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## Possibility of Producing Activated Carbon from Mango Trees Wastes Using Physical Activation

El Bessoumy, R. R.<sup>1\*</sup> and Enas L. A. Salem<sup>2</sup>

<sup>1</sup>Ag. Structures and Environmental Control Dept., Fac. of Ag., Eng., Al-azhar Univ., Nasr City, Cairo, Egypt.

<sup>2</sup>Agric. Eng. Res. institute (AEnRI), Agric. Res. Center (ARC), Egypt.

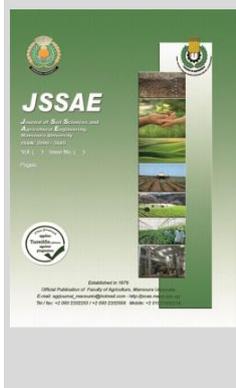


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### ABSTRACT

Biomass is widely recognized as a sustainable resource for producing activated carbon for environmental applications and energy. However, the main disadvantage remains the production of activated carbon from biomass with a large surface area that can be effectively used for ecological treatment. The present study shows the effect of different activation methods to produce activated carbon from mango trees' wood residues. Pre-treatments for mango tree wood residues were done with carbonization at 400 °C for 30 min. The produced was impregnated in KOH solution (1:1 ratio) for chemical activation. Afterward, the char created was dried at 105 °C for 60 minutes in the oven. Four different diameters of 3, 4, 5, and 6 mm of produced char were achieved. Samples of 100 g from char were activated at four different temperature levels of 300, 350, 400 and 450 °C for activation times of (15, 20, 25 and 30 min), respectively. The gage pressure of the atmosphere was recorded in the activation process at a constant nitrogen flow rate of 50cm<sup>3</sup>/min. The results showed an effect on the surface area of produced activated carbon. The best surface area for produced activated carbon was 1199.8m<sup>2</sup>/g at maximum methylene blue absorption of 528.9 mg/g, the temperature of 400 °C, activation time of 30 min and under atmospheric pressure for the specimens of 3 mm in diameter.

**Keywords:** Activated carbon, physic-chemical methods, Mango tree wood, agricultural residues.



### INTRODUCTION

The activated carbon which considers a functional carbon material has characteristics of strong adsorption capacity, a large specific surface area, and renewable, stable physical & chemical properties and so on. It has been widely used in the fields of petrochemical, medical and health, power electronics, environmental protection and even Astronautics and Aeronautics, and the demand is increasing day by day (Longmei *et al.*, 2016). One form of wastewater treatment is using activated carbon. However, the production of such activated carbon increases the cost. Which it puts an additional burden on the consumer. Therefore, methods are needed to minimize the production cost of activated carbon. At such a cost, a cheaply available starting material is to be chosen. Another way to reduce costs is through a good choice of production method. In addition, by choosing an appropriate starting material, its use can reduce solid waste pollution while reducing the raw material cost for the synthesis-activated carbon manufacturing process (Lafi, 2001). Agricultural waste materials have negative environmental impacts and have traditionally been chosen over adsorbents because they are inexpensive or no-cost materials.

In addition, these materials have excellent impurity removal properties such as their large surface area, pore size distribution and functional groups on their surface (Mohamed *et al.*, 2016).

Agricultural by-products and waste materials such as coconut shells (Olafadehan *et al.*, 2012), wood (Choy *et al.*, 2005), and rice husks (Yalcin and Sevinc, 2000) are among of low-carbon precursors for activated carbon manufacture.

Vital criteria when selecting a carbonaceous material as a precursor to activated carbon include; low inorganic content, low cost, process-ability of the material, minimal environmental impact, and durability (Choy *et al.*, 2005). A good activated carbon precursor should also have high carbon content (Hiremath *et al.*, 2012). Waste materials may be divided into two categories; conventional and unconventional wastes. Conventional waste from agricultural and timber industries like coconuts and olives are non-conventional waste refers to industrial waste and municipal activities, such as polyethylene terephthalate (PET) and polyvinyl chloride (PVC). In that sense, our precursor is grated coconut, a by-product of the coconut industry that came from activated carbon precursors. It is believed that the use of chemical activation was more prevalent in the manufacture of activated carbon for conventional waste (Dias *et al.*, 2007). Activated carbon is porous material which can be used for a variety of purposes (i.e., adsorption, elimination, purification, catalyst, etc.). Two typical processes produce activated carbon from organic materials: physical activation and chemical activation (Rashidi and Yusup, 2020). Activated carbon is used as an adsorbent in lots of commercial fields. In latest years, it's been used for controlling air pollution, water pollution, odors, etc., the research is growing on activated carbons, which use natural waste sludge for environmental protection (Demirbas *et al.*, 2008) Activated carbon is used in guard beds to adsorb contaminants in gas production. Activated carbon is a general term used to describe a family of carbonaceous adsorbents. These adsorbents have a highly crystalline form and a high volume fraction of very small

\* Corresponding author.

E-mail address: rizkjuly\_74@azhar.edu.eg

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pores, resulting in a high specific surface area, typically from 500 to 1500 m<sup>2</sup>/g. The chemical production of activated carbon with a high specific surface area of up to 3000 m<sup>2</sup>/g has been reported by (Marsh and Rodríguez, 2006). Activated carbon has a carbonaceous and porous structure with heteroatoms, especially oxygen, adding functional groups. CA from piassava (palm fiber, broom straw residues) had a pronounced porous structure with surface areas of 658 to 1190 m<sup>2</sup>/g (Avelar *et al.*, 2010). Among the components of wood, cellulose which is crystalline, while, hemicellulose and lignin are amorphous (John and Thomas, 2008). Depending on intended use of activated carbon, environmental issues involved, availability and cost, many chemical reagents have been used effectively as activating agents, the common examples include: zinc chloride (ZnCl<sub>2</sub>), phosphoric acid (H<sub>3</sub>PO<sub>4</sub>), aluminum chloride (AlCl<sub>3</sub>), sulfuric 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>). In industry, the most commonly used are phosphoric acid, zinc chloride and potassium hydroxide (Marsh and Rodríguez, 2006; Foo and Lee, 2010 and Yusufu *et al.*, 2012). The goal of the carbonization stage is to preserve the carbonaceous structure of the material, which is achieved by burning material at a temperature range from 400 to 850 °C. In addition, the properties of activated carbon depend on the physical and chemical properties of the precursor, as well as the method of activation (Demiral *et al.*, 2008). The basic objective of this work is to use some agricultural residues like mango trees wood residues to produce activated carbon by the physical activation method. Also, is to investigate the effect of activation temperature, particle size and resident time on the yield and characteristics of the activated carbon.

**MATERIALS AND METHODS**

The practical part of this research was carried out at the Faculty of Agricultural Engineering, Al- Azhar University, in 2022. The main aim of the present work is to study some factors which affect activated carbon production from agricultural residues (Mango trees wood residues, MTWR) using the chemical-physical method. For achieving activated carbon production from mango tree

wood residues, specific goals were pursued: Producing and studying some characteristics of activated carbon from mango tree wood residues. Calculate bulk density, yield, methylene blue and surface area of activated carbon. For producing activated carbons from mango tree wood residues, two stages should be carried out through the chemical-physical method. The first stage is a thermal treatment of raw material which implies dehydration and where most non-carbon elements such as volatile substances and dust are eliminated by the heating source under anaerobic conditions. The next stage is the activation in a range of different levels of temperatures and pressures.

**Raw materials**

Mango tree wood residues used in this work were collected from a mango trees farm. Wood residues from collected mango trees were thoroughly washed with deionized water to remove dirt particles adhering to their surface. The mango wood waste was dried naturally by being spread outdoors during the intense hours of sunshine of the day, after unwanted particles and dust were removed using a screen mesh. The following table (1) shows the chemical composition of mango tree wood residues before carbonization.

**Table 1. Chemical composition of mango trees wood residues, cited from (Ahmad *et al.*, 2015)**

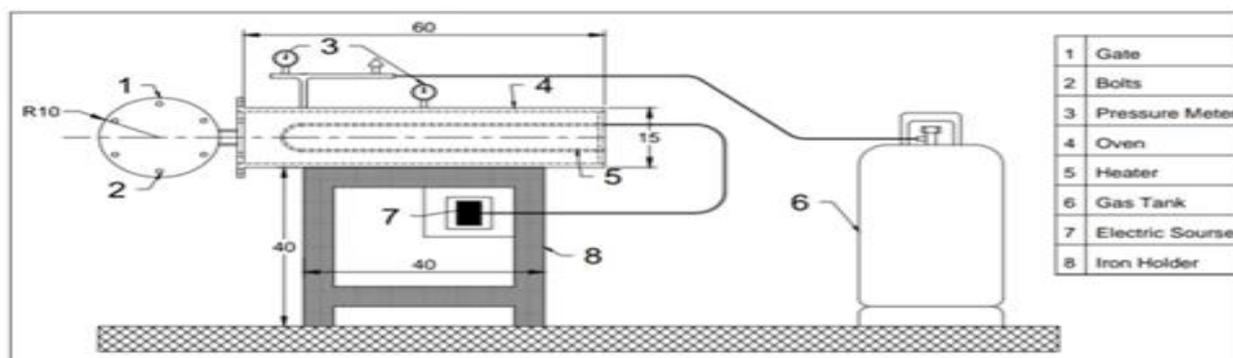
Elements (%)	N	C	O	H	S	Ash
Mango wood	1.57	68.61	9.92	0.29	31.2	2.7

**Potassium hydroxide (KOH)**

It is one amongst chemical substances and an inorganic compound with the formula KOH. Also, it is a strong base used in many industrial applications. Some of its properties are its corrosive nature and its high reactivity to acids. The solution of potassium hydroxide (99.9%) used in this study was obtained commercially from a chemistry laboratory at Faculty of Science, Al-azhar University, Assuit Branch.

**Horizontal metal Furnace**

The furnace used to produce activated carbon was constructed of high heat-resistant and mild steel sheet; its parts are shown in the following figure (1).



**Fig. 1. Schematic diagram of the horizontal furnace, constructed by (Gomaa *et al.*, 2022) and its parts: (1) Sample, (2) Furnace, (3) N<sub>2</sub> gas cylinder and (4) electric source.**

**Test procedures**

The mango tree wood residues were carbonized and activated in an electrical oven and the constructed metal furnace, respectively. The mango tree wood residues were carbonized at 400°C for 30 min. Four different activation

temperatures of (300, 350, 400 and 450 °C) were used at different activation times of (15, 20, 25 and 30 min). The produced char after the carbonization process was sieved to four different diameters. The produced activated carbons

were used to measure yield, density, surface area and methylene blue.

**Carbonization**

The carbonization process has been performed by putting about one kg of dried mango tree wood inside an electrically oven, its temperatures ranged from 250 - to 750 °C and heated up to the desired carbonization temperature of 400 °C for 30 min. The produced char after carbonization was left to cool, after that it was sieved manually to its desired different diameters of (3, 4, 5 and 6 mm). About 25 kg of the carbonized materials (char) give 192 samples of each sieve. These samples have been carbonized according to Yang *et al.*, (2020)

**Activation**

A sample of 100 g was put into the previous horizontal metal furnace and heated to different temperatures for different times. The temperature levels of 300, 350, 400 and 450 °C were used for activation times of 15, 20, 25 and 30 min, respectively. The activation process was done at a pressure Gage of (atmosphere) and a constant nitrogen flow rate 50 cm<sup>3</sup>/min. During the activation process a constant flow of 50 mL/min of nitrogen gas was assured to keep inert atmosphere. The activation temperature and heating gradient were obtained according to Yang *et al.*, (2020). Four samples were selected randomly of each parameter to determine the moisture content according to ASAE, (2011); the moisture content was determined after activation process and before the measurements.

**Experimental design**

In the present work, mango tree woods residues were collected from a mango tree farm; it was washed and dried at 110 °C for 60 min then carbonization at 400 °C for 30 min. A combination of four particles diameters (3, 4, 5 and 6 mm) heated at different activation temperatures of (300, 350, 400 and 450 °C) for four activation times of (15, 20, 25 and 30 min) at a constant pressure gage of (Atmosphere), resulting in 64 treatments. Each treatment was repeated three times to give three replicates resulting in, 192 specimens.

**Measurements**

All the following measurements were carried out at the ambient room temperature of 25°C and relative humidity of 40 %.

**Activated carbon yield**

The mass of each sample (initial weigh) was recorded before activation process and after it. The mass yield of each sample was calculated as mass of wood-based activated carbon divided by the initial sample mass of 100 g used for carbonization and activation. The yield percent was expressed using following equation:

$$Yield = \frac{W_c}{W_o} \times 100 \dots \dots \dots (1)$$

**Where :** *W<sub>c</sub>* and *W<sub>o</sub>* are final dry product mass (g) and dry raw material mass (g), respectively.

**Activated carbon bulk density**

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

$$B = \frac{W_a}{V_o} \dots \dots \dots (2)$$

**Where:** *B* is bulk density (g/cm<sup>3</sup>), *W<sub>a</sub>* mass of sample and *V<sub>o</sub>* is the volume occupied by the packed sample.

**Methylene blue number**

According to Genli *et al.*, (2021) & Kumar and Kumaran, (2005), total methylene blue adsorbed carbon microspheres at time *t*, *qt* (mg/g) could be calculated using the following equation:

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

**Where:** *qt* (mg/g) is equilibrium adsorption capacity, *C<sub>o</sub>* represents initial concentration of solution (mg/L), *C<sub>t</sub>* represents concentration of solution at time *t*, (mg/L), *V* is volume of the solution (L), and *M* is the mass of dry adsorbent (g).

**Surface area**

The surface area was determined by the adsorption of the iodine method (ASTM, 2006). An instrument was used to determine the specific surface area of activated carbon samples. The Surface area was calculated using the following equation:

$$S = \frac{X N}{M} \dots \dots \dots (4)$$

**Where:** *S*, Surface area (m<sup>2</sup>/g), *X*, iodine value (mg/g), *N*, Avogadro number and *M*, Mass of activated carbon used

**Hardness test**

The hardness property of the activated carbon reinforced polymer composites was recorded using a digital Shore "D" scale with reference to ASTM, (2010). The test was carried out to measure penetration of a specified indenter into material under specified conditions of force and time.

**SEM analysis**

SEM images have been obtained by a scanning electron microscope (JEOL; JSM6380A) powered by a ground voltage of 20.0 kV and equipped with an electron probe analyzer system, it is agreed with Samorm *et al.*, (2011). SEM images were performed at the Electron Microscope Unit, Faculty of Science, Alexandria University.

**RESULTS AND DISCUSSIONS**

**Activation time via yield**

Figure (2) shows the relationship between yield (%) and activation time (min) at different activation temperatures of (300, 350, 400 and 450 °C). It shows that the yield of activated carbon is affected by the activation time and temperatures. Figure (2) shows that the yield of activated carbon is linearly decreased by increasing activation time and activation temperature with the coefficient of determination ranging between 64.29 to 44.6 %. The obtained data indicated that the mass reduction ratio decreased from 37.1 to 24%. The rate of high weight loss is 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; it was matching with Yakoot and El-Deen, (2016).

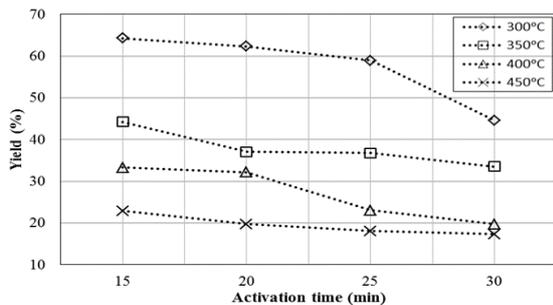


Fig. (2): Effect of activation time on activated carbon yield

**Activation time via bulk density**

Figure (3) shows the relationship between activation time (min) and bulk density (g/cm<sup>3</sup>) at different activation temperatures. It shows that the bulk density of activated carbon is affected by the activation time and temperatures. The bulk density of activated carbon decreases with increasing both activation time and temperature. The obtained data illustrated that the bulk density decreases from 0.52 to 0.45 g/cm<sup>3</sup> with increasing activation time from 15 to 30 min at activation temperatures ranging from 300 to 450 °C, respectively. This may be due to the increase in loss of volatile solids with an increase in the temperature and activation time, or the porosity development during the activation process. These results were matching with Abechi *et al.*, (2013). Also, the same previous results of reduction in bulk density with increasing activation temperature and time of activating agent were recorded by Yakoot and El-Deen, (2016).

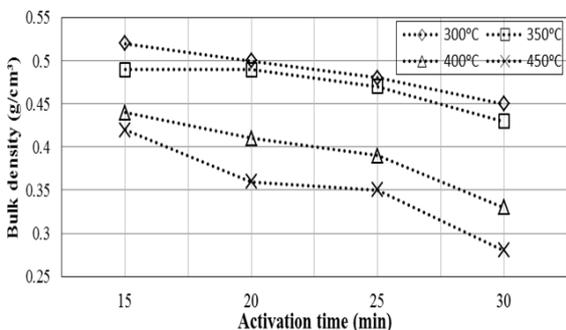


Fig. (3): Effect of activation time on activated carbon bulk density

**Activation time via surface area and methylene blue**

Figure (4) shows the relationship between activation time (min) and surface area (m<sup>2</sup>/g) at different activation temperatures. The following figure shows that the surface area of activated carbons was affected by activation time and temperatures. The AC surface area increases with increasing each activation time and temperature. The obtained data showed that the maximum surface area of (1199.8 m<sup>2</sup>/g) was forming when the carbonization temperature was recorded to be 400 °C, activation temperature of (450 °C) and activation time of 30 min, respectively. This may be because of enhancing activation reactions that caused burn-off of structure to form larger pores. Thus, the carbon content in produced activated carbons was increased and porous structures were built by removing non-carbon atoms, the same results was matching with Mashkoor and Nasar, (2019).

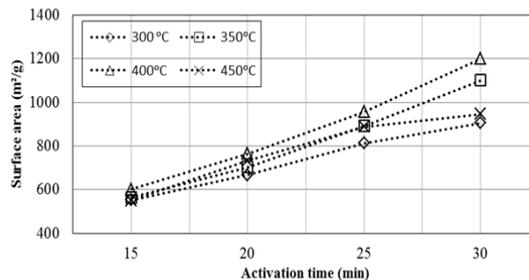


Fig. (4): Effect of activation time on activated carbon surface area

Also, figure (5) shows the relationship between activation time (min) and blue methylene (mg/g) at different activation temperatures. Also, figure (5) shows, that the blue methylene of activated carbon is affected by activation time and different temperatures. The obtained data showed that the initial absorption rate in blue methylene increases from 152 to 267.3 mg/g with an increasing initial activation time from 15 to 25 min and thereafter, it gradually decreased at above 400 °C. This may be due to that increasing activation time will give more chance for hot flowing gases to do their work to some extent in increasing pores and surface area i.e. higher time will increase the amount of volatile matter during the activation process while, higher removing gives more pores and higher surface area (Hameed and Salman, 2009).

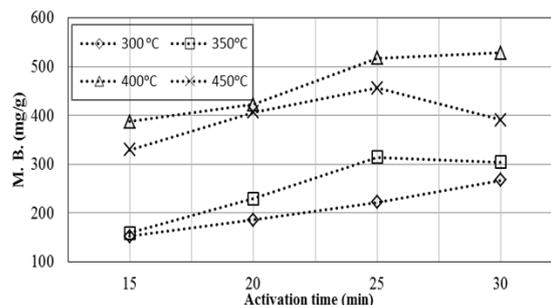


Fig. (5): Effect of activation time on methylene blue (M.B.)

**Correlation between activation temperature with surface area and yield**

Figure (6) shows the relationship between the surface area of activated carbons and yield at different activation temperatures. The activation temperature which ranged from 300 to 450 °C slightly increased porosity with a much evident effect on the miso-porosity volume. On the hand, the increased activation temperature leads to decreased activated carbon yield. Also, it appears in the figure that there is a high reduction in activated carbon yield at activation temperatures from 300 to 400 °C.

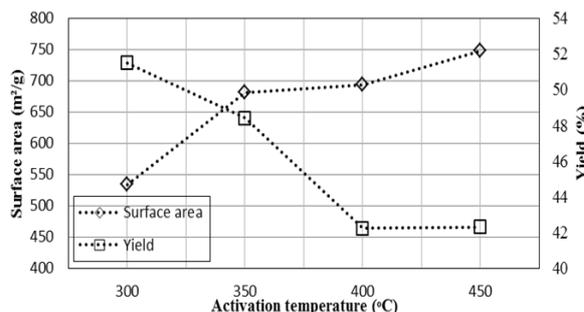


Fig. (6): Relationship between surface area and yield at different activation temperatures

### Activation time via hardness

Figure (7) shows average values of hardness for composite at room temperature. It was observed that the hardness values at room temperature increased from 80.3 (25 - 75 % sample) to 93.3 (30 -70 % sample). This result was supported by study of Brostow *et al.*, (2010).

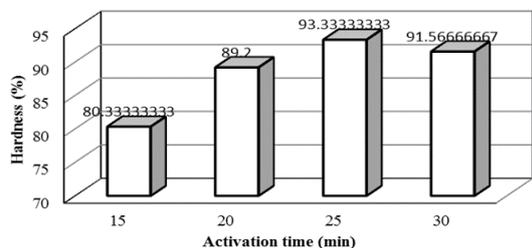


Fig. (7): Effect of activation time on hardness

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

Figure (8) shows the produced activated carbon SEM morphology which revealed that the combined activation process of temperature and time were effective in creating well developed pores on produced activated carbon surface area. From figure (8), it was observed that the production of activated carbon samples had a wide variety of pores looked like a well-opened structure.

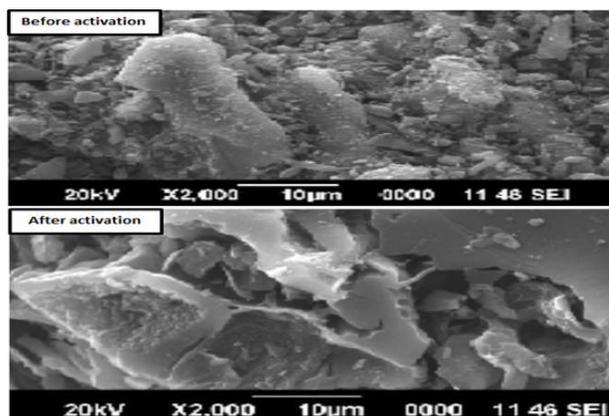


Fig. 8. SEM micrographs of activated carbon for mango tree wood residues before and after activation, respectively

### CONCLUSION

This work is suggesting an environmentally friendly process for utilizing agricultural residues for activated carbon production and removing methylene blue from aqueous solutions onto the produced carbon. The following conclusions can be drawn based on this work: The surface area of chemi-physical treated activated carbon "AC" from mango trees wood residues was high relatively, (1199.8 m<sup>2</sup>/g). The results are shown that the optimum conditions of produced AC from mango tree wood residues as 450 °C at 30 min of temperature and time from the carbonization process, respectively. The influence of various pre-treatment methods on the physico-chemical properties of carbon-based adsorbents was summarized, along with related effects on pollutant adsorption. As a result, this work represented that agricultural residues could be converted to effective and low cost adsorbent materials in an eco-friendly

manner since activated carbon derived from mango tree wood residues showed a satisfactory adsorption performance because of its, high surface area and its high capacity of adsorption.

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## إمكانية إنتاج الكربون النشط من مخلفات أشجار المانجو باستخدام التنشيط الفيزيائي

رزق ربيع كامل البسومي<sup>1</sup> و إيناس لقمان عبد اللطيف سالم<sup>2</sup>

<sup>1</sup>قسم هندسة المنشآت الزراعية والتحكم البيئي - كلية الهندسة الزراعية - جامعة الأزهر - مدينة نصر - القاهرة - مصر

<sup>2</sup>قسم بحوث القوى والطاقة - معهد بحوث الهندسة الزراعية - مركز البحوث الزراعية - مصر

### المخلص

يقدم هذا البحث دراسة إمكانية إنتاج الكربون النشط من المخلفات الزراعية (بقايا خشب اشجار المانجو) للمساعدة في حل مشاكل التلوث البيئي الناتج من تراكم المخلفات الزراعية. تم كربنة خشب المانجو عند درجة حرارة ثابتة مقدارها 400 م° لمدة نصف ساعة ثم طحنها لأربع اقطار مختلفة (6,5,4,3 مم) ثم نقعها في محلول هيدروكسيد البوتاسيوم لمدة نصف ساعة بنسبة 1:1 ثم تنشيطها تحت تدفق غاز النيتروجين بمعدل ثابت 50 سم<sup>3</sup>/د عند أربع مستويات من حرارة التنشيط (450,400,350,300 م°) خلال أربع مراحل زمنية هي (15,20,25,30 دقيقة), وضغط جوي ثابت, 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 لتطير بعض المواد بسبب الإحترق الغير كامل للفحم مما يؤدي إلى زيادة المسامية بفتح أكبر قدر من الثغور. كانت أفضل معاملة عند درجة حرارة 400 م° وزمن قدره 30 دقيقة في القطر 3 مم حيث بلغت أعلى قيمة لمساحة السطح 1199,8 م<sup>2</sup>/جم وبالتالي كانت أعلى قيمة لإمتصاص ازرق الميثيلين 528,9مجم/جم ومن هذه الدراسة يمكن استخدام بقايا خشب اشجار المانجو في إنتاج كربون النشط نوعاً اقتصادياً كبير يمكن استخدامه في تنقية المياه بأنواعها والمحافظة على البيئة من الملوثات ونوصي باستكمال هذه الدراسة مستقبلاً.