CONTINENTAL J. APPLIED SCIENCES
Nuhu and Oluwadayo (2017) 12 (1): 62 – 74 DOI: 10.5281/zenodo.546470

Research Article

The Synthesis of Activated Carbon from Coconut Shell for an Adsorbent Using Iron Chloride

Nuhu, Steven Kuba[1] and Oluwadayo, Francis Asokogene[2]
[1]Department of Minerals and Petroleum Resources Engineering, School of Engineering, Plateau State Polytechnic, B/Ladi, P. M. B. 02023, Via Bukuru, Jos, Nigeria. [2]Department of Minerals and Petroleum Resources Engineering Technology, Auchi Polytechnic, Auchi, Edo State, Nigeria
Corresponding author: kubsteven3@mail.com

Abstract

Coconut shell obtained from Chorbe in Jos, Plateau state, Nigeria was carbonized and char formed at a temperature of 655°C. The char was ground and sieved into different sizes of 7, 14, 25 and -25mm. These sizes were activated using iron chloride solution and then treated with hydrochloric acid, sodium hydroxide and ammonia solution and dried in a furnace to a temperature of 750°C to increase the pores in each of the sizes. The bulk densities of 7, 14, 25 and -25mm particle sizes were calculated and found to be 0.399, 0.587, 0.619 and 0.383g/cm^3 in that order. While the corresponding values for the pore volume and porosity were obtained to be 248.07, 212.45, 204.80 and 247.73cm^3 and 77.8, 74.9, 72.22 and 77.0% respectively. The percentage yield was also found to be 97.04, 65.80, 62.50 and 100.0% corresponding with particle sizes of 7, 14, 25 and -25mm. When proximate analysis was conducted on the four different particles sizes, the results of moisture content, volatile matter, ash content and fixed carbon were obtained to correspond with 14.84%, 22.4%, 34.25%, 15.8%: 22.14%, 22.4%, 29.4%, 57.72%: 0.8%, 1.4%, 1.3%, 7.8% and 62.21% 53.8%, 35.50%, 18.7% respectively. The overall result shows that the activated carbon with the four different particle sizes are good but best with 7 and -25mm particles sizes for water treatment, purification of gases, chemicals and other gaseous and liquid substances.

KEY WORDS: Carbonization, Activation, Iron chloride, Activated carbon and Porosity

Received: 31 January 2017 Accepted: 10 March 2017

Introduction

The ever increasing in population has stimulated the growth of various industries leading to discharge of pollutants into water bodies. Water treatment processes such as sedimentation, filtration, clarification, coagulation, flocculation, chemical oxidation, adsorption and biodegradation has been in practice to address the persistent pollution of water bodies. Among these methods, chemical adsorption is the technology employed in the removal of two types of impurities from water sources, which include; suspended solids and microbial pathogens present in the water. However, as industrialization and high-input agriculture has been extended into the developing world, chemical impurities such as pesticides, herbicides, fertilizers and other harmful substances have found their way into the drinking water supplies and have been linked to severe ill health-related issues. The removal of these chemicals, suspended solids and microbial pathogens employs the use of activated carbon obtained from some agricultural wastes for the treatment of waste water (United States Environmental Agency, 2012).

Nowadays, carbon has been one of the excellent elements which have changed material science. A good porous activated carbon is gotten from carbon with better properties for wide range of industrial applications. Activated carbon is the usual name used for a group adsorbing substances of crystalline form having a wide internal pore structures that makes the carbon more adsorptive (Shoba-Jhadhav, 2008). These properties are obtained when char is subjected to controlled gasification by oxidizing gases or when a raw material impregnated with dehydrating agent is subjected to carbonization. The feed for the production of activated carbon are the types with high carbon but less inorganic content such as wood, lignite, peat and coal (Guo and Lue, 2009). Apart from these, much agricultural wastes and by-products such as macadamia nut shell, paper mill sludge and peach stones have successfully been converted to activated carbon. In Nigeria there are potential raw materials for the production of the activated carbon such as coconut shell, wood etc. Khalili *et al*. (2000) and Shoba-Jhadhav (2015) explained that, one local agricultural waste which is coconut shell is used to produce an activated carbon due to its availability and inexpensiveness with high carbon and low inorganic content.

A number of researchers have been reported to have used coconut shell as raw material. Table 1.0 summarizes their works with reference to the raw materials they used, methods and their application of activated carbon produced.

Table 1.0: Summary of Earlier Work on Activated Carbon Using Coconut Shell

Author	Year	Raw Material	Method of Activation	Application
Mozammel *et al*.	2002	Coconut shell	$ZnCl_2$	Iodine
McEnancy& Devaston	2002	Coconut shell	$ZnCl_2$ Physical	Phenol & Dry Nitrogen
Baker *et al*.	2002	Coconut shell	(N_2 gas)	Adsorption
Rodriguez-Reinoso *et al*.,	2005	Coconut shell	$ZnCl_2$ and CO_2	Phenol Methylene Blue

Source: (Nurul'ain, 2007)

By now many researches have been conducted on the activation of carbon to improve its local production and application. One of the fastest growing areas where activated carbon is utilized is in the environmental application in the areas of wastewater treatment where it is used for purification, decolourization and the removal of toxic organics, heavy metal ions such as copper, zinc, chromium and mercury. Only few researches have used activated carbon on the removal of cyanide pollutants (Kim *et al*., 2006).

The surface oxygen functional groups can be easily introduced to the carbon by different activation methods including dry and wet oxidation agents. Dry oxidation methods involve the reaction with hot oxidizing gas such as steam and carbon (IV) oxide at temperature above 700^0C (Smisek and Cerney, 2000). While wet oxidation methods involves the reaction between the carbon surface and solutions of oxidizing agents such as phosphoric acid (H_3PO_4), nitric acid (HNO_3,) hydrogen peroxide (H_2O_2), zinc chloride ($ZnCL_2$), potassium permanganate ($KMNO_4$), ammonium persulphate $(NH_4)_2SO_8$), potassium hydroxide (KOH), etc. From the above oxidizing agents, phosphoric acid and zinc chloride are usually used for the activation of lignocelluloses materials which have been carbonized (Puziy *et al*., 2002). On the other hand, potassium hydroxide is usually used to activate coal or char. It has been reported that zinc chloride produces activated carbon with higher specific area than that produced by phosphoric acid. However, the use of phosphoric acid in activation is widely preferred over zinc chloride because of its offensive environmental impact and the activated carbon produced from it cannot be used in food and pharmaceutical industries (Moreno-Castilla *et al*., 2001). A large variety

of agricultural wastes and by-products have been examined as cellulosic precursors for the manufacture of activated carbon including hard wood and bituminous coal. These precursors encompass coconut shell and wood, olive stones, bagasse, pecan shells, palm seed, apple pulp, rubber seeds and molasses (Rodriguez-Mirasol *et al*., 2003). Carbonaceous materials such as coal, wood and peat are usually used for the production of active commercial carbons.

Due to increase need for activated carbon in our society and the high cost of raw materials for its production, many researchers have attempted various wastes such as tyres resins, agricultural by-products; and dried sewage sludge as raw materials and proposed new production methods for activated carbon with potential applications in pollution control. The choice of a cheap precursor for the production of activated carbon profoundly increases savings in the production and a way of utilizing the waste material, hence reducing its disposal problem (Rozada *et al.*, 2003).

Therefore, this research is aimed at using iron chloride ($FeCl_3$) for the synthesis of coconut shell into activated carbon.

This research was aimed at the production of activated carbon from coconut shell in order to:
(i) Test for the activity (porosity and strength) of iron chloride instead of the zinc chloride and phosphoric acid which pollute the environment and become harmful to the filtrate when used in the purification of waste water, gasses, pharmaceuticals etc.
(ii) Cut down the cost of importing activated carbon.
(iii) To utilize the abundant waste agricultural resources
(iv) To help in the cheap purification and re-utilization of used or waste water as encouraged by sustainable development goals.

Research Methodology

The coconut shell samples were obtained from a local seller and processor of coconut at Chorbe in Jos, Plateau State. The dirt was removed from the samples after which it was washed and sun dried for 24 hours. These samples were crushed and further dried in the furnace. The pre-treatment of the waste was done using the thermal pre-treatment; the ground coconut shell was autoclaved at 120^{o}C and 1.034 bar for 15 minutes to remove excessive moisture.

In the carbonization process, pyrolysis was performed using a muffle furnace in the absence of oxygen at a temperature of 655°C for 2½ hours. During the carbonization of the coconut shell, it was expected that volatiles amounted to 60% of the mass of coconut shells on dry weight basis be released to the atmosphere, yielding 40% of coconut shell mass of charcoal. The volatile released during the carbonization process was methane, carbon dioxide and a wide range of organic vapours. The char was then crushed and separated by sieving into different particle sizes using four different sieves (17, 14, 25 and -25mm). A weight of 50g char was weighed from each of the four sieves and poured into four different beakers where it was soaked by a chemical solution of $FeCl_3$ for 8 hours to become an activated carbon. They were then separately rinsed, dried, weighed and recorded. The activated carbon in each of the beaker was further treated with 0.1M hydrochloric acid (HCL), 0.1M sodium hydroxide (NaOH) and 1.0M ammonia solution (NH4). The resulting activated carbon in each of the four beakers were washed with distilled water then weighed and dried in the furnace at 110°C for 2 hours. It was then poured into four separate crucibles and kept in a muffle furnace at 750°C for2 hours to increase the porosity of the activated carbon. After removing them from the furnace they were allowed to cool, weighed and recorded each. They were then packed in different dry containers.

Determination of Bulk Density

Firstly, the mass of the beaker was measured, and then the known sample of the activated carbon was poured into the cylinder and weighed. It was transferred into an aluminium plate and then dried in a muffle furnace at a temperature of 105°C for 1 hour. After drying, the weight of the sample was measured and recorded. This was done for each of the four samples.

The test of bulk density represents the flow consistency and packaging quantity of solid sample. Bulk density is defined as weight per unit volume of material.

$$\text{Bulk Density (BD)} = \frac{M_2 - M_1}{V}:$$

Where: DB = bulk density (kg/m^3)

M_1 = mass of the cylinder (g)

M_2 = mass of measuring cylinder + its content (g)

V = volume of the measuring cylinder in litres (L)

Determination of Pore Volume

A mass of 50g of the char was saturated with a known volume of water and the total volume weighed and recorded. This mixture was dried in a muffle furnace, when cooled the difference in weight of volume was weighed and recorded. The procedure was repeated on all the other particle sizes.

Pore volume = Volume at saturation – volume at furnace dry

Determination of Porosity

Porosity, also known as void fraction, is a measure of the empty spaces in a material, and is a fraction of the volume of voids over the total volume, between 0 and 1 or as a percentage between 0 and 100%. The porosity of a medium tells the fraction of void spaces in the material where the void may contain substances such as air or water.

$$\text{Porosity (P)} = \frac{V_P}{V_T} \times 100 \quad \text{(Ng and Chong, 2002)}$$

Where: V_p = total pore volume of sample
V_T = total volume of sample

But, Total Volume of sample = $\pi r^2 h$:
Where: h = height of the sample in the cylinder
r^2 = radius of the cylinder

Determination of Percentage Yield

This is the ratio of the quantity of feed sample first introduced for activation and the quantity of the product after activation. Percentage yield is used in a place where chemical transformation occurs.

$$\text{Percentage Yield} = \frac{\text{Amount of sample introduced}}{\text{Amount of sample obtained}} \times 100$$

Determination of Moisture Content

A sample of 1g of activated carbon was measured and then poured into a petri dish, spread nicely on the dish and then heated open in an oven at a temperature of between 105 and 110^{o}C for 1½ hours. After heating, the petri dish was removed, cooled in desiccators, measured and recorded.

$$\text{Moisture Content } (M_c) = \frac{100\,(B-F)}{(B-G)}:$$

Where: B = weight of Petri dish + original sample
F = weight of Petri dish + dried sample
G = weight of Petri dish

Determination of Volatile Matter

The weight of an empty dry crucible and its lid was taken. A weight of 1g of the sample of activated carbon was fed into the crucible, closed with the lid and inserted into a muffle furnace and then heated to 925°C for exactly 7 minutes. The crucible was removed, cooled in a dessicator and weighed.

$$\text{Volatile Matter } (V_m) = \frac{100\,(B-F) - M(B-G)}{(B-G)(100-M)}$$

(Yang and Lua, 2003)

Where: B = Mass of crucible and lid + sample before heating
F = mass of crucible and lid + content after heating
G = mass of empty crucible and lid
M = % of moisture determined above

Determination of Ash Content

The weight of a crucible was measured and 1g of sample of activated carbon was poured into it, placed in a muffle furnace and heated to 750°C for 1½ hours. The crucible was removed, cooled in a dessicator and the weight of the ash was measured and recorded.

$$\text{Ash Content}(A) = \frac{100\,(F-G)}{B-G}$$ (Elliott et al., 2009)

Where: G = mass of empty crucible
B = mass of crucible + sample
F = mass of crucible + ash sample

Fixed Carbon Determination

This is difference between hundred percent and the summation of moisture content, ash content and volatile matter.

Fixed carbon (FC) = 100 - (moisture content + ash content + volatile matter)

Results

Table 1.0: Sample Soaked in a Chemical Solution of Iron Chloride

S/N	SIEVE SIZE (mm)	SATURATED CARBON (g)	DRIED CARBON (g)
1	7	318.2	72.13
2	14	318.7	106.25
3	25	316.8	112.0
4	-25	317.0	69.27

Table 1.1: Bulk Density

S/N	MESH SIZE (mm)	BULK DENSITY (g/cm^3)
1	7	0.399
2	14	0.587
3	25	0.619
4	-25	0.383

Table 1.2: Pore Volume

S/N	MESH SIZE (mm)	PORE VOLUME (cm^3)
1	7	248.07
2	14	212.45
3	25	204.80
4	-25	247.73

In Table 1.2, it is observed that the pore volume decreases from particle sizes of 7 to 25mm and increased at particle size of -25mm. The higher the pore volume of an activated carbon the more its porosity, hence the pore volume is a function of porosity. The more volume of pores the faster and more gaseous or liquid substances are purified.

Table 1.3: Porosity

S/N	MESH SIZE (mm)	POROSITY (%)
1	7	77.80
2	14	74.90
3	25	72.22
4	-25	77.00

Table 1.3 clearly shows decrease in the porosity as the mesh size of the particles of the activated carbon decreases to 25mm particle size. Mesh sizes 7 and -25mm have the best porosity (that is more empty spaces for the passage of substances during purification).

Table 1.4: Percentage Yield

S/N	MESH SIZE (mm)	PERCENTAGE YIELD (%)
1	7	97.04
2	14	65.86
3	25	62.50
4	-25	100.0

Percentage yield is the creation of pores or chemical transformation of material after activation. Table 1.4 above shows samples from mesh size of -25mm having the highest chemical transformation followed by mesh size of 7mm. Mesh sizes of 14 and 25mm having less pore creation, meaning some of is particles are still blind.

Table 1.5: Moisture Content

S/N	MESH SIZE (mm)	MOISTURE CONTENT (%)
1	7	14.85
2	14	22.40
3	25	34.25
4	-25	15.80

Table 1.5 indicates sample sizes 14 and 25mm having the highest moisture content perhaps because more of the particles that are being blinded trapped moisture in them or

are capable of absorbing moisture and retained it. While samples of sizes 7 and -25mm have low moisture content simply because of the number of pores they have which may not allow moisture be absorbed or retained in them.

Table 1.6: Volatile Matter

S/N	MESH SIZES (mm)	VOLATILE MATTER (%)
1	7	22.14
2	14	22.40
3	25	29.40
4	-25	57.72

The volatile matter in the particles sizes of 7 and 14mm in Table 1.6 are fewer than those in particle size -25mm. Looking at the pattern above, increase in particle size decreases the amount or percent of the volatile matter.

Table 1.7: Ash Content

S/N	MESH SIZES (mm)	ASH CONTENT (%)
1	7	0.8
2	14	1.4
3	25	1.3
4	-25	7.8

The lower the ash content in an activated carbon the more effective it is used as an adsorbent or the higher the mechanical strength. Table 1.7 shows that samples of size 7mm has lower ash content hence higher mechanical strength, followed by that of 25mm and 14mm with relatively lower ash content which makes its desirable for activated carbon. Particle size -25mm with the highest ash content but still within the allowable limit of <8.0% according to Nurul'ain (2007) will equally not reduced mechanical strength nut might not be as strong as others.

Table 1.8: Fixed Carbon

S/N	MESH SIZES (mm)	FIXED CARBON (%)
1	7	62.21
2	14	53.80
3	25	35.50
4	-25	18.70

The more the fixed carbon in an activated carbon the higher the purification effectiveness or strength of that activated carbon. Table 1.8 depicts that sample of size 7mm has the highest fixed carbon which clearly tells that it can give high purification effectiveness. The purification strength reduces as the particle size of the activated carbon reduces.

Conclusion

The study used many experimental techniques like porosity, percentage yield, bulk density, pore volume and proximate analysis on different particle sizes of samples of activated carbon produced from coconut shell.

The bulk density of the particle sizes of 14 and 25mm are quite higher than the other particle sizes thus implying better flow consistency and packaging quantity of solid fuel material.

The pore volume and the porosity which are some of the most important parameters in an activated carbon production were studied. The activated carbon with particle sizes of 7 and -25mm have higher pore volumes and porosity respectively. These are very important in the speed and quantity of the purification of substances. The percentage yield in Table 1.4 equally confirms the activity of the chemical transformations on particle sizes of 7 and -25mm having more pores opened.

Proximate analysis of activated carbon provided a good idea about the physical properties of the activated carbon produced. From these analyses it was found out that, the moisture content of activated carbon with particle sizes of 7 and -25mm are lower probably because of the higher pore volumes created by the activation which may not retained any moisture. The volatile matter of the activated carbon is minimal in particle sizes 7 and 14mm, in this case, perhaps because of the coarse nature of the particle sizes. The ash content here is very low in particle size 7mm which makes the activated carbon of this particle size having higher mechanical strength than others. As it is said the more

the fixed carbon in an activated carbon the higher its purification effectiveness or strength. Particle sizes of 7 and 14mm have higher amounts of fixed carbon making them as having more purification strength or better adsorbent than others.

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