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Effect of Process Factors on the Quality of Activated Carbon Produced From Coal by Microwave-Induced Chemical Activation

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***ABSTRACT**: This work involves the study of the effect of process parameters on the characterized properties of activated carbon (AC) produced from microwave-induced KOH-Chemical activation of Coal. The pretreated Coal sample was subjected to carbonization at 800^0C for 2 hours. The char produced was activated in microwave oven at various conditions of power output, radiation time and impregnation ratio of KOH/Char. The result showed that the effect of these preparation parameters on the adsorption capacity of AC followed the same pattern. The adsorption characteristics monitored increases with each of these process variables up to an optimum value and later decreases after exceeding the maximum limits. The optimum values of microwave power output, radiation time and impregnation ratio were recorded at 726W, 8 mins and 1/2 respectively for all the adsorption potentials investigated. The carbonization of coal was established to be the major determinant in the production of high quality activated carbon.The tremendous improvement of the quality of AC produced is highly encouraging whereas its rating in adsorbent ranking is a remarkable feat of salient achievement.*

***KEYWORDS:** Process factors, Quality, Activated carbon, Coal, Microwave-Induced, Chemical activation.*

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I. INTRODUCTION

Microwaves are forms of electromagnetic energy in which the applied energy is converted into heat by mutual interaction between medium consisting of electric field component of the wave and the charged particles [Foo and Hameed, 2009] Microwave radiation is part of the electromagnetic spectrum with frequencies ranging from 300MHz to 300GHz, lying between infra-red and radio frequencies and wavelength (λ) of 1mm to 10cm. Microwave heating has wide applications in areas such as material sciences, telecommunications, information technology, food processing, organic and polymer synthesis, desulphurization of coal, vitrification of radioactive wastes, metal fractionation in sewage sludge, wood drying, curing as well as preheating of ceramics, plastics and rubber treatment etc [Adekunle and Farid, 2017].

Based on the mode of interaction with the microwave, materials can be classified into three broad groups as conductors, insulators and absorbers. The absorbers consists of carbon-based solid such as Coal, Peat, wood, sawdust and some agricultural biomass etc. These absorbers are high loss materials which absorbs microwave radiation and cause direct energy transfer at room temperature [Foo and Hameed, 2009]. Microwave absorbers are also known as dielectric materials which are characterized by possessing very few free charge carriers and exhibiting a dipole movement [Hesaset al, 2013]. The ability of the dielectric material to be polarized and heated using microwave field is defined by its dielectric loss tangent (Menendez et al, 2010) as

$$\tan\delta = \varepsilon''/\varepsilon'$$

whereε' is the dielectric constant or real permittivity. It is a measure of the material's ability to retard microwave energy passing through it. Whereas ε'' the dielectric loss factor (or imaginary permittivity) which measures the materials ability to dissipate energy [Haque, 1999]. Thus

$$\varepsilon = \varepsilon' - j\varepsilon''$$

ε is complex permittivity and j is equal to $(-1)^{1/2}$.

Researches on microwave heating of carbon-based materials can be classified into two types depending on the phase of the microwave absorber (Menendez et al, 2010). The first category called solution heating comprises those from microwave-assisted organic synthesis, while the others involve solid state carbon materials and metals. In solution heating, the energy absorbed is converted to heat within the particles themselves by dipole rotation and ionic condition. Unlike the organic substances, the carbon-based solids consist of fewer or no rotating dipoles. As reported by Menendez et al (2010), the microwave heating leads to delocalization of Pi-electrons from SP^2-hybridized carbon network.

AC is a carbonaceous substance obtained from special treatment of solid state carbon material. The production objective is intended towards achieving large surface area, high pore volume distribution, controllable pore structure, thermal stability, low acid/base reactivity and rapid adsorption capacity [Echegi, 2017].Basically, it is a crude form of graphite with a random or amorphous structure, which is highly porous, exhibiting a broad range of pore sizes from visible cracks, crevices and slits of molecules dimensions (Foo and Hameedm 2009). Over the years, the preparation of AC is dominated by conventionalmethod of heating process. But due to its inherent limitations, this method of production is now given way to a more efficacious microwave heating process. In conventional heating method, the heating is from the surface to the interior part of each particle resulting in temperature gradient. Even though, temperature gradient can be avoided by slow heating, the result will lead to a long period of preparation with the consequences of greater energy consumption and its attendant cost implications. Succinctly, thermal heating involves high energy consumptions, requires large equipment size and generates undesirable heating rate leading to the detrimental effect on the quality of the AC [Ejikeme et al, 2015]. In addition, the overheating and thermal induced attrition of the sample might result in complete combustion of the carbon [Polart et al, 2010].

However, in microwave heating, there is a direct interaction between the microwaves and the particles inside the pressed compact material leading to quick volumetric heating. (Adekunle and Farid, 2017). The volumetric heating and deep energy penetration produce more uniform heating inside the material, minimize waste and increase the rate of chemical reactivity [Ebere et al, 2015]. Similarly, the consumption of gases reduces due to the reduction in the treatment time. Based on these inherent advantages, microwave is now becoming a viable alternative for the production of AC.

But the successful application of microwave is strongly dependent on several operational parameters such as normal power output, radiation time, nature of preliminary treatment of the precursor, and concentration of activating agents etc. In this study, the effects of these parameters were investigated on the quality of AC produced from Enugu sub-bituminous coal using KOH as activating agent.

II. MATERIALS AND METHOD

2.1. Production of the Coal Sample

The sourced coal from Enugu coal mine was subjected to preliminary treatment of cleaning, drying, size reduction and sieving. The coal sample was washed with water to remove dirt particles from the surfaces and sun-dried for 2 days at the interval of 8 hours each. The dried sample was crushed and sieved to the mesh size of 1-2mm.

2.2 Carbonization

50g of the ground coal sample was placed into the electric furnace and heated at the rate of 10^0C per minute from room temperature to 800^0C. After attaining the above temperature, the heating was maintained for 2 hours. Later, the carbonized sample was allowed to cool gradually to ambient temperature.

2.3 Activation

The carbonized sample was ground and sieved to a particle range of 0.75 to 1.00mm. The char product was impregnated with activating agent of KOH at KOH/Char ratios of 1/4, 1/3, 1/2, 1/1 and 2/1. The resulting samples were designated M1, M2, M3, M4, and M5 respectively. The mixture was stirred for 2 hours and later filtered, dried over-night at 110^0C and stored in desiccators.

For the microwave activation, 20g of KOH – impregnated char of sample1 coded MI was added to a ceramic crucible in a cylindrical quartz reactor, placed in a 2450MHz microwave with a nominal power output of 726W. The radiation process was conducted for 8 minutes under a 0.5L/min flow of humidified N_2. After the microwave activation, the KOH activated carbon was cooled to room temperature using a flow of humid N_2 at the rate of 0.5L/min. The activated carbon sample was then mixed with 0.1N HCl and stirred for 1 hour. Later, it was washed with de-ionized water until a pH range of 6.5 – 7.5 was reached. Finally, the AC sample was dried at $100^0 \pm 5^0$C for 24 hours. The same procedure was repeated with the other samples of M2, M3, M4 and M5 of KOH –impregnated char. In the same vein, the effect of microwave power output was studied by conducting the same experiment at different nominal power output of 162W, 324W, 520W, & 726W and 900W at constant impregnation ratio of 1/2 and radiation time of 8 mins. Also at the constant microwave power of 726W and impregnation ratio of 1/2, the effect of radiation time was investigated at the exposure time of 4, 6, 8, 10 and 12 minutes. Prior to this, series of experiments were conducted using the same procedure at process conditions of power output of 726W, radiation time of 8 minutes and impregnation ratio of 1/2 but with the omission of the carbonization of the raw coal. Finally, the characterized properties of surface area, iodine number, pore volume, moisture content, porosity, fixed carbon content etc of the AC were conducted and evaluated.

III. RESULT AND DISCUSSION

3.1 Effect of Non-Carbonization of Raw Coal on the Quality of AC

Parameter	Moisture content (%)	Ash Content (%)	Bulk density (g/cm^3)	pH	Iodine number (mg/g)	Surface area (m^2/g)	Volatile matter (%)	Fixed carbon (%)	Pore volume (cm^3/g)	porosity
Value	2.86	14.04	0.54	7.1	429	501	24.63	58.47	0.39	0.52

3.2 Effect of Power Output on the Quality of AC

Power output / Parameter	162W	324W	520W	726W	900W
Moisture Content (%)	2.91	2.43	2.28	2.16	1.85
Ash Content (%)	10.87	10.99	11.07	11.26	13.32
Bulk density (g/cm^3)	0.55	0.55	0.54	0.53	0.54
pH	6.9	6.9	6.9	7.1	6.8
Iodine Number (mg/g)	299	792	984	1225	972
Surface Area (m^2/g)	360	896	1074	1380	1026
Pore Volume (cm^3/g)	0.38	0.42	0.47	0.51	0.45
Porosity	0.41	0.48	0.58	0.64	0.60
Volatile Matter (%)	30.12	27.26	20.83	12.57	12.44
Fixed Carbon (%)	56.20	60.84	65.82	74.01	72.39

3.3 Effect of Impregnation Ration on the Quality of AC

Impregnation Ratio / Parameter	1/4	1/3	1/2	1/1	2/1
Moisture Content (%)	2.85	2.46	2.28	2.09	1.89
Ash Content (%)	10.76	12.06	12.01	12.85	12.77
Bulk density (g/cm^3)	0.54	0.55	0.52	0.50	0.50
pH	7.0	7.0	7.2	7.1	7.2
Iodine Number (mg/g)	497	909	1258	986	809
Surface Area (m^2/g)	680	1008	1436	1007	964
Pore Volume (cm^3/g)	0.38	0.52	0.61	0.54	0.50
Porosity	0.45	0.56	0.68	0.61	0.58
Volatile Matter (%)	31.25	24.61	10.25	11.97	10.20
Fixed Carbon (%)	55.14	60.83	75.46	73.09	73.54

3.4 Effect of Radiation Time on the Quality of AC

Radiation Time / Parameter	4 mins	6 mins	8 mins	10 mins	12 mins
Moisture Content (%)	2.67	2.48	2.14	2.01	1.98
Ash Content (%)	12.86	14.01	14.41	15.24	15.44
Bulk density (g/cm^3)	0.57	0.55	0.53	0.54	0.54
pH	6.8	7.0	7.0	6.9	7.0
Iodine Number (mg/g)	618	891	1132	1016	987
Surface Area (m^2/g)	699	1082	1405	1178	1064
Pore Volume (cm^3/g)	0.36	0.54	0.62	0.60	0.55
Porosity	0.48	0.56	0.69	0.64	0.63
Volatile Matter (%)	27.82	20.59	15.26	13.92	14.01
Fixed Carbon (%)	56.65	62.92	68.19	68.83	68.57

The result of the microwave-induced chemical activation of coal using KOH at various process conditions are presented in Tables 3.1 – 3.4. The analysis reveals that after the carbonization and activation, the adsorption characteristics of AC such as surface area, pore volume, iodine number, porosity, moisture content, fixed carbon etc improved tremendously. The observation shows that the values of these assessed properties recorded are dictated by the specific process variables of power output level, radiation time and impregnation ratio of KOH/Char employed in the experiment. In addition to the process parameters, the nature of activation is also one of the key factors in the determination of the quality of the AC. For instance, in Table 3.1, the values of these characterized properties of AC obtained were relatively low mainly due to the omission of carbonization process prior to activation in microwave oven. This report is corroborated by Adekunle et al, (2017) on the analysis of the properties of AC produced frombiomass using microwave heating. Therefore, with a view to enhancing the quality of the AC, the pretreated coal sample was subjected to complete carbonization at elevated temperature of 800^0C for 2 hours. At the first instance, preliminary experiments on the effects of power output, heating time and impregnation ratio, were conducted to investigate the range of applicability of the process variables vis-a-viz the quality of the AC produced. This indeed, provided proper direction to the approach of some experimental intrigues encountered and led to the simplification of the work.

3(i) Effect of Power Output level on the Properties of AC

Based on preliminary studies conducted earlier on, the other identifiable two parameters of radiation timeand impregnation ratio were maintained constant at 8 mins and 1/2 respectively during the investigation of the effect of power output level on AC. The increase in microwave nominal power output from 162W to 726W resulted in substantial improvement of the adsorption capacity of the prepared AC. For instance, the surface area recorded at 162W is a mere value of $360m^2/g$ which is remarkably low in adsorbent rating. The value increased with the power output and reaching a maximum value of $1380m^2/g$ at 726W. However, further increase in power level to 900W, the surface area obtained, declined to $1026m^2/g$. Similarly, the iodine number increased from 299 to 1225 mg/g and thereafter took a down turn and decreased to 972mg/g at the above mentioned power output range respectively. Accordingly, the investigation of the effect on the pore structure development in terms of pore volume and porosity of the AC exhibited the same phenomenon of occurrence. Like the surface area and iodine number, the pore structure development and ultimately the adsorption capacity of AC as depicted in Table 3.2 increases with microwave power output up to and optimum mark at the neighbourhood of 726W. Beyond the optimum power output, all the assessed properties of AC were observed to decline with the increase in nominal microwave power output level. These findings are in consonance with the reports of Hesas et al, (2013) and Foo et al, (2012) on the analysis of quality of AC produced from microwave-induced chemical activation of agricultural wastes and biodiesel.

At the high power output level, the reaction between the char and activating agent intensifies due to increase in temperature. This leads to higher rate of volatilization of the melted ash and tar, and subsequent creation of large amounts of micropores and mesopores [Echegi et al, 2018]. The decrease in these adsorption potentials beyond the optimum power output of the microwave activation may be due to the burning of the carbon and destruction of the pore structures by higher level of radiation. The significant melting of the ash that plausibly blocked the pores in char when cooled to ambient temperature as reported by Zawawi et al, (2017) and corroborated by Amir (2012) arose from the structural deformation and collapse of pore walls at excessive temperature of activation.

3(ii) Effect of Impregnation Ratio on the Quality of AC

From Table 3.3, it was observed that the adsorption capacity of the prepared AC was appreciably affected by the changes in the concentration of KOH-activating agent. The effects were investigated at various impregnation ratios of KOH/Char at 1/4, 1/3, 1/2, 1/1 and 2/1 under the experimental conditions of constant power output level of 726W and radiation time of 8 mins. With the increasing ratio from 1/4 to 1/2, all the adsorption properties of AC increased progressively. For instance, the iodine number recorded a significant increment of 761mg/g from 497mg/g at the range of the ratio of 1/4 to 1/2. This prove of positive effect could not be extended beyond 1/2 as the increase to 1/1 resulted in reduction to 986mg/g. In the same fashion, the surface area increased to a maximum of $1436m^2/g$ from $680m^2/g$ at the ratio of 1/4 to 1/2. Subsequently, it decreased to $964m^2/g$ as the ratio increased to 2/1. The effect on pore volume development and porosity is also remarkable and thus a confirmation of the factor that concentration of activating agent is one of the key determinants in the production of high quality AC from microwave activation. Notably, the concentration of KOH-activating agent produced significant variations on most of the characterized parameters monitored. Generally, the results of these adsorption properties investigated exhibited the same trend of steadily rise to optimum and thereafter a decrease to a minimum as the concentration of KOH increases. This pattern of occurrence is in agreement with the work of Bouchelta et al (2012) on the study of effect of NaOH-activating agent on AC produced from Petro-chemical Industrial Waste under microwave-induced chemical activation. Similarly, Deng et al (2010) and Liu et al (2010), in their separate works, obtained the same results on the investigation of the effects of microwave power output on the pore structure of AC produced from cotton stalk and bamboo by microwave induced chemical activation.

The role of impregnation agent is to minimize the formation of tars and other liquids that could possibly clog up the pores and inhibit the development of porous structures of AC (Bouchelta et al, 2012). At high concentration of impregnation ratio, it may be inferred that the reaction is highly strengthened. The developed of porosity in KOH activation is attributed to the reduction of KOH to potassium by this spontaneous reaction [Lillo-Rodena et al, 2004; Mao et al, 2015].

$$4KOH + C \rightleftharpoons 4K + CO_2 + 2H_2O$$

Therefore, by increasing the KOH/Char ratio, the activation process is intensified. The potassium species formed during the activation stage diffuse into the internal structure of the char matrix leading to the creation of new pores and widening of the existing ones (Mao et al, 2017; Ejikeme et al, 2015).

3(iii) Effect of Microwave Radiation Time on the Quality of AC

Based on the findings of the control experiment, the effect of heating time was conducted at the constant microwave power output of 726W and impregnation ratio of 1/2 while varying the radiation time from 4 to 12 minutes. From the results in Table 3.4, the characterized adsorption parameters of surface area, pore volume, iodine number, porosity etc are increased as the radiation time is increased from 4 to 8 minutes. But when the radiation time is increased from 8 to 12 minutes, a drop in adsorption capacity was noticed. At this range, the values of these assessed parameters decreased progressively to minimum. Thus, the finding indicates the attainment of optimum adsorption capacity at the neighbourhood of 8 minutes. Remarkably, the analysis of the result shows that these adsorption parameters do not vary monotonically with the heating time. For instance, the variation of microwave heating time from 4 to 6 minutes resulted in a substantive increment of about 44% in iodine number. But increasing the heating time from 6 to 8 minutes increased the iodine number by 27%. Similarly, for the surface area, the increments recorded at the above mentioned rates are 57% and 30% respectively. While at the reverse side of the optimum attainment, the iodine number decreased by 11% from 8 to 10 minutes, whereas the surface area also produced a decrease of 16%. The same trend was observed in other adsorption parameters investigated in this work such as pore volume, porosity, moisture content, fixed carbon etc.

The findings obtained in this work correlated with the earlier studies by Deng et al (2010), Foo and Hameed (2012) and Li et al (2009) in their respective research works on the effect of heating time on the adsorption capacity of AC prepared by the method of microwave-induced chemical activation. Also Hesas et al (2013), observed that the adsorption capacity of AC decreases when the activation time increases beyond the optimum value in the activation stage. The same adsorption trend was reported by Mao et al (2017) for the AC's produced at different radiation times in microwave-assisted KOH activation of Pinewood and Wheat straw. These findings indicate that prolonging exposure to microwave radiation results in an increase in temperature which in turn increases the reaction rate, thus improving those aforementioned adsorption parameters. It was attributed that the drop in adsorption capacity of AC after the optimum time is due to drastic rise in temperature and localization of hot spot in the char and the subsequent ablation as well as shrinkage of the AC channels and skeleton.

IV.CONCLUSION

In this study of microwave-induced chemical activation of Enugu sub-bituminous coal using KOH, the results show that the AC obtained possessed high adsorption characteristics that classify it accordingly as highly rated adsorbent in quality ranking. Each of the process parameters of power output level, radiation time and impregnation ratio of KOH/Char had significant impacts on the adsorption capacity of the prepared AC. The effects revealed that the values of the assessed properties of surface area, iodine number, pore volume, porosity etc obtained increased with power output level, radiation time and impregnation ratio up to an optimum value. Further increase beyond the established maximum led to a decrease in value due to excessive carbon burnt off. However, these properties of AC monitored do not vary monotonically with the process variables. Whereas the carbonization of the raw coal is one of the decisive factors for the production of high quality AC from microwave activation of coal.

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