

PRODUCTION OF ACTIVATED CARBON FROM INDUSTRIAL BAMBOO WASTES

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MAHANIM SMA, WAN ASMA I, RAFIDAH J, PUAD E & SHAHARUDDIN H. 2011. Production of activated carbon from industrial bamboo wastes. Activated carbon was produced from bamboo residues using carbonisation and steam activation in a high temperature reactor. Tests were carried out to study the effects of two processing parameters, namely, activation time (30, 60, 90, 120 and 150 min) and activation temperature (650, 700, 750 and 800 °C) on the properties of activated carbon produced. Activated carbon properties were analysed based on iodine number and Brunauer-Emmett Teller (BET) surface area. Increasing activation temperature gave higher iodine values. Optimum conditions for producing activated carbon from bamboo wastes were at activation temperature 800 °C and activation time 120 min that resulted in bamboo activated carbon with the highest iodine number of 823 mg g⁻¹ and BET surface area of 719 m² g⁻¹. With these parameters, waste bamboo has the potential to be a promising precursor for the production of activated carbon. The characteristics of the activated carbon in this study was comparable with commercial ones.

Keywords: Activation time, activation temperature, iodine number, carbonisation, steam activation

MAHANIM SMA, WAN ASMA I, RAFIDAH J, PUAD E & SHAHARUDDIN H. 2011. Penghasilan karbon teraktif daripada sisa perindustrian buluh. Karbon teraktif dihasilkan daripada sisa buluh menggunakan karbonisasi dan pengaktifan secara stim di dalam reaktor bersuhu tinggi. Kajian dijalankan untuk menyelidiki kesan dua parameter pemrosesan iaitu masa pengaktifan (30 min, 60 min, 90 min, 120 min dan 150 min) dan suhu pengaktifan (650 °C, 700 °C, 750 °C dan 800 °C) terhadap ciri-ciri karbon teraktif yang dihasilkan. Ciri-ciri karbon teraktif dianalisis berdasarkan nombor iodin dan luas permukaan Brunauer-Emmett Teller (BET). Suhu pengaktifan yang meningkat memberikan nilai iodin yang tinggi. Keadaan optimum untuk menghasilkan karbon teraktif daripada sisa buluh adalah pada suhu pengaktifan 800°C dan masa pengaktifan 120 min yang menghasilkan nilai iodin tertinggi 823 mg g⁻¹ dan luas permukaan BET 719 m² g⁻¹. Sisa buluh mempunyai potensi untuk menjadi pelopor bagi penghasilan karbon teraktif. Ciri karbon teraktif dalam kajian ini adalah setanding dengan karbon teraktif komersial.

INTRODUCTION

Activated carbon is an important component of filter material for the removal of hazardous components in exhaust gases for the purification of drinking water and for waste water treatment. The demand for activated carbon will continue to rise due to its wide range of use as a result of environmental compliances in many countries (Elisabeth et al. 2007).

Biomass has become one of the main sources of carbon for the production of activated carbon. The biomass used is usually waste materials or by-products in commercial activities. By reusing or recycling these low cost materials to produce activated carbon, we are providing another environmentally friendly alternative to dispose of the waste and by-product (Cheong 2006).

Researchers have studied the production of activated carbon from various types of biomass such as palm-tree cob, plum kernel, cassava peel, bagasse, jute fibre, rice husk, olive stone, date pit, fruit stones and nutshell. In the present work, a by-product from the bamboo industry was used as the precursor. Bamboo is a natural resource in Malaysia and has been traditionally used to construct various living facilities and tools. Bamboo has been used as structural material at construction sites in China, India, Malaysia and other countries because it is strong, tough and cheap (Hameed et al. 2007).

Bamboo can be converted into charcoal and activated carbon via carbonisation followed by activation (Keith et al. 2005, Young et al. 2005,

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Chan et al. 2006, Hameed et al. 2007, Edward et al. 2010). As landfill or open-dumping is no longer economically or legally feasible, thermal processing (example carbonisation) of bamboo is considered as one of the possible solutions since it does not only reduce the quantity of waste in landfill but also converts these materials to an adsorbent for wastewater treatment or other applications (Hameed et al. 2007, Edward et al. 2010).

The carbonisation process is to enrich the carbon content and create an initial porosity, and the activation process helps in enhancing the pore structure. Carbonisation takes place in the temperature range of 300–400 °C. During carbonisation, primary carbonisation gases are produced which can be categorised as permanent gases and oils (tars) if they are cooled to ambient temperature. The residue of the carbonisation process is the primary charcoal which serves as base material for the activation step (Elisabeth et al. 2007).

The charcoal is then activated by steam in the same reactor at 650–800 °C. Some of the carbon is oxidised which leads to the generation of pores. It is very important at this step to keep the activation conditions constant. Steam activation at higher temperatures gave better activation and enhanced widening of the narrow pore network (Prakash Kumar et al. 2006).

The objective of this study was to obtain data on surface and adsorption properties of activated carbon prepared from bamboo residues. The present work also studied the influence of various process parameters such as activation time and activation temperature on activated carbon.

MATERIALS AND METHODS

The precursor material was bamboo waste from a chopstick factory in Felda Mempaga 3, Bentong, Malaysia. Experiments were conducted in a high temperature reactor with dimensions of 2.1 × 9.5 × 1.2 m (length × width × height). The reactor could produce 400 g of activated carbon/day from 3 kg of raw materials.

Carbonisation and activation processes

For the carbonisation and activation processes, bamboo wastes were cut and air dried to moisture contents of 13–15%. Three kg of dried samples were taken for each run and fed into the reactor after which the vessel was heated to temperature

between 300 and 400 °C in the absence of air. The carbonisation reaction was completed when the emitted gas turned bluish in colour. The charcoal prepared from the carbonisation process was activated using steam in the same reactor at different activation times (30, 60, 90, 120 and 150 min) and temperatures (650, 700, 750 and 800 °C).

Characterisation

Composition of ethanol–toluene extraction, holocellulose, α-cellulose and lignin content

The internodes of bamboos at different portions (basal, medium, top) were cut into small strips, ground into 250 μm size and used for chemical analysis. The resulting material was placed in sealed plastic container and labelled for chemical analysis. The chemical analysis was conducted using the test methods in Table 1.

Proximate and ultimate analysis

Proximate analysis (moisture content, ash content, volatile matter and fixed carbon) of bamboo, bamboo charcoal and bamboo activated carbon were carried out using MS 873:1984 (MS 2004). For ultimate analysis, nitrogen content was determined using nitrogen analyser and the determination of elements was carried out using inductive couple plasma spectrometer.

Thermogravimetric analysis

Thermal decomposition was observed in terms of global mass loss using thermal gravimetric analyser. This apparatus detects mass loss as the specimen is heated. The samples were evenly and loosely distributed in an open sample pan of 6.6 mm diameter and 4.1 mm deep with initial sample amounts of 15–20 mg. The temperature change was controlled from room temperature to 900 °C at a heating rate of

Table 1 Test methods

Component	Reference
Holocellulose	Wise et al. (1946)
Alpha-cellulose	TAPPI (2009)
Lignin	TAPPI (2002)
Ethanol–toluene	TAPPI (1997)

20 °C min⁻¹. Oxygen free nitrogen gas of 99.995% purity was continuously passed into the furnace at flow rate of 100 ml min⁻¹ at room temperature and atmospheric pressure.

Iodine number determination

Iodine number is a relative indicator of porosity in an activated carbon sample. The iodine value (mg of iodine adsorbed/g of carbon) of the activated carbon produced was determined according to ASTM 4607-86 (ASTM 2011) and MS 873:1984 (MS 2004). Analysis was carried out by mixing 1–1.6 g of 212 µm powdered sample with 10 ml HCl and 100 ml 0.1 N iodine solutions, shaken occasionally in a 250 ml Erlenmeyer flask (MS 2004). The solution was filtered and titrated against standard 0.1 N Na₂S₂O₃ solution to a colourless end point.

Burn-off weight percentage

The burn-off weight percentage measures the degree of activation process. It is defined as the ratio of percentage weight decrease of the material during preparation to the original weight of the raw material. It is mathematically expressed as

$$\text{Percentage burn-off} = \frac{W_i - W_f}{W_i} \times 100$$

where W_i is the initial weight of the raw material and W_f is the final weight of product.

BET surface analysis

The active surfaces of the charcoal and activated carbon were calculated from N₂ adsorption isotherms using Brunauer-Emmett Teller (BET) surface analyser. The nitrogen adsorption–desorption data were recorded at liquid nitrogen temperature 77 K. Prior to measurements, samples were outgassed at 573 K under nitrogen

flow for at least two hours. About 0.5 g of sample was used in each adsorption experiment. Nitrogen adsorption isotherm was measured over relative pressure range from approximately 10⁻² to 1.

RESULTS AND DISCUSSION

Chemical composition of bamboo waste

There were variations in the compositions of ethanol–toluene extraction, holocellulose, α-cellulose and lignin from base to top of the bamboo (Table 2). The amounts of ethanol–toluene extraction, holocellulose, α-cellulose increased from base to top. However, the amount of lignin decreased from base to top.

The ethanol–toluene extractives of bamboo consisted of soluble materials not generally considered part of the bamboo substance, i.e. waxes, fats, resins, gums and some water-soluble substances. Ethanol–toluene extractive contents of bamboo increased from the base to the top (3.74–4.45%).

Holocellulose includes α-cellulose and hemicellulose. Alpha-cellulose is the main constituent of bamboo. Approximately 40–55% of the dry substance in bamboo is α-cellulose. Cellulose is a homopolysaccharide composed of β-D-glucopyranose units which are linked together by (1–4)-glycosidic bonds. Cellulose molecules are completely linear and have strong tendency to form intra- and intermolecular hydrogen bonds. Bundles of cellulose molecules are thus aggregated in the form of microfibrils, in which crystalline regions alternate with amorphous regions. Hemicelluloses are heterogeneous polysaccharides. Like cellulose, most hemicelluloses function as supporting materials in the cell walls.

Height had a significant effect on the holocellulose content. The top portion had the highest holocellulose content while the

Table 2 Chemical composition of bamboo

Bamboo	Alcohol–toluene extraction (%)	Holocellulose (%)	Alpha-cellulose (%)	Lignin (%)
Base	3.74	63.04	46.14	16.12
Middle	4.09	77.84	46.38	13.71
Top	4.45	79.64	46.53	12.48

bottom portion, the lowest. There was significant difference in α -cellulose content along the height of the culm. It increased gradually from the bottom to the top. Holocellulose content increased from the base to the top (63.04–79.64%). Alpha-cellulose content also increased from the base to the top (46.14–46.53%).

The lignin content decreased from the base to the top (16.12–12.48%). Lignin is a polymer of phenylpropane units. Lignin can be isolated from extractive-free wood as an insoluble residue after hydrolytic removal of the polysaccharides. Lignin is obtained after removing the polysaccharides from extracted (resin free) wood by hydrolysis with 72% sulphuric acid. Bamboo lignin is made from three phenyl-propane units, p-coumaryl, coniferyl and sinapyl alcohols interconnected through biosynthetic pathways. The lignin present in bamboo is unique. The lignification process undergoes changes during elongation of the culm. The full lignification of the bamboo culm is completed within one growing season with no further ageing effect (Li 2004).

Proximate and ultimate analyses

Table 3 shows that the fixed carbon content for bamboo is 11.32%. The bamboo sample was rich in volatiles (70%) but low in ash content (2%). The low ash content would result in minimal effects of inorganic impurities on pore development during activation process.

Previous studies suggested that suitable activated carbon should be low in ash but rich in carbon and volatiles. The ash consists of mainly minerals such as silica, alumina, iron, magnesium and calcium. Ash in activated carbon is undesirable and taken as impurity. Ash may interfere with carbon adsorption through competitive adsorption and catalysis of adverse reactions. Usually materials with the lowest ash content produce the most active products.

Fixed carbon content in samples changes when exposed to high temperatures during carbonisation and activation. The fixed carbon content increased steadily while volatiles declined with temperature. At higher temperatures, organic substances become unstable as the heat provides energy to the molecules to break their bonding and linkages and once these are broken, the substances will be discharged both as gas and liquid products. Similar trend was reported

by Azam (2005) in the preparation of activated carbon from biomass.

Ultimate analysis gives the elemental content of activated carbon such as carbon, nitrogen and sulphur. The analysis is very useful to investigate the purity of total carbon in activated carbon. The carbon content in activated carbon was more than 60%. Some can be as high as 90% (Azam 2005).

Thermogravimetric analysis

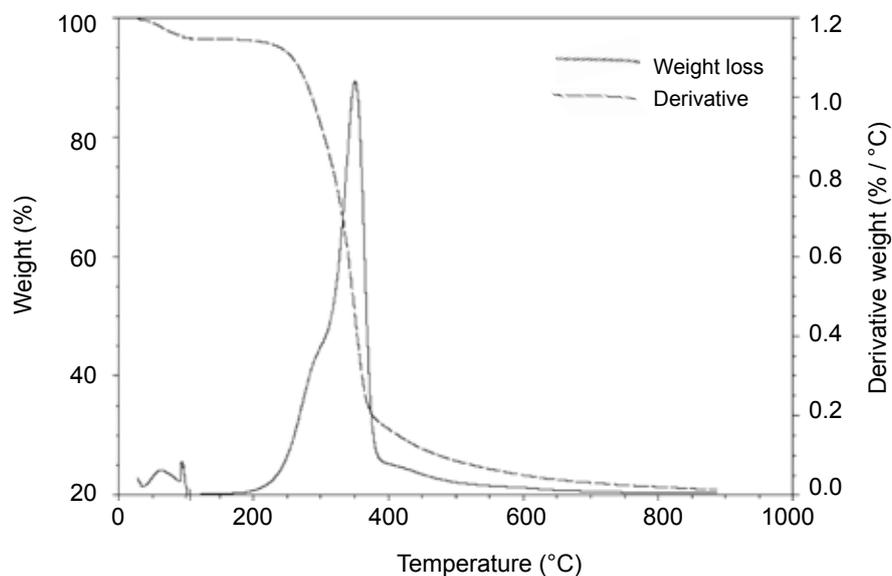
Figure 1 shows the measured thermogravimetric (TG) and derivative thermogravimetric (DTG) curves (obtained by calculation of the first derivative from the TG lines) plotted for raw bamboo. The DTG curve shows the two thermal decomposition steps.

The first TG curve corresponded to the desorption of water under temperature range of 30–100 °C. The second curve covered temperature range of 250–400 °C and consisted of two peaks: weak at 290 °C and strong at 350 °C, corresponding to weight losses of 10 and 45% respectively. Keith et al. (2005) observed only one peak at 350 °C for common bamboo. From this study, hemicellulose started to decompose at the temperature range of 250–320 °C with maximum derivative weight of 0.35% at 280 °C. However, after the temperature had been raised to 900 °C, the solid residue left was 21%. Cellulose pyrolysis took place at a higher temperature range, i.e. 320–380 °C with maximum derivative weight of 1.05% at 350 °C. At temperatures higher than 380 °C, almost all cellulose was pyrolysed. Lignin was reported to be the most difficult component to decompose. Lignin decomposed slowly throughout the whole temperature range from ambient to 900 °C.

The thermogram of raw bamboo is almost the same as those determined for woods (Yang et al. 2007, Krzesinska et al. 2008), with the following steps: (1) loss of absorbed water, (2) polymer decomposition begins with hemicellulose component, (3) cellulose and lignin contribute to weight loss after hemicellulose has decomposed, (4) most rapid rate of decomposition owing to the amount of cellulose and lignin, and (5) the remaining lignin continues to decompose above 400 °C. In general, the pyrolysis of wood occurs in a step-wise manner with hemicellulose breaking down first at 200–260 °C, cellulose next at 240–350 °C and lignin at 280–500 °C (Krzesinska et

Table 3 Proximate and ultimate analyses of bamboo raw material, bamboo charcoal and activated carbon

Proximate and ultimate analyses (%)	Bamboo (%)	Bamboo charcoal (%)	Bamboo activated carbon (%)
Moisture	15.30	4.26	5.27
Volatile matter	70.12	16.21	10.64
Ash	1.76	5.14	7.22
Fixed carbon	11.32	73.15	76.86
Nitrogen	–	0.54	0.23
Sulphur	0.07	0.16	1.71
Carbon	38.40	74.27	69.85

**Figure 1** TGA thermogram of raw bamboo

al. 2008). It can be seen from Figure 2 that the thermal decomposition of raw bamboo is similar to that of wood, as they are materials containing the same organic constituents, i.e. cellulose, hemicellulose, lignin, although in different proportions.

Activation

Bamboo-based activated carbons that are produced from carbonisation and activation processes can be used as a potentially commercially available activated carbon for the treatments of gaseous and liquid pollutants in industrial effluents and drinking water filtration applications. The use of bamboo waste from various industries will be a valuable outlet for waste products. Many industries (textiles, clothing, microelectronics, electroplating, chemical, food, drink, petrochemical) have

problems finding economically feasible method to remove pollutants from effluents.

The choice of raw material has an important impact on the economy of activated carbon. The criteria used when choosing a carbonaceous raw material include potential for obtaining high quality of activated carbon, low inorganic content, volume and cost of the raw material, workability of the raw material, minimum environmental impact and storage life of the raw material (Keith et al. 2005).

The development of activated carbon in Malaysia will encourage local industries to utilise biomass resource. It will also give the industry a chance to recover, recycle and reuse these wastes. Currently the government is looking at a number of options to reduce wastes (municipal solid waste, sewage sludge, hazardous chemical wastes) by incineration. It is well established that such

incineration processes produce dioxins which can be most effectively removed by adsorption onto activated carbons.

Effect of activation temperature

Figure 2 shows the effect of activation temperature on iodine number at different activation times. The iodine number increased with increase in activation temperature. The highest value of iodine number 823 mg g⁻¹ was obtained at 800 °C. Carbon biomass with iodine number between 600 and 1450 mg g⁻¹ is considered activated carbon (ASTM 2011). This indicates the development of pores. Meanwhile, the lowest value of iodine number 237 mg g⁻¹ was obtained at 650 °C with activation time of 30 min. When the activating agent comes in contact with the char, it reacts both with the exterior and interior of the particle, in which most of the disorganised carbon is removed. When the activation temperature increased above 800 °C, the particles were burned out completely due to very high gasification rates (Prakash Kumar et al. 2006). The variation of pores with the burn-off shows that there is an evolution of pores with activation before reaching the rupture of the pore structure, which happens probably at 150–180 min of activation (Zabaniotou et al. 2004).

Effect of activation time

The effect of activation time on iodine number at various temperature is shown in Figure 3.

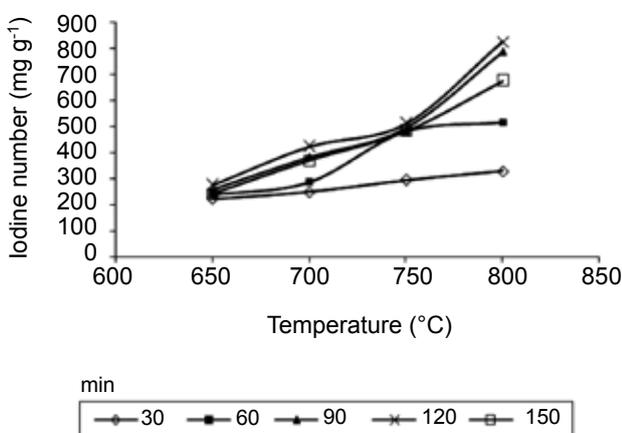


Figure 2 Effect of activation temperature on iodine number at different activation times

Increasing activation time increased the iodine number. The highest iodine number was 823 mg g⁻¹ at 120 min activation time. Meanwhile, the lowest iodine number at 30 min was 237 mg g⁻¹. Further increase in activation time at 800 °C led to complete burn-off due to very high gasification rates. The observed trend agreed with that reported by Prakash Kumar et al. (2006).

Burn-off weight percentage

The degree of activation or burn-off can be quantified by means of the weight loss of char during the activation process, that is, the weight difference between the char and the activated carbon recovered. Burn-off as an indicator of the severity of activation is one of the important factors that determine the production of activated carbons of different qualities from the raw material (Lazaro et al. 2007). The results indicate burn-off range of 73–88% (Table 4).

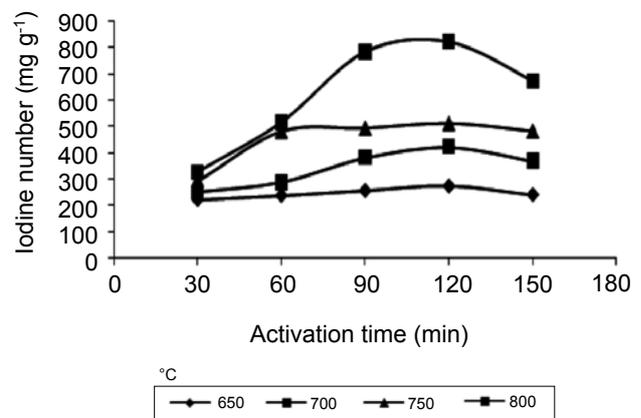


Figure 3 Effect of activation time on iodine number at different activation temperature

Table 4 Burn-off weight percentage of bamboo activated carbon at different temperatures

Temperature (°C)	Burn-off (%)
650	73
700	75
750	79
800	88

Textural characteristics of the prepared activated carbon from BET analysis

The structural changes of activated carbon prepared are summarised in Table 5. The volume of adsorbed nitrogen specific seemed to increase with increasing activation temperature. The BET surface areas increased with increase in evaluated activation temperature. This agrees with the findings of Chan et al. (2006).

CONCLUSIONS

Activation temperature and activation time were important parameters affecting the activated carbon produced. The best conditions were at 800 °C activation temperature and 120 min activation time. This gave the highest iodine number of 823 mg g⁻¹ and BET surface area of 719 m² g⁻¹. Increasing temperature and time resulted in increased iodine number which subsequently increased adsorption capacity of the activated carbon. When the temperature exceeded 800 °C, the particles were burned out completely.

The activated carbon prepared from bamboo exhibited good surface characteristics and porosity properties. This indicates that bamboo has the potential to be a promising precursor for the production of activated carbon.

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Table 5 Surface characterisation from BET analysis

Sample	BET surface area (m ² g ⁻¹)	Langmuir surface area (m ² g ⁻¹)	Micropore area (m ² g ⁻¹)	External surface area (m ² g ⁻¹)	Micropore volume (cm ³ g ⁻¹)
Bamboo charcoal	385	564	338	47	0.1752
Bamboo AC (750 °C/120 min)	464	680	407	57	0.2111
Bamboo AC (800 °C/120 min)	719	1056	553	165	0.2867

AC = activated carbon

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