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Synthesis of Granular Activated Carbon from *Ficus Carica* Wood by Chemical Treatment

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ABSTRACT

The study focused on the preparation of activated carbon and the examination of its properties. Activated carbon was produced by soaking *Ficus carica* L. wood in a sodium hydroxide (NaOH) solution. The mixture of wood and NaOH was then filtered, dried, and thermally carbonized until all vapors and gases were expelled. Soaking the wood in varying concentrations of NaOH for 48 hours resulted in an increased retention of the base within the wood. The findings indicated that Sample 5 (with a base-to-wood ratio of 2:1) exhibited the highest adsorption capacity for Methylene Blue dye, achieving a rate of 298 mg/g. In contrast, Sample 4 (with a base-to-wood ratio of 1.5:1) demonstrated superior iodine adsorption, reaching 853 mg/g. Additionally, other measured parameters such as density, humidity, and ash content were found to be within acceptable limits.

Keywords: Granular activated carbon, *Ficus Carica* wood, Chemical treatment

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INTRODUCTION

Activated carbon is the common term used for a group of absorbing substances of crystalline form, having large internal pore structures that make the carbon more absorbent. Activated carbon is manufactured according to the Ostreijkos patents of 1900 and 1902. Every year, approximately one hundred fifty thousand metric tons of granules and thirty thousand metric tons of pellets/rods [1].

Activated Carbon is prepared from most carbonaceous substances used as raw materials to make activated carbon such as coal (lignite, sub-bituminous and bituminous), coconut shell and wood (hard wood and soft wood), most activated carbons are made from raw materials such as nutshells, wood, coal and petroleum. Other types of raw materials used, but to a lesser extent, include fruit pits, nut shells and rice hulls. The most common types used for water treatment are coal and coconut shells. The most common method to produce activated carbon is steam activation. The first stage of this method is carbonization, in which the majority of the volatile components present in the raw materials are removed, leaving a carbon char. The char is then "activated" in a second stage at temperatures of 600- 900 C° with steam in a low-oxygen environment. Activated carbon is the most popular and the cheapest material used in purification. Much of activated carbon is regenerated (cleaning/desorption) and is used hundreds or even thousands of times [2,3].

We often speak of the absorption surface of carbon, which can vary from 400-1600 m/g, as a measure of the effectiveness of carbon. This is incorrect. The effectiveness of carbon depends on its ability to absorb a certain substance or substances, depending on the chemical and physical properties that carbon possesses. Activated carbon can be manufactured for different purposes [4].

Chemical activation is principally used for the activation of wood-based activated carbon and activated carbon made from stones. This differs from steam activation in that carbonization and activation occur simultaneously. The raw material, usually wood chips, is mixed with an activating and dehydrating substance, usually phosphoric acid or zinc chloride. The activation takes place at a low temperature,500ºC is the normal condition, but sometimes it can go up to 800ºC [5].

Phosphoric acid causes the wood to swell and open its cellulose structure. During the activation, the phosphoric acid acts as a stabilizer and ensures that the carbon does not collapse again. The result is a very porous activated carbon full of phosphoric acid. This is later washed out and re-used in the next production. As a result of the manufacturing process, no "chips" (crystalline plates) are to be found in this carbon. Instead, the carbon acquires a very open pore structure, which is ideal for the absorption of large molecules. As a rule, this carbon is ground down to powdered carbon [6].

Activated carbon is a solid, porous, carbonaceous material created through the carbonization and activation of organic substances. This process can occur at high temperatures, with or without the addition of inorganic salts, and typically involves exposure to activating gases such as steam or carbon dioxide. Alternatively, carbonaceous matter may be treated with a chemical activating agent such as phosphoric acid or zinc chloride and the mixture carbonized at an elevated temperature, followed by removal of the chemical activating agent by water washing [7].

Characterization of activated carbons is driven by the need to have qualitative and quantitative information which serve as the basis for comparison and selection of activated carbon for specific applications. Such data are also useful for modeling the behavior and performance of activated carbons. Furthermore, characterization provides feedback for use in the design and preparation of activated carbon. The Characteristics often measured are density, abrasion resistance, surface area, average pore size, pore size distribution, pore shape, pore volume, and the surface chemistry of the carbon. Whilst there are well-established standard methods for measuring the density and abrasion resistance, scientists and industry are still grappling with what accurate methods to use for measuring the othersecondary data on the activated carbon. As a result, there are still concerns with the accuracy of values determined for these parameters. The most popular method for Characterizing activated carbon is through the measurement of adsorption data and application of mathematical models that relate the adsorption data to such Characteristics as pore volume of the adsorbent and the properties of the adsorptive.(1) the two principal mechanisms by which activated carbon removes contaminants from water are adsorption and catalytic reduction. Organic are removed by adsorption and residual disinfectants are removed by catalytic reduction [8,9].

Powdered activated carbon is an attractive approach for improving refinery-activated sludge effluent. Its use is a viable alternative to granular activated carbon tertiary treatment for meeting proposed 1983 Best Available Technology Economically Available (BATEA) effluent quality standards as required by the Environmental Protection Agency (EPA). The proposed process involves adding powdered activated carbon to the aeration tank of the activated sludge process, achieving cost effectiveness by operating at a very high sludge age and a low carbon dose. Effective removal of oil and colloidal solids in the pretreatment step is necessary for successful operation [10].

Studied the adsorption characteristics of cadmium and zinc onto a granular activated carbon. Cadmium and zinc removals increased on raising pH and temperature and decreased on raising the molar metal/carbon ratio. The adsorption processes were modelled using the surface complex formation (SCF) Triple Layer Model (TLM) with an overall bidentate species. A dependence of the SCF constant on pH, the molar metal/carbon ratio and temperature was observed, and a correlation for log K_{ads} was determined. The SCF model successfully predicted cadmium and zinc removals [11,12].

Granular activated carbon (GAC) is commonly used for removing organic constituents and residual disinfectants in water supplies. This not only improves taste and minimizes health hazards; it protects other water treatment units such as reverse osmosis membranes and ion exchange resins from possible damage due to oxidation or organic fouling. Activated carbon is a favored water treatment technique because of its multifunctional nature and the fact that it adds nothing detrimental to the treated water [13].

Typical surface area for activated carbon is approximately 1,000 square meters per gram (m^2/g). However, different raw materials produce different types of activated carbon varying in hardness, density, pore and particle sizes, surface areas, extractable, ash and pH. These differences in properties make certain carbons preferable over others in different applications [14].

A powdered carbon‘s ability to remove impurities from a liquid is evaluated using laboratory-scale batch treatment tests or "Isotherms". Data from these laboratory adsorption tests may be used to calculate the carbon‘s capacity for impurity removal, or its "adsorptive capacity". These laboratory scale tests are also useful to identify the best-performing carbon type and to define the relative economics of powdered carbon treatment [15].

The ash content of activated carbon is a measure of the mineral content (Ca, Mg, Si, Fe, salts, etc.) left in the carbon after the manufacturing process. Therefore, activated carbon used for purification of water, alcohol, and other foodstuffs is cleaned with acid, often followed by water, to get rid of most of these substances [16].

Carbon has been used as an adsorbent for centuries. Early uses of carbon were reported for water filtration and for sugar solution purification. Activated carbons ability to remove a large variety of compounds from contaminated waters has led to its increased use in the last thirty years. Recent changes in water discharge standards regarding toxic pollutants have placed additional emphasis on this technology [17].

The American Water Works Association (AWWA) sets standards on the amount of moisture content that GAC contains (no more than 8% by weight). The percent of ash content should also be considered, as a high percentage can cause problems in hard water. Particle size should also be considered, as it impacts the pressure drop across the filter, the backwash rate, the carbon usage rate (CUR), and the overall filtration capabilities. Powdered activated carbon (PAC) is also an option but is mainly used when carbon filtration is necessary on a seasonal basis [18].

Experimental

Preparing Samples of wood for Carbonization Purposes

A fig wood was taken from the perennial parts and left in the air till dryness then, it was cut into homogeneous pieces, then the pieces put in a 110-130 °C furnace until got equilibrium weight, thereafter, the pieces were made small to get the carbonize able size, by using the base NaOH.

Primary Carbonization by Soaking Method

The primary material was taken (the wood), by various ratios (zero, 0.5, 1, 1.5, 2, 2.5) from the base (NaOH), (the Carbonizing) and activating material for 48 hours, then all the samples were filtered in a normal way without using any filtering papers. All the models were dried and their weights were calculated (dried at 110-130C^{o}), and all dried samples were kept in for final carbonizing.

Final Thermal Carbonization and the Final Activation of the Various Samples

The resultant material from the primary carbonization has been taken and warmed up to a temperature of about (550 ± 50 °C) by a direct flame for 3-hour period of time. Then the samples were cooled off to room temperature[19].

The Carbon Activation and Purification

The prepared carbon samples were washed with distilled water to remove any unreacted sodium hydroxide. This washing process continued until the equivalence of the resulting water indicated complete removal of the sodium hydroxide, as confirmed using filter paper. Subsequently, all samples were dried in a furnace at a temperature of 110-130 °C for 3 hours to achieve a constant weight.

After completing the washing and drying processes, the carbon sample was placed in a round-bottom flask. A solution of 10% hydrochloric acid was then added, and the mixture was boiled for two hours. Following this, the carbon was filtered and washed with distilled water until the washing water reached equivalence.

After this process, the sample was dried at a temperature of 110-130 °C until a constant weight was achieved. The samples were then stored in a sealed container for future measurements.

Determination of the Internal and External Pores in Activated Carbon

Measurement of Internal area for Activated Carbon using Iodine adsorption from Aqueous Solution

The measurement of the adsorbed iodine amount by the activated carbon samples is regarded as an easy and quick method to obtain information about the inner surface dimension, and the determination of the amount of the adsorbed iodine by milligrams by one gram of the activated carbon, and procedure implies the following [20]:

1. Transfer one activated carbon gram in a conical flask with 250ml capacity, an amount of 10ml of 10% HCl acid.
2. Boil the mixture for half an hour, then the solution left to cool off to the laboratory temperature.
3. 100 ml of iodine solution (0.1 N), transferred to a flask, then sealed by a light stopper the flask was put in the electrical agitating device for 30 minutes, then all its contents were filtered material and remainder was collected in dry flask.
4. 50 ml of the filtered transferred, and was put in a conical flask of 250ml capacity, then diluted with a standard solution of the sodium thiosulfate dihydrate (0.1N), till it became of pale yellow color.

Finally, we added one milliliter of starch pilot the color of the solution changed the blue color and according to the size of the sodium thiosulfate dihydrate (0.1N), till it acquired the yellow-pale color, then one ml of starch pilot was added (the solution color will shift to blue), and the process of admixing is continued until the blue color disappear according to the consumed sodium thiosulfate dihydrate through the sizes differences.

5. The following formula has been applied for the account of adsorbed iodine weight by the activated carbon:-

$X = A - [2.2B \times \text{ml volume of sodium thiosulfate dihydrate}]$

$A = N1 \times 12693$…..

$B = N2 \times 126.93$….

Whereas:-

X = the adsorbed iodine weight in ml, by the activated carbon

N1 = Iodine solution concentration

N2 = sodium thiosolfate dihydrate concentration which (N1 = N2) equivalent to (0.1N)

As for the Iodine number, it is calculated by the following equation:-

$$\text{I.N} = \frac{X}{M}\, D \ \ldots..$$

M = the activated carbon sample weight used (1gm)

D = correction factor

Measurement of External Surface Area For Activated Carbon Using Adsorption of Methylene Blue From Aqueous Solutions

The study of methylene blue adsorption from its aqueous solution gives an idea about the molecules that have a high molecular weight, this method expresses the outer surface area of the activated carbon. We can define this value as the number of the milligrams of methylene blue which removed from its aqueous solution, by its adsorption on the outer surface of the activated carbon by one gram of the activated carbon, whereas (0.1) gram of the activated carbon was taken after adding a certain amount of methylene blue (20 ppm) and put in a dry conical flask, then the flask attached to the electrical shaker device for (24) hours at the laboratory temperature until the color is disappeared, at this case, an additional amount of the pigment solution is added, and the shaker continues until the color is fixed, then a certain amount of the solution is taken and put in a centrifugal apparatus for (3-5) minutes to get rid of the carbon particles, after that the purified solution is situated in an absorption cell, then the absorption is measured, and the value of the absorption is determined concerning the pigment solution at (665 nm) wave length (the wave length where the pigment is being absorbed).

The calculation of the removed pigment concentration, from the aqueous solution, is done by the standard curve, which is prepared for this purpose, by taking various standard concentrations from pigment solution (5, 10, 15, 20, 25 ppm), and by measuring the absorption of these solutions at the wavelength (665 nm) and by drawing a diagrammatic line between the absorption lines and the concentrations [21].

Measurement of Some Activated Carbon Physical Properties

Determination of Humidity Content

One gram of the wet activated carbon samples was weighed and put in an oven at 150 Celsius for three hours. The percentage of the humidity was measured according to the difference in the weight [22].

$$\%\text{Humidity} = \frac{\text{weight of the wet activated carbon} - \text{weight of the dry activated carbon}}{\text{weight of the dry activated carbon}} \times 100$$

Percentage of Ash

One gram of the activated carbon was taken and put in a crucible and the crucible was put in an electric oven at 1000 °C for three hours, Then it was left to be cool for weight of different materials including the ash for each sample of the greenish activated carbon samples and finally to measure the percentage of ash in each sample [23].

$$\%\text{Ash} = \frac{\text{weight after born}}{\text{weight befor born}} \times 100$$

Determination of apparent Density [24]

The density is measured by putting any amount of activated carbon (after being crashed and sieved in specific sieves size 80 mash) in volumetric bottle is weighed by using sensitive scales and the density is measured as follows:-

$$\text{Density} = \frac{\text{mass}}{\text{volume}} \ \text{g/cm}^3$$

RESULTS AND DISCUSSION

It was observed during the preparation of activated carbon from *Ficus carica* wood using the soaking method that a soaking period of 48 hours with sodium hydroxide (NaOH) was optimal. After soaking, the wood was transformed into sawdust and subsequently dried at a temperature of 110-130 °C. The wood ratio to NaOH base was evaluated, and the ratios ranged from (0.0-2.5 g) base to (1 g) wood. The soaking process was conducted with gentle agitation for 48 hours. During this time, a noticeable change in the color of the mixture occurred, transitioning from colorless to pale yellow, then to red, and finally to dark brown. This color change indicates the breakdown of cellulose, hemicellulose, and lignin molecules that make up the wood, as well as the formation of various sodium salts that contribute to the observed coloration. After the completion of the soaking period, the five samples that had been prepared were filtrated, and the filtrated materials were left to be dried at room temperature. It appears from the table that the increase in the base weight NaOH leads to reactions with wood which cause significant increases in the wood weight can be seen in Table (1). *The weight of each sample before soaking is 10 g.

Table 1 Change in weight activated carbon sample after soaking method

Samples	Ration NaOH:Wood	Weight after soaking (g)	Increase in the weight (%)
1-	Without base	-	-
2-	0.5 : 1	10.7	7
3-	1.0 : 1	11.2	12
4-	1.5 : 1	11.8	18
5-	2.0 : 1	12.1	21
6-	2.5 : 1	12.6	26

Table 2 value of the measurement for activated carbon obtained soaking with different concentration NaOH and heat carbonization

Ash (%)	M.B (mg/g)	I.N (mg/g)	Density (g/cm^3)	Humidity (%)	Samples
2.70	22	162	0.097	0.8	1*
2.82	72	543	0.118	1.1	2
2.82	163	740	0.125	1.0	3
2.92	292	853	0.103	2.3	4
2.50	298	814	0.082	2.7	5
2.58	188	389	0.115	2.5	6

Weight of each sample before soaking is 10 g.
*Sample Without base (NaOH).

Table 3 Adsorption of Methylene Blue dye

Adsorption of Methylene Blue dye (mg/g)	Sample
22	1
72	2
163	3
292	4
298	5
188	6

Table 4 Iodine adsorption from aqueous solution

Iodine Adsorption from Aqueous Solution (mg/g)	Sample
162	1
543	2
740	3
853	4
814	5
389	6

Table 5 Density of the samples

Density (g/cm^3)	Sample
0.097	1
0.118	2
0.125	3
0.103	4
0.082	5
0.115	6

Dried samples were taken and carbonized at temperature of 550C°±25C° on direct flame in a container isolated from the air until the completion of the liberation of all fumes and gases. Note that the samples swelled during the process of carbonization and activation by the base. Samples were transferred to a filter paper and washed in distilled water carefully until the equivalent of the washing water (equivalent of Wash Solution with sunflower paper was detected), after that all samples were activated by using 30% HCL acid and thermal escalation under reflux condenser for half an hour to remove the remnants of NaOH base and some of the products of the corrosion process, if any.

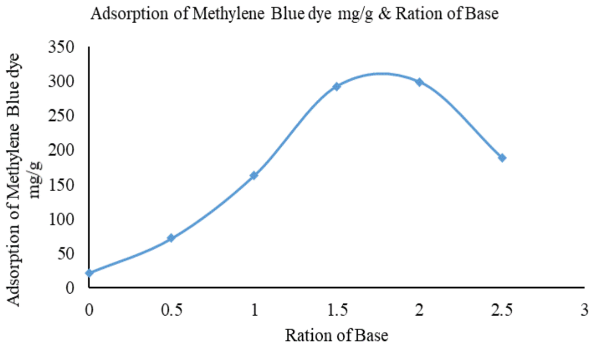

Fig. 1 Ratio of Base & M.B

Then the samples were infiltrated and transformed into an Erlenmeyer flask in fixed weights to measure the iodine number (IN) and adsorption of Methylene Blue dye (MB) in order to identify the surface area of the State (through the measurement of adsorption Methylene Blue) and the interior space (by measuring the adsorption I.N) and thus calculating the total area of the samples.

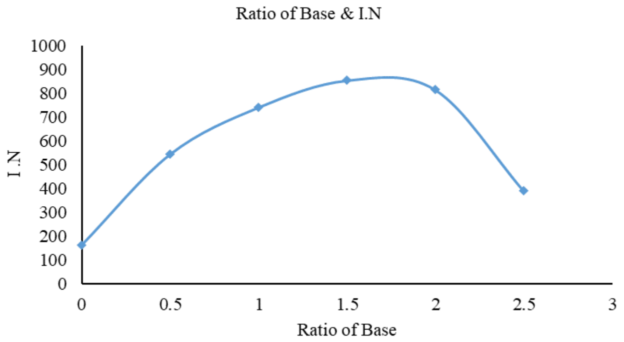

Fig. 2 Ratio of Base & I.N

It was clear from measuring the six samples that carbonizing the samples without NaOH base gave a little iodine number (IN = 162) and also a little adsorption (MB = 22.30) (the adsorption of the blue methylene dye from its water solution). When using different ratios of NaOH base (These percentages ranged from zero to 2.5 of NaOH to 1 of wood) showed an increase in the absorption of MB nearly doubled when increasing NaOH base to 2 (wood 1:2 NaOH), And began to recede (decrease) to the extent of (wood 1:2.5 base) which indicates poisoning stomata are also external and internal.

The change can be observed in the values of (MB) and (IN) with a base ratios increase in Table 3, chart 1 and Table 4, figure 2, respectively. The measurement of physical properties of the samples shows that the hydrophilic groups in activated carbon increase by increasing the ratio of the base, which has been done in a standard method.

While ratios of the density, Humidity and Ash content were economically and academically accepted as in tables (5, 6, 7) and Charts (3, 4, 5) respectively

Fig. 3 Density & Ratio of Base

Table 6 Humidity content of each samples

Humidity Content (%)	Sample
0.8	1
1.1	2
1.0	3
2.3	4
2.7	5
2.5	6

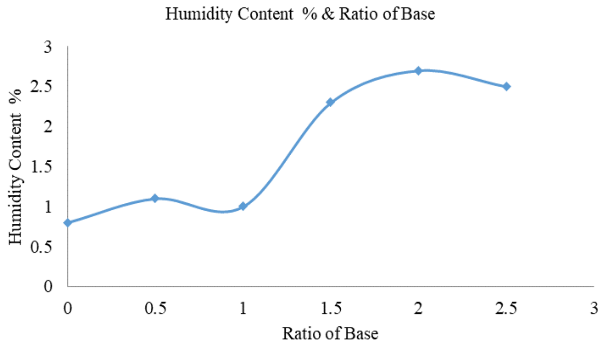

Fig. 4 Humidity & Ratio of Base

Table 7 Ash content of each samples

Ash content (%)	Sample
2.70	1
2.82	2
2.82	3
2.92	4
2.50	5
2.58	6

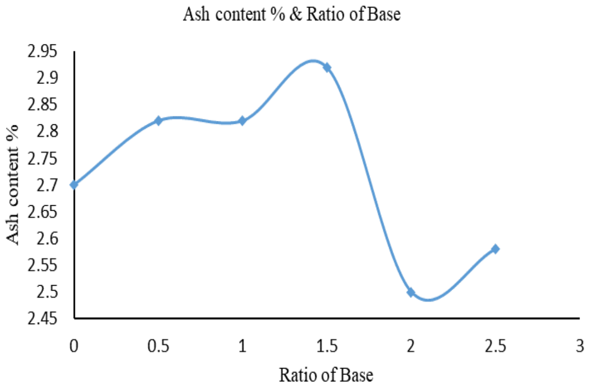

Fig. 5 Ash & Ratio of Base

CONCLUSION

Model 4, which contains a ratio of 1.5 bases: 1 wood, showed the best test for absorption of methyl blue dye as well as absorption of iodine.When observing Model 6 showed a clear decrease in the absorption capacity of methylene blue dye as well as iodine, which indicates the breakage and destruction of the pores formed inside the activated carbon when the base ratio exceeds 2.5:1 wood.

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