



PRODUCTION OF A NEW ACTIVATED CARBON PREPARED FROM PALM FRONDS BY THERMAL ACTIVATION

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Abstract:

A new activated carbon has been thermally produced from palm fronds, obtained from palm trees, cultivated widely in desert areas. A (21.75%) yield of Palm Fronds Activated Carbon (PFAC) was obtained from via a physical method. Characterization of PFAC was studied. Well-developed porosities verified by SEM were about 14% higher than that of commercial activated carbon. The nature of the product was identified by XRD. The activation process caused both the formation of graphite layers and the increase in bulk density, the graphite layers occurred due to breaking of chemical bonds and carbon burn-off through weak carbon –CO₂ oxidation. The development of micro porosity will lead to promising applications for the removal of metal ions, that can be achieved through the surface functional groups of PFAC, detected by FTIR. PFAC is very effective for the adsorption of methylene blue dye from aqueous solutions with great removal (99.5%).

Keywords: Adsorption; Activated Carbon; Thermal Activation; Palm Fronds.

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1. Introduction

Adsorption onto activated carbon has been found to be superior for wastewater treatment compared to other physical and chemical techniques, such as flocculation, coagulation, precipitation and ozonation as they possess inherent limitations such as high cost, formation of hazardous byproducts and intensive energy requirements [1].

However, commercially available activated carbons are still considered expensive. This is due to the use of non-renewable and relatively expensive starting material such as coal, which has difficulty in pollution control [2, 3].

Therefore, in recent years, many researchers have tried to produce activated carbons for removal of various pollutants using renewable and cheaper raw materials which were mainly industrial and agricultural byproducts, such as coconut shell [4], waste apricot [5], sugar beet bagasse [6], molasses [7], rubber wood sawdust [8], rice straw [9], bamboo [10], rattan sawdust [11], oil palm fiber [12], coconut husk [13], date stones [14], and Algerian date stones [15].

As one of the oldest known fruit crops, the date palm has been cultivated in desert in North Africa and the Middle East for at least 5000 years [16, 17]. It is also found in Spain, Australia and American continent [18].

Number of palm trees in the world is estimated to be about 100 million trees; more than 80% of them are planted in the Middle East. In Egypt, there are about 11 million palm trees representing 9% of the palm trees census in the world [18, 19].

Palm fronds are byproducts of the cultivation of palm trees. An adult date palm tree can produce approximately 13.5-20 kg of dry fronds annually [19]. These fronds are disposed however it is considered a big fortune, as 1.15 million hectare (ha) (1 ha = 10,000 m²) were planted with palm trees in 2009 [20] and assuming an average density of 100-125 trees/ha, it can be estimated that 1.9-2.4 million tons of dry fronds are available each year [21].

Many studies have been made on the parts of the palm tree like petiols [22], palm tree leaves [23], and oil palm fronds by pyrolysis and impregnation using sodium carbonate where the oil palm tree grows in West Africa and Malaysia [24].

Although Hassan Al-Swaidan et al; has studied the preparation of activated carbon from palm fronds by chemical activation using H₃PO₄ [25], we studied this preparation by physical activation as it gives more accurate pore size distribution [26] and more economical production.

In this study, palm fronds (PF) were used to prepare activated carbon (PFAC) using thermal activation method at high temperature [27]. The characterization was carried on the prepared activated carbon to study its properties. The application of PFAC as an adsorbent for the adsorption of methylene blue dye was studied to detect its efficiency and adsorption capacity. The results are promising compared to the commercial activated carbon in sight of economy benefit. We will keep researching to improve the adsorption ability of this new activated carbon.

2. Materials and Methods

2.1. Sample Collection and Preparation

Palm fronds were collected and prepared by chopping up the upper segment of a palm tree trunk; collected samples were washed with distilled water to remove dirt and clipped into smaller pieces. The samples were dried in oven over 70°C for 8 hours. They were ground using mortar and pestle into different sizes.

2.2. Thermal Activation of the Palm Fronds

A 50 g dried sample of palm fronds was put in a burning crucible, placed in a fixed-tube furnace for thermally activation 800°C. This was allowed to cool and washed with distilled water to a pH of 7, oven dried till dehydration then grounded. It was sieved with a 53µ mesh to obtain a fine powdered activated carbon and it was kept in an air tight container and used for the various experiments.

2.3. Characterization of Activated Carbon

The prepared carbon was subjected to characterization such as surface area, bulk density, moisture content, ash content, water absorption, matter soluble in water, matter soluble in acid, PH, X-ray diffraction (XRD), and Scanning electron microscope (SEM). They were tested to determine the suitability of this carbon for water and waste water treatment. The results were compared with commercially available activated carbon.

The surface area and pore characteristics which include the pore volume and pore size distribution were analyzed using Micrometrics (Model ASAP 2020, USA surface area and porosity analyzer through nitrogen adsorption isotherm at 77 K).

In order to study the structure sight of the activated carbon, Scanning Electron Microscopy (SEM) was employed to visualize morphology of an activated carbon. XRD is also used for the study of nature of activated carbon.

The analysis was conducted using a Scanning Electron Microscope (SEM) (JEOL-JSM-5700F) The FT-IR spectra (KBr) were recorded on a Shimadzu (4000-400 cm^{-1}) spectrometer.

2.4. Testing the Adsorption Capacity

To ensure the ability of the activated carbon prepared from palm fronds (PAC) to be used as a good adsorbent, it was tested for the adsorption of methylene blue.

2.4.1. Adsorbate

Methylene blue dye (99% purity) obtained from Sigma–Aldrich, Egypt, was used as adsorbate. Distilled water was used to prepare all solutions.

2.5. Adsorption Studies

Batch adsorption was performed in 20 sets of 100mL flasks. In a typical adsorption run, 50 mL of methylene blue solutions with initial concentration of 300 mg/L was placed in a flask. 0.1 g of PAC was added to the flask and kept in an isothermal shaker (120 rpm) at 25°C until equilibrium was attained. The obtained equilibrium time was 150min. It was used for other adsorption experiments.

The concentrations of methylene blue solutions before and after adsorption were determined using a double beam UV–vis spectrophotometer (UV-1601 Shimadzu, Japan) at its maximum wavelength of 668 nm. The same experiment was repeated but with different concentrations of methylene blue from 50 ppm to about 650 ppm to study its isotherm. The percentage removal at equilibrium was calculated by the following equation:

$$\% \text{ Removal} = \frac{(C_o - C_e) \times 100}{C_o} \quad (1)$$

Where C_o and C_e are the liquid- phase concentrations of methylene blue at initial state and at equilibrium (mg/L), respectively [28].

3. Results and Discussions

3.1. Characterization of the Adsorbent

Activated carbon is widely used as adsorbent due to its high adsorption capacity, high surface area, and micro porous structure. The wide usefulness of carbon is a result of specific surface area, high chemical and mechanical stability. The chemical nature and pore structure usually determine the sorption activity. The physiochemical properties of the activated carbon prepared from palm fronds (palm fronds activated carbon or PFAC) and commercial activated carbon were compared and listed in table (1).

Table 1: Charactrization of adsorbent (PFAC) compared to (CAC).

No	PARAMETERS	CAC	PFAC
1	Bulk density (g/cc)	0.5	0.47
2	Moisture content (%)	5.01	5.69
3	Ash content (%)	2.91	9.12
4	Matter soluble in water (%)	1.55	11.11
5	Matter soluble in acid (%)	4.58	29.6
6	PH	8-9	7
7	Surface area (m ² /gm.)	11x10 ²	66.49
8	Porosity (%)	52	66

CAC- Commercial activated carbon, PFAC- palm frond activated carbon.

The pH of PFAC is near to natural which will be helpful for the treatment of all classes of waste water and the carbon can also be used for drinking water purification.

Even though moisture content of the carbon has no effect on its adsorptive power, it dilutes the carbon and necessitates the use of additional weight carbon during treatment process. PFAC has low moisture content (5.69%) comparing to other carbons. Any porous material will have the tendency to absorb moisture. The moisture content of this carbon is near normal and comparable with the values reported elsewhere [28].

Ash content shows the amount of inorganic substituent present in the carbon. From Table (1), it was found that PFAC has low ash content which can increase the fixed carbon value.

Water soluble matter and acid soluble matter give the information about the amount of impurities present in carbon which affect the quality of activated carbon for water treatment. In our analysis, the data shows that PFAC contains amount of impurities and needs further purification.

Porosity is the main factor for increasing the adsorptive power of an activated carbon. Porosity is related to the bulk density and specific gravity of activated carbon. PFAC has high porosity 66% which is about 26% higher than the commercial AC.

PFAC has high surface area $66.49 \text{ m}^2 \text{ g}^{-1}$ showed that it is highly porous carbon compared to other activated carbons such as Activated Carbon Prepared from Algerian Dates Stones by H_3PO_4 Activation which has surface area $54.93 \text{ m}^2 \cdot \text{g}^{-1}$. However obtained value can be increased by followed chemical activation.

Bulk density indicates the fiber content of the precursor. From the data it is shown that PFAC has good bulk density. Generally, an adsorbent with high bulk density need not be regenerated frequently because it can hold more adsorbate per unit weight [29].

3.2. Scanning Electron Microscopy (SEM)

Figure 1(a) shows the scanning electron microscopy (SEM) image of the produced PFAC. It depicts a surface containing a well-developed pores expected of a good adsorbent, in which the carbonaceous matters and salts that could have blocked the pores as seen had been leached off by the activation process (Figure 1(b)), showing the efficacy of the thermal activation and it shows fibrous cylindrical channels in which the carbon atoms are arranged in a honeycomb lattice that is considered helpful for the accessibility of metal ions to the adsorbent surface.

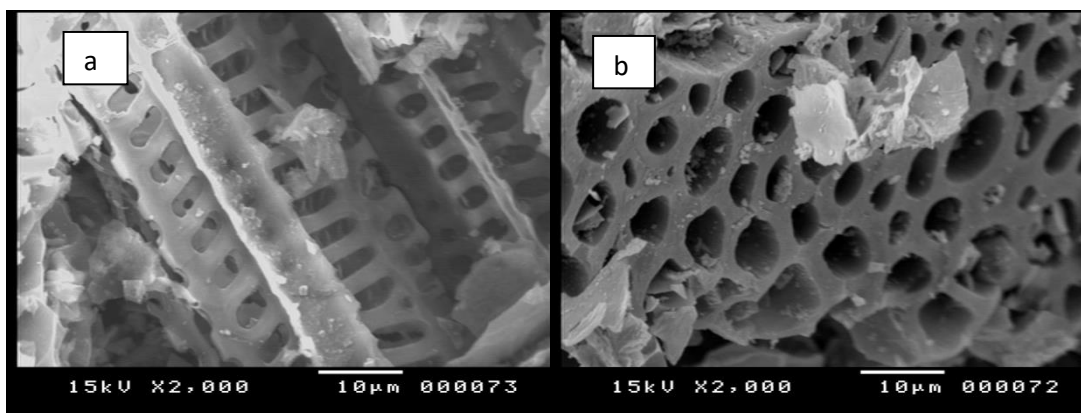


Figure 1: Scanning Electron Microscope (SEM) of PFAC (magnification: 2000x)

(a) palm fronds raw material, (b) palm fronds activated carbon.

3.3. X-Ray Diffraction Studies

Figure (2) illustrates the XRD pattern of PFAC with an activating temperature 800°C . The activated carbon exhibited sharp peak at around $2\theta = 29^\circ$ which corresponds to the peak of graphite which is the most stable form of carbon under standard conditions. Graphite has a layered structure that consists of rings of six carbon atoms arranged in widely spaced horizontal sheets. Graphite thus crystallizes in the hexagonal system.

At the same time, there are two very broad diffraction peaks around $2\theta = 24^\circ$ and 43° in spectrum. This reveals an amorphous structure of activated carbon. The appearance of the peak at around 24° signifies an increasing regularity of crystalline structure, which will result in a better layer alignment.

In this result, it can be explained that the pyrolytic reaction of organic compounds consists of the breaking of chemical bonds by temperature and condensing further into active compounds. These compounds form typical graphitic layers and stack of planes during carbonization and a higher density of amorphous unsaturated active sites available for adsorption.

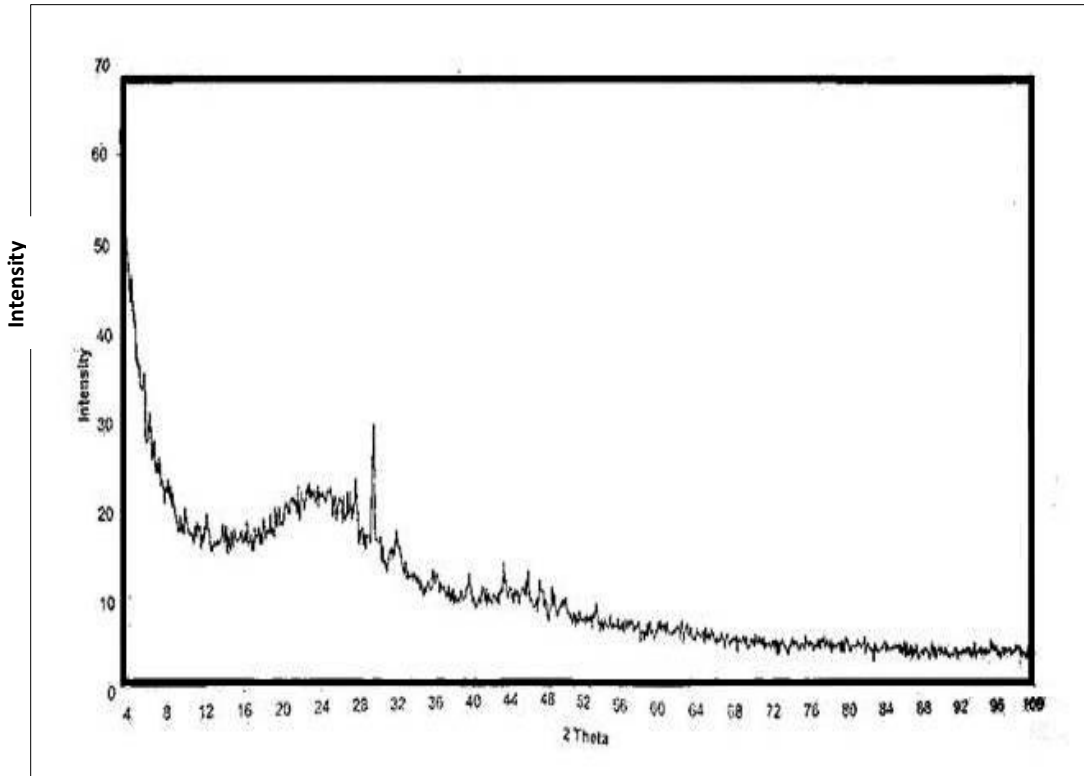


Figure 2: The X- Ray Diffraction (XRD) pattern of Palm fronds Activated Carbon.

3.4. FTIR

Fourier Transform Infrared (FT-IR) spectrum analysis was used to investigate the functional groups of the Palm Fronds Activated Carbon and its surface properties. (FT-IR) spectra shown in Figure (3) was measured in the range of (400-4000) cm^{-1} wave number. The FT-IR spectrum reveals the complex nature of the adsorbents as evidenced by the presence of a large number of peaks. The strong peak obtained at 3443.31 cm^{-1} indicates the existence of free and intermolecular bonded hydroxyl O–H stretching vibration of alcohols, phenols and carboxylic acids as in cellulose, and lignin, thus showing the presence of “free” hydroxyl groups on the adsorbent surface. Peak around 1626.95 cm^{-1} corresponds to the C=C bending that may be attributed to the lignin aromatic groups. The peak at 1433.69 cm^{-1} indicates the presence of C=O of carboxylic groups. The peak around 1109.88 cm^{-1} reveals the appearance of C–OH stretching of phenolic group present in surface of activated carbon.

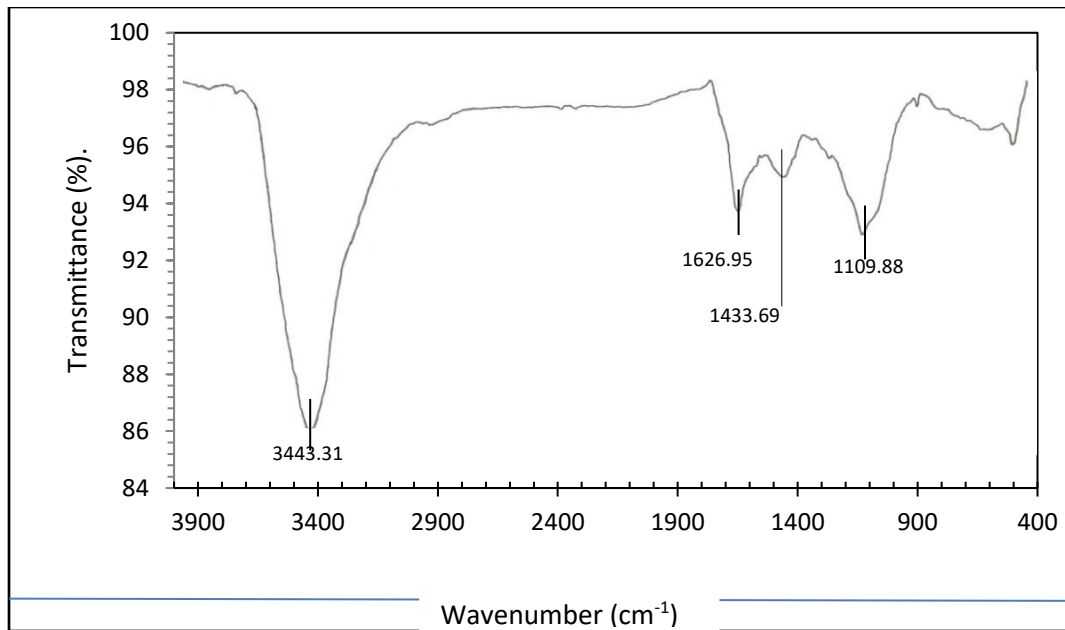


Figure 3: FTIR spectra of Palm fronds Activated Carbon.

3.5. Application of the Prepared Activated Carbon

Data presented in Fig.(4) showed the relationship between the amount adsorbed by a unit weight of solid adsorbent PFAC (q_e (mg/g)) in comparison with the commercial activated carbon (CAC) against the amount of solute (methylene blue) remaining in the solution (C_e (mg/L)) at equilibrium.

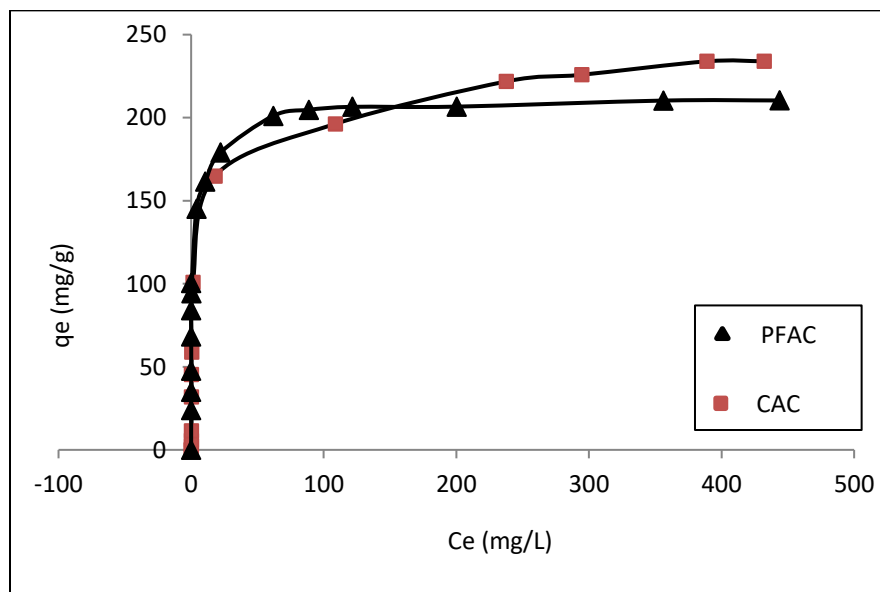


Figure 4: Adsorption isotherm studies of methylene blue onto palm-fronds activated carbon (PFAC) and commercial activated carbon (CAC) ($m=0.1g$, $V=0.05L$, $pH=7$, $T=25\pm 1$).

The experimental maximum adsorption capacity (q_e (mg of methylene blue per g of PFAC)) of palm-fronds activated carbon for removing methylene blue at equilibrium was $q_{max}= 210.375$ (mg/g) compared to 233.945(mg/g) for the commercial activated carbon CAC.

The percentage removal of methylene blue onto PFAC reached 99.95% in the concentration range (40-290(mg/g)) then it decreases gradually to about 70% at concentration 600 (mg/L) (Fig. (5)).

This large ability to adsorb methylene blue could be attributed to the formation of graphite crystals during the thermal activation. Atoms in the plane are bonded covalently, with only three of the four potential bonding sites satisfied. The fourth electron is free to migrate in the plane resulting in an increased adsorption of methylene blue due to an increase.

in the electrostatic attraction between positively charged dye and negatively charged adsorbent.

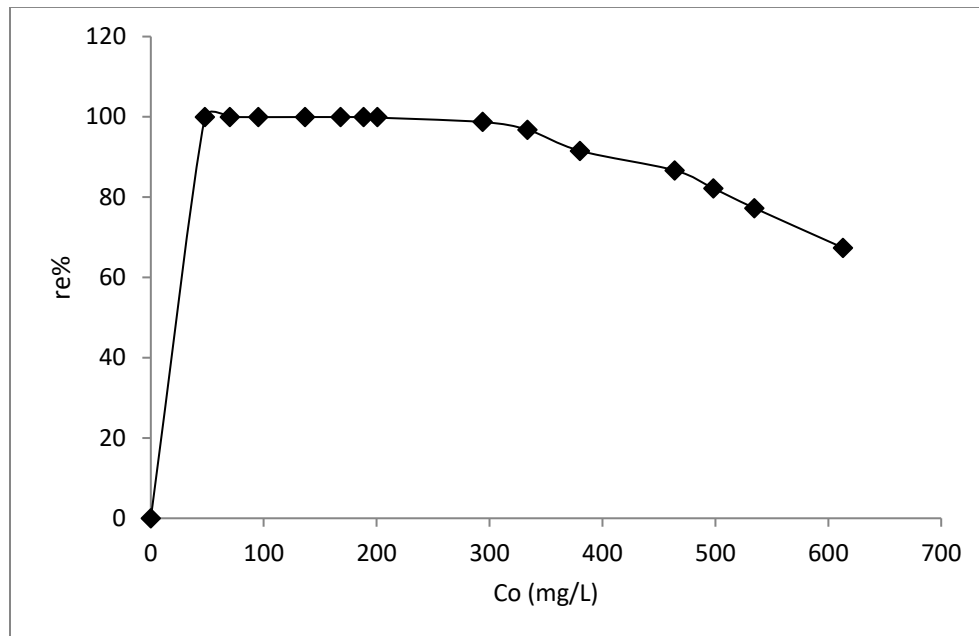


Figure 5: Effect of initial concentration of methylene blue on its percentage removal onto palm-fronds activated carbon (m=0.1g, V=0.05L).

4. Conclusions

Activated carbon could be successfully prepared from palm fronds under thermal activation at 800°C after physical treatment.

It showed a higher porosity 66% compared to 52% of commercial activated carbon and a moderate surface area (66.49 m²/g)

Activated carbon prepared from palm fronds showed promising maximum adsorption capacity (about 210.375 (mg/g)) compared to 233.95(mg/g) for the commercial activated carbon and amazing percentage removal (about 99.95% for initial methylene blue concentration range (40-290 (mg/L))).

Thus, an activated carbon prepared from palm fronds was used as an alternative for commercial activated carbon for removal of impurities from aqueous solutions. From this it has been concluded

that activated carbon prepared from palm fronds can be used as a new, low-cost, easily available, and eco-friendly adsorbent.

Abbreviations

- PF palm fronds
 PFAC. Palm fronds activated carbon
 CAC commercial activated carbon
 XRD. X ray diffraction
 FTIR courier trasmittance infrared
 SEM. Scanning electron microscope
 m. mass of adsorbent (g)
 V. Volume of adsorbate (L)

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