Pore Size Distribution and Microstructure of Oil Palm Shell Heat Treated at 300°C Followed by Slow- or Fast Heating Treatment

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Abstract

Pore size distribution and microstructure development of oil palm shell heat treated at 300°C and treated at 300°C and recarbonization at 600°C followed by slow- or fast heating treatment up to 700°C were investigated by small angle X-ray scattering (SAXS), N\textsubscript{2} gas adsorption and Raman spectroscopy. On oil palm shell heat-treated at 300°C, slow heating treatment gave the widening micropore along with the ordering microstructure; but fast heating treatment produced charcoal with a narrow diameter of micropore with wider pore size distribution and the disordering microstructure. On oil palm shell heat treated at 300°C and recarbonization at 600°C, slow heating treatment contributed on the opening new micropore with ordering microstructure, but some parts of micropore showing inaccessible for N\textsubscript{2} gas. Meanwhile, fast heating treatment with the heating rate from 75 to 250°C/min increased BET surface area with similar pore size distribution and the disordering microstructure.

Keywords: Oil palm shell charcoal, pore size distribution, microstructure, slow- fast heating treatment, SAXS, Raman spectroscopy.

Introduction

Charcoal is carbonaceous material produced from carbonization process of biomass including wood and other biomass such as oil palm shell. The microstructure of charcoal consists of porosity which is the gaps between elementary graphitic crystallites and carbon skeleton which is formed by a graphitic sheet with a random orientation. The microstructure of charcoal from biomass is considered to provide an important contribution for the control in the development of biomass carbon based material (Ishimaru et al. 2007). In recent years, the microstructure of biomass based carbon has been used as the template for the formation of SiC, SiC/C and SiC/SiO\textsubscript{2}/C composites (Greil 2001; Fujisawa et al. 2004; Vyshnyakova et al. 2006; Sulistyo et al. 2010). A detailed microstructural study of softwood pyrolysis performed by Paris et al. (2005) showed that the charring process commenced with the formation and growth of aromatic structures and nanopores at temperature higher than 327°C. The ordering of the carbon structures occurred with the increase of heat treatment temperature from 400-800°C (Yamauchi and Kurimoto 2003). This report had a good agreement with previous studies which defined that the formation microporosity system at 500°C and used of the temperature range in the preparation of charcoal for activated carbons (Mackay and Roberts 1981; Pastor-Villegas et al. 1993; Lua et al. 2006). Carbonization at a temperature of 700°C was conducted to prepare the charcoal for porous SiC ceramic (Fujisawa et al. 2004). Therefore, a condition of the carbonization especially temperature, obviously very much influence charcoal with the proper microstructure for production of carbon based materials.

Charcoal with a low degree of order of microstructure is generated from the production of smoke liquid from oil palm shell. The smoke liquid production is run at low temperature heat treatment of 300°C to maximize the yield of liquid product and to minimize the content of polyaromatic hydrocarbons (Hattula et al. 2001; Demirbas 2001). A large amount of this solid waste charcoal material with high carbon content is potentially used as precursors for the preparation of adsorbents and for lignocellulosic ceramics such as SiC composites. However, the microstructure in the charcoal limits the further application and utilization. Until now, fewer studies have been reported on the utilization of the charcoal heat-treated at 300°C.

Heat treatment of the solid waste charcoal oil palm shell was necessary to be carried out to improve a degree of the order of the microstructure in the material. The heat treatment of rockrose wood with a heating rate of 10°C/min develops the charcoal with pores in the range of micro- to macropores (Pastor-Villegas et al. 1993). Heat treatment at a slow heating rate up to 800°C removes volatile matters with a series of chemical reactions such as dehydration, breaking the C-O, C=O and C-C bonds, or repolymerization (Kurosaki et al. 2007). On the other hand, the fast heating with the rate of 6000°C/min up to 800°C made the chemical reactions occur concurrently and produced the charcoal with macroporous structure. In this study, the heat treatments with large different heating rates were carried out in order to produce the charcoal with different pore structures and microstructures from oil palm shell heat-treated at 300°C. The pore size distribution and microstructure of oil palm shell heat-treated at 300°C followed by slow-or fast heating treatments up to 700°C were investigated by small angle X-ray scattering (SAXS) and Raman spectroscopy. The
effect of reaction times in slow heating treatment and that of heating rates in fast heating treatment were examined in this study.

**Experimental**

**Heat Treatment**

Air dried oil palm shell (OPS) with the characteristics as shown in Table 1 was heat-treated at a rate of 10°C/min and was maintained constant at 300°C for 180 min in an electric cylindrical steel furnace of 1 kg capacity. The charcoal heat-treated at 300°C noted as HT1. The furnace was connected to a water-cooled condenser to entrap liquid by cooling smoke. After heat treatment, HT1 charcoals were granulated by a hammer mill to be sieved to 63-150 μm. As the comparison of the effect of heating at 600°C, part of HT1 samples were re-carbonized up to 600°C at 100°C/min and were then held at this temperature for 60 min in a muffle furnace. The charcoal from the heat treatment at 300°C and then followed by re-carbonization at 600°C noted as HT2.

**Slow Heating Treatment**

Dry weight of 20 g of HT1 or HT2 charcoal was mixed with 40 ml distilled water containing 4 g of ZnCl₂ at 60°C. The small amount of ZnCl₂ was used in the mixture to aid in porosity development in charcoal (Rodriguez-Reinoso and Molina-Sabio 1992). The dehydrated materials were dried at 115°C for 3 h in an electric oven and then were heat-treated at 10°C/min up to 700°C, and then kept for 60, 120 and 180 min in a muffle furnace. After cooling, charcoals were washed with 0.1 N HCl and water and then were dried at 115°C.

**Fast Heating Treatment**

Dry weight of 0.2 g of HT1 or HT2 charcoal was put into a 10 mm of the diameter of graphite die with a length of 5 cm (point 1 in Fig. 1) which was closed with two graphite punches (point 2 in Fig. 1) with a length of 2.5 and 2.2 cm. The charcoal in the graphite die was placed in a pulse current sintering apparatus (VCSP-II, SS-Alloy Co. Ltd., Hiroshima) as sketched in Fig. 1. The charcoal was heat-treated at 75, 250, 1000 and 2000°C/min up to 700°C and was kept for 15 min under N₂ gas flow with a rate of 100 mL/min. A pressure of 12 MPa on the graphite die which had minimum effects on the charcoal was applied from the start of the heating and was released after cooling.

Tabel 1. Proximate and ultimate analysis of raw and carbonized material oil palm shell.

| Samples | Volatile matter (%) | Fixed carbon (%) | Ash (%) | C (%) | H (%) | N (%) | Odiff (%) |
|---------|---------------------|------------------|---------|-------|-------|-------|----------|
| OPS     | 73.08               | 13.30            | 2.37    | 49.64 | 6.76  | 0.20  | 41.03    |
| HT1     | 22.31               | 36.52            | 5.69    | 77.95 | 2.05  | 0.22  | 14.09    |
| HT2     | 10.81               | 69.73            | 6.45    | 89.39 | 2.44  | 0.16  | 1.56     |

Note: OPS is raw oil palm shell. HT1 charcoal is oil palm shell heat-treated at 300°C for 180 min; HT2 charcoal is oil palm shell heat-treated at 300°C for 180 min and then followed by re-carbonization at 600°C for 60 min. Odiff is estimated by difference from 100-C-H-N-Ash elements.

![Figure 1. Schematic of a pulse current sintering apparatus.](image-url)
Pore Distribution, Surface Area, and Microstructure Analysis

The pore size and distribution were analyzed by a three-slit optical systems with a line focus of SAXS (small-angle X-ray scattering, Rigaku Corporation, Japan). X-ray generator was operated at 50 kV and 200 mA. The intensity was measured with a scintillation counter. The scattered intensity was recorded as a function of the scattering wave vector,

\[ q = \frac{4\pi}{\lambda} \sin \theta \]  

(1)

where \( \lambda \) is the X-ray wavelength and \( 2\theta \) is the scattering angle. The scattered intensity of charcoal which was put into a transparent capillary (Borokapillaren glas, Germany) with a diameter of 1 mm and a length of 80 mm was corrected by that of the transparent capillary without charcoal.

The pore size distribution was analyzed by using NANO-Solver Ver. 3.4 (Rigaku Corporation, Japan). This software program conducted profile fittings over the entire measurement range of scattered intensity in order to improve reproducibility in analysis. Least-squares methods were adopted to optimize the fitting parameters for a spherical model, assuming dispersed spheres. The spherical model which was fitted to the measurement data calculated the scattering intensity which depended on the form factor and structure factor using the following equation (Rigaku Corporation 2006):

\[ I(q) = |F(q)|^2 S(q) \]  

(2)

where \( F(q) \) is the form factor of the pore with a sphere shape possessing a radius \( R \), and \( S(q) \) is the structure factor which is equal 1 when the pores are randomly distributed in the medium. The spherical model with a radius of \( R \) is used in the description of form factor (Rigaku Corporation 2006) as follow

\[ F(q, D) = \Delta \rho \frac{4\pi}{q^3} (\sin(qR) - qR \cos(qR)) \]  

(3)

where \( \Delta \rho \) is electron density difference at position \( R \). The structure factors are expressed by following equations (Rigaku Corporation 2006):

\[ S(q) = 1 + \int (n(r) - n_0) e^{iqr} dr \]  

(4)

where \( n_0 \) is average particle density. The pore distribution assumes \( \Gamma \) distributions which are expressed by the following equation (Rigaku Corporation 2006):

\[ P_{MR}^M(R) = \frac{1}{\Gamma(M)} \left( \frac{M}{R_0} \right)^M e^{-\frac{MR}{R_0}} R^{-1-M} \]  

(5)

where \( R_0 \) is the average pore radius and \( M \) is the shape parameter in the software. The shape parameter \( M \) is expressed by \( \sigma \), the normalized dispersion normalized by average size, as follows (Rigaku Corporation 2006):

\[ \sigma = \sqrt{\frac{\delta R^2}{R_0}} x100 = \frac{1}{\sqrt{M}} x100 \]  

(6)

The pore size of HT1 and HT2 charcoals was also analyzed by \( N_2 \) adsorption at 77 K by a NOVA 2000 (Quantachrome Instruments, USA). The pore size was determined by the Barret-Joyner-Halenda (BJH) models (Barrett et al. 1951). In the case, surface area using Brunauer-Emmett-Teller (BET) equation (Brunauer et al. 1938) and adsorption isotherm in HT2 charcoals prepared by slow- and fast heat treatment were determined from \( N_2 \) adsorption at 77 K by a TriStar II 3020 (Micromeretics Instrument Corporation, USA).

A Raman spectroscopy (Renishaw inVia, England) equipped with an air-cooled CCD detector was used to analyze the carbon microstructure of the charcoals. An argon laser (514.5 nm) was adopted as an excitation source. The laser was focused to approximately 1 μm in diameter at a power of less than 1 mW on the charcoal surface in order to prevent irreversible thermal degradation. Spectra were measured in the 1,100-1,800 cm\(^{-1}\) range. Six 10-second accumulations gave adequate signal-to-noise ratio of the spectra. The wave number was calibrated using the 520 cm\(^{-1}\) line of a silicon wafer. Spectral processing was performed using WiRE 2 software. The crystallite size or coherence length \( (L_o) \) was determined from Raman peak data by an empirical formula developed by Tuinstra and Koenig (1970):

\[ L_o = \frac{(43.5 \, \AA)}{I_d/I_g} \]  

(7)

Proximate and Ultimate Analysis

Proximate analysis, including moisture, ash content, and volatile matter, were determined by ASTM D 2867-70, D 2866-70, and D 1762-64, respectively (1970; 1977). The fixed carbon content was obtained by difference. Ultimate analyzes was carried out by Perkin Elmer elemental analyzer 2400. The oxygen content was obtained by difference. The proximate and ultimate analyses were conducted on oil palm shell, HT1, and HT2 charcoals.

Results and Discussion

Pore Size Distribution

Fig. 2 shows an example of pore distribution analysis of a SAXS profile of HT2 charcoal by NANO-Solver, which was calculated by a curve fitting method where a simulated curve was to be fitted to a SAXS profile from scattering. In the SAXS profile, the scattering intensity in the low and high degree regions of 2θ attributed to inter- and intraparticle disorder, corresponding to meso- and micropore, respectively. The curve fitting analysis showed that the dominant pore structures of HT2 charcoal were micropores.
distibution using NANOler distributions. On the other hand, distribution of charcoal. The intensity, a.u.) internal diameter less than 2 nm. Mesopore is between 2 and 50 nm. The meaning of HT2 charcoal is referred to that in Table 1.

Table 2 lists the pore distribution, the volume fraction of pore and the average pore diameter of HT1 and HT2 charcoals measured by BJH method and analyzed by SAXS. The average pore diameter of HT1 and HT2 charcoals showed a minor difference and good agreement with that calculated by BJH method using N2 adsorption analysis at 77 K (Barrett et al. 1951). On the other hand, wider distribution of mesopores was found in both HT1 and HT2 charcoals.

The size distribution of micropore in HT1 and HT2 charcoals are shown in Fig. 3. The average micropore size in HT1 charcoal was 0.376 nm which was smaller than that of 0.5 nm in the pine wood charcoal heat treated at 347°C reported by Paris et al. (2005). The micropores were formed in HT1 charcoal when gas and tars evolved up to 300°C (Rodriguez-Reinoso and Molina-Sabio M 1992). In the case HT2 charcoal, the slope of SAXS profile increased slightly, equivalent to the greater diameter in the pores of 0.917 nm, which was greater than that of HT1 charcoal. The wider pore diameter with smaller distributions in HT2 charcoal was indicated by the slight increase in the SAXS signals. The heat treatments at 300°C and followed by re-carbonization at 600°C on HT2 charcoal released pyrolysis products which may influence on widening micropores already existed in the material. The micropore distribution of HT2 charcoal was smaller than that of HT1 charcoal, meaning that the widening of micropore might be occurred by connecting bigger pores leading to a certain pore size.

Figure 4a and c show the size distribution of micropore in HT1 charcoals in slow- and fast heating treatments. In slow heating treatment with additional ZnCl2, lower scattering intensity of reaction time 120 and 180 min indicates lower porosity of the samples than that of 60 min. It is suggested that a prolonged reaction time from 60 min to 120 and 180 min caused the conversion of some micropore into wider pore sizes which reduced the porosity of the sample. The prolonged reaction time from 60 min to 120 min and 180 min also increased the diameter of micropore from 0.347 to 0.842 and 0.511 nm. In case of fast heating treatment, HT1 charcoals treated at different heating rates showed quite a similar scattering intensity which exhibited downward-convex profiles corresponding to a wide size distribution. The scattering intensity profiles of HT1 charcoals in fast heating treatment were lower than that in

Table 2. BET Surface area and pore diameter analyzed by BJH model and by SAXS of HT1 and HT2 charcoals.

| Charcoal | BET surface area (m²/g) | BJH pore diameter (nm) | Diameter of pore I (nm) | Vol. (%) | Diameter of pore II (nm) | Vol. (%) | Avr. Pore diameter (nm) |
|----------|-------------------------|------------------------|-------------------------|----------|------------------------|----------|------------------------|
| HT1      | 2.302                   | 5.148                  | 0.376                   | 94.5     | 87.47                  | 5.5      | 5.140                  |
| HT2      | 28.915                  | 4.533                  | 0.917                   | 90.1     | 41.84                  | 9.9      | 4.959                  |

Note: The meaning of HT1 and HT2 is referred to that in Table 1. Pore I and II are referred to that in Fig. 2.
slow heating treatment indicates lower porosity of the samples than that of HT1 charcoals in slow heating treatment.

Figure 4b and d show the size distribution of micropore in HT2 charcoals in slow- and fast heating treatments. In slow heating treatment, highest scattering intensity of reaction time 120 min and 180 min indicates higher porosity in the samples than that of 60 min. Heat treatment at 300°C and then at 600°C in the preparation HT2 charcoal may increase the release of volatile matters, resulting in widening micropore in the charcoal. Subsequent heat treatment at 700°C for 120 and 180 min with additional ZnCl₂ enhanced the pores and created new micropore, which showed good agreement with the previous study (Lua et al. 2006) that reported the optimum temperature of carbonization at 600°C for preparation of activated carbon. In the case of fast heating treatment, similar with previous discussion, the scattering intensity of samples was almost identical. The scattering intensity of HT2 charcoals in fast heating treatment was remarkable lower than that in slow heating treatment. However, fast heating treatment in a pulse current apparatus formed high distribution of homogeneous narrow micropore around 0.15-0.28 nm for HT1 charcoals and around 0.1 for HT2 charcoals which were different with an earlier investigation which reported that the fast heating produced macroporous carbon (Kurosaki et al. 2007).

Fig. 3. Size distributions of micropores in HT1 and HT2 charcoals. The meaning of HT1 and HT2 is referred to that in Table 1.
Surface Area

Surface area and adsorption isotherm were determined on HT2 charcoals in slow- and fast heating treatments due to the much different characteristics of micropore distribution. Figure 5a shows BET surface area of HT2 charcoals in slow heating treatment with additional ZnCl₂ in relationship to reaction time. HT2 charcoals prepared with reaction time 120 min and 180 min exhibited comparable BET surface area to the charcoal prepared with reaction time 60 min. The increasing porosity of HT2 charcoals in slow heating treatment due to the increasing reaction time from 60 min to 120 min and 180 min as shown by SAXS analysis was unable to be confirmed by gas adsorption analysis. A possible explanation is the closed porosity occurred in the charcoals prepared with reaction time 120 min and 180 min, which is not accessible for gas adsorption analysis.

Figure 5b shows BET surface area of HT2 charcoals in fast heating treatment in relationship to a heating rate. BET surface area of HT2 charcoals slightly increased with the increase of heating rate from 75 to 250°C/min. Both BET surface area of HT2 charcoals prepared with heating rate 75 and 250°C/min were comparable to HT2 charcoals in slow heating treatment. However, the increase of heating rate from 250 to 1000 and 2000°C/min abruptly decreased BET surface area of HT2 charcoals with the value only 6.2 and 1.3 m²/g, respectively. Fast heating may cause the surface of carbonized wood melted during the heat treatment with the rate of thousand degrees per minute (Diebold and Bridgwater 1997) which close or block the micropore which become inaccessible for gas adsorption analysis.

Figure 6a and b show the adsorption isotherm of HT2 charcoals in slow- and fast heating treatment. The shape of adsorption isotherms suggested that these charcoals exhibited Type I adsorption isotherms (Marsh and Rodriguez-Reinoso 2006) which containing microporosity only. Type I adsorption isotherm are typical of microporous solids in that micropore filling occurs significantly at relatively low partial pressure of < 0.1 P/Po.[10]. HT2 charcoals in fast heating treatment prepared with reaction time 1000 and 2000°C/min showed a low adsorption isotherm.
Microstructural Change with Heat Treatments

Figure 7 shows the Raman spectra of HT1 and HT2 charcoals which exhibited D-band and G-band attributed to graphitic sp²-bonded carbon. D-band is usually associated with the disordered structure of turbostratic carbon and G-band corresponds to the crystalline vibration (Ishimaru et al. 2007; Paris 2005). HT1 charcoal prepared from a heat treatment at 300°C exhibited broad D-band and G-band with full width at half maximum (FWHM) of 279 and 125 cm⁻¹, respectively, corresponding to a typical of least ordered carbon material. The appearing of D-band and G-band on HT1 charcoal showed the aromatization occurred at heat treatment at 300°C. HT2 charcoal prepared from a heat treatment at 300°C and followed by re-carbonization at 600°C showed narrowing D-band and G-band with FWHM of 206 and 82 cm⁻¹ at positions shifted to 1362 and 1601, indicating the ordering of microstructure in the charcoal which was agreed with a previous study (Rodriguez-Reinoso and Molina-Sabio 1992) that reported the charcoal structure was consolidated at temperature range between 770-1120K. However the FWHM of D-band of HT2 charcoal was broader than that of carbonized wood reported previously (Yamauchi and Kurimoto Y 2003).
Effect of Slow Heating Treatment on Microstructural Change

Figure 8a and b show the plot of D-band and G-band width (FWHM) of HT1 and HT2 charcoals in slow heating treatment in relationship to reaction times. The FWHM of D-band and G-band of HT1 charcoals decreased with the increasing reaction time. The narrowing of band width suggests the structural arrangement or the ordering carbon element with the increase in reaction time (Yamauchi and Kurimoto Y 2003). The D-band and G-band of HT2 charcoals slow-heat-treated were narrower than that of HT1 charcoals slow-heat-treated for the reaction time of 60 and 120 min. It is suggested that re-carbonization at 600ºC, as second step of the slow heat treatment on HT2 charcoals contributed to the ordering microstructure. Prolonged reaction time of 180 min influenced significantly on the ordering of microstructure HT1 charcoal slow-heat-treated.

Effect of Fast Heating Treatment on Microstructural Change

The FWHM of D-band and G-band of HT1 and HT2 charcoals in fast-heating treatment in relationship to heating rates are shown in Fig. 9a and b. The FWHM of D-band and G-band of both HT1 and HT2 charcoals in fast heating...
treatment became broader with the increasing heating rate. Meanwhile, HT1 charcoals fast-heat-treated showed broader D-band which indicated strong defects occurred compared with fast-heat-treated HT2 charcoals. As discussed on previously, fast heating treatment promotes the simultaneous chemical reactions including fragmentation and re-polymerization leading to a large formation of volatile matter (Kurosaki et al. 2003). HT1 charcoals released more intensively volatile matters during the fast heating treatment than that of HT2 charcoals. It is suggested that the fast volatilization contributed to more defects occurrence in the microstructure of HT1 charcoals. The defects in the microstructure of the charcoals intensively occurred with the increase of heating rate as shown by the broadening of the FWHM of D band. Therefore the fast heat treatments produced charcoal with lesser microstructural ordering than that of the slow heat treatments.

Figure 10a and b show the coherence length ($L_a$) of HT1 and HT2 charcoals in slow- and fast heating treatments, respectively. The $L_a$ was reported to dependence on temperature with a decreasing trend with the increase of temperature (Paris 2005). In this study, $L_a$ was insensitive to the increasing reaction time in slow heating treatments, as shown in Fig 10a and to the increasing heating rate in fast heating treatments, as shown in Fig 10b. HT1 and HT2 charcoals slow heat-treated at 10ºC/min up to 700ºC possessed higher $L_a$ than that of HT1 and HT2 charcoals fast-heat-treated. It is suggested that the different chemical reaction occurred during the volatilization in the slow- and fast heating treatment as discussed previously may affect the growth of turbostratic crystallites

![Figure 9](image)

Figure 9. FWHM of D-band and G-band of a HT1 and b HT2 charcoals in fast heating treatment with the function of heating rate. The meaning of HT1 and HT2 is referred to that in Table 1.
Figure 10. Coherence Length (Lc) of HT1 and HT2 charcoals a in slow- and b fast heat-treatment. Filled circles, HT1 chars; empty squares, HT2 chars. The meaning of HT1 and HT2 is referred to that in Table 1.

Conclusions

A slow heating treatment up to 700°C on HT1 charcoal with the increasing reaction time for 120 and 180 min lead to the widening of micropore diameter without the opening of new micropores indicating a reducing porosity and with the ordering microstructure comparing with those in HT1 charcoal with the reaction time for 60 min. Fast heating treatment on HT1 charcoal created micropore with narrower diameter and wider size distribution, but with lesser porosity and the disordering microstructure comparing with those in HT1 charcoal slow heat treated. In fast heating treatment on HT1 charcoal, the increase of heating rate produced charcoal with the decreasing micropore diameter and quite similar micropore size distribution, but with the disordering microstructure.

Re-carbonization at 600°C in the preparation of HT2 charcoal contributed on the opening the new micropore with average diameter around 0.452-0.474 nm and on the ordering microstructure in the charcoals after the slow heat treatment up to 700°C for 120 and 180 min. But some parts of the micropore in HT2 charcoal slow heat treated were inaccessible for N₂ gas. The increase of heating rate in fast heating treatment of HT2 charcoal was unchanged the distribution of micropore size and affected on the disordering microstructure. But BET surface area of HT2 charcoals slightly increased with the increase of heating rate from 75 to 250°C/min then decreased with the increase of heating rate to 1000 and 2000°C/min.

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