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POWDERED ACTIVATED CARBONS FROM *PINUS CARIBAEA* SAWDUST

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ABSTRACT

Activated carbons with different porous structures have been prepared from *Pinus caribaea* sawdust through the use of CO_2 and steam as activating agents. The evolution of the Brunauer-Emmett-Teller (BET) surface area upon activation becomes fairly similar in both cases, and values above 1000 m^2/g can be reached at high burn-off levels. Activation with steam produces a more developed porous structure, with a substantially higher contribution of mesoporosity, than does activation with CO_2. Increasing the activation temperature leads in both cases to a wide pore-size distribution. The presence of a well-developed mesoporosity makes the resulting products good candidates for adsorbents for water and wastewater treatment. For these purposes, the characteristics of these activated carbons are comparable to those used as commercial adsorbents in those fields, showing in the case of the steam-activated carbons, a somewhat higher mesopore volume than many of the commercial products while maintaining similar surface area values

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to them. The relative simplicity of the process makes the production of steam-activated carbons a feasible and economically valuable alternative for sawmill wastes.

Key Words: Sawdust waste; Activated carbon; Porous structure; Surface area; Pore size distribution

INTRODUCTION

Activated carbon adsorption is gaining application as a polishing technique for water and wastewater treatment. Residual chlorine, halomethanes, and a variety of pollutants in municipal and industrial effluents can be removed by carbon adsorption. Nevertheless, the extensive use of this operation is still limited by economic factors, mainly related to the relatively high prices of commercial adsorbents. Thus, less expensive products are needed, and the use of biomass wastes as raw materials is of potential interest in areas where they are available, especially in developing countries. Sawdust residues from wood-processing plants represent good candidates from which to produce inexpensive, powdered, activated carbons usable for water and wastewater cleaning if the sawdust is composed of appropriate porous structures.

In this work we report the results obtained on the preparation of different types of activated carbons from *Pinus caribaea* sawdust through carbonization followed by partial gasification of the resulting chars with CO_2 and with steam. Gasification was conducted at a low (725–750°C) and a high (850–875°C) temperature. A wide range of burn-off values, between approximately 25 and 75%, was used to obtain a variety of products with different surface area and pore size distributions, each of which may have use in different environmental applications, especially in water and wastewater treatment. The species investigated is fairly abundant in the region of Pinar del Rio, Cuba, where a number of sawmills operate. The development of potential uses for the wood-processing wastes has been an important socioeconomic concern.

The scientific literature on activated carbon preparation includes a number of references on the use of different lignocellulosic wastes as precursors. Experiments cited in references (1–4) are, among others, examples of this work. Previously, we reported our results on physical and chemical activation of waste lignin precipitated from kraft black liquors (5,6).

EXPERIMENTAL

The raw material used in this work was sawdust of *Pinus caribaea* from Guane (Pinar del Río, Cuba). The 0.6–1.6 mm particle size fraction was selected by sieving. The typical analyses of the raw material are summarized in Table 1.

Table 1. Typical Analyses of the Sawdust Used as Raw Material

Chemical Analysis (%, d.b.)	
Cellulose	48.0
Hemicellulose	29.2
Lignin	26.9
Proximate Analysis (%, d.b.)	
Fixed carbon	15.7
Volatile matter	83.9
Ash	0.40
Ultimate Analysis (%, d.a.f.)	
C	44.2
H	6.7
N	0.14
O (by difference)	48.9

The sawdust was carbonized in an N_2 atmosphere (2.5 cm^3/s continuous flow) and the resulting chars were activated. Both carbonization and activation were carried out in a conventional static furnace that consisted of a stainless steel tube of 750 mm in length and 75 mm i.d. and heated by an electric system with temperature and heating rate controls. Samples of approximately 10 g were used in the carbonization runs and approximately 2-g samples were used in the activation experiments. In both processes, the samples were placed in a flat ceramic pan located in the central part of the furnace tube where an isothermal zone of approximately 350 mm in length could be maintained. A thermocouple connected to the temperature controller was placed slightly above the sample.

In the carbonization runs, the system was heated at 10°C/min until a temperature of 700°C was reached. This final temperature was maintained for 2 hours and then the sample was cooled while the N_2 flow was maintained through the furnace tube. Different carbonization temperatures and times were previously checked to establish optimal experimental conditions. Typical yield (dry char to initial dry sawdust weight) in the carbonization step was between 22 and 23%. The C content of the chars was typically around 93%.

Activation was accomplished through partial gasification of the resulting chars. Two activating agents, CO_2 and steam, were studied. In all the experiments the furnace tube was first flushed with N_2 and then heated at 10°C/min up to the activation temperature. Once this temperature was reached, the feed was switched to CO_2 (2.5 cm^3 STP/s) or water (0.15 cm^3/min as liquid water) and maintained for different times to reach different burn-off values (activated carbon to initial char weight) and as a consequence a wide range of activated carbons were produced. Burn-off values in the range 27 through 77% were obtained.

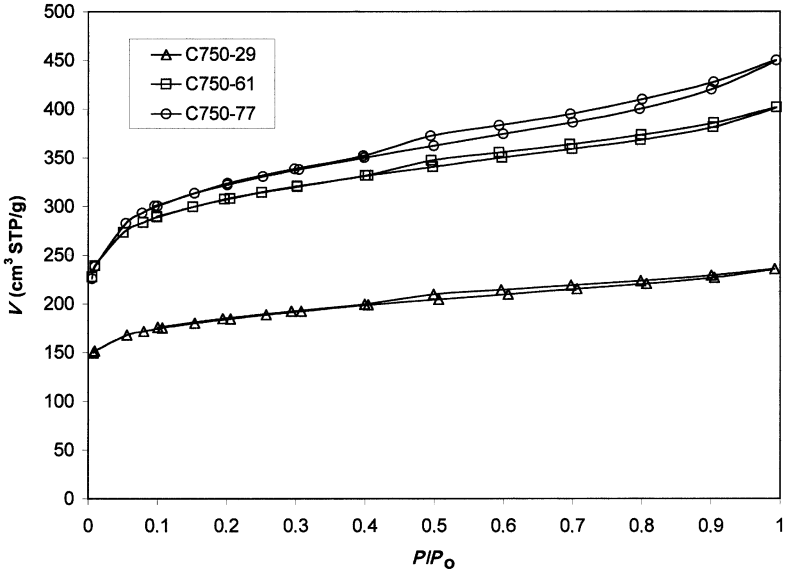
The porous structure of the activated carbons was characterized. The adsorption-desorption isotherms of N_2 at 77 K as well as the adsorption isotherms of CO_2 at 273 K were obtained through the use of a Quantachrome Autosorb-1 apparatus. The samples were previously outgassed at 453 K and 0.05 torr for 8 hours. Mercury porosimetry was carried out by means of a Carlo Erba porosimeter 4000. To have a picture of the changes in the external surface of the particles as activation develops, we used a JEOL JSM-840 instrument to obtain scanning electron microscope (SEM) graphs of the activated carbons.

From the N_2 isotherms, we obtained the BET surface area. Two values of micropore volume were obtained using the D-R equation (7) applied to the N_2 and to the CO_2 adsorption isotherms. The CO_2 isotherms are more related to the narrow microporosity of the activated carbons than are the N_2 isotherms, and the comparison of both micropore volumes serves to show more detailed information about the microporous structures of the activated carbons. The micropore size distribution was obtained from the MP method (8). The mesopore volume was assessed through a combination of mercury porosimetry and N_2 adsorption (isotherm) values. The porosimetry data allowed us to assess the volume of mesopores wider than 8 nm in diameter whereas the fraction between that size and 2 nm was assessed from the N_2 isotherm data. The mesopore size distribution within the low mesopore range (below 7 nm diameter) was obtained from the BJH method (9) applied to the desorption values of the N_2 isotherms. The α_s method was used to obtain the values of the external, nonmicroporous, surface area (10,11). The macropore volume of the activated carbons was obtained from the mercury intrusion curves. The shape of the mercury intrusion curves, in this case, may lead to some value errors caused from the uncertainty associated with the lower intrusion pressure region. We did not consider the volume values below 2 atm of intrusion pressure, which is a reasonable approximation based on analysis of curve shapes and the particle sizes of these powdered activated carbons.

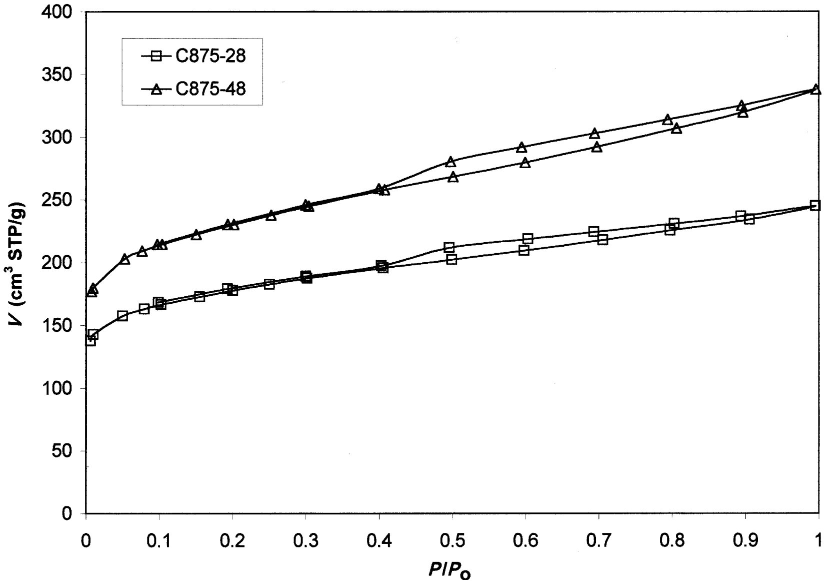
The activated carbons are identified throughout this article with a "C" (CO_2 activation) or an "S" (steam activation) followed by the activation temperature in °C and the burn-off value in percentages.

RESULTS AND DISCUSSION

Figure 1 shows the 77 K N_2 adsorption-desorption isotherms of the activated carbons obtained with CO_2 as activating agent at 1 relatively low (750°C) and 1 relatively high (875°C) temperature. The isotherms reveal in all the cases a mostly microporous structure that exhibits high relative adsorption values at very



(a)



(b)

Figure 1. N_2 adsorption-desorption isotherms of the CO_2-activated carbons: (a) 750°C and (b) 875°C.

low relative pressures. Nevertheless a widening of micropore size distribution and an increasing contribution of mesoporosity at increasing burn-off values are suggested from the shape of the curves.

The transition from the initial steep region of the isotherms to the linear branch becomes less sharp as burn-off values increase. This change in slope is indicative of the aforementioned widening of the micropores. To confirm and establish this in a more conclusive way, we obtained the CO_2 adsorption isotherms at 273 K and a final pressure of 1 atm (relative pressure close to 0.03). From these and use of the D-R equation we obtained the values of micropore volume, which are reported in Table 2, together with the micropore values derived from the D-R equation applied to the N_2 adsorption isotherms. The values determined by the isotherms provide a good value of the total micropore volume, whereas those from the equation correspond to the narrow micropores of approximately 1 to 2 times the molecular dimensions of the adsorbate, which are approximately 0.3–0.7 nm (12). Table 2 shows that both values of micropore volume are quite similar at low burn-off levels, whereas the differences among the larger volumes, obtained by N_2 isotherms, increase with increased burn-off values, confirming the aforementioned widening of the micropores. Figure 2, which shows the micropore size distribution obtained from the MP method for a high and a low burn-off carbon, also supports this conclusion.

Table 2 includes the values of apparent surface area of the activated carbons as obtained from the BET method. A significant increase can be observed as burn off increases, and values in the vicinity of 1000 m^2/g can be obtained at a approximate 65% burn off, which is equivalent to an approximate 8% total yield (activated dry carbon to initial dry sawdust weight).

Figure 3 shows the 77 K N_2 adsorption-desorption isotherms obtained for the activated carbons prepared from steam partial gasification of *Pinus caribaea* chars at 725 and 850°C. An important development of microporosity was observed upon activation, as revealed from the increase of N_2 uptake at low relative pressures as burn off increased. Nevertheless, some important differences arise

Table 2. Values of BET Surface Area (m^2/g) and Micropore Volume (cm^3/g) of the CO_2-Activated Carbons

Carbon	BET Surface Area	V_{mic} (N_2)	V_{mic} (CO_2)
C750-29	575	0.27	0.25
C750-61	969	0.46	0.35
C750-77	1021	0.48	0.33
C875-28	631	0.26	0.24
C875-48	783	0.38	0.30

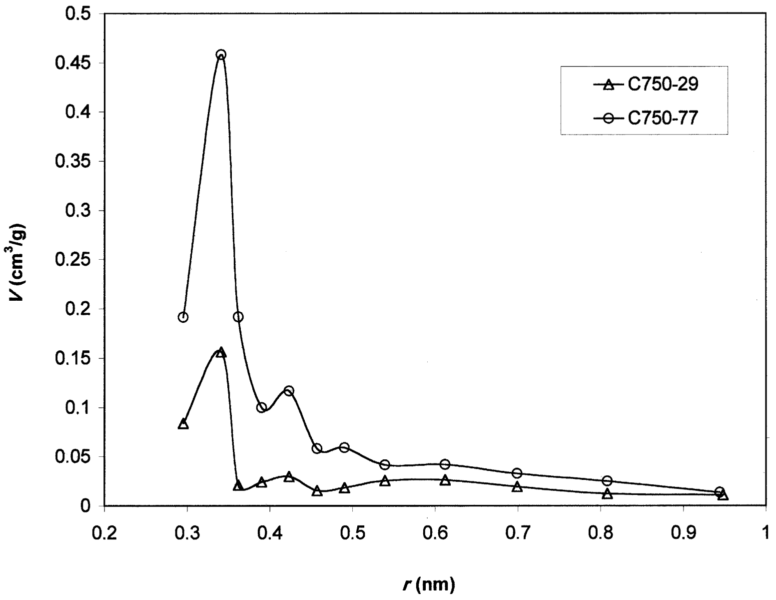


Figure 2. Micropore size distribution of CO_2-activated carbons at low and high burn off.

when the values from steam-activated carbons are compared with those from CO_2 activation.

The isotherms for steam-activated carbon clearly show a more rounded "knee" even at low burn-off degrees, which indicates a broader micropore size distribution. To better establish this, we proceeded as with the CO_2-activated carbons. Table 3 summarizes the results obtained concerning the microporosity and

Table 3. Values of Surface Area (m^2/g) and Micropore Volume (cm^3/g) of the Steam-Activated Carbons

Carbon	BET Surface Area	V_{mic} (N_2)	V_{mic} (CO_2)
S725-27	570	0.28	0.21
S725-47	732	0.31	0.24
S725-61	895	0.37	0.25
S725-71	1038	0.43	0.22
S850-29	617	0.27	0.22
S850-49	790	0.34	0.25
S850-68	937	0.38	0.24
S850-75	924	0.39	0.22

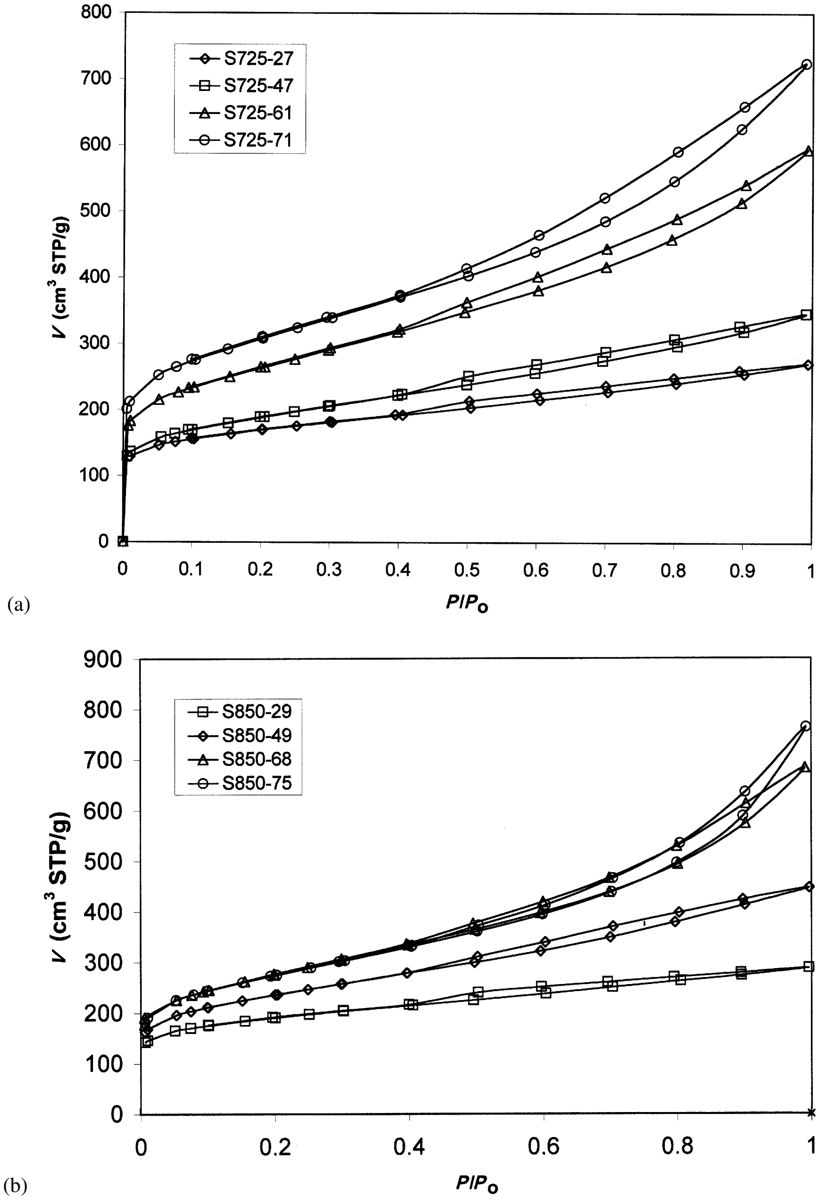


Figure 3. N_2 adsorption-desorption isotherms of the steam-activated carbons: (a) 725°C and (b) 850°C.

also includes the BET surface area values. As can be seen, the BET surface area values are quite similar to those obtained from CO_2 activation. The values of micropore volume are somewhat lower for the CO_2-activated carbon than for the carbon activated by steam; this result is particularly evident in the data of high burn-off products. The differences of the micropore size distribution are of some importance because even at low burn-off levels the micropore volumes obtained with N_2 are higher than those obtained with CO_2, the difference increasing with increasing burn off. The MP distribution curves confirmed this conclusion. As an example, we present in Fig. 4 the curves corresponding to activated carbons obtained at 725°C with low, medium, and high degrees of activation (burn-off). The widening of the microporous structure as steam activation proceeds is clearly seen, as is a significant broadening of the micropore size distribution, relative to CO_2-activated carbons. These differences between CO_2 and steam activation with regard to the development of microporosity are in agreement with the results of previous works in which other precursors of different natures were used (13,14).

An important feature of activated carbons is a well-developed mesoporosity because this has practical implications with regard to their potential uses as adsorbents, particularly in liquid-phase applications, such as water and wastewater treatment. The presence of a sufficient number of mesopores (transition pores) is a common requirement for commercial adsorbents used in treating water. Meso-

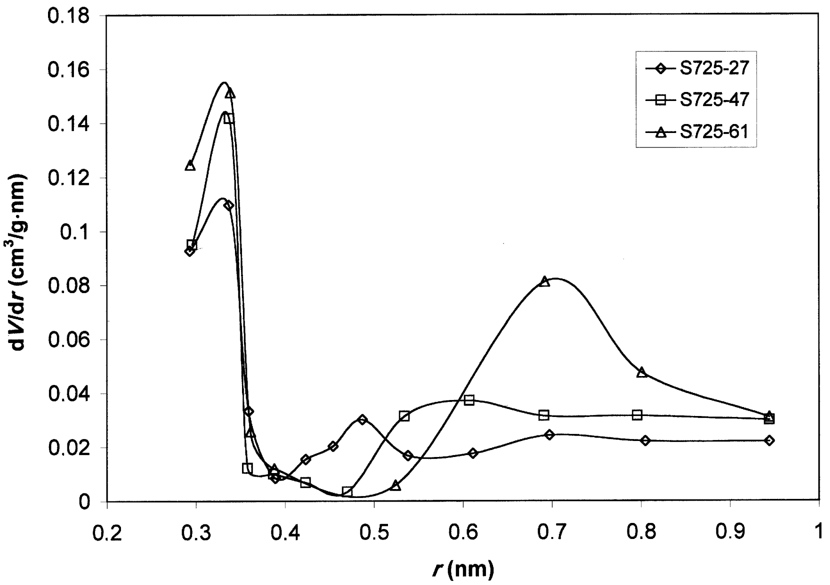


Figure 4. Micropore size distribution of steam-activated carbons.

pores eliminate or diminish diffusional limitations that may lead to the need for very high contact times and/or inefficient use of the adsorbent.

The isotherms of Figs. 1 and 3 clearly reveal a development of mesoporosity upon activation. The increase of the slope of the linear branch of the adsorption curves as burn off increases and the presence of increasingly wider hysteresis loops, which are more evident in the case of steam activation than in CO_2 activation, support this conclusion.

To acquire conclusive knowledge of the mesoporous structure of these activated carbons, we obtained the mercury intrusion curves. From them and the N_2 adsorption isotherms we calculated the mesopore volume values reported in Table 4. As can be seen, the development of mesoporosity remains moderate in the CO_2-activated carbons, whereas the steam-activated carbons show values of mesopore volume well above those of many of the commercial adsorbents presently used in water and wastewater treatment applications (15).

Besides the higher mesopore volumes, the steam-activated carbons show a wider distribution of mesopore size; the difference becomes more pronounced as burn off increases. This can be seen from the values of Table 4, in which we have included the fraction of mesopore volume corresponding to mesopores smaller than 7 nm diameter (low mesopore range). Figure 5 presents the size distribution curves within this low mesopore range as obtained from the BJH method.

Thus, the results of the activated-carbon characterizations led to the conclusion that steam activation produces a more open porous structure than does CO_2 activation. The steam-activated carbons have a wider distribution of micropore size as well as higher values of mesopore volume, and they have a wider size distribution within the whole mesopore range. On looking at the gasification reaction, which gives rise to the development of porosity, we note that steam has a higher reactivity than CO_2 (16,17) and thus particle-diffusion limitations are more likely in steam activation. Under steam activation, gasification takes place according to the relative accessibility of active sites, which gives rise to the devel-

Table 4. Values of Mesopore Volume (cm^3/g) and Fraction of Low-Size Mesopores, F_{lm} (% of the Total Mesopore Volume)

Carbon	V_{meso}	F_{lm}	Carbon	V_{meso}	F_{lm}
C750-29	0.064	50.2	C875-28	0.075	46.7
C750-61	0.10	48.4	C875-48	0.21	42.5
C750-77	0.17	49.5			
S725-27	0.15	59.3	S850-29	0.11	49.5
S725-47	0.23	45.2	S850-49	0.30	39.7
S725-61	0.35	39.3	S850-68	0.58	34.8
S725-71	0.52	37.7	S850-75	0.60	33.2

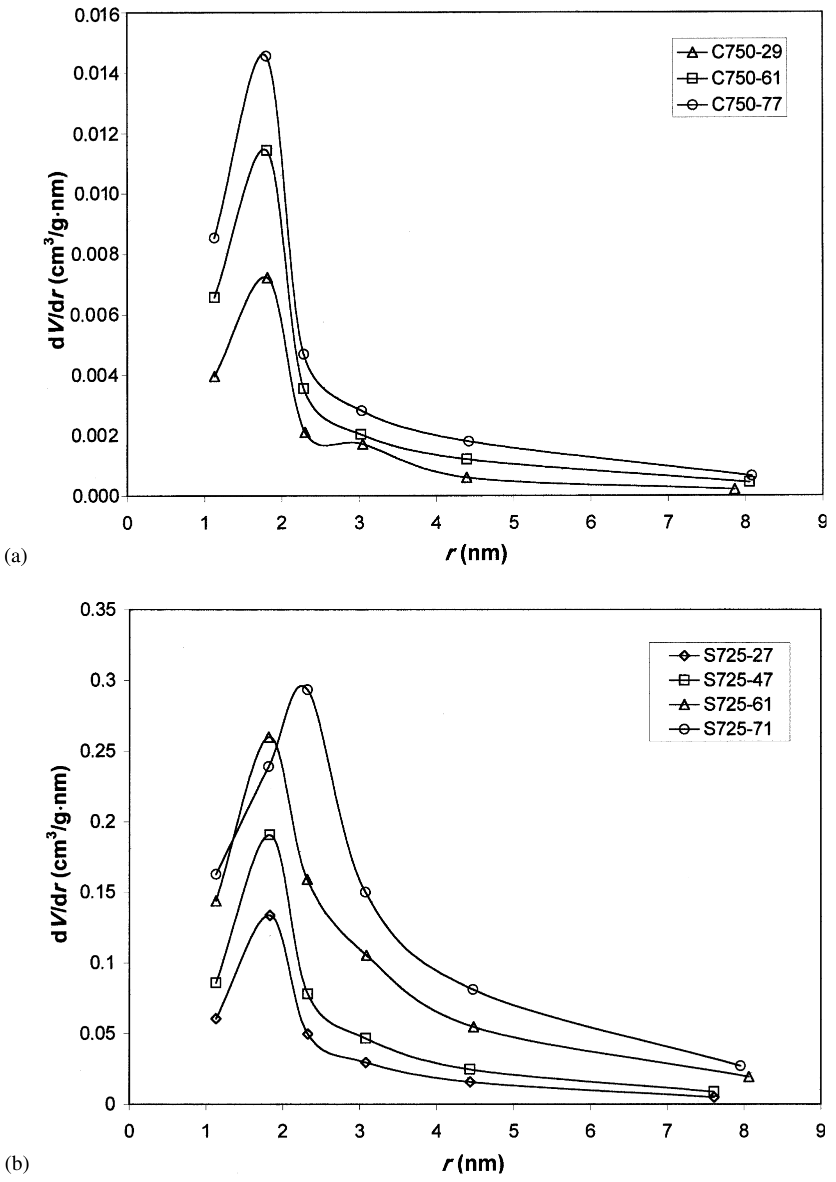


Figure 5. Mesopore size distribution of (a) CO_2- and (b) steam-activated carbons within the low mesopore range.

opment of a wider porous structure. Walker (18) attributes the differences between CO_2 and steam activation to a stronger inhibiting effect of H_2 in the steam-gasification reaction than the inhibition by CO in CO_2 gasification. This inhibition results in a less uniform reaction, which causes a wider pore size distribution. However, the inhibiting effects of CO_2 and H_2 have been a matter of controversy (19).

As indicated before, both steam and CO_2 activation of the raw material used in this work led to quite similar developments of total surface area (measured as BET surface area), and the distribution of this surface area must be characterized for researchers to gain a better knowledge of the structural characteristics of these activated carbons. We obtained the values of the external surface area, that is, the fraction of surface area not associated with the microporosity. This can be estimated from the slope of the linear multilayer region of the α_s plots. The values obtained, which are reported in Table 5, confirm the former results for the more open porous structure of the steam-activated carbons relative to those activated with CO_2.

One possible inconvenience for those who wish to use these activated carbons as powdered adsorbents in water and wastewater treatment arises from the adsorbent's relatively low density, which in the case of the steam-activated products at the higher burn-off values, falls below 0.4 g/cm^3 (as determined with mercury at 2 atm). This low density is mainly due to the high macropore volume, especially in the case of steam activation. Table 6 summarizes the macropore volumes of the activated carbons obtained from mercury porosimetry. A sharp increase of macropore volume is noticed at burn-off values above approximately 70%. Figure 6 shows SEM micrographs of 2 samples of highly activated products in which one can see the aforementioned high macroporosity and its quite homogeneous distribution over the particle surface. This high macroporosity arises in part from the precursor material itself. The char products, before activation through partial gasification, have a macropore volume close to 0.45 cm^3/g.

Table 5. Values of External Surface Area (m^2/g) (S) of the Activated Carbons

Carbon	S_{ext}	S_{ext}/S_{BET} (%)	Carbon	S_{ext}	S_{ext}/S_{BET} (%)
C750-29	72	11.1	C875-28	111	17.6
C750-61	114	11.8	C875-48	162	20.7
C750-77	165	16.2			
S725-27	162	28.4	S850-29	167	27.1
S725-47	324	44.3	S850-49	334	42.3
S725-61	519	58.0	S850-68	551	59.6
S725-71	635	67.7	S850-75	698	67.2

Table 6. Values of Macropore Volume (cm^3/g) of the Activated Carbons

Carbon	V_{macro}	Carbon	V_{macro}
C750-29	0.50	C875-28	0.52
C750-61	0.82	C875-48	0.67
S725-27	0.66	S850-29	0.63
S725-47	0.84	S850-49	0.82
S725-61	0.96	S850-68	1.00
S725-71	1.61	S850-75	1.97

With regard to the activation temperature, no significant effect was noticed on the development of the porous structure as far as total surface area and micropore volume are concerned. The meso- and macroporosity seem to be slightly more developed at higher activation temperature, but this cannot be conclusively established.

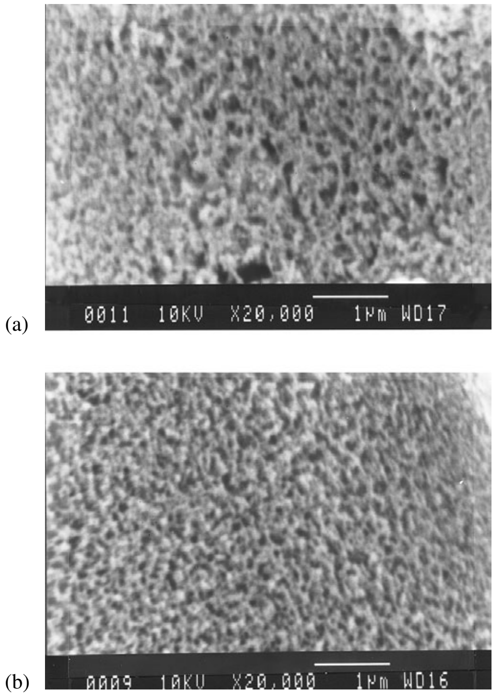


Figure 6. SEM micrographs of (a) CO_2- and (b) steam-activated carbons at a high burn off.

Table 7. Values of the Methylene Blue Number (mg/g) of the Activated Carbons

Carbon	Methylene Blue Number
C750-77	220
S725-27	111
S725-71	241
S850-29	128
S850-75	392

The methylene blue number is a widely used parameter for assessment of activated carbons for water and wastewater treatment. In Table 7 we report the values of this parameter obtained in this study. These values fall well within the common range of commercial activated carbons.

CONCLUSIONS

The manufacture of activated carbon provides an interesting way of adding economic value to sawmill wastes. The results of this work prove that *Pinus caribaea* sawdust can be transformed into activated carbons usable for water and wastewater treatment. The process follows a well-known and established technology consisting of carbonization and controlled partial gasification with steam or CO_2. The use of steam enhances the development of mesoporosity, which in the case of the precursor used in this work reaches values well above those of many of the commercial adsorbents presently used in the aforementioned fields, while maintaining comparable surface area values (15). This feature is of practical relevance because it allows the adsorption process to be carried out at relatively low contact times without losing adsorption capacity.

The activated carbons prepared from the sawdust are in powdered form, which would limit their application as liquid-phase adsorbents in stirred tank operations. For use in column applications, they must be pelletized to a convenient particle size. Further work must be done in which we will check the possibility of using pitch from wood carbonization as a binder.

The high macropore volume of these activated carbons leads to relatively low values of apparent density, which may be inconvenient with regard to the potential use of the powdered form in water and wastewater treatment. This problem can be controlled by using a CO_2-steam mixture as activating gas with a convenient steam partial pressure. Under such conditions the rate of the steam-gasification reaction is lower so that it becomes more comparable to that of particle diffusion. This decreases the relative importance of gasification in the outer part of the carbon particles, which are responsible for macropore creation.

The activation process could even be completed with combustion gases. According to our previous results with some other related precursors (4), the presence of up to approximately 5% O_2 in those gases has had very little effect on the porous structure of the activated carbons.

Besides water and wastewater treatment, these activated carbons may have other environmental applications, such as in gas cleaning processes, where the microporous carbons obtained from CO_2 activation would be preferable over those of steam. Nevertheless, the mesoporosity of the steam-activated carbons is again of practical interest in some emerging applications, such as the removal of dioxins and furans from the off-gases of incineration plants. In gas-phase operations the activated carbons could be directly used in powdered form (injection in ducts or settling chambers) or after agglomeration to granules or channeled monolites of convenient size. The direct application of powdered activated carbon is gaining interest for situations in which high flow-rate gas streams must be treated. In this case, the mesoporosity of the carbon particles becomes an important feature.

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