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Preparation of activated carbon from forest and agricultural residues through CO_2 activation

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Abstract

An attempt was made to convert some agricultural and forest residues, including oak wood waste, corn hulls, and corn stover, into activated carbons. Microporous activated carbons were produced by physical activation with CO_2 as the activation agent; specifically, the effects of activation temperature and duration were explored. The resultant activated carbons were characterized by measuring their porosities and pore size distributions. The activated carbons produced had the BET surface areas ranging from 400 to 1000 m^2/g, and ratios of micropore volume to total pore volume ranging from 0.38 to 0.66. The pore size distributions (PSDs) were narrow and peaked approximately at a pore diameter of 0.5 nm. Both the surface area and the nature of porosity were significantly affected by the conditions of activation, the extent of which depended on the nature of the precursors. In general, the higher the activation temperature, the greater are the surface areas and micropore volumes of the resultant activated carbons. Nevertheless, various precursors were affected differently by the duration of activation. For oak, the longer the duration of activation, the greater the adsorption capacity of resultant activated carbons, and vice versa for corn hulls and corn stover.

Keywords: Activated carbon; CO_2; Oak; Corn hulls; Corn stover

1. Introduction

Activated carbons are highly porous materials that are found in numerous applications. For instance, they often serve as adsorbents, catalysts, and catalyst supports. The market for activated carbons is indeed vast. The global consumption of activated carbons was 750,000 t in 2002. The estimated growth of worldwide demand is 4–5% per year, with higher growth rates of 5–6% per year projected for the U.S. between 2002 and 2005 [1]. Currently, activated carbons are manufactured from a variety of starting materials, i.e., precursors, including different types of wood [2–6], nutshells [7–10], coal [11–13], and polymers [14,15]. These precursors are usually high in carbon content.

Commercially viable internal surface areas are generated in carbon precursors almost always through physical or chemical activation. The bulk of these internal surface areas is present in the micropores of the carbon. High surface areas are required for large sorptive capacities. In industrial practice, physical activation is carried out most frequently by burning off some of the raw carbon in an oxidizing environment to create micropores. The usual commercial choices of activation gas are steam, CO_2, air, or their mixtures. Activation normally takes place at temperatures between 700 and 1000 °C in steam and CO_2, and lower temperatures in air. Chemical activation was not considered in the current work. It entails the addition of materials such as zinc salts or phosphoric acid to the carbon precursors, thereby possibly generating secondary environmental pollution during disposal.

In the present study, CO_2 was selected as the activation gas: CO_2 is clean and easy to handle. Moreover, CO_2 has

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been a preferred choice as the activation gas on the laboratory scale: it facilitates control of the activation process due to the slow reaction rate at temperatures around 800 °C [16]. González et al. [17] conducted their investigation of CO_2 activation with both vertical and horizontal furnaces. They have concluded that a horizontal furnace is advantageous for micropore development.

Significant quantities of forest and agricultural residues are not recycled; consequently, they are wasted. The aim of this study is to investigate the conversion of some of these abundant forest and agricultural residues into high-quality activated carbons that might be commercially viable. The residues that served as precursors included oak wood waste, corn hulls, and corn stover. The activated carbons were prepared through carbonization in a fluidized bed followed by CO_2 activation, which was accompanied by heat treatment, over a range of activation conditions. The activation temperature and duration were explored for their effects on the properties of the activated carbons; this rendered it possible to determine optimum activation conditions. The activated carbons were characterized according to their physical adsorption properties by the conventional gas sorption methodology.

2. Experimental

2.1. Materials

The residues studied included oak wood waste, corn hulls, and corn stover. Table 1 lists the elemental analysis of each precursor. The table reveals that these residues are high in carbon content; this indicates that they are suitable precursors for preparing activated carbons.

Carbon dioxide with a purity of 99.5%, supplied from a compressed gas cylinder, was used as the activation gas for physical activation. Liquid nitrogen served as the coolant, and ultra-pure carrier grade compressed nitrogen with a purity of 99.9995% served as the adsorbate for the gas adsorption characterization.

2.2. Preparation of chars

The residues were fed into a fluidized sand-bed reactor, operating at approximately 500 °C, with inert nitrogen as the fluidizing agent. The typical feed rate of residue was 7 kg/h. The process typifies fast pyrolysis because of the high heating rates of the rather small particles (less than 1 mm). Once the residue was fed into the sand bed, it pyrolyzed, and the resulting gas stream and entrained fine char exited the freeboard of the reactor and entered two heated cyclones in series. The cyclones were enclosed in a heated jacket at 450 °C. The vast majority (95%+) of the char was collected in the first cyclone. At the end of the run, the char was removed from the cyclone collection bins, weighed, and sealed in containers. The remaining hot gas flow continued through a set of condensers and exited the system. The char yields from oak, corn hulls, and corn stover were 10, 32, and 22%, respectively. Table 1 also lists the results of elemental analysis of the resultant char obtained from each precursor. It is clear that the carbon content of char was significantly higher after carbonization, and the oxygen and hydrogen were lower. The surface areas and total pore volumes for the chars produced were very low as listed in Table 2.

Table 1
Elemental analysis of raw materials and their chars

Elements	Elements in the materials (wt.%)					
	Corn hull	Corn hull char	Corn stover	Corn stover char	Oak	Oak char
C	44.100	70.330	39.980	59.360	45.610	70.840
H	6.975	3.226	5.811	3.060	6.755	3.165
O	41.109	16.733	43.517	16.936	47.209	13.225

Table 2
Properties of char

	S_{BET} (m^2/g)	V (cm^3/g)
Oak	92	0.1458
Corn hulls	48	0.0581
Corn stover	38	0.0538

S_{BET}: surface area calculated using the BET equation; V: total pore volume estimated by converting the amount of N_2 gas adsorbed at a relative pressure of 0.99 to liquid volume of N_2.

Fig. 1 presents the pore size distributions (PSDs) of the chars determined by the non-local density functional theory (NLDFT). Obviously, the chars possess wide PSDs ranging from 0.4 to 6 nm centered around 3–4 nm.

2.3. Physical activation

Physical activation was performed in a quartz tube reactor vessel, which was contained in a horizontal three-zone laboratory tube furnace (model HTF55437C, Lindberg/Blue).

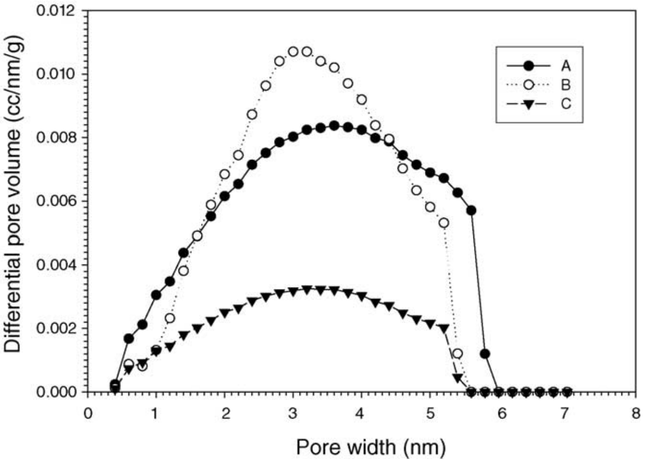

Fig. 1. Pore size distributions of chars: (A) char from oak; (B) char from corn hulls; (C) char from corn stover.

The reactor's inner diameter was 6 cm and its length was 60 cm. A quartz boat containing char was positioned centrally within the reactor. Temperature control for the reactor consisted of a three-zone temperature control system (model CC58434C, Lindberg/Blue); it contained three independent microprocessor-based digital temperature controllers. First, about 0.7 g of char was weighed to the nearest one-tenth of a milligram and placed in a thin layer in the sample boat. The boat was then centered in the furnace, which was turned on after CO_2 with a purity of 99.5% purged the system for about 2 h to exclude O_2. It took approximately 15 min for the furnace to reach the set temperature. After a specified duration for activation, the furnace was turned off. Carbon dioxide continued to flow through the system for about 2 h until the sample cooled down. The gas volumetric flow rate was maintained at 24 ml/min (STP) for all experimental runs. On completion, the resultant activated carbon was weighed to determine its burn-off. Here, the burn-off refers to the weight difference between the original char and the activated carbon divided by the weight of original char with both weights on a dry basis, i.e.,

$$\text{burn-off} = \frac{W_0 - W_1}{W_0} \times 100\% \tag{1}$$

where W_0 is the weight of char and W_1 the mass of the carbon after activation. The activation was performed at temperatures of 700 and 800 °C and activation durations of 1 and 2 h. Each experiment was replicated 3–5 times to ensure the reproducibility of the resultant data. The sample standard deviation, s, of each set of data in terms of weights never exceeded 0.05 g. The minimum of the mean, m, of each set of data was 0.6 g; this has given rise to the maximum relative standard deviation, or coefficient of variation, CV, of 0.08.

2.4. Characterization

N_2 adsorption isotherms of the activated carbons at 77 K were obtained with a gas sorption analyzer, Autosorb-1-MP-LP (Quantachrome). Each sample's surface area, pore volume, and PSD were determined from nitrogen adsorption data and the Quantachrome software (adsorption data were obtained over the relative pressure, p/p_0, range from 10^{-6} to 1 with nitrogen adsorption at 77 K). Two minutes were allowed for each adsorption point. The samples were degassed under high vacuum at 120 °C for 8 h prior to measurement. The surface area, S_{BET}, was calculated using the BET (Brunauer, Emmett and Teller) equation [18,19] from selected N_2 adsorption data within the range of relative pressure, p/p_0, from 0.1 to 0.3. The total pore volume, V, was estimated by converting the amount of N_2 gas adsorbed (expressed in cm^3/g STP) at a relative pressure of 0.99 to liquid volume of the adsorbate (N_2). The micropore volume, V_0, was determined according to the DR (Dubinin–Radushkevich) method [20–22]. Pore size distributions were computed by the NLDFT method [23–27] by minimizing the grand potential as a function of the fluid density profile.

3. Results and discussion

CO_2 activation was performed at temperatures of 700 and 800 °C and for durations of 1 and 2 h. Table 3 lists the activation conditions, burn-off, BET surface area, total pore volume, micropore volume, and ratio of micropore volume to total pore volume of each sample. Comparison of the data in Tables 2 and 3 reveals that the activation process has a notable effect on the properties of carbons. The results are further elaborated and analyzed in the following sections.

3.1. Burn-off

Fig. 2 depicts the relationships between the ratio of micropore volume to total pore volume, V_0/V, and the burn-off in activating char from oak, corn hulls, and corn stover at activation temperatures of 700 and 800 °C and for durations of 1 and 2 h. The data plotted in the figure have been extracted from those given in Table 3. Obviously, for chars from oak and corn hulls, the greater the burn-off, the smaller the V_0/V, up to the burn-off of about 43%; thereafter, this trend reversed, thereby indicating that the development and widening of microporosity exceeded the destruction of porosity. For char from corn stover, the greater the burn-off, the higher the V_0/V, up to a burn-off of 46%, due to the continuous development of microporosity. In a previous study [28], greater degrees of burn-off always generated activated carbons with higher microporosity, when producing activated carbons from cereal grains such as corn and hard red winter wheat by CO_2 activation.

3.2. Influence of activation temperature

The BET surface areas of all activated carbons obtained at 700 °C were lower than those obtained at 800 °C. For in-

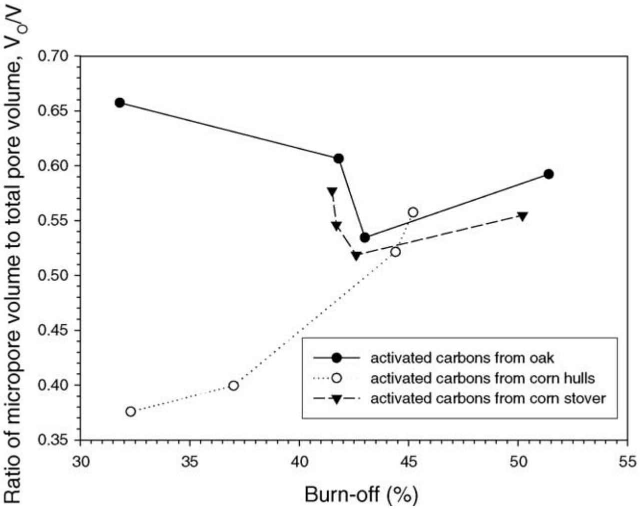

Fig. 2. Relationship between the ratio of micropore volume to total pore volume and the burn-off of the chars: the activation temperature and duration of individual data points from the left to the right are 700 °C and 1 h; 700 °C and 2 h; 800 °C and 1 h; and 800 °C and 2 h, respectively.

Table 3
Properties of activated carbons through carbon dioxide activation

Activation temperature (°C)	Activation time (h)	Burn-off (%)	S_{BET} (m^2/g)	V_0 (cm^3/g)	V (cm^3/g)	V_0/V
Oak						
700	1	31.8	642	0.2704	0.4113	0.6574
700	2	41.8	644	0.2450	0.4040	0.6064
800	1	43.0	845	0.3212	0.6011	0.5344
800	2	51.4	985	0.3792	0.6403	0.5922
Corn hulls						
700	1	32.3	977	0.3352	0.8923	0.3757
700	2	37.0	902	0.3284	0.8223	0.3994
800	1	44.4	1010	0.4348	0.8341	0.5213
800	2	45.2	975	0.3792	0.6803	0.5574
Corn stover						
700	1	41.5	660	0.2817	0.4879	0.5774
700	2	41.7	432	0.1818	0.3329	0.5461
800	1	42.6	712	0.2849	0.5494	0.5186
800	2	50.2	616	0.2343	0.4223	0.5548

S_{BET}: surface area calculated using the BET equation; V_0: micropore volumes determined according to the DR method; V: total pore volume estimated by converting the amount of N_2 gas adsorbed at a relative pressure of 0.99 to liquid volume of N_2; V_0/V: the ratio of micropore volume to total pore volume.

stance, the surface areas were 642 and 845 m^2/g for the activated carbons from oak for 1 h of activation at 700 and 800 °C, respectively (see Table 3). The extent of burn-off of products from the activation at 700 °C was less than the amount at 800 °C. Thus, the products from the former contained less volatile materials than those from the latter. The loss of volatile materials was probably responsible at least partially for the creation of both macro- and micropores, resulting in samples with high surface areas. In addition, the activation of char with CO_2 must have involved the C–CO_2 reaction [29]. This would lead to the removal of carbon atoms and also cause the burn-off, thereby contributing to the development of micropore structure.

The production of activated carbons with high adsorption capacity entails activating of chars at a high temperature, thus giving rise to the combined effects of volatile material loss and C–CO_2 reaction as demonstrated by the results from the current work. Similar results were obtained by Lua and Guo [30] who prepared activated carbons from oil-palm shell by CO_2 activation; the higher the activation temperature, the greater the surface areas of the resultant activated carbons. As can be discerned from the data in Table 3, both the surface areas and the micropore volumes were higher for all activated carbons obtained at the higher activation temperature compared to those obtained at the lower activation temperature. The ratios of micropore volume to total pore volume of activated carbons from corn hulls generated at 700 °C was lower than those of activated carbons prepared at 800 °C, thus indicating an increase in the fraction of micropore volume with increasing temperature. Sometimes, however, a higher temperature causes micropores to widen because it destroys the walls between adjacent pores, thereby resulting in the enlargement of pores. This leads to a decrease in the fraction of micropore volume and an increase in the total pore volume as exhibited by the samples from oak and corn stover. For example, when the activation lasted 1 h, the total pore volume of activated carbons from oak obtained at 700 °C was less than that obtained at 800 °C; the former was 0.4113 cm^3/g, and the latter was 0.6011 cm^3/g.

3.3. Influence of activation duration

The properties of carbons from oak obtained upon 1 and 2 h of activation at 700 °C were very similar (see Table 3). Fig. 3 also illustrates that their isotherms almost coincide. Apparently, the activation durations of 1 and 2 h did not appreciably affect the properties of activated carbons from oak at 700 °C. In contrast, the surface areas, total pore volume, and micropore volume of activated carbon obtained upon 1 h of activation were much less than those upon 2 h of activation at 800 °C. Obviously, the pore structure of carbons from oak altered substantially for different durations of activation at 800 °C.

As revealed by the data in Table 3, the surface areas, micropore pore volumes, and total pore volumes of activated carbons from chars generated from corn hulls as well as those from corn stover were appreciably greater after 1 h of activation than after 2 h of activation. This is in sharp contrast to the results from the activation of char from oak. Plausibly, in activating the chars from both corn hulls and corn stover, the rate of pore structure formation exceeded that of the destruction due to the pore enlargement and collapse at the earlier stage and vice versa at the later stage.

3.4. Comparison with blank experiments

Blank experiments were carried out to determine the effects of heating alone. Char placed in N_2 atmosphere was heated under the same experimental conditions as those adopted for the CO_2 activation experiments. Heat treatment alone led to burn-off of about 20% in comparison to 30–50% with CO_2 activation at 700 °C for a duration of 1 h. The sur-

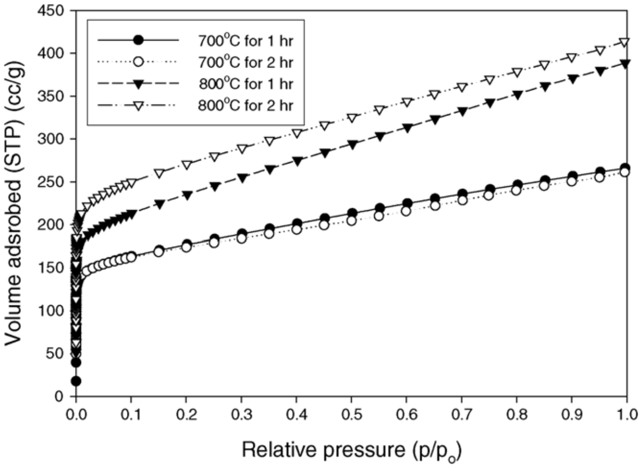

Fig. 3. Nitrogen adsorption (77 K) isotherms of activated carbons from oak through CO_2 activation.

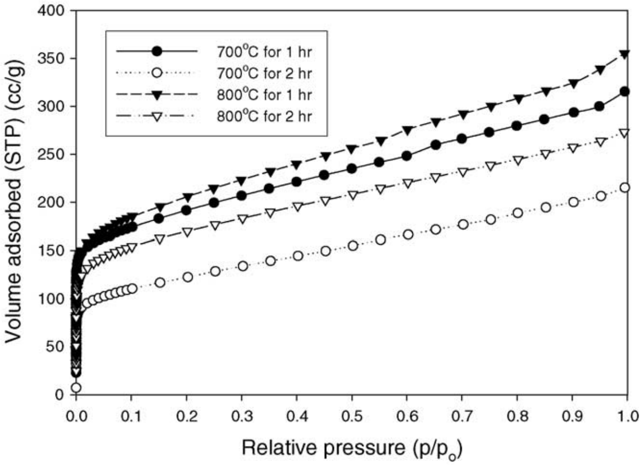

Fig. 5. Nitrogen adsorption (77 K) isotherms of activated carbons from corn stover through CO_2 activation.

face areas, total pore volumes, and micropore volumes of the activated carbons resulting from heat treatment alone were significantly lower than those of activated carbons obtained through CO_2 activation. For instance, the surface areas of the activated carbons from corn hulls generated at 700 °C with 1 h CO_2 activation was more than twice of that with heat treatment alone; the former was 977 m^2/g, and the latter, 411 m^2/g. According to Rodríguez-Reinoso et al. [31], CO_2 can open closed pores as well as widen existing pores by the activation, thus increasing the accessibility of the small pores to the molecules of an adsorbate. Consequently, nitrogen molecules can more easily penetrate into the interior of activated carbons where the pore structure is well developed.

3.5. Isotherms

Figs. 3–5 exhibit the N_2 adsorption isotherms for the activated carbons resulting from the CO_2 activation of chars at an activation temperature of either 700 or 800 °C, and for an activation duration of either 1 or 2 h. All these isotherms are clearly of Type I [32,33] that are typical of microporous materials. The flatter the isotherm, the more dominant the micropores, and vice versa. Regardless, the values of V_0/V, i.e., the fraction of micropore volume, usually exceed 0.5. Isotherms always contain two parts, i.e., adsorption isotherm and desorption isotherm; for convenience, only adsorption isotherms were plotted in Figs. 3–5. To illustrate the existence of the hysteresis loop, one of the isotherms in Fig. 3 has been expanded and is exhibited as Fig. 6.

As can be seen from Fig. 3 for oak, the development of mesoporosity relative to that of microporosity became appreciable at an activation temperature between 700 and 800 °C for a given activation duration: the higher the temperature, the steeper the isotherm branches except at the very low relative pressures (approximately less than 0.1). This is also evident from the higher values of V_0/V of the activated carbons ob-

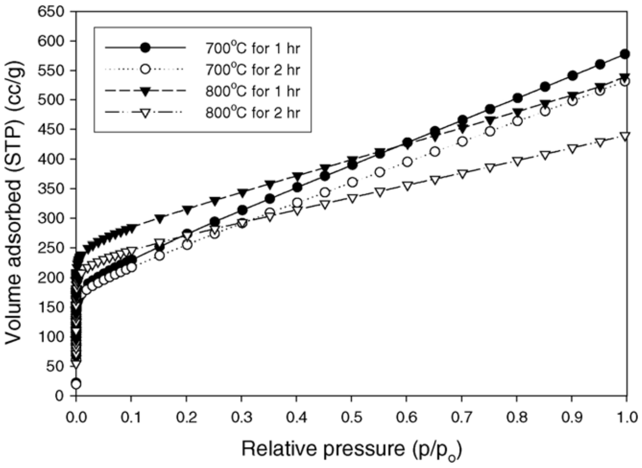

Fig. 4. Nitrogen adsorption (77 K) isotherms of activated carbons from corn hulls through CO_2 activation.

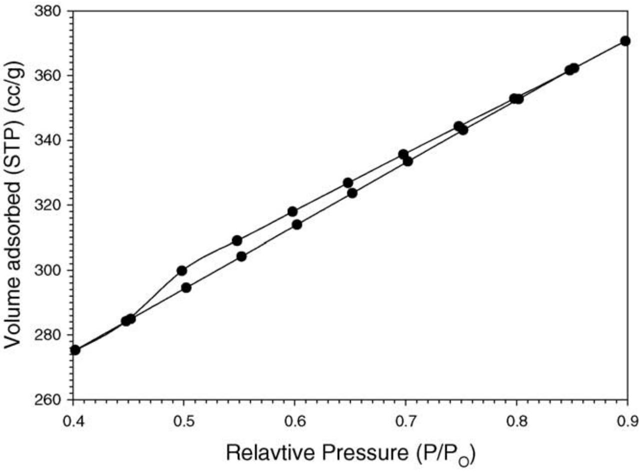

Fig. 6. Hysteresis loops shown in the nitrogen adsorption (77 K) isotherm of activated carbons from oak through CO_2 activation at 800 °C for 1 h: the lower branch represents the adsorption isotherm and the upper branch represents the desorption isotherm.

tained at 700 °C than those obtained at 800 °C, as can be discerned from the data in Table 3. At a given activation temperature, however, the slopes of isotherm branches of these activated carbons obtained for the durations of 1 and 2 h were almost identical.

As can be discerned from Fig. 4 for corn hulls, the lower the activation temperature, the steeper the isotherm branches for a given activation duration; the shorter the activation duration, the steeper the isotherm at a given activation temperature. The development of microporosity, therefore, must have been favored by the higher temperature and longer duration. This is also evident from the increase in V_0/V listed in Table 3. The higher temperature and longer duration often cause greater extent of burn-off, as discussed earlier in Section 3.1.

Fig. 5 for corn stover indicates that the higher the activation temperature, the steeper the isotherm branches at a given activation temperature. This appears to imply that the development of mesoporosity relative to that of microporosity became significant at temperatures between 700 and 800 °C. At a given activation temperature, the shorter the activation duration, the steeper the isotherm branches, thereby indicating that the microporosity continuously developed between the durations of 1 and 2 h.

The hysteresis loop is plotted in Fig. 6 for the isotherm of activated carbons from oak generated by CO_2 activation at 800 °C for 1 h. The N_2 adsorption isotherm was measured at 77 K. The hysteresis loops in the isotherms are associated with mesopores; for the given amount of the adsorbent in the pores, capillary evaporation during desorption takes place at a lower relative pressure than capillary condensation during adsorption. In general, hysteresis loops close prior to reaching a relative pressure of ca. 0.4 for N_2 adsorption at 77 K because of the termination of desorption from mesopores.

Three models are available for sorption hysteresis. These are the independent (single) pore model [34], the network model [34], and the disordered porous material model [35]. According to Thommes et al. [36], the third model presents the most realistic view. It takes into account the fact that the thermodynamics of the pore fluid is determined by phenomena spanning the complete porous system. Hysteresis is associated with thermodynamic metastability of low- and high-density phases of the pore fluid.

4. Conclusions

Activated carbons with well-developed microporosity and high surface areas can be manufactured from agricultural and forest residues such as corn hulls, corn stover and oak; they can be generated by CO_2 activation of the chars from these residues. The activated carbons produced had BET surface areas ranging from 400 to 1000 m^2/g, and ratios of micropore volume to total pore volume ranging from 0.38 to 0.66. The PSDs of activated carbons, calculated by the NLDFT method, are much narrower than those of the starting chars. The differential pore volume of each of these activated carbons peaks approximately at a pore diameter of 0.5 nm, thus indicating its potential as a gas adsorbent.

Both the surface area and the nature of porosity are significantly affected by the conditions of activation, the extent of which depends on the nature of the precursors. In general, the higher the activation temperature, the greater are the surface areas and micropore volumes of the resultant activated carbons. Nevertheless, various precursors are affected differently by the activation duration. For oak, the longer the activation duration, the greater the adsorption capacity of the resultant activated carbons, and vice versa for corn hulls and corn stover.

References

[1] The Economics of Activated Carbon, 7th ed., Roskill Information Services, London, UK, 2003, http://www.roskill.com/reports/activated.

[2] G.R. Moore, P.R. Blankenhorn, F.C. Beall, D.E. Kline, Wood Fiber 6 (1974) 193–199.

[3] P.R. Blankenhorn, D.R. Barnes, D.E. Kline, W.K. Murphy, Wood Sci. 11 (1978) 23–29.

[4] R.T. Baileys, Master Thesis, Pennsylvania State University, University Park, PA, 1979.

[5] R.T. Baileys, P.R. Blankenhorn, Wood Sci. 15 (1982) 19–28.

[6] K. Anuar, G. Joseph, F.B.H. Ahmad, Z. Zulkarnain, Z.M. Hussein, H. Abdullah, Res. J. Chem. Environ. 5 (2001) 21–24.

[7] H.H. Montenegro, J. Am. Oil Chem. Soc. 62 (1985) 259–261.

[8] A. Arulanantham, N. Balasubramanian, T.V. Ramakrishna, Mater. Finishing 87 (1989) 51–55.

[9] C.J. Kirubakaran, K. Krishnaiah, S.K. Seshadri, Ind. Eng. Chem. Res. 30 (1991) 2411–2416.

[10] R. Torregrosa, J.M. Martin-Marinez, Fuel 70 (1991) 1173–1180.

[11] F. Prinsloo, D.P.J. Opperman, C. Budeli, D. Hauman, Annu. Int. Pittsburgh Coal Conf. 6 (1999) 697–712.

[12] A. Linares-Solano, I. Martin-Gullon, C.S.D. Lecea, B. Serrano-Talavera, Fuel 79 (2000) 635–643.

[13] Y. Zou, B. Han, Energy Fuels 15 (2001) 1383–1386.

[14] N.T. Kartel, N.V. Gerasimenko, N.N. Tsyba, A.D. Nikolaichuk, G.A. Kovtun, Russ. J. Appl. Chem. 74 (2001) 1765–1767.

[15] S. Park, W.J. Jung, Colloid. Interf. Sci. 250 (2002) 196–200.

[16] F. Rodríguez-Reinoso, Controlled gasification of carbon and pore structure development, in: Fundamental Issues in Control of Carbon Gasification Reactivity, Kluwer Academic Publishers, Dordrecht, Boston, 1991.

[17] M.T. González, F. Rodríguez-Reinoso, A.N. Garcia, A. Marcílla, Carbon 35 (1997) 159–162.

[18] D.M. Yong, A.D. Crowell, Physical Adsorption of Gases, Butterworths, Washington, 1962.

[19] G.D. Parfitt, K.S.W. Sing, Characterization of Powder Surfaces, Academic Press, New York, 1976.

[20] M.M. Dubinin, Chem. Rev. 60 (1960) 235–241.

[21] M.M. Dubinin, J. Colloid. Interf. Sci. 23 (1967) 487–499.

[22] M.M. Dubinin, H.F.J. Stoeckli, Colloid. Interf. Sci. 75 (1980) 34–42.

[23] N.A. Seaton, J.P.R.B. Walton, N. Quirke, Carbon 27 (1989) 853–861.

[24] C. Lastoskie, K.E. Gubbins, N.J. Quirke, Phys. Chem. 97 (1993) 4786–4796.

[25] C. Lastoskie, K.E. Gubbins, N. Quirke, Pore size distribution analysis and networking: studies of microporous sorbents, in: Characterization

of Porous Solids. III. Studies in Surface Science and Catalysis, vol. 87, Elsevier, New York, 1994.
[26] J.P. Oliver, W.B. Conklin, M.V. Szombathely, Determinant ion of pore size distribution from density functional theory: a comparison of nitrogen and argon results, in: Characterization of Porous Solids. III. Studies in Surface Science and Catalysis, vol. 87, Elsevier, New York, 1994.
[27] B.P. Russell, M.D. LeVan, Carbon 32 (1994) 845–855.
[28] A. Venkatraman, W.P. Walawender, L.T. Fan, ACS Div. Fuel Chem. 41 (1996) 260–264.
[29] F. Rodríguez-Reinoso, M. Molina-Sabio, Carbon 30 (1992) 1111–1118.
[30] A.C. Lua, J. Guo, Langmuir 17 (2001) 7112–7117.
[31] F. Rodríguez-Reinoso, M. Molina-Sabio, M.T. González, Carbon 33 (1995) 15–23.
[32] S.J. Gregg, K.S.W. Sing, Adsorption, Surface area and Porosity, Academic Press, London, 1982.
[33] S. Lowell, J.E. Shields, Powder Surface Area and Porosity, 2nd ed., Chapman & Hall, London, 1984.
[34] D.H. Everett, Adsorption hysteresis, in: The Solid–Gas Interface, vol. 2, Marcel Decker, New York, 1967.
[35] L. Sarkisov, P.A. Monson, Capillary condensation and hysteresis in disordered porous materials, in: Characterization of Porous Solids V: Proceedings of the Fifth International Symposium on the Characterization of Porous Solids (COPS-V), Series: Studies in Surface Science and Catalysis, vol. 128, Elsevier, Amsterdam, New York, 2000.
[36] M. Thommes, R. Köhn, M. Fröba, Appl. Surf. Sci. 196 (2002) 239–249.