Effect of Activation Method and Agent on the Characterization of Prewinkle Shell Activated Carbon

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Abstract

The effect of activation method (physical and chemical) and the concentration of the chemical activating agents on the characterization of periwinkle shell activated carbon was investigated. The periwinkle shell was pyrolysed and activated by physical activation (using an oxidizing gas - steam) and chemical activation (using base: ZnCl₂, CaCl₂ and acid: H₂SO₄, HCl). The produced activated carbons were characterized to determine the bulk density, moisture content, pore volume, porosity, ash content, iodine number and surface area following standard ASTM procedures. The effects of concentration of the activating agents on these properties were also studied. Effects of activation method on activation carbon properties showed that the chemical activated carbon had higher bulk density, pore volume, porosity, ash content, iodine number, and surface area, while the physical activated carbon had higher moisture content. The effect of activating agent concentration showed that the bulk density, pore volume and porosity increased with concentration for base activated carbon and decreased with concentration for acid activated carbon. The ash content and iodine number fluctuated (initial increase then decrease and increase) for base activated carbon; while these parameters increased continuously and fluctuated (initial decrease the continuous increase) with concentration for acid activated carbon. The moisture content and surface area decreased with concentration for base activated carbon; these parameters fluctuated (initial increase, decrease then increase) and increased continuously for acid activated carbon. Periwinkle shell carbon chemically activated with ZnCl₂ was the best activated carbon with highest density, pore volume, porosity, iodine number and surface area.

Keywords: Activated carbon, Physical and Chemical activation, effect of activation agent concentration.

1. INTRODUCTION

Activated carbon (activated charcoal) is a form of carbon processed to be extremely porous, increasing its surface area for adsorption or chemical reactions. It is composed primarily of carbon atoms and can be produced from any material with high carbon content. Such material includes hard/soft wood, lignite and coal. The use of waste materials that have no competing demands and environmental concerns has necessitated the use of such waste materials with high carbon content for the production of activated carbon. These materials include industrial waste, agricultural by-products and residual wastes such as paper mill sludge, bagasses fly ash-a sugar industry waste, rice husk, date and peat stones, oil palm waste, palm kernel fiber and shell, coconut shell, periwinkle shell, snail shell, waste bamboo etc. The intrinsic pore network in the lattice of these materials after been processed ensures the removal of impurities from gaseous and liquid media.

Activated carbons are generally prepared by the carbonization of raw organic, carbonaceous starting materials in an inert atmosphere followed by the activation of carbonized product/char.

The carbonization creates initial porosity and orders the carbon structure to enrich the carbon material; the activation enhances the carbon structure by widening the pores, making it more porous (Daud & Ali, 2004). Carbonation involves pyrolysis of the raw material at a temperature range of 600 - 900°C in the absence of oxygen (in inert atmosphere with gases like argon or nitrogen) during which volatile components are removed, producing a residual carbonaceous product with low surface area.

Activation can be performed either by physical/thermal or chemical methods (Rahim, et al., 2008). Physical activation process involves treatment of the char obtained from carbonization with gasification reactants/oxidizing gases such as steam, carbon dioxide, air or a suitable combination (Ahmida, et al., 2015) at high temperature (400 - 1000°C) (Ahmad, et al., 2015). The porous activated carbon is produced when the oxidant converts the carbon materials to form carbon mono-oxide (CO) and carbon dioxide (CO₂) thus opening pores in the activated carbon materials. Chemical activation process involves treatment of the char obtained from the carbonization with chemicals such as alkali and alkaline earth metal containing substances (KOH, NaOH, and Na₂CO₃), acid (H₃PO₄, HCl, H₂SO₄ and C₂H₄O₂) and other chemicals/salts (ZnCl₂, CaCl₂ etc), the chemicals functioning as dehydrating or oxidizing agents, influencing the pyrolytic decomposition and inhibiting the formation of tar. This process is usually done at lower temperature (500°C to 800°C) and activation time, giving higher carbon yields, surface area, better porosity and lower energy cost as compared to physical activation (Ahmad, et al., 2015).

Activated carbon is widely used in domestic, commercial and industrial settings (Mendez et al., 2006), these include the food industry where activated carbon is used in de-colourization, deodorization and taste removal; to

remove heavy metals and organic contaminants from liquids, in water de-chlorination and processing of foods, in gas cleaning applications and air filters. The efficiency of an activated carbon particularly its adsorption capacity had been reported by Hayashi, et al., (2002) and Kwaghger, & Ibrahim, (2013) to be a function of the type of precursor, preparation methods and conditions used in its production. Parameters such as impregnation ratio (Ahmadpour & Do, 1997), method of mixing, activation temperature and activated carbon. Hence optimizing these parameters (Olorundare, et al., 2014) will ensure the production of high quality activated carbon.

Therefore, the objectives of this study were to investigated the efficiency of the pretreatment methods, the activating agents used and their concentration (impregnation ratio) on the overall performance – the physiochemical properties of the produced activated carbon. These were achieved by characterizing the activated carbon produced from periwinkle shell by physical (steam) activation and chemical activation with zinc chloride (ZnCl₂), calcium chloride (CaCl₂), sulphuric acid (H₂SO₄) and hydrochloric acid (HCl) at varying concentrations for the adsorption of methyl blue from its aqueous solution.

2. MATERIALS AND METHODS

2.1 Material

The raw material (periwinkle shells) used for production was obtained from a local market at Eagle Island in Port Harcourt, Rivers State. The chemical activation agents, zinc chloride ($ZnCl_2$), calcium chloride ($CaCl_2$), sulphuric acid (H_2SO_4) and hydrochloric acid (HCl) were purchased from a chemical shop at the industrial chemical section of Mile 3 market, while distilled water was obtained from the Department of Chemistry laboratory, Rivers State University. All chemical reagents used were of analytical grades. The pyrolysis reaction was performed using the "pyrolysis set up" in the reaction kinetics laboratory of the Department of Chemical/Petrochemical Engineering, Rivers State University, Port-Harcourt, Rivers State, Nigeria. The activation of the carbon and characterization of the produced activated carbon were also performed at the reaction kinetics laboratory.

2.2 Methods

Pretreatment of Raw Material

The periwinkle shell samples were washed several times with distilled water to remove sand and other surface impurities and sun dried.

Pyrolysis/Carbonization

The periwinkle shells were heated in a muffle furnace for 30 minutes to dry (free of water). A measured weight

of the shells was introduced into the reactor and pyrolized at 300° for three hours in the absence of air. A receiver was connected to the condenser to receive the distillates formed during the pyrolysis. The char material was cooled at room temperature before discharging into containers.

Activation of Produced Charcoal

a. Physical Activation

A measured weight of the crushed sample was soaked in distilled water and heated at a temperature of 800°C in a furnace for two hours to form slurry. The slurry was washed and the activated carbon allowed to air dry.

b. Chemical Activation

The carbonized periwinkle shell was chemically activated using four different activating reagents: two bases $(ZnCl_2 \text{ and } CaCl_2)$ and two acids (HCL and H_2SO_4)) at different concentrations (0.1, 0.25, 0.45, and 0.65 mol.). The carbonated material (carbon) from the periwinkle shells was crushed into powdered form using a crusher. Measured weights of the crushed sample were soaked in a varying concentrations of the activating agents in conical flasks and stirred until the mixtures turned a paste. The pastes were heated in a muffle furnace at 550°C for 2 hours. The activated carbons were cooled at room temperature, washed with distilled water until pH were approximately 7 (no change in color when tested with red litmus paper) indicating no trace of the activating agent used. The chars were dried for 6 hours and stored in tight nylons.

Characterization

The characterization of different physio-chemical properties of the produced activation carbon was performed following standard procedures. All results were the average of duplicate analysis.

i. Bulk Density

The Bulk density was determined using ASTM 2854-09 method (ASTM D2854, 2014). 10g of the sample into a centrifugal tube and tapped on bench top until the volume of the sample stops decreasing. The initial weight of the empty centrifugal tube was determined by weighing, the final weight of both the tube and the sample was determined after tapping.

Bulk Density =
$$\frac{m}{x_1 - x_2} X 100$$

(1)

Where: x_1 is the displaced volume of water

 x_2 is the initial volume of water m is the mass of sample (AC) used

ii. **Pore Volume and Porosity**

The pore volume and porosity of the activated carbons were obtained using the bulk data.

Pore volume was calculated using the expression:

$$Pore Volume = \frac{Bulk Density}{Density of water} \times 100$$
The porosity was calculated using the expression:
Pore Volume
(2)

$$Porosity = \frac{Pore \, Volume}{Weght \, of \, carbon} \, x \, 100 \tag{3}$$

iii. Ash Content

Ash content determination was performed following ASTM D2866-11 method (ASTM D2866, 2011). 5g of the sample was placed in a crucible and reweighed with its content. The initial weight of the sample and crucible was determined before heating. The sample was then heated in a furnace at a temperature of 900°C for 3 hours in an open crucible. The sample was cooled to room temperature (dry) and reweighed. In this test, the amount of residual substance is equal to the ash present in the sample. The ash content was then calculated using the formula:

$$\% Ash = \frac{(wt.crucible + AC - wt.crucible + ash)}{(wt.crucible + AC - wt.crucible)} \times 100$$
(4)

iv. Moisture Content

Moisture content will be determined using ASTMD 2867- 99 "oven drying method" (ASTM D2867, 2009). 2.0 grams of sample (W_1) was dried using a moisture analyzer at a certain temperature of about 150°C, until the weight of sample was constant (W_2). The moisture content was determined using the equation.

$$X_{o} = \frac{W_{1} - W_{2}}{W_{1}} \times 100$$
(5)

Where: X_0 = moisture content on wet basis

 W_1 = initial weight of sample in grams

 W_2 = final weight of sample in grams after drying

v. Iodine Number

The iodine number is an index that is used to express the adsorption capacity, surface area, and porosity of the activated carbon. The iodine number was determined following ASTM D4607-94 method (ASTM D4607, 2014). 5g of activated carbon was measured; 10ml of HCl was boiled at 60° C for 20minutes and allowed to cool to room temperature. 100ml (v) of 0.1N (m) prepared iodine solution was added to the sample and stirred for 1minute. The solution was filtered using a filter paper. 50ml of the filtrate was measured and 10ml of starch was added to the filtered solution as indicator. The sample was then titrated against 0.1N or 0.05 mol/L sodium thiosulphate solution using a burette. The iodine value (IV) was calculated using the formula:

$$IV\left(\frac{mg}{g}\right) = \left(\frac{y-x}{yv}\right)\left(\frac{1}{w\,m}\right)$$

Where:

Y = volume of thiosulphate for blank

X = Titer value

V = volume of iodine solution used

W = weight of sample

M = molarity of iodine solution

vi. Surface Area

The determination of surface area was performed by the adsorption of nitrogen at 196°C using a micromeristic ASAP 2000 instrument to determine the specific surface area of the activated carbon samples. The samples were out gassed overnight at 180°C prior to adsorption measurements. The surface area equation is applied to fit the nitrogen adsorption isotherms and evaluate the specific surface area of the samples. The Surface area was calculated using:

$$S_{A} = \frac{X_{M}N_{A}}{M}$$
 Where: X_{M} = Adsorption value from spectrophotometer N_{A} = Avogadro's No.
M = Mass of Activated carbon used

(7)

(8)

3. DISCUSSION OF RESULT

Five grades of activated carbon were produced. AC1: Periwinkle shell carbon physically activated with steam; AC2a & AC2b: Periwinkle shell carbon chemically activated with two bases: $ZnCl_2$ & $CaCl_2$; AC3a & AC3b: Periwinkle shell carbon chemically activated with two acids: H_2SO_4 & HCl. The physio-chemical properties of these activated carbons were determined and presented in Table 1. The optimum value of the properties of the chemically activated carbon at the optimum concentration (within the range considered) of the respective activating agent was used in Table 1 as the bases for comparison with the physically activated carbon. Table 1: Physio-Chemical Properties of the Produced activated carbon

Tuble 1.1 Hysio Chemiear Troperties of the Troduced activated earbon.						
SN	Physio-Chemical Property	ACTIVATED CARBON				
		PHYSICAL CHEMICALLY ACTIVATED				
		STEAM	BASE		ACID	
		AC1	AC2a	AC2b	AC3a	AC3b
1.	Bulk Density (gm/L)	1.12	1.59	1.49	1.38	1.34
2.	Moisture Content (%)	1.80	1.77	1.15	1.14	2.3
3.	Pore Volume	0.127	0.159	0.149	0.138	0.134
4.	Porosity (%)	2.54	3.18	2.98	2.76	2.68
5.	Ash Content (%)	2.00	5.20	5.80	3.00	2.60
6.	Iodine Number (mg/g)	0.034	0.074	0.319	0.123	0.198
7.	Surface Area (m^2/g)	7.319	7.79	7.719	7.695	7.759

Table 1 shows that: the bulk density of the physical activated carbon was lower than those of the chemically activated carbon. This is in agreement with the work of Prauchner & Rodríguez-Reinoso, (2012) who reported that coconut shell activated carbons prepared using physical activation with carbon dioxide (CO_2) had lower bulk density than that chemical activated with H₃PO₄ or ZnCl₂. The moisture content of the chemically activated carbon were lower than those of the physically activated carbon except for chemically activated carbon with HCl. This is due to the dehydrating effect of the activating chemicals used. The ash content of the chemically activated carbon were higher than those of the physically activated carbon. Similar result was obtained by Hu, et al., (2001) where impregnated carbon with H₃PO4 had higher ash content than the physically activated carbon. The chemically activated carbon had higher iodine number. Baseri, et al., (2012) also reported that the physical activated carbon of yellow oleander (thevetia peruviana) had lower iodine number than those chemically activated with Na₂SO₄, H₃PO₄, ZnCl₂, KOH, HCl, and H₂SO₄. The pore volume, porosity and surface area of the chemically activated carbons were higher than that of the physically activated carbon. This according to Malik, et al., (2006) is due to the dehydration and decomposition of the organic matter by the chemicals during chemical activation causing high porosity of activated carbon. Similar result was obtained by Hidayu, & Muda, (2016) who reported that the pore volume and surface area of chemically (ZnCl₂) activated palm shell carbon were higher than that of the physically activated carbon. The pore volume and porosity of the base $(ZnCl_2 \&$ CaCl₂) activated carbon were higher than those of the acid (H₂SO₄ & HCl) activated carbon. These results are in agreement with those of Gin, et al., (2014) where the pore surface and structure of ZnCl₂ activated carbonized cassava peel was found to be the best developed compared to those of sulphuric acid and hydrochloric acid activated carbonize cassava peel. Periwinkle shell carbon activated with $ZnCl_2$ had the highest surface area. In a similar work by Hidayu & Madu, (2016) this was because ZnCl₂ activating agent had contributed to create more new pores and widen the existing pores.

In chemical activation, different activating agents are expected to significantly affect the extent of activation (Subramani & Revathi, 2015). Therefore, the effects of the concentrations of the activating agents on these properties were studied and presented as follows:

3.1 Bulk Density

The effects of varying the concentrations of the activating agent (base and acid) on the Bulk density of the chemically activated carbon are shown in Figure 1A and 1B.



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Figure 1B: Effect of Concentration of Acid Activating Agent on Bulk Density of Activated Carbon

Figures 1A showed that the bulk density of base (ZnCl₂ and CaCl₂) activated carbon increased with increase in concentration. This trend is in agreement with Pugmire, et al., (1991) where the concentration of activating chemicals had been reported to affect cross-linking reactions which leads to growth of molecules and hence increase in density. Figures 1B showed that the bulk density decreased with increase in concentration for the acid (H₂SO₄ and HCl) activated carbon. Similar results of decrease in bulk density with increase in concentration of activating agent were obtained by Yakoot & El-Deen, (2016) using phosphoric acid for the activation of olive stones and Adib, et al., (2016) using phosphoric acid for the activation of sugarcane bagasse. Bulk density is the mass of a unit volume of the sample in air, including both the pore system and the voids among the particles (Zhonghua, et al., 2001). Hence the bulk density determines the amount of carbon that can be contained in a filter of a given solid and the quality of liquid that is retained. The results showed increasing carbon particle in base activated carbon and a decreasing carbon particle in acid activated carbon. Higher density provides greater volume activity hence indicates better quality of an activated carbon (Laine, et al., 1989). Therefore, the carbon should be activated at concentration that gives the highest density.

3.2 Moisture Content

The effects of varying the concentrations of the activating agent (base and acid) on the moisture content of the chemically activated carbon are shown in Figure 2A and 2B.



Figure 2A: Effect of Concentration of Base Activating Agent on Moisture Content of Activated Carbon





Figure 2A showed that the moisture content decreased then gradually became almost constant with concentration of the activating agent for the base $(ZnCl_2 \text{ and } CaCl_2)$ activated carbons. Similar results were obtained by Owabor & Iyaomolere, (2013); the decrease was due to dehydration rate which increased with concentration of activating agent to a point after which increase in concentration had little effect on the moisture content.

Figure 2B showed that the moisture content initially increased then decreased with concentration after 0.25 mol. and increased continuously with concentration of the activating agent after 0.45 mol. for the acid (H2SO4 and HCl) activated carbons. Similar result was obtained by Ugwoha, et al., (2017). The moisture content is the amount of water physically bound to the activated carbon under normal condition. Zhou, et al., (2001) observed that water vapors compete in adsorption process and fills the adsorption sites within the pores, thus reducing the efficiency of the activated carbon. Similarly, Rengaraj, et al., (2002) showed that lower moisture content increases the rate of adsorption of contaminants resulting in better activated carbon. Hence the carbon should be activated at concentration of the activating agent that gives the lowest moisture content of the activated carbon.

3.3 Pore Volume and Porosity

The effects of varying the concentrations of the activating agent (base and acid) on the Pore volume and Porosity of the chemically activated carbon are shown in Figures 3A, 3B and 4A, 4B respectively.





Figure 3B: Effect of Concentration of Acid Activating Agent on Pore Volume of Activated Carbon







Figures 3A and 4A showed that the pore volume and porosity increased with increase in concentration of the activating agent for base activated carbon. These results were in agreement with those of Ahmad, et al., (2015) in the activation of raw date palm fronds carbon with $ZnCl_2$ where mesopore volume increased with increase in $ZnCl_2$ concentration; Owabor & Iyaomolere, (2013) who observed increase in the bulk volume (due to increase in pore volume) and porosity (due to elimination of large amount of internal carbon mass) with increasing concentration of activating agent for periwinkle shell carbon activated with $ZnCl_2$ within the concentration range considered. The increased porosity of pores created by the reactant as a result of the spaces left by zinc chloride after washing.

Figures 3B and 4B showed that the pore volume and porosity decreased with concentration for acid activated carbon. Similar results were obtained by Ugwoha, et al., (2017) for periwinkle shell carbon activated with phosphoric acid for the removal of ammonia in industrial waste water.

3.4 Ash Content

The effects of varying the concentrations of the activating agent on the ash content of the chemically activated carbon are shown in Figure 5A and 5B.





Figure 5A showed that the ash content increased, decreased after 0.25 mol., then increased after a concentration of 0.45 mol. of the activating agent for base activated carbon. A similar trend was obtained for periwinkle shell carbon activated with $ZnCl_2$ by Owabor & Iyaomolere, (2013) where the ash content increased, reached an optimum point then declined as the impregnation ratio increased. The increase in ash content was the result of increased formation of insoluble inorganic compounds with increase in impregnation ratio, while the drop observed was due to low char burn off at the respective impregnation ratio. Figure 5B showed that the ash content increased continuously with concentration of activating agent for acid activated carbon. This trend is consistent with results of Adel *et al*, (2010) where ash content increased with concentration for carbon produced from rice husk and activated with HCl. Ash content of an activated carbon is the reminant of the carbonaceous portion or the undesirable inorganic residues (silica, alumina, iron etc) that remains after carbonization. Hence high ash content raw materials contain high level of impurities that lead to blocking of pores during the activation process, thus reducing surface area and overall activity of the activated carbon. Materials with the lowest ash content are therefore the most active (Khalili, et al 2000). Therefore, the carbon should be produced at activating agent concentration that gives the lowest ash content which for base and acid activated carbons was 0.1 mol.

3.5 Iodine Number

The effects of varying the concentrations of the activating agent (base and acid) on the Iodine number of the chemically activated carbon are shown in Figure 6A and 6B.



agent on Iodine Number of Activating agent

Figure 6A showed that the iodine number increased, decreased after 0.25 mol. then increased after 0.45 mol. concentration of the activating agent for base activated carbon. Similar results were obtained for periwinkle shell carbon activated with $ZnCl_2$ by Owabor & Iyaomolere, (2013) where the amount of iodine adsorbed increased rapidly with increasing $ZnCl_2$ impregnation, attained an optimum then reduced. The increase in the iodine number was due to increase in pore formation and hence adsorptive capacity of the activated carbon as impregnation increased, while the drop may be due to abundant increase in the pore volume which eventually resulted to internal collapse of the pores. Similar fluctuations in iodine number with activating agent concentration was obtained by Subramani & Revathi, (2015).

Figure 6B showed that the iodine number initially decreased then increased continuously after a concentration of 0.25 mol. of the activating agent for acid activated carbon. Similar fluctuations in iodine number with activating agent concentrations was reported by Adib et al., (2016) for sugarcane bagasse carbon activated with phosphoric acid.

Iodine number is the amount of iodine in milligrams, adsorbed per gram of carbon when the equilibrium concentration (Ce) of iodine is 0.02M. Hence it is a measure of the iodine adsorbed in the pores of the activated carbon and an indication of the pore volume available in the activated carbon. Therefore, the carbon should be activated at concentration that gives the highest iodine value.

3.6 Surface Area

The effects of varying the concentrations of the activating agent on the Surface area of the chemically activated carbon are shown in Figure 7A and 7B.



Figure 7B: Effect of Concentration of Acid Activating Agent on Surface Area of Activated Carbon

Figure 7A showed that the surface area initially increased then decreased continuously after a concentration of 0.25 mol. of the activating agent for base activated carbon. These trends are in agreement with the work of Ahmad, et al., (2015), an initial increase in surface area with increase in concentration of $ZnCl_2$ which decrease at higher concentrations due to the destruction of the porous structure at high concentrations.

Figure 7B showed that surface area increased continuously with concentration of the activating agent for acid activating agent. Similar results were obtained by Kwaghger & Ibrahim (2013) and Yakout & El-Deen, (2016) which reported the surface area of activated carbon from mango nuts using HCl and activated carbon from olive stone using phosphoric acid increased with increase in activating agent concentration. The increasing trend of surface area observed was due to carbon gasification enhanced by increase in impregnation ratio and residence time, causing the removal of carbon atoms on pore walls, resulting in increased surface area (Teng, et al., 1996).

4. CONCLUSION

The effects of the activation method and concentrations of the chemically activated periwinkle shell carbon were investigated. Periwinkle shells were processed, carbonized and activated using the physical and chemical methods. The physical activation was performed using steam while the chemical activation was performed using base ($ZnCl_2$ and $CaCl_2$) and acid (H_2SO_4 and HCl). The activated carbons were characterized to determine its

physio-chemical properties, the effect of varying concentration of the activating agent of the chemically activated carbon was also investigated. The results showed that the chemical activated carbon had better properties compared to the physical activated carbon; higher bulk density, pore volume, porosity, iodine number and surface area. Periwinkle shell carbon activated with $ZnCl_2$ was the best with the highest values of these properties. The effect of activating agent concentration showed that the bulk density, pore volume and porosity increased with concentration for base activated carbon and decreased with concentration for acid activated carbon. The moisture content and iodine number fluctuated with concentration of the activated carbon. The surface area increased with concentration for acid activated carbon but fluctuated for acid activated carbon. The surface area increased with concentration for acid activated carbon but fluctuated for base activated carbon. Periwinkle shell carbon chemically activated with $ZnCl_2$ was the best activated carbon with highest density, pore volume, porosity, iodine number and surface area.

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