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Analysis and production of activated carbon from carica papaya seed by chemical activation

F.S.Omeiza[1]*, P.A.Ekwumemgbo[1], J.A.Kagbu[1], K.O.Israel[2]

[1]Department of Chemistry, Ahmadu Bello University, Zaria, (NIGERIA)

[2]School of Basic and Remedial Studies, Ahmadu Bello University, Funtua, (NIGERIA)

E-mail : omsafo@yahoo.com

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ABSTRACT

The production of chemically activated carbon from Carica Papaya Seed (CPS) was experimentally investigated using laboratory scale muffle furnace. The effects of process variables such as time, temperature and chemical reagents were explored. In this research work, physicochemical properties, effect of time and temperature on devolitization of Carica Papaya Seed (CPS) were determined using standard methods previous used for similar determination before it was used to produce activated carbon by 2-step method using Activating Agents (AA) ZnCl2 and 0.1M H3PO4.

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KEYWORDS

CPS;
Activated carbon;
pH;
Activating agents;
FTIR.

INTRODUCTION

Activated carbon is an amorphous form of carbon in which a high degree of porosity was been developed during manufacturing or treatment. It has a high level of porosity and associated large surface area which makes it an excellent adsorbent for a wide variety of substances from both the liquid and gaseous phases[1]. Activated carbon has a large internal surface and porosity which is the primary reason for its excellent adsorption capacities; hence most practical applications require an activated carbon with large volume of fine pore. In order to obtain activated carbon with such properties, carefully controlled processes of dehydration, carbonization and activation of raw material must be undertaken[2]

EXPERIMENTAL

Sample collection

Samples of CPS were collected from Samaru, Sabo, and Giwa markets in Kaduna State and Yalemu market in Kano State, Nigeria

Processing of samples

CPS samples were washed thoroughly with distilled water and sun dried and powdered into small particle sizes with a mortar and pestle. The CPS samples were then sieved into different particle sizes

Methodology

Preliminary studies

The methods used for the preliminary studies are those used by[3,4].

Moisture and dry matter content determination

Clean crucibles were dried in desiccators and 2.00g CPS samples were weighed into the crucibles and dried in an oven at 105°C for 3hrs after which they were cooled in a desiccators and then weighed

$$\%\ \text{Moisture content} = \frac{\text{Loss in weight on drying (g)}}{\text{Initial sample weight (g)}} \times 100$$

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$$\text{Dry matter}(\%) = \frac{\text{Oven dry weight (g)}}{\text{Initial sample weight (g)}} \times 100$$

Ash content determination

Oven dried samples from moisture content determination was used. The crucibles containing the dry samples were placed in a muffle furnace and temperature was allowed to rise to 500°C and after 2hrs the crucibles were removed and allowed to cool in a dessicator and weighed.

$$\%\ \text{Ash} = \frac{\text{Ash weight (g)}}{\text{Oven dried weight (g)}} \times 100$$

pH determination

1% solution of the raw and carbonized samples was prepared and allowed to stay for 2hrs. The pH of the supernatant was taken using Crisson pH meter at room temperature.

Bulk density

This was done by measuring the volume of water displaced when a known weight of raw and carbonized samples is dropped into a measuring cylinder containing known volume of water.

$$\text{Bulk density} = \frac{\text{Mass of sample taken}}{\text{Volume of water displaced}}$$

Determination of surface area

The surface areas of the raw and the carbonized sample were determined using the methylene blue adsorption test (MBT) method described by[5]. 2.00g of each adsorbent was dissolved in 200ml of dionized water and 10pgm of methylene blue was added to the adsorbent suspension and agitated in a shaker for 2hrs. Thereafter it was kept for 24hrs to reach equilibrium, after which aliquot was taken, and analyzed using CORNING colorimeter. To determine the amount of methylene blue adsorbed. A graph of concentration of methylene blue added versus the amount of adsorbed methylene blue was used to identify the point of complete cation replacement as shown below

$$\text{Specific surface area} = \frac{{}_{M}M_{B} \times Av \times AMB}{319.98} \times 1/Ms$$

${}_{M}M_{B}$ = Amount of methylene blue adsorbed at point of complete cation replacement

Ms= mass of adsorbent

Av = 6.02 × 1023

AMB = Area covered by one methylene (assumed to be 130 Å2)

Elemental analysis

Elemental analysis was done at the reactor facility section, centre for energy research and training (cert). Ahmadu Bello University, Zaria-Nigeria by Neutron Activation Analysis (NAA). The analysis was done on the raw and carbonized samples.

Effect of temperature variation and duration on ash formation

Six 2.00g of CPS samples were carbonized at 300°C,450°C,500°C,600°C,700°C for 2hrs and allowed to cool in a dessicator. They were all weighed using analytical balance, while four 2.00g CPS samples were at 500°C for different times.

Procedure for carbonization and activation

The samples of CPS were carbonized and activated by 2- step method[4]. 2.00g of raw CPS samples was weighed into preweighed crucibles and placed in a carbolite furnace at 500°C for 3minute to carbonize as shown below

$$\text{Raw-CPS} \xrightarrow[\Delta]{500^{o}C,\ 3\text{minutes}} C + CO_2\ \text{(CARBONIZATION)}$$

2.00g of carbonized CPS samples were mixed Separately with 2cm^3 of each Activating Agents (AA) i.e 0.1M $ZnCl_2$ and 0.1M H_3PO_4.

The samples were heated in a furnace at 750°C for 5minutes after they were cooled with ice cold water and allowed to dry at room temperature. They were stored in sealed air tight polythene bag

$$\text{Carbonized-CPS} \xrightarrow[\Delta]{AA,\ 750^{o}C,\ 5\text{minutes}} AC + CO_2\ \text{(ACTIVATION)}$$

The above was repeated until substantial amount of activated CPS was obtained. It was allowed to cool in H_2O and allowed to dry at room temperature and stored in polythene bags.

RESULTS AND DISCUSSION

TABLE 1 : Physicochemical properties of raw and carbonized CPS samples

Parameter	
% Moisture	14
% Dry matter	86
% Ash Content	2.93
% yield carbon	21.83
pH measurement	
(i) Raw	6.90
(ii) Carbonized	7.54
Bulk density (g/cm^3)	
(i) Raw	0.66
(ii) Carbonized	0.72
Surface area(m^2g^{-1})	
(i) Raw	183
(ii) Carbonized	268

% Moisture and dry matter

The yields and quality of activated carbon produced can be improved by the removal of moisture[6]. The lower the % moisture the better the yield and quality of the product. The % moisture of the CPS was 14%. The value obtained for CPS is higher than for coconut and groundnut shell samples[7] Cassava peels[8]. However, % dry matter for CPS was 86 % and this gives a substantial amount of organic matter needed for conversion to carbon by carbonization process.

% Ash content

This is a measure of the mineral content of the samples[3] and the amount of ash depends on the carbon source[9]. The ash content was found to be 2.93%. This is favourable because the ash serves as interference during the adsorption process. The lower the ash content, the better the material[9]

pH

The pH of AC depends on the nature of the activation process and AA used. The pH can be modified by washing[1] and that the pH of carbon in water suspension can vary between 4 and 12. Alkaline surfaces are characteristic of carbons having vegetable origin and this affects adsorption of dyes, colour and unsaturated organic compounds[10]. It is seen that for CPS sources the changes from acidic to alkaline as it goes from raw to carbonize. This is favourable for adsorption process where it can be used to remove toxic materials.

Percentage yield carbon

The yield is defined as the ratio of final weight of the obtained activated carbon after washing and drying to the weight of dried CPS initially used. As shown in TABLE 1 above, the %yield carbon from CSP is 21.83%. This is in agreement with reports that acceptable average of 70% weight of raw samples is lost as volatiles during the carbonization process[11].

TABLE 2 : Elemental analysis of raw-CPS and Carbonized-CPS samples

Element(mg/l)	Raw-CPS	Carbonized-CPS
K	257±4.00	420±1.60
Na	4±0.06	3±0.03
Mg	12±4.00	28±11.0
Fe	0.1±0.00	0.3±0.01
Ca	26±6.00	102±12.00
Cr	BDL	BDL
As	BDL	BDL
Al	101±5.00	157±0.60
P	5±0.03	11±0.01

BDL= Below Detection Limit

The result from TABLE 2 shows that the elemental quantities in the carbonized samples were generally much higher than the amount found in the raw samples. It may be that the carbonization process made the elements more susceptible to detection by getting rid of interfering masking agents such as organic substance like protein. Samples showed comparatively high elemental quantities for Potassium (K), Aluminum (Al), calcium (Ca) while Arsenic (As) and Chromium (Cr) are below the detection limit.

TABLE 3: Effect of temperature variation on devolatilization and ash formation

Temperature (°C)	Devolatilization (g)
300	1.33
450	1.56
500	1.61
600	1.62**
700	1.63**

****ash formation sets in**

From the above TABLE 3, it is evident that at temperature 600°C and 700°C, there was ash formation.

Hence, the optimum temperature for carbonization was experimentally determined to be 500°C.

TABLE 4: Effect of duration on devolatilization

Time (hrs)	Devolatilization(g)
2	1.61
2 ½	1.66
3	1.73**
31/2	1.87**

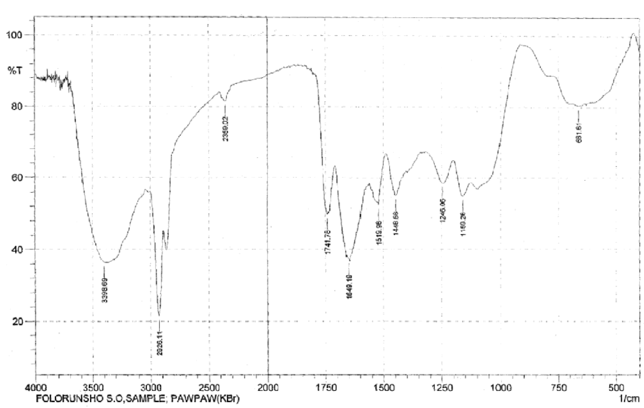
****ash formation sets in**

From TABLE 4, it is also evident that at about a period of 3hrs, ash formation sets in.

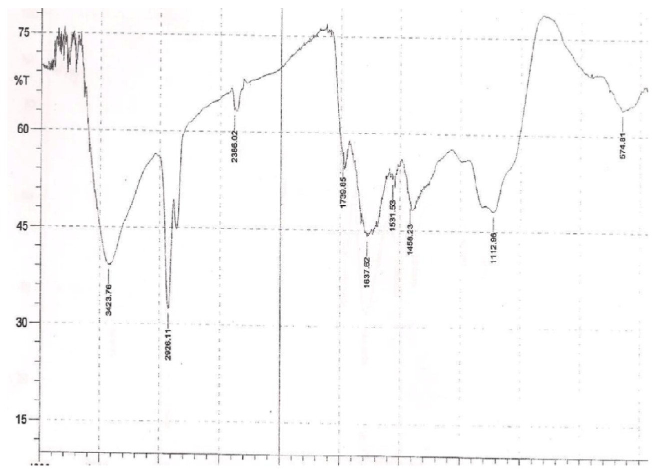
TABLE 5 : Band assignment for raw and carbonized CPS samples

Position	Assignment	References
3300-3500cm^{-1}	N-H stretching	[12,13]
2900- 2950cm^{-1}	C-H Stretching	[12-14]
1720-1745cm^{-1}	C=O stretching from ester	[12,15]
1630-1649cm^{-1}	Water	[12-15]
1 500- 1600cm^{-1}	aromatic ring vibration	[12,15]

FITR technique was used to identify those functional groups of the adsorbent produced. The infra red spectrum of the raw and carbonized sample was recorded in the 4000-400cm^{-1} region using KBr as solvent. An FTIR spectrum was also obtained to understand the surface structural chemistry of both the raw and the carbonized samples. Bands assignment is predicted in TABLE 4. The IR band consisted of four region: the broad N-H band (3300-3500cm^{-1}), C-H stretching region (2900-2950cm^{-1}),C=O stretching region (1720-1745cm^{-1}), water (1630-1649cm^{-1}), aromatic ring vibration(1500-1600cm^{-1}) and fingerprint band (below 1500). The most remarkable difference



FITR spectra for raw-CPS in KBr



FITR spectra for raw-CPS in KBr

between the two spectra is the intensity of the N-H stretching band for the raw and carbonized samples. The intensity of this band was much greater for raw than for the carbonized samples. This may be associated with differences in concentration of respective functional groups.

CONCLUSION

From the obtained results and discussions, it can be concluded that the various parameters determined are favourable and the 2-step methods was feasible for the production of activated carbon from carica papaya seed

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