



EFFECTS OF TEMPERATURE AND IMPREGNATION RATIO OF ACTIVATING AGENT ON THE ACTIVATED CARBON PRODUCED FROM ENUGU SUB-BITUMINOUS COAL

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Abstract The effects of temperature and impregnation ratio of activating agents on the quality of activated carbons produced from Enugu Coal were investigated. For the impregnation ratio, the preheated coal sample was combined with given activating agent at various impregnation ratios and carbonized at 900⁰C for a specific time interval. While in temperature analysis, the impregnation ratio was maintained constant and the carbonization conducted at various temperatures. The activated carbons produced were characterized in terms of iodine value, pore volume, surface area, porosity, pH, fixed carbon, ash content and moisture content. The results revealed that the values of each of these adsorptive properties vary with temperature and also with the impregnation ratio. The optimum adsorptive characteristics of both activating agents were recorded at the temperature of 900⁰C. The KOH exhibited an outstanding performance over others in all the conditions monitored. FTIR test indicated significant reduction in surface acidic functional groups leading to the establishment of surface basicity on the synthesized activated carbon.

Keywords: Activated Carbon, Coal, Carbonization, Activation, Fourier Transmittance Infrared Spectroscopy

1. Introduction

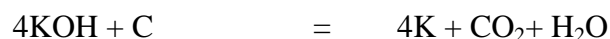
In the recent years, activated carbon (AC) has attracted global attention in its production and utilization. AC, a specially modified adsorbent with large porous surface area, effective pore distribution network, desirable surface reactivity and high mechanical strength has wide range of applications in the treatment of domestic and industrial waste waters, production of refined sugar, decolourization of petroleum products, purification of vegetable oils, solvent and metal recovery, catalysis, remediation of contaminated soil as well as in pharmaceutical industries (Foo et al, 2009; Echegi, 2017). The physical and chemical characteristic of AC lies on the selection of

precursors, activation method employed and nature of activating agent.

The AC can be produced from any carbonaceous materials such as various ranks of coal, sugar, bones, petroleum heavy oils as well as wood, sawdust, rice husk, corn cobs, coconut shells etc. In all these, coal has been found to be the most favoured precursor based on the differences in composition and organic matter which modify porosities, textural and surface properties of the AC (Rafsanjanj et al, 2013). The two basic methods of activation comprising physical and chemical activation produce AC of high adsorptive capacities but with distinct characteristics in quality assessment. In physical activation, the first step which is carbonization is conducted in an oxygen free

environment at temperature range of 400⁰ – 900⁰C at a slow heating rate so as to optimize the yield of char (Cuhadaroglu et al, 2008; Echegi, 2017). Due to blockage of the pores by tars and the irregular arrangement of void spaces, the carbonized product exhibits a lower adsorptive capacity with a surface area of only several meter squared per gramme (Echegi et al 2018). Therefore, the char should be subjected to activation in order to increase porosity, create and widen the existing pores. According to Cuhadaroglu et al (2008) and Yusuf and Co-workers (2012), activation can either be physical process with the use CO₂, water vapour etc or a physio-chemical process employing activating agents such ZnCl₂, H₃PO₄, HCl, among others. Secondly, the operation should be conducted at relatively high temperatures. As reported by many researchers, activation temperature significantly affects the yield and adsorption capacity of AC with the optimum temperature ranging from 500⁰ – 800⁰C (Yusuf et al, 2012; Marsh et al, 2006). In chemical activation, both carbonization and activation processes are conducted simultaneously at specified temperature. It involves the impregnation of precursor-based material by mixing with an excess amount of activating agent such as KOH, NaOH, H₃PO₄, ZnCl₂, HCl, K₂CO₃, Na₂CO₃, etc. The nature of activating agent, impregnation ratio and temperature of activation are the key factors to the determination of the quality of AC in chemical activation. Alkali compound such as hydroxides or carbonates of potassium and sodium develop micropores while transition metals and alkali-earth compounds promote the formation of mesopores (Echegi, 2018). The ACs produced by ZnCl₂ activation are predominantly micro-porous, but with significant mesopores which increases with the impregnation ratio. However, its uses are restricted due to hazardous nature of the product. H₃PO₄ produces finer pores than ZnCl₂. In addition to recent improvement in acid recovery, it is widely applied to the activation of coal-based materials (Echegi, 2017). The KOH⁻ and NaOH⁻ activations

produce highly developed micropores AC with large surface area greater than 1000m²/g (Hesas et al, 2017). The development of porosity in KOH activation which is different from that of ZnCl₂ and H₃PO₄ may be attributed to the reduction of KOH to potassium by this spontaneous reaction (Ejikeme et al, 2015).



Then, this free metal potassium K, penetrates between the graphene layers of the carbon which leads to the expansion of the layers and erosion of structural network. Finally, with increasing temperature, the free metal is removed rapidly from the graphene layers thus creating porosity.

In this work, the effects of temperature and impregnation ratio of activating agents on the quality of AC produced from Enugu sub-bituminous coal were investigated. Chemical activation was employed due to the economy of the process in addition to the expected quality of AC product.

2. Materials and Method

2.1 Pretreatment of Coal

The coal sample sourced from Onyeama Mine in Enugu, Enugu State was ground with a rock crusher/grinder to a reduced particle sizes for 10 minutes. The ground coal was dried in an oven at 100⁰C for 24 hours and later sieved to a mesh size of 450µm.

2.2 Effect of Impregnation Ratio of Activating Agent on the Quality of AC Produced

The activating agent and the pretreated coal sample were measured out and mixed together in a given impregnation ratio of 4/1, 2/1 and 1/1 in a beaker. The resulting mixture was stirred continuously in each activating agent of 50% solution at a temperature of 80⁰C for 30 minutes. The sample was later filtered by vacuum and dried in oven at 100⁰C for 24 hours.

The impregnated sample was transferred to a heating crucible and placed in a programmeable tube furnace. The furnace was programmed in such a way that the heating rate of 5⁰C per minute was maintained. During this

carbonization, nitrogen was introduced to the system at a flow rate of 1000cm³/min and the heating continued to a temperature of 900⁰C. 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 85⁰C 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 100⁰C 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.

2.3 Effect of Temperature on the Quality of AC Produced

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 150⁰C for 5 hours in an oil bath. At the end of this period, the mixture was cooled to ambient temperature, filtered by vacuum and the dried at

100⁰C 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 5⁰C per minute. At a final temperature of 600⁰C, 700⁰C, 800⁰C, 900⁰C, and 1000⁰C, 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 85⁰C 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 100⁰C for 24 hours. Thereafter, the characterized properties of surface area, iodine value, pore volume, porosity, FTIR tests, pH, density, fixed carbon, ash content and moisture content of the AC were conducted and evaluated. The same procedure was repeated using activating agent of NaOH, ZnCl₂ and H₃PO₄. In the same vein, the raw coal sample was characterized as described above.

3 Results and Discussion

Table 1 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 2 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 Properties of Activated Carbon at varying impregnation ratio of activating agents

PARAMETERS	ZnCl ₂ 4/1	ZnCl ₂ 2/1	ZnCl ₂ 1/1	H ₃ PO ₄ 4/1	H ₃ PO ₄ 2/1	H ₃ PO ₄ 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

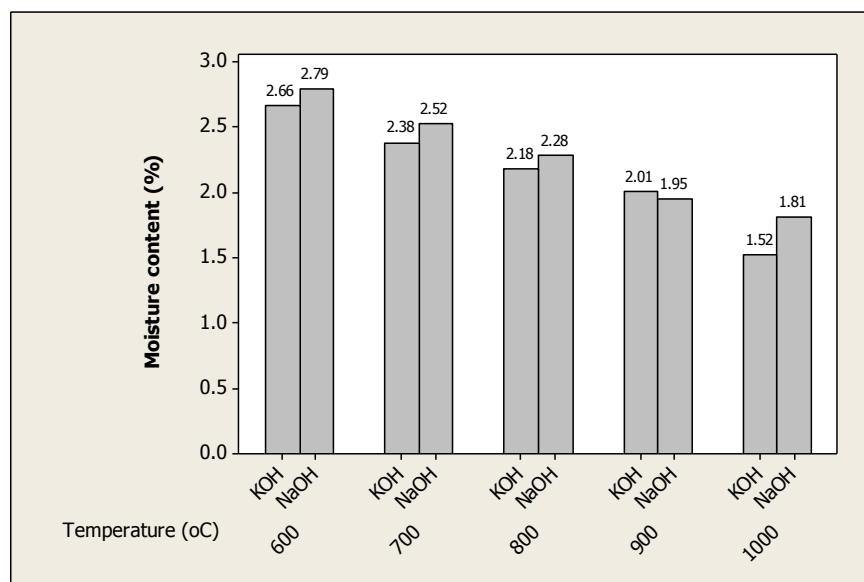


Figure 3.1A Effect of temperature on Moisture Content

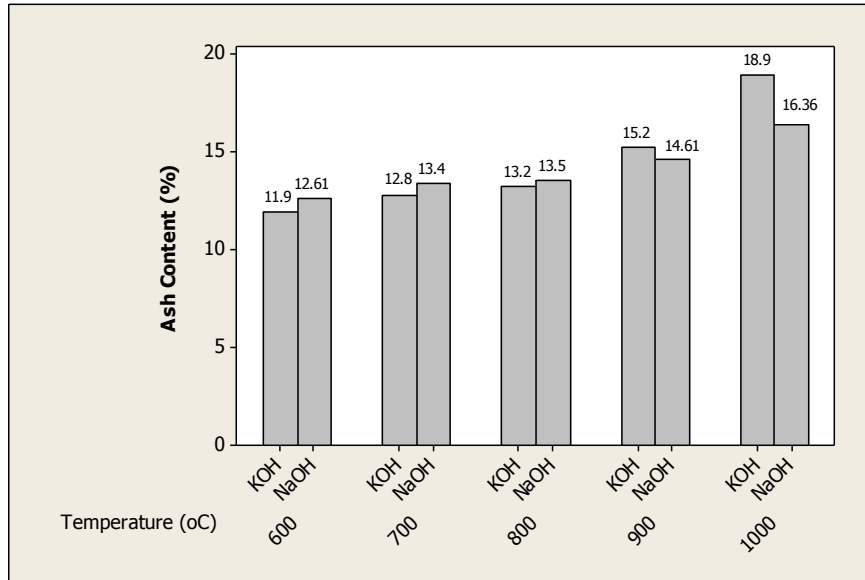


Figure 3.1B Effect of temperature on Ash Content

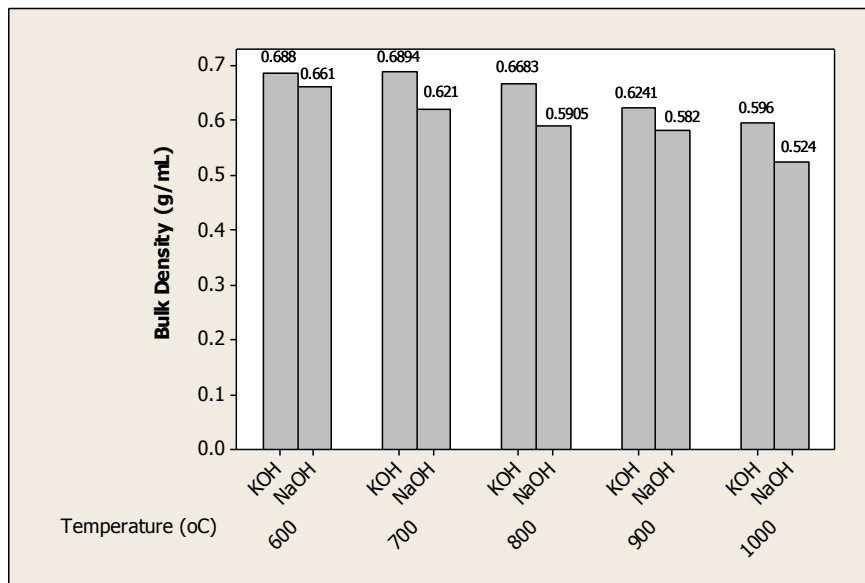


Figure 3.1C Effect of temperature on Density

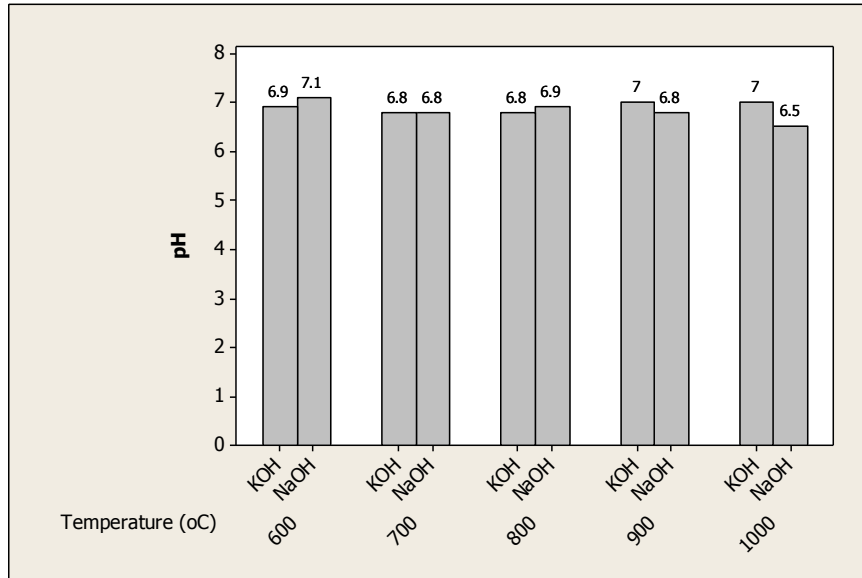


Figure 3.1D Effect of temperature on pH

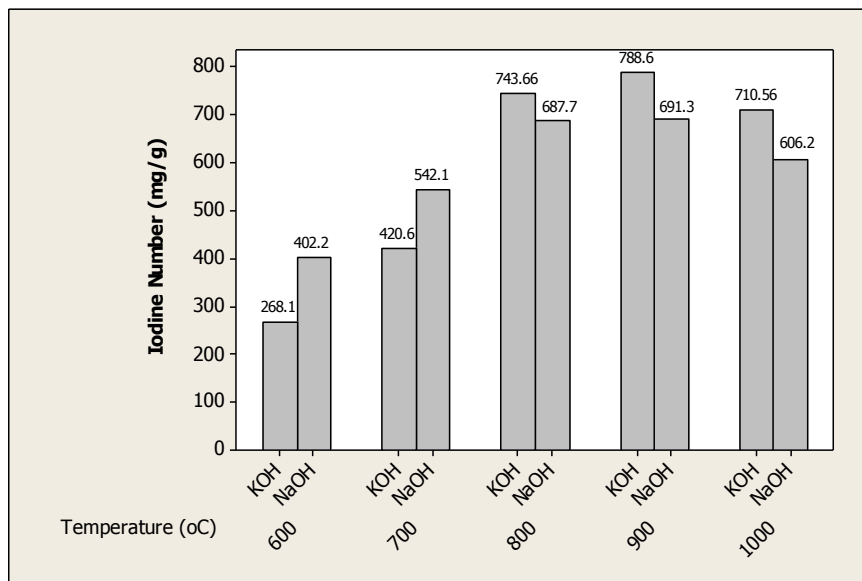


Figure 3.1E Effect of temperature on Iodine number

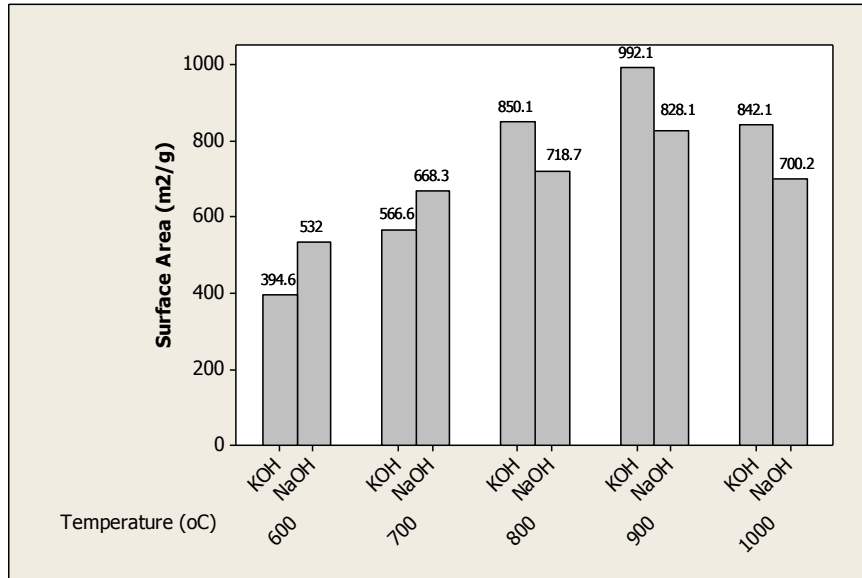


Figure 3.1F Effect of temperature on Surface Area

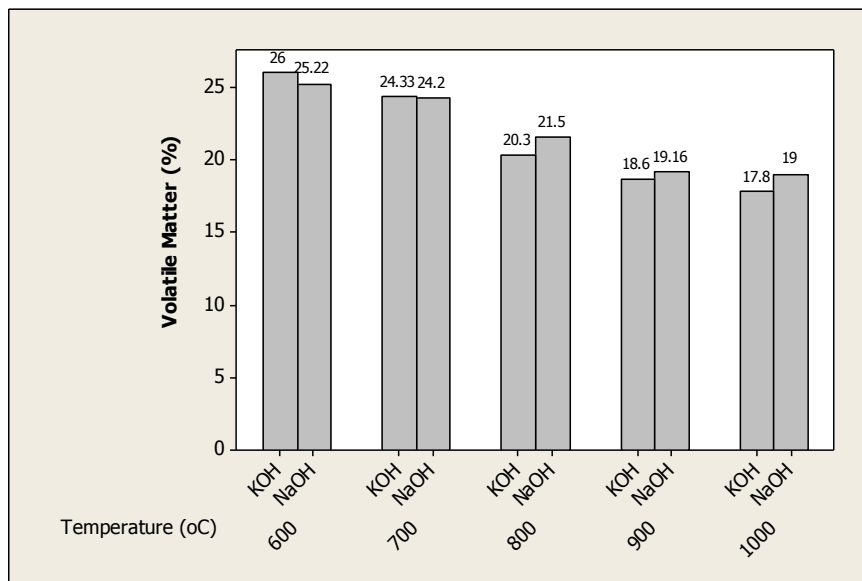


Figure 3.1G Effect of temperature on Volatile Matter

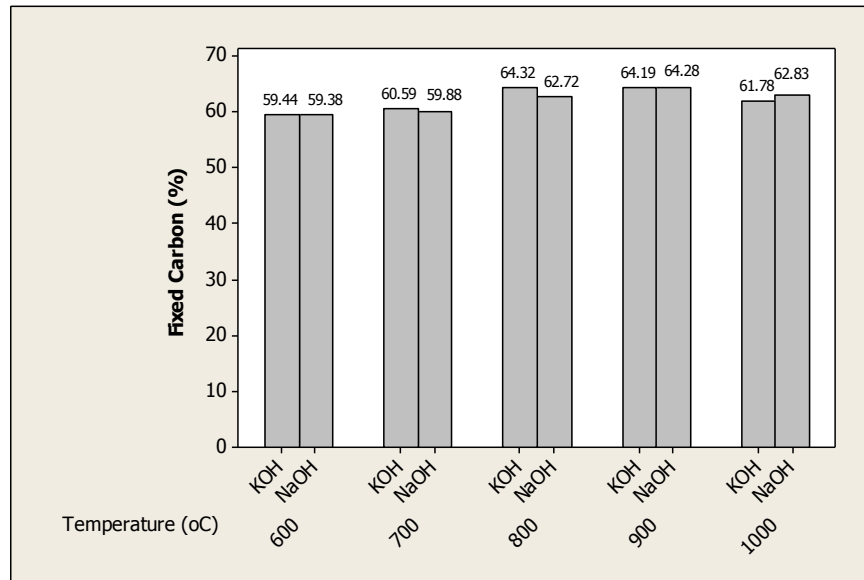


Figure 3.1H Effect of temperature on Fixed Carbon content

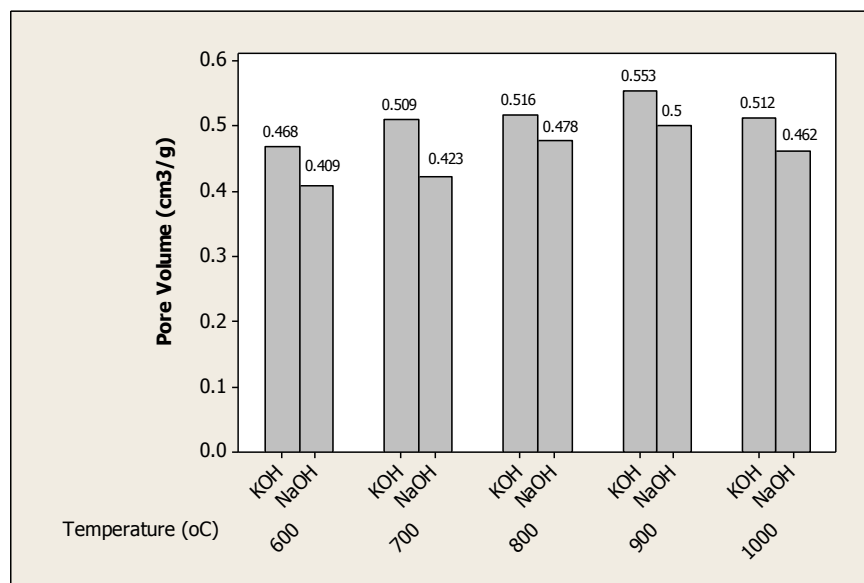


Figure 3.1I Effect of temperature on Pore Volume

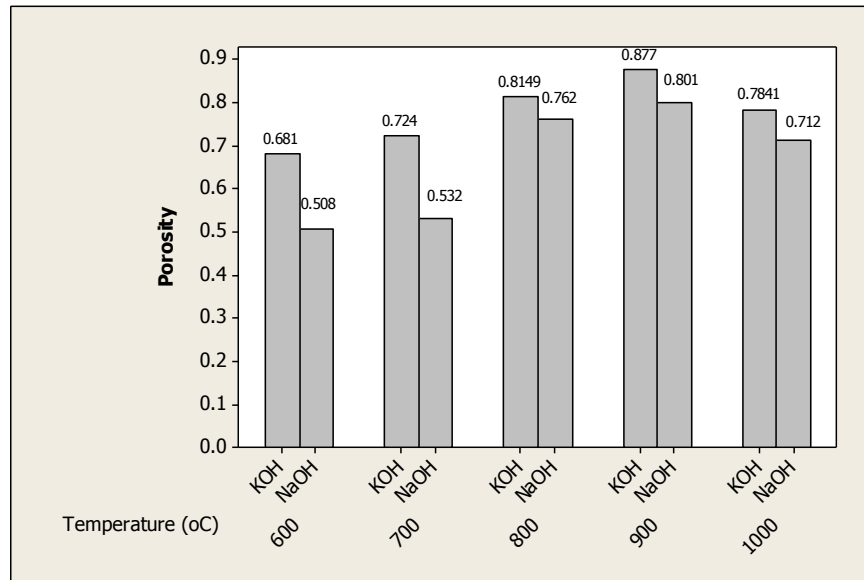
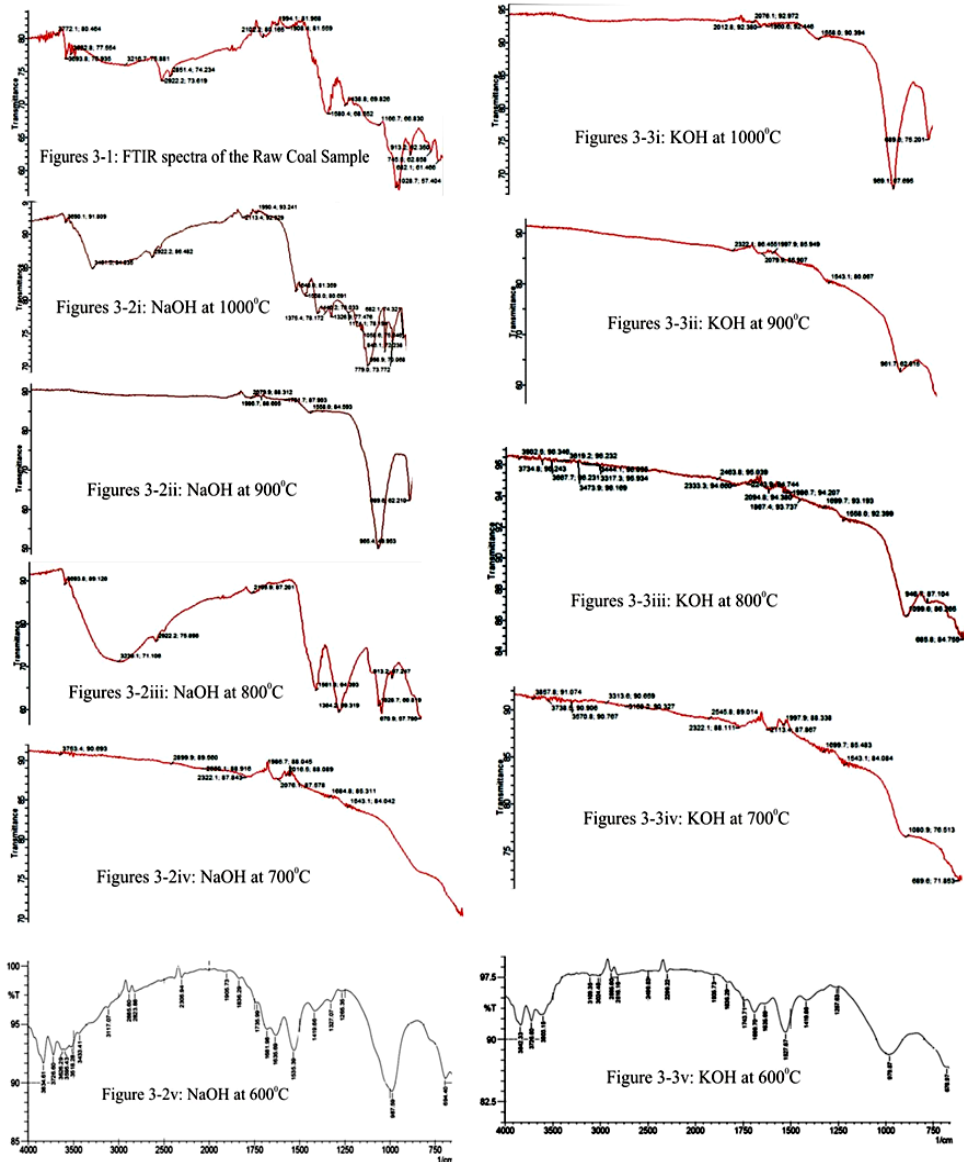


Figure 3.1J Effect of temperature on Porosity



4. Results and Discussion

The results of the properties of raw coal and AC produced from Enugu Coal are presented in Tables 3.1 – 3.2 and in figures 3.1(A-J). The analysis reveals a tremendous improvement in all the assessed properties of AC when compared to the raw coal counterpart. In experiment 2 (Tables 3.1 – 3.2), the effects of activating agent and the impregnation ratio were investigated. From the results, these important adsorptive characteristics of AC such as surface area, iodine value, porosity, bulk density etc increased with the impregnation ratio of each of the activating agents. For instance, in NaOH-activation process, the surface area increased by 43% from 718 m²/g at the impregnation ratio of 1/1 to 2/1. Similarly, the iodine value recorded a substantial increment of 40% at the same range of impregnation. The same trend was obtained for the other adsorptive parameters studied such as moisture content, pore volume, fixed carbon etc. But the KOH activating agent was observed to have displayed an outstanding performance over others in all the conditions investigated. At the impregnation ratio of 4/1, the value of surface area of 1862 m²/g recorded is simply remarkable, likewise other parameters such as iodine value, pore volume etc which recorded values that are far above average in adsorptive rating of standard adsorbent. Nonetheless, ZnCl₂ and H₃PO₄ which produced the least values in that order are however observed to have met the standard specifications of adsorbent. In a similar manner, these assessed variables progressively swift downwards as the impregnation ratio decreases. The observed differences in adsorption characteristics exhibited by various activating agents are in agreement with the work of Fierro and Co-workers (2007), who reported that BET surface area, pore volume, iodine value etc of AC produced from Kraft lignin by chemical activation using KOH are higher than those from NaOH. These findings were also corroborated by other researchers including Mousa et al (2017) and Lillo-Roden and Co-

workers (2004) in their individual works on the comparative analysis of various AC produced from different activating agents on carbonaceous materials. The variation in properties may be due to the nature and degree of interactions occurring between the carbonized chars and the individual activating agents. The oxidizing action of these activating agents leads to decreitive removal of successive layers of atoms from the carbon matrix. The oxidation selectively erodes the surfaces and creates spaces between the elementary crystallites (Echegi, 2017). 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 pores structure with large internal surface areas (Rafsanjani et al, 2013).

The mode of interaction of acid with coal was reported by Baba et al (2017) to be mainly through oxygen functional groups leadings to dehydration and repolymerization reactions with carbon structure. On the other hand, KOH and NaOH, acting as bases, involve the process of both dehydrogenation and oxidation reactions. According to Ejikeme et al (2015), the overall stoichiometric reaction of KOH involves direct attack on the carbon centre resulting in more aliphatic chains and formation of potassium species. These species formed during the activation stage diffuse into the internal structure of the char matrix and widen the existing pores in addition to creating new ones (Mao et al, 2015). By increasing the KOH/Char ratio, the activation process is intensified. Accordingly, the pore sizes are successively broadened and new micropores and mesopores are created in the original pore walls.

In the third group of experiments, figure 3.1(A-J); the effect of temperature on the quality of AC produced at constant impregnation ratio of 1/1 was studied. From the results it was observed that iodine value, pore volume, surface area, porosity etc of the AC, vary with temperature of activation. Each of these

parameters increases with temperature within the range of 600°C – 900°C , establishing maximum values at 900°C . Thereafter, the trend takes a down turn and decreases at the temperature of 1000°C . Thus, it may be deduced that the optimum activation temperature for KOH and NaOH is at the neighbourhood of 900°C . For instance, the iodine value increased from 268 to 789 mg/g at the temperature of 600°C – 900°C and later decreased to 711 mg/g at 1000°C for KOH activation. Similarly, the pore volume for NaOH activation recorded significant increment of $0.100\text{cm}^3/\text{g}$ from $0.409\text{cm}^3/\text{g}$ within the temperature range and subsequently nose-dived to $0.462\text{cm}^3/\text{g}$ at 1000°C . This remarkable trend is indeed a confirmation of the substantial development of adsorptive potentials in AC. These findings are in consonance with the reports of Yusuf et al (2012) on AC produced from coconut shells and cattle bones using chemical activation. The observations may be due to the devolatilization which increases with temperature until some of the linkages in the impregnated sample attain their limit of thermal stability. The mechanism of decomposition according to Baba et al (2007), involves the initiation of volatile formation, release of the free radicals and reactions that form cross-linkages within the char structure. The reaction increases with temperature more especially, for a slow heating rate.

The reduction in adsorptive properties after attaining the maximum value at the temperatures above 900°C , is an indication of occurrence of structural deformation in the already formed AC. The structural deformation may have arisen from the collapse of the pore walls which subsequently created resistances to the diffuse of molten ash out of the pore channels of the AC. The decreases in volume of pores and surface area in AC beyond the optimum temperature may be attributed to significant melting of the ash fractions in the char that might have probably blocked the pores after the char has been cooled to ambient

temperature (Echegi, 2017; Zawawi et al, 2017).

Ash is one of the impurities that forms complexes on the surface of AC leading to alteration in surface characteristics. In addition to the creation of hydrophilic-hydrophobic imbalance, it covers the existing pores and reduces the adsorptive power of the AC as well as the efficiency of reactivation. From the results, the effect of temperature and impregnation ratio indicates appreciable variation in ash content of AC produced. At temperature of 600°C , the least value of 11.9% was recorded by using KOH, while the value increased to 18.9% at the highest temperature 1000°C . However, the variation is relatively less significant with the impregnation as the highest differential value of 1.8% was recorded from 2/1 to 1/1 for NaOH. Comparatively, the ash content for all the varieties of synthesized AC were lesser values but within the permissible range for standard activated carbons (Echegi, 2017). This may be attributed to lower inorganic content and higher fixed carbon which is inherent to the organic nature of the raw material (Orkun et al, 2012).

In both forms of activation, there is significant improvement in density recorded for all the AC. The decrease in density with the temperature and the impregnation ratio is an indication of pore volume development. The lower density leads to greater volume and thus a confirmation of better quality of the AC (Mohammed-Khah et al, 2009). At increasing temperature, the interacting particles are highly activated and repositioned for higher rate of devolatilization which creates structural expansion, widening and re-opening of existing pores and hence a decrease in bulk density (Echegi, 2017). A report from Zawawi et al (2017) corroborated this established evidence in this work although the analysis is on bulk density instead.

The IR absorption bands of oxygen groups on the surface of raw coal are affected by the conditions of two experiments conducted (experiments 2.2 - 2.3). The FTIR of raw coal

shows many peaks belonging to different functional groups. A strong and broad absorption peak appeared between $3770 - 3680\text{cm}^{-1}$ related to hydrogen bonding of O-H stretching. The intense band located at the range of $2920 - 2850\text{cm}^{-1}$ corresponding to C - H asymmetric stretching of methyl and aldehyde groups. A peak at 1680cm^{-1} is associated with C = O stretching, indicating esters, carboxylic acid and conjugated Ketonic structures. The raw coal spectrum also reveals a band between $1440 - 1420\text{cm}^{-1}$ which can be attributed to the presence of C - H asymmetric bonding.

Most of the AC produced from experiment 2.3 (figures 3.3 and 3.4) exhibited similar IR spectroscopic features. The reduction, and in some cases virtual absence of hydrogen bonding at the corresponding range of precursor shows that the activating agent acted as dehydrating agent in the interaction with raw coal (Saurez-Garcia et al, 2002). Similarly, the absence of the band in AC equivalent to the absorption peak at 1680cm^{-1} in raw coal is an indication that chemical activation broke many bonds in aliphatic and aromatic species, thereby eliminating many volatile and light substances (Hesas et al, 2013). At temperature of activation above 700°C , the observation reveals that the characteristic broad coal absorptions which occurred in precursor has disappeared which also suggest a complete carbonization of the samples. From $800^{\circ} - 1000^{\circ}\text{C}$, the two activating agents displayed symbolic differences in spectra absorption. At this 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 vibrations $3500-3400\text{cm}^{-1}$ and C - H aliphatic stretching absorption bands from $2930-2920\text{cm}^{-1}$ (Zawawi et al, 1999). This observation shows that the intensity of spectra absorption bands increases with temperature for both NaOH and KOH reagents. At 900°C , similarities in spectra occurred at $965-960\text{cm}^{-1}$. However, the analysis reveals that the intensity of these wave bands

varies significantly. Contrary to the observation of Cuhadaroglu and Co-workers (2007), KOH-activated carbon recorded higher intensity than the NaOH AC counterpart. This is a corroborative evidence of higher degree of activation in favour of KOH that has been reported earlier in this work. Furthermore, the analysis shows that the main difference between the various AC from different carbonization methods occurred at the band of C - H aromatic stretching. From figure 3.3 and 3.4, the results show that the NaOH - activated carbon has much higher content to C - H than the KOH activated ones.

5 Conclusion

From the results obtained, the adsorptive characteristics of AC produced vary with the temperature of activation and also with the impregnation ratio. The optimum temperatures for all the activating agents were recorded at the neighbourhood of 900°C . The KOH and NaOH established distinctive performance in adsorptive rating which may be described as a rare commending feat in industrial accomplishment. The other activating agents which recorded relatively lower values were equally observed to have met the standard specifications of adsorbent. The FTIR analysis indicated significant reduction in oxygen containing surface groups that lead to the development of aromatic structures after carbonization and deactivation

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