

# Characterisation and utilisation of a new activated carbon obtained from Moroccan olive wastes

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**ABSTRACT:** A comparative study of the adsorption of some organic compounds (methylene blue, iodine, phenol, atrazine, diuron and sodium dodecylsulphate (DSS)) was carried out using activated carbon obtained by the utilisation of Moroccan olive wastes and commercial carbon from wood, coal and black plant. This new carbon was prepared by physical activation in the presence of steam. Our results show that the prepared carbon performs very well; it has a specific surface determined by adsorption of nitrogen (N<sub>2</sub>) and isotherm BET, of 1200 m<sup>2</sup>/g. The maximum capacities of its adsorption according to the Langmuir model are 1.26 mmol/g (403 mg/g) for methylene blue, 4.46 mmol/g (1131 mg/g) for iodine, 1.95 mmol/g for phenol, 1.56 mmol/g for atrazine, 1.98 mmol/g for diuron and 0.82 mol/g for DSS superior to those obtained with commercial carbon. Tests on the discolourisation of sugar syrup have shown that it is as satisfactory as the commercial carbon used by sugar mills in Morocco.

## INTRODUCTION

At present, activated carbon can be derived from many different materials—those which are based on coal, wood or coconut are the most widely used. These types of carbon are expensive and are often imported, increasing further the cost of water treatment, and hence making it necessary that developing countries find a cheaper material for the preparation of activated carbon for use in industry, drinking water purification and waste water treatment.

Some authors have studied the feasibility of the preparation of activated carbon from by-products of agricultural and industrial wastes, for example, the case of olive stones [1], ears of palm tree [2], waste apple pulp in cider production [3], agricultural by-products [4], macadamia nut shells [5], waste husks of *Moringa oleifera* [6], fertiliser waste slurry [7], straw [8], and apricot stones [9,10].

The aim of this study was to test a new activated carbon prepared using waste from the farm food industry (olive wastes from Morocco) by physical activation in the presence of steam. Adsorption experiments were carried out in a discontinuous reactor with the organic molecules: methylene blue, iodine, phenol, two herbicides (atrazine and diuron), an anionic surfactant (sodium dodecylsulphate, DSS) and sugar syrup.

The choice of these molecules was justified by their properties: the macroporosity of carbon is often tested by its affinity to the methylene blue molecule, the iodine and phenol molecules give interesting information on the microporosity of the material, while herbicides and surfactants are polluting elements which cause problems for the environment.

In Morocco, the industrial preparation of white sugars includes manufacturing processes which ultimately eliminate

the colour of the sugar. The discolourisation step using activated carbon or black plants is highly necessary for this process.

The results of adsorption experiments were compared to those obtained from commercially available activated carbon (Filtrisorb F400 from Chemviron, Ceca Anticarbon 25K, Picazine and the black plant used by the Tadla sugar mill (SuTa)).

## MATERIALS AND METHODS

### Preparation and characterisation of the new activated carbon

#### Carbonisation

The carbonisation of raw material is a method for eliminating volatile products by the degradation of organic material in such a way as to retain—in addition to mineral elements—only a carbonised skeleton. The operational conditions which are used during carbonisation determine the porous texture which cannot be modified later.

The experiments were carried out in a 125 mL stainless steel Prolabo autoclave. The reactor was heated by means of an electrical oven, whose temperature was controlled by a programmer linked to a thermocouple. The equipment also included a system of inert gas sweeping (using nitrogen), which permitted a rapid evacuation of any volatile elements into a trap kept at 0 °C, thus facilitating the retrieval of the condensable products of the carbonisation.

The olive waste was dried at 120 °C overnight, and then placed in the reactor vessel which was then raised to the final (maximal) temperature. After the carbonisation was complete, the passage of the vector gas and its evacuation was halted to

avoid any entry of air. After the apparatus had cooled, the carbonised carbon was retrieved, dried, weighed and retained for the activation process.

#### Activation

All the activation reactions were performed in a thermolyne silice electric oven with a temperature control device linked to a thermocouple, a silicon reactor and a steam generator was placed at the entrance of the reactor (Fig. 1).

The carbonised carbon was placed in the reactor and the system was set at the temperature of  $900\text{ }^{\circ}\text{C}$  ( $\pm 2\text{ }^{\circ}\text{C}$ ), which was maintained during the injection of the steam. At the end of activation, the oven was cooled to the ambient temperature. The activated carbon was retrieved and boiled for 30 min in distilled water. It was then washed, dried, and weighed, and then ground in a mortar (90% of the final particles had an average diameter of about  $54\text{ }\mu\text{m}$ ). It was then retained for the subsequent analyses.

#### Characterisation

The activated carbon which had been prepared was then examined by scanning electron microscopy (SEM) (a Philips XL 30) in order to observe its porosity and texture.

The specific surface of the new activated carbon was determined by the adsorption of nitrogen at  $77.3\text{ K}$  and isotherm BET. The appliance which was used for these measurements was a discontinuous volumetric instrument (Micrometrics ASAP 2000 V2.02). Degassing was carried out for 18 h under a primary dynamic vacuum at a temperature of  $300\text{ }^{\circ}\text{C}$  and a

pressure of  $8 \times 10^{-3}\text{ mmHg}$ . The tolerance on the relative pressure ( $P/P_0$ ) was 5%, the interval between measurements of  $P_0$  (pressure of saturation) was 120 min

The dosage of the surface functions of activated carbon was carried out according to the method of Boehm [11].

The electrokinetic potential (zeta potential) of the CAP particles was determined using shaken suspensions of  $50\text{ mg/g}$  CAP using a Pen Kem Lazer Zee-Meter apparatus, model 500 (at a voltage of  $100\text{ V}$ ). The zeta potential values ( $\pm 5\%$ ) were corrected for all variations in temperature in the course of the experiment, and are therefore expressed at a reference temperature of  $20\text{ }^{\circ}\text{C}$ .

#### Commercial products and chemicals

##### Commercial activated carbons

- Activated carbon, Filtrasorb-400 from Chemviron, which has a mineral origin, and has been activated at a high temperature ( $900\text{--}1000\text{ }^{\circ}\text{C}$ ) in the presence of oxygen, is a mesoporous carbon whose specific surface, determined by the adsorption of nitrogen and isotherm is  $1050\text{ m}^2/\text{g}$ .
- Anticarbon 25K carbon from Ceca, which has a plant origin (the wood of maritime pine trees) is activated with steam at a high temperature ( $900\text{--}1000\text{ }^{\circ}\text{C}$ ). Its specific surface (provided by the manufacturer) is  $950\text{ m}^2/\text{g}$ .
- Activated carbon from Picazine is manufactured from charcoal which has been activated at  $500\text{ }^{\circ}\text{C}$  and has a specific surface of  $640\text{ m}^2/\text{g}$ .

Activated carbon used in the sugar industry in Morocco (SuTa).

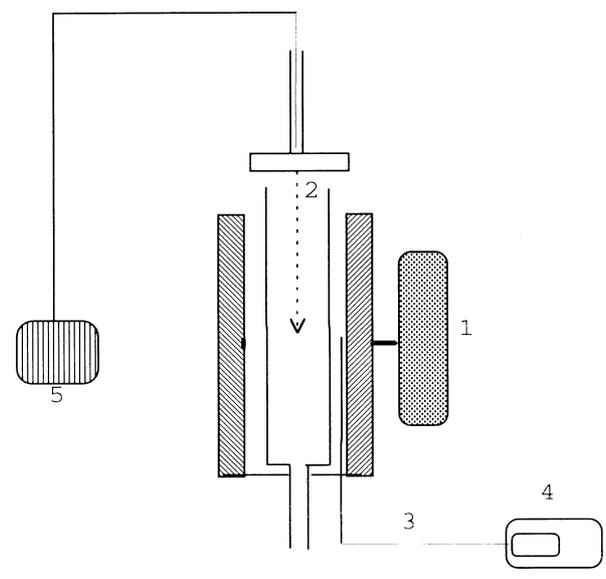
All the powdered activated carbons (CAP), all of which had grains smaller than  $50\text{ }\mu\text{m}$ , were dried for 24 h at  $105\text{ }^{\circ}\text{C}$ , and then kept in a sealed bottle. They were used without further treatment for the adsorption tests.

##### Chemical used

- Methylene blue:  $\text{C}_{16}\text{H}_{18}\text{ClN}_3\text{S}$ ; a 99.5% pure RP Prolabo product.
- Iodine: a 99.5% pure RP Prolabo product.
- Phenol:  $\text{C}_6\text{H}_6\text{O}$ ; a pure RP Prolabo product (99%).
- Atrazine: 2-chloro 4-N-ethylamino 6-N-isopropylamino-S-triazine (Chem service, France), 99.5% pure. Its solubility in water was  $35\text{ mg/L}$ .
- Diuron 3-(3,4-dichlorophenyl)-1,1-dimethyluree (Chem service, 98% pure). Its solubility in water was  $42\text{ mg/L}$ .
- Sodium dodecylsulphate (DSS):  $\text{CH}_3(\text{CH}_2)_{11}\text{SO}_4\text{Na}$  (Aldrich), 98% pure.
- The sugar which was used was a first extraction type, exhibiting an initial colour of 480 ICUMSA units [14].

#### Analytical methods

Atrazine and Diuron were dosed by high pressure liquid chromatography (HPLC). Dosing was carried out by an



**Fig. 1** The activation device. 1, furnace; 2, reactor; 3, thermocouple; 4, temperature controller; 5, water vapour generator.

	CAP-GO	F400	Céca	SuTa	Picazine
Origin	Olive wastes	mineral	wood	vegetal	wood
Activation	physical	physical	physical	–	chemical
Surface function					
Acid (mmol/g)	1.07	1.05	0.3	0.81	3.5
Basic (mmol/g)	2.67	0.45	0.59	0.33	0.02
$\zeta$ (mV)	–34.2	–21	–21	–30	–21
SSA (m <sup>2</sup> /g)	613	565.9	537.6	226.4	289.2
SS (m <sup>2</sup> /g)	1200	1050	950	–	640

SSA: apparent specific surface calculated [16].

SS: Specific surface (BET).

$\zeta$ : Electrokinetic potential.

Hitachi L-6200A fitted with an L-4000 UV detector, an Hitachi 2500 integrator and an Hitachi AS-200A auto-sampler. The mobile phase was a mixture of methanol and water (80/20). The flow rate was set at 0.5 mL/min. The injection volume was 50  $\mu$ L for Atrazine and 100  $\mu$ L for Diuron. Their detection wavelengths are 222 nm and 248 nm, respectively.

After determining their range of adsorption in the UV–visible spectrum, the aqueous solutions of methylene blue and phenol were measured by spectrometry at 680 nm (270 nm for the phenol) using a Shimadzu model A160.

Iodine was dosed by the sodium thiosulphate volumetric method.

In aqueous solution, DSS combines with methylene blue to form a complex which is extractable by chloroform, and is dosed by spectrometry at 650 nm [12,13].

The discolourisation test sugar was performed using a 420 nm colourimeter, according to the method developed by ICUMSA [14].

Metallic ion levels were dosed by atomic graphite furnace adsorption spectrometry (Varian, spectra AA-800).

### Operation proceedings

All the solutions were prepared from distilled water (pH = 5.5,  $\chi = 2 \mu$ S/cm). Adsorption tests were carried out in a discontinuous reactor at  $20 \pm 2^\circ\text{C}$  using 100 mL volumes, constantly shaking them for a variable period, and fixing them by kinetic tests in the presence of a variable quantity of CAP ( $m$ ) and adsorbates at an initial concentration =  $C_0$ . No variation in pH was observed during the suspension of small quantities of CAP by the end of the adsorption tests.

When the contact time (determined by kinetic testing) had elapsed, the CAP suspensions were filtered on 0.3  $\mu$ m cellulose nitrate. The filter element was then measured in order to evaluate the residual concentration ( $C_r$ ) in solution. The adsorbed concentration ( $C_{ad}$ ) was obtained by subtracting the difference:

$$C_{ad} = C_0 - C_r$$

**Table 1** Characteristics of activated carbon prepared both commercially and from olive wastes

Control tests were carried out for each mixture and showed no detectable loss of adsorbed material by filtration for the concentrations which we employed.

All results were based on triplicates of each test.

### The Langmuir mathematical model

The adsorption results were compared by means of Langmuir model in the form of a linearised saturation function [15].

$$\Gamma = \Gamma^\infty K C_r / (1 + K C_r)$$

or

$$1/\Gamma = 1/\Gamma^\infty + 1/(\Gamma^\infty K C_r)$$

$\Gamma^\infty$  ( $C_{ad}/m$ )<sub>max</sub> = maximal capacity of adsorption in (mmol/g)

$\Gamma$  ( $C_{ad}/m$ ) = capacity of adsorption in (mmol/g)

$C_r$  residual concentration in solution in (mmol/L)

$K$  thermodynamic constant of adsorption in (L/ $\mu$ mol<sup>–1</sup>)

## RESULTS AND DISCUSSIONS

The best conditions for activated carbon preparation were found to be: a carbonisation temperature of 400  $^\circ\text{C}$ , an activation temperature of 900  $^\circ\text{C}$ , an activation time of 30 min, a grain size before activation of between 0.08 and 0.125 mm, a sample mass of 5 g (corresponding to the reactor being half full), a steam flow of 0.1 cm<sup>3</sup>/min, and a rate of increase in temperature of 25  $^\circ\text{C}/\text{min}$ .

After activation, the carbon was boiled for 30 min in distilled water, and then washed, dried, weighed, ground and sifted to obtain a powder with a grain size < 54  $\mu$ m. This powder was then dried at 110  $^\circ\text{C}$  until it remained a constant weight, and was kept in hermetically sealed bottle.

### Behaviour of the activated carbon prepared in water

A record of the pH variations and the conductivity of the distilled water in contact with the prepared CAP (CAP-GO) showed that the suspensions of this carbon have a basic characteristic of greatly increasing the pH value (from 5 to

**Table 2** Analysis of washing water of CAP-GO

Metallic ions (mg/g)	Ca <sup>2+</sup>	Mg <sup>2+</sup>	Fe <sup>3+</sup>	Al <sup>3+</sup>	Cr <sup>3+</sup>	Cu <sup>2+</sup> , Cd <sup>2+</sup> Pb <sup>2+</sup> , Zn <sup>2+</sup>
CAP washed with distilled water	14	0.8	ND*	ND	ND	ND
CAP washed with HCl (1 M)	20	1.0	0.83	5.5	0.007	<5 × 10 <sup>-4</sup>

\* ND: not detected.

8.5). In contrast, the surface pH of the carbon which is used by SuTa is slightly acidic (pH 5.5–6.2).

The pH value reached a maximum after a contact time of 20 min in both cases. This variation in pH was accompanied by an increase in the conductivity of the middle, from 2 to 125  $\mu\text{S}/\text{cm}$  for the prepared CAP and from 2 to 175  $\mu\text{S}/\text{cm}$  for the SuTa CAP. This rise was related to another release of metallic ions into solution.

The increase in the quantity of metallic ions released by the carbon after washing demonstrates the ability of this material's surface to release these ions again by way of eventual ionic exchange with the aqueous middle (adsorption of protons and release of cations), which leads to a reduction in surface pH.

Table 2 presents the results of an analysis of the washing water (300 mg of CAP-GO in 100 mL water) using distilled water and HCl (1 M). It can be seen that the Ca, Mg and Al ions are again released in significant quantities and must therefore constitute the principal elements which contribute to the increase in conductivity.

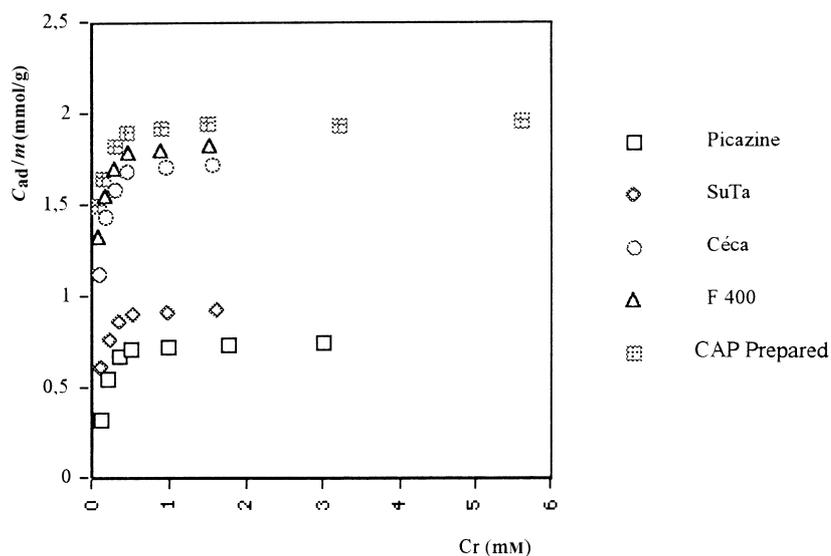
In order to compare the prepared carbons, called CAP-GO, with the commercial carbons which are used in water purification and the sugar industry for sugar discolourisation, we carried out some adsorption tests.

### Adsorption of organic molecules

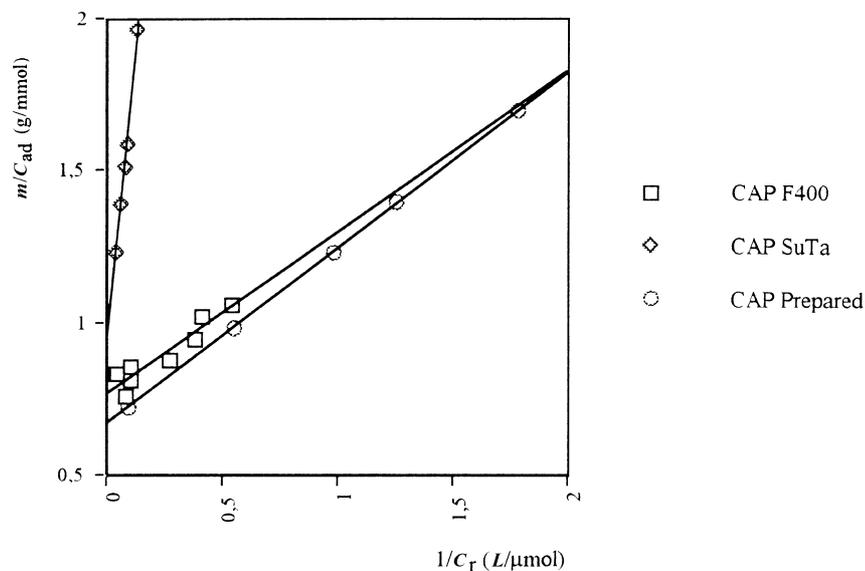
Contamination by organic micropollutants (pesticides, surfactants, phenol, etc.) from the aquatic milieu is a problem for the officials responsible for the protection of the environment, and more particularly for those who have to maintain water quality. If these pollutants resist the standard treatments, adsorption on activated carbon is one of the most suitable procedures likely to eliminate the majority of them.

The results of adsorption of organic molecules on different activated carbons in the form of Langmuir's adsorption isotherm, will demonstrate the efficiency of the prepared carbon. In Fig. 2, the results of the adsorption of phenol on the different types of carbon are presented. These results show that the carbon prepared from olive wastes has a high adsorption capacity, superior to that which was obtained with other carbons.

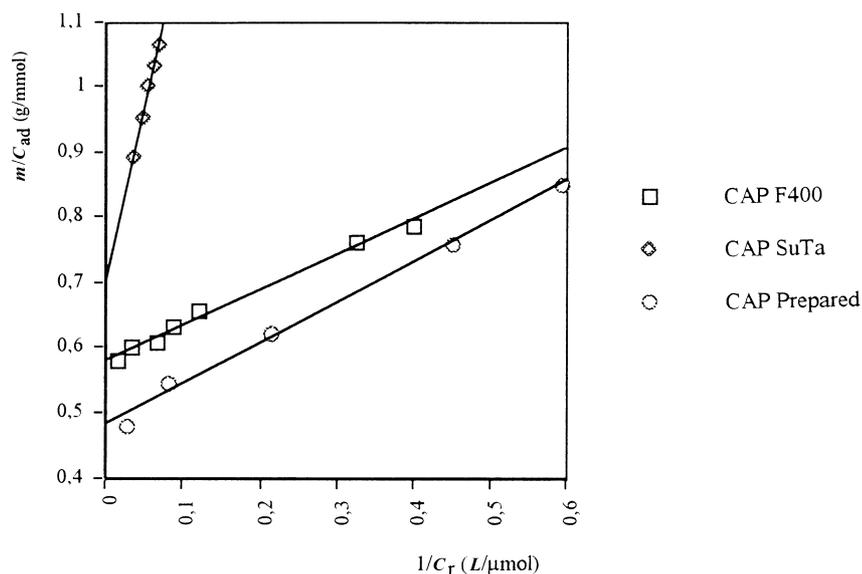
Figures 3 and 4 and Table 3 show the Langmuir isotherms of adsorption of the other organic compounds which we studied, and also some of the characteristics of the activated carbons. We can see that the prepared activated carbon is always more satisfactory than the other types of carbon—the adsorption in this case is of a hydrophobic nature by way of interactions of the van der Waals type and hydrogen bonds.



**Fig. 2** Phenol adsorption: experimental isotherms of the various adsorbents at  $20 \pm 2^\circ\text{C}$ , contact time 3 h.



**Fig. 3** Atrazine adsorption: Langmuir isotherms of the various adsorbents.



**Fig. 4** Diuron adsorption: Langmuir isotherms of the various adsorbents.

**Table 3** Comparison of the adsorption capacity  $\Gamma^\infty$  (mmol/g) of the molecules on the prepared activated carbon and on commercial activated carbon

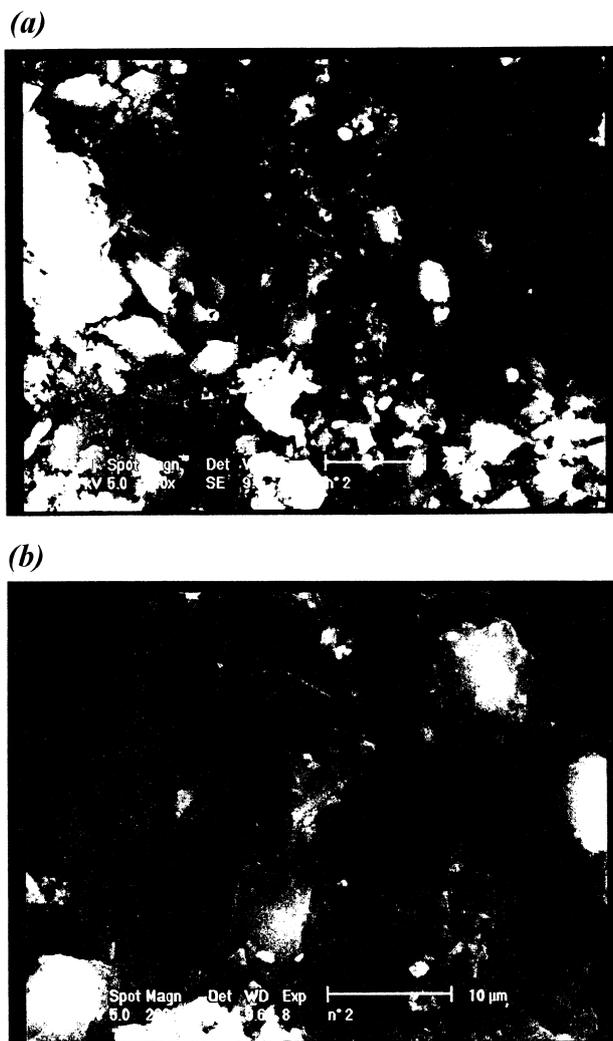
	CAP-GO	F400	Céca	SuTa	Picazine
Methylene blue	<b>1.26</b>	0.88	–	0.38	–
Iodine	<b>4.46</b>	4.33	4.13	1.57	3.63
Phenol	<b>1.95</b>	1.8	1.70	0.92	0.72
Atrazine	<b>1.56</b>	1.42	0.70	1.0	1.07
Diuron	<b>1.98</b>	1.72	1.39	1.4	1.25
DSS	<b>0.82</b>	0.68	0.43	0.46	0.26

An estimate of the apparent specific surface (SSA) has been calculated on the basis of the adsorption of phenol (method of Giles & Nakhwa [16]) using the equation:

$$SSA = \Gamma^\infty N_A A_m$$

where  $\Gamma^\infty$  = maximal adsorption capacity obtained at saturation;  $N_A$  = Avogadro's number;  $A_m$  = Surface occupied by molecule of phenol  $0.255 \times 10^{-8} \text{ m}^2$ .

The values of  $\Gamma^\infty$ , and of the apparent specific surface calculated on the basis of the adsorption of phenol, show the development of the porosity of the prepared carbon. This was



**Fig. 5** Surface structure of the prepared carbon determined by scanning electron microscopy. Magnification: (a)  $\times 800$ , (b)  $\times 2000$ .

confirmed by the skeletal morphology observed in representative SEM micrographs of the activated olive wastes (Fig. 5).

The main characteristics of CAP-GO obtained were compared to those of the other carbons (Table 1).

#### Adsorption of nitrogen

The adsorption/desorption of nitrogen at 77 K on the prepared activated carbon, and the application of the BET theory show a predominant presence of micropores, which justifies the use of the concept of 'equivalent BET area'. It can be seen that the mathematical treatment leads to an equation called 'transformed linear BET', which is illustrated in Fig. 6. This equation has been verified for values of  $P/P_0$  which lie between 0.05 and

0.20. The value of  $V_m$  is  $273.2 \text{ cm}^3/\text{g}$  and the correlation coefficient is 0.999.

This apparent specific surface of CAP-GO (prepared from olive wastes) of  $1200 \pm 20 \text{ m}^2/\text{g}$  demonstrates the high quality of olive wastes as a material likely to be used for the preparation of activated carbon.

According to Sing [17], the BET model takes no account of micropore filling processes, and the true surface area may be significantly greater than the apparent value determined by this model.

#### Test on sugar syrup

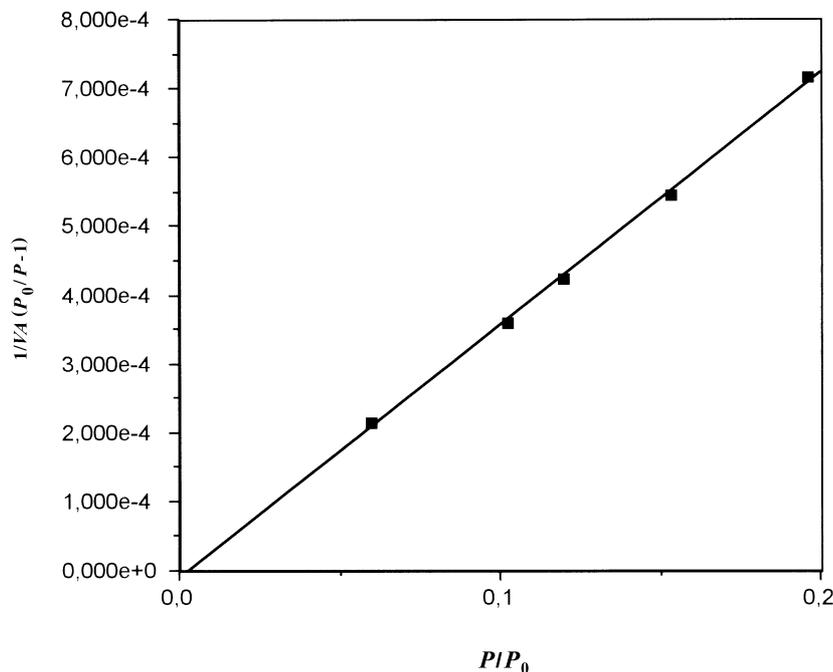
Information about the nature of the most common colouring materials will be of great importance for the sugar industry, mainly during the discolourisation stages. In the present case, recent studies have shown the acid characteristic of the colouring elements, and their high molecular masses which can exceed  $100\,000 m_a$  [18]. Indeed, the composition of the juice combined with the manufacturing process favour the formation of colouring materials up to the high point of polymerisation.

In order to study the effect of the prepared activated carbon on the discolourisation of sugar syrup, the results of the investigation are compared with the activated carbon used in the sugar industry in Morocco (the SuTa case). Table 4 illustrates the results of discolourisation using two concentrations for each type of activated carbon (4 and  $6 \text{ mg}_{\text{sugar}}/\text{g}_{\text{carbon}}$ ).

These results show that the activated carbon (CAP-GO) has an important discolouring effect (61%) but that it remains slightly inferior to CAP-SuTa (78%), although this latter absorbs less methylene blue and iodine. This variation can be explained by the difference in porosity (the CAP-SuTa is macroporous while the CAP-GO is microporous) on the one hand, and by the nature of the carbon surface, which is basic (increase of pH of distilled water in contact with carbon) while that of SuTa carbon is slightly acidic. This has been confirmed by the dosage surface functions according to the method of Boehm; indeed, the surface of the prepared carbon contains  $1.07 \text{ mmol/g}$  of acid sites and  $2.67 \text{ mmol/g}$  of basic site (Table 1) The dominant colouring elements are negatively loaded, which makes it possible to have an additional adsorption which is would be the result of the favourable interactions

**Table 4** Percentage of discolourisation of sugar syrup after treatment by activated carbon

CAP	SuTa	CAP-GO	CAP-GO treated with HCl (1 M)
4 ( $\text{mg}_{\text{CAP}}/\text{g}_{\text{sugar}}$ )	69	48	67
6 ( $\text{mg}_{\text{CAP}}/\text{g}_{\text{sugar}}$ )	78	61	76



**Fig. 6** Nitrogen adsorption: experimental isotherm at  $-196^{\circ}\text{C}$  on activated carbon prepared from olive wastes.

between the negative loads of the colouring elements and the surface of the acidic carbons.

In a second phase, and to study the effect of the pH of the surface, we have undertaken a treatment of the carbon CAP-GO (300 mg CAP in 100 mL chlorhydric acid, 1 mol/L) in order to bring the surface pH closer to that of CAP-SuTa.

The results of discolourisation by activated carbon (CAP-GO washed by HCl) show that the acid treatment improves the discolourisation of the sugar (from 61% to 76%), which displays the effect of  $\text{H}_3\text{O}^+$  ions in favouring the attraction of the negatively-loaded colouring elements, while the effect of the metallic ions found on the surface is negligible.

## CONCLUSION

The adsorption tests on the new activated carbon obtained from olive wastes has shown that this material performs well. It can be put to various uses: drinking water treatment (the elimination of pesticides, surfactants, etc.), and the treatment of both waste water and industrial water (elimination of phenol and other polyaromatic molecules such as colouring elements). Our results confirm the adsorbant qualities and performances of this activated carbon as compared to other, more widely used, commercial carbons which are prepared from coal, wood or vegetal coal. The adsorption of nitrogen, and the BET isotherm of CAP-GO shows that it has a very large specific surface ( $1200\text{ m}^2/\text{g}$ ) which is superior to that found in commercial carbons. This material has a large capacity for the adsorption of organic molecules which we studied. The discolourisation tests which we carried out on the sugar syrup show that it has a great affinity for the compounds which are

responsible for colourisation and that it can be satisfactory in sugar industry.

These results demonstrate the various applications of carbon prepared from olive wastes. In addition it has the following advantages: the utilisation of waste, the preservation of the environment by guarding against deforestation, its availability, the low raw material cost, and the possibility of a local production, implying a reduction in the cost of water treatment.

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