



## Comparison of Activated Carbons Prepared from Indonesian Forest and Agricultural Residues

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Activated carbons were prepared from biomass waste of bengkirai wood (*Shorea laevifolia*) and coconut shell using carbonization process. The carbonization temperature and carbonization holding time were tested in the ranges of 400-600 °C for 2-6 h. The activated carbons resulted from this process were evaluated for BET surface area, pore size distribution and adsorption or iodine number. The results revealed that carbonization temperature and carbonization holding time gives significant effect on the pore structure of activated carbon produced. The highest BET surface area (271 m<sup>2</sup>/g) was obtained for coconut shell-activated carbon at the activation conditions of 800 °C for 4 h.

**Key Words:** Activated carbon, Biomass waste, Carbonization, Bengkirai wood, Coconut shell.

### INTRODUCTION

Activated carbons have well-developed pore structures and high internal surface area, so that they have been employed in a wide number of applications on an industrial scale, such as technologies for the purification of gases, the removal of organic pollutants from water for purification of drinking water and wastewater, the use for a catalyst or a catalyst support in the catalytic processes and for electrode materials in electrochemical devices and processes. Thus an activated carbon has played an important role in the chemical, pharmaceutical and food industries<sup>1</sup>.

Evaluation of waste biomass is getting increased attention in all over the world as it is renewable, widely available, low cost and environmental friendly. Indonesia, as an agricultural country, is one of the world's largest biomass resources, so there is a big potential of agricultural and forest residues. Coconut trees are grown in tropical areas and after taking coconut meat it will leave coconut shell as agricultural waste. While, bengkirai wood is a tropical hardwood found and developed at forest in South East Asia. The wood is a tremendously popular export and widely used as a slightly low cost alternative to teak and after processing the wood leaving a small chip timber as forest waste. However, they are potential sources of raw materials for the activated carbons.

Preparation of activated carbon from biomass wastes have been studied by several authors. Daud and Ali<sup>2</sup> compared the

pore development in palm and coconut-shells based activated carbons under identical experimental conditions. Carbonization and activation processes were carried out at 850 °C using a fluidized bed reactor. They found that the activation rate of coconut-shell based char is significantly higher than palm-shell based char implying less time required to reach a certain level of burn-off during activation process. On pore development, at any burn-off, the micropore and mesopore volumes created in palm-shell based activated carbon were always higher than those of coconut-shell based activated carbon. On macropore volume, for palm-shell based activated carbon, the volume increased with increase in burn-off up to 30 % and then decreased. However, for coconut-shell-based activated carbon, the change in macropore volume with burn-off was almost negligible but the absolute macropore volume decreased with burn-off. Reed and Williams<sup>3</sup> studied five types of biomass, *i.e.*, hemp, flax, jute, coir and abaca. They concluded that the characteristics of activated carbons are influenced by their feedstock. These results show that the development of the surface area and porosity characteristics of an activated carbon is influenced by the nature of the original biomass feedstock and the process conditions of carbonization and activation.

Activated carbons with well-developed microporosity and high surface areas prepared from agricultural and forest residues, *i.e.*, corn hulls, corn stover and oak studied by Zhang *et al.*<sup>4</sup> using CO<sub>2</sub> activation. The activated carbons produced

had BET surface areas ranging from 400-1000 m<sup>2</sup>/g and ratios of micropore volume to total pore volume ranging from 0.38-0.66. Li *et al.*<sup>5</sup> prepared activated carbon from coconut shell using steam activation. They reported maximum BET surface area and pore volume of 1512 m<sup>2</sup>/g and 0.46 cm<sup>3</sup>/g. While, in another study conducted by Guo *et al.*<sup>6</sup> on production of activated carbon from coconut shell by CO<sub>2</sub> activation found that enhancing activation temperature was favourable to the formation of pores, widening of pores and an increase in mesopores. It was also indicated that increasing activation time was favorable to increasing of micropores and mesopores but longer activation time would cause pores to collapse. Ahmadpour and Do<sup>7</sup> studied the effects of carbonization temperature on pore volume evolution of macadamia nut shell. The samples were chemically pretreated with ZnCl<sub>2</sub> and KOH before carbonization. Maximum surface area of 1718 and 1169 m<sup>2</sup>/g was obtained for ZnCl<sub>2</sub> and KOH impregnated samples, while the carbonization temperature was set at 500 and 800 °C for the samples, respectively. In these carbonization temperatures, the micropore volumes of 0.723 and 0.529 cm<sup>3</sup>/g for ZnCl<sub>2</sub> and KOH impregnated samples were achieved. Foo and Hameed<sup>8</sup> studied preparation of activated carbon from oil palm empty fruit bunch (EFB) via KOH chemical activation using a microwave device operating at 2.45 GHz. The activation process was performed at an input power of 360 W and an irradiation time of 15 min. They found that the BET surface area, total pore volume and average pore size were 807.54 m<sup>2</sup>/g, 0.45 cm<sup>3</sup>/g and 21.93 Å, respectively. The production of activated carbons from almond shell, using physical activation by CO<sub>2</sub> is reported by Nabais *et al.*<sup>9</sup>. The method has produced activated carbons with apparent BET surface areas and micropore volume as high as 1138 m<sup>2</sup>/g and 0.49 cm<sup>3</sup>/g, respectively. The activated carbons produced have essentially primary micropores and only a small volume of wider micropores.

Another investigation was conducted by Youssef *et al.*<sup>10</sup> to evaluate the effect of impregnation ratio of ZnCl<sub>2</sub> on textural properties of the activated carbon obtained from apricot stone. It was observed that as the impregnation ratio was increase from 25 to 75 %, the carbon BET surface area increased from 311 to 728 m<sup>2</sup>/g. In their experiments, maximum BET surface area of 728 m<sup>2</sup>/g and micropore volume of 0.3 cm<sup>3</sup>/g were achieved at impregnation ratio of 75 % and carbonization temperature of 600 °C. Munoz-Gonzalez *et al.*<sup>11</sup> investigated the preparation of activated carbon from peach stone by phosphoric acid activation. They also concluded that as the concentration of H<sub>3</sub>PO<sub>4</sub> in the solution was increased from 2 to 8 mol/L, the porosity of the carbon was improved. Maximum micropore volume of 0.4 cm<sup>3</sup>/g was obtained at carbonization temperature of 450 °C.

The aim of this study is to investigate the conversion of forest and agricultural residues into high-quality activated carbons that might be commercially viable in one step activation. Biomass wastes from agricultural and forest residues, *i.e.*, coconut shell and bengkirai wood were selected in this study. The activated carbons were prepared through carbonization in a fluidized bed accompanied by heat treatment, over a range of activation conditions. The activation temperature and duration time were explored for their effects on the properties of the activated carbons, then characterized according

to their physical adsorption properties by the conventional gas sorption methodology.

## EXPERIMENTAL

**Preparation of activated carbon:** Bengkirai wood (*Shorea laevifolia*) and coconut shell utilized in the present study were obtained from Sleman in Yogyakarta Province of Indonesia. The starting materials were manually chosen, cleaned with deionized water, dried at 110 °C for 12 h and sieved to a size range of 5.0-7.5 mm and stored in sealed containers for experimentation.

Carbonization of the materials carried out in a vertical stainless-steel reactor, which was placed in an electrical furnace. During the carbonization process, about 10 g of raw materials was used to prepare the chars. The materials were heated up to a carbonization temperature (400, 600, 800 °C) at a heating rate of 10 °C/min and were held for at least 2 h and held at this temperature for certain time at the carbonization temperatures under N<sub>2</sub> gas flow. After carbonization, the furnace was cooled down to room temperature with N<sub>2</sub> flushing through the sample.

### Physical characterization

**N<sub>2</sub> adsorption of activated carbon:** The pore structure characteristics of activated carbons were determined using N<sub>2</sub> adsorption (Quantachrome NovaWin2). Pore structures of sample were characterized by nitrogen adsorption at 77 K with an accelerated surface area and porosimetry system. Prior to gas adsorption measurements, the carbon was degassed at 300 °C in a vacuum condition for a period of at least 3 h. Nitrogen adsorption isotherm was measured over a relative pressure (P/P<sub>0</sub>) range from approximately 10<sup>-6</sup> to 1. The BET surface area, microspore volume and microspore surface area of the activated carbons were determined by application of the Brunauer-Emmett-Teller (BET) analysis software available with the instrument.

**Iodine number of the activated carbons:** The end product is characterized for yield and iodine number. In this case, yield is defined as the ratio of the dry weight of activated carbon obtained to the dry weight of the raw material taken for activation. The iodine number indicates the porosity of the activated carbon or surface area contributed by the pores larger than 10 Å and it was define as the milligrams of iodine adsorbed per gram of carbon This number is defined as the milligrams of iodine adsorbed by 1 g of carbon. The iodine adsorption was determined using the sodium thiosulfate volumetric method (ASTM D4607-94) and estimated by mixing the activated carbon with 0.02 N iodine solution shaken occasionally and then by titration of the solution against Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>.

## RESULTS AND DISCUSSION

**Effect of carbonization temperature and holding time of carbonization on the yield of activated carbon:** The yield is an important factor to be considered in the production of activated carbon. Figs. 1 and 2 shows the effect of carbonization temperature and holding time of carbonization on the yield of activated carbon with different raw material, *i.e.*, bengkira wood and coconut shell. Fig. 1 showed that the yields

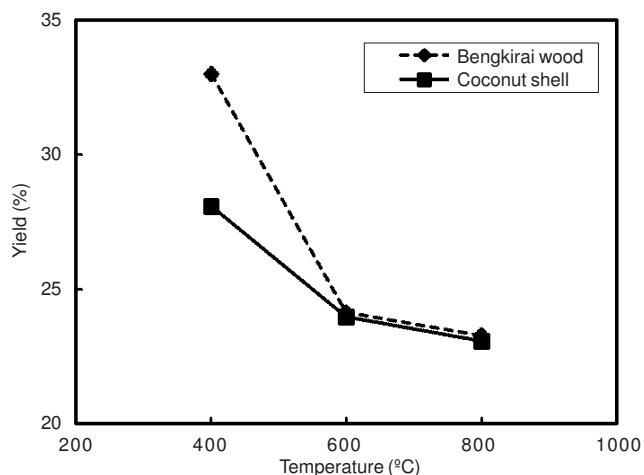


Fig. 1. Effect of carbonization temperature on the yield of activated carbon

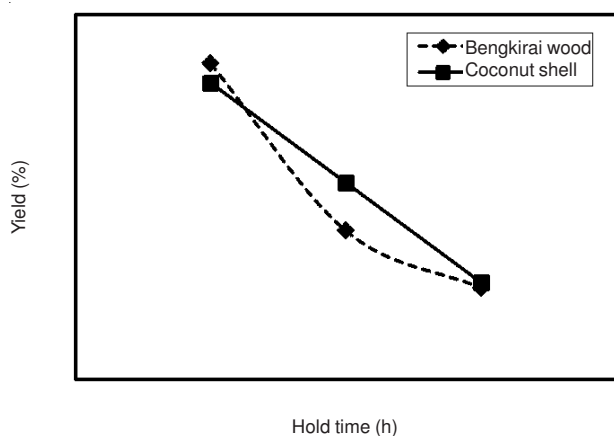


Fig. 2. Effect of holding time on the yield of activated carbon

of all activated carbons derived from different raw material had the similar trends. The yields decreased with increasing carbonization temperature. These findings are consistent with the general concept that increasing the carbonization temperature decreases the amount of the unstable volatiles on the activated carbon and eventually influences the product yield and porosity<sup>12</sup>. The yields of activated carbons derived from bengkirai wood were larger than those activated carbons derived from coconut shell.

Generally, carbonization process applied to generate char, oil or gaseous product from biomass. During carbonization, moisture and volatile compounds are removed from the biomass and solid chars are generated whose properties are different from the nature origin of the biomass materials<sup>13</sup>. The lignocellulosic composition of the biomass has a significant effect on the yield of the activated carbon. Researchers confirmed that lignin is the main source of char, whereas cellulose and hemicelluloses are the volatile fractions of biomass. Therefore, the composition of the lignocellulosic biomass is an important parameter in char formation and the higher lignin content results in higher char yield<sup>14,15</sup>. From Fig. 2, the holding time of the carbonization process has an effect on the yield of activated carbon. The time should just be enough to eliminate all the moisture and most of the volatile components in the precursor to give high yield of the activated

carbon. Since the end of the volatile evolution marks the formation of the basic pore structure, activation should be limited up to that point.

#### Effect of carbonization temperature on the pore structures and pore size distribution of activated carbon:

The adsorption capacity of an activated carbon largely depends on the amount of micropores that are present in the solid and surface area of carbon. There are several critical parameters in the preparation of activated carbon that would affect its structure and one of them is carbonization temperature. While, one of the methods for estimating the type of pores present in a solid is by analyzing the isotherm curve. The N<sub>2</sub> adsorption isotherms at 77 K for activated carbons derived from bengkirai wood and coconut shell at different carbonization temperatures were shown in Figs. 3 and 4.

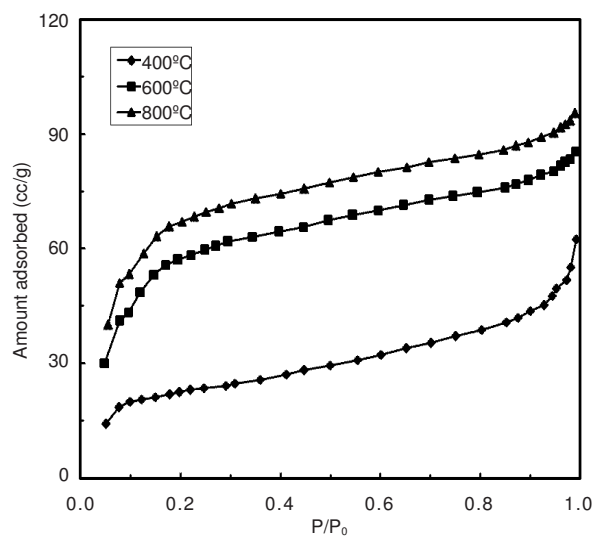


Fig. 3. N<sub>2</sub> adsorption isotherms at 77 K for activated carbons derived from bengkirai wood under different carbonization temperatures

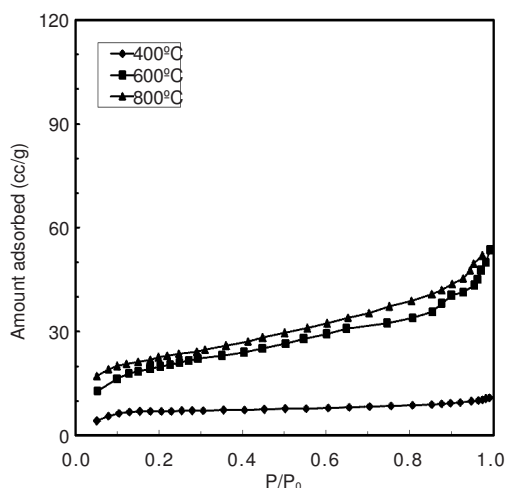


Fig. 4. N<sub>2</sub> adsorption isotherms at 77 K for activated carbons derived from coconut shells under different carbonization temperatures

As can be seen from Fig. 3 for bengkirai wood, the isotherms may be considered as the intermediate between types I and II according to BDDT (Brunnauer-Deming-Deming-Teller) classification<sup>16</sup>, with a noticeable amount of N<sub>2</sub> retained

not only at very low values of  $P/P_0$  as usual in type I isotherms, but also at higher values of  $P/P_0$  as characterized by type II isotherms. The initial part of the type I isotherm at low relative pressure represents micropore filling and the slope of the plateau at high relative pressure is due to multilayer adsorption on the nonmicroporous surface, *i.e.*, in mesopores, in macropores and on the external surface. At low-pressure region or  $P/P_0 < 0.2$  significant uptake of nitrogen was occurred. This means that nitrogen molecules were adsorbed in the microporous structure. The adsorption in micropores was interpreted according to the pore filling mechanism, thus result in highly adsorbed volume.

Similar to activated carbon derived from bengkirai wood, it was indicated in Fig. 4 that the isotherms of activated carbon derived from coconut shells belonged to the intermediate between types I and II. From these results, it could be inferred that a significant amount of microporosity and a small amount of mesoporosity appeared to be formed in the activated carbons prepared. As shown in Fig. 3, the initial part of the type I isotherm for the chars at low relative pressure represents micropore filling and the slope of the plateau at high relative pressure is due to multilayer adsorption on the nonmicroporous surface, *i.e.*, in mesopores, in macropores and on the external surface. By comparing the isotherm curves of activated carbon derived from coconut shell and activated carbon derived from bengkirai wood, it can be concluded that for the same carbonization temperature, activated carbon derived from coconut shell has a higher adsorption capacity as compared to that derived from bengkirai wood. For example, at carbonization temperature of 800 °C, the adsorption capacity of activated carbon derived from coconut shell is approximately 190 cc/g at STP, while 65 cc/g at STP for that derived from bengkirai wood.

The structural heterogeneity of a porous material is generally characterized in terms of the pore size distribution. This pore size distribution represents a model of a solid internal structure and closely related to both kinetic and equilibrium properties of the porous material and perhaps is the most important aspect for characterization<sup>17</sup>.

In accordance to the classification adopted by the IUPAC, adsorbent pores are classified into three groups: micropores (size < 2 nm), mesopores (2-50 nm) and macropores (> 50 nm). Micropores can be divided into ultramicropores (width

less than 0.7 nm) and supermicropores (width from 0.7-2.0 nm). The size of pores that are formed would have an effect on the porosity, the total surface area is available for adsorption and most importantly, the size of molecules that can diffuse into the solid. Thus the development of an appropriate pore structure is necessary if the activated carbon produced is to be used for a particular application<sup>12</sup>.

Table-1 shows pore characteristic of activated carbon derived from bengkirai wood and coconut shell at different carbonization temperature and carbonization holding time. It was found in Table-1 that when the carbonization temperature increased there is no significant effect on the BET surface area both on the coconut shell activated carbon and the bengkirai wood of activated carbon, however total volume and micropore volume increased. In general, the higher BET surface area is an indication of the existence of a significant amount of microporosity. With increasing carbonization temperature, the adsorption capacity of the activated carbon increased. At high carbonization temperatures, a more ordered structure is likely to be developed in the char that leads to a slower rate of gasification in the interior of the particle. As could be seen in Table-1, the maximum BET surface area of the coconut-shell activated carbon was 264 m<sup>2</sup>/g (carbonization temperature: 800 °C; holding time: 4 h), which was higher than that of bengkirai wood activated carbon, which has 80 m<sup>2</sup>/g (carbonization temperature: 600 °C; holding time: 4 h).

Figs. 5 and 6 showed that the pore size distributions of activated carbons derived from coconut shell and bengkirai wood prepared at different carbonization temperature by BJH<sup>18</sup> method. From Figs. 5 and 6, it can confirm the existence of a limited pore size distribution for all samples, showing pores mainly in the micropore range. On the coconut shell activated carbons, it can be observed that the pore distribution became wider for samples with higher carbonization temperature. However for the bengkirai wood activated carbons, there is no significant effect of carbonization temperature to develop microporosity.

**Effect of carbonization holding time on the pore structures and pore size distribution of activated carbon:** The duration of the activation has a significant effect on the development of the carbon's porous networks. The time should just be enough to eliminate all the moisture and most of the volatile components in the precursor to cause pores to develop.

TABLE-1  
PORE CHARACTERISTIC OF ACTIVATED CARBON DERIVED FROM BENGKIRAI WOOD AND COCONUT SHELL AT DIFFERENT CARBONIZATION TEMPERATURE AND CARBONIZATION HOLDING TIME

Biomass	T (°C)	Holding time (h)	S <sub>BET</sub> (m <sup>2</sup> /g)	S <sub>micro</sub> (m <sup>2</sup> /g)	V <sub>T</sub> (cm <sup>3</sup> /g)	V <sub>micro</sub> (cm <sup>3</sup> /g)
Coconut shell	400	4	263	260	0.0403	0.0260
Coconut shell	600	4	250	236	0.0649	0.0253
Coconut shell	800	4	271	264	0.0757	0.0409
Bengkirai wood	400	4	26	23	0.0062	0.0022
Bengkirai wood	600	4	80	59	0.0153	0.0042
Bengkirai wood	800	4	74	50	0.0130	0.0037
Coconut shell	400	2	100	84	0.0240	0.0086
Coconut shell	400	4	251	237	0.0653	0.0255
Coconut shell	400	6	249	235	0.0647	0.0253
Bengkirai wood	400	2	26	24	0.0070	0.0029
Bengkirai wood	400	4	81	67	0.0178	0.0061
Bengkirai wood	400	6	74	59	0.0153	0.0054

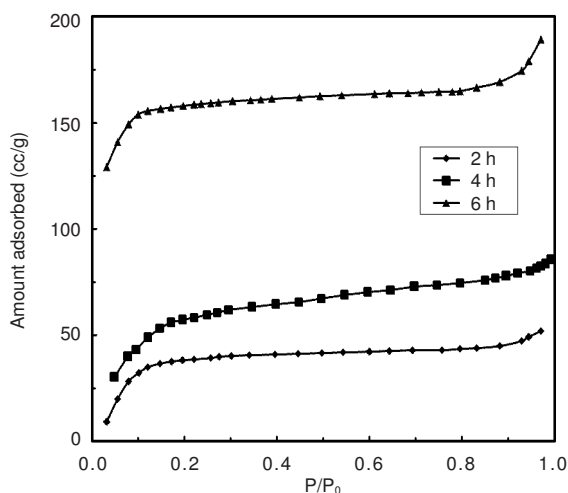


Fig. 5. Pore size distribution of coconut shell activated carbon prepared at different carbonization temperatures

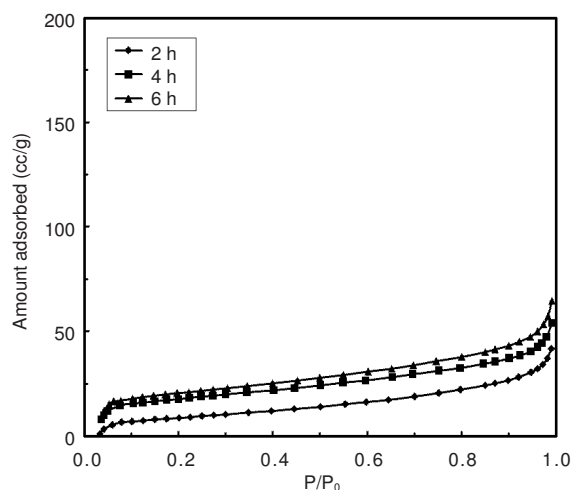


Fig. 6. Pore size distribution of bengkirai wood activated carbon prepared at different carbonization temperatures

Since the end of the volatile evolution marks the formation of the basic pore structure, activation should be limited up to that point. Longer durations cause enlargement of pores at the expense of the surface area. Also, the control of the activation time is of economic interest since shorter times are generally desired as it equates to reduction in the energy consumption<sup>19</sup>.

Fig. 7 shows the  $N_2$  adsorption isotherms at 77 K for activated carbons derived from bengkirai wood under different holding time of carbonization. It was indicated in Fig. 7 that the isotherms of all activated carbons prepared belonged to the intermediate between types I and II. The results were similar to those of activation temperature. From these results, it could be inferred that a significant amount of microporosity and a small amount of mesoporosity appeared to be formed in the activated carbons prepared.

Fig. 8 shows the  $N_2$  adsorption isotherms at 77 K for activated carbons derived from coconut shell under different holding time of carbonization. As shown in Fig. 8, the activated carbon derived from coconut shell exhibit the intermediate between types I and II isotherm according to the IUPAC classification<sup>20</sup>. The presence of hysteresis and an increase of the adsorbed nitrogen volume values for higher relative pressures

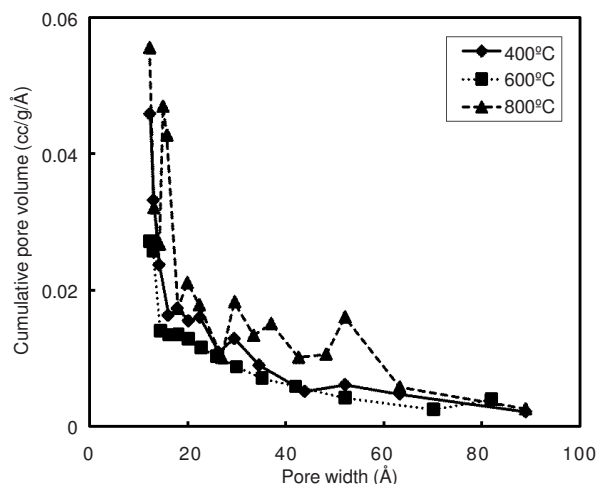


Fig. 7.  $N_2$  adsorption isotherms at 77 K for activated carbons derived from bengkirai wood under different holding time of carbonization

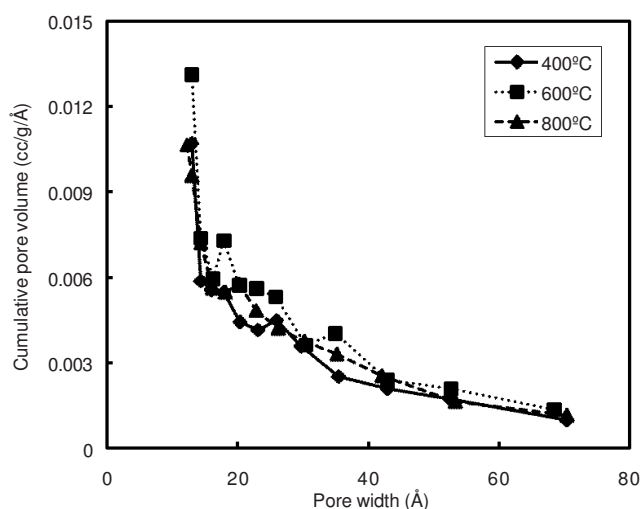


Fig. 8.  $N_2$  adsorption isotherms at 77 K for activated carbons derived from coconut shells under different holding time of carbonization

reveal meso and macroporosity. In a comparison between coconut-shell-based activated carbon in Fig. 6 and bengkirai wood-based activated carbon in Fig. 5 prepared by different carbonization holding time, coconut-shell-based activated carbon was shown higher adsorption capacity based on the  $N_2$  isotherm curve.

Figs. 9 and 10 illustrated the pore size distributions (PSD) of the activated carbon using BJH method for various carbonization holding time at carbonization temperature 800 °C. From Fig. 9, it is obvious that the impregnation ratio gives significant effect on the pore structure of activated carbon produced. At 2 h carbonization holding time, the pore structure of activated carbon produced mainly consists of micropore, however with the increase of carbonization holding time, the creation of micropore structure and widening of micropores to mesopores also increase. The result from Fig. 10 also shows the same result as Fig. 9. This figure confirms that with the increase of carbonization holding time on bengkirai wood activated carbon, the creation of micropore structure occur. This conclusion was also supported by the measurement of BET surface area, total pore volume and micropore volume as shown in Table-1. It also can be seen from Table-1 that the BET

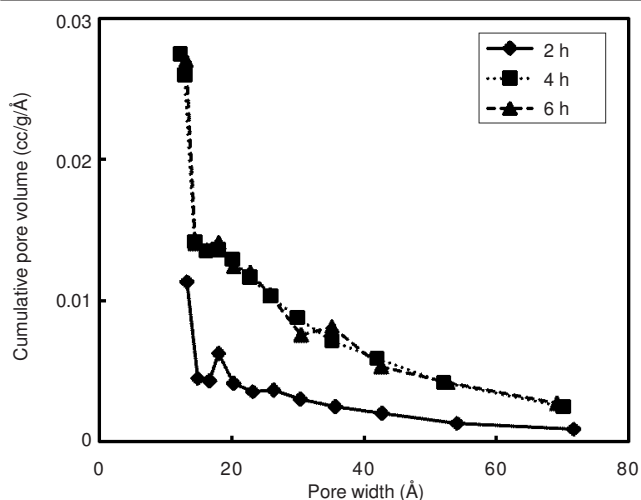


Fig. 9. Pore size distribution of coconut shell activated carbon prepared at different carbonization holding time

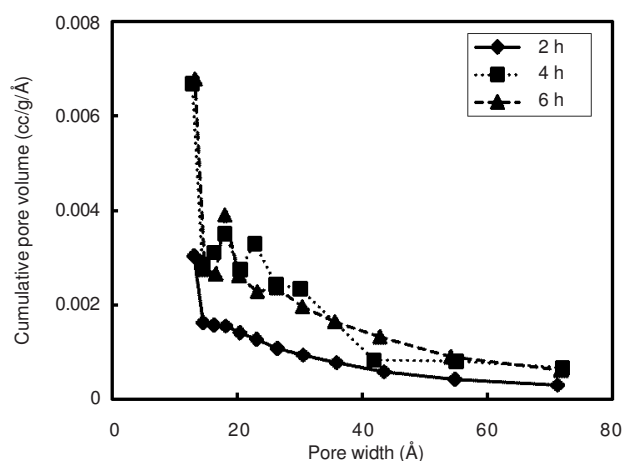


Fig. 10. Pore size distribution of bengkirai wood activated carbon prepared at different carbonization holding time

surface area, total pore volume and micropore volume of the activated carbons increase with increasing the carbonization holding time from 2-6 h. The conclusion is that the coconut shell activated carbon has larger BET surface area, total pore volume and micropore volume as compared compare to the bengkirai wood activated carbon.

**Iodine number of the activated carbons:** Iodine number was measured to evaluate the adsorptive capacity of the activated carbon. It has been established that the iodine number (in mg/g) measures the porosity for pores with dimensions  $\geq 1.0$  nm. The iodine numbers were determined by using the single bottle point uptake from 0.02 M iodine solution.

Figs. 11 and 12 illustrate the effect of carbonization temperature and carbonization holding time on iodine number. From Fig. 11, it can be seen that the iodine number increase significantly with increase in carbonization temperature for both bengkirai and coconut shell activated carbons. The increase in carbonization time increases the micropore development, which increases the adsorption capacity of the activated carbon. The highest iodine number was reached at 800 °C carbonization temperature for both materials. As shown in Fig. 12, similar result was shown where increasing

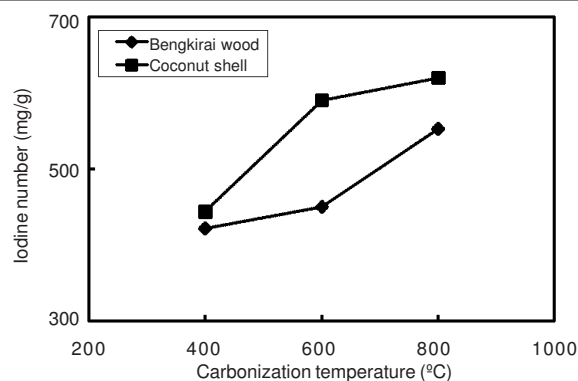


Fig. 11. Relation between carbonization temperature and iodine number

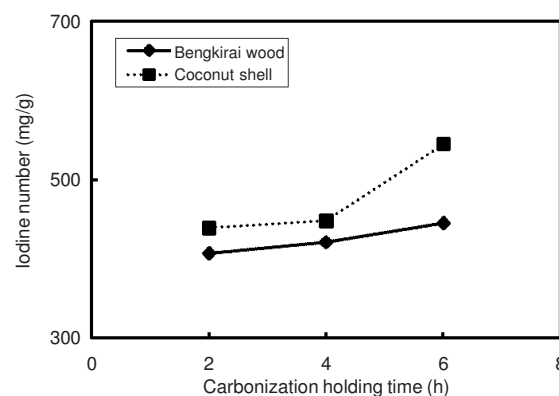


Fig. 12. Relation between carbonization holding time and iodine number

carbonization holding time increase the iodine number. The higher iodine number may be the indication of the existing of highly developed microporous structure.

## Conclusion

The main conclusion of this study is that we were able to produce activated carbons from bengkirai wood and coconut shell with good physical properties, in terms of adsorption properties, surface area measurements and pore size distributions. The carbonization temperatures and carbonization holding time had great effects on the yields and pore development of the chars prepared. With increasing carbonization temperatures and carbonization holding time, more volatiles were released and more micropores were formed. All of the isotherms for all activated carbons prepared may be considered as the intermediate between types I and II according to BDDT (Brunnauer-Deming-Deming-Teller) classification, with a noticeable amount of  $N_2$  retained only at very low values of  $P/P_0$  (as usual in type I isotherms). According to the BET surface area, the coconut-shell activated carbon is significantly higher than the bengkirai wood activated carbon. On pore development, at any carbonization temperatures and carbonization holding time, the micropore created in coconut-shell activated carbon is always higher than that of bengkirai wood activated carbon. The iodine number increases with increasing carbonization temperatures and carbonization holding time. By comparing the iodine number of coconut-shell activated carbon and bengkirai wood activated carbon, it can observe that for any any carbonization temperatures and carbonization holding time, coconut-shell activated carbon has a higher adsorption capacity as compared to bengkirai wood.

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