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Synthesis and characterization of activated carbon made from coconut Shell

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Abstract:*Coconutshellisused to produceactivatedcarbon, by the chemicalmethodwithphosphoricacid and 48 hourssoaking time. Carbonizationwascarried out at twodifferenttemperatures of 500 to 700 ° C. Lateractivatedwith standards ASTM D 1348-94 and 1755-95 D to determine the moisture and ashsamplewascharacterized. The textural properties of activatedcarbonwith the method of Brunauer-Emmett-Teller (BET), using N_2 adsorption weredetermined, the best resultwas for the activatedcarbontreated to 700 °C, whichgives as a resultone'sisotherm of type II according to the IUPAC adsorption is a macroporoussolid. X ray diffraction shows peaksindicating the presence of crystalline phases type graphite and fullerene. The scanning electronmicroscopyshowed the presence of micropores and mesoporeswithhoneycomb type morphology.*

Keywords*: activated carbon, adsorption, coconut shell, carbonization*

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I. Introduction

In Mexico, the coconut-producing States are Campeche, Colima, Guerrero, Jalisco, Michoacan, Oaxaca, Tabasco, Nayarit, Sinaloa, Quintana Roo, Veracruz and Yucatan. The coconut groves in the country area is 162, 224 hectares, of which only 12,400 hectares are destined to the coconut production, which is where detaches the fiber, as well as the husk or shell of the coconut [1-2].

The most important products derived from coconut are fiber, coconut oil, dust, bone, pulp, coconut water and coconut fruit or pulp. In particular, the producers of coconut in the state of Tabasco just focus on water, copra, coconut oil extraction since it is of great importance in the commercial sector. on the other hand, because that is not given an industrial use to shell or coconut bone, is thrown to the environment by causing a strong impact of air pollution due to burning it, as it is considered him trash [2].

According to studies about the coconut shell, it is a biomass with high carbon content material, so it is used as raw material for production of activated carbon, due to its high hardness, volatility and low ash content, as well as good performance in surface area specific [3-5].

Activated carbon is used in different processes, but their cost is very high so it is needed to investigate low-cost sorbents made from biomass waste or synthetic materials. The porous structure of activated carbon is the main physical characteristic to determine its adsorptive performance, so it is important to the study of activated carbon from waste for use as adsorbent. Economic and environmental problems of the coconut-producing region are associated with the generation of pollutants [6-9] coconut shell residue. The objective of this study is the synthesis and characterization of activated carbon using as raw material Coconut shell.

II. Methodology

Activation of the samples by the chemical method

Coconut shell that we used in this study they obtained from the municipality of Paraíso, Tabasco, Mexico. Prepared 4 samples, two were pyrolysisto 500°C and two to 700°C for an hour in a ramp heating of 10 °C x min. For the activation of the samples, they used as an agent of activation the H_3PO_4. The samples were sealed completely to leave free acid and thus kept for 48 hours, then washed with distilled water to a pH between 6 and 6.5 washing once coal proceeds to dry with the help of the muffle for 8 hours at a temperature of 100 °C, is subsequently placed in the desiccator until use.

Determination of moisture and ash

Standard ASTM D 1348-94 [10] quantifies the percentage of moisture; ash determination we performed with standard ASTM D 1755-95 [11].

Characterization of the adsorbent

After obtaining carbon from coconut shell, were analyzed samples using the following techniques: X-ray diffraction, physical adsorption of N_2 and scanning electron microscopy.

X-ray diffraction (XRD)

Composition and crystalline phases formed during the process of activation of activated carbon samples they evaluated with the technique of XRD, with the team "Diffraction solutions D8 advance".The characterization was performed directly on the surface of specimens of activated carbon, using the following conditions: flush angle 2θ at a voltage of 35 kV tube, using a current of 20 mA, with a number of steps from 5° to 95° degrees, 0.020°, 2 s. time step size For the qualitative analysis of the XRD patterns were applied the XRD Wizard (measuring programming) software and XRD Commander (execution of measurement) identification of phases was carried out with the database of the EVA05 team.

Physical adsorption of N_2

Nitrogen adsorption measurements they carried out using a static volumetric procedure in a Micrometrics Tristar II. The gases used are N_2 and have (Air Liquide, 99.999% quality). Measurements they made at the normal boiling temperature of the N_2 (77 K at atmospheric pressure).

Using this technique can be determined some textural characteristics of the solid as the specific surface area (BET, in m^2/g) the total specific pore volume or distribution diameter and equivalent pore diameter [12].
The most solid surfaces are not completely flat, especially in the case of porous solids and more specifically the case of zeolites. IUPAC recognizes three types of pores according to their size [13]: Macropores (dp> 50 nm), Mesopores (2 <dp<50 nm) and micropores (dp< 2nm), as it is the case of zeolites.

Scanning electron microscopy

The analysis of morphology of the burned coals to temperatures of 500-700°C they examined with the scanning electron microscope (SEM) "Analytical Scanning Electron Microscope, JEOL JSM-6010LA mark".

III. Result

Determination of moisture (%)

For the determination of the percent of humidity, crucibles for the samples we weighed to 500 and 700°C. Subsequently weighed the amount of activated carbon. After weights, the samples in a heating furnace at a temperature of 110 °C we placed for a period of 4 hours, with the following results (Table 1):

Table1: Results of moisture at 500°C

Crucibles weight (g)	Coal samples (g)	Crucible weight + carbon (g)	Percent moisture (%)
1: 33.626	4.542	36.130	5.27 %
2: 30.260	4.453	32.770	5.59 %

Samples of coal at 700 °C were used crucibles small following the same procedure as above (Table 2).

Table 2: Results of moisture at 700°C

Crucible	Weight crucibles (g)	Carbon (g)	Crucible weight + carbon (g)	Moisture
1	24.935	5.236	30.171	9.13%
2	21.444	5.122	26.566	9.93%

Determination of ash (%)

Approximately 2 g of sample with known moisture they weighed in a crucible previously calcined at 800 °C and weighed. The sample they calcined at a temperature according to the product, i.e. 500 and 700 °C. After calcination, crucible is cooled and weighed. The analysis is performed in duplicate (Table 3).

Table 3: Results of the determination of percentage ash of coconut Shell at 500°C

	Mass of the empty crucible (g) m^2	Mass pot + ash (g) ml	% of ash
1	35.136	35.463	16.35
2	26.426	27.151	16.25
		Average	16.3

The above procedure reproduced only that a temperature of 700 °C for this case had the following results (Table 4):

Table 4: Results of the determination of % ash of coconut shell at 700°C

	Mass of the empty crucible (g) m^2	Mass pot + ash (g) ml	% of ash
1	27.136	26.463	14.35
2	23.426	22.151	15.25
		Average	14.3

The characterization of chemically-activated carbons are fundamental to know the morphological and structural characteristics that prevail in the adsorbent materials for our research were characterizations by XRD, area specific BET and scanning electron microscopy. Obtained the following results:

Through the analysis of XRD made of activated carbon samples shown having peaks of graphite and coal very well defined, which means that you its samples of activated carbon which have types of porous structures characteristic of these materials, which are listed below (Fig. 1):

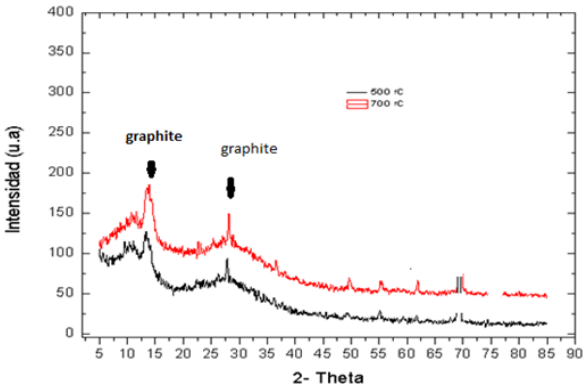

Fig1: Difractogram XRDactivated carbon at 500ºC and 700 ºC

The first type of structure is formed by microcrystalline which are similar to graphite consisting of parallel layers of carbon atoms arranged hexagonally which are located at 28º in 2-theta in two planes.

The second type of structure is described as a three-dimensional lattice of hexagons of carbon disordered because of the system randomly from condensed benzene structures that are formed during the carbonization process.

In these patterns also seen, some other peaks marked both in activated carbon at 500 ° C as in the 700 °C, which correspond to H_3PO_3, H_3PO_4 and Na_3PO_4.

The presence of these peaks in the analysis is due to the activation of precursor material with phosphoric acid, which I react in pyrolysis process and the active coal washing with water and sodium hydroxide resides on the plane between 10-15 degrees in 2-theta.

We can also realize that two activated carbons have practically the same crystalline account that the components that were found in this characterization are the expected since in a charcoal and according to the conditions (Table 6) in which was performed this single adsorbent material can be expected to find components present (Fig. 3)phases which tells us that higher temperatures increased the intensity of these will be.

Determination of the surface area (BET)

According to the results obtained by the technique of adsorption-desorption of N_2 (BET specific area) catalysts, which determined the area, the diameter of the pore and pore volume, is observed differences in the behavior of the isotherms of adsorption and distribution of specific Area (S_{BET}), diameter of pore (D_P) and total volume of pore (V_T) for every activated carbon.Results are observed in the Table 5 and Fig. 2 shows the isotherms.

Table 5: Textural parameters obtained from N_2 adsorption to 77 K samples activated physically.

Sample	**S_{BET} (m^2/g)**	**D_P (Å)**	**V_T (cm^3/g)**	**V_{Micro} (cm^3/g)**
CA-500 ºC	417.34	38.23	0.19859	0.1687
CA-700 ºC	701.19	45.58	0.47548	0.1025

In the table above can realize that the highest temperature is the one with the best features of both surface area and pore volume since I present a surface area of 701.19 m^2/g being an acceptable area to the end that will be to the activated carbon.

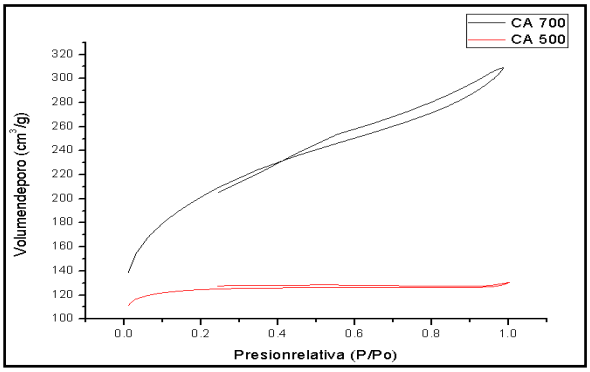

Fig. 2: Adsorption isotherm to 500 °C and 700°C

Activated carbons are isotherms with widening of elbow and a rise of the adsorption curve at pressures close to saturation in the case of activated at 700 ° C otherwise the carbon at 500 ° C since this was kept in the curve of adsorption and not full desorption. This increase in the quantity adsorbed near P/P_0=1 suggests a capillary condensation of N_2 in the region corresponding to the mesoporos, indicating the presence of certain mesoporosityin these samples.

Scanning electron microscopy (for the carbon to 700 °C)

We can give us account that the components that were found in this characterization are the expected since in a charcoal and according to the conditions (Table 6) in which was performed this single adsorbent material can be expected to find components present (Fig. 3).

Carbon is the element that should have greater composition of the material within the ranks of the 75-85% of all the material, otherwise could be an activated carbon. As is the case of our material.

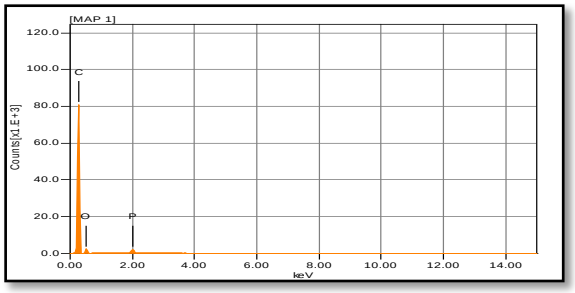

Fig 3: EDS activated at 700°C

In the Table 6 we can see the mass compositions and mol of our activated carbon prepared at 700 °C with this we can also say that percentages are consistent with what was expected and that it has the lowest concentration the phosphorus which is due to the dehydrating agent used and which is responsible for the widening of the fibers of the raw material (coconut shell).

Table 6: Chemical composition of activated carbon

Formula chemical	% mass	% mol
C	89.78	92.42
O	9.37	7.24
P	0.85	0.34
Total	100.00	100.00

Mapping is one of the ways through which we can realize how each component of our study, in the case of our material is distributed us to count that all the elements are well distributed making this material will be suitable for the purposes set out and that can be a good adsorbent material according to the morphological features found in this characterization (Fig. 4).

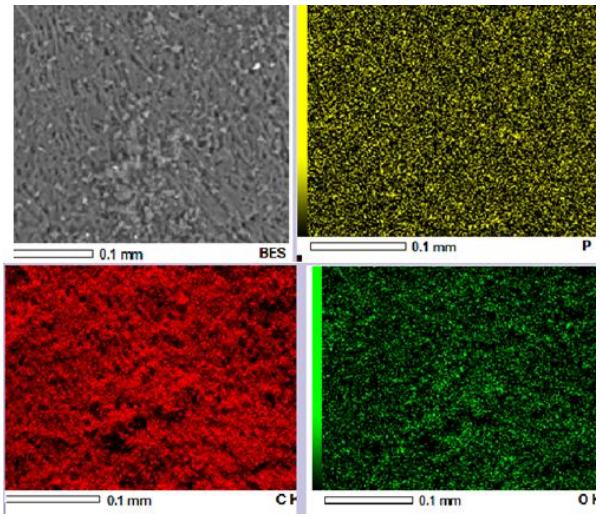

Fig. 4: Mapping at 700°C for the activated carbon from coconut shell

IV. Conclusion

According to the results obtained in the characterizations in the laboratory equipment came to the conclusion that the adsorbent material produced is a product that is in the range (500-1500 m^2/g) needed to adsorb metals and fats in wastewater, which could be one of the uses that can be given to this material, and that in addition to being a matter of scrap predecessor makes it a suitable process.

The adsorbent material presented better results was that made at 700°C by chemical method since in the characterizations of area surface BET was obtained 701.1974 m^2/g, also in the SEM morphology test show qualities and pore diameters larger on the material from 500 °C by using the same method of preparation.

In the study of X-ray results were expected since the present this characterization in crystalline phases correspond to the elements with which they work, i.e., only obtained peaks corresponding to elements like carbon, phosphorus, sodium, among others themselves which are the substances used as it is the case of phosphoric acid the forerunner (coconut shell), and the base for neutralization.

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