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PREPARATION AND CHARACTERIZATION OF ACTIVATED CARBON FROM JATROPHA WASTE CAKE

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ABSTRACT

The present work investigated the use of Jatropha waste cakes, a by-product of the manufacture process of biofuels, as a potential feedstock for the preparation of activated carbon. Chemical activation of this precursor, using phosphoric acid as dehydrating agent, was adopted. The effect of the main process parameters, acid concentration, impregnation ratio, and temperature of pyrolysis step, on the performances of the obtained activated carbons was studied. The Jatropha waste cake was carbonized at 300 ^oC and activated with H_3PO_4 at 400 ^oC. The microstructural and structural properties of the adsorbent were studied using Nitrogen gas adsorption analysis, SEM and FTIR. The surface areas and pore size of the jatropha activated carbon were 868 m^2g^{-1} and 2.128 nm respectively. The FTIR results showed slight changes in the absorption band of the precursor material compared to that of the activated jatropha carbon. This showed that the activation process was successful.

Key words: Activation, adsorbent, furnace, jatropha, surface.

INTRODUCTION

In aquatic environments, pharmaceutials are often detected [1]. The conventional wastewater treatment technologies have not always effectively removed pharmaceutical compounds from wastewater before their discharge into streams and surface water [2]. Therefore, more effective and specific treatment options are required to adequately reduce the amount of these pollutants in effluents, and hence minimize their effects on the environment. Activated carbon possesses a large surface to volume ratio and this makes it a viable option to effectively treat waste water. Activated carbons are the most commonly used adsorbents. Different pollutants such as pesticides, phenolic compounds, dyestuffs, and metals can be removed from liquid streams using these solids [3, 4].

Jatropha waste cake, which is an agricultural waste generated by the biofuel industry, can be considered as a potential low-cost adsorbent for the removal of pharmaceutical compounds from hospital effluents. Research has been carried out using *Jatropha* seed hull for the adsorption of a dye (malachite green) and heavy-metal ions, such as zinc and cadmium, and it has shown a remarkable adsorption capacity. Due to its high lignin content, *Jatropha* hull has the potential to be a good precursor for the production of activated carbon [5].

Thus activated carbon prepared from *Jatropha* waste cake is being proposed as a probable adsorbent for removing pharmaceutical compounds from wastewater before their eventual discharge into water bodies.

MATERIALS AND METHODS

Sample Collection and Preparation

Jatropha curcas waste cake was obtained from the National Research Institute for Chemical Technology (NARICT), Zaria. The *Jatropha* waste cake collected was sundried for three days to remove moisture according to the method described by Itodo *et al* [6]. The sun-dried samples were properly washed with water to remove dust and water-soluble impurities. The samples were allowed to drain off excess water accumulated during washing. Thereafter, the samples were introduced into a thermostatic laboratory oven set at 105 ºC for 24 hours. The dried samples were gently crushed using a laboratory mortar and pestle followed by sieving with a < 400µm aperture sieve. The sieved samples were stored in airtight containers for further use.

Carbonization of *Jatropha* waste cake

The prepared *Jatropha* waste cake was carbonized at 300 ºC in a muffle furnace using the method described by Gimba *et al* [7]. About 50 g of the *Jatropha* waste cake was placed in large sized ceramic crucibles and carbonized in the furnace. The carbonized samples were allowed to cool in desiccators prior to activation.

Chemical activation of the carbon

Chemical activation of the carbonized sample was done using H_3PO_4 as dehydrating agent as described by Baccar *et al* [8]. Fifty grams of the crushed and dried precursor (carbonized *Jatropha* waste cake) was mixed with H_3PO_4. The impregnation ratio defined by the weight of the impregnant (H_3PO_4) to the precursor was 1:1. Stirring was used to ensure the access of the acid to the interior of the precursor. The pyrolysis of the impregnated material was conducted in a regulated furnace at temperature of 400 ºC while the adsorption time was maintained at 2 hours. After cooling down to room temperature, the prepared adsorbent was thoroughly washed with hot distilled water until a neutral pH was obtained. The prepared adsorbent was then dried at 105 ºC overnight, ground and stored in hermetic bottle for subsequent analyses.

Characterization of Prepared Adsorbent

Specific Surface Area

Specific surface area of the prepared activated carbons was evaluated through N2 adsorption at 77 K, using an Autosorb1-Quantachrome instrument (NOVA 4200e USA). The BET (Brunauer–Emmet and Teller) model was applied to fit Nitrogen adsorption isotherm and to evaluate the surface area (S_{BET}) of the sorbent [9]

Morphology analysis

The morphology of the raw material (jatropha waste cake) and the activated carbon prepared in the optimal conditions were analysed by SEM analysis. This was done using Quanta Scanning Electron Microscope (FEI Quanta 250 USA).

Infra-Red Spectroscopy Analysis

The surface functional groups and structure were studied by FTIR spectroscopy. The FTIR spectra of the raw material andthe resulting activated carbon were recorded between 400 and 4000 cm−1 in an FTIR spectrometer (Agilent Technologies Cary 630 USA).

RESULTS AND DISCUSSION

The BET Surface Area and Pore Characterization of the prepared Activated Carbon

The BET surface area, pore volume and pore diameter of the jatropha carbon activated with H_3PO_4 are presented in Table 1. The BET surface area (S_{BET}]) was found to be 868 m^2g^{-1}, pore volume was observed to be 0.4866 cm^3g^{-1} and the pore diameter was found to be 2.128 nm. The results were linearly regressed using the BET equation.

Scanning Electron Microscope (SEM)

The micrographs of the transverse section of the jatropha activated carbons are presented in Plates 1 – 2.

Fourier Transform Infrared Analysis

The FTIR spectra of the jatropha cake and activated jatropha carbons were shown in Figures 1and 2 respectively. The major bands observed for the jatropha cake were between 3000 – 3300 cm^{-1}, 2800 – 2900 cm^{-1}, 1400 – 1700 cm^{-1} and 1000 -1300 cm^{-1} while that observed for the prepared activated carbon were at 3463 cm^{-1}, 2700-2100 cm^{-1}, 1625 cm^{-1}, 1208 cm^{-1}, 1080 cm^{-1} and less than 1000 cm^{-1}. The band frequencies and their assignments for the jatropha cake and prepared activated carbons over the range of 1000 – 3500 cm^{-1} are presented in Tables 2 and 3.

Table 1: Textural characteristics of the Jatropha Activated Carbon (JAC)

Parameters	Value
$S_{BET}{}^{a}$(m^{2}g^{-1})	868
Pore volume (cm^{3}g^{-1})	0.4866
Average pore diameter (nm)	2.128
R^2	0.9960

[a] *Specific surface area (multipoint BET method).*

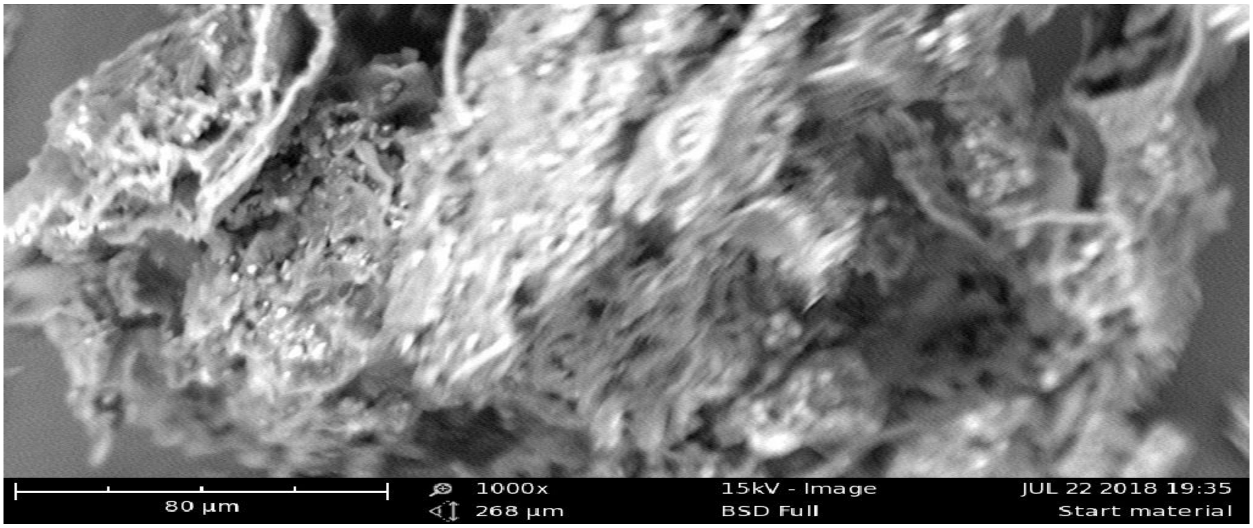

Plate 1: SEM Micrograph of jatropha waste cake

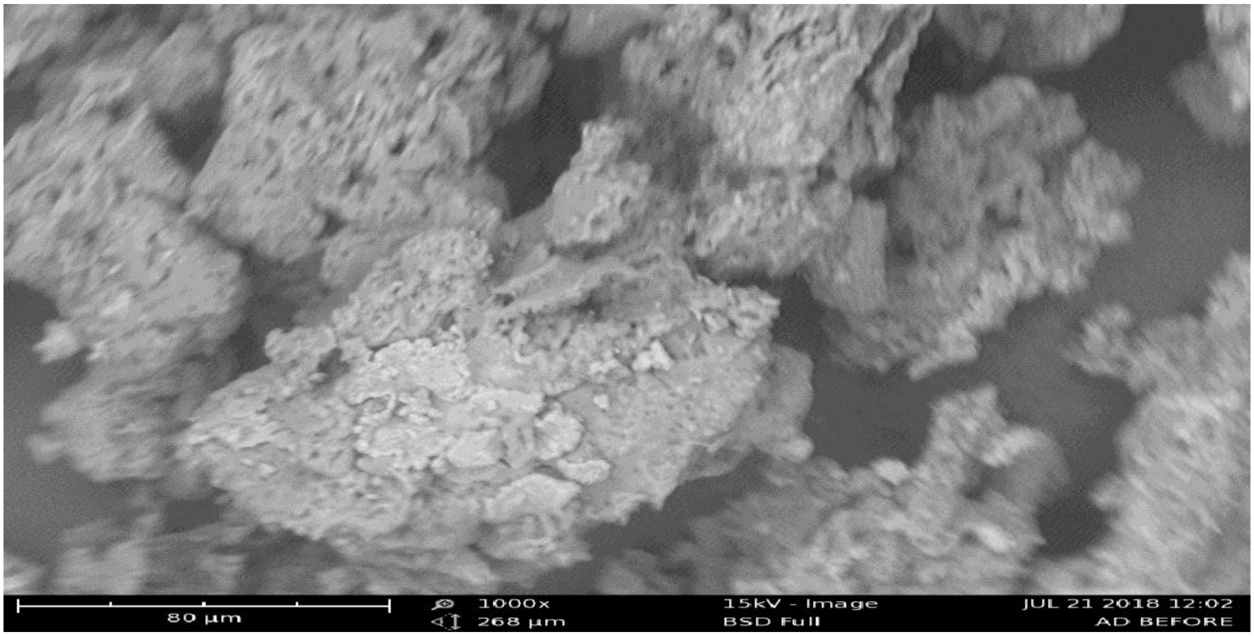

Plate2: SEM micrograph of prepared jatropha activated carbon

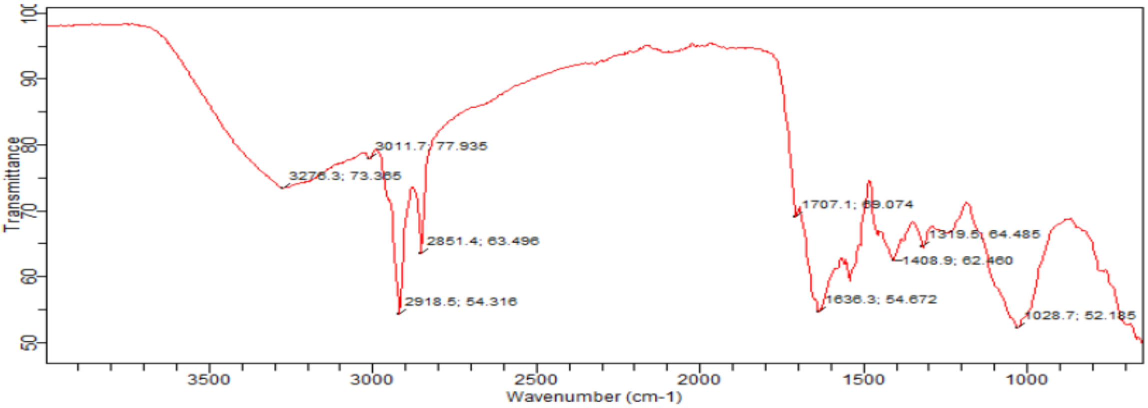

Figure 1: FTIR Spectrum of *Jatropha* Waste Cake.

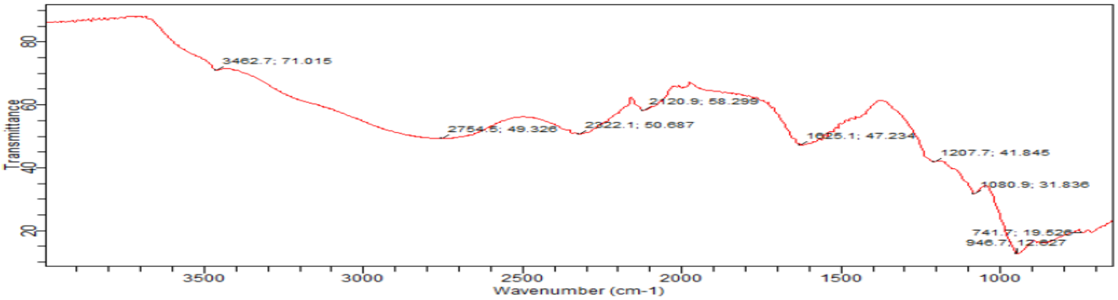

Figure 2: FTIR Spectrum of *Jatropha* Activated Carbon

Table 2: FTIR Table of Assignment for the *Jatropha* Cake (Precursor)

Wavenumber (cm^{-1})	Origin	Assignment
3276.3	O-H Stretch	Hydroxyl group
2918.5-2851.4	C=O	Carbonates
1707.1	C=O Stretch	Carboxylic acids and esters
1636.3	C=O	Amide
1408.9-1319.5	C=O	Carboxylate group
1028.7	Si-O-Si	Organic siloxane

Table 3: FTIR Table of Assignment for the *Jatropha* Activated Carbon

Frequency ([cm^{-1})	Origin	Assignment
3462.7	O-H Stretch	Hydroxyl group
2754.5-2322.1	C=O	Carbonates
2120.9	C≡C	Terminal alkyne
1625.1	C=O	Conjugated ketone
1207.7	P-O-C	Phosphates
1080.9	Si-O-C	Organic siloxane or silicone
946.7	PO_4^{3-}	Phosphates
741.7	C-H	Aromatic C-H

The textural characteristics of the prepared jatrophaactivated carbon (JAC) showed that the adsorbent has a large specific surface area (868 m^2g^{-1}) and the pore diameter was 2.128 nm. The adsorbent prepared from *jatropha* waste was mainly mesoporous and it is favourable for the adsorption of large molecules.

Large pores in a honeycomb shape were clearly found on the surface of the jatropha activated carbon (Plates1 and 2). The well-developed pores had led to the large surface area and porous structure of the activated carbon. Comparison of the precursor morphology with that of the prepared carbon showed substantial changes occasioned by phosphoric acid activation. The phosphoric acid incorporated into the interior of the precursor particle restricted the formation of tar as well as other liquids such as acetic acid and methanol, present in the raw jatropha material, and inhibited the particle shrinkage or volume contraction during heat treatment as stated by Bansal and Goyal [10]. A distinguishing feature of the JAC micrograph is the cracks in the carbon matrix probably developed as a result of thermal stress on the carbon matrix due to temperature changes in the activation process. The cracks contribute to the overall surface of the activated carbon which in turn increases it adsorption capacity.

Infrared spectroscopy was used to obtain information about the chemical structure and functional groups of the raw material and the prepared activated carbon. The FTIR spectrum of the Jatropha-waste cakes is quite similar to those of other lignocellulosic materials such as pistachio-nut shell, rockrose, and neem waste [11 - 13]. The bands located at 2918.5 and 2851.4 cm^{-1} correspond to the C=O vibrations in carbonates. The band at 1707.1cm^{-1} is ascribed to carbonyl C=O groups in carboxylic acids and esters. The C=O vibrations in amides may have caused the emergence of the band at about 1639.3cm^{-1}, while the vibrations at 1408.9 and 1319.5cm^{-1} are assigned to the bands C=O in carboxylate groups. The band at 1028.7 cm^{-1} can be attributed to Si-O-Si which may indicate the presence of silica. When comparing the two spectra (the raw material and the prepared activated carbon), some noticeable modifications took place in the wave number range of 3500–1000 cm^{-1}. These modifications make the spectrum of the activated carbon less complicated than that of the precursor.

Considering the spectrum of the prepared carbon, it was observed that there was a decrease in intensity of the band corresponding to O-H groups (~3500 cm^{-1}) and this may have resulted from the hydrolysis effect of H_3PO_4. Bands between 1408.9 and 1319.5 cm^{-1} , corresponding to the C=O, disappeared which suggests that phosphoric acid activated carbon contains less C=O groups than the raw material. The decrease of the amount of carbonyl groups may be due to hydrolysis effect of H_3PO_4, resulting in the decomposition of these groups and subsequent release of their byproducts as volatile matter. A disappearance of the weak band located between 3011 cm^{-1} and a shift of the C=O band from between 2918.5 and 2851.4 to 2754.5 and 2322.1 cm^{-1}were also observed. The appearance of a band (946 cm^{-1}) corresponding to PO_4^{3-} shows the presence of phosphates as a result of the activation and the appearance of a strong absorption band (741 cm^{-1}) corresponding to aromatic C-H groups. It can be implied from this findings that the activated carbon structure is richer in aromatics.

Thus it can be inferred that the acid impregnation of the rawmaterial followed by the pyrolysis step led to a more carbonaceous and aromatic structure due to the dehydration effect of H_3PO_4

and evolution of volatiles during pyrolysis. The IR analysis suggests that phosphoric acid chemical activation led to the incorporation of phosphorous element in the structure of the obtained carbon.

CONCLUSION

Based on the results obtained within the framework of this study, it appears that jatropha cake may constitute a suitable precursor for the manufacture of an effective activated carbon through chemical activation with phosphoric acid. The most efficient activated carbon is that obtained under the following optimal conditions: an acid concentration equal to 50% H_3PO_4, an impregnation ratio of 1:1, and a pyrolysis temperature of 400 ˚C. The adsorption characteristics of the activated carbon prepared under these conditions compared well, and sometimes more favourably than the previous reports for activated carbon. The results of the nitrogen gas adsorption analysis showed that the activation process produced high surface area materials with well-developed pores and volume. The surface areas and pore diameter of the jatropha activated carbon was 868 m^2g^{-1} and 2.128 nm respectively. FTIR showed the changes on the surface functional groups between the raw and activated jatropha carbons.

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