

## Manufacture of Activated Carbon by Chemical Maceration and Some of Its Physical Properties

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### Abstract:

A carbonaceous substance with a very porous structure is activated carbon. Its surface may be given numerous physical and chemical treatments to provide various functionality. Various precursors can produce activated carbon, including palm fibers, agricultural waste, and fossil fuels. In this article, particular focus has been placed on the fundamental characteristics of activated carbon and how physical and chemical processing affects its surface chemistry. An overview of the adsorption process, several adsorption isotherms, and adsorption kinetics are also provided. A brief explanation of the adsorption mechanism onto activated carbon is provided. Activated carbon is used, such as the adsorption of water contaminants. X-ray diffraction and electron microscopy (FE-SEM) were used to study the crystal structure of the activated carbon, where it was found that the crystal size of palm fiber and activated carbon ranges between (91.1- 35.9 nm), and (44.5- 10.9 nm) respectively. The SEM images showed surface distortion was due to impurities, and the average crystal size was 38.89 nm.

**Keywords:** Activated Carbon ( AC ), Chemical Maceration, Fibers palm, Azo Dye, Adsorption .

### تصنيع الكربون المنشط عن طريق النقع الكيميائي وبعض خصائصه الفيزيائية

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### مستخلص:

الكربون المنشط هو مادة كربونية ذات بنية مسامية للغاية يمكن معاملة سطحه في العديد من العلاجات الفيزيائية والكيميائية لتوفير وظائف مختلفة. يمكن أن تنتج السلالات المختلفة للكربون المنشط من مواد متعددة ، بما في ذلك ألياف النخيل والنفايات الزراعية والوقود الأحفوري. في هذه المقالة ، تم التركيز بشكل خاص على الخصائص الأساسية للكربون المنشط وكيف تؤثر المعالجة الفيزيائية والكيميائية على كيمياء سطحه. كما يتم توفير نظرة عامة على عملية الامتزاز ، والعديد من ايزوثيرمات الامتزاز ، وحركية الامتزاز. يتم توفير شرح موجز لآلية الامتزاز على الكربون المنشط. يستخدم الكربون المنشط ، مثل امتصاص ملوثات المياه. تم استخدام حيود الأشعة السينية XRD والمجهر الإلكتروني SEM لدراسة التركيب البلوري للكربون المنشط. وأظهرت صور ال SEM تشويه السطح كان بسبب الشوائب، وكان متوسط حجم الكريستال 38.89 نانومتر.

الكلمات المفتاحية: الكربون المنشط ، النقع الكيميائي ، ألياف النخيل ، صبغة الأزو ، الامتزاز.

## 1. Interdiction:

Different definitions of activated carbon (AC), also known as activated charcoal [1]. According to the definition, a class of porous carbonaceous materials can never be adequately represented by a structural formula or chemical analysis [2]. Additionally, AC is a solid, microcrystalline, non-graphitic form of a black carbonaceous substance with a very porous structure that is unflavored [3, 4]. Additionally, according to some researchers, AC is a porous substance made from a carbon metamaterial that has millions of microscopic holes between its carbon atoms opened as a result of the activation process [5]. The term “AC” refers to a group of carbon-black, porous materials with a large surface area that are produced via the carbonization of any elemental carbon-rich substance. Due to its excellent adsorption characteristics, activated carbon is a flexible adsorbent. According to the kind of feedstock, particle size, and preparation technique, activated carbon’s adsorption abilities vary [6, 7]. The active reagents, activation duration, impregnation ratio, carbonization temperature, and presence of inorganic impurities

are further factors that impact the adsorption quality of the resultant activated carbon [8]. There are two ways to make activated charcoal: physically and chemically. Carbonization of the precursor is the first step in physical activation, followed by activation utilizing various gases. The material is first carbonized at a high temperature between 500 and 900 °C without air. Next, the material is subjected to an oxidizing atmosphere, such as carbon dioxide, oxygen, or steam, at temperatures between 800 and 1000 degrees Celsius [9]. This is known as the activation stage, sometimes known as oxidation. The requisite porosity of the material has been effectively obtained. At the same time, the activation phase makes sure that [10]. When chemical activating agents are present, sometimes referred to as the one-step AC preparation procedure, chemical activation takes place [11]. First, chemical activation often demands a lower activation temperature and shorter material activation durations. Second, because of the action of chemicals, chemical activation can encourage the formation of holes in the carbon structure. In general, chemical activation yields more carbon generation than physical acti-

vation [12]. After heat treatment, however, it requires a thorough procedure that restores chemical agents [13]. Due to environmental concerns, this discovery could restrict its use [14]. Activated carbon is often produced using rocks that are high in carbon [15]. Coal and biomass rich in lignocellulose materials from agriculture or agricultural waste are two significant sources of activated carbon preparations. Commercial activated carbon is made from costly, non-renewable feedstock such as petroleum waste, lignite, wood, coal, and peat [16]. Contrarily, it has been claimed that low-cost activated carbon may be effectively made from agricultural by-products, including Marula nutshells, grains, maize cobs, almond shells, and bamboo [17, 18]. Adsorption is an important technique in separation and purification processes. Among the many types of adsorbents, activated carbon is the most widely used [19, 20]. The aim of the study is to obtain activated carbon with good adsorption specifications, will be used to remove the methyl blue dye .

## 2. Experimental:

**2.1. Material:** All chemicals were used through this work purchased from BDH Company. Use the fibers from

palm trees in Diyala Governorate - Iraq. Hydrochloric acid (HCl), Sodium hydroxide (NaOH), Distilled water purity is 98%, Deionized water purity is 99.99% .

**2.2. Devices used:** UV-Vis. spectra were recorded with spectrophotometer type: SHIMADZU UV spectrophotometer-1800. Centrifuge KUBOTA2010 Japan. XRD - diffraction SHIMADZU-6000. SEM ZEISS Sigma 300-Drying oven .

## 2.3. Preparation of activated carbon:

Three groups of fiber washed with distilled and ionized water were taken. The first group was calcined at 600 °C for two hours. After burning the first group, it was divided into three samples. Each sample was soaked in an alkaline medium of sodium hydroxide at a concentration of (1M, 2M, and 3M) for two hours and washed with hydrochloric acid concentration of (0.1M) to remove the base and wash with distilled water. The three samples are dried at 105 °C for 24 hours to obtain activated charcoal to obtain a neutral PH. As for the second group of the three aggregates, a calcining process was also carried out by an oven at a temperature of 600 °C for two hours. After burning, the second group was divided into three samples,

and each sample was soaked in an alkaline medium of sodium hydroxide (1M, 2M, 3M), and the samples were dried. For 24 hours after drying, the samples are burned for one hour at a temperature of 600 degrees Celsius, washed with hydrochloric acid to get rid of the base, and washed with distilled water to obtain a neutral pH. The three samples are dried at 105 degrees Celsius for 24 hours to obtain activated charcoal. The third group of the three aggregates is divided into three sections, and each is soaked in an alkaline medium of sodium hydroxide (1M, 2M, 3M). The three sections are dried at 105 ° C for 24 hours. The three samples were dried at a temperature of 105 C for 24 hours to produce activated charcoal after being calcined at a temperature of 600 °C for two hours, rinsed with hydrochloric acid to remove the base, and washed with distilled water to achieve a neutral pH.

#### **2.4. Preparation of an aqueous solution of methyl blue dye:**

A standard 600mg/L of methyl blue solution was prepared in a volume of 1 L. The solution was diluted in three beakers containing a constant concentration of 500 mg/L.

#### **2.5. Applications:**

To determine which of the three groups is activated charcoal, 0.1 gm of each group was taken and placed in a 500 ml beaker containing previously prepared methyl blue dye concentrations and placed in a requesting device at room temperature. Samples were taken at different times, the samples were placed in a centrifuge, and the absorbance was measured for each sample. Figure 1 shows the removal efficiency results by activated carbon prepared by the first method soaked at a concentration of NaOH 3M. It is the best type prepared for this study, as the results are shown in Table (1). These results showed that the removal percentage of the dye prepared with an aqueous solution increased with time, as shown in Figure (1)

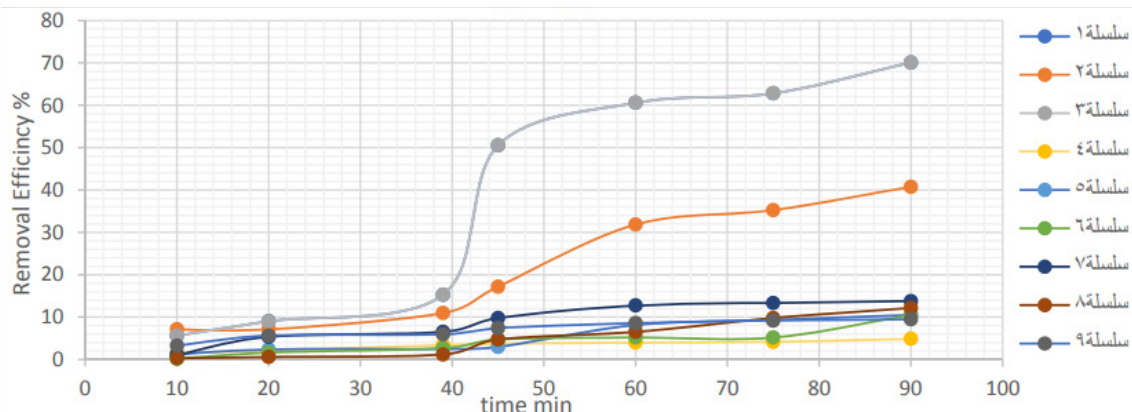


Figure (1): Shows the efficiency of absorption of Activated Carbon over time for methylene blue dye

Table ( 1 ) shows the results of adsorption of methyl blue dye on activated charcoal

Absorptivity Series 3	Absorptivity Series 2	Absorptivity Series 1	Activated charcoal soaked with NaOH base in different concentrations	Time
0.662	0.689	0.628	1M بتركيز NaOH	After 10 minutes have passed
0.681	0.674	0.580	2M بتركيز NaOH	
0.685	0.662	0.622	3M بتركيز NaOH	
0.636	0.649	0.616	1M بتركيز NaOH	After 20 minutes have passed
0.629	0.664	0.574	2M بتركيز NaOH	
0.610	0.130	0.079	3M بتركيز NaOH	
0.645	0.636	0.597	1M بتركيز NaOH	After 30 minutes have passed
0.654	0.613	0.563	2M بتركيز NaOH	
0.621	0.536	0.590	3M بتركيز NaOH	
0.644	0.659	0.411	1M بتركيز NaOH	After 45 minutes have passed
0.611	0.638	0.442	2M بتركيز NaOH	
0.619	0.433	0.577	3M بتركيز NaOH	
0.624	0.614	0.405	1M بتركيز NaOH	After 60 minutes have passed
0.639	0.603	0.318	2M بتركيز NaOH	
0.635	0.422	0.307	3M بتركيز NaOH	
0.595	0.611	0.400	1M بتركيز NaOH	After 75 minutes have passed
0.655	0.600	0.302	2M بتركيز NaOH	
0.630	0.417	0.300	3M بتركيز NaOH	
0.587	0.608	0.390	1M بتركيز NaOH	After 90 minutes have passed
0.663	0.598	0.297	2M بتركيز NaOH	
0.648	0.417	0.300	3M بتركيز NaOH	



### 3. Results and Discussion:

#### 3.1. XRD diffraction patterns:

The XRD diffraction profile of activated carbon is shown in Figure (2, A). This activated carbon has very broad diffraction peaks, and the absence of a strong peak indicates that the structure is essentially amorphous [21]. Two large diffraction peaks can be found in the spectra at  $2\theta=24^\circ$  and  $43^\circ$ , which are related to the (020) and (100) diffractions, respectively. The peak at about  $24^\circ$  at the activation temperature ( $600^\circ\text{C}$ ) indicates that the crystal

structure has become more regular, which will improve the layer alignment [22]. The crystal size of NO palm fiber and activated carbon prepared by treating palm fibers in an alkaline medium was (91.1- 35.9 nm), and (44.5-10.9 nm). The X-ray diffraction curves for raw palm fiber are displayed in Figure (2 B); the palm fiber diffraction peaks for the designs (101), (002), and (101) are relative to  $2\theta=16.38^\circ$ ,  $22.43^\circ$ , and  $34.47^\circ$ . According to reports in the literature [23], the observed peaks are indicative of crystalline cellulose.

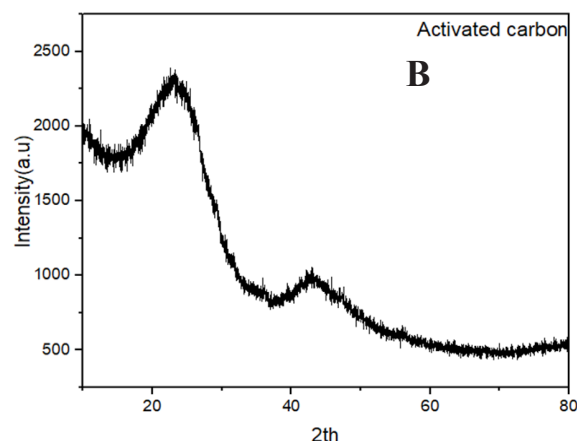
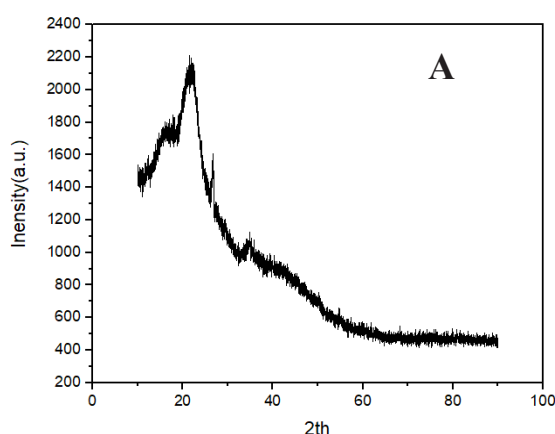


Figure (2): A: XRD for synthesized activated carbon, B: XRD for Palm fibers

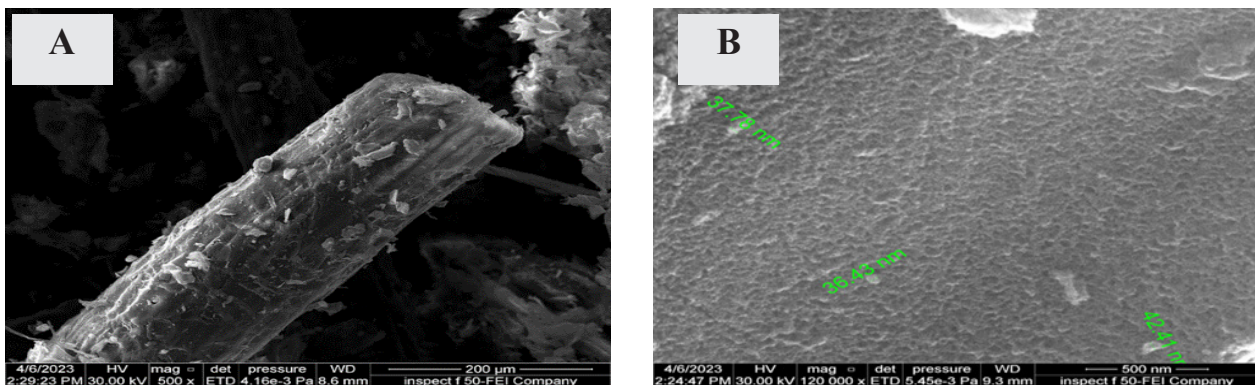
#### 3.2. Scanning electron microscope SEM:

Regarding Palm fibers, SEM can be a valuable tool for studying their microstructure and surface character-

istics. Palm fibers are obtained from various parts of the palm tree, such as the leaves or the fruit bunch, and are used for multiple applications, including in the production of ropes, mats,

brushes, and even in some construction materials. Figure 4A depicts the longitudinal surface and cross-section of palm fibers. Here, it can be seen that the exterior cell wall of the fibers includes lignocellulosic material, non-cellulose hydrophobic substances (like waxes), and surface contaminants. These substances create a smooth, protective covering on the fibers' surface. Additionally, a thin layer of parenchyma-like cells can be seen. As a result,

this surface is formed. FE-SEM images show the surface shape of activated carbon before curing is more uniform and uniform. In addition to the fine pore structure, the coated fiber showed a porous shape, compared with the row fiber, and the surface morphology is irregular and inconsistent; This may be due to surface scattering impurities, as in Figure 4B, with an average crystal size of 38.89 nm [24]



**Figure (4): Scanning electron microscope images of carbon-activated palm fibers, 4A palm fibers before to treatment, and 4B palm fibers following treatment**

#### 4. Conclusion:

The creation of activated carbon from palm fibers was the main topic of this investigation. This was accomplished by extracting palm fibers using a technique involving sodium hydroxide. After XRD techniques, FE-SEM confirmed the activated carbon synthe-

sis. According to the study's findings, natural palm fibers utilized as reinforcement in composite materials may be compared to those taken from the palm stalk. The crystalline size of palm fibers prepared by alkaline and acidic media ranges from (91.1-35.9 nm) to (44.5-10.9 nm) as shown by XRD. The

average crystalline size of the fibers after treatment was 38.89 nm, as shown by the results of FE-SEM. After that, adsorption applications were carried out on methyl blue dye, and good results were shown.

### Reference

1. Yahya, M. A., Al-Qodah, Z., & Ngah, C. Z. (2015). Agricultural bio-waste materials as potential sustainable precursors used for activated carbon production: A review. *Renewable and sustainable energy reviews*, 46, 218-235.
2. Prabu, P., & Raghu, K. (2017). Synthesis and characterization of cotton stalk activated carbon by chemical activation using H<sub>3</sub>PO<sub>4</sub>. *Synthesis*, 2(5).
3. Ravichandran, P., Sugumaran, P., Seshadri, S., & Bařta, A. H. (2018). Optimizing the route for production of activated carbon from Casuarina equisetifolia fruit waste. *Royal Society open science*, 5(7), 171578.
4. Yahya, M. A., Mansor, M. H., Zolkarnaini, W. A. A. W., Rusli, N. S., Aminuddin, A., Mohamad, K., ... & Ozair, L. N. (2018, June). A brief review on activated carbon derived from agriculture by-product. In *AIP conference proceedings* (Vol. 1972, No. 1, p. 030023). AIP Publishing LLC.
5. Hagemann, N., Spokas, K., Schmidt, H. P., Kägi, R., Böhler, M. A., & Bucheli, T. D. (2018). Activated carbon, biochar and charcoal: linkages and synergies across pyrogenic carbon's ABC s. *Water*, 10(2), 182.
6. Chen, R., Li, L., Liu, Z., Lu, M., Wang, C., Li, H., ... & Wang, S. (2017). Preparation and characterization of activated carbons from tobacco stem by chemical activation. *Journal of the Air & Waste Management Association*, 67(6), 713-724.
7. Salgado, M. D. F., Abioye, A. M., Junoh, M. M., Santos, J. A. P., & Ani, F. N. (2018, January). Preparation of activated carbon from babassu endocarp under microwave radiation by physical activation. In *IOP Conference Series: Earth and Environmental Science* (Vol. 105, p. 012116). IOP Publishing.
8. Grima-Olmedo, C., Ramírez-Gómez, Á., Gómez-Limón, D., & Clemente-Jul, C. (2016). Activated carbon from flash pyrolysis of eucalyptus residue. *Heliyon*, 2(9),



- e00155.
9. Shamsuddin, M. S., Yusoff, N. R. N., & Sulaiman, M. A. (2016). Synthesis and characterization of activated carbon produced from kenaf core fiber using H<sub>3</sub>PO<sub>4</sub> activation. *Procedia Chemistry*, 19, 558-565.
  10. Mkungunugwa, T., Manhokwe, S., Chawafambira, A., & Shumba, M. (2021). Synthesis and characterisation of activated carbon obtained from Marula (*Sclerocarya birrea*) nutshell. *Journal of Chemistry*, 2021, 1-9.
  11. Vidal, R. V., García, R. V., Martínez, A. F., & Estébanez, F. J. D. L. S. (2019). Activated carbon from almond shells for water treatment: A mini review. *Agricultural Reviews*, 40(4), 271-280.
  12. Moon, S., Ryu, J., Hwang, J., & Lee, C. G. (2023). Efficient removal of dyes from aqueous solutions using short-length bimodal mesoporous carbon adsorbents. *Chemosphere*, 313, 137448.
  13. Song, T., Liao, J. M., Xiao, J., & Shen, L. H. (2015). Effect of micropore and mesopore structure on CO<sub>2</sub> adsorption by activated carbons from biomass. *New Carbon Materials*, 30(2), 156-166.
  14. Chung, K. T. (2016). Azo dyes and human health: A review. *Journal of Environmental Science and Health, Part C*, 34(4), 233-261.
  15. Ali, A. E., Chowdhury, Z. Z., Faisal, A. N., Das, R., Wahab, Y. A., & Ramakrishnan, S. (2022). Thermochemical Conversion of Lignocellulosic Waste to Activated Carbon: A Potential Resource for Industrial Wastewater Treatment. In *Rapid Refrigeration and Water Protection: Next Generation Adsorbents* (pp. 153191-). Cham: Springer International Publishing.
  16. Yetri, Y., Hoang, A. T., Mursida, Dahlan, D., Muldarisnur, Taer, E., & Chau, M. Q. (2020). Synthesis of activated carbon monolith derived from cocoa pods for supercapacitor electrodes application. *Energy Sources, Part A: Recovery, Utilization, and Environmental Effects*, 1-15.
  17. Cheng, J., Hu, S. C., Sun, G. T., Kang, K., Zhu, M. Q., & Geng, Z. C. (2021). Comparison of activated carbons prepared by one-step and two-step chemical activation process based on cotton stalk for supercapacitors application. *Ener-*

- gy, 215, 119144.
18. Bilal, M., Ihsanullah, I., Younas, M., & Shah, M. U. H. (2021). Recent advances in applications of low-cost adsorbents for the removal of heavy metals from water: A critical review. *Separation and Purification Technology*, 278, 119510.
  19. Yağmur, H. K., Kaya, İ., & Özer, H. K. (2023). Formulation and evaluation of peel-off gel mask with St. John's Wort oil and activated carbon from pinecone.
  20. Bora, M., Tamuly, J., Benoy, S. M., Hazarika, S., Bhattacharjya, D., & Saikia, B. K. (2022). Highly scalable and environment-friendly conversion of low-grade coal to activated carbon for use as electrode material in symmetric supercapacitor. *Fuel*, 329, 125385.
  21. Abdulrazak, S., Hussaini, K., & Sani, H. M. (2017). Evaluation of removal efficiency of heavy metals by low-cost activated carbon prepared from African palm fruit. *Applied water science*, 7, 3151-3155.
  22. Magbitang, R. D. S., Zaine, S. N. A. B., Osman, N. B., & Ang, G. (2023, May). Natural Dye and Activated Carbon from Theobroma Cacao as Photosensitizer and Counter Electrode for Titania-Based Dye-Sensitized Solar Cell. In *Proceedings of the 1st International Conference of New Energy: ICNE 2022, 1-2 Dec, Sarawak, Malaysia* (pp. 7387-). Singapore: Springer Nature Singapore.
  23. Georgin, J., da Boit Martinello, K., Franco, D. S., Netto, M. S., Piccilli, D. G., Foletto, E. L., ... & Dotto, G. L. (2021). Efficient removal of naproxen from aqueous solution by highly porous activated carbon produced from Grapetree (*Plinia cauliflora*) fruit peels. *Journal of Environmental Chemical Engineering*, 9(6), 106820.
  24. Kumar, D., Kumar, A., Prakash, R., & Singh, A. K. (2019, August). X-ray diffraction analysis of Cu<sup>2+</sup> doped Zn<sub>1-x</sub>Cu<sub>x</sub>Fe<sub>2</sub>O<sub>4</sub> spinel nanoparticles using Williamson-Hall plot method. In *AIP Conference Proceedings* (Vol. 2142, No. 1). AIP Publishing.