal conditions, surface area and pore volume were estimated to be $3,441 \text{ m}^2/\text{g}$ and 2.093 ml/g , respectively. The characterization of FTIR illustrated that surface of activated carbon had abundant functional groups. The yield of activated carbon decreased with microwave heating time and microwave power increased. SEM analysis shows that activated carbon has a clear pore structure.

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Table 3: Comparison surface area of activated carbon using KOH activating agent.

Activating agent	Precursors	Heating method	S_{BET} (m^2/g)	References
KOH	Bamboo	Microwave heating	3,441	Present study
	Coconut husks	Microwave heating	1,356	[14]
	Isotropic petroleum pitch	Conventional heating	2,992	[15]
	Rambutan	Microwave heating	971.54	[16]
	Anthracite	Microwave heating	3,451	[17]
	Rice husk	Conventional heating	2,696	[18]

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