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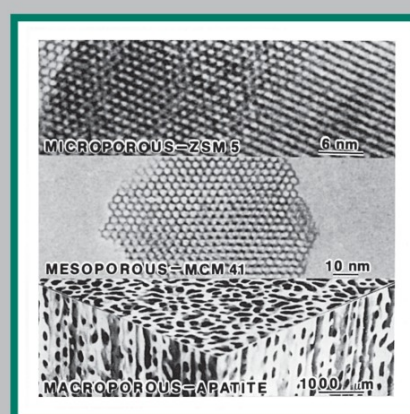
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Preparation and pore characterization of activated carbon from Ma bamboo (*Dendrocalamus latiflorus*) by H₃PO₄ chemical activation

Su-Ling Liu · Ya-Nan Wang · Kun-Tsung Lu

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Abstract High specific surface area activated carbon materials have been produced from the naturally occurring Ma bamboo (*Dendrocalamus latiflorus*) using phosphoric acid (H₃PO₄) as the activating agent. The effects of different sizes of raw materials, H₃PO₄ concentrations, and activation temperatures on the specific surface area, pore morphology, and mass yield of activated carbon are presented. A high specific surface area for activated carbon derived from Ma bamboo was achieved under the optimized conditions of 45 wt% H₃PO₄ impregnation concentration, activation temperature of 400 °C, and a holding time of 120 min. Chemical activation of Ma bamboo by H₃PO₄ is a useful technique for obtaining activated carbon with desired pore size distributions and pore morphologies from low cost precursors and at low activation temperatures.

Keywords Activated carbon · Ma bamboo · Phosphoric acid · Chemical activation · Pore characterization · Adsorption–desorption

1 Introduction

Activated carbon possesses a pore structure favorable for the formation of a larger specific surface area and high adsorption capacities; accordingly, the pore structure of activated carbon plays an important role on its adsorption properties [1]. Activated carbon can be prepared from biomass by pyrolysis, a very complex process, and is subject to the influence of many factors such as the type of raw materials, activated methods, pyrolysis temperature, the holding time, and heating rate [2–4]. In general, activated carbon can be prepared by either physical or chemical activation. In contrast to physical activation, which generates higher amounts of volatile matter remaining in the activated carbon at lower temperatures in comparison to high temperatures [5], chemical activation can, for instance, use dehydrogenation reactions, which inhibit tar formation and reduces the volatile substances generated. Therefore, the advantages of chemical activation include high yield, low activation temperature, short reaction time, and the development of a well-defined pore structure. Among the various chemical activations of biomass, the promotion of hydrolysis and dehydration by phosphoric acid as the chemical agent and its stimulation of cross-linking reactions give rise to an increase in the solid yield, and a decrease in pyrolytic tar yield [4]. Chemical activation of biomass materials by phosphoric acid is a useful technique for obtaining activated carbon materials with desired pore size distribution and surface morphology at low temperatures [6].

Bamboo is one of the most important forest resources, as it is a fast-growing plant, possesses a short cultivation span of about 3–6 years, and is an abundant source of biological materials. Ma bamboo (*Dendrocalamus latiflorus*), native to South China and Taiwan, is second in total yield among

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the six major species of bamboo found in Taiwan. However, except for collection of the edible bamboo shoots, Ma bamboo plants are not as widely used for furniture, construction, and handcrafts as other species; hence, the reduced utilization of Ma bamboo must be addressed to maximize its applications at present. In order to diversify the applications of Ma bamboo to increase and enhance its utilization to the greatest advantage, this study is mainly focused on the preparation of activated carbon from this raw material, using phosphoric acid as the activating agent, in order to obtain activated carbon with high specific surface area at reduced reaction temperatures (350–500 °C). Toward this end, the influence of various raw material sizes of Ma bamboo, phosphoric acid concentrations, activation temperatures, and holding times on pore structure of the activated carbon were investigated. It is anticipated that this examination of Ma bamboo-derived activated carbon will comprise a significant contribution to existing knowledge and applications of activated carbon with respect to adsorption of industrial pollutants such as metallic ions.

2 Materials and experimental methods

2.1 Materials

Ma bamboo specimens (*Dendrocalamus latiflorus* Munro) of 3–5 years growth were collected at Compartment 16 of the Suili Tract located in the National Taiwan University Experimental Forest College, Nanto, Taiwan. The density of Ma bamboo collected was 0.51. Bamboo culms were cut at about 15 cm above ground level. Concentrated phosphoric acid (H₃PO₄; 85 %; Sigma Aldrich) was used in experiments.

2.2 Experimental methods

2.2.1 Preparation of activated carbon

For preparation of activated carbon, Ma bamboo was first placed in an oven at 105 ± 5 °C for 24 h. The material was divided into three sizes, denoted as bamboo pieces (3 cm × 12 cm), shavings, and particle (20–40 mesh). Three different concentrations of H₃PO₄ were employed, i.e., 30, 45, and 60 wt%. The different sizes of dried Ma bamboo were impregnated with solutions of H₃PO₄. For the impregnation, 25 g of Ma bamboo was added to a 200 mL solution of H₃PO₄ and stirred at 85 °C, maintaining this temperature for 2 h to ensure the penetration of H₃PO₄ to the interior of the bamboo. Next, the samples were evaporated to dryness. All impregnated materials were then placed in a carbonization furnace (CB 56,

CHI-HOW Heating Co., Ltd.) under a nitrogen flow of 100 mL/min at a heating rate of 10 °C/min, activation temperatures were varied within the range of 350–500 °C at holding times of 30, 60, 90, and 120 min. After this time, the samples were cooled to room temperature under the same flow of nitrogen to obtain activated carbon prepared under the various conditions [7, 8]. The samples were washed with hot distilled water twice and then dried overnight in an oven at 105 ± 5 °C.

2.2.2 Measurement of pore morphology

Prior to gas adsorption measurements, the activated carbon was first degassed in a processor (BELPREP-vac II, BEL Japan, Inc.) at 300 °C under vacuum (1×10^{-2} kPa) for 3 h.

After completion of pre-treatment, pore texture measurements of the prepared activated carbon samples were carried out in a porosity analyzer (BELSORP-mini 2, BEL Japan, Inc.) with nitrogen (99.9999 % purity) at a temperature of 77 K for adsorption–desorption experiments. According to the data, the specific surface area (S_{BET}), total pore volume (V_T) and mean pore diameter (D_p) of activated carbon were calculated by the Brunauer–Emmett–Teller (BET) method. The micropore surface area (S_{mi}) and micropore volume (V_{mi}), as well as mesopore surface area (S_{me}) and mesopore volume (V_{me}) of activated carbon were determined by the t-plot and Barrett–Joyner–Halenda (BJH) methods, respectively, according to the BELSORP analysis program software.

All characterizations of specific surface area, pore volume and pore size distribution of the resulting activated carbon samples from N₂ adsorption–desorption measurements were performed in at least triplicate. The Microsoft Excel[®] software package was employed to estimate the mean and standard deviation statistical parameters of the collected data [9].

3 Results and discussion

3.1 Effects of raw material sizes on porosity development

Phosphoric acid activation of lignocellulosic materials generally involves two processes: impregnation and heat treatment. Impregnation forms a phosphoric acid and lignocellulosic macromolecule composite; the properties of this composite significantly affect the properties of the activated carbon [10–12]. Therefore, materials of different sizes impregnated by an activating agent exhibit differences in pore development during carbonization. Figure 1 shows the N₂ adsorption–desorption isotherm curves of

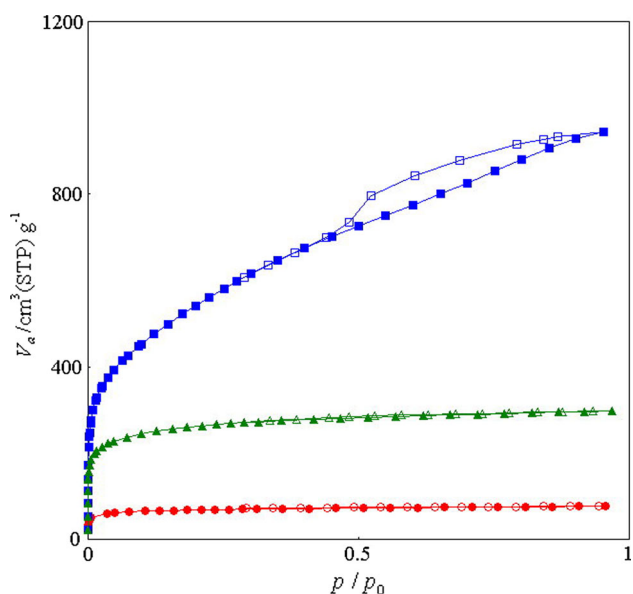


Fig. 1 N_2 adsorption–desorption isotherm curves of Ma bamboo activated carbon for different raw material sizes (filled and open circle: bamboo pieces; filled and open triangle: shavings; filled and open square: particle (20–40 mesh); filled symbols represent adsorption curve; hollow symbols represent desorption curve; activation conditions: 400 °C, 30 wt% H_3PO_4 and 120 min holding time)

activated carbon derived from Ma bamboo for the various raw material sizes examined. According to the International Union of Pure and Applied Chemistry (IUPAC) classifications, a Type I isotherm is associated with microporous structures, and a Type IV isotherm indicates a mixture of microporous and mesoporous materials. The results demonstrated that the pores found in both bamboo pieces and shavings were filled completely and attended to the adsorption amount of saturation, as in the case of mainly microporous materials at high relative pressures. These results indicate a Langmuir monolayer adsorption and Type I isotherm, meaning the materials possess a micropore pattern. However, the isotherm curve of bamboo particle incorporated both a broader “knee” (point of flexion) and more obvious hysteresis loop. The broad knee generally results from the co-existence of both micropores and mesopores in large amounts; the hysteresis loop is typically brought about by capillary condensation in the mesopores of activated carbon materials [13, 14].

The yields and pore characteristics of activated carbon prepared from Ma bamboo of different raw material sizes at an activation temperature of 400 °C, 30 wt% H_3PO_4 concentration, and holding time of 120 min are shown in Table 1. The yields of activated carbon derived from particles (20–40 mesh), shavings, and bamboo pieces were 9.14, 29.47, and 34.04 %, respectively. As can be seen, the specific surface area (S_{BET}), micropore surface area (S_{mi}) and mesopore surface area (S_{me}) were all observed to

decrease with an increase in bamboo size. The phosphoric acid was incorporated into the Ma bamboo cell wall interior, forming a phosphoric acid–biopolymer composite that could eventually be converted into a solid product with a highly developed pore structure through carbonization [12, 15–17]. The agent would have greater difficulty penetrating the cell walls of the larger bamboo specimens, resulting in inadequate material activation. Although the activated carbon prepared from particle-sized Ma bamboo exhibited the highest S_{BET} , S_{mi} , and S_{me} of pore structure, its activated carbon yield of 9.14 % was the lowest among the three different raw material sizes under the same activation conditions. Therefore, considering the balance between pore structure and yield, bamboo shavings were identified as the superior material for activated carbon preparation, and were chosen for subsequent experiments.

3.2 Effects of phosphoric acid concentrations on porosity development

The effect of phosphoric acid concentration on porosity development was examined by subjecting raw material shavings samples to concentrations of 30, 45, and 60 wt% H_3PO_4 during preparation, at an activation temperature of 400 °C, heating rate of 10 °C/min and holding time of 120 min.

As shown in Fig. 2, the N_2 adsorption–desorption isotherm curve for activated carbon prepared using 30 wt% H_3PO_4 was associated with a Type I adsorption isotherm. In contrast, a Type IV isotherm was observed for materials prepared with 45 and 60 wt% H_3PO_4 ; however, the hysteresis loop of the sample using 45 wt% H_3PO_4 was broader than that of 60 wt%, indicative of a higher mesopore surface area (S_{me}) and mesopore volume (V_{me}). The development of porosity is related to the formation of phosphate linkages such as phosphate and polyphosphate esters, which can serve to connect and crosslink biopolymers. These linkages expand the structure of the bamboo, resulting in the retention of phosphate compounds in the expanded structure, which act to hinder material shrinkage during high-temperature treatment [16, 18].

The yields and pore characteristics of Ma bamboo activated carbon materials prepared with different phosphoric acid concentrations are listed in Table 2. The yields of activated carbon treated with 30, 45, and 60 wt% H_3PO_4 were 29.47, 28.03, and 22.09 %, respectively. The yield was observed to decrease with an increase in phosphoric acid concentrations. The values for the specific surface area (S_{BET}), total pore volume (V_T), micropore surface area (S_{mi}), and micropore volume (V_{mi}) varied with the phosphoric acid concentration as follows: 45 > 60 > 30 wt%. The phosphoric acid introduced within the material plays a double role. Firstly, it induces hydrolysis of the

Table 1 Yields and pore characteristics of Ma bamboo activated carbon for different raw material sizes

Size ^a	Yield (%)	S_{BET} (m ² /g)	V_T (cm ³ /g)	D_P (nm)	S_{mi} (m ² /g)	V_{mi} (cm ³ /g)	S_{me} (m ² /g)	V_{me} (cm ³ /g)
PM	9.14	1,994 ± 12	1.46 ± 0.01	2.93 ± 0.05	1,863 ± 12	1.35 ± 0.01	1,078 ± 24	1.02 ± 0.02
SV	29.47	912 ± 4	0.46 ± 0.00	2.02 ± 0.00	603 ± 17	0.45 ± 0.00	130 ± 1	0.11 ± 0.00
BP	34.04	233 ± 5	0.12 ± 0.00	2.06 ± 0.01	239 ± 1	0.11 ± 0.00	27 ± 0	0.02 ± 0.00

^a Activation conditions: 30 wt% H₃PO₄; 400 °C; heating rate, 10 °C/min; holding time, 120 min

^b PM particle (20–40 mesh), SV shavings, BP bamboo pieces

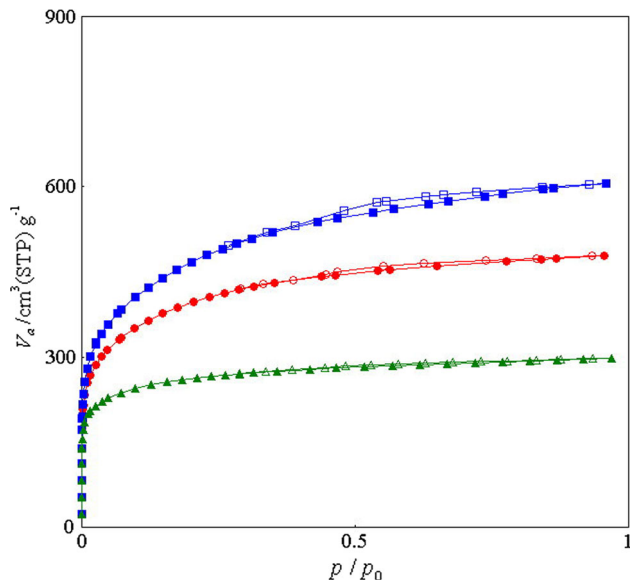


Fig. 2 N₂ adsorption–desorption isotherm curves of activated carbon from Ma bamboo shavings for different H₃PO₄ concentrations at an activation temperature of 400 °C (▲△: 30 wt%; ■□: 45 wt%; ●○: 60 wt%; filled symbols represent adsorption curve; hollow symbols represent desorption curve; activation conditions: 400 °C, 120 min holding time)

lignocellulosic material, thus weakening bonding within the structure and favoring swelling of the biomaterial. This effect was more evident in samples treated with 45 wt% H₃PO₄, thus favoring the development of porosity when compared to those samples treated with 30 and 60 wt% H₃PO₄. Secondly, the phosphoric acid occupies a certain volume, which inhibits the contraction of the structure during the heat treatment, thus leaving behind a porous structure when it is extracted by washing after carbonization [19]; an increase in concentration to 60 wt% was accompanied by a decrease in development of the porous structure. In connection with this interpretation of the results, it is relevant to note that a characteristic of H₃PO₄ is its high viscosity at high solution concentrations, which may cause pore blockage. It may be deduced from the values of V_{mi} and V_{me} , that this activated carbon prepared using 45 wt% H₃PO₄ showed an acceptable microporosity and mesoporosity. The impregnation of 45 wt% H₃PO₄ into

the lignocellulosic structure of Ma bamboo shavings appears to be the cause of micropore creation. In sum, a superior pore structure was obtained with 45 wt% H₃PO₄; this concentration was determined as optimal in subsequent experimental conditions.

3.3 Effect of activation temperature on porosity development

The specific surface area of activated carbon depends greatly on the activation temperature. An increase in activation temperature is accompanied by an increase in the reaction rate between the H₃PO₄ agent and carbonized materials. In this study, the progression of porosity development of bamboo shavings impregnated with 45 wt% H₃PO₄ at activation temperatures of 350, 400, 450, and 500 °C was investigated.

For insuring the bio-materials were transferred to carbon material after chemical activation at such lower temperature. The weight percentages of carbon, hydrogen, and oxygen were measured by elemental analysis using Elementer vario EL III CHN-OS Rapid F002 equipment. The results are shown in Table 3 and indicated that the contents of carbon, oxygen and hydrogen of activated carbon with different temperatures were 74–83, 14–20 and 3–6 %, respectively. The contents of carbon increased gradually with increasing activation temperatures. The results also showed that the organic bamboo had already become to a carbon material after chemical activation even at such lower temperature of 350 °C. AL-Othman et al. used peanut shell as a raw material and obtained contents of carbon was 80.16 %, oxygen was 22.11 % and hydrogen was 1.76 %, when the activation temperature at 450 °C [20]. Gundogdu et al. [21] obtained contents of carbon, oxygen and hydrogen were 79.32, 15.21 and 1.79 %, respectively, which used tea industry waste as a raw material and the activation temperature at 700 °C. The results of the elemental analyses also indicated that carbon, oxygen, and hydrogen accounted for over 99 % of sample weight for different activation temperatures, confirming the effectiveness of the purification [22].

The N₂ adsorption–desorption isotherm curves of the activated carbon produced from Ma bamboo at varying

Table 2 Yields and pore characteristics of activated carbon from Ma bamboo shavings for different H₃PO₄ concentrations

Conc. (wt%)	Yield (%)	S_{BET} (m ² /g)	V_T (cm ³ /g)	D_p (nm)	S_{mi} (m ² /g)	V_{mi} (cm ³ /g)	S_{me} (m ² /g)	V_{me} (cm ³ /g)
30	29.47	912 ± 4	0.46 ± 0.00	2.02 ± 0.00	603 ± 17	0.45 ± 0.00	130 ± 1	0.11 ± 0.00
45	28.03	1,692 ± 9	0.94 ± 0.01	2.21 ± 0.01	1,464 ± 14	0.91 ± 0.01	518 ± 15	0.42 ± 0.01
60	22.09	1,428 ± 6	0.74 ± 0.00	2.07 ± 0.01	1,167 ± 12	0.72 ± 0.01	335 ± 5	0.26 ± 0.01

Activation conditions: 400 °C; heating rate, 10 °C/min; holding time, 120 min

Table 3 Elemental analyses of activated carbon from Ma bamboo shavings for different activation temperatures

Temp. (°C)	Elemental composition (wt%)		
	C	O	H
350	74.22 ± 0.86	19.99 ± 1.41	5.38 ± 0.76
400	79.44 ± 0.09	16.51 ± 0.29	3.69 ± 0.24
450	81.46 ± 0.36	14.54 ± 0.26	3.72 ± 0.22
500	82.16 ± 1.56	14.41 ± 1.65	3.20 ± 0.07

Activation conditions: 45 wt% H₃PO₄; heating rate, 10 °C/min; holding time, 120 min

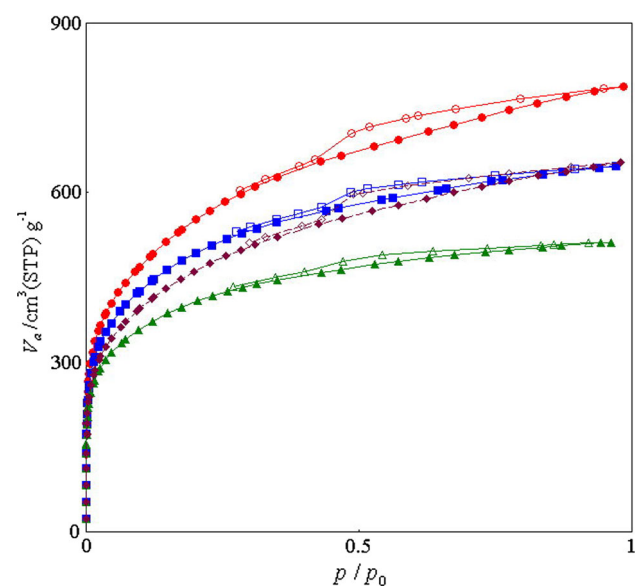


Fig. 3 N₂ adsorption–desorption isotherm curves of activated carbon from Ma bamboo shavings for different activation temperatures (▲△: 350 °C; ●○: 400 °C; ■□: 450 °C; ◆◇: 500 °C; filled symbols represent adsorption curve; hollow symbols represent desorption curve; activation conditions: 45 wt% H₃PO₄, 120 min holding time)

carbonization temperatures are shown in Fig. 3. The adsorption–desorption isotherms of activated carbon at carbonization temperatures of 350–500 °C corresponded to a Type IV isotherm, indicating that they were composed of a mixture of microporous and mesoporous materials. The plateau of this isotherm commenced at high relative pressures (P/P_0); toward the end of the isotherm, a steep gradient was observed as a result of limited uptake of nitrogen,

indicating capillary condensation in the mesopores. This characteristic indicates the development of micro- and mesoporous structures on the char during the carbonization process [23]. The hysteresis loop was brought about by capillary condensation in the mesopores of activated carbon, thus, the maximum volume of mesopores was observed at 400 °C. Increasing the activation temperature to 450 °C decreased the micropores and mesopores, an observation that is likely due to the thermal degradation of pore walls caused by collapsing micropores as well as mesopore burn-off.

The yield, specific surface areas, and pore volumes of activated carbon prepared at different activation temperatures are summarized in Table 4. The yields of activated carbon subjected to activation temperatures of 350, 400, 450, and 500 °C were 32.16, 28.03, 26.75, and 24.05 %, respectively. It was found that increasing the activation temperature decreased the yield of activated carbon, likely due to the loss of a large portion of volatile matter and phosphate-containing compounds through volatilization of carbon and protective phosphate groups [18]. The specific surface areas and pore volumes of the activated carbon samples increased gradually with increasing activation temperatures over the range 350–400 °C, and then decreased with further increases in activation temperature over 450–500 °C. At an activation temperature of 350 °C, the extent of activation of the carbonized Ma bamboo material was relatively low, which was demonstrated by smaller surface areas and pore volumes for both micropores and mesopores. It is thought that an increase in temperature to 450 and 500 °C results in violent gasification reactions that might cause part of the pore structure to collapse; this destruction of pore structure contributes to the reduction in surface area [14]. From Table 4, it can be seen that the pore volume decreased upon an increase in temperature to 450 °C, which could be deduced from the collapsing of micropores. This result was in accordance with the higher activation temperature for the larger pore diameter (D_p). From the results mentioned above, the optimum temperature for activation was determined as 400 °C.

In others study, Ip et al. used bamboo scaffolding waste as a raw material and obtained the highest specific surface area of 2,123 m²/g at the conditions of using the 85 wt% H₃PO₄ concentration, the activation temperature of 600 °C

Table 4 Yields and pore characteristics of activated carbon from Ma bamboo shavings for different activation temperatures

Temp. (°C)	Yield (%)	S_{BET} (m ² /g)	V_T (cm ³ /g)	D_P (nm)	S_{mi} (m ² /g)	V_{mi} (cm ³ /g)	S_{me} (m ² /g)	V_{me} (cm ³ /g)
350	32.16	1,614 ± 9	0.84 ± 0.01	2.08 ± 0.01	1,391 ± 6	0.81 ± 0.00	341 ± 7	0.28 ± 0.01
400	28.03	2,190 ± 6	1.24 ± 0.02	2.27 ± 0.03	1,932 ± 16	1.15 ± 0.12	796 ± 3	0.63 ± 0.02
450	26.75	1,797 ± 9	1.05 ± 0.02	2.35 ± 0.05	1,539 ± 22	1.00 ± 0.01	655 ± 12	0.55 ± 0.03
500	24.05	1,637 ± 6	1.04 ± 0.04	2.54 ± 0.09	1,331 ± 57	0.89 ± 0.10	701 ± 5	0.61 ± 0.04

Activation conditions: 45 wt% H₃PO₄; heating rate, 10 °C/min; holding time, 120 min

with a heating rate of 1 °C/min and the acid to bamboo ratio of 2 [18]. However, in the study the phosphoric acid possessed a higher viscosity of its concentrated solutions and hindered the pore structure development during pyrolysis process [7], therefore it need a higher activation temperature of 600 °C to obtain the highest specific surface area of 2,123 m²/g. In our study, the similar result of specific surface area of 2,190 m²/g was observed only with a lower concentration of 45 wt% H₃PO₄ and at a lower activation temperature of 400 °C. From the result, we can summarize that using a suitable concentration of H₃PO₄ solution treatment the highest specific surface area of bamboo made activation carbon could be obtained even at a lower temperature.

Wang had prepared activation carbon by waste bamboo culms soaked in a phosphoric acid solution at 253 K (−20 °C) for 2 days with the assistance of ultrasonication and carbonized at 973 K (700 °C). The specific surface area of the obtained activation carbon was 747 m²/g [24]. It was quite lower than that of our study of 2,190 m²/g. The bio-materials soaked with phosphoric acid which can catalyze the primary pyrolysis reactions of biopolymers, and the impregnated temperature of a high viscosity of phosphoric acid will affect the soaked amount in the interior cell walls of raw materials and furthermore affects the pore structure development of activation carbon. In our study, the bamboo was dipped in 45 wt% H₃PO₄ and stirred at 85 °C for 2 h, which ensured the penetration of H₃PO₄ to the interior of the bamboo cell wall very well and the highest specific surface area of 2,190 m²/g obtained at low activation temperature of 400 °C.

It was noted that the highest S_{BET} of activated carbon was 2,190 m²/g, which, remarkably, was prepared at a lower activation temperature at 400 °C. This provided further indication that chemical activation by H₃PO₄ is a useful technique for obtaining activated carbon materials with desired pore size distribution and pore characteristics at a low activation temperature.

3.4 Effect of holding time on porosity development

The aim of activation is to remove all kinds of volatiles produced from the raw material during thermal degradation

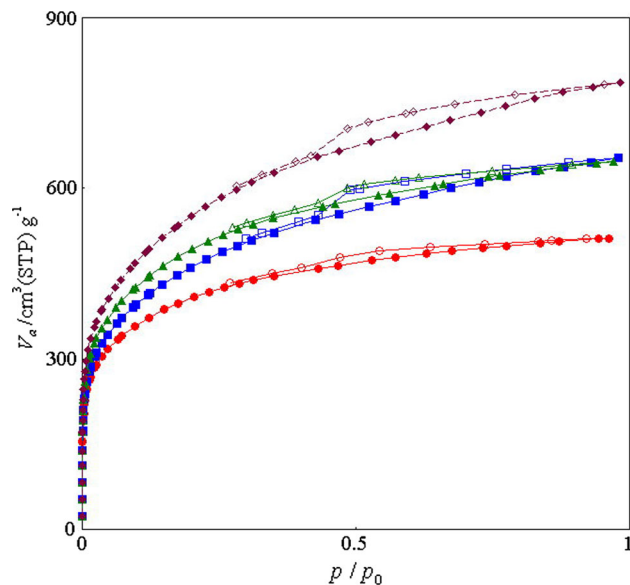


Fig. 4 N₂ adsorption–desorption isotherm curves of activated carbon from Ma bamboo shavings for different holding times (●○: 30 min; ■□: 60 min; ▲△: 90 min; ◆◇: 120 min; filled symbols represent adsorption curve; hollow symbols represent desorption curve; activation conditions: 400 °C, 45 wt% H₃PO₄, 120 min holding time)

in order to obtain a suitable activated carbon material possessing a specific C/H ratio. The reactions occurring during the activation process are rather complicated such as the reaction of cellulose and lignin, which results in the removal of C, H, and O elements and formation of char with cyclic and aromatic structures. Therefore, the structure of the activated carbon obtained under different holding times is also related to the specific surface area and pore characteristics of activated carbon. Interestingly, no obvious differences were observed in the N₂ adsorption–desorption isotherm curves of activated carbon for different holding times (as shown in Fig. 4); all curves were well matched to a Type IV isotherm, i.e., they possessed micropores and mesopores simultaneously.

The yields and pore characteristics of Ma bamboo activated carbon at different holding times are shown in Table 5. Under the same reaction conditions (45 wt% H₃PO₄, 400 °C), the yields at holding times of 30, 60, 90, and 120 min were 31.13, 30.52, 30.12, and 23.38 %, and

Table 5 Yields and pore characteristics of activated carbon from Ma bamboo shavings at different holding times

Holding times (min)	Yield (%)	S_{BET} (m ² /g)	V_T (cm ³ /g)	D_P (nm)	S_{mi} (m ² /g)	V_{mi} (cm ³ /g)	S_{me} (m ² /g)	V_{me} (cm ³ /g)
30	31.13	1,532 ± 5	0.83 ± 0.00	2.17 ± 0.01	1,332 ± 11	0.80 ± 0.00	410 ± 2	0.34 ± 0.00
60	30.52	1,748 ± 4	1.04 ± 0.00	2.39 ± 0.00	1,510 ± 3	0.99 ± 0.00	638 ± 2	0.56 ± 0.00
90	30.12	1,848 ± 3	1.03 ± 0.00	2.22 ± 0.00	1,646 ± 12	0.98 ± 0.00	565 ± 2	0.47 ± 0.00
120	23.38	2,032 ± 7	1.22 ± 0.00	2.40 ± 0.01	1,797 ± 7	1.15 ± 0.00	757 ± 1	0.65 ± 0.00

Activation conditions: 400 °C; 45 wt% H₃PO₄; heating rate, 10 °C/min

the corresponding specific surface areas of the activated carbon were 1,532, 1,748, 1,848, and 2,032 m²/g, respectively. The results indicated that an increased holding time is advantageous to pore structure development. The activated carbon obtained at a holding time of 120 min had higher S_{BET} , V_{mi} , and V_{me} ; however, the pyrolysis was more time-consuming. Although the activated carbon yielded at a holding time of 30 min had relatively inferior pore structure development, it was obtained in the highest yield, and its S_{BET} value was still comparable with that of commercially available activated carbon ($S_{BET} > 1,000$ m²/g).

Liu et al. [25] had prepared activated carbon from bamboo waste handicrafts, which performed at the conditions of heating rate 10 °C/min, phosphoric acid/carbon ratio 1:1, activation temperature at 600 °C and activation time were 30 and 60 min. The specific surface areas of the activated carbon were 1,215 and 1,416 m²/g, respectively. The results of our study, four different holding times from 30 to 120 min (45 wt% H₃PO₄, 400 °C and heating rate 10 °C/min) and the specific surface area increased from 1,532 to 2,032 m²/g, which was in accordance with the Liu et al. study. However, in our study, using a lower activation temperature had the similar results, indicating it is energy saving and favor activated carbon making.

4 Conclusions

An examination of activation conditions for the preparation of activated carbon derived from Ma bamboo was performed. Penetration of the activating agent (H₃PO₄) into the cell walls of Ma bamboo was difficult for larger raw material sizes; bamboo shavings were found to be a suitable size, which maintained a balance between mass yield and desired pore characteristics of the obtained activated carbon. A phosphoric acid concentration of 45 wt% was effective in promoting the development of lignocellulosic material porosity by complete penetration of the internal cell wall; activated carbon prepared with 45 wt% H₃PO₄ exhibited the highest specific surface area of 1,692 m²/g and possessed ideal pore characteristics. An increase to 60 wt% resulted in pore blockage and a decrease in pore structure due to the higher viscosity of the reagent. Among

the different activation temperatures investigated, the highest specific surface area and best porosity characteristics were observed at 400 °C. In contrast to known physical activation methods, the chemical activation process described here can be conducted at lower temperatures to obtain activated carbon with desirable pore morphologies. In addition, pore development was assisted by an increase in holding time. Notably, even at a reduced holding time of 30 min, the specific surface area obtained was still comparable with that of commercial activated carbon.

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