

Valorisation of the Olive Cake Residue by Pyrolysis Under Water Vapour Current

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Abstract: Our principal aim is to contribute to the durable development by the use of the local biomass and more particularly to the valorisation of the olive cake residue while realizing a raw material autonomy local, a clean, sparing process of atoms and an energy autonomy. Within this framework and considering the availability of the olive cake residue and the absence of its thermochemical valorisation (pyrolysis, gasification and combustion), we were interested in this research in the study of slow pyrolysis with 400-800°C of this biomass and its activation in only one stage in a continuous pipe still equipped with a quartz tube under a water vapour current. The characterizations of the activated carbons obtained are carried out by various techniques of analyses such as: FTIR, DSC, ATD-ATG, CHN, BET and NMR. The products obtained are characterized by porous volumes from 0.5-0.7 cm³ g⁻¹, of average porosities from 15-20 Å° and of surfaces specific from 300-900 m² g⁻¹. The increasing request of the activated carbons however, relates to the field of water and the environment.

Key words: Olive cake residue, pyrolysis, activated carbon, water vapour, FTIR

INTRODUCTION

We tried to outline the possibilities offered by the use of the biomass to reach the activated carbon. The activated carbon is the universal standard for the purification and the displacement of the organic contaminants of the liquids and gas, gifted with particular adsorbent properties because of their great porosity (Sun and Meunier, 1999). Its significant specific surface also allows its use as support in heterogeneous catalysis (Perrin and Scharff, 1999; Papin, 1989). The activated carbon particle presents 2 types of pores responsible for the adsorbent properties. They are the macropores and the micropores (Perrin and Scharff, 1999; Lartey *et al.*, 1999; Goulson and Richardson, 1979; Young and Crowell, 1997).

Coal is obtained by pyrolysis of organic substrates (biomass) which is activated at high temperature under oxidants (chemical activation, oxidizing gas reagents, solid reagents or desiccants tells that: CO₂, H₂O, H₃PO₄, ZnCl₂, MgCl₂, etc.) to lead to the strongly porous end product by increasing its specific surface and to multiply its capacity of adsorption. The activated carbon is one insoluble blasting powder in water, without odour and very flexible adsorbent.

Used for there purification of foodstuffs and medicinal, there purification of waste water and recovery of solvents, there purification of gases, there discolouration of oils and vegetable fat, it digestive purification in the acute intoxications, etc.

The activated carbon are manufactured starting from various materials of vegetable and mineral origin of high carbon contents like the coconut shells, the seeds, wood, the peat, fossil coal, even cores of olives and in our case that the olive cake residue.

The olive cake residue is the result of by-product derived from the triturating (the report olive cake residue/fruit, is 33%), it represents the olive paste after crushing and extraction of the olive oil and it contains most of the dry olive matter (skin, pulp, small pieces of the cores: Table 1). Its modest heating value is produced mainly by the shell which represents 60% of the mass (Nefzaoui, 1999).

The cellular wall of the olive cake residue is made up mainly of 3 essential components, the cellulose, hemicelluloses and lignin (Vigouroux, 2001) (Table 2).

The local availability of the olive cake residue and the absence of study of its pyrolysis in the bibliography led us to start the study of the possibility of carbonization

Table 1: Chemical composition of the olive cake residue

Composition	(%)
Shells (crumbs + endocarps)	56
Skin and pulp (mesocarpe + epicarpe)	28
Oil and almond	5
Ash	4
Water	7

Table 2: Constitution of the cellular wall of the olive cake residue in MS%

Cellulose	Hemicelluloses	Lignin	Ashes
24.0	23.6	48.4	4.0

and activation of this raw material. The choice of the method of activation is guided by the preoccupation with a development of a simple process of application.

Methodology used is as follows:

- The pyrolysis and the activation of the olive cake residue out of activated carbon are carried out under a water vapour current
- The quality of the product obtained is studied according to the conditions of the reaction pyrolysis-activation (temperature, flow of water vapour, duration of carbonization and activation, etc.)

Experimental study: In what follows, we treat a pyrolysis under a water vapour current, which follows primarily a radicalizing mechanism in channel. Initially, the molecules are duplicated by forming free radicals, which propagate the channel by adapting atoms of hydrogen and by forming new radicals (Fillippov, 1991; Minkova *et al.*, 1999, 2001). The chemical conversions during pyrolysis are primarily the decomposition of the large molecules and the secondary transformations of the products of decomposition, which lead to a solid (coal), a liquid (pyroligneous liquor) and to gases (CO_2 , CO , H_2 , etc.). Its components undergo degradation and a chemical conversion pushed by varying the temperature and the residence time of the products of decomposition in the reactional zone (Benabdaah *et al.*, 1994).

Calorimetric analysis differential DSC: With an aim of studying the decomposition of the olive cake residue according to the temperature, we examine by DSC of the behaviour of this biomass beyond the temperature of carbonization (Fig. 1).

The cellulose, hemicelluloses and lignin undergo a thermal decomposition in a well defined temperature range. The mechanism of conversion of carbon cellulose consists of 4 successive stages (Tang and Bacon, 1964):

- The desorption of the water adsorbed starting from 150°C
- The loss of the structural water of cellulose between 150 and 300°C

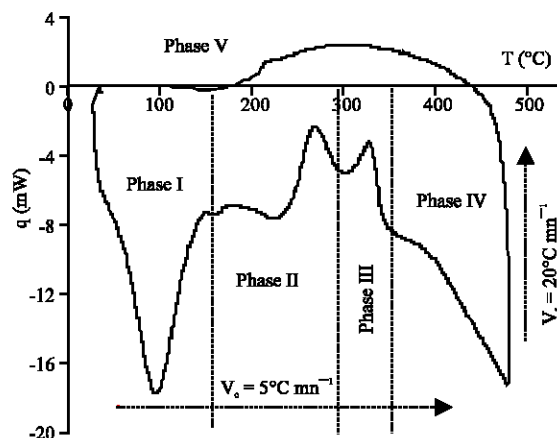


Fig. 1: Variation of the heat of carbonization of olive cake residue according to the temperature

- Rupture of the channels or depolymerization and rupture of the connections C-O and C-C between 240 and 400°C
- Finally aromatization starting from 400°C , which led to the formation of the layers of the graphitic type

However, we can admit that the thermal decomposition of the olive cake residue is the result of the separate decomposition of each one of its components. One can thus follow this process by 3 great stages according to Fig. 1: thermal decomposition of hemicelluloses between 200 and 260°C , followed by that of cellulose between 240 and 350°C and finally the decomposition of lignin between 280 and 500°C (Soltes and Elder, 1981).

To ambient temperature until 160°C (Phase I): Phase of drying. The olive cake residue loses water like some volatile compounds (ethers, phenols, etc.) caused by the produced vapour.

To 160°C with 300°C (Phase II): Phase of torrefaction. The compounds of the olive cake which are most unstable thermally (hemicelluloses) breaks up in particular into xylenes, from which are formed water (which is added to that coming from the moisture of the olive cake residue), of the amino acids, oleic acid, linoleic acid, palmitic acid, lauric acid, acetic acid and formic acid, etc. Oxygenated gases (CO , CO_2) and methanol also get clear. With the exit of this phase, the olive cake residue is known as to torrefy.

Starting from 300°C (Phase III): Exothermic phase, it occurs a clear exothermic reaction which rises abruptly until 350°C without contribution of external energy. It is

during this phase that the compounds with strong energy content leave the olive cake residue (lignin), that the effluents are most abundant and that the olive cake residue is transformed into coal.

Between 350°C and 500°C (Phase IV): Carbonization requires a new contribution of energy. It is during this phase that the cellulose breaks up especially, giving water, CO₂, of coal and an unstable intermediate product, the levoglucosane, which breaks up into products similar to those obtained with hemicelluloses. It is also during this phase that the heavy tar and the phenolic compounds are produced (phenol, ortho-phenols, derived from cinnamic acid, glycosides, flavonoïdes, etc.) resulting from the decomposition of the lignin, this is also accompanied by a production of methanol.

Beyond that 500°C (Phase V): There is a phase of dissociation. The less abundant gases grow rich in hydrogen. The departure of hydrogen involves a relative enrichment out of carbon of the solid residue.

To 500°C at the ambient temperature (Phase IV): Phase of cooling of the sample.

Analysis thermogravimetric differentials ATG-ATD: The examination of the variation of the mass of the olive cake residue according to the temperature was measured by ATG-ATD. This technique enables us to give the loss of absolute weight of the olive cake residue introduced into an aluminium oxide crucible according to the temperature, under a current of argon, using a high performance thermobalance and according to a program of temperature. The resolution of the microbalance is of $\pm 0.01 \mu\text{g}$. The results of measurement are represented by Fig. 2a and b.

The carbonization of the olive cake residue is an endothermic phenomenon.

The variation of the mass between the ambient temperature (25°C) and that of carbonization (700°C) is 13.243 mg (moisture 7%) for an initial mass of the olive cake residue equal to 17.370 mg. The mass output of carbonization: $\rho_{\text{mh}} = 23.76\%$ (or $\rho_{\text{ms}} = 25.55\%$).

The heating rate is of 5°C mn^{-1} , it is the same speed, which will be used in the continuous pipe still during preparation of our samples.

It is starting from 400°C that the phase of aromatization starts, which led to the formation of the layers of the graphitic type. From this temperature the thermal decomposition is assured.

The curve of variation of the mass according to the temperature and time (Fig. 2) indicates a significant

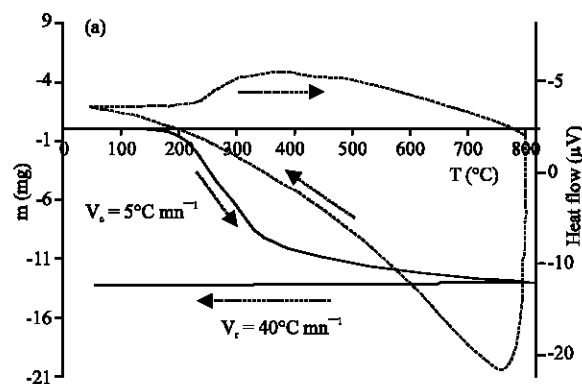


Fig. 2a: Variation of m and Heat flow according to T under inert gas (Helium and Argon)

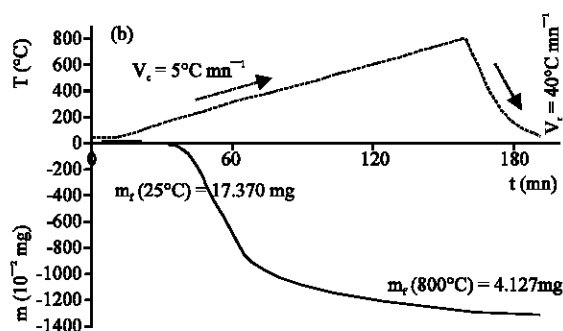


Fig. 2b: Variation of m according to T and t under inert gas (Helium and Argon)

reduction towards 400°C. The output in pyrolysats becomes almost constant starting from 500°C. Thus, we can affirm that carbonization is complete at this temperature and that aromatization with place starting from 400°C. The departure of the totality of the volatile compounds seems completed towards 500°C. Outputs corresponding to an average value of 26% mass.

Pyrolysis of the olive cake residue: The preparation of the activated carbon obtained is based on slow traditional pyrolysis and activation by water in only one stage. Several tests were carried out (Table 3) by introduction of a quantity of approximately 45 g of starting material into a tight continuous pipe still equipped with a quartz tube, under a water vapour current with constant flow (0.75 l h^{-1}). The unit is heated at the desired temperature. The device used is characterized by a speed of heating linear of 5°C mn^{-1} with a resolution $\pm 10^\circ\text{C}$.

The stage of the final temperature is maintained for one duration ranging between 5 and 30 mn. At the end of the reaction, the heating is stopped and the

Table 3: Summary table of the experimental results

Experiment	m_i (g)	T_a (°C)	t_c (mn)	m_f (g)	ρ_{mh} (%)	ρ_{ms} (%)
CG-400	45.20	400	20	13.39	29.62	31.85
CG-450	44.83	450	20	11.68	26.05	28.01
CG-500	44.83	500	20	10.97	24.47	26.31
CG-550	44.83	550	20	10.69	23.84	25.64
CG-600	44.83	600	20	9.77	21.79	23.43
CG-700-05	44.83	700	05	7.58	16.91	18.18
CG-700-11	44.83	700	11	7.56	16.86	18.13
CG-700-20	44.83	700	20	7.52	16.77	18.04
CG-700-29	44.83	700	29	7.46	16.64	17.89
CG-800	44.83	800	20	4.93	10.99	11.82
CG-ASHE	20.00	550	225	0.78	3.90	4.19

cooling of the furnace until the ambient temperature is accomplished under the same water vapour current with a speed of 40°C mn^{-1} (Benabdaah *et al.*, 1994).

RESULTS AND DISCUSSION

The methods of analyses necessary to characterize the activated carbon, concern, the examination of the composition during the preparation and the studies of the texture of the products obtained. Specific surface, porous volume and the capacity of adsorption are the characteristics aimed in our study. A test of examination of the organic groupings related to surface was undertaken by the NMR of the solid.

In all the analyses, the activated carbon samples were crushed, filtered to $80\ \mu\text{m}$ and then dried with the drying oven with 120°C during 24 h. Their cooling was carried out in a desiccator's.

Evolution of the olive cake residue according to the temperature: Study by FTIR spectra: The spectrum of olive cake residue (Fig. 3) is characterized by the presence of 2 intense tapes with 2853 and $2924\ \text{cm}^{-1}$ characteristics of aliphatic CH vibration whose content decreases as the temperature of pyrolysis increases. To $3006\ \text{cm}^{-1}$ one observes a tape of low intensity which can allot to aromatic $\nu\text{C-H}$, which can come from phenolic group. Groupings O-H gives place to a wide strip centred around $3369\ \text{cm}^{-1}$. The presence of the groupings ester in the olive cake residue had with the olive oil residues, appears by 2 intense $\nu\text{C}=\text{O}$ at 1712 and $1652\ \text{cm}^{-1}$. Around $1163\ \text{cm}^{-1}$ appear vibrations $\nu\text{C-O-C}$ resulting from the ether groups or phenolic (Serrano *et al.*, 1999).

The tape corresponding to vibrations $\nu\text{C-C}$ in the plan of the aromatic cycles is located at $1575\ \text{cm}^{-1}$, this tape is observed for the pyrolysats obtained with 400°C .

The vibration of deformation of CH_2 to $1462\ \text{cm}^{-1}$ undergoes a decrease of the intensity with the increase in the temperature of pyrolysis (Zawadzki, 1997).

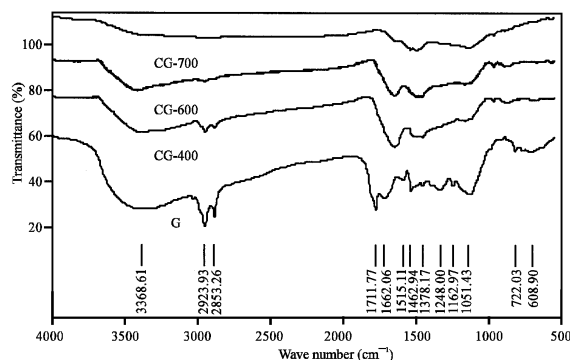


Fig. 3: FTIR Spectra of the olive cake residue G and of carbonisats CG-400, CG-600 and CG-700

The evolution of FTIR spectra of the olive cake residue according to the temperature makes it possible to highlight the fast disappearance of the esters groupings at 400°C , the tapes γCH continuously undergo a reduction in the intensity until the activated carbon formation.

In field $3180\text{--}3600\ \text{cm}^{-1}$, the vibration of valence νOH undergoes a reduction in intensity, its presence in the activated carbon reflects the hydroxylic existence of group OH on the surface, as well as the possibility of chemically absorbed water. The displacement of this tape towards numbers of lower waves indicates the presence of bridges hydrogen.

The progressive aromatization of material is in agreement with the presence of aromatic band $\nu\text{C-C}$ in all the spectra in the neighbourhoods of $1575\ \text{cm}^{-1}$. Its relative intensity believes with the increase in the temperature.

FTIR Spectra at various temperatures make it possible to identify connections C-H, O-H, C=O, aromatic C-C and C-O-C. Formation of the graphitic structure during pyrolysis and the activation and description by the increase of the intensity of $\nu\text{C-C}$ to $1575\ \text{cm}^{-1}$.

The outputs of carbonization, as well as the chemical nature of surface, are strongly influenced by the temperature of pyrolysis. We note the increase in the carbon contents with the temperature and the reduction in the hydrogen content and oxygen (Table 4).

Analyses Carbon Hydrogen Nitrogenizes (CHN): Ultimate analysis CHN of carbonisats obtained at various temperatures of pyrolysis, shows the reduction in the contents in hydrogen and oxygen (Table 4), in the shape of CO , CO_2 and mainly H_2O . We can conclude that:

- The carbonisat arrives more or less at 85% of the contents out of carbon and tiny room to 1/10 its oxygen contents

Table 4: Evolution Carbon, Hydrogen, Nitrogen, Oxygen and Ashes of the pyrolysats according to the temperature

Coal	C (%)	H (%)	N (%)	Ashes (%)	O (%) per diff
Cake Res. TI	48.20	6.3	3.1	4.0	38.4
CG-400	73.40	5.3	-	1.1	20.2
CG-550	86.30	3.4	-	1.4	8.9
CG-600	89.50	2.8	-	1.7	6.0
CG-700	92.10	1.9	-	2.1	3.9
CG-800	93.40	1.6	-	2.9	2.1

- The olive cake residue is definitely lignocellulosic
- The temperature of carbonization has which the thermal decomposition is reached, starts starting from 500°C

Capacity of adsorption of nitrogen by ATD-ATG: The activated carbon are dried and degassed with 150°C during 30 mn under a vacuum of 10^{-4} Torr, follow-ups of a cooling with 25°C. The phase of adsorption of nitrogen is to realize at ambient temperature to the stage of saturation (Table 5). Although, these measurements do not make it possible to determine the specific surface of the samples, they are useful for a relative classification of the capacities of adsorption. These properties are in relation to specific surfaces of the pyrolysats.

The product resulting from pyrolysis and the activation of the olive cake residue with 800°C (pyrolysats CG-800) present the best performance. The samples obtained until the 500°C are characterized by a very weak adsorption from N_2 . This is the results of absence of activation at this temperature.

Determination of the capacity of adsorption per infra-red spectroscopy: The infra-red spectroscopy is the method, which we used for the quantitative determination of the adsorbents, methylene blue, benzene and iodine.

To carry out calibration, we must have a set of samples with concentrations different from the component to quantify which interests us. The concentrations must be known (by means of the weighing of the ingredients during the preparation of the samples). We carry out calibration using the quantities of known components and using the totality of their spectra (Fig. 4). The calculation of the calibration curves is done starting from concentrations known by linear regressions.

The infra-red spectra used are of absorptions type. In the ideal case, one can obtain a linear relationship between the values of concentration and the values of intensity (law of Beer-Lambert). The analysis is done inside the interval of the beforehand gauged concentrations. The software will calculate the unknown concentrations automatically.

The properties of a porous solid depend primarily on the geometry and the size on the pores like their distributions. In order to characterize our samples in

Table 5: Quantity of nitrogen adsorbed by the activated carbon with 25°C and under P_{atm}

CG	m_i CG (mg)	N_2 adsorbed (mg)	N_2 adsorbed (10^{-3} moles g^{-1} CG)	V_{ad} ($cm^3 g^{-1}$)	Mass (%)
CG-400	11.346	0.021	6.610	1.481	0.185
CG-550	10.332	0.025	8.641	1.935	0.242
CG-700	33.720	0.364	38.552	8.636	1.479
CG-800	8.536	0.175	73.219	16.401	2.038
CATG-800	4.070	0.072	63.180	14.152	1.769

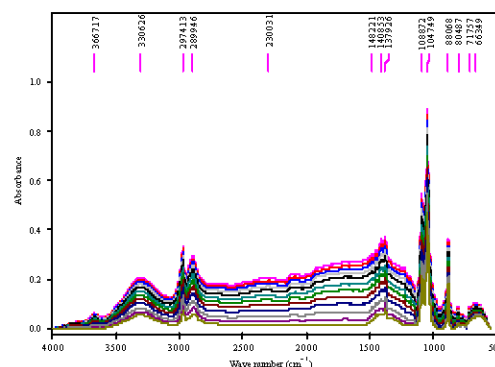


Fig. 4: Spectra of calibration of methylene blue in anhydrous ethanol (resp. upwards, 0, 50, 100, 200, 300, 400, 500, 600, 700, 800, 900 and 1000 mg L⁻¹)

relation to the size of the pores, we measured the capacities of adsorption with liquid molecules of different sizes, methylene blue, iodizes and benzene (Table 6a and b).

The activated carbon are immersed in a quantity of ethanol solution containing adsorbed it with a given concentration. The difference between the initial concentrations and final concentrations is representative for the adsorbed quantity.

Measurement was made so that the adsorbed final quantity is in the horizontal part of the asymptote of the isotherm of adsorption.

The mixture coal credit-solution adsorbed, is placed in a bath thermostated with 25°C and agitated during 30 mn. The solution obtained by filtration of the activated carbon after 24 h have undergoes a quantitative analysis using spectroscopy FTIR. The experimental results are consigned in Table 6b.

NB: The intensity of adsorption of ethanol is very low (Zoulalian, 1997).

In agreement with the bibliography, the development of microporosity starting from 600°C, as well as the evolution of S_{mi} in the prepared samples are characterized by a weak variation between 700 and 800°C. The values of S_{mi} make it possible to conclude that between 600 and 800°C, the increase in the duration of activation can compensate for the reduction in the temperature of activation (Fig. 5).

Table 6a: Equations of the calibration curves $Y = f(X)$ established starting from the spectra of calibration of the aqueous solutions in anhydrous ethanol with 25°C

Aqueous solution	Concentration	$Y = f(X)$	r
$C_{16}H_{18}ClN_3S$	50-600 mg L ⁻¹	$Y = +515.31 - 0.1608900 * X$	0.9993
C_6H_6	20-500 mL L ⁻¹	$Y = +721.43 - 0.2252500 * X$	0.9992
I_2	2-20 g L ⁻¹	$Y = +25.765 - 0.0080447 * X$	0.9993

Table 6b: Quantity of benzene, iodine and methylene blue adsorbed by the activated carbon with 25°C

Coal	C_6H_6		I_2		$C_{16}H_{18}ClN_3S$	
	w w ⁻¹ (%)	cm ³ g ⁻¹	mg g ⁻¹	cm ³ g ⁻¹	mg g ⁻¹	cm ³ g ⁻¹
CG-400	9.66	0.109	212.25	0.043	43.62	0.073
CG-550	16.07	0.182	538.27	0.109	74.33	0.124
CG-700	25.33	0.288	686.91	0.139	145.07	0.242
CG-800	27.03	0.307	736.33	0.149	170.13	0.283
CATG-800	17.23	0.196	579.11	0.117	124.92	0.208

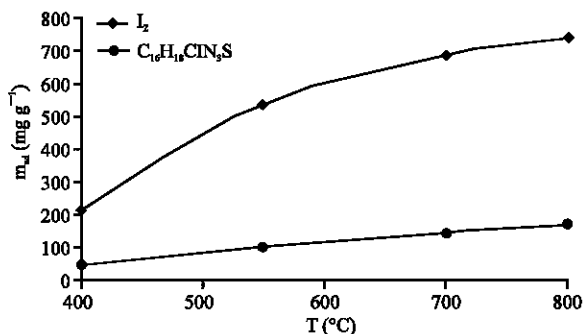


Fig. 5: Evolution of m_{ad} of Iodine and Methylene blue according to T

Specific surface and porosity (Table 7): The isotherms of adsorption of diazotizes on coals, deferred on the Fig. 6 and 7, are apparently of the type I in which adsorbed overlaps to the maximum of a monomolecular layer, characteristic of a nonporous solid or microporous adsorbent, whose diameter of the pores is lower than 25 Å° (Fig. 8).

In fact, the stages of saturation are not horizontal: it would have to be said that they are the composite isotherms of type I+II (Jean-Paul, 1997). The solids overlap with several layers of molecules of adsorbents. This form is found if the solid is macroporous ($D_p > 200 \text{ Å}^\circ$).

The curve representative by the Fig. 8 of porous volume V_p according to the diameter D_p of pore (called cumulative curve of volumes of pores according to the rays r of pores) gives access specific surface $S_i = 2V_p/r_i$ one supposing the cylindrical shape of the pores.

By using given surfaces microporous S_{mi} previously, have can deduce that our conditions lead to an average rate of activation (Table 8).

Correlation and estimate: Stoekli and coll (Ibanez, 2002), established an empirical relation m_i and m_f called usually Burn-off:

$$\text{Burn-off (\%)} = \left(1 - \frac{m_f}{m_i}\right) \times 100 \quad (3.1)$$

Table 7: Specific surfaces, porous volumes and average diameters of the pores

Specification	Carbonisat	CG-700-30	CG-700-05
Mass sample (mg)		109.2	106.5
Open space (cm ³)		31.6680	19.5204
Equilibrium time (sec)		15	15
Temp. desorption (°C)		150	150
Specific surface (m ² g ⁻¹)	Langmuir	471.7589	346.8557
	BET	290.0235	211.5619
	At point P/P°	313.1790	227.6432
		P/P°=0.3178	P/P°=0.3183
Porous volume (cm ³ g ⁻¹)	Adsorption		
	D_p : 10-500 Å°	161.4244	149.2932
	Desorption		
	D_p : 10-500 Å°	120.2447	80.0510
	Microspore	225.2515	146.9080
	At point P/P°		
	and D_p in Å°	0.1822	0.1711
		P/P°=0.9929	P/P°=0.9773
		D_p : 2722 Å°	D_p : 867 Å°
	Adsorption		
Porous average diameter (Å°)	D_p : 10-500 Å°	0.0736	0.0868
	Desorption		
	D_p : 10-500 Å°	0.0472	0.0720
	Microspore	0.1308	0.0863
	Langmuir	15.4466	19.7338
	Adsorption	18.2397	23.2661
	Aesorption	15.7045	35.9995

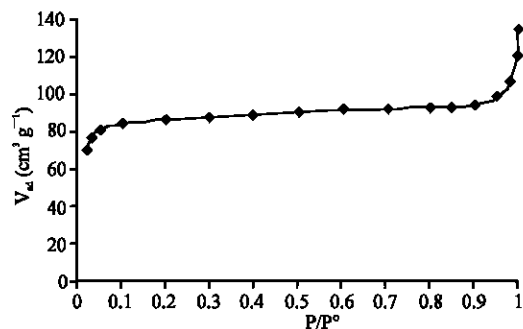


Fig. 6: Isotherm of adsorption of carbonisat CG-700-05 with 150°C

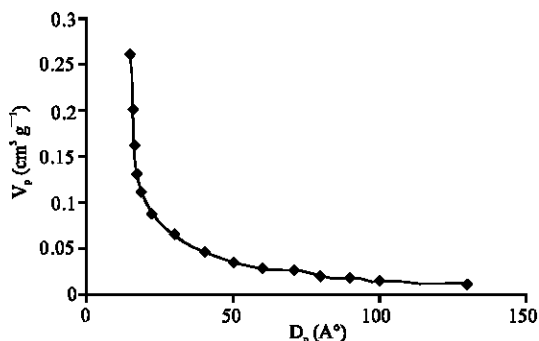


Fig. 7: Distribution of V_{ad} according to D_p of carbonisat CG-700-30

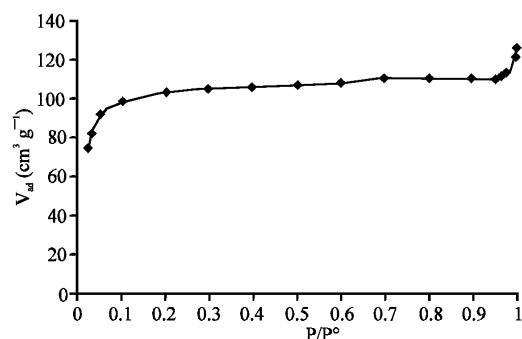


Fig. 8: Isotherm of adsorption of carbonisat CG-700-30

Table 8: Estimate of volumes and microporous surfaces of coals

Coal	Burn-off (%)	W_o (cm³ g⁻¹)	L_o (nm)	S_{mi} (m² g⁻¹)
CG-400	68.15	0.558	1.5-1.9	587-744
CG-550	74.36	0.596	1.5-1.9	627-794
CG-700	81.96	0.641	1.5-1.9	674-854
CG-800	88.18	0.679	1.5-1.9	714-905

A relation Eq. 3.2 between W_o and Burn-off was established (Ibanez, 2002):

$$W_o \text{ (cm}^3 \text{ g}^{-1}\text{)} = 0.15 + 0.006 \times (\text{burn-off } \%) \quad (3.2)$$

Ibanez (2002) established a simple geometrical relation Eq. 3.3 between S_{mi} and W_o :

$$S_{mi} \text{ (m}^2 \text{ g}^{-1}\text{)} = \frac{2000 \times W_o}{L_o} \quad (3.3)$$

The application of these models to our samples obtained starting from the olive cake residue by using the average diameters of the microspores establish by the models of Langmuir and of BET, the results consigned in Table 8 reveal. The extension of these models to sample CG-700, makes it possible to find the same tendency noted in the determination of the indices of iodine, benzene and methylene blue by FTIR (S_{mi} CG-700: 693-878 m² g⁻¹).

Study of the groups of surface of the activated carbon by NMR: In general, the surface of the activated carbon carries sites oxygenated and possibly of the amino sites. They can be of acid, basic or neutral nature. Sites of the acid types can be groupings carboxylic, phenolic, anhydrides, ethers, quinones and lactones, they rendrent the more absorbent activated carbon.

We undertook the characterization of the organic groupings of surface by NMR of the solid. Indeed spectrum NMR-¹H of sample CG-700 (Fig. 9) presents a wide strip at 0.30 ppm, which can be allotted to groupings methyl. It is visible in spectrum NMR-¹³C with 11 ppm. Graphitic carbon sp² appears with 122.64 ppm. The groupings carbonyls are not visible (Fig. 10).

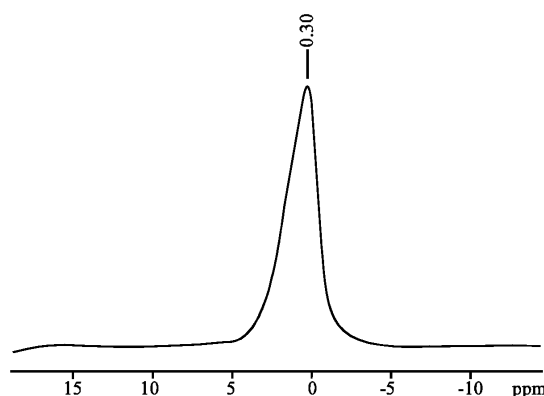


Fig. 9: Spectrum NMR-¹H of carbonisat CG-700

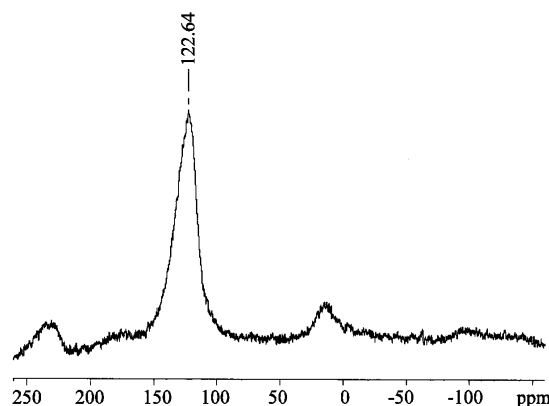


Fig. 10: Spectrum NMR-¹³C of carbonisat CG-700

CONCLUSION

The process of carbonization and activation was carried out in only one stage at a temperature of 700°C under a water vapour current. The pyrolysats obtained were characterized by ultimate analysis, spectroscopic methods (FTIR and NMR), specific surface and porous volume.

The determination of the capacities of adsorption of the activated carbon was carried out with assistance FTIR, by using the methylene blue, benzene and iodine. The characteristics of the products obtained are comparable with the commercial activated carbon.

The result from the whole of this research that the olive cake residue is a biomass which can be useful for manufacture of the activated carbon with acceptable properties.

Finally the know-how acquired in this research should enable us to approach the study of modelling and the optimization of the method of activation with the addition of polymers and the study of the physico-chemical properties (standard of the specific pores, surfaces, porosity, etc.) according to the reactional parameters.

The study of the volatile compounds formed during the preparation of the activated carbon represents the 2nd future aspect of this research.

ACKNOWLEDGEMENT

Participations of Mrs. Pr. Faouzi Bouachir, Pr. Abdelhamid Ghorbal, Pr. Moheddine Fourati and our Japanese friend H. Sido in experimental research are acknowledged.

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