International Research Journal of Environmental Sciences ISSN 2319–1414
Vol. **14(1),** 10-18, January **(2025)** *Int. Res. J. Environmental Sci.*

Towards designing activated carbons with specific surface characteristics from agro waste of *Telfairia Occidentalis* Hook. F.

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Available online at: www.isca.in, www.isca.me
Received 29th July 2024, revised 28th September 2024, accepted 5th November 2024

Abstract

A careful study of the surface properties under different reagents and various materials could avail scientists the luxury of determining the nature with respect to intended use of particular activated carbon and then design the adsorbent according to a specified need. In this paper surface chemistry of five activated carbon prepared with various activating reagents was studied with the aim of identifying the surface functional groups and acid-base characteristics. IR Spectroscopy, elemental analysis and Boehm titration methods were used and data subjected to some chemometrics. Results revealed IR spectra typical of activated carbon materials with C=C, O-H and C-H stretches of benzene, acids and esters and unsaturated systems present. The H/C ratio of FAC_6 *decreased rapidly as in all other carbons. Oxygen containing active sites were predominant even though acidic sites showed high concentration than basic sites except for* $FAC_{6,}$ FAC_7 *and* FAC_9*. Total active sites showed that* FAC_{10} *(3.84 ± 0.36) >* FAC_8 *(2.47 ± 0.15) >* FAC_9 *(1.70 ± 0.41) >* FAC_7 *(1.48 ± 0.26) >* FAC_6 *(0.96 ± 0.18) whereas acid / base ratios showed that* FAC_6 *(31) >* FAC_7 *(2.7) >* FAC_8 *(0.89) >* FAC_{10} *(0.79) >* FAC_9 *(0.75). Physicochemical properties have an influence on the acid/base properties of adsorbents studied. Such information when added to the many pieces already existing could assist in predicting the characteristic properties of a particular activated carbon.*

Keywords: Active sites, Activated carbon, Chemistry, Heteroatom, Infra-red, Spectroscopy.

Introduction

Surface characteristics of activated carbons can contribute significantly to their adsorption ability[1]. The activated carbon total surface area per unit weight, molecular surface attraction and contaminant concentrations in the medium correlates with the efficiency of an activated carbon in adsorbing compounds[2]. However, the applicability and selectivity of activated carbon (ACs) is strongly influenced by its surface chemistry[3,4]. It has been shown that through suitable chemical reaction, the surface can be modified to be either more acidic, basic or neutral[5]. The chemical composition of the original material additionally impacts the chemistry of these surface.

It is well understood now that the active sites which make up the functional groups are concentrated on the ends of grapheme layer[6]. Since these surface groups are the first point of contact for adsorbent – adsorbate interaction, research has soon that there exists strong relationship of the surface chemistry of activated carbon and its adsorption capacity. Some studies recommend that the appropriation of the functional groups on the outside of ACs relies upon pore size[7].

A lopsided appropriation of functional groups makes surface heterogeneity. Another justification, heterogeneity is fundamentally in the presence of heteroatom[8]. ACs have complex nature displayed as high surface region coming about because of an unpredictable pore structure, huge contrasts in the physicochemical parameters among the heterogeneous surface locales, the event of fractional delocalization of the π-electrons and the capacity of certain surface destinations to respond with water and different solvents.

Surface oxides are crucial in moderating the chemical behavior of activated carbons. Though a relatively minor component of the carbon, surface oxides provide acid-base character, redox and catalyst oxidative properties. In addition, Surface oxides are responsible for imparting a hydrophilic character to hydrophobic carbon[10].

Some of the functional groups that have been reported on the outside of activated carbon include: carboxylic, phenolic, quinone, lactone and anhydride. These groups contain locales of high electron thickness there by making them great surfaces for adsorption.

Albeit the presence of surface useful groups on carbonaceous materials have been for some time researched, the discussion is progressing to find out which activation cycle or surface treatment will deliver the best functional groups reasonable for explicit applications[11]. Makers of enacted carbon could give specialists data concerning the nature and properties of practical gatherings required, which would empower their items to be more widely utilized if the subtleties given are useful[12,13]. Evidence abounds to confirm the versatility of adsorption by activated carbon in both water and waste water[14]. Therefore, knowledge of surface chemistry could assist in preparing activated carbons that meet more specific demands in industries. This could have a ripple effect on the economy since cheap materials would lead to cheap activated carbon thereby reducing the huge foreign exchange used in the importation of activated carbon in Nigeria.

Fluted pumpkin is a sprawling vegetable shrub that grows low to the ground, characterized by its large, lobed leaves and elongated, twisting tendrils. This plant thrives in the moist regions of Nigeria and across Africa as a whole. It is classified under the family Cucurbitaceae[16]. In local dialects, it is referred to as 'ugu' by the Ibos in Eastern Nigeria, 'egusi iroko' by the Yoruba, and 'uwmenkhen' in Benin. The seed shell of the fluted pumpkin represents the largest morphological component of the plant. Researchers report that more than 80 tons of waste are generated annually within the eastern states of Nigeria[17,18]. A review of literature suggests that waste from *Telfairia occidentalis* can be a valuable source for production of high-quality activated carbons. Therefore, addressing the existing challenges such as tailoring the Acs for specific uses, and exploring new avenues, research can contribute to the development of sustainable and cost-effective materials for various applications.

Therefore, current research aimed to determine some surface functional groups information of ACs obtained in contribution to efforts towards undertaking the surface of AC prepared from fluted pumpkin seed shell. The data obtained here could add to existing data thereby enhancing further understanding of the surface chemistry of carbons from agro waste produced by one step activation. By using such low-cost materials, waste from *Telfairia occidentalis* could be removed from the environment and used to create wealth. Specific surface-active sites are identified on the surfaces in simple methods that could be reproduced towards creating ACs according to specific needs.

Materials and Methods

Preparation of FACs: Chemical activation and carbonization with, H_3PO_4, H_2SO_4, Na_2CO_3, NaOH and NaCl was carried out according to the methods[19] with slight modification. The numbers 6 to 10 were added as subscripts to the activating agents. Thus, FAC produced from H_3PO_4 acid will be referred to as FAC_6 for short. Hence FAC_6 to FAC_{10}. Briefly, 30% wt of aqueous solution of each reagent was mixed with 50g sample in ratio of 1:6. The mixture was left to soak for 12 hours and later heated to form a paste. The paste was placed in a pre-programmed furnace and carbonized at 500^oC for 1 hour in an inert atmosphere using carbon dioxide gas. The sample was allowed to cool overnight and then neutralized with HCl and washed with distilled water till the pH was constant. Then it was dried at 105^oC in an oven and later removed to cool at room temperature. The AC produced were sieved with 106 µm, put in air tight, labeled bottles and collectively called chemically prepared fluted pumpkin mesocarp activated carbon (CFAC). For brevity, CFAC will simply be referred to as FAC.

Physicochemical characteristics of FAC: pH values and electrical conductivities (EC) of samples were measured by use of method according to Castilla, C. M.[20], while iodine value, bulk density, pore volume, moisture and ash contents were and determined according to Vicete G. S.[21]. Eelemental composition of the various carbons was done by using the Perkin-Elmer CHN analyzer at Warri town in Mail infield laboratory, where oxygen was determined using method by ASTMD 5622 of 1995-98.

Fourier transforms infrared spectroscopy: Each sample of FAC was dissolved in Nujol, and a drop of each solution was placed between two polished flat sodium chloride plates (cells). The plates were compressed together and positioned in the IR spectrophotometer, where they were scanned 16 times at a resolution of 4 with the instrument model IR, Prestige 21 Schumadzu Corporation S/N A21004200709. Spectra of the prepared samples were obtained within the range of 500–4000 cm^{-1}, which were subsequently printed and analyzed using library values[22].

Determination of active sites: The determination of acidic and basic sites on FACs was carried out using acid-base titration based on Boehm's method as referenced by Bandoz et al.[15]. Each carbon sample, weighing 0.2g, was placed into a bottle, to which 20 ml of 0.1M NaOH and $NaHCO_3$ were added. A blank was created for every experiment conducted. The bottles were then purged with nitrogen (N_2), sealed, and shaken for a duration of 48 hours. Following this, the solutions were filtered, and the quantities of each absorbed reagent were assessed through titration with 0.1M HCl.

Surface Area Determination: An amount of 1g of dried activated carbon was placed in an elemenyer beaker, followed by the addition of 3.0ml of ethylene glycol, which was carefully measured. The resulting mixture was shaken gently to create a uniform slurry. This slurry was then positioned in desiccators along with anhydrous calcium chloride and ethylene glycol serving as desiccants, and the lid was secured. The weight of the carbon and ethylene glycol mixture was recorded at ten-hour intervals until the difference between consecutive measurements fell below 0.001g. The weight noted at this point was documented as the final weight of the slurry.

The specific surface area was calculated as

$$SSA = \frac{W_{FS} - W_S}{2.86\times10^{-4}} = \frac{W_a}{2.86\times10^{-4}} \qquad (1)$$

SSA refers to specific surface area, while W_F is final of the final slurry, then, Wsrefers to the weight sample used, and Ware present weight of ethylene glycol left on the carbon sample.

Wa = W_{F} – Ws,2.86×10^{-4} is a constant for the weight of ethylene glycol used in forming a monomolecular of surface layer of square meter (g/m^2).

Chemometric methods: Atomic ratios (AR): Using the equation

$$\frac{Y}{C} = \frac{\%Y/M_{m(Y)}}{\%C/M_{m(C)}} \ldots\ldots\ldots\ldots \qquad (2)$$

Atomic ratios were calculated and used in plotting figure 1, where Y/C is atomic ratio for the element y (Hydrogen (H), Oxygen (O) and Nitrogen (N) and Y%, % Carbon (C) - the mass percentages (obtained by elemental analysis of activated carbon) of the element y and carbon, respectively; Mm(Y) and Mm(C), the molar masses for the element Y and C – carbon[23].

Elemental ratios (ER): Elemental ratios of all metals determined in FACs were calculated as the percent composition of one element to that of another. The ER obtained were plotted to determine the trends.

Plotting surface active sites: Concentrations of surface-active sites were plotted as a bar chart and then compared with each other using error base percentage to obtain a trend.

Acid/Base ratios versus physicochemical properties: In other to determine trends in Acid/Base ratios with physicochemical properties of chemically prepared activated carbon samples, scatter plots were obtained using excel Microsoft program.

Results and Discussion

Physicochemical properties-characteristics of FACs: The table shows the mean values of physicochemical characteristics of prepared activated carbon (Table-1). All samples were alkaline except FAC_7. Other characteristics compared well with each other and were within acceptable limits. However, the conductivities of all FACs were significantly higher than that for commercial activated carbon (CAC), but it was observed that surface areas were significantly lower than for CAC.

IR spectrum of CFACs: Figure-1 shows 1R spectrum of FAC_6 to FAC_{10}. The IR spectrum of all CFACs showed presence of C=C of benzene ring (680-730cm^{-1}) and O-H of alcohol and ester at 3320cm^{-1}. There was a C=N group at 1620cm^{-1}. No other CFAC sample showed presence of C=N group. IR spectroscopy showed that H_3PO_4 acid treatment promotes a more extensive loss of hydrogen and a marked decrease in aliphatic groups increased aromaticity and increased stability of carbonyl functional groups for samples activated H_3PO_4. These results are similar to those reported in literature[6,11].

The IR spectrum of FAC_7 showed a total of eight peaks, including the unidentified peak at 590cm^{-1} and the O-H Stretch of alcohol 329cm^{-1}. Notice that an aldehydic group has been introduce into FAC_7. Of all 9 peaks on the spectrum of FAC_8 the peak between 1310-1340 to 1950 on all confirm the presence of C=C – functional group arising from an alkene were 2680-2680cm^{-1} showed the presence of C-H Stretch. In FAC_8 as in FAC_9 the aldehydic group were absent.

The peaks were sharper and with less broadening shoulders as were the case in other CFACs. The region between 400-1300cm^{-1} was vituallly peakless exept for a weak peak at 730cm^{-1} indicating a monosubstituted benzene. Not common to most of the CFACs, IR spectra of showed more peaks inFAC_9, including the unidentified peak at 500cm^{-1}. The O-H bend of acid according to Uzoukwu, 2009[24] was observed as small medium weak peak at 990cm^{-1}.

FAC_7 showed total of eight peaks including the unidentified peak at 590cm^{-1}. An aldehydic group was found in FAC_7 in addition to the other functional groups. Of all nine peaks on the spectrum of FAC_8 the peak between 1310-1340 to 1950cm^{-1} on all confirm the presence of C=C – functional group arising from an alkene were 2680- 2680cm^{-1}. The peaks were sharper with less broadening shoulder. The region between 400 - 1300 was virtually peak less except for a weak peak at 730cm^{-1} indicating monosubstituted benzene. Like most of the FAC the I.R spectrum of showed more peaks in FAC_9, including the peak that is unidentified at 500cm^{-1}. The O-H bend of acids according to was observed as a small medium weak peak at 990 cm^{-1}.

Table-1: Physicochemical characteristics of prepared activated carbon.

AR	Code	pH	Cond.	IV	Bd	PV	MC	Ash	SFA
NS	CAC	8.8±0.2	184±4	220±2	0.65±0.1	-	-	-	1200±10
H_3PO_4	FAC_6	8.2±0.1	265±22	240±2	0.36±0.1	$3.8\pm0.2\times10^6$	4.2±0.1	16±2	634±22
H_2SO_4	FAC_7	6.2±0.1	295±5	246±7	0.38±0.1	$3.4\pm0.1\times10^6$	3.0±0.2	13±2	670±15
Na_2CO_3	FAC_8	8.6±0.2	310±16	245±9	0.36±0.1	$4.0\pm0.1\times10^6$	2.7±0.2	11±2	763±20
NaOH	FAC_9	8.6±0.1	311±14	245±9	0.32±0.1	$4.0\pm0.2\times10^6$	2.7±0.1	11±2	788±22
NaCl	FAC_{10}	7.6±0.1	301±12	268±4	0.35±0.1	$4.1\pm0.1\times10^6$	3.6±0.1	09±1	801±21

AR: Activation reagent, NS: Not specified, Cond.: Conductivity (µS), IV: Iodine value (mg I /g), Bd: Bulk density (g/ml), PV: Pore volume (m^3/g), MC: Moisture content (%), Ash: Ash content (%). SFA: surface area (g/m^2), CAC: commercial activated carbon.

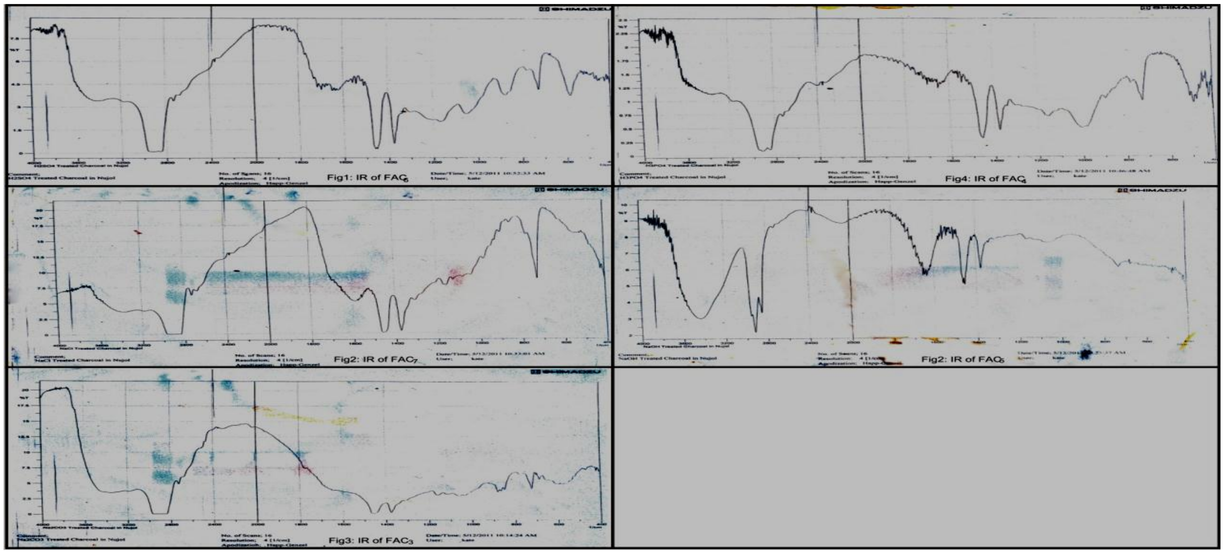

Figure-1: IR spectra of chemically activated carbon samples (top left to right the IR spectra are numbered FAC_6 to FAC_{10} from top left to right).

FAC_{10} Showed more peaks than all other nine AC samples, and as usually the C=C and O-H stretch of alcohol and was observed at 730, 870 and 1050, 3400cm^{-1} respectively. The shifting of towards higher energy levels may be due to hydrogen bonding. Every sample had a weak and broad peak at 3290-3600cm^{-1}. Some researchers[16,17] had observed similar peaks, and explained that they indicate the presence of moistureas confirmed by hydroxide ions as moisture was trapped on their surfaces during sample preparation and handling as a result of high adsorptive nature of the activated carbons.

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Table-2 shows data interpreted from IR spectra of FAC_6. The IRspectrum of all FAC showed presence of C=CH of benzene ring at 680-730 and O-H of alcohol and ether as was found in FAC_5. However the O-H stretch of alcohol and ether was found at 3320cm^{-1}, far higer than all other O-H groups found in FAC.Another anomally was the presence of C=N group found at 1620cm^{-1}. This does not aggree with any of the AC samples but seems to agree with result of elemental analysis and was found to be highest amongst all other FACs. It should be emphasised that no other FACs sample showed presence of C=N group, a hetero atom functional group. IR spectroscopy showed that H_3PO_4 acid as activation agent enhances an extensive hydrogen loss, leading to higher stability of carbonyl functional groups due to decreasing aliphatic groupsfor samples.

Elemental composition of FACs: The mean values of elemental constituents of FACs are presented in Table-3. The percent carbon content of AC based on processed activated carbon ranged from FAC_8 (89.00±0.30) to FAC_{10} (94.00±0.40). The carbon content followed the order: FAC_{10}>FAC_9> FAC_6 and FAC_7> FAC_8. These lower values of carbon content are considered high[18]. The hydrogen content ranged from FAC_9 (0.29 ± 0.02) % to FAC_7 (0.40 ± 0.02) %. These values showed high hydrogen content of FACs. The nitrogen content ranged from FAC_8 (0.10±0.01) % to FAC_6 (0.18±0.02) with a mean of 0.19. Percent oxygen in all FACs ranged from FAC_8 (0.10 ± 0.0) to FAC_{10} (3.0±0.20)[27].

The mean percent oxygen for FACs (2.14) was lower than those values reported by Carrot et al.[19]. Ash content followed the same trend even though it was for all samples. High ash contents are not desirable for AC being used as adsorbents[19].

The atomic ratios for FACs are plotted against the FACs in Figure-2a. Ratios used here are those of O/C, O/H, H/C and N/C. The O/C ratio increased from FAC_6 (0.008) FAC_7 (0.010) and then decreased to FAC_9 (0.005) and after, increased slowly to FAC_{10}. This is expected to lead to oxygenated functional groups[20]. The H/C ratios for all five FACs decreased continually from FAC_7 (0.028) to FAC_{10} (0.1) except for FAC_6 (0.207). It could be explained that dehydrogenation predominated cyclization in going from acid to basic and finally a neutral impregnation reagent. The O/H ratio of FACs (Figure-2a) decreased continually from FAC_6 (0.45) to FAC_7 (0.008) and then increased to FAC_{I0} (0.012).

Table-2: Surface functional groups present on various FACs.

Sample	C=C of benzene	C-H saturated	C-O aldehyde and ketone	O-H alcohol and ether	C-O acid & ester
FAC$_6$	+	-	-	+	-
FAC$_7$	+	-	+	+	-
FAC$_8$	+	+	-	+	-
FAC$_9$	+	+	-	+	-
FAC$_{10}$	+	-	+	+	-

\+ indicates presence of functional group, - absent.

Table-3: Elemental composition and ash content of chemically prepared activated carbon.

Element (%)	FAC$_6$	FAC$_7$	FAC$_8$	FAC$_9$	FAC$_{10}$	Mean
C	90 ±0.30	90 ± 0.30	89± 0.30	92 ± 0.40	94± 0.40	91.00
H	0.31± 0.02	0.40 ± 0.02	0.3 ± 0.01	0.29± 0.02	0.3± 0.01	0.32
N	0.18± 0.02	0.16 ± 0.02	0.10 ± 0.01	0.14 ± 0.02	0.17± 0.02	0.19
O	1.8 ± 0.5	2.0 ± 0.30	1.1 ± 0.10	2.8 ± 0.10	3.0± 0.20	2.14
Ash	1.6 ± 0.01	2.0 ± 0.02	1.8 ± 0.01	1.3 ± 0.01	1.2± 0.01	1.34
Total	93.09	94.56	91.3	96.53	98.67	94.83

Mean ± SEM = Mean values ± Standard error of means for triplicate analysis.

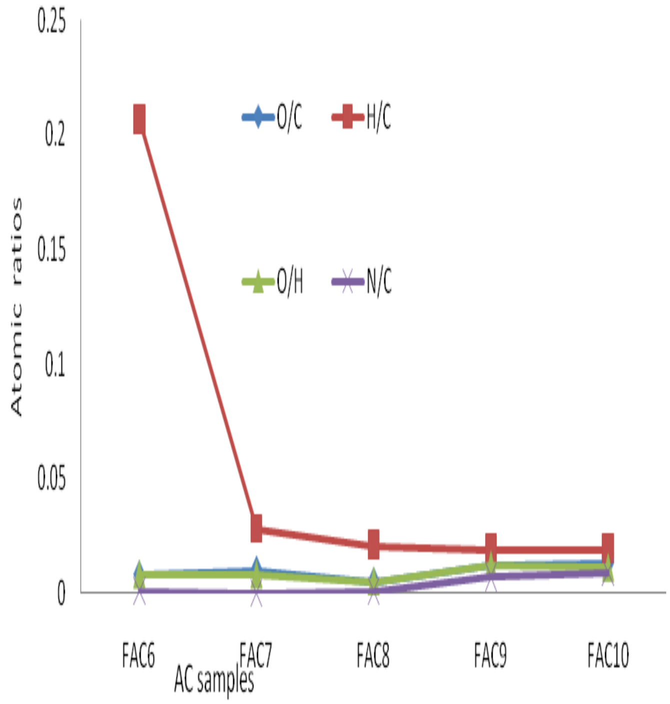


Figure-2a: Graph of atomic ratios of FACs.

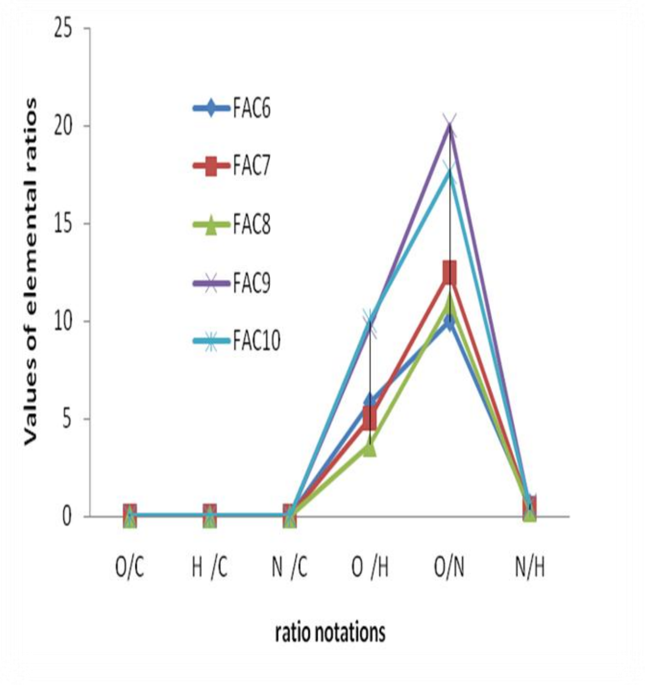


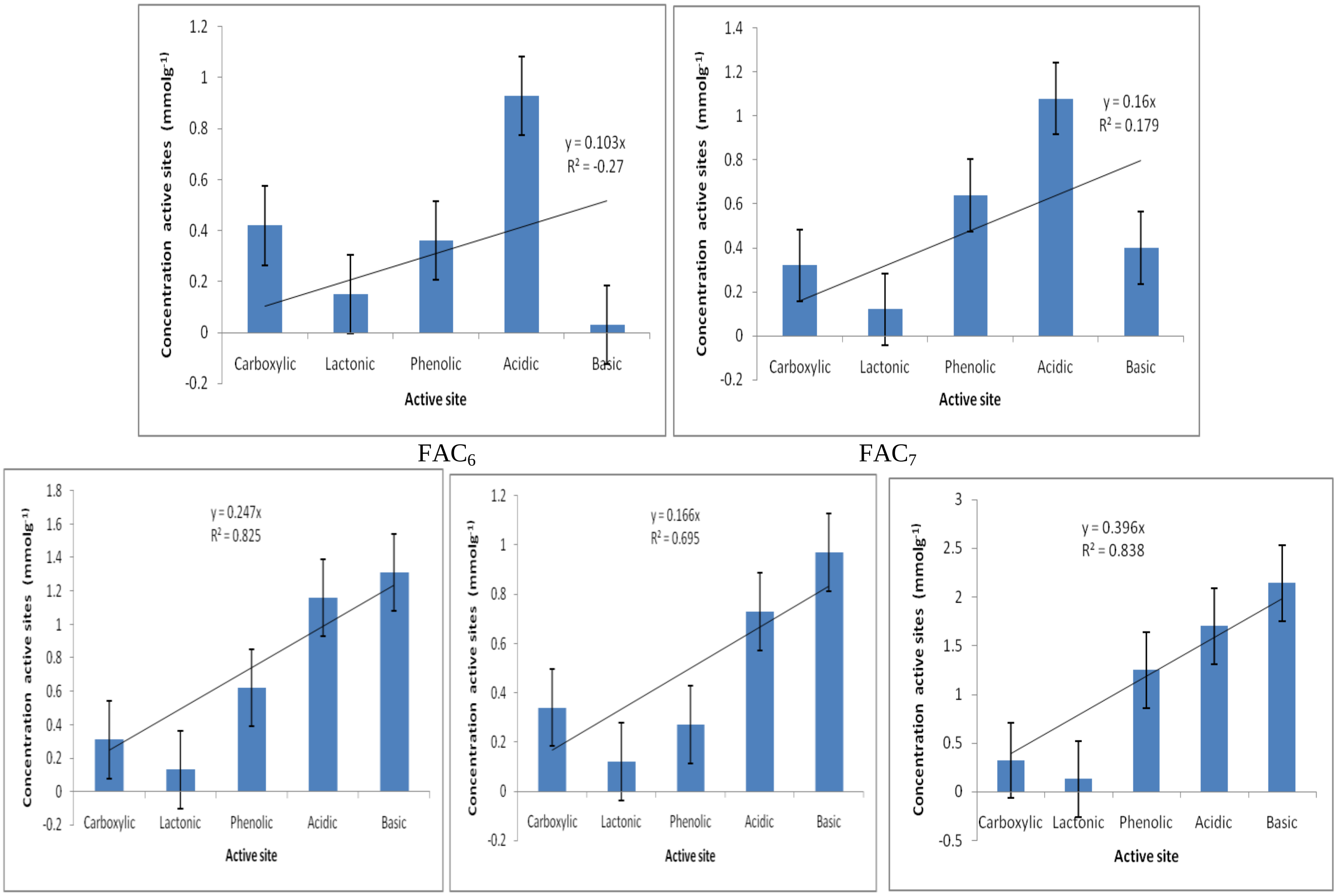
Figure-2b: Graph of elemental ratios of FACs.

The N/C ratios showed a steady increase from FAC$_6$ (0.01) to FAC$_{10}$ (0.009). As explained that a constant N/C ratio indicates stabilization during carbonization, stage of producing activated carbon, there is an appreciable concentration of nitrogen in the precursor. In this case it is not volatilized and only little HCN is formed during the formation of a more stable functional group contain nitrogen function group in FAC[20]. The atomic ratios for FACs showed that H/C ratio for FAC$_6$ was higher than all others.

Values of elemental ratios (ERs) for all elements determined in this work ranged from H/C (0.0003) to O/N (20). When plotted on a trend chart (Figure-2b) revealed that O/C, H/C, N/C were at same level 0, converging on the y-axis prior to O/H at the ascend to the peak. At O/H the ERs followed the decreasing order: FAC$_{10}$ > FAC$_9$ > FAC$_6$ > FAC$_7$ > FAC$_8$. The peak was observed at O/N with elemental ratio corresponding to FAC$_9$. The order of decreasing values of ERs at the peak was FAC$_9$> FAC$_{10}$ > FAC$_7$ > FAC$_8$ > FAC$_6$. At the N/H values ERs converged again at 0 levels on the y-axis.

Data for the various active sites have been presented as mean values plus or minus standard deviation for the triplicate experiments. Amongst all five FACs, FAC$_{10}$ (1.41) exhibited highest concentration of surface groups (Table-4). This was followed by FAC$_7$ (0.78) while FAC$_9$ (0.28) was the lowest. Adsorption capacity is enhanced by high surface group concentration[21,22]. This suggests that carbon surface -charge transfer interactions that occur between adsorbate and groups on the fused ring were more concentrated on FAC$_{10.}$ The order of Boehm titration results reveal that the nature of surface groups concentrated on FAC$_{10}$ (3.84) amongst FACs. However, FAC$_7$, FAC$_8$, FAC$_9$ and FAC$_{10}$ showed good concentrations of active sites.

Bar plots (Figure-3) of active site concentrations show general positive increase from carbocylic acid group to base groups. This trend is emphasized in FAC$_8$, FAC$_9$ and FAC$_{10}$. All equations had slops ranging from 0.103 to 0.396. R^2 values for FAC$_8$, FAC$_9$ and FAC$_{10}$ were above 6.00 while FAC$_6$ and FAC$_7$ had values of 0.27 and 0.179 respectively. Errors bars were uniform except for lactonic concentration of lactonic sites in all FAC$_S$. Concentration active sites for FAC$_S$ showed fairly good regression parameters[30-32].



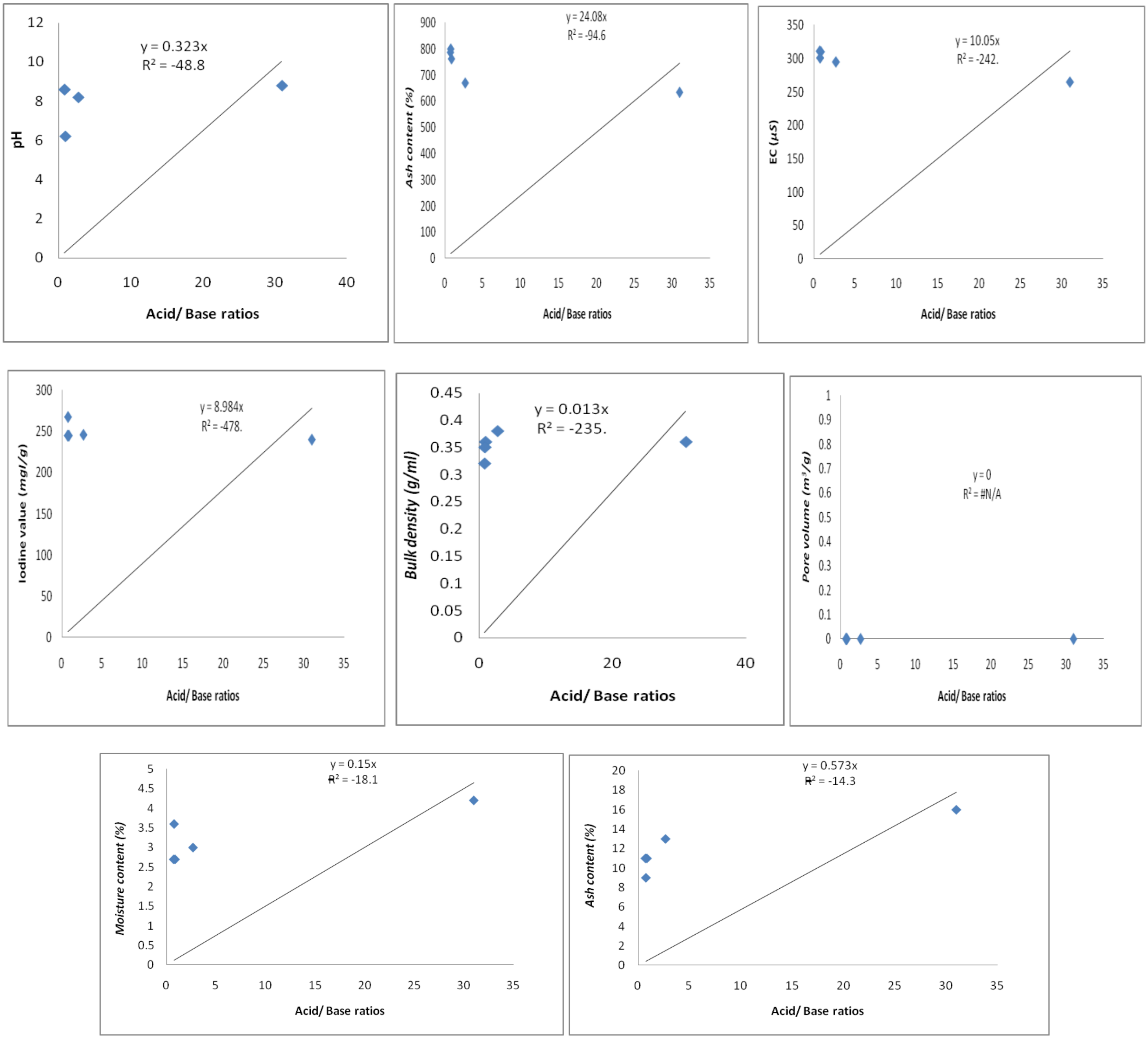
FAC$_6$ FAC$_7$

FAC$_8$ FAC$_9$ FAC$_{10}$

Figure-3: Bar plots of concentrations of surface-active sites showing percent error bars for comparing.

Table-4: Concentration of various active sites on FACs in mmolg^{-1}.

	Carboxylic	Lactonic	Phenolic	Acidic	Basic	Total	Acid/base
FAC_6	0.42 ±0.01	0.15 ±0.1	0.36 ±0.21	0.93 ±0.34	0.03 ±0.01	0.96 ±0.18	31
FAC_7	0.32 ±0.02	0.12 ±0.01	0.64 ±0.08	1.08 ±0.71	0.40 ±0.21	1.48 ±0.26	2.7
FAC_8	0.31 ±0.10	0.13 ±0.02	0.62 ±0.13	1.16 ±0.28	1.31 ±0.61	2.47 ±0.18	0.89
FAC_9	0.34 ±0.02	0.12 ±0.01	0.27 ±0.03	0.73 ±0.22	0.97 ±0.23	1.70 ±0.41	0.75
FAC_{10}	0.32 ±0.02	0.13 ±0.01	1.25 ±0.26	1.70 ±0.77	2.14 ±0.81	3.84 ±0.36	0.79



Figures-4: Trends in activated carbon characteristics in relation to increasing acid/base ratio.

Scatter plots of physiochemical properties and acid/base ratios (Figures-4) revealed direct relationship with large values of gradients in the case of ash content, EC, iodine value. However, scatter plots for pH, bulk density with acid/base showed low gradients while pore volume and acid/base showed zero gradients. Equally there was no R^2 value, whereas all other scatter plots had R^2 values ranging from that were negative and low. It could be observed that physiochemical properties of FAC_S had good regression parameters with acid/base ratios. It was observed that pore volume had no relationship with acid / base ratios[33-35].

Conclusion

The use of chemical activation procedures in this research has revealed improved chemistry of the surface observed on activated carbons from fluted pumpkin seed shell. However, there was no significant difference between oxygen containing surface groups. There were more stable alkaline groups then an increase from acid to basic and finally neutral activation reagents. Active site concentrations showed general positive increase from carboxylic acid group to base groups. Functional groups developed here suggested that chemically prepared fluted pumpkin activated carbons are promising as potential adsorbents. Though large enough error bars were uniform and suggest a bias in the results obtained, while physicochemical properties have some influence on surface properties of the activated carbons studied. By conducting careful studies of the surface groups and optimizing chemical activation procedures, researchers could generate information that can enable selection and production of activated carbons for specific needs and applications.

Acknowledgments

The authors acknowledge gratefully the undergraduate students of Pure and Industrial Chemistry Department, Madonna University, and to the Head of department who provided funds for chemicals and the IR analysis at Fugro Laboratory Ltd, Port Harcourt, Nigeria.

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