



The Quality of Activated Carbon Produced from Enugu Sub-Bituminous Coal by Chemical Activation

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Abstract In this study, the quality characteristics of activated carbon produced from sub-bituminous coal were investigated. Two sets of experiment were designed to ensure effective study of this work. Chemical activation method was employed. The effects of the process variables such as chemical reagents, activation temperature, impregnation ratio and carbonization temperature were investigated to optimize these parameters. The assessed adsorptive characteristics of surface area, iodine number, porosity and fixed carbon content etc recorded high values for most of the activating agents. The KOH activating agent displayed an outstanding performance over others in all the conditions investigated. Acidic surface groups were established by FTIR technique while SEM analysis shows increased pore volume distribution and improved surface activity for all the samples of activated carbon.

Keywords coal, activated carbon

1. Introduction

Activated carbon (AC) is a generic term for a family of highly porous carbonaceous materials, none of which can be characterized by a structural formula or by a chemical analysis [1]. AC is a microcrystalline, non-graphitic form of carbon with porous structure that has been processed to develop its internal porosity [2]. The different types of activated carbon can be distinguished from one another only by reference to certain physical, chemical and adsorptive properties. Some important physical properties are the number and size distribution of the pores, bulk density, dry impact hardness, abrasion resistance, particle size distribution and moisture content. AC is characterized by a vast system of pores of molecular size within the carbon particles. The pores which are classified as micropores, mesopores and macropores may be cylindrical or rectangular in shapes [3]. Each of the pores has its specific function during adsorption. The most important are the micropores, located deep under the surface that provides the largest surface area for adsorption processes. Micropores provide the adsorptive sites for small molecules and are also the primary sites of chemisorptions since they are sites with the highest adsorption energies [4]. The mesopores also known as transitional pores serve two functions. Besides their significant contribution to the adsorption of larger molecules, they act as transport arteries to the micropores. The large macropores do not contribute greatly to surface area and the amount of material adsorbed in them is insignificant. Their main function is to allow adsorbate molecules to pass rapidly to the deeper pores [5].

The chemical property assessment of AC is based on the ability of the carbon to adsorb different kinds of adsorbates from gaseous phase (benzene, carbon tetrachloride and nitrogen) and aqueous phase (such as iodine, methylene blue, molasses and phenol). Due to high degree of microporosity, AC possesses high internal surface area which is the basis of its adsorptive property rating. However, the degree of utilization of the available surface area in adsorption



is mostly affected by the molecular architecture of the adsorbate vis-a-viz the pore dimensions of the AC [6-7]. The ash content, ash composition, nature of the surface chemistry and pH of the carbon are other important chemical properties of the activated carbon. All AC possess not only carbon but also small amounts of chemically bonded oxygen and hydrogen in the form of various functional groups that usually gives acidic character to solid carbon plus mineral matters which is indicated as ash or residue after combustion [2-8]. The ash which reflects the purity of carbon emanates mainly from the formation of oxides of silicon, aluminum and iron during carbonization. The deposited ash covers the surface and thereby blocking and reducing the dimensions of the pores. In addition, it will lead to the alteration of the other surface characteristics especially the hydrophilic/hydrophobic existing balance and hence the selectivity trends of the carbon product and its intrinsic adsorptive behaviour [5].

In general, the factors that influence the adsorptive characteristics of AC from aqueous solution are temperature, pH value of the solution, chemical nature and concentration of the species present as well as its particle-size distribution [1]. For the uptake of adsorbates, they must be transported to the internal surfaces of the pores. Therefore, the adsorption time will be influenced by the duration of the diffusion path while the kinetics of adsorption increases with decrease in particle size.

AC is a very versatile adsorbent with respect to its interactions with various organic and inorganic compounds. However, carbon is generally a non-polar material and therefore, it adsorbs organic compounds in preference to polar inorganic species. The adsorption of organic species onto carbon involves a simple mechanisms comprising terms of a balance between hydrogen-bonding forces in solution and Van der Waals attraction between the carbon surface and the organic compounds [1-5]. As the adsorption by AC is seriously affected by the solubility power of solvent for solute, application involving organic materials will be less attractive when the adsorbate has high molecular mass, low polarity and a low degree of ionization because these properties generally signify low aqueous solubility.

In chemical activation, both carbonization and activation are conducted simultaneously at the same temperature. It involves the impregnation of the carbonization material by mixing it with an excess amount of the given chemical, usually in the form of concentrated solution. Like the physical activation, the commonly used activating agents are KOH, NaOH, H_3PO_4 , $ZnCl_2$, H_2SO_4 , HCl, K_2CO_3 , Na_2CO_3 etc. The impregnation is conducted in such a way to ensure intimate contact between the reagent and the starting material. The combination leads to dehydration/erosion of the carbon skeleton and expansion or swelling of the interior canals of the existing structure with the resultant formation of porous structure. The degree of erosion of carbon structure and the nature of surface oxides that form on the carbon during activation depend mostly on the composition of the activating agent. Alkali compounds such as hydroxides and carbonates of potassium and sodium develop higher percentage of micropores, whereas transition metals and alkali-earth compounds (compounds) promote the formation of mesopores [3-5]. In this research work, the study is focused on the characterization of quality of AC produced from sub-bituminous coal obtained from Onyema mine, Enugu, Nigeria by chemical methods of activation.

2. Materials and Method

2.1. Pre-activation Treatment

The coal sample obtained from Onyema mine in Enugu was ground with a rock Crusher/grinder for about 10 minutes. The ground coal was dried in an oven at 100 °C for 24 hours and later sieved to a mesh size 500 μ m.

2.2. Chemical Activation

Chemical activation process was carried out in two steps, namely activation followed by carbonization. Activation studies were conducted at low temperature and high temperature conditions as described in experiments 1 and 2. The details are outlined below.

2.3 Experiment 1

The pre-treated coal sample was activated by mixing and heating it with activating agent at low temperatures. The activating agents employed were KOH, NaOH, $ZnCl_2$ and H_3PO_4 .



The activating agent and the precursor were measured out and mixed together in a given impregnation ratio as 4/1, 2/1 and 1/1. The resulting mixture was stirred continuously in each activating agent of 50% solution at a temperature of 80 °C for 30 minutes. The activated sample was later filtered by vacuum and dried in oven at 100 °C for 24 hours. After the activation process, the sample was subjected to carbonization. The sample was placed in a programmable tube furnace. The furnace was programmed in such a way that the heating rate of 5 °C per minute was maintained. During carbonization, nitrogen was introduced to the system at a flow rate of 1000 cm³/min and the heating continued to a temperature of 800 °C. The heating was maintained constant at this temperature for 2 hours. Later, the furnace and its content were allowed to cool to ambient temperature.

The carbonized sample was added 0.5N HCl and heated at 85 °C for a period of 30 minutes accompanied with constant stirring. After filtration, the sample was rinsed with distilled water several times until the pH of the activated carbon-distilled water mixture has exceeded 6. Then, the sample of activated carbon obtained was dried at 100 °C for 24 hours in an oven. Finally, it was subjected to characterization process in which the value of the following properties such as surface area, iodine value, pore volume, porosity etc were determined.

2.4. Experiment 2

In this experiment, reference was made to the first experiment with regards to the characteristic properties of the activated carbon obtained from different activating agents vis-a-viz their impregnation ratio. As observed, the activating agents of H₃PO₄ and ZnCl₂ produced lower values of the parameters assessed irrespective of the impregnation ratio employed. Therefore, they are considered unsuitable for this process and subsequently eliminated for further works.

A measured amount of 2g of pre-treated coal sample was put into a volumetric flask and mixed with 50% activating agent solution of KOH at a ratio of 1/1. A condenser was mounted to the flask to prevent evaporation. The mixture was boiled at a temperature of 150 °C for 5 hours in an oil bath. At the end of this period, the mixture was cooled to ambient temperature, filtered by vacuum and dried at 100 °C for 24 hours in an oven. A similar procedure of carbonization as described in first experiment was adopted as follows:

The sample was placed in a tube furnace where N₂ was introduced to the system at a flow rate of 1000cm³/min. The heating of the furnace was maintained at 5 °C per minute. At a final temperature of 600 °C, 700 °C, 800 °C, 900 °C, and 1000 °C, the heating was allowed to remain constant for 2 hours. At the end of this period, the furnace was left to cool to room temperature. The carbonized sample was mixed with 250cm³ of 0.5N HCl and the mixture was subjected to heating accompanied by constant stirring at 85 °C for 30 minutes. Later, it was filtered by vacuum and the sample of activated carbon obtained rinsed with distilled water several times until pH of the activated carbon-distilled water mixture has exceeded 6. The activated carbon obtained was dried in an oven at 100 °C for 24 hours. Thereafter, the characterized properties of surface area, iodine value, pore volume, porosity etc of the AC evaluated. The same procedure was repeated using activating agent of sodium hydroxide NaOH.

3. Results and Discussion

Table 3: Properties of Raw Coal

Parameter	Moisture Content (%)	Ash Content (%)	Bulk Density (g/cm ³)	pH	Iodine Value (mg/g)	Surface area (m ² /g)	Volatile Matter (%)	Fixed Carbon (%)	Pore Volume (cm ³ /g)	Porosity
Value	9.86	15.63	0.798	6.8	138	206	33.71	41.80	0.111	0.441

Table 3.1A: Properties of Activated Carbon at varying impregnation ratio of activating agents

Parameters	NaOH 4/1	NaOH 2/1	NaOH 1/1	KOH 4/1	KOH 2/1	KOH 1/1
Moisture content (%)	1.3475	1.7403	2.231	1.3261	1.536	2.175



Ash content (%)	15.2	15.0	13.2	14.8	14.2	13.5
Bulk Density (g/ml)	0.3317	0.5050	0.5905	0.3575	0.6518	0.6683
pH	7.2	7.0	7.0	7.1	6.9	7.0
Iodine number (mg/g)	1419.7	962.57	687.64	1711.74	1013.69	743.67
Surface Area (m ² /g)	1530.4	1030.0	718.2	1862.3	1138.1	850.0
Volatile matter (%)	18.4	19.5	20.2	19.2	20.5	21.7
Fixed carbon (%)	65.05	63.76	64.37	64.67	63.76	62.63
Pore Volume (cm ³ /g)	0.560	0.540	0.479	0.697	0.589	0.516
Porosity	0.8174	0.8063	0.7617	0.8737	0.8688	0.8149

Table 3.1B: Properties of Activated Carbon at varying impregnation ratio of activating agents

Parameters	ZnCl ₂	ZnCl ₂	ZnCl ₂	H ₃ PO ₄	H ₃ PO ₄	H ₃ PO ₄
	4/1	2/1	1/1	4/1	2/1	1/1
Moisture content (%)	2.536	3.318	3.317	3.348	4.361	4.362
Ash content (%)	14.0	13.3	13.1	13.8	13.4	13.6
Bulk Density (g/ml)	0.5820	0.5800	0.5799	0.6210	0.6521	0.6681
pH	7.1	6.9	7.2	7.1	6.8	6.9
Iodine number (mg/g)	455.66	423.68	327.64	416.28	306.1	281.0
Surface Area (m ² /g)	522.0	490.0	358.0	442.0	352.0	296.0
Volatile matter (%)	25.1	26.3	27.4	26.7	27.3	30.2
Fixed carbon (%)	58.36	57.08	56.18	56.15	54.40	52.84
Pore Volume (cm ³ /g)	0.448	0.432	0.338	0.408	0.388	0.307
Porosity	0.7714	0.7216	0.6243	0.6132	0.5961	0.5832

Table 3.2A: Properties of Activated Carbon at varying temperatures of activation with KOH

Parameters	KOH				
	600 °C	700 °C	800 °C	900 °C	1000 °C
Moisture content (%)	2.66	2.38	2.18	2.01	1.52
Ash content (%)	11.9	12.8	13.2	15.2	18.9
Bulk Density (g/ml)	0.688	0.6894	0.6683	0.6241	0.596
pH	6.9	6.8	6.8	7.0	7.0
Iodine number (mg/g)	268.1	420.6	743.66	788.6	710.56
Surface Area (m ² /g)	394.6	566.6	850.1	992.1	842.1
Volatile matter (%)	26.00	24.33	20.30	18.60	17.80
Fixed carbon (%)	59.44	60.59	64.32	64.19	61.78
Pore Volume (cm ³ /g)	0.468	0.509	0.516	0.553	0.512
Porosity	0.681	0.724	0.8149	0.877	0.7841

Table 3.2B: Properties of Activated Carbon at varying temperatures of activation with NaOH

Parameters	NaOH				
	600 °C	700 °C	800 °C	900 °C	1000 °C
Moisture content (%)	2.79	2.52	2.28	1.95	1.81
Ash content (%)	12.61	13.4	13.5	14.61	16.36
Bulk Density (g/ml)	0.661	0.621	0.5905	0.582	0.524
pH	7.1	6.8	6.9	6.8	6.5
Iodine number (mg/g)	402.2	542.1	687.7	691.3	606.2
Surface Area (m ² /g)	532.0	668.3	718.7	828.1	700.2
Volatile matter (%)	25.22	24.20	21.50	19.16	19.00
Fixed carbon (%)	59.38	59.88	62.72	64.28	62.8
Pore Volume (cm ³ /g)	0.409	0.423	0.478	0.50	0.462
Porosity	0.508	0.532	0.762	0.801	0.712



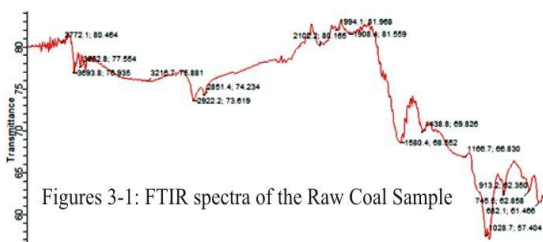
The results of characteristic properties of the raw coal and its derivable AC as obtained in this work are presented in Tables 3.0 – 3.4. The results revealed that most of the adsorptive properties of the activated carbon recorded exponential increases from those of the precursor material. The analysis shows that the least percentage increase of 44 percent was obtained in surface area of H_3PO_4 activating agent. While the iodine value and pore volume produced maximum increase of 143mg/g from 138mg/g and $0.196 \text{ cm}^3/\text{g}$ from $0.111 \text{ cm}^3/\text{g}$ respectively. Similarly all other parameters of AC investigated produced the same progressive trend of substantial increment when compared to the precursor. Also, the observation shows a variation in properties from one method of activation to another. These variations were dictated by the process factors involved in the carbonization and activation such as the nature of the activating agent, temperature of carbonization and impregnation ratio of activating agent to precursor material, among others. In the first experiment, the effects of the nature of the activating agent and the impregnation ratio were studied. The results obtained revealed that the value of these properties increased with the impregnation ratio for all the activating agents. For instance, the surface area from NaOH-activating agent increased from 718 to $1530 \text{ m}^2/\text{g}$ from the ratio of 1/1 to 4/1. Similarly, iodine value for KOH- activating agent recorded 744 and 1712 mg/g at the same range of impregnation ratio mentioned above. The same trend was obtained for other characteristics of pore volume, moisture content, bulk density, porosity etc. The effects of these activating agents vis-a-viz their impregnation ratio, show that KOH recorded the best performance in adsorptive rating followed by NaOH and then ZnCl_2 while H_3PO_4 takes the ray. This observation is in agreement with the work of Fierro and co-worker [9] who reported that BET surface areas of activated carbon produced from Kraft lignin by chemical activation using KOH is higher that of NaOH-activating agent. These findings were also corroborated by other researchers including Amir [10] and Lillo-Roden and Co-workers [11] in their individual works on the comparative analysis of various AC produced from different activating agents. The oxidizing action of these activating agents leads to discrete removal of successive layers of atoms from the carbon matrix. The oxidation selectively erodes the surfaces and creates spaces between the elementary crystallites.

The less organized, loosely bound carbonaceous materials are oxidized and cleared with the creation of channels, together with fissures within and parallel to graphitic planes, constituting the porous structure with large internal surface areas [10-12]. The mode of interaction of acid with coal was reported [6] to be mainly through oxygen functional groups leading to dehydrogenation and repolymerization reactions with carbon structure. On the other hand, KOH and NaOH, acting as bases, involve the process of both dehydrogenating and oxidation reactions. According to Azargohar [3], the overall stoichiometric reaction of KOH involves direct attack on the carbon centre resulting in more aliphatic chains, hence higher porosity and surface area, among others. In this first experiment, the values of the properties of this adsorbent such as surface area, porosity, iodine value, pore volume etc obtained from using ZnCl_2 and H_3PO_4 were low in comparison to those of KOH and NaOH. Consequently, further studies were shifted to the activating agents of KOH and NaOH.

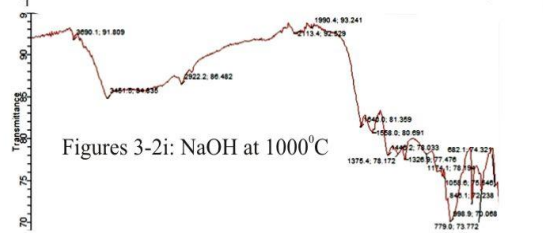
The second group of experiments was conducted to investigate the effects of activation variables and carbonization temperature on the quality of activated carbon at constant impregnation ratio of 1/1. The results as contained in Tables 3.3- 3.4 showed that the temperature of activation has a considerable influence on all the properties of the AC as produced. In both activating agents, for instance, the surface area obtained increases with temperature from 600 °C to a maximum value at 900 °C. Thereafter, it decreased after exceeding the temperature of 900 °C. Thus, the optimum temperature is established at the neighbourhood of 900 °C. In the same vein, the porosity of AC from NaOH-activating agent increased from 0.508 to 0.801 at a temperature range from 600 °C to 900 °C and later decreased to 0.702 at 1000 °C. While the surface area of AC from KOH-activating agent recorded a substantial increment of 150% and a decrease of about 15% at the above temperature ranges respectively. The same pattern of variation was obtained for all the properties investigated. These observations are in consonance with the results reported for AC produced from coconut shells and cattle bones using chemical activation [13]. In this work, the surface area increases with temperature up to a maximum of 900 °C and thereafter decreased beyond 900 °C. These findings may be due to the volatilization of melted ashes that results in opening of blocked pore channels which subsequently lead to the creation of larger amount of micropores and mesopores [10]. But the reduction in value



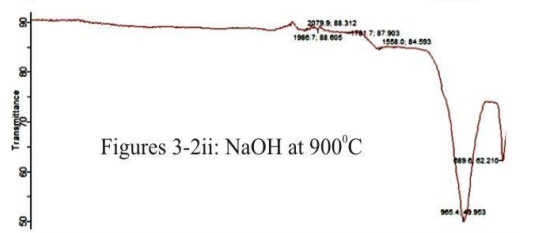
after exceeding the optimum temperature of 900 °C is an indication of the occurrence of structural deformation of the already formed AC. Thus, structural deformation might have risen from the significant melting of the ash fraction in char at a temperature greater than 900 °C that plausibly blocked the pores in the char when the char was cooled to ambient temperature. This may have been enhanced by the collapse of the pore walls that created resistance to diffusion of the molten ash out of the pore channels of the AC [5].



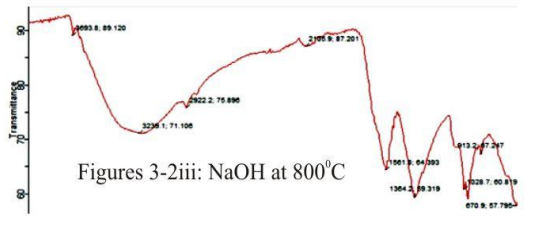
Figures 3-1: FTIR spectra of the Raw Coal Sample



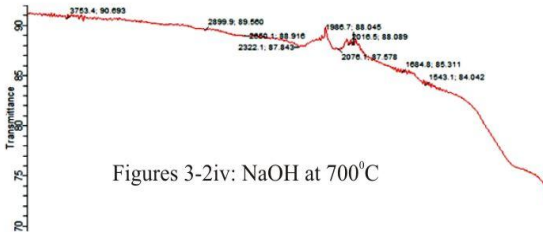
Figures 3-2i: NaOH at 1000°C



Figures 3-2ii: NaOH at 900°C



Figures 3-2iii: NaOH at 800°C



Figures 3-2iv: NaOH at 700°C

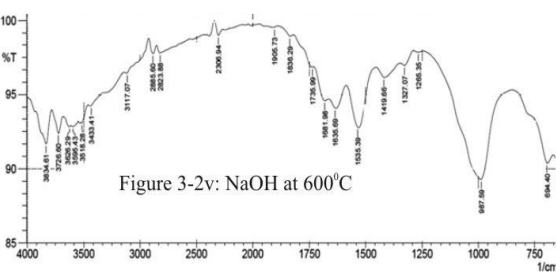
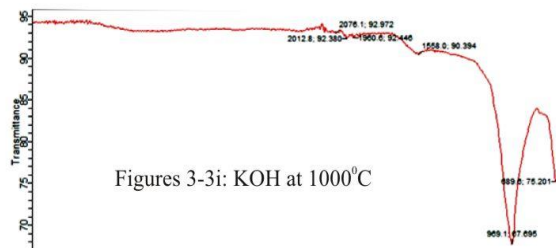
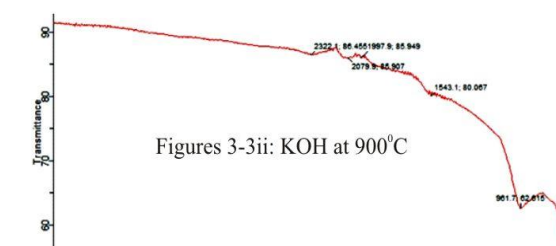


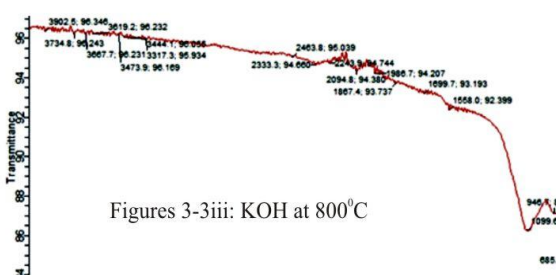
Figure 3-2v: NaOH at 600°C



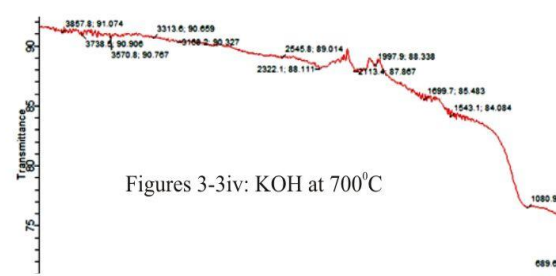
Figures 3-3i: KOH at 1000°C



Figures 3-3ii: KOH at 900°C



Figures 3-3iii: KOH at 800°C



Figures 3-3iv: KOH at 700°C

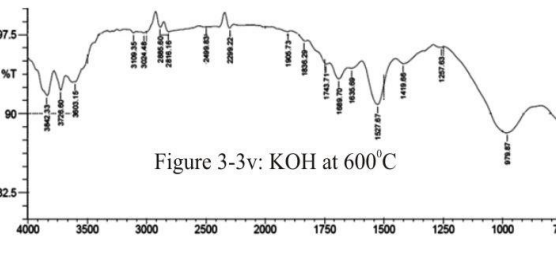


Figure 3-3v: KOH at 600°C

Generally, the AC obtained exhibited true densities which are higher than the precursor. The bulk density decreases with the concentration of the activating agents and with the temperature of activation. From Table 3.2B, the density of NaOH activated carbon at a temperature of 600 °C was recorded as 0.661 g/cm³. The value decreased to 0.524 g/cm³ at higher temperature of 1000 °C. In the same vein, the results in Table 3.2A revealed that at the impregnation ratio of 1/1, the KOH activated carbon produced a density of 0.668 g/cm³, while at the impregnation ratio 4/1, it decreased to 0.358 g/cm³. A similar trend of result was exhibited at various impregnation ratios for each of the activating agents investigated. The findings may be due to the devolatilization which increases with temperature until some of the species and their linkages in the impregnated sample attain their limit of thermal stability. This leads to structural expansion, widening and re-opening of existing pores and hence a decrease in true density [5-10]. After the release of lighter components and thorough oxidative action on the sample, the remaining solid matrix developed a relatively increased porosity thereby exhibiting increase in pore volume.

This invariably leads to a decrease in bulk density. The lower density leads to greater volume activity and thus a confirmation of better quality of the AC [2-7]. However, this observation is contrary to the work of Baba et al [6] that obtained reduced volume of activated carbon produced from the activating agents of KOH and H₃PO₄ on the pyrolysis of bituminous coal.

Ash is one of the impurities that form complexes on the surface of the AC leading to alteration in surface characteristics. In addition to the creation of hydrophilic-hydrophobic imbalance, it covers the existing pores and reduces the pore volume distributions [3-5]. From the results obtained, the effect of temperature and impregnation ratio of reagent indicates significant variation across the different activating agents. At high temperature, the ash content increases as observed in Tables 3.3 and 3.4, reaching a maximum value at 1000 °C for all the activating agents. In the same manner, the value of ash content increases with the impregnation ratio of activating agent, the least value obtained at 1/1 [Tables 3.1 A and 3.1B]. Generally, the result analysis shows that NaOH recorded the highest percentage ash content while the least comes from H₃PO₄. The yield of ash is also an indication of the high rate of devolatilization of carbonaceous materials of the coal which increases with temperature. However, the effect of ash may not have serious impact on the adsorptive properties of the activated carbon as much as other major determining factors of surface area, iodine value, pore volume etc maintained their standard values [5].

Fourier transform infrared (FTIR) analysis was conducted to analysis the functional groups of raw coal and activated carbon samples. The raw coal exhibited hydroxyl groups, including hydrogen bonding O – H stretching between 3770 – 3680 cm⁻¹, which is much less prevalent in all AC samples. The reduction in hydrogen bonding shows that the activating agents acted as dehydrating agents in reacting with the raw coal during the production of AC [14-15]. The intense band at the range of 2920 – 2850 cm⁻¹ in raw coal is assigned to asymmetric C – H stretching, which is reduced in the derived AC as shown in figures 3-2 – 3-3.

This observation is a true reflection of the confirmation that activation process removed a substantial amount of hydrogen. The stretching absorption band at 1680cm⁻¹ observed in the raw coal is associated with C = O group in configurations which indicates the presence of esters, carboxylic acid and conjugated ketonic structures. The band was virtually absent in the prepared AC, indicating that chemical activation broke many bonds in aliphatic and aromatic species and eliminated many volatile and light substances [14]. The peak at approximately 1440 – 1420 cm⁻¹ in the raw coal may be attributed to the presence of C – H asymmetric bending while the peaks at the range of 1580 – 1520cm⁻¹ obtained in AC with KOH and NaOH activating agents indicate the C = C stretching band and aromatic skeletal stretching vibration of lactone and carbonyl groups [8-16].

Most of the AC produced from different modes of activation exhibited similar IR spectroscopic features. In figures 3-2 and 3-3, the characteristic broad coal absorptions which were observed in precursor are virtually absent at the temperatures of activation above 700°C which is an indication of the complete carbonization of the samples. Also, the analysis of figures 3-2 and 3-3 reveals that the two activating agents of KOH and NaOH exhibited remarkable differences in FTIR spectra absorption at the activation temperatures of 800-1000 °C. At this temperature range, the AC produced with the activating agent of NaOH is characterized with various wave bands of different functional groups. There are high intensity H – bond and –OH stretching of carboxyl, phenol and alcohol vibration of 3500 –



3400cm^{-1} and C – H aliphatic stretching absorption from $2930 - 2910\text{cm}^{-1}$ [4]. The observation shows that the intensity of the spectra absorption bands increases with temperature for both NaOH and KOH reagents. But at $900\text{ }^{\circ}\text{C}$, similarities in spectra absorption occurred at $965 - 960\text{cm}^{-1}$. However, the intensity of these wave bands varies significantly. Contrary to the observation of Cuhadaroglu and Co-workers [8], KOH-activated carbon recorded higher intensity than its counterpart of NaOH-activated carbon, therefore producing a corroborative evidence of higher degree of activation in favour of KOH. But the main differences between the carbonization products are observed from the characteristics of the band of C – H aromatic stretching. From figures 3-2 and 3-3, the results show that NaOH- activated carbon samples have much higher content to C – H than the KOH-activated ones. Some weak bands also appeared in the range of $900 - 600\text{cm}^{-1}$ which are associated with out of plane bending mode of C – H and O – H groups.

Scanning electron microscopy (SEM) was carried out to investigate the variation in the surface of derived carbon samples. Figure 3-4 shows the SEM images of the microstructure of the raw coal while figures 3-5 to 3-6 are those of the AC samples obtained from different modes of activation. From the analysis, the surface topology of the precursor differed significantly from the activated samples. The surfaces of the raw coal were fairly smooth with few cracks or voids, but in activated carbon, the external surfaces were characterized by irregular cavities and pores. Thus, the activation process produced many large pores of varying shapes and sizes at its entire surface.

In this experiment, the nature of the pores was observed to depend on the temperature of activation as depicted in figures 3-4 to 3-5. At the temperature of $600 - 1000\text{ }^{\circ}\text{C}$, the pore channels become wider and the pore walls become thinner as the temperature increases. This may be due to increase in the burn-off of the volatile substances. The more organic matter is removed from the activated carbon, the wider and thinner will be the pore channels and the walls respectively. The well developed pores had led to increased surface area and pores structure of the derived activated samples. The intensity of porosity is much higher at high temperatures of carbonization. Hence, the observation reveals that the carbonization temperature of $1000\text{ }^{\circ}\text{C}$ seemed to produce the climax of pore development. The reaction rate between the activating agent of KOH (or NaOH) and carbonaceous material of the precursor increases with temperature and thus leading to the formation of well development pores at higher temperatures. According to Zawawi and Co-workers [17], the pore development may also be caused by the breakdown of some materials in the precursor due to thermal expansion during the activation stages.

4. Conclusion

The study revealed that the characteristic quality of the AC produced from this sub-bituminous coal depends on the nature of the activating agent, impregnation ratio, carbonization temperature etc. The assessed characteristics of surface area, pore volume, porosity, fixed carbon among others recorded high values for most of the activating agents. The analysis shows that KOH produced the best performance in all the conditions investigated while H_3PO_4 takes the ray. An optimum temperature of $900\text{ }^{\circ}\text{C}$ was established for all the reagents. SEM micrograph of the AC samples indicates higher population of pores with wider size distribution which increases with the temperature of carbonization.

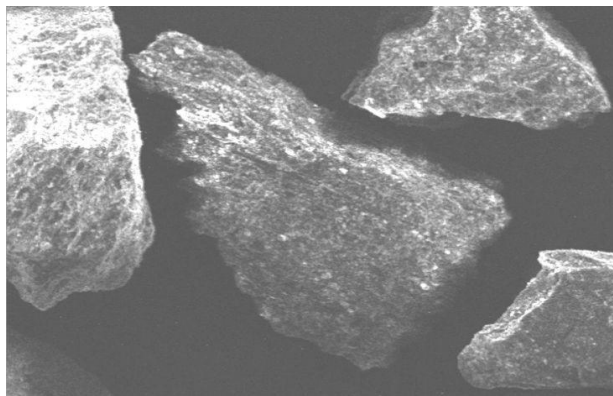


Figure 3-4: SEM of Raw Coal Sample

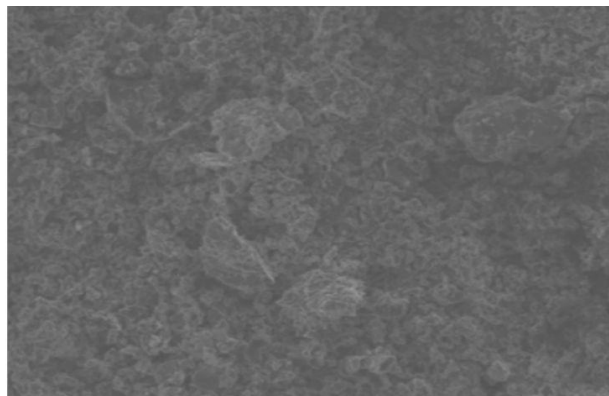


Figure 3-5i: SEM of NaOH - activated at 1000°C

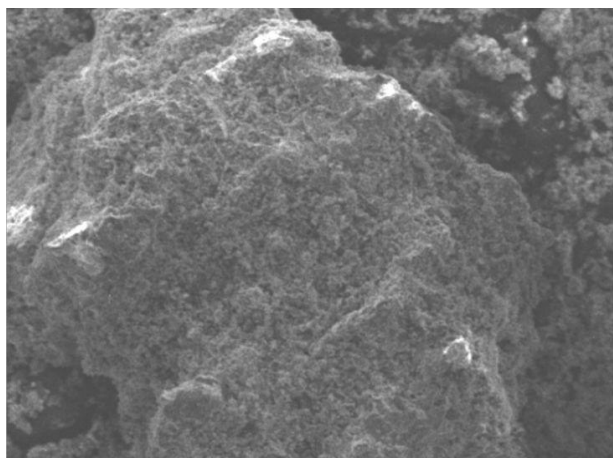


Figure 3-5ii: SEM of NaOH - activated at 900°C

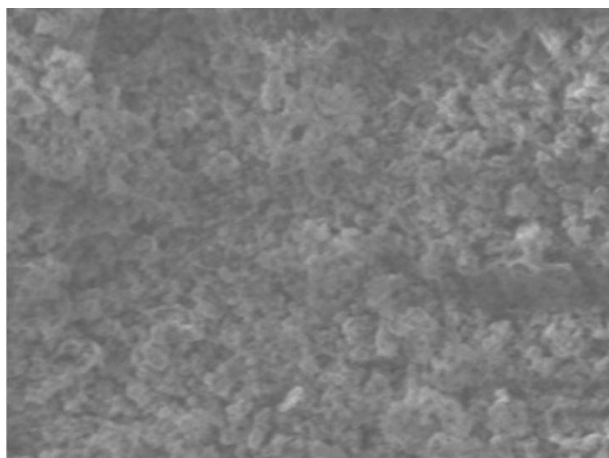


Figure 3-5iii: SEM of NaOH - activated at 800°C

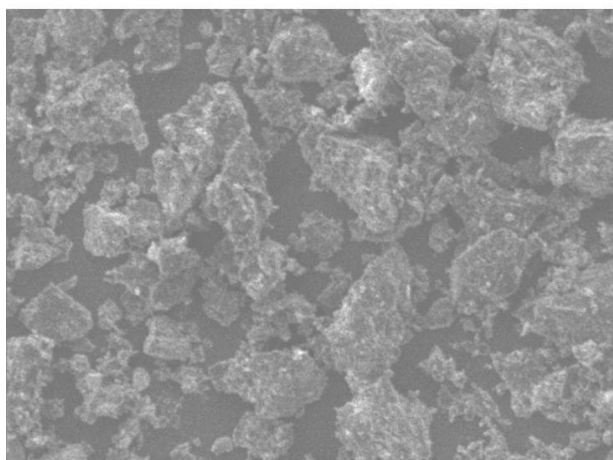


Figure 3-5iv: SEM of NaOH - activated at 700°C

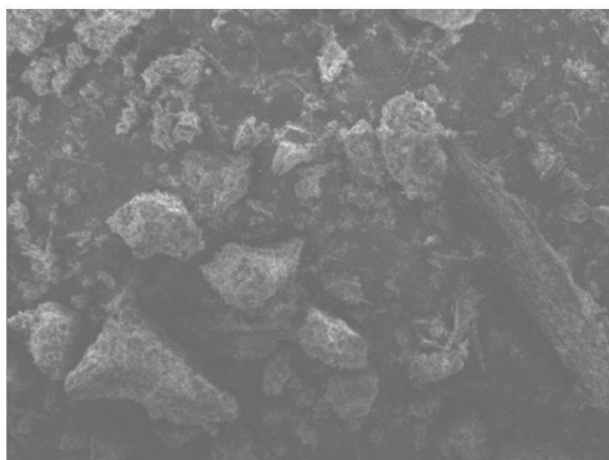


Figure 3-5v: SEM of NaOH Activated at 600°C

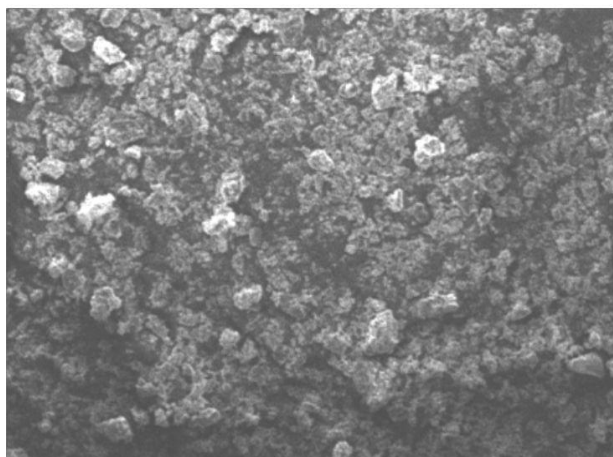


Figure 3-6i: SEM of KOH - activated at 1000°C

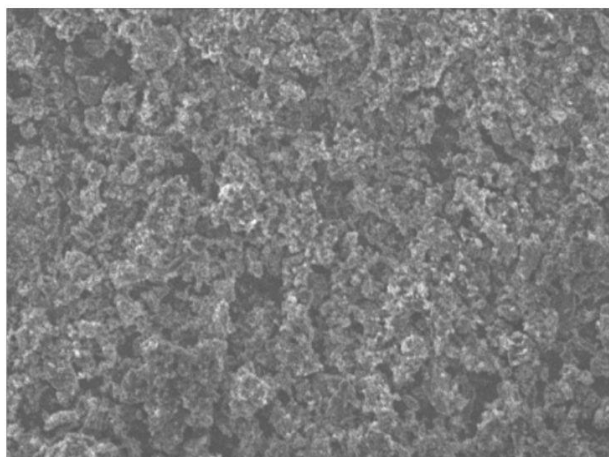


Figure 3-6ii: SEM of KOH - activated at 900°C



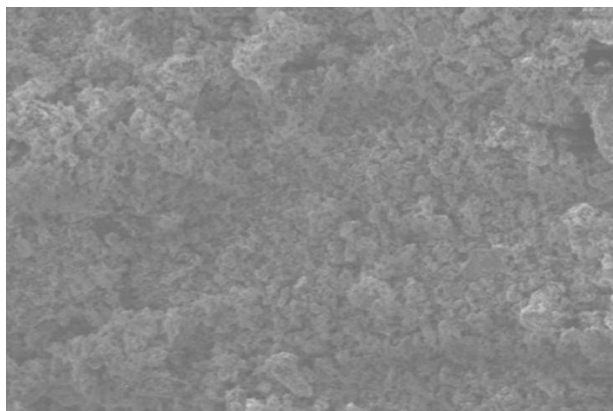


Figure 3-6iii: SEM of KOH-Activated at 800°C

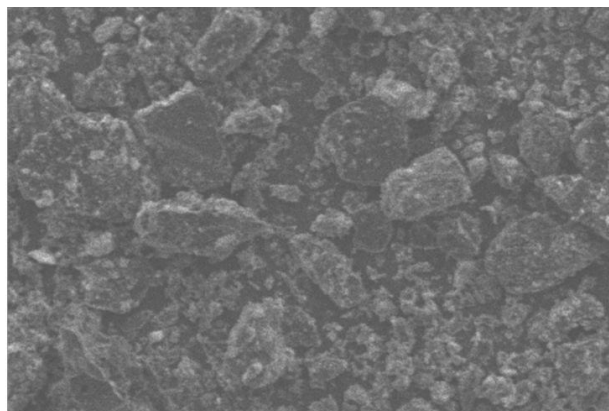


Figure 3-6iv: SEM of KOH-Activated at 700°C

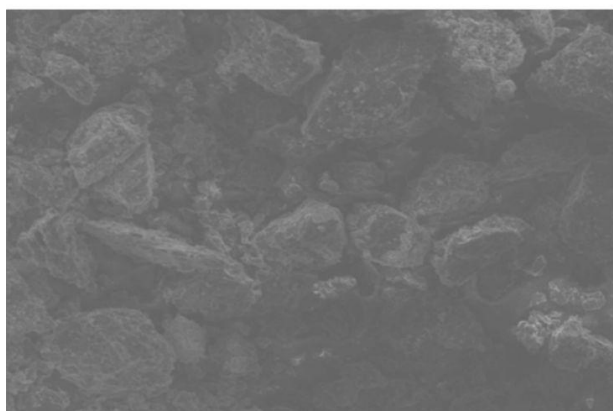


Figure 3-6v: SEM of KOH-Activated at 600°C

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