Study of commercial wood charcoals for the preparation of carbon adsorbents

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Abstract

We have studied the chemical composition and the physical characteristics of wood charcoals manufactured in Extremadura (Spain) as far as their potential production of carbon adsorbents is concerned. The samples were characterized by proximate and ultimate analyses, densimetric measurements, adsorption (carbon dioxide, 273 K; nitrogen, 77 K) and scanning electron microscopy. The characteristics of the wood charcoals depend not only on the wood used, but also the carbonization system. In particular, the eucalyptus charcoal produced in continuous furnace has interesting characteristics: the lowest ash content ($\sim$2%), the highest fixed carbon content ($\sim$90%), the highest total pore volume ($\sim$1 cm^3 g^{-1}) and an initial development of its pore structure in the range of narrow micropores to wide macropores.
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1. Introduction

According to the International Union of Pure and Applied Chemistry (IUPAC), charcoal is a traditional term for a char obtained from wood, peat, coal or some related natural organic materials [1]. This solid product is obtained by carbonization, a process by which solid residues with an increasing content of the element carbon are formed from organic material, usually by pyrolysis in an inert atmosphere [1].

Wood can be viewed as a polymeric structure consisting of carbohydrates (cellulose and hemicellulose) and lignin, with small amounts of extraneous organic chemicals and minerals [2]. Consequently, it is reasonable to expect that after wood carbonization will give rise to a solid mainly composed of carbon atoms, heteroatoms (mainly hydrogen and oxygen) and mineral matter (usually given as ash content). Such a solid will be wood charcoal.

The pyrolysis of lignocellulosic materials is a complex process; a variety of products result, which are broadly classified as char, tar, volatile (liquids) and gases [3]. Heating

wood to a temperature slightly above 100 °C already initiates some thermal decomposition; the hemicelluloses are degraded at 200–260 °C; the cellulose at 240–350 °C and the lignin at 280–500 °C [4]. As described in earlier works [5,6], volatile matter is lost during the wood carbonization and it is formed a carbon skeleton and a rudimentary pore structure. Moreover, it must be taken into account that the residual elementary carbon atoms, visualized as stacks of flat aromatic sheets cross-linked in a random manner [7], leave interstices which may become filled with the tars and other decomposition products or at least partially blocked by the disorganized carbon [8]. The surface of non-graphitized carbons, such as wood charcoals, consists of the faces of graphene sheets and of the edges of such layers, with heteroatoms, in particular oxygen, which is predominantly located on the edges in the form of various functional groups [9,10].

Adsorption is of a great importance from a technological standpoint and for the characterization of a diverse range of powders and porous materials [11]. All industrial adsorbents are composed of very fine particles (nanometric particles) or are highly porous, with pore structures more or less organized which are composed of nanometric pores in internal widths: micropores (pores of internal width less than 2 nm), mesopores

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(pores of internal width between 2 and 50 nm) and macropores (pores of internal width greater than 50 nm) [11,12]. Wood charcoal is a porous carbon material with more or less adsorbent properties; pores in the range of micro- to macropores are expected on studying its pore structure.

Wood charcoal has been manufactured during millennia for different applications. In a historical perspective, the use of carbonized wood as an adsorbent material dates of ancient times [7,13]. The first activated carbons, under the name of Eponit, were manufactured in 1909 according to a patent of R.V. Ostrejko [14–16], by heating wood charcoal with steam and carbon dioxide in a special furnace [16]. Today activated carbon is considered an unique or exceptional versatile adsorbent [8,15]; it is the most versatile adsorbent because of its large surface area, polymodal (but essentially micro-porous) porous structure and surface chemistry [16]. In other words, activated carbon is a fascinating material [17].

In the field of carbon adsorbents, wood is widely used for industrial-scale production of activated carbons following either physical or chemical activation. Well now, wood charcoal is cheaper than activated carbon and may be sufficient as adsorbent for some applications. The characterization of a wood charcoal is necessary to evaluate its applicability as adsorbent. Obviously, the initial adsorption capacity of a wood charcoal may be enhanced by adequate treatments.

Pulido et al. [18] studied the removal of mercury and other heavy by carbonized wood from aqueous solutions of their salts. They found that mercury is preferentially removed, even better than using a commercial activated carbon. There is, however, few studies of this type.

In the last years, we have studied the use of commercial wood charcoals manufactured in Extremadura as adsorbents, mainly adsorption of radionuclides from aqueous solutions. This region currently leads the Spanish production of wood charcoal for use as barbecue charcoal and for the manufacture of barbecue charcoal briquettes. Such domestic fuels are used nationally and in other European countries [19]. The mean exportation of wood charcoal between 1996 and 2000 exceededs 70% of Spanish exports [20]. The exportation of wood charcoal manufactured in Extremadura for 2000 was 1.7×10^7 kg.

This paper presents a study of wood charcoals manufactured in different carbonization systems, and their characterization, for a potential production of carbon adsorbents.

2. Experimental

2.1. Commercial wood charcoals

Four commercial charcoals manufactured from holm-oak and eucalyptus woods have been characterized. Table 1 gives the notations used for the wood charcoals studied.

The first charcoal was manufactured by partial combustion in a carbonization system called "carbonera" (a charcoal kiln), which is completely craft, from holm-oak wood. This carbonization system is a carefully constructed firewood pile on a prepared piece of land. Its form is approximately semispherical, having a circular base and a height dependent on

Table 1
Manufacture of wood charcoals and notations

Sample	Wood	Carbonization system
EnC	Holm-oak	Charcoal kiln
EnHD	Holm-oak	Discontinuous brick furnace
EuHD	Eucalyptus	Discontinuous metallic furnace
EuHC	Eucalyptus	Continuous furnace similar to a Lambiotte furnace

the firewood mass to be carbonized; a charcoal kiln of 140,000 kg has a circle of radius about 8 m and a height about 4 m. It has well distributed hollows between the wood pieces for a adequate air circulation during the carbonization process; the thicker piece is vertically placed in the centre of the circle. The firewood pile is carefully covered with thin branches or other lignocellulosic materials to achieve a uniform support for a topsoil layer of about 10–20 cm; the topsoil acts as a physical barrier to separate the firewood to be carbonized from exterior air. A hole in the top of approximately 20 cm in diameter serves to initiate the partial combustion, putting in thin wood or wood charcoal burning, and furthermore as an initial smokestack. The length of the carbonization process is approximately of 4 weeks. This process is controlled by opening and closing of holes from the top to the base of charcoal kiln; covering with topsoil if takes place the formation of fissures and observing the smokes (white colour indicates that the carbonization process takes place without excess of air). After cooling, the charcoal kiln is carefully opened to separate the charcoal manufactured. The yield of holm-oak charcoal depends on the mass of holm-oak wood, the atmospheric conditions and the control during the carbonization process; it is about 20%.

The second and third charcoals were manufactured by partial combustion in two discontinuous furnaces from holm-oak and eucalyptus woods. Both furnaces are partially craft carbonization systems, having rectangular bases, walls con-structed with bricks or steel sheets, smokestacks and a regular distribution of channels close to the bases. In both furnaces, the walls act as physical barrier to separate the firewood to be carbonized from exterior air, and the channels serve to initiate the partial combustion and to admit atmospheric air during the wood carbonization. The total time of carbonization and subsequent cooling was about 3 weeks in both furnaces, but the yield of wood charcoal was different.

The fourth wood charcoal was industrially manufactured in a continuous furnace, similar to a Lambiotte furnace. The yield of charcoal from eucalyptus wood (moisture content 20%) was about 30%.

As-received wood charcoals were first crushed and sieved. The size fraction of 1–2 mm was used in this study.

2.2. Characterization of the samples

2.2.1. Chemical characterization
The chemical composition of the samples was determined by chemical analyses, as follows. In the determination of proximate composition, the moisture and volatile matter contents were determined using a Mettler (Toledo 851^e) thermobalance, the ash

content was obtained by heating the samples at 650 °C in air to constant mass in a muffle furnace, and the fixed carbon was estimated by difference. The determinations of the contents of carbon, hydrogen, nitrogen and sulphur were performed by using a LECO CHNS-932 elemental analyser, and the oxygen content was estimated by difference.

2.2.2. Physical characterization

The wood charcoals were characterized by densimetric measurements, adsorption (carbon dioxide, 273 K; nitrogen, 77 K) and scanning electron microscopy (SEM).

Three density values were determined: bulk density (ρ), density measured by mercury displacement (ρ_{Hg}) and density measured by helium displacement (ρ_{He}). The ρ values were calculated from the masses of particles making up a bed divided by the volume of the bed. The ρ_{Hg} values were determined as described in previous papers [21,22]. The ρ_{He} values were determined using a Quantachrome stereopycnometer, as described in the literature [23]. The total pore volume accessible to helium at room temperature, V_p (cm^3 g^{-1}), was obtained by the expression $V_p = 1/\rho_{Hg} - 1/\rho_{He}$ [16,24].

The micropore structure of the wood charcoal with the highest open pore volume (to helium) was characterized by adsorption (carbon dioxide, 273 K; nitrogen, 77 K) using a Micromeritic apparatus. The Dubinin–Radushkevich (DR) equation was applied in the modern formulation [25] to obtain the micropore volumes, $W_{DR}(CO_2)$ (cm^3 g^{-1}) and $W_{DR}(N_2)$ (cm^3 g^{-1}), as well as to obtain the characteristic energy values, $E_0(CO_2)$ (kJ mol^{-1}) and $E_0(N_2)$ (kJ mol^{-1}). Adsorption (nitrogen, 77 K) was also used to obtain the equivalent surface area, $S_{BET}(N_2)$ (m^2 g^{-1}), by applying the Brunauer, Emmett and Teller (BET) equation [26].

The non-micropore structure of the wood charcoals was studied by mercury porosimetry and scanning electron microscopy. As described in previous papers [21,22,27,28], the cumulative pore volume of macro- and mesopores, V_{Hg} (cm^3 g^{-1}), was determined by mercury porosimetry using a Quantachrome porosimeter (Autoscan-60); the macropore volume, V_{ma} (cm^3 g^{-1}), was regarded as being equal to the cumulative pore volume at the pore radius of 25 nm. As in previous papers [27,28], SEM micrographs were obtained by means of a JEOL (JSM-5 400) scanning microscope.

3. Results and discussion

3.1. Chemical composition

Data from the proximate and ultimate analyses are listed in Table 2. The analyses are given on dry basis since the moisture

Table 2
Chemical analyses of wood charcoals

Sample	Proximate analysis (%, dry basis)			Ultimate analysis (%, dry basis)				
	Volatile matter	Ash	Fixed carbon	C	H	N	S	O$_{diff.}$
EnC	23.70	5.00	71.30	73.67	3.08	0.80	0.00	17.45
EnHD	21.90	4.55	73.55	84.44	2.45	0.60	0.00	7.96
EuHD	11.21	2.17	86.62	88.56	2.39	0.51	0.00	6.37
EuHC	8.01	2.06	89.93	90.06	1.70	1.30	0.06	4.82

content of a wood charcoal depends both on its nature and storage ambient.

As can be seen from Table 2, the proximate composition depends not only on the starting wood, but also the carbonization system by which they were manufactured. The charcoals manufactured from holm-oak wood show higher values of volatile matter and of ash contents in comparison with the charcoals manufactured from eucalyptus wood. Consequently, the eucalyptus charcoals have the highest contents of fixed carbon.

Concerning the ultimate composition, the content values of carbon are consistent with those of fixed carbon; for each sample the fixed carbon is lower compared with the carbon content. The carbon content follows the sequence EnC < EnHD < EuHD < EuHC. Both hydrogen and oxygen contents follow the opposite sequence. As expected, the nitrogen content is low for each sample and the sulphur content is nil or very low.

Activated carbon consists, of course, principally of carbon (over 90% of its mass), oxygen (the second most abundant element of activated carbon) and other heteroelements (hydrogen, nitrogen, etc.), as well as mineral matter occluded in the pores (the ash content varies widely with the raw material used in the manufacture, up to 20%; for activated carbons made from wood the ash content is 3–8%) [29]. Taking into account these indications, it is interesting to note that the production in continuous furnace yields an eucalyptus charcoal with the best chemical composition as a precursor of activated carbon.

3.2. Physical characteristics

Table 3 lists the values of densities and of pore volumes determined for each wood charcoal. With regard to the bulk density, there are considerable differences between the charcoals EnC (0.43 g cm^{-3}) and EuHC (0.30 g cm^{-3}); bulk density values given in the literature for carbons used in gas adsorption range from 0.40 to 0.50 g cm^{-3}, while for carbons

Table 3
Densities and pore volumes of wood charcoals

Sample	ρ (g cm^{-3})	ρ_{Hg} (g cm^{-3})	ρ_{He} (g cm^{-3})	V_p (cm^3 g^{-1})	V_{ma} (cm^3 g^{-1})	V_{Hg} (cm^3 g^{-1})	$V_p - V_{Hg}$ (cm^3 g^{-1})
EnC	0.43	0.89	1.54	0.475	0.276	0.407	0.068
EnHD	0.37	0.65	1.42	0.834	0.565	0.797	0.037
EuHD	0.33	0.62	1.53	0.959	0.662	0.824	0.135
EuHC	0.30	0.61	1.68	1.044	0.661	0.864	0.180

used in decolourization, the range is 0.25–0.75 g cm^{-3} [16]. As for the mercury density, the values determined range from 0.6 to 0.9 g cm^{-3}; the values of this density for activated carbons range from 0.6 to 0.8 g cm^{-3} [30]. As for the helium density, the values determined range from 1.50 to 1.70 g cm^{-3}; for activated carbons the helium density values range from 2.0 to 2.10 g cm^{-3} [30]. The eucalyptus charcoal manufactured in continuous furnace has the lowest values of ρ and ρ_{Hg} and the highest ρ_{He} value.

The pore volume of activated carbon usually exceeds 0.2 cm^3 g^{-1} but in many instances it is greater than 1 cm^3 g^{-1} [14]. All the wood charcoals studied have relatively high values of open pore volume (to helium) (>0.400 cm^3 g^{-1}); the EuHC charcoal has the highest value of V_p (1.044 cm^3 g^{-1}) and the EnC charcoal has the lowest (0.475 cm^3 g^{-1}). The different V_p values fit in with the fact that both the volatile matter content and the ash content of holm-oak charcoals are higher than those of eucalyptus charcoals (Table 3). The V_p values mean that the porosity has evolved differently; the pores must contain different amounts of tarry matter or products of decomposition formed during the wood carbonization processes. Different mineral matter may also be occluded in the pores.

Table 3 also lists the $V_p - V_{Hg}$ differences. These differences indicate that the wood charcoals have different micropore structures accessible to helium at room temperature. In the case of EuHC charcoal, such difference has the maximum value, indicating a better development of its micropore structure in comparison with other charcoals.

The micropore structure of the EuHC charcoal has been studied by adsorption (carbon dioxide, 273 K; nitrogen, 77 K). The adsorption isotherms are plotted in Figs. 1 and 2. According to the classification of physisorption isotherms recommended by the IUPAC [12], the shape of the nitrogen isotherm (Fig. 2) may to be considered of type I, characteristic of microporous solids. According to the refined classification proposed by Rouquerol et al. [31], the nitrogen isotherm is of type Ib.

Because of the relatively low pressure range covered (up to $p/p^0 \approx 0.03$), adsorption of carbon dioxide is used to determine the exact volume of narrow micropores (up to two molecular
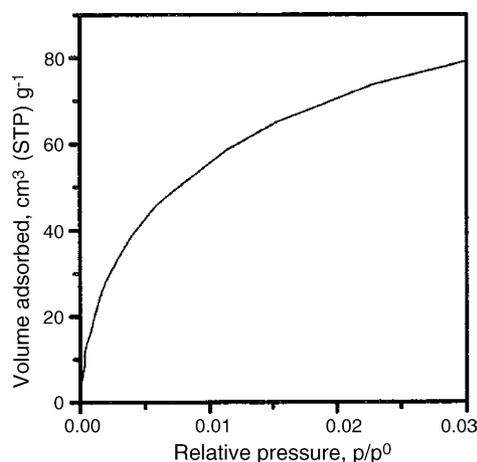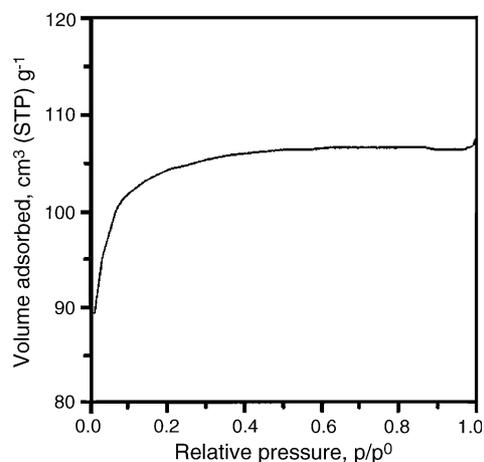

Fig. 2. Adsorption isotherm (nitrogen, 77 K) for EuHC.

dimensions, about 0.7 nm) and adsorption of nitrogen to determine the volume of wide micropores [16]. From Table 4, the micropore structure of the EuHC charcoal is mostly formed by narrow micropores, because $W_{DR}(CO_2) > W_{DR}(N_2)$, with significant values of narrow ($W_{DR}(CO_2) = 0.255$ cm^3 g^{-1}) and wide ($W_{DR}(N_2) = 0.165$ cm^3 g^{-1}) micropore volumes. The characteristic energy values determined (Table 4) are included in the range 30–35 to 17–18 kJ mol^{-1} given for activated carbons, which corresponds to average pore widths of approximately 0.4–2 nm [32]; $E_0(CO_2) > E_0(N_2)$, this means that each fraction of micropores has a different average pore width.

Concerning the non-micropore structure of the wood charcoals, the V_{Hg} values are considerable, ranging from 0.407 cm^3 g^{-1} for EnC charcoal to 0.864 cm^3 g^{-1} for EuHC charcoal (Table 3). The cumulative intrusion curves (Fig. 3) are almost coincident for the eucalyptus charcoals, but are very different for the holm-oak charcoals. Such curves show a wide range of pore widths and a bimodal size distribution, from pore radius values of approximately 3000 (EnC plot) and 5000 nm (EuHC plot) to approximately 2 nm. Clearly, the influence of the carbonization system on the non-micropore structure is not significant when the raw material is eucalyptus wood, while there are considerable differences when the raw material is holm-oak wood. The non-micropore structure is more developed in eucalyptus charcoals (EuHC ≈ EuHD) than in holm-oak charcoals (EnHD > EnC).

From Table 3 and Fig. 3, it can be seen that most of the pore volume determined by mercury porosimetry corresponds in each case to macropores; 0.276 cm^3 g^{-1} for EnC charcoal, 0.565 cm^3 g^{-1} for EnHD charcoal and approximately

Fig. 1. Adsorption isotherm (carbon dioxide, 273 K) for EuHC.

Table 4
Parameters of the DR equation and BET surface area

Sample	EuHC
$W_{DR}(CO_2)$ (cm^3 g^{-1})	0.255
$E_0(CO_2)$ (kJ mol^{-1})	31.45
$W_{DR}(N_2)$ (cm^3 g^{-1})	0.165
$E_0(N_2)$ (kJ mol^{-1})	21.40
$S_{BET}(N_2)$ (m^2 g^{-1})	387
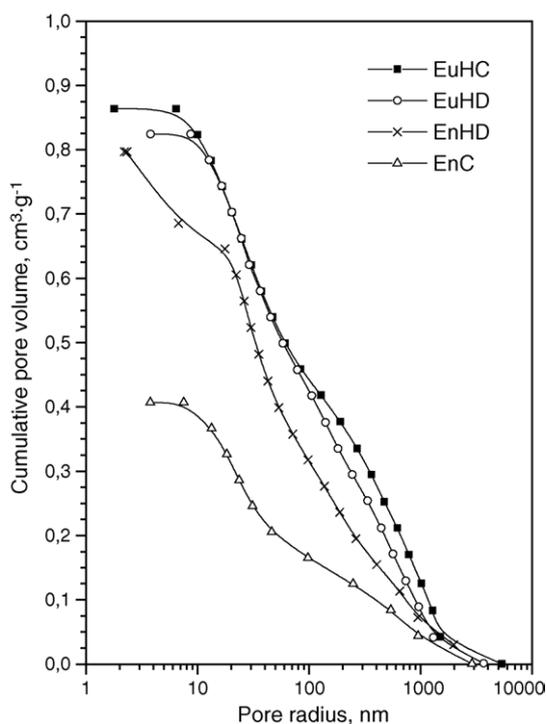
Fig. 3. Plots of the pore size distribution of wood charcoals determined by mercury porosimetry.

Fig. 4. SEM micrographs of two wood charcoals: EnC (above) and EuHC (below).

0.660 cm^3 g^{-1} for eucalyptus charcoals. These values are within the range of macropore volumes for activated carbons, usually 0.2–0.8 cm^3 g^{-1} [29]. In addition, the SEM micrographs (Fig. 4) show two different macropore structures. The macropore structure is more rudimentary in the case of EnC charcoal; mineral matter can be observed within the macropores of this sample. In contrast, the macropores of EuHC charcoal are very different in widths and lack mineral matter.

The great development of the macropore structure of the wood charcoals studied is interesting. In the literature it is pointed out that most of the adsorption takes place in the micropores of an activated carbon, but meso- and macropores play a very important role in any adsorption process; they serve as a passage for the adsorbate to the micropores, since only a few of these are placed on the outer surface of the carbon particle [16]. Wildman and Derbyshire [33] described and discussed the importance of the existence of an appreciable macropore volume in applications of activated carbons concerning vapour and liquid phase adsorption.

All industrial adsorbents (highly porous or composed of very fine particles) have large specific surface areas (generally well in excess of 100 m^2 g^{-1}) [11]. The equivalent surface area (BET method; nitrogen, 77 K) of EuHC charcoal is 387 m^2 g^{-1} (Table 4), which is relatively high compared with those of other charred products prepared in the laboratory scale. Values of 69 and 110 m^2 g^{-1} were obtained for charcoals prepared by treatment of rockrose and extracted rockrose in the atmosphere formed when the temperature increased [21,22]. For some lignocellulosic chars prepared by pyrolysis in a nitrogen atmosphere these maximum values have been reported: 359 and
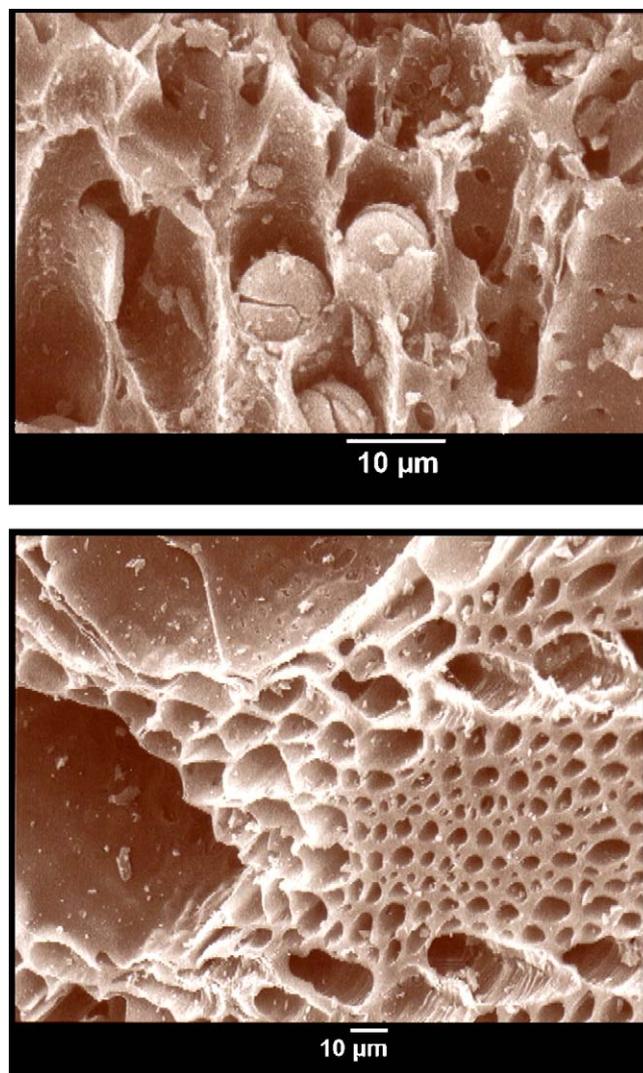
379 m^2 g^{-1} for chars prepared from rockrose and extracted rockrose [21,22]; 245 m^2 g^{-1} for an eucalyptus char [34] and 318 m^2 g^{-1} for a char prepared by pyrolysis of oil palm stones [35]. The inner specific surface area of an activated carbon is generally greater than 400 m^2 g^{-1} [14]; the equivalent surface area of EuCH charcoal indicates that the manufacture in continuous furnace yields an eucalyptus charcoal with an interesting initial equivalent surface area, which may be enhanced by activation.

4. Conclusions

For a potential production of carbon adsorbents, the wood charcoals manufactured in Extremadura present characteristics dependent not only on the starting wood, but also on the carbonization system. In particular, the production in continuous furnace yields an eucalyptus charcoal with interesting characteristics: the lowest ash content (∼2%), the highest fixed carbon content (∼90%), the highest total pore volume

($\sim$1 cm^3 g^{-1}) and an initial development of its pore structure in the range of narrow micropores to wide macropores.

Acknowledgements

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