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Activated carbon from vetiver roots: Gas and liquid adsorption studies

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Abstract

Large quantities of lignocellulosic residues result from the industrial production of essential oil from vetiver grass (*Vetiveria zizanioides*) roots. These residues could be used for the production of activated carbon. The yield of char obtained after vetiver roots pyrolysis follows an equation recently developed [A. Ouensanga, L. Largitte, M.A. Arsene, The dependence of char yield on the amounts of components in precursors for pyrolysed tropical fruit stones and seeds, Micropor. Mesopor. Mater. 59 (2003) 85–91]. The N_2 adsorption isotherm follows either the Freundlich law $K_F P^\alpha$ which is the small α equation limit of a Weibull shaped isotherm or the classical BET isotherm. The surface area of the activated carbons are determined using the BET method. The K_F value is proportional to the BET surface area. The α value increases slightly when the burn-off increases and also when there is a clear increase in the micropore distribution width.
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1. Introduction

Vetiver grass (*Vetiveria zizanioides*) is a tall (1–2 m), fast-growing, perennial tussock grass. It has a long (3–4 m) massive and complex root system. It is grown for soil conservation, water conservation and stabilization, and it is used for roof thatching, fodder for livestock. Volatile extracts of vetiver roots are used in the perfume, soap and related industries. Its root aromatic oil can also be used as a repellent for Formosan subterranean termite and represents a promising natural alternative for the control of this invasive pest [2]. Vetiver roots are grown in tropical areas including Haiti for industrial production of essential oil, resulting in lignocellulosic residues. After oil extraction, 98% of the starting material remains, leaving huge amounts of residues which are not used as industrial material, but burnt in fields or at the road side. Thus, investigations aiming to improve the value of such residues are of interest. To promote an economic utilization of this cheap and abundant agriculture waste, it is proposed to use it as a prospective starting material for the preparation of activated carbon (AC).

ACs are well-known adsorbents extensively used for purification and separation in many industrial processes and for effluent treatment, owing to their ability to adsorb a large variety of compounds from both gaseous and liquid streams. World consumption of ACs is steadily increasing, as applications concerning environmental pollution remediation are emerging. ACs can be virtually manufactured from any carbonaceous precursor, but the most commonly used raw materials are wood, coal, coconut shells and some polymers. The prospective demand of ACs has led to an increasing search toward plentiful, renewable supplies of low cost feedstock for ACs production. The suitability of several agricultural by-products, e.g. nutshells, fruit stones, sugarcane wastes [3–9] has been analyzed. Nevertheless, to our knowledge, the use of *Vetiveria zizanoides* as a precursor for activated carbons preparation has not been investigated previously.

The present work examines the feasibility of using *vetiver roots* as a potential feedstock for ACs preparation by pyrolysis and physical activation. Preparation of activated carbon by a physical method involves use of oxidizing agents such as H_2O, CO_2 or air. The influence of the heat treatment duration (HTD) on the development of porous structure of the carbons is investigated.

The major objectives of this study were to obtain data on porosity development, surface and adsorption properties of acti-

vated carbons prepared from vetiver roots. Traditional BET method in conjunction with the use of the Freundlich isotherm [9] was used to analyze the experimental data.

2. Methods

2.1. Chemical composition

Lignocellulosic materials are a complex matrix consisting of varying percentages of the major organic components: holocellulose (cellulose and hemicellulose) and lignin of which the cell wall is largely comprised and intracellular substance, commonly referred to as extractives. The chemical composition of the vetiver roots was determined by performing standard analyses for lignin (Klason lignin), pentosan (as hemicellulose) and cellulose (Kursshner and Hoffer method), for which the technical procedures has been already described [10]. Five grams of the precursor were incinerated to constant weight in dry air to determine the ash content. Elemental analysis of the precursor and of its inorganics content (ash) was performed with classical techniques by the Service Central d'Analyse, CNRS (Vernaison France). The sample was burned out at $1015\,^{\circ}C$. The combustion products, CO_2, and H_2O were quantified by using an appropriate infra-red analyser. NO_x were reduced to N_2 which was quantified in a conductivity cell. Sulfur content was determined with a micro-analyser type LECO (SC 144). In order to determine the oxygen content, the carbon sample was pyrolised at $1080\,^{\circ}C$ under a nitrogen flow and the oxygenated gaseous product: CO was quantified by using an appropriate infra-red analyser.

2.2. Preparation of vetiver roots activated carbon

The starting material, vetiver roots, was first dried at $105\,^{\circ}C$ for 24 h in order to reduce the moisture content. It was then ground and sieved. The fraction with a particle size ranging from 0.4 to 1 mm was used for carbonization. Approximately 2 g of sample was placed in a quartz tube and inserted into a horizontal furnace (Thermolyne F 21100) under a nitrogen flow of 80 ml/min. The isothermal experiments were carried out at 650, 700, 750, 800, 850 and $900\,^{\circ}C$ for holding times of 2 h, with a heating rate of $10\,^{\circ}C/min$ starting at room temperature to the final temperature. Preparation of the activated carbon entailed holding the chars at $820\,^{\circ}C$ for 1 h. In a second stage, they were subjected to activation at $820\,^{\circ}C$, for different holding times, with two oxidizing agents, steam or CO_2, at a gas flow rate of 80 ml/min. The samples prepared with steam and CO_2 are named VHx and VCx, respectively. The number x indicates the oxidation duration in hours.

2.3. Characterization of the activated carbons

2.3.1. Fourier transform infra-red (FTIR) spectroscopy

The functional groups on the surface of the carbons were determined by FTIR spectroscopy. The spectra were recorded using a Nexus Nicolet spectrophotometer. In preparation for measurements, activated carbon samples were dried overnight at $110\,^{\circ}C$ and the spectrophotometer was pre-calibrated. The spectrum was obtained by co-adding 32 scans, at a resolution of $4\,cm^{-1}$ over the range of 400–$4000\,cm^{-1}$.

2.3.2. Iodine index

Iodine index of the prepared activated carbons was determined by using the ASTM procedure modified as follows: using a starting material weight of 0.17 g instead of 0.5 g, the iodine concentration was determined by the sodium thiosulfate method. The volume of the reactants was reduced by a factor of 4. Using a commercial activated carbon as reference, we could confirm that iodine index obtained by this modified procedure was four times smaller than the one obtained with the original ASTM procedure. Therefore it was necessary in the calculations of the iodine index of our samples, to multiply the values obtained by a factor of 4.

2.3.3. Adsorption studies from solution

Phenolic compounds and organic dyes are common pollutants in wastewaters. Thus phenol (molecular size <1 nm [11]) and methylene blue (molecular size <1.4 nm [12]) were chosen as target adsorbates to test the adsorption properties of the vetiver roots activated carbons. Adsorption experiments were carried out at $27\,^{\circ}C$ using batch equilibration techniques. Initial concentrations were $25\,mg/dm^3$ for phenol and $15\,mg/dm^3$ for methylene blue. A series of 8–12 Erlenmeyer flasks containing 3–15 mg of activated carbon and $100\,cm^3$ of solution were sealed and shaken at room temperature until equilibrium was obtained. The equilibrium time was determined by kinetic tests. At preset times, aqueous samples were taken and the concentration of dye or phenol was determined. The adsorbent was removed by centrifugation. Measurement of the adsorbates concentration was carried out by a direct ultraviolet absorbance method (Anthelie Advanced 5 Secoman, UV–vis instrument). Calibration curves were used to calculate the concentration of adsorbates in the equilibrated solutions. For comparison, the same experiments were carried out with a commercial activated carbon Picactif TE60.

The experimental data were fitted with the Freundlich equation isotherm, represented as: $q_e = K_F C_e^n$ or the Langmuir equation isotherm: $q_e/q_m = (K_L\ C_e)/(1 + K_L\ C_e)$ The amount of compound adsorbed q_e was determined from the initial liquid phase concentration and the equilibrium concentration, $q_e = V(C_0 - C_e)/m$. C_0 and C_e are the initial and equilibrium concentration, respectively, V the volume of solution, and m is the mass of activated carbon. K_F is the Freundlich constant, q_m, the amount of adsorbate per unit mass of carbon corresponding to complete monolayer coverage and K_L, the Langmuir constant represents the equilibrium constant.

2.3.4. Nitrogen adsorption studies

The BET surface area of the activated carbons (S_{BET}) was measured with a sorptiometer Omnisorp 100 CX (Beckman Coulter). Samples were outgassed at $250\,^{\circ}C$ for 8 h at a pressure of 10^{-5} Torr before adsorption. BET areas were calculated from adsorption data in P/P_0 range of 0.001–0.1 at 77 K. Micropore volumes (total volume of pores <2 nm diameter) were estimated from the Dubinin–Radushkevich plots and mesopore volumes

Table 1
composition of vetiver roots and vetiver roots ashes

Vetiver roots		Vetiver roots ashes	
Constituents	Weight %	Constituents	Weight %
Hemicellulose	20.09	Ca	24.91
Cellulose	24.51	Mg	2.99
Lignine	33.07	K	8.33
Extract	20.6	Na	1.54
C	46.66	P	7.17
H	6.18	Mn	630 ppm
N	1.15	Ti	0.11
O	42.18	Si	6.27
Ash	1.67	Al	3.18
		Fe	1.60
		Zn	<100 ppm

Fig. 1. Char yields obtained as a function of pyrolysis temperature.

were obtained by BJH treatment of desorption data. In order to correlate gaseous and liquid phase data, N_2 adsorption data were fitted using the Freundlich equation and the physical interpretation given by Brouers et al. [9]. The mean pore diameter, D, was calculated from $D = 4V_{N_2}/S$ [13], where V_{N_2} was used to designate the total volume of pores of diameter of less than 30 nm and S being the BET surface area.

3. Results and discussion

3.1. Chemical analysis

Results of the chemical analyses of vetiver roots are given in Table 1. Holocellulose which is cellulose plus hemicellulose represents the major component at 44.6%, and the content of extractives and ash are 20.6% and 1.67%, respectively. The vetiver roots has, however a lignin content of 33.07%. As high lignin content material is preferred for activated carbon preparation [14,15], this value allows us to predict that vetiver roots could be a good candidate for activated carbon preparation. The chemical analysis gives a moderate content of C (46.66%) and O (42.18%), H (6.18%), N (1.15%). The chemical analysis of the vetiver roots ash shows that Ca (24.91%), and other inorganic elements such as K (8.33%), P (7.17%) and Si (6.27%) are the major inorganic elements in vetiver roots. Other elements, Al (3.18%), Mg (2.99%), Na (1.54%), Fe (1.6%), Ti (0.11%) are present in relatively smaller amount. Mn (630 ppm) and Zn (<100 ppm) are present as trace elements.

3.2. Carbonization studies

A feasibility study of activated carbon production from vetiver roots requires data on activated carbon yield. The yield of char produced after pyrolysis at different temperatures from 670 to 900 °C ranged from 25 to 28%. Those values are similar to those generally found for other lignocellulosic material [8]. Fig. 1 shows that increasing the temperature leads to a decrease of char yield and at 900 °C, almost 75% of the material is volatilized. Among several studies correlating the yield of charcoal obtained from pyrolysis of different tropical lignocellulosic precursors with the content of their botanical component
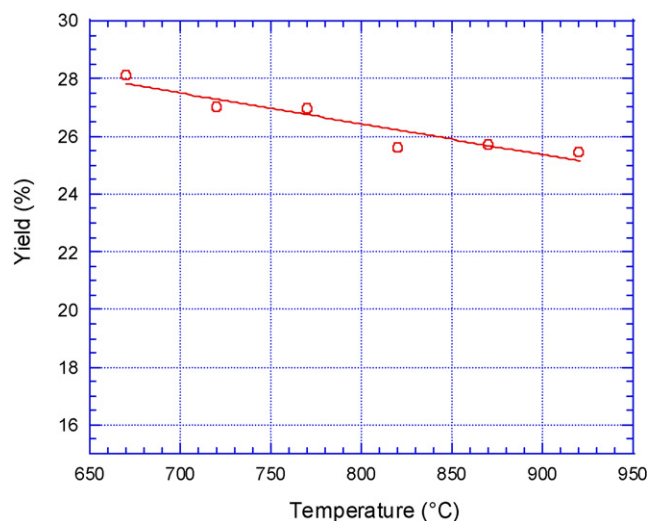
in the starting material [14–16], a recent work [1] proposed a new equation. The proposed model sums the carbon yields from the different components in order to predict the carbon yield of a lignocellulosic material. In this model, the precursor is considered as a composite material with four organic components, lignin, cellulose, hemicellulose and extractives, each component pyrolyzing independently with the mass of ash remaining almost constant during the thermal transformation process. The yield of char was obtained as a summing up of the contribution of the different parts obtained after thermal treatment. The authors proposed the following equation for modelling Y, the yield of char: Y (wt.%) $= L[0.59 - 2.7 \times 10^{-4} (t °C - 600) + 0.22 C + A + yE$, where L, C and A are, respectively the percentage (wt.%) of lignin, cellulose and ash in the precursor. The term, yE takes account of the contribution of the extractives that partly volatilize at medium temperature (500–600 °C), whereas carbonaceous deposits formed via cracking and condensation reactions led to stable tars. The expression for y is given by $y = a + b (600 - t °C)$, where a corresponds to the vaporized fraction of extractives and b to the carbonaceous deposit resulting from condensation and cracking reactions. The contribution yE was taken account only for precursor having a significant amount of extractive. Extractives represent a class of component that may vary considerably with the parent material, therefore their volatization and the formation of secondary products may vary with the parent material. As shown in Table 1, for vetiver roots the contribution of the extractive is high, 20.6%, thus it can be taken into account. In our work, the yields Y, obtained for vetiver roots can be fitted using the expression: Y (wt.%) $= L[0.59 - 2.7 \times 10^{-4} (t °C - 600) + 0.22 C + A + E [a + b (600 - t °C)]$, with: $L = 33.07$, $C = 24.51$, $A = 1.67$, $E = 20.6\%$. A good correlation ($R = 0.99$) was found and the values $(-0,102 \pm 0.008)$ and $(-1,1 \pm 0.4 \times 10^{-4})$ °C^{-1}, for a and b were similar to the one obtained for guava seeds where (-0.1) and (-2×10^{-4}), were obtained for a and b. The equation previously proposed for tropical fruit stones and seeds can thus be
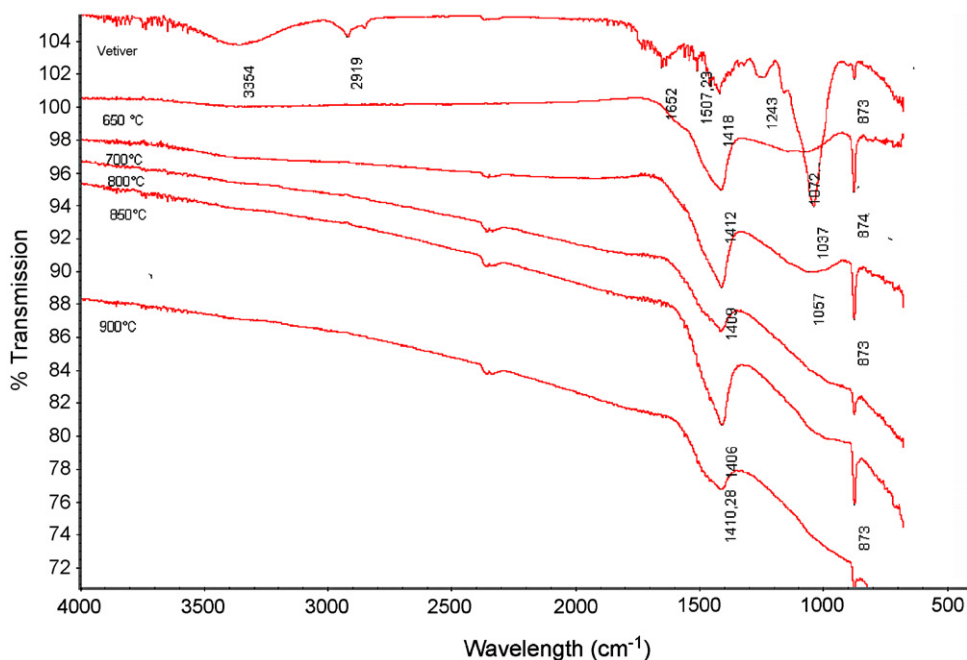
Fig. 2. FTIR spectra of vetiver roots chars.

used to predict the char yield obtained from the roots of vetiver which is a herbaceous plant.

3.3. FTIR studies

FTIR spectra of the vetiver roots, of the chars prepared at different temperatures and of the activated carbons are displayed in Figs. 2 and 3, respectively. The spectrum of the vetiver roots exhibited bands at $3354 \, cm^{-1}$ which can be attributed to the OH stretching vibrations in hydroxyl groups [17]. The low frequency values for these bands suggest that the hydroxyl groups may be involved in hydrogen bonds. The position of the band due to non-bonded OH groups is usually above $3500 \, cm^{-1}$. The band at $2919 \, cm^{-1}$ is due to C–H vibration of aliphatic groups. C=C absorptions occur between 1700 and $1400 \, cm^{-1}$ [18]. Specifically olefinic vibrations could be the cause of the band at $1652 \, cm^{-1}$, and the skeletal C=C vibrations in aromatic rings bands in the $1600–1400 \, cm^{-1}$ region of the spectrum. Absorption due to C–O vibrations occurs between 1300 and $850 \, cm^{-1}$ [18]. The band at $1243 \, cm^{-1}$ may be due to esters and the strong one at $1037 \, cm^{-1}$ to C–O vibration in C–OH moiety. The pyrolysis of the vetiver roots leads to a progressive decrease

of the intensity of the latter band absorbing at $1037 \, cm^{-1}$ when the temperature increases. Only bands attributed to aromatic C=C and C–O vibrations absorbing between 1400 and $850 \, cm^{-1}$ and a band at $873 \, cm^{-1}$ due to aromatic C–H vibrations are clearly displayed in the spectra of all chars prepared [18]. Thus, the number of oxygen containing groups decreased and aromatisation of the samples occurred during pyrolysis. The spectra of the activated carbons VH1, VH2, VH4, VH8, VC2 and VC3 contains principally the bands associated to the C=C, C=O and C–H aromatic vibrations between 1400 and $850 \, cm^{-1}$.

3.4. Activation: porosity development during activation

3.4.1. Nitrogen adsorption study

Table 2 shows that as burn-off increases from 20 to 32, for VH1, VH2 and VH4, BET surface increases with the corresponding values of 452, 501 and $576 \, m^2/g$. The micropore volume also increases, with values of 0.17, 0.19 and $0.22 \, cm^3/g$ for VH1, VH2 and VH4, respectively. For VH8, with a burn-off of 60% a lower BET surface of $306 \, m^2/g$ and a micropore volume of $0.10 \, cm^3/g$ are obtained. The BET surface area value

Table 2
Textural characteristics of vetiver roots activated carbons

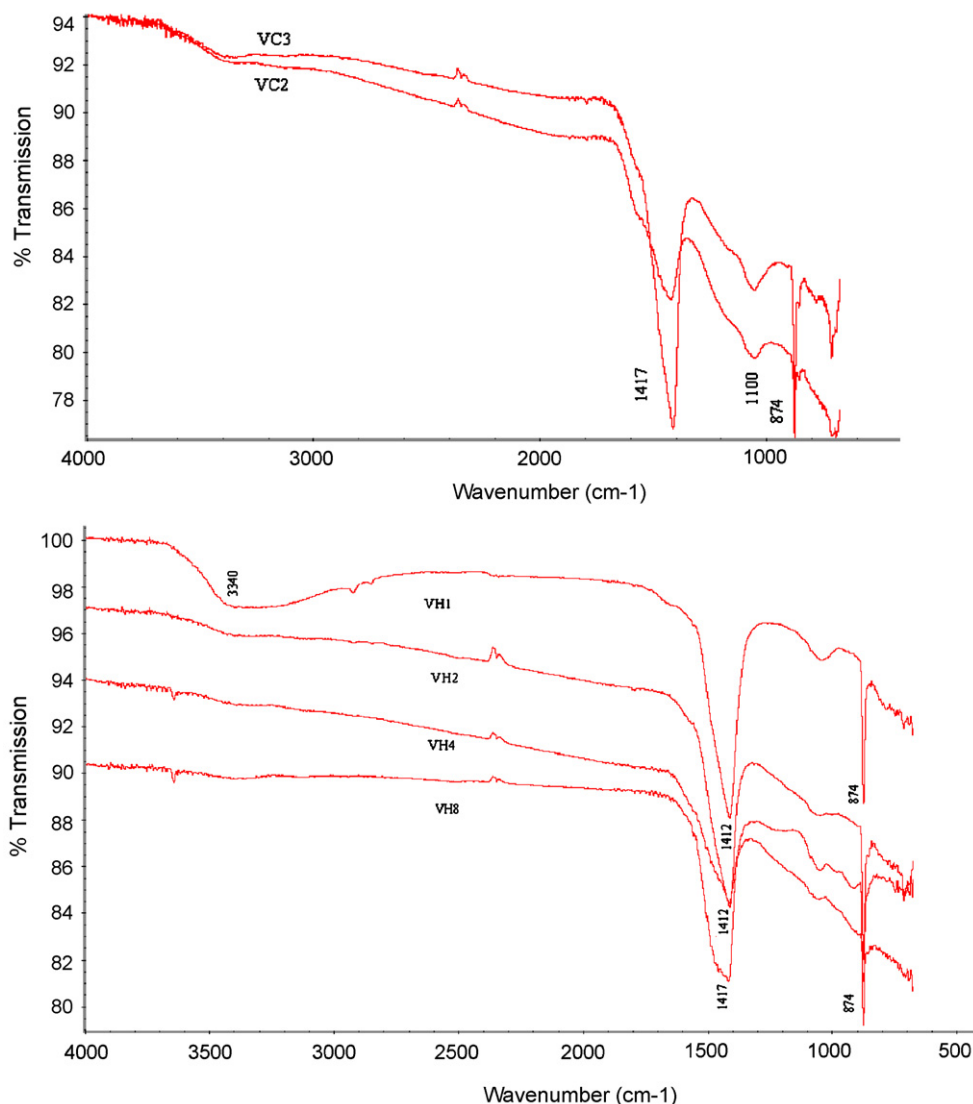| Sample	Burn-off	Mean pore diameter (nm)	BET surface area (m²/g)	Total pore volume (cm³/g)	Micropore volume (cm³/g)	Mesopore volume (cm³/g)	K_F	α
VH1	21	1.28	452	0.23	0.17	0.06	128	0.048
VH2	25	1.27	501	0.25	0.19	0.06	143	0.050
VH4	32	1.35	576	0.31	0.22	0.09	165	0.054
VH8	60	1.78	306	0.23	0.10	0.13	97	0.103
VC2	37	1.23	576	0.27	0.22	0.05	162	0.050
VC3	42	1.19	735	0.34	0.28	0.06	205	0.050

Fig. 3. FTIR spectra of vetiver roots activated carbons.

is smaller than the values obtained for the other VHx carbons due to mesopore formation. The mesopore volume of $0.13 \, cm^3/g$ found for VH8 is two times higher that the one found for VH1 and VH2, and still higher than the one found for VH4. This indicates that increasing activation by water vapour leads to a loss of microporosity, whereas mesopore volume increases concomitantly. The carbon dioxide activated carbons are essentially microporous and their BET surface areas are 576 and 735 m^2/g, and micropore volumes 0.22 and 0.28 cm^3/g for VC2 and VC3, respectively.

The N_2 adsorption isotherms are presented in Fig. 4. The transition from the initial steep region of the isotherms to the linear branch becomes less sharp as the burn-off value increases, due to the widening of the micropores. In a recently published article [9] it is shown that at low pressure N_2 adsorption data of many microporous materials, can also be fitted by using either the Freundlich law $K_F P^\alpha$, which is the small α equation limit of a Weibull shaped isotherm, with $\alpha \leq 0.2$ or the Weibull isotherm itself. Our data were thus fitted by using the Freundlich law

(Fig. 4). It was shown that the Freundlich isotherm exponent can be directly mathematically related to the adsorption energy distribution and indirectly to the micropore distribution. The exponent α increases with an increase of the micropore distribution width and a corresponding decrease of the adsorption energy. K_F values of 123, 143, 165, 97, 162 and 206 were found for VH1, VH2, VH4, VH8, VC2 and VC3, respectively. α values of 0.048, 0.050, 0.054, 0.103, 0.05 and 0.05 are obtained for VH1, VH2, VH4, VH8, VC2 and VC3, respectively. As shown in Fig. 5, a similar micropore size distribution is obtained for VH1, VH2, VH4, with a slight increase of the mean pore diameter when the burn-off increases. This is in agreement with the slight increase of the α value when the burn-off increases. Increasing the burn-off leads to formation of mesoporosity and to the enlargement of the micropore distribution width (Fig. 5), consequently, the α value increases for VH8. As previously described, the K_F value is proportional to the BET surface area (Fig. 6) whereas increase of α value is related to the burn-off, and can be attributed to the development of wider pores. These two quan-
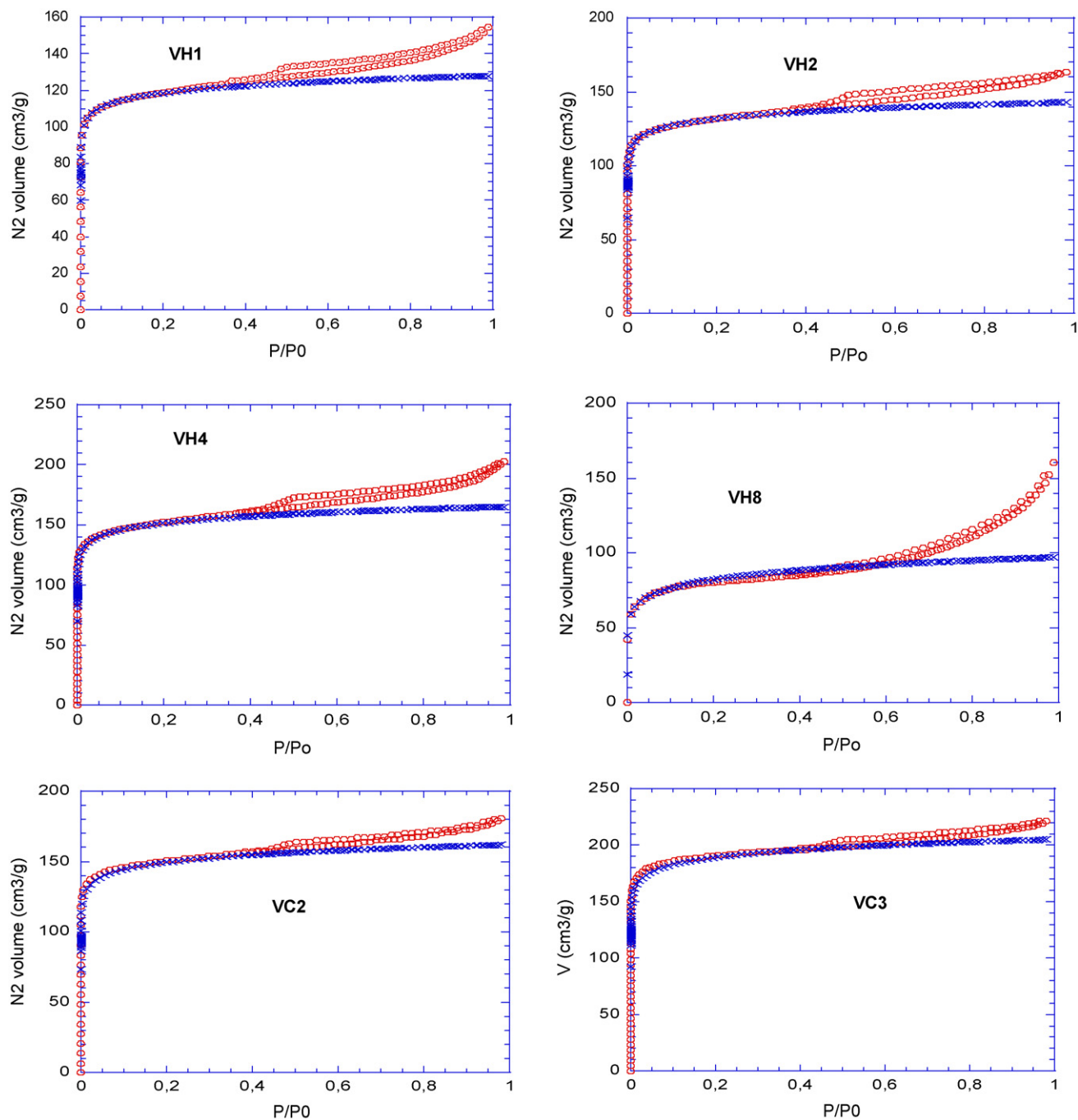
Fig. 4. N_2 adsorption isotherm of the vetiver roots activated carbons: experimental data ($\bigcirc$), Freundlich isotherm fit ($\times$).

Table 3
Adsorption parameters at 27 °C of liquid adsorption studies

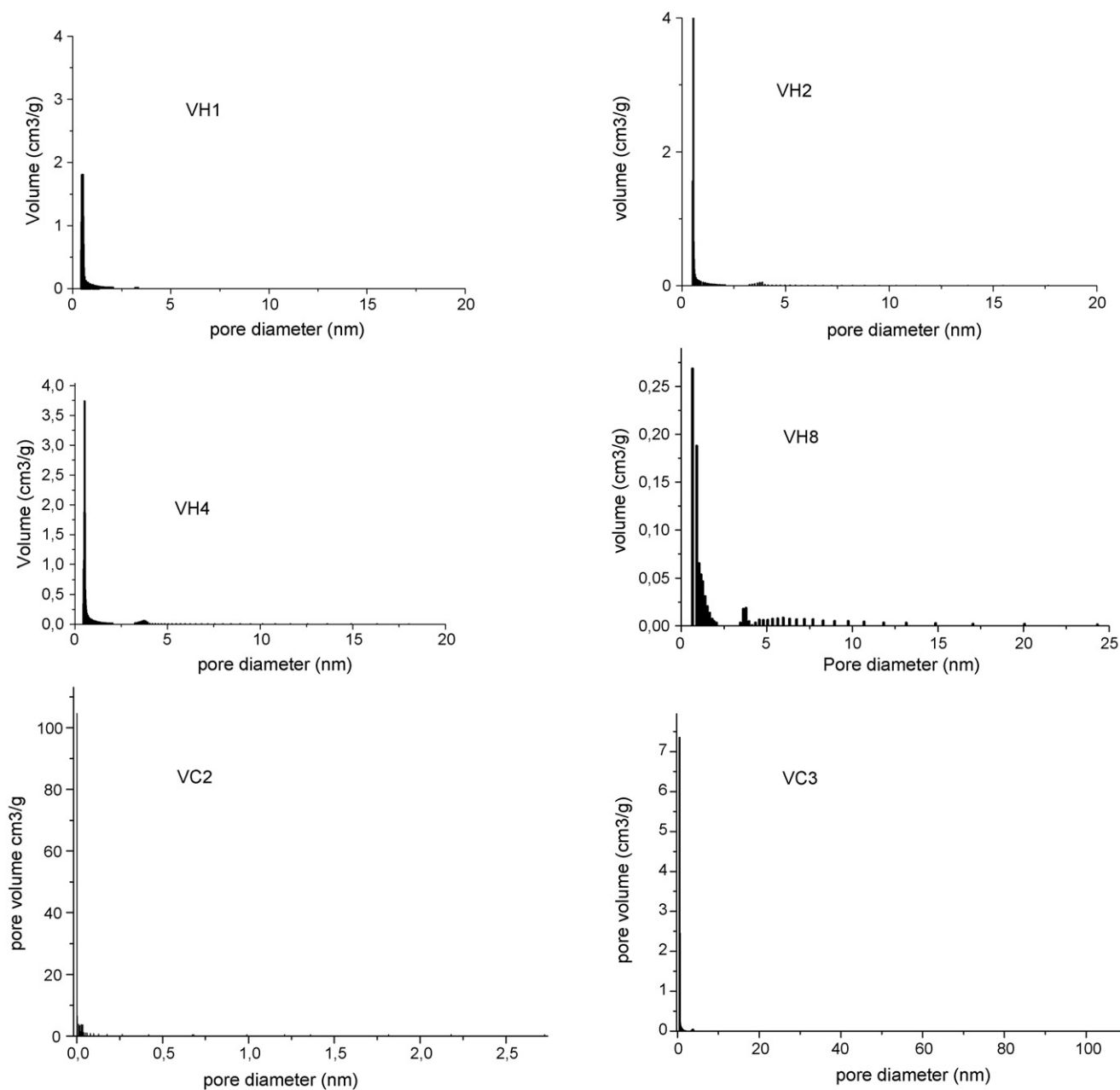| Sample	Burn-off	Phenol K_F (mg/gAC)	MB K_F (mg/gAC)	Iodine index (mg/gAC)
VH1	21	11.2	2.7	356
VH2	25	9.8	31.9	417
VH4	32	7.7	133.5	575
VH8	60	1.8	174.4	687
VC2	37	21.46	31.01	701
VC3	42	25.87	40.30	730

Fig. 5. Pore size distribution of the vetiver roots activated carbons.

tities measure the change of adsorbing power and adsorbing energy distribution resulting from the change of the microporous heterogeneity when the activation energy is modified. The α value increases slightly when the burn-off increases as shown in the previous work [9]. A net increase is obtained when there is a clear change in micropore distribution.

3.4.2. Liquid adsorption studies

The iodine index increases with the burn-off indicating that the activation process leads to development of a new porosity (Table 3). The iodine index increases with the BET surface area for the steam activated carbons VH1, VH2 and VH4 and although the surface area of VH8 is lower than the one calculated for the other VHx samples, the VH8 iodine index is higher than the one obtained for the others VHx activated carbons. This value, is similar to the iodine index obtained, for the high surface area carbon dioxide activated carbons, VC2 and VC3 (701 and 731 mg/g, respectively).

The Freundlich and the Langmuir isotherms were tested to fit the data obtained for adsorption of phenol on the vetiver roots activated carbons. Only the Freundlich isotherm provided an acceptable fit (Fig. 7) indicating an heterogeneous sorption mechanism.

The phenol adsorption capacities are low for all the steam-activated samples (Table 3) and decrease from 11.23 to 1.3 mg/g when the burn-off increases. There is however no correlation between the phenol adsorption capacity and the micropore volume, indicating that chemical interactions may also be involved
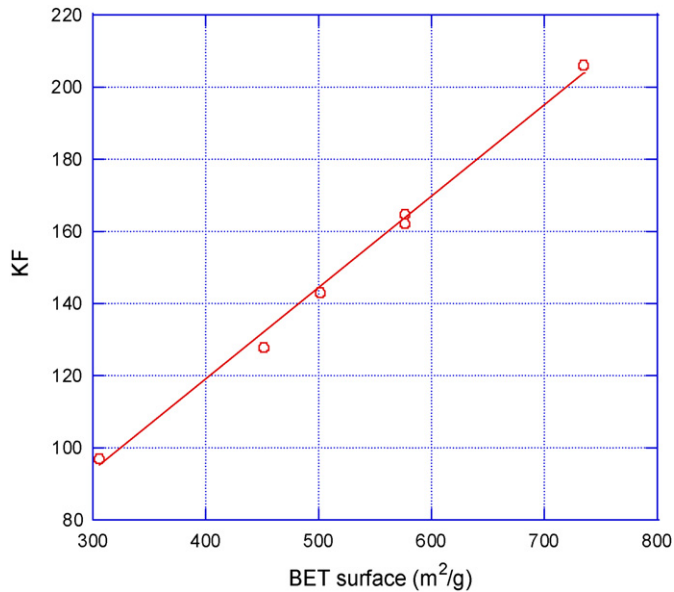
Fig. 6. Freundlich constant of VH1, VH2, VH4, VH8, VC2 and VC3 as a function of BET surface.

in the adsorption mechanism of phenol on the VHx activated carbons [19,20]. The acidic character of a carbon is associated with oxygen surface complexes or oxygen functionalities such as carboxyls, lactones and phenols. Basic properties of the carbon surface are likely to be due to: (1) functional groups like pyrones, chromenes, ether and carbonyls, that contain less oxygen, (2) protonated amino groups for nitrogen-rich carbons, and (3) grapheme layers acting as Lewis bases and forming electron donor-acceptor complexes with H_2O molecules [19]. The phenol adsorption capacities are higher for the carbon dioxide activated carbons, VC2 (21.46 mg/g) and VC3 (25.87 mg/g). This observation could be explained by the fact that, steam-activated carbons generally have higher acidity values (lower pH at zero
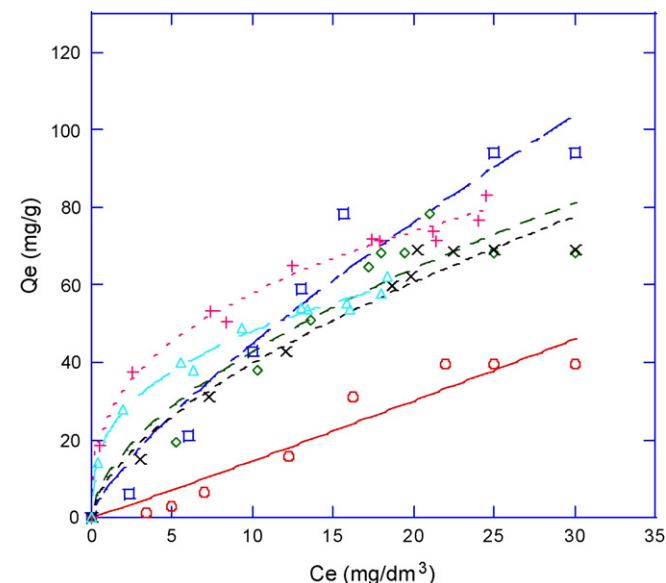

Fig. 7. Adsorption isotherm at 27 °C of phenol on VH1 ($\bigcirc$), VH2 ($\square$), VH4 ($\diamond$), VH8 ($\times$), VC2 ($\triangle$), VC3 (+).
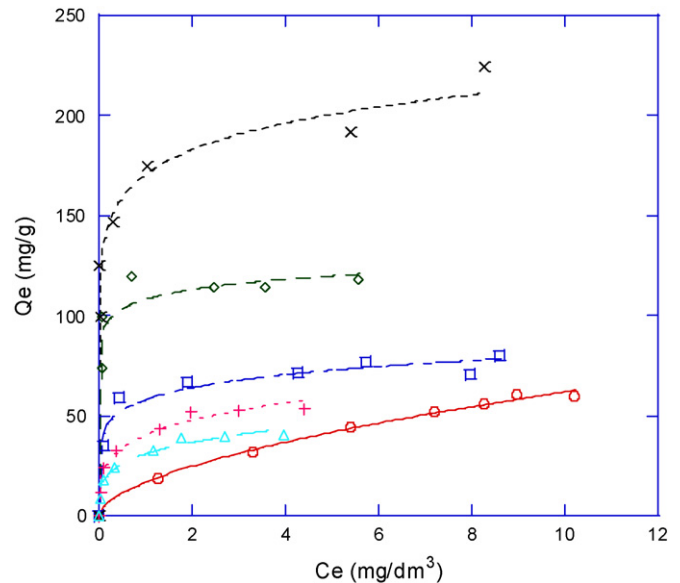
point charge) than carbon dioxide activated carbons [21,22] and that the most basic carbons present the better adsorption capacity for phenol [19,20].

The methylene blue adsorption capacity, increases with the burn-off indicating that the activation process leads to development of new porosity (Table 3). All the data obtained for adsorption of methylene blue on the vetiver roots activated carbons could be fitted by the Freundlich isotherm (Fig. 8). The highest methylene blue adsorption capacity values, $K_F = 174$ mg/g, is obtained for VH8. This value is higher than the one calculated for the commercial activated carbon PicaTE60 (158 mg/g). Due to their low mesoporosity, the microporous activated carbons VC2 and VC3 have much lower methylene blue adsorption capacities than the one obtained for VHx activated carbon with similar burn-off. The Langmuir isotherm which is a typical model for monolayer adsorption provided a satisfactory fit to the adsorption data of methylene blue on VH4, VC2 and VC3 only. The q_m value determined from the Langmuir isotherm represents the amount of solute adsorbed per unit mass of adsorbent corresponding to complete monolayer coverage of the adsorptive sites. The surface area estimated by the adsorption of methylene blue is given by: $S_{MB} = 1000. S°_{MB}q_m$, where $S°_{MB}$ is the surface area of methylene blue (1.93 m^2/g) [23]. The q_m values obtained for adsorption of methylene blue on VH4, VC2 and VC3 are, 172.95, 40.04 and 53.40 mg/g, respectively. The corresponding S_{MB} values calculated are 333, 77.3 and 103.4 m^2/g for VH4, VC2 and VC3. The ration S_{MB}/S_{BET} is an indication of the fraction of porosity accessible to methylene blue. Due to its molecular size, <1.4 nm [12], the dye molecule can access about 58% of the porosity of VH4, with a mean pore diameter of 1.35 nm whereas only 13.4% and 14% of the porosity of VC2 and VC3 respectively is accessible to the methylene blue molecule. Indeed, the mean pore diameters, 1.23 and 1.20 nm for VC2 and VC3 are lower than the one of VH4 and it has been estimated that the minimum pore diameter methylene blue

Fig. 8. Adsorption isotherm at 27 °C of methylene blue on VH1 ($\bigcirc$), VH2 ($\square$), VH4 ($\diamond$), VH8 ($\times$), VC2 ($\triangle$), VC3 (+).

molecules can enter is 1.3 nm [24]. Overall, activated carbon prepared by steam activation of vetiver roots have a good ability for the adsorption of methylene blue.

4. Conclusion

Vetiver roots can be used to prepare high surface area activated carbons. Steam activated carbons prepared from vetiver roots are however, not suitable for adsorption of classical phenolic pollutants, due to their low adsorption capacity. However, high burn-off activated carbons obtained by steam activation of vetiver roots can be used for specific applications such as the adsorption of large molecules such as methylene blue. The role played by activated carbon surface groups of vetiver roots activated carbons, for phenol and methylene blue adsorption needs to be further investigated. Nevertheless, their potential utilization as sorbents will be fully dependant of their mechanical properties, notably, the dusting attrition of these ACs. This parameter has now been fully investigated and the results obtained will be the subject of a future paper. The potential of vetiver roots activated carbons for adsorption of other dyes and recalcitrant macromolecules from molasses wastewaters will also be studied in the future.

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