



Full length article

# Possibility of using olive-seeds residues as a source of activated carbon

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## ABSTRACT

Presents study shows the effect of different activation methods to produce activated carbon from olive seeds residues. Pre-treatment of the seeds with carbonization at 400 °C for 30 min. The produced char impregnate in KOH solution (1:1 ratio) for chemical activation. After that, the char was dried at 105 °C for 60 min in the oven. Four different sizes (3, 4, 5, and 6 mm) of the produced char were achieved. Samples of 100 g of char were activated at four different temperature levels of (250, 350, 450 and 550 °C) for activation time of (15, 20, 25 and 30 min) respectively. Four gage pressures of (atmosphere, 1.3, 1.6 and 1.9 bars) were achieved in the activation process at constant nitrogen flow rate 50 cm<sup>3</sup>/min. The results showed that there is a clear effect on the surface area of the produced activated carbon, the best value of surface area for the produced activated carbon was 1091.58 m<sup>2</sup>/g at maximum iodine absorption of 1099.9 mg/g, activation temperature 450 °C, activation time 25 min, practical size 3 mm and atmosphere pressure.

## 1. Introduction

The olive tree is considered one of the first trees cultivated in human history, with implications for religion (Greek mythology and the Old Testament) and social life of our ancestors. For example, the olive plant has been a symbol of peace and friendship since ancient Greece, when the Olympic Games winners were honoured with a garland of olive branches (Kapellakis et al., 2008; Valvez et al. 2021). The interest in the activated carbon production has grown significantly in the whole world, mainly because the properties of activated carbon can be tailored for many applications and it can be produced from bio-based material, such as different agricultural residues types such as (date palm stone, olive stone and woods) (Davide et al., 2022). The carbonization creates initial porosity and orders the carbon

structure to enrich the carbon material; the activation enhances the carbon structure by widening the pores, making it more porous (Daud and Ali, 2004). Carbonation involves pyrolysis of the raw material at a temperature range of 600 – 900 °C in the absence of oxygen (in inert atmosphere with gases like argon or nitrogen) during which volatile components are removed, producing a residual carbonaceous product with low surface area. Activation can be performed either by physical/thermal or chemical methods (Subramani and Revathi, 2015). Physical activation process involves treatment of the char obtained from carbonization with gasification reactants/oxidizing gases such as steam, carbon dioxide, air or a suitable combination at high temperature (400 – 1000 °C) (Ahmida et al., 2015). The considering the development of porous structure of bituminous coals

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under carbon dioxide atmosphere or inert gas and elevated pressure are available. The porous activated carbon is produced when the oxidant converts the carbon materials to form carbon mono-oxide (CO) and carbon dioxide (CO<sub>2</sub>) thus opening pores in the activated carbon materials (Natalia, 2019). Chemical activation process involves treatment of the char obtained from the carbonization with chemicals such as alkali and alkaline earth metal containing substances (KOH, NaOH, and Na<sub>2</sub>CO<sub>3</sub>), acid (H<sub>3</sub>PO<sub>4</sub>, HCl, H<sub>2</sub>SO<sub>4</sub> and C<sub>2</sub>H<sub>4</sub>O<sub>2</sub>) and other chemicals/salts (ZnCl<sub>2</sub>, CaCl<sub>2</sub> etc), the chemicals functioning as dehydrating or oxidizing agents, influencing the pyrolysis decomposition, and inhibiting the formation of tar. This process is usually done at lower temperature (500 to 800°C) and activation time, giving surface area, better porosity and lower energy cost as compared to physical activation alone (Ahmad et al., 2015). The carbonization step consists of a pyrolysis procedure, in which, volatile compounds are released due to several complex, rival, and consecutive reactions leading to the achievement of fixed carbon in an inert atmosphere and at temperature range of 400–500 °C. In the second step of the physical activation process, the precursor is placed under a heat treatment at high temperatures from 700 to 1100 °C in the presence of oxidizing agents, such as CO<sub>2</sub>, water steam, air, or a mixture of them (Pezoti et al., 2014). Depending on the intended usage of the activated carbon, the environmental issues involved, cost and availability, several chemical reagents have been effectively used as activating agents, common examples include phosphoric acid (H<sub>3</sub>PO<sub>4</sub>), zinc chloride (ZnCl<sub>2</sub>), aluminum chloride (AlCl<sub>3</sub>), sulphuric acid (H<sub>2</sub>SO<sub>4</sub>), potassium hydroxide (KOH), sodium hydroxide (NaOH) and potassium carbonate (K<sub>2</sub>CO<sub>3</sub>). Among these, the most commonly used, industrially are zinc chloride, phosphoric acid and potassium hydroxide (Marsh and Rodriguez, 2006; Foo and Lee, 2010; Yusufu et al., 2012). The aim of the carbonization stage is to conserve the carbonaceous structure of the material which is achieved by burning off the material in a range of temperatures from 400 to 850°C (Refas et al., 2010).

## 2. Materials and methods

This work was carried out in the Faculty of Agricultural Engineering, Al azhar University, Assiut branch during (2020). The main objective of the present work is to study some factors affecting the activated carbon production from agricultural wastes using physicochemical process. In order to achieve the production of activated carbon from agricultural waste (olive seeds residues) the following specific objectives were pursued:

Producing and studying some characterization of activated carbon from olive seeds. Determination the yield, density and the surface area per iodine number of

activated carbon. For producing activated carbons from olive seeds residues, two-steps should be carried out through the physiochemical method. The first step is a thermal treatment of the raw material that implies dehydration and where most of the non-carbon elements as dust and volatile substances are eliminated by heating the source in anaerobic conditions. The second step is activation in range of different levels of temperatures and pressures.

### 2.1. Material

#### 2.1.1. Raw material

The olive seeds, residues used in this work were collected from the food manufacturing industries the collected olive seeds residues were washed exhaustively with deionized water to remove adhering dirt particles from the surface. The olive seeds residues were dried naturally in the sun, after that undesired particles and dust were removed by screening mesh. Table 1 shows the chemical composition of the seeds before carbonization.

**Table 1**

Chemical composition of the olive seeds residues

Elements	N	C	O	H	S	Ash
Olive seeds residues	1.87	48.67	7.92	0.19	41.2	3.7

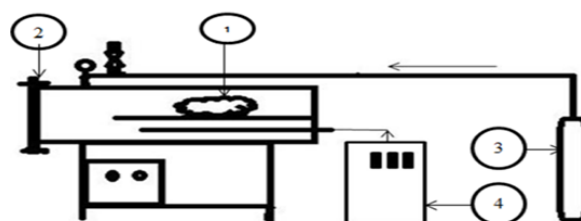
Source: Ahmad et al. (2015).

#### 2.1.2. Potassium hydroxide (KOH)

This chemical agent is an inorganic compound with the formula KOH. It is a strong base and is used in many industrial applications. Some of its characteristics are its high reactivity toward acids and its corrosive nature. The potassium hydroxide solution (99.9) used in this work were obtained from the chemical laboratory of sciences college Al-Azhar University Assiut branch.

#### 2.1.3. Horizontal metal Furnace

The furnace was constructed using a metal sheet made of mild steel of high heat resistance. Fig. 1 show that schematic diagram of the used furnace.



**Fig. 1.** Schematic diagram of horizontal furnace 1) Sample. 2) Furnace. 3) Gas N<sub>2</sub> cylinder. 4) electric resource

## 2.2. Test procedures

The olive seeds residus were carbonized and activation in the electrical oven and constructed metal furnace respectively. The seeds were carbonized at 400°C for 30 min. Four different activation temperatures were used at for different activation time and gage pressure. The produced char after carbonization process was sieved to four different sizes. The produced activated carbons were studied to measure yield, density, surface area and iodine value.

### 2.2.1. Carbonization

Were left for cooling, the carbonization process was performed by loading 1 kg of dried olive-seeds into an electrical oven ranged (250-750 °C), and heated up to a carbonization temperature of 400 °C for 30 min. the produced char after carbonization were left to cool after that the produced char screen manually desired different sizes of (3, 4, 5 and 6 mm) in the laboratory of soil and water department in collage agricultural Al-Azhar University Assuit branch. About 25 kg carbonized materials (char) give 256 samples of each sieved product. The samples were carbonized according to (Yang et al., 2020).

### 2.2.2. Activation

A sample of 100 g was loaded into the horizontal metal furnace and heated up to different temperatures for different times. Temperature levels of (250, 350, 450 and 550 °C) for activation time of (15, 20, 25 and 30 min) respectively. Four gage pressures of (atmosphere, 1.3, 1.6 and 1.9 bars) were achieved in the activation process at constant nitrogen flow rate 50 cm<sup>3</sup>/min. During the activation a constant flow of 50 mL/min of nitrogen gas was assured to keep inert the atmosphere. The choice of activation temperature and heating gradient were made according to (Davide et al., 2022). Four samples were selected randomly of each parameter to determine the moisture content according (ASAE, 201; Gottipati 2012); the moisture content was determined to produced activated carbon after activation process and before measurement's.

## 2.3. Experimental design

In the present work, olive-seeds were collected from the food manufacturing industries, washed and dried at 110 °C for 60 min then carbonization at 400 °C for 30 min, a combination of four practical sizes (3, 4, 5 and) and activation temperature for (250, 350, 450 and 550 °C) four activation time (15, 20, 25 and 30 min) and four gage pressures (Atmosphere, 1.3, 1.6 and 1.9 bar) resulting, in 256 treatments. Each treatment was repeated three times to give three replicates resulting in 768 samples.

## 2.4. Measurements

The following Measurements were carried out at ambient room temperature of (25°C), and relative humidity of (40 %).

### 2.4.1. Activated carbon yield

According to Adinata et al., 2007 the mass of each sample was recorded after activation as before it (initial weigh). The mass yield from each sample was calculated as the mass of olive seeds-based AC divided by the initial sample mass (100 g) used for carbonization and activation. The percent yield was expressed using the following equation:

$$\text{Yield} = \frac{W_c}{W_o} \times 100 \quad \dots [1]$$

Where:  $W_c$  and  $W_o$  are the final dry product weight (g) and the dry raw material weight (g), respectively.

### 2.4.2. Activated carbon bulk density

The Bulk density was determined using method (ASTM D2854, 2014). The initial weight of the empty tube was determined by weighing. Sample of 100 g was put into a tube and tapped on bench top. The final weight of both the tube and the sample was determined after tapping. The bulk density was calculated according to following equation:

$$B = \frac{W_a}{V_o} \quad \dots [2]$$

where: B is bulk density (g/cm<sup>3</sup>),  $W_a$  weight of sample and  $V_o$  is the volume occupied by the packed sample

### 2.4.3. Iodine value

According to (Genli et al., 2021) the total IN adsorbed carbon microsphere at time, t, qt (mg/g) can be determine using the following equation:

$$qt = \frac{(C_o - C_t) V}{M} \quad \dots [3]$$

where :qt (mg/g) is the equilibrium adsorption capacity,  $C_o$  represents the initial concentration of the solution (mg/L),  $C_t$  represents the concentration of the solution at time t (mg/L), V is the volume of the solution (L), and M is the weight of dry adsorbent (g).

### 2.4.4. Surface area

The determination of surface area was performed by the adsorption of iodine method (ASTM D4607, 2014) instrument to determine the specific surface area of the activated carbon samples. The Surface area was calculated using:

$$S = \frac{X N}{M} \quad \dots [4]$$

where:  $S$ = Surface area ( $m^2/g$ ),  $X$ = iodine value ( $mg/g$ ),  $N$ = Avogadro number and  $M$  = Mass of Activated carbon used.

#### 2.4.5. SEM analysis

The SEM images were recorded with Scanning Electron Microscope (JEOL; JSM-6380A) equipped with an electron probe analyzer system (Landing Voltage 20.0 kV) agreed with (Samorm et al., 2011). The SEM images carry out in the Faculty of Electron Microscope Unit Faculty Of Science Alexandria University.

### 3. Results and discussions

#### 3.1. Activation time via yield

Fig. 2 Showed that the yield of activated carbon is linearly decrease by increasing activation time, activation temperature and pressure with coefficient of determination range between 52.57 to 19.5 %. The obtained data indicated that the mass is decrease 24 to 37.1%. The rate of weight loss is high primarily due to the initial large amount of volatiles that can be easily released with increasing temperature as well as the loss of moisture to a lesser extent agreed with Adinata et al., 2007.

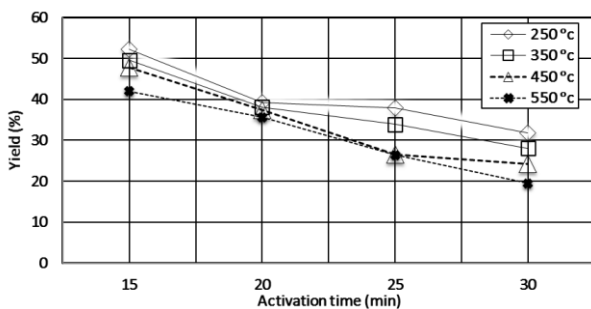


Fig. 2. Effect of parameters condition on yield.

#### 3.2. Activation time via bulk and true densities

The bulk and true densities of activated carbon shown in Figs. 3 and 4 decreased from 0.85 to 0.44  $g/cm^3$  and from 1.26 to 1  $g/cm^3$  respectively with increased activation time from 15 to 30 min at activation temperature constant. This may be due to the increase in the loss of volatile solids with an increase in the temperature and activation time, or the porosity development during activation process and these results are consistent with Abechi et al., 2013. Fig. 3 showed that the bulk density decreased with increase in activation time for the activated carbon. Similar results of decrease in bulk density with increase activation time and temperature of activating agent were obtained by Yakoot and El-Deen, 2016.

#### 3.3. Activation temperature via surface area and iodine value

The initial increase of surface area could be due porosity of the poor volume increase. Fig. 5 shows the

effect of activation time along with the rest of the other factors on the surface area and iodine value of the activated carbon. In general surface area of the activated carbon was increased from 15 to 25 min for all samples but with increased activation time to 25 min we noticed a decrease in the surface area of carbon, and thus affected the amount of iodine absorbed. The carbon contents in the activated carbon were increased and the porous structure was built by eliminating non-carbon atoms agreed with (Mashkooor and Nasar, 2019). Also figure 5. Showed that the initial sorption rate in iodine increased with increasing initial activation time from 676.1 to 1099.9  $mg/g$  at 15 to 25 min and thereafter gradually decreased at above 450 °C to 995.32  $mg/g$ . The optimum value for surface area 1091  $m^2/g$  and iodine uptake 1099.9  $mg/g$  at temperatures 450 °C and time 25 min. This is because increasing activation time will give more chance for hot flowing gases to do its work to some extent in increasing the pores and surface area i.e. higher time will increase the amount of volatile matter during activation process and higher removal giving more pores and higher surface area (Hameed et al., 2009).

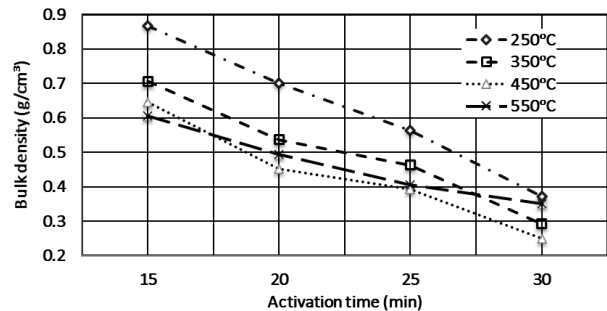


Fig. 3. Effect of parameters condition on bulk density.

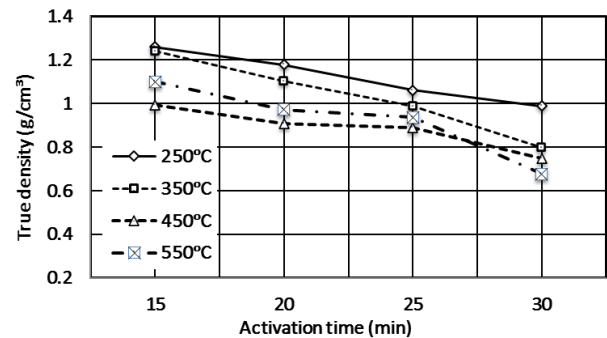


Fig. 4. Effect of parameters condition on true density.

#### 3.4. Correlation between surface area with iodine value and bulk density

Figs. 6 and 7 showed that relationship between surface area and iodine number. The surface area and iodine number of AC have a positive relationship with activation temperatures over 250 to 550 °C. The following linear regression equation described the relationship between each of surface area, iodine value and between surface areas with bulk density respectively.



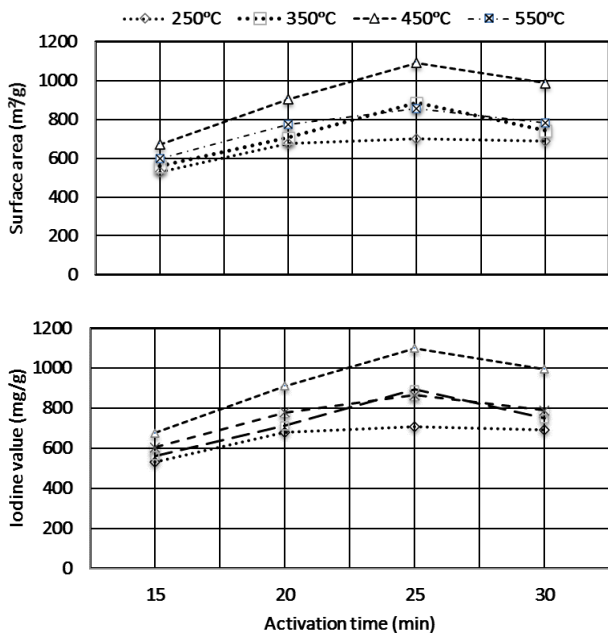


Fig. 5. Effect of activation time on surface area and iodine value.

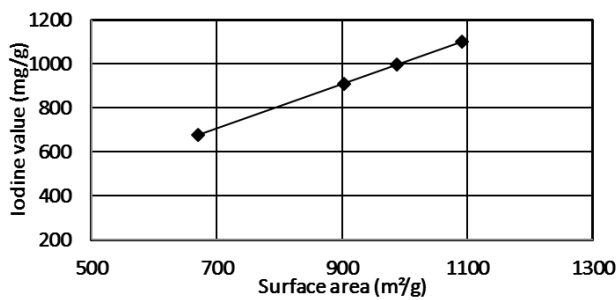


Fig. 6. Relationship between surface areas and iodine value.

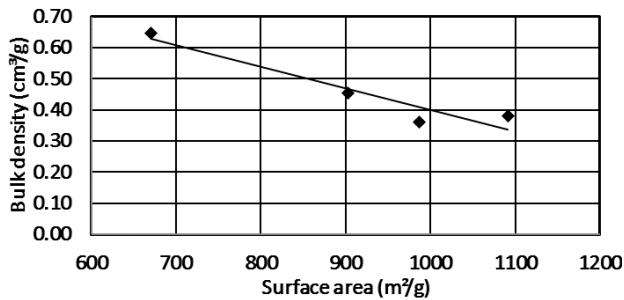


Fig. 6. Relationship between surface area and bulk density.

3.5. Scanning electrical microscopy (SEM) and electrical diagram X. (EDX) analysis

Figs. 8 and 9 show the SEM and EDX morphology revealed that the combination activation process of temperature and time were effective in creating well developed pores on the surface area of activated carbon. It was observed that sample AC had a wide variety of pores with look like structure opening. There were cavities and regular at the external surface of the AC. Results also showed that pressure played an important

role in porosity developments. It was also found that all selected AC had homogenous types of pore structure distribution. The EDX spectrum indicated that carbon content of the AC is very high (86.75%) greed with Yagmur et al., 2008.

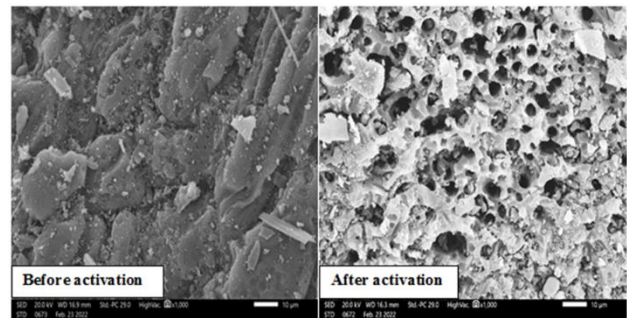


Fig. 8. SEM micrographs of activated carbon from olive seeds before and after activation respectively.

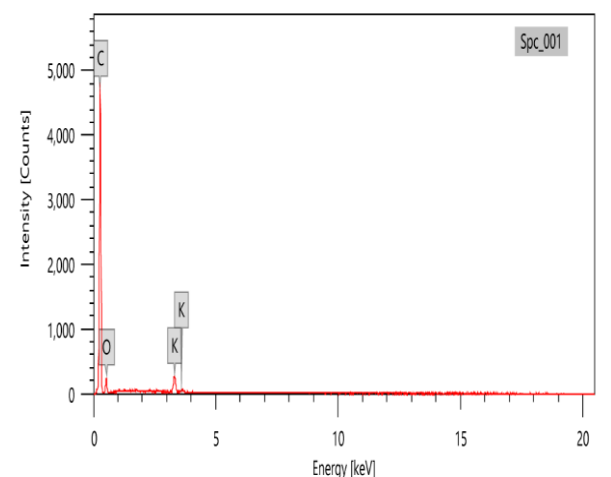
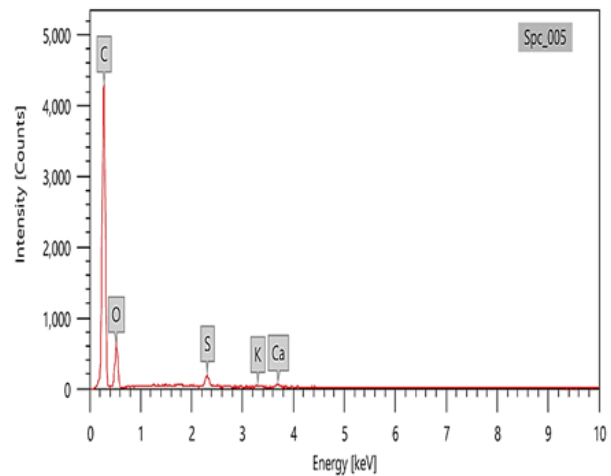


Fig. 9. EDX spectrum of carbon material before and after activation.

4. Conclusions

This study focuses on the production of activated carbon from olive seeds by physicochemical activation method. Activated carbon with high surface area can be developed by controlling the pressure gage during activation process. The optimum activation condition for

surface area development of olive seeds derived activated carbon were found to be impregnation ratio 1:1, activation temperature 450 °C, activation time 25 min and particle size 3 mm. the obtained surface area were 1091 m<sup>2</sup>/g.

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## إمكانية استخدام بذور الزيتون كمصدر لإنتاج الكربون النشط

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## الملخص العربي

يقدم هذا البحث دراسة إمكانية إنتاج الكربون النشط من المخلفات الزراعية (بذور الزيتون) للمساعدة في حل مشاكل التلوث البيئي الناتج من تراكم المخلفات الزراعية. تم كربنة بذور الزيتون عند درجة حرارة ثابتة ٥ م لمدة نصف ساعة ثم طحنها لأربع مستويات (٦,٥,٤,٣) مم ثم نقعها في محلول هيدروكسيد البوتاسيوم لمدة نصف ساعة بنسبة ١:١ ثم تنشيطها تحت تدفق غاز النيتروجين بمعدل ثابت ٥٠ سم<sup>٣</sup>/دقيقة عند أربع مستويات من حرارة التنشيط (٥٥٠, ٤٥٠, ٣٥٠, ٢٥٠) م خلال أربع مراحل زمنية (٣٠, ٢٥, ٢٠, ١٥) دقيقة، مستويات ضغط (جوى، ١,٣, ١,٦, ١,٩ بار) أظهرت النتائج أن هناك تأثير واضح على العائد من الكربون

النشط وزيادة في مساحة سطح الكربون الناتج مع زيادة درجات حرارة التنشيط وزمن التنشيط ويرجع ذلك لتطاير بعض المواد بسبب الاحتراق الغير كامل للفحم مما يؤدي إلى زيادة المسامية بفتح أكبر قدر من الثغور. كانت أفضل معاملة عند درجة حرارة ٤٥٠ م° وزمن قدره ٢٥ دقيقة حيث بلغت أعلى قيمة لمساحة السطح ١٠٩١,٥٨ م<sup>٢</sup>/جم وبالتالي كانت أعلى قيمة لامتصاص اليود ١٠٩٩,٩ مجم/جم ومن هذه الدراسة يمكن استخدام بذور الزيتون في إنتاج كربون نشط ذو عائد اقتصادي كبير يمكن استخدامه في تنقية المياه بأنواعها والمحافظة على البيئة من الملوثات ونوصى باستكمال هذه الدراسة مستقبلا.