



Preparation and Characterization of Activated Carbon from Palm Kernel Shell by Chemical Activation

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Abstract

This work investigates the effect of change in temperature and time of activation on physicochemical properties of activated carbon prepared from palm kernel shell by chemical activation. The experimental design yielded nine activated carbons which were characterized by standard methods, including SEM examinations and FTIR analysis to study the surface morphology and to analyze the functional groups on the prepared carbons surface respectively. The BET surface area, obtained from nitrogen gas adsorption ranged from 17 to 217 g/m². The BET surface area of the prepared activated carbons was in the order: A1 > A2 > A3 > C1 > B3 > B1 > B2 > C3 > C2. The nitrogen gas adsorption also showed that samples A1, A2, A3, B1, B2, B3 and C1 conform to type I of IUPAC isotherm classification while samples C2 and C3 conform to type II isotherm. The carbonized sample, (CC), exhibit type III isotherm. Result shows that the best condition for the preparation of activated carbon for adsorption from palm kernel shell using KOH as activating agent be 800°C for 45 minutes, taking into cognizance the feasibility of scaling up production.

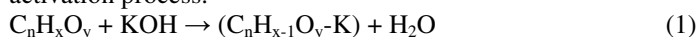
Keywords: Palm kernel shell, adsorption, chemical activation, surface chemistry, activated carbon.

Introduction

Activated carbon is a carbonaceous material which is predominantly amorphous in nature and in which a high degree of porosity is developed by the process of manufacturing and treatment. Every activated carbon has a memory which largely depends on the source and the preparation conditions¹. Activated carbon can be manufacture from virtually all carbonaceous materials. However agricultural wastes offer the most available and cheapest of all the known raw materials. Activated carbon is inexpensive and hence very widely used adsorbent^{2,3}.

Kernel shell is an abundant solid waste from oil processing mills in Nigeria. This shell may be a good source of quality activated carbon. Nigeria rank third largest producer of palm products in the world after Malaysia and Indonesia. Nigeria production of palm kernel oil increased from 190 000 ton in 1999 to 203 000 ton in 2003 with a growth rate of 1.3%. This is an indication that a huge volume of kernel shell are being generated without due consideration to the environmental problems. This waste can be a renewable source of activated carbon. Several uses are attributable to activated carbon, ranging from removal of undesirable odor, color, taste and other organic and inorganic impurities from domestic and industrial wastewater. The extraordinarily large internal surface and pore volume are developed during the preparation stages. The preparation involves two main steps which are carbonization and activation of the carbonized product. The carbon is usually charred at temperature below 600°C during the carbonization⁴. The char is then subjected to chemical treatment such as KOH impregnation to develop porosity, and increase adsorption affinity⁴

The following reactions occurred during impregnation and activation process:



The first stage which is semi carbonization process can be represented thus:

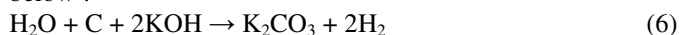


Char usually contains 75-80% carbon; some oxygen, (15-17%) and some hydrogen, (<3%)⁵

Tars consists of phenols, CH₃COOH, CH₃OH, (CH₃)₂CO, and other high molecular weight hydrocarbons. The gaseous components include among others H₂O, CO₂, CO. The following reaction occurred during the activation (2nd stage) process⁵:



Potassium intercalated with the carbon matrix during activation, resulting in the widening and formation of new pores. Secondary reaction that could possibly take place is given below⁵:



Potassium carbonate is known to prevent excessive sample burn-off, as such, a well developed porosity and high yield resulted⁵. Potassium carbonate is known to prevent excessive sample burn-off, resulting in high yield and well developed internal porosity⁵. The structure of the carbonaceous material became reorganized in a fashion characterized by large internal surface through the activation process⁶. The intrinsic properties

of the activated carbon are dependent on the raw material, the activating agent, and the conditions of chemical treatment^{1, 7}. However the most important factor in making active carbon of good quality activated carbon, besides the properties of the precursor is the preparation conditions¹. Activated carbon has been produced from palm kernel shell by several methods and the optimum carbonization temperature and time for the source material has been reported. However the optimum KOH activation conditions which influence the final adsorption properties the most have not been investigated. In this work the influence of temperature of activation and time variation on adsorption capacity of KOH activated kernel shell were investigated.

Material and Methods

Palm kernel shell was collected from Sabo market in Zaria. The palm kernel shell was removed, washed, dried and crushed using a locally made grinder. This was sieved to 1.18mm and carbonized at 400°C for 1 hour. A portion of the carbonized material (20 g) was mixed with 200 cm³ KOH solution at impregnation ratio of 1:1(KOH pallet: Char). Impregnation at 80°C with continuous stirring for 2 hours was employed. The sample was filtered using a vacuum pump and dried overnight at 120°C. The dried sample was then pyrolysed at temperatures of 800°C, 900°C and 1000°C. The activation times at each temperature were 15 minutes, 30 minutes and 45 minutes in a furnace. The activated carbons were gradually cooled to room temperature. It was washed using distilled water and then 0.1 M HCl until the washing water had a pH 7.0⁸. The prepared activated carbons were dried at 120°C overnight, cooled and stored for further studies.

The Brunauer-Emmett-Teller (BET) Surface areas were determined by nitrogen adsorption at 77 K. The nitrogen gas adsorption measurements were done after the carbon was degassed at 300°C in an inert condition for 24 hours. A relative pressure of between 10⁻⁵ and 0.995 of nitrogen gas was used to obtain the N₂ adsorption isotherm. The BET surface area, mesopore volume and micropore surface area of the activated carbon were determined by the application of the Brunauer-Emmett-Teller (BET) and Dubinin-Asthakov (DA) analysis software respectively. The pore size distribution was calculated using the method of Guzel and Uzun¹. Frouer Transform Infrared Spectrometer (Themo Nicolet, model magna 760) was used to determine the functional groups of the adsorbents using the pellet press disk technique. The the adsorbent/KBr mass ratio was 100:1. The spectral scanned at the rate of 10 nms⁻¹ ranged from 4000 to 400 cm⁻¹. A scanning electron microscope (model SEM-50, Philips, Holland) was used for obtaining micrographs of the prepared activated carbons.

Results and Discussion

The kernel shell was converted to activated carbon using potassium hydroxide as activating agent. Potassium hydroxide is

a common activating agent in activated carbon manufacturing industries. The choice of this chemical in this work was informed by the fact that it is more eco-friendly compared to the ZnCl₂, H₃PO₄ more frequently used. The experimental design to optimize the preparation conditions yielded nine (9) distinct activated carbons from palm kernel shell. Characterizations of the carbons were by standard methods, including SEM examination, nitrogen gas adsorption for surface area measurement and FTIR for surface chemistry studies.

The morphological structures on the carbons were analyzed with the aid of SEM. The surface structures of the activated carbons have burnt out pores with tunnel or honeycomb-like structures as shown in figure 1. The surface of the activated carbons changed with temperature and time of activation. The SEM photographs for samples B1 and sample C1 are characterized by a smooth surface with many orderly pores developed, compared to the others. This is as a result of lack impurities such as tar that could clog up the pores and inhibit good pore structure development.

The effect of change in activation time and temperature, which most significantly influence the physicochemical characteristics including; surface area, bulk density and yield of the adsorbent were studied and discussed. The physicochemical properties are a pointer to whether the experimental carbons can be used commercially⁹. Physicochemical properties describe the suitability of adsorbent for a particular process¹⁰. The yield of any chemical process is an important variable to be considered when such process is to be scaled up. The yield is the quantity of the final product formed from the starting raw material on a scale of 100. A high yield is required for a feasible economic production of activated carbon. The yield of the prepared carbon varied widely with the temperature and time of activation. Table 1 showed that carbons activated at 1000°C have the least percentage yield.

The percentage yield also decreased as the activation time for the carbon increased from 15 to 45 minutes. This is because increase of temperature and time of activation resulted in more volatile component being lost, and hence a decreasing percentage yield. The highest observable percentage yield was 46.33% and was obtained at an experimental condition of 800°C for a time of 15 minutes. This was about twice the yield obtained at activation condition of 1000°C for 45 minutes which was 19.25%. The time of activation does not influence the yield remarkably, relative to the activation temperature. The percentage yield increased from 19 to 29, 38 to 41 and 43 to 46% as the time of activation was increased from 15 to 45 minutes for activation temperature of 1000°C, 900°C and 800°C, respectively. It is also clear that the influence of time was more pronounced at higher temperatures (table-1). However the percentage yield decreased from 46 to 38 and to 19 as the temperature of activation increased from 800°C to 900°C and to 1000°C, respectively. This is significant because a rise of temperature by hundred degrees resulted in a decrease of ten

percent yield. The effect of activation temperature and time is therefore most felt at the highest experimental temperature of 1000°C. Similar trend were reported by literature¹¹⁻¹³. It has also been reported that increasing the KOH-kernel shell ratio increases the percentage yield⁵. This is because the potassium ions attached onto the carbon surface act as catalyst to accelerate direct reaction between the carbon and KOH. Bulk density is an important characteristic of activated carbon as it is a measure of the amount of adsorbate the carbon can hold per unit volume. It is one of the variables that must be considered in the design of adsorption column and it also affected the overall cost of the adsorption process¹⁴. The bulk density of the prepared carbon is in the range between 0.6233 and 0.6952 (table-1). The very narrow range in the values is due to the fact that the parameter depends largely on the starting raw material (palm kernel shells). It is observed that as the temperature and time of activation increased, the bulk density decreased but only slightly. The influence of time is much less significant than that of temperature. Lima *et al.*¹⁵ reported that bulk density decreased with progression of activation due to porosity development. Bulk density is also very closely related to and

affected by particle size; as such the similar values obtained for the nine samples may not be unconnected with the similar particle size (855µm) used for the determination of the parameter for all the samples. The slight variation in value may be as a result of varying experimental conditions (varying temperature and time of activation). In general, the bulk density of the prepared carbons is high and similar to those reported in literature. Vitidsant *et al.*¹¹ reported bulk density of 0.5162 g/cm³ for palm oil shell pyrolysed at 800 °C for 60 minutes. Bulk density of commercial carbons in the range of 0.47 to 0.54 gm⁻³ have been reported by researcher¹⁶. Higher bulk density also imparts more mechanical strength¹⁷. The surface area obtained from N₂ adsorption ranged from 17 to 217 g/m² (table-2). Large surface areas show possibility of high adsorption of organic compounds. Surface area is therefore an important attribute for consideration in terms of applications of activated carbon¹⁶. Figure-2 shows the nitrogen gas adsorption isotherm for the prepared activated carbon. The BET surface area (Table 2) is in the order: A1 > A2 > A3 > C1 > B3 > B1 > B2 > C3 > C2

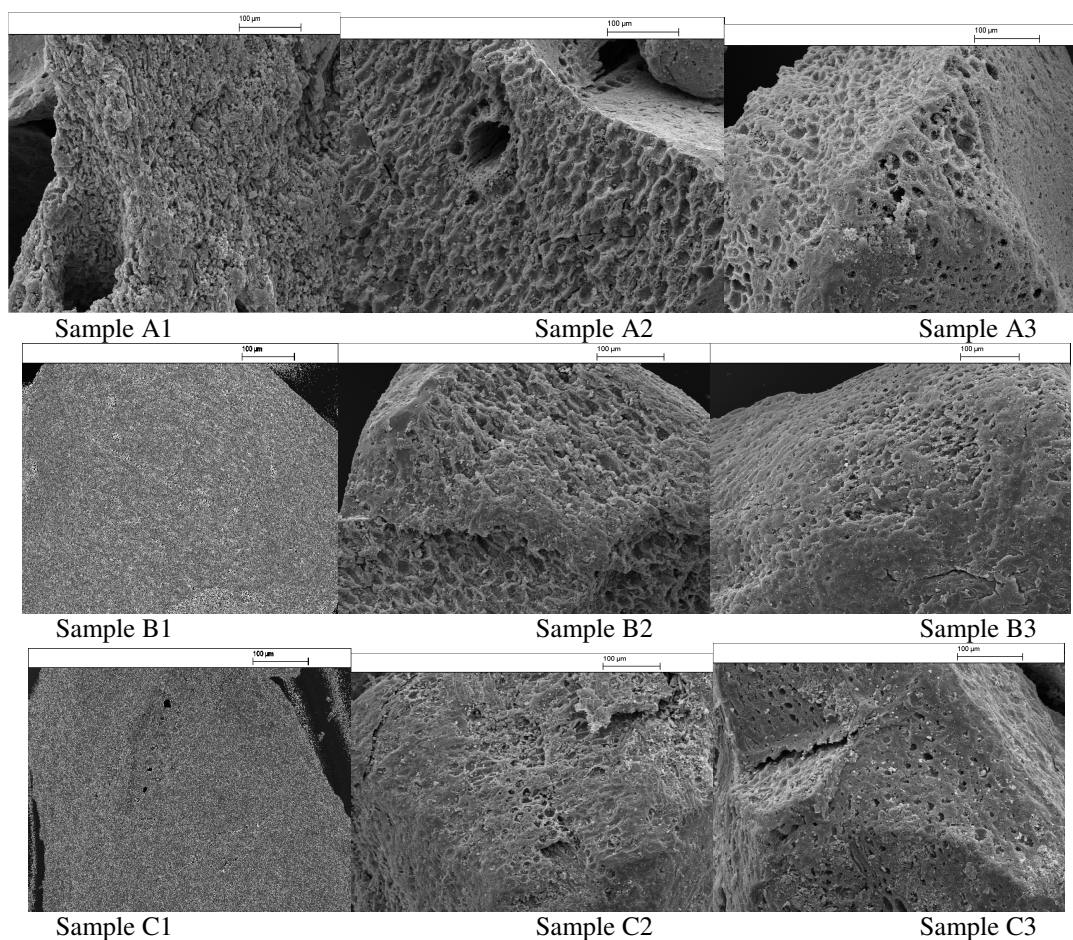


Figure-1
The SEM photograph of the prepared activated carbons

Table-1
Physicochemical properties of the prepared activated carbons

Sample code	A1	A2	A3	B1	B2	B3	C1	C2	C3
Activating Temp(°C)	1000	1000	1000	900	900	900	800	800	800
Activating time (mins)	45	30	15	45	30	15	45	30	15
Yield (%)	19.25	24.14	29.84	38.59	38.83	41.21	43.38	44.35	46.33
Bulk density (gcm ⁻³)	0.62	0.64	0.68	0.63	0.64	0.69	0.63	0.65	0.70

Table-2
The surface area and pore size characterization of the prepared activated carbons

Sample code	A1	A2	A3	B1	B2	B3	C1	C2	C3
S _{N₂} (gm ⁻²)	217	155	141	72	54	73	127	17	20
V _μ (cm ³ g ⁻¹) x10 ⁻²	11.00	7.80	6.70	3.80	3.00	3.80	6.30	0.92	1.1
V _m (cm ³ g ⁻¹) x10 ⁻²	1.00	1.20	0.40	1.00	0.70	0.50	0.30	0.58	0.50
V _T x 10 ⁻²	12.00	9.00	7.10	4.80	3.70	4.30	6.60	1.50	1.60

S_{N₂}=N₂ BET surface Area, V_μ=Micropore Volume, V_m=Mesopore Volume, V_T=Total pore volume

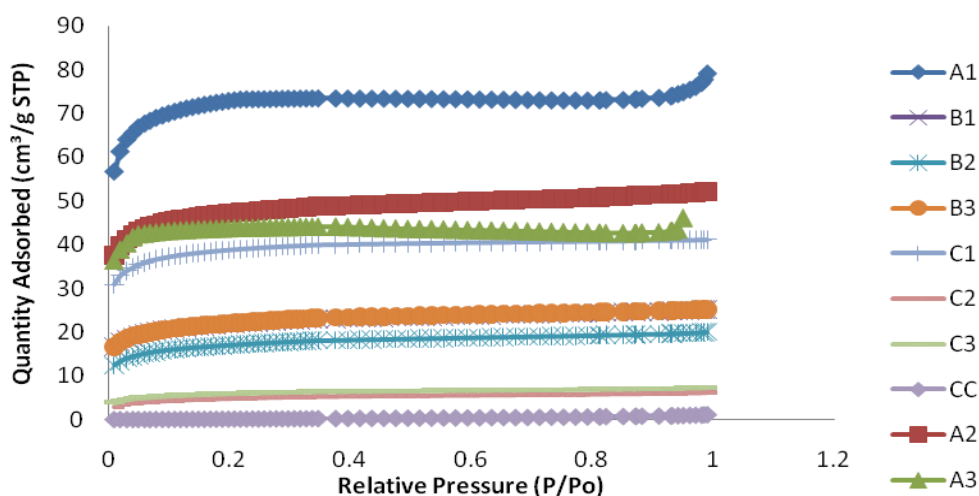


Figure-2
N2 - Adsorption isotherm for the samples at 77K

The total pore volume ranged from 0.015 to 0.12 cm³g⁻¹ (table 2). Most isotherms have been shown to conform to one of the five types of IUPAC classification¹³. Figure 2 showed that, samples A1, A2, A3, B1, B2, B3 and C1 conform to type 1 isotherm. This indicates that the carbons contain well structure micropores with pores dimensions < 20 Å. The interaction energy between the solid surface and the adsorbate molecules increased because of the overlapping potential energy force of the pores. As a result, an increased adsorption was observed especially at low concentration⁷. This may bring about complete pore fillings at low concentration. However at high concentration, adsorption is small and tending to a constant as shown by the plateau that is more or less parallel to the concentration axis. This is due to narrow pores that they can accommodate only a single molecular layer. The type 1 isotherm is as such common to Chemisorptions. The samples prepared at the higher temperatures of 900°C and 1000°C therefore exhibit type 1 isotherm indicating that they are overwhelmingly microporous and there is a near absence of mesopores in them. It was also observed that samples C2 and

C3 conform to type II isotherm. These samples were prepared at a relatively lower temperature of 800°C and activated at a relatively low resident time of 30 and 15 minutes respectively. The conformation to type II isotherm is an indication that samples C2 and C3 are nonporous, where the surface is whole external. This explains why the BET surface areas are relatively low. The inflection point or kneel of the isotherm usually mark the point of completion of monolayer adsorption. Monolayer is formed when the solid reaches saturation¹⁸. Increasing the relative pressure lead to additional layer of adsorbate until the number of adsorbed layer becomes infinite. The carbonized sample, (CC), exhibit type III isotherm as shown in Figure 2. The type III also corresponds to nonporous or highly macroporous adsorbent. This, again, accounted for the very low BET surface area of 3 gm⁻². The isotherm is convex shaped relative to the concentration axis. The convexity continued throughout the isotherm because the net heat associated with the adsorption is very small.

The effects of variation of temperature and time of activation on the surface area of the carbons are depicted in figures 3 and 4.

Temperature and time of activation which are key factors in the preparation steps were varied to monitor the pore development in the carbon and in order to optimize the conditions for the preparation of the product. The trend in pore development in the precursor is such that the tiny micropores formed increased in number with the time of activation increased from 15 to 30 minutes. The very numerous pores formed collapse to form larger ones of mesopores as the time was increased to 45 minutes. These developments are reflected by the surface areas (table 2). It is clear that as the temperature rose to 900°C, the mesopores collapse back to form once again micropores, the number of which increased with time of activation and temperature.

The FT-IR has been employed for the investigation of activated carbon surface chemistry. The FT-IR spectra of the activated carbons and that of carbonized carbon (CC) revealed complex surface as by presence of several peaks. Tables 3, 4 and 5 present the peaks wavelength and possible assigned functional groups. The carbonized samples exhibit more peaks than that found in the activated carbons. This is an indication that some reorganization of the surface oxides on subjection to activation at the high temperature occurred. The absorption peak observed around 3400 cm^{-1} on the surface of carbonized sample on the activated carbon indicates the presence O-H stretching vibration in the carbons¹⁹. The peak at 2922.25 and 2855.71 were assigned to C-H stretching of aliphatic compounds¹⁹.

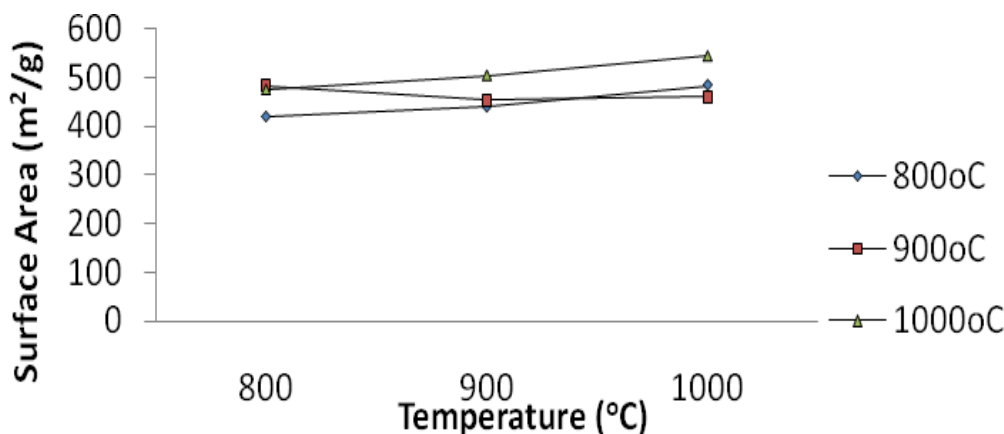


Figure-3
Effects of temperature on surface area of activated carbons

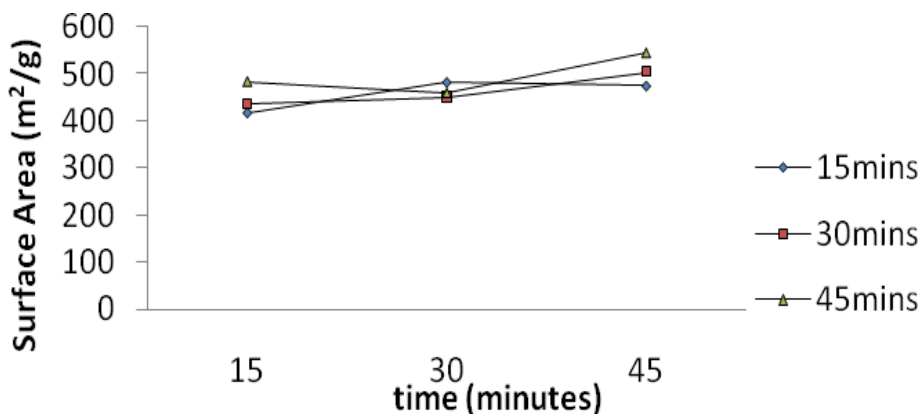


Figure-4
Effects of activation time on surface area of activated carbons

The O-H stretching of carboxylic acid in the 23700 region disappeared after activation at high temperature of 800 °C while the C=O stretching of carboxylic acids around 1594 cm⁻¹ shifted to 1629.9 after activation. The peaks around 1400, a finger print of C-O stretching and peaks at 1200 and 1000 which are finger print of and O-H bending of alcohol and carboxylic acid also

underwent various shifts. The band around 3400 was present in all the samples while the bands around 3700 which can be ascribed to O-H of phenol were not found on samples A1, A2, B1 and C1. The band at 2922.25 for C-H stretching was lost in sample A1, B1 and B3.

Table-3
Peak assignments of functional group of samples A1, A2 and A3

Peak positions/samples				Possible assignments	References
CC	A1	A2	A3		
3431.48	3418.94	3436.3	3436.3	O-H str	20
2922.25	-	-	2923.22	C-H str	21, 26
2855.71	-	2882.71	-		
2372.52	2360.95	2357.09	2374.45	C=O str of carboxylic acids	20, 22
-	-	-	2082.23		
-	1646.3	1694.52	1634.73	C=O str of carbonyl group	23,24
1594.22	-	1547.93	-		
1430.26	1409.05	-	1423.51	C-O str and OH bending of alcohol and carboxylic acids	19,24
1214.23	-	1221.95	-		
1035.81	-	1069.56	1025.2		
803.38	995.3	-	-	Bending mode of aromatic compounds	23,24,25

Table-4
Table 3; Peak assignments of functional group of samples B1, B2 and B3

Peak positions			Possible assignments	References
B1	B2	B3		
3415.08	3417.01	3443.05	O-H str	20
-	-	-	C-H str	21, 26
-	2882.71	-		
-	-	2341.66	C=O str of carboxylic acids	20
2619.42	2603.99	-		
1641.48	1642.44	1650.16	C=O str of carbonyl group	23, 24
1566.25	1561.43	1556.61		
1406.15	1409.05	1343.46	C-O str and OH bending of alcohol and carboxylic acids	24
-	1227.73;	1220.98		
1004.95	1003.9	-		
-	-	990.48	Bending mode of aromatic compounds	23, 24
833.28	830.38	828.45		

Table-5
Table 3; Peak assignments of functional group of samples C1, C2 and C 3

Peak positions			Possible assignments	References
C1	C2	C3		
3447.87	3426.66	3423.76	O-H str	20
2923.22	2924.18	2925.15	C-H str	21, 26
2853.78	-	-		
-	-	2370.59	C=O str of carboxylic acids	20
-	-	2082.23		
1629.9	1634.73	-	C=O str of carbonyl group	23,24
-	-	1566.25		
1408.08	1407.12	1402.3	C-O str and OH bending of alcohol and carboxylic acids	24
1199.76	1248.95	-		
1011.7	1002.05	1003.98		
830.38	825.56	827.49		
			Bending mode of aromatic compounds	23,24

The band 2922.25 was also lost in sample B2. The C=O of carboxylic acids peak split into two in samples A3 and C3, shifted to 2619.42 in B1, 2603.99 in B2 but lost completely in sample C1. It can be observed that dominant activities on the surface occurs with C=O stretching of carboxylic acids and carbonyl group. The C=O stretching band of carbonyl group around 1629.9 was introduced by the activation process and found in all the samples but shifted to 1566.25 in sample C3. In all, similar functional groups were observed on the surface of the activated carbons due to their exposure to similar chemical treatment and narrow variation of temperature of activation.

Conclusion

A low cost and high quality active carbon should be of high surface area, produced at low temperature¹⁰. Among the prepared carbons, sample A1 has the highest surface area. However, sample C1 has slightly higher bulk density, relatively high surface area (127 m²/g) and a percent yield of twice that of sample A1. It also has the advantage of being prepared at lower temperature than sample A1 and therefore more feasible to produce on industrial scale. It is therefore recommended that the best condition for the preparation of activated carbon for adsorption from palm kernel shell using KOH as activating agent be 800°C for 45 minutes, taking into cognizance the feasibility of scaling up production. This meets up with the needed requirement of lower temperature of production, high bulk density and high % yield that are key factors in large scale production and usage. Further increase in temperature and time of activation was detrimental because it led to highly reduced yield with little increase in the surface area of the carbon. Significantly reduced yield of product was observed for activation temperature beyond 800°C.

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