CHAPTER ONE

INTRODUCTION

1.1 BACKGROUND OF STUDY

Activated carbon, also widely known as activated charcoal or activated coal is a form of carbon which has been processed to make it extremely porous and thus to have a very large surface area available for adsorption or chemical reactions (Mattson *et al.,* 1971). The word active is sometimes used in place of activated. It is characterized by high degree of micro porosity. A gram of activated carbon can have a surface area in excess of 500 m^2. Sufficient activation for useful applications may come solely from the high surface area, though further chemical treatment generally enhances the adsorbing properties of the material. Activated carbon is most commonly derived from charcoal.

Waste biomass is getting increasing attention all over the world for activated carbon development as it is renewable, widely available, cheap and environmentally friendly resource. The common method of development is thermochemical (*Kumar et al.,* 2005). The main concern is the removal of chemical component by adsorption from the liquid or gas phase (Bansal *et al.,* 1988). Today, activated carbon has been produced from various biomass such as corncob, rice husk, cherry stones, coconut shells, palm shells, to mention but a few.

Preparation of activated carbon with ultra-high specific surface area from biomass such as lignin, corncob, cornstalk, dates, etc., has attracted much attention. Among these carbon sources, corncob is a good precursor for preparing carbon with ultra-high specific surface area (Li, 2007). The carbons prepared from corncob have been used in wastewater treatment such as removal of organic pollutants (Sun *et al.,* 2006).

However, a comprehensive study of activating corncob with different activation strategies to prepare carbon with ultra-high specific surface area and pore volumes, and their subsequent performance in water purification as the impurity adsorption has not to our knowledge been reported. Therefore, in this study we report the synthesis of ultra-high surface area carbon materials using two preparation strategies namely, chemical activation procedure using a chemical activator such as ammonium sulphate $((NH_4)_2SO_4)$ and microwave-synthesized activation procedure. We also report the adsorption capacity of those carbons for water purification.

To prepare activated carbon, conventional heating method is usually adopted, in which the heat is produced by electrical furnace. However, in some cases, the thermal process may take several hours, even up to a week to reach the desired level of activation (Yuen *et al.,* 2009). Another problem related to the furnace is that the surface heating does not ensure a uniform temperature for different shapes

and sizes of samples. This generates a thermal gradient from the hot surface to the kernel of the sample particle, blocks the effective diffusions of gaseous products to its surroundings and finally results in activated carbon quality decrease (Peng *et al.,* 2008). Furthermore, there is a considerable risk of overheating or even thermal runaway (exothermic process) of portion of sample, leading to the complete combustion of the carbon (Williams *et al., 2008*).

Recently, microwave has been widely used in preparation and regeneration of activated carbon. The main difference between microwave devices and conventional heating systems is heating pattern. In microwave device, the energy is directly supplied to the carbon bed. The conversion of microwave energy is not by conduction or convection as in conventional heating, but by dipole rotation and ionic conduction inside the particles (Jones, 2002). Therefore, the treatment time can be significantly reduced through microwave heating.

1.2 STATEMENT OF PROBLEM

In recent years, increasing awareness of environmental impact of organic and inorganic compounds has prompted the purification of waste water prior to discharge into natural waters. A number of conventional treatment technologies have been considered for treatment of waste water contaminated with organic substance. Among them, the adsorption process has been found to be the most

effective method while activated carbon is regarded as the most effective material for controlling this organic load. Common active carbons available are usually developed by thermochemical means using activating agents and heating ovens, thus producing activated carbons which take a longer time with limited pore structures. With the advent of microwave technology, a better and efficient activated carbon can be produced within a short period and a cheaper cost.

1.3 OBJECTIVE OF THE RESEARCH

The aim of this research project is to determine and compare the performance of chemically and microwave synthesized activated carbon from corn cob.

1.4 SIGNIFICANCE OF THE RESEARCH

When this research project is successfully completed, it will provide the following benefits:

i. Corn cobs are abundant in Nigeria.

ii. Encourage the establishment of industries that will use Agricultural waste materials to produce activated carbon.

iii. It will create job opportunities, thereby reducing unemployment in the country.

iv. It will attract foreign exchange for Nigeria as activated carbon has very wide industrial applications.

1.5 SCOPE OF RESEARCH

This research work focuses on the following:

i. Preparation of activated carbon from corn cob by thermal and microwave means

ii. Comparative study of the adsorption capacities of chemically and microwave synthesized activated carbon.

CHAPTER TWO

LITERATURE REVIEW

2.1. ADSORPTION

The term adsorption refers to the accumulation of a substance at the interface between two phases such as solid and liquid or solid and gas. The substance that accumulates at the interface is called 'adsorbate' and the solid on which adsorption occurs is 'adsorbent'. Although certain phenomenon associated with adsorption were known in ancient times, the first quantitative studies were reported by C.W. Scheele in 1773 (Mantell, 1951) on the uptake of gases by charcoal and clays. This was followed by Lowitz' observations who used charcoal for decolorization of tartaric acid solutions. Larvitz in 1792 and Kehl in 1793 observed similar phenomenon with vegetable and animal charcoals, respectively. However, the term 'adsorption' was proposed by Bois-Reymond but introduced into the literature by Kayser (Abrowski, 2001). Ever since then, the adsorption process has been widely used for the removal of solutes from solutions and gases from air atmosphere.

At the surface of the solids, there are unbalanced forces of attraction which are responsible for adsorption. In cases where the adsorption is due to weak van der Waals forces, it is called physical adsorption. On the other hand, there may be a chemical bonding between adsorbent and adsorbate molecule and such type of adsorption is referred as chemisorption.

2.2 ACTIVATED CARBON DEVELOPMENT

Activated carbon is nothing but carbon produced from carbonaceous source materials like corn cob, nutshells, peat, wood, coir, lignite, coal and petroleum pitch. It can be produced by any one of the following described processes:

2.2.1 PHYSICAL REACTIVATION

By this process precursor is developed into activated carbons using gases. This is generally done by using one or a combination of the following processes:

➢ *Carbonization*: Material having appreciable carbon content is pyrolyzed at temperature ranging between 600–900 °C, in the absence of oxygen (usually in inert atmosphere with gases like argon or nitrogen) using a furnace.

➢ *Activation/Oxidation*: in this process raw material or carbonized material is exposed to oxidizing atmospheres (carbon monoxide, oxygen, or steam) at temperatures above 250°C, usually in the temperature range of 600–1200°C.

2.2.2 CHEMICAL ACTIVATION

Before carbonization, the raw material can be impregnated with certain chemicals. The chemical needs to be typically an acid, strong base, or a salt (ammonium sulphate, phosphoric acid, potassium hydroxide, sodium hydroxide, zinc chloride, respectively). The role of the activating agent is to improve the pore size of the activated carbon in order to improve its adsorption capacity. After impregnation, the raw material needs to be carbonized at lower temperatures (450–900 °C). It is

believed that the carbonization / activation step proceeds simultaneously with the chemical activation. Chemical activation is preferred over physical activation owing to the lower temperatures and shorter time needed for activating material.

2.2.3 STEAM ACTIVATION

The use of steam for activation can be applied to virtually all raw materials. Varieties of methods have been developed but all of this shares the same principle of initial carbonization at 500°C to 600°C followed by activation with steam at 800°C to 1100°C. Since the overall (converting carbon to carbon dioxide) is exothermic, it is possible to utilize this energy and have a self-sustaining process. Initial, gasification of the carbonize material with steam occurs and is shown in the following reaction known as water-gas reaction.

$$C \ + \ H_2O \ \longrightarrow \ CO \ + \ H_2 \ (- 31 \ Kcal)$$

This reaction maintains temperature by partial burning of the CO and H_2

$$CO \ + \ \tfrac{1}{2}O_2 \longrightarrow \ CO \ (+ 67 \ Kcal)$$

$$H_2O + \ \tfrac{1}{2}O_2 \ \longrightarrow \ H_2O \ (+ 58 \ Kcal)$$

$$C \ + \ O_2 \ \longrightarrow \ CO_2 \ (+ 94 \ Kcal)$$

2.3 PROPERTIES OF ACTIVATED CARBON

The properties of activated carbon can be discussed under physical and chemical properties.

2.3.1 PHYSICAL PROPERTIES

The most important physical property of activated carbon is the surface area of the activated carbon. For specific applications, the surface area available for adsorption depends on the molecular size of the adsorption and the pore diameter of the activated carbon. Generally, liquid-phase carbons are characterized as having a majority of pores of gas phase adsorbents are 3mm in diameter and smaller. They require larger pores due to the essence of rapid diffusion of the liquid.

The density of activated carbon, together with its specific adsorptive capacity for a given substance can be used to determine grades of activated carbon required for an existing system.

The mechanical strength and the resistance of the particles are important where pressure drop and carbon losses are concern.

2.3.2 CHEMICAL PROPERTIES

1. IODINE NUMBER: Iodine number is defined as the milligrams of iodine adsorbed by one gram of carbon when the iodine concentration in the residual filtrate is 0.02 normal. Iodine number is the most fundamental parameter used to characterize activated carbon performance. It is a measure of activity level (higher number indicates higher degree of activation), often reported in mg/g (typical range 500–1200 mg/g). It is equivalent to surface area of carbon between 900m²/g and 1100m²/g. It is the standard measure for liquid phase applications.

2. MOLASSES NUMBER: Some carbons are more adept at adsorbing large molecules. Molasses number or molasses efficiency is a measure of the mesopore content of the activated carbon by adsorption of molasses from solution. A high molasses number indicates a high adsorption of big molecules (range 95–600). Molasses efficiency is reported as a percentage (range 40%–185%). The European molasses number (range 525–110) is inversely related to the North American molasses number.

3. TANNIN ADSORPTION: Tannins are a mixture of large and medium size molecules. Carbons with a combination of macropores and mesopores adsorb tannins. The ability of a carbon to adsorb tannins is reported in parts per million concentrations (range 200 ppm–362 ppm).

4. DECHLORINATION: Some carbons are evaluated based on the dechlorination half-life length, which measures the chlorine-removal efficiency of activated carbon. The dechlorination half-value length is the depth of carbon required to reduce the chlorine level of a flowing stream from 5 ppm to 3.5 ppm.

5. APPARENT DENSITY: Higher density provides greater volume activity and normally indicates better-quality activated carbon.

6. HARDNESS/ABRASION NUMBER: It is a measure of the activated carbon's resistance to attrition. It is an important indicator of activated carbon to maintain its physical integrity and withstand frictional forces imposed by backwashing, etc. There are large differences in the hardness of activated carbons, depending on the raw material and activity level.

7. ASH CONTENT: Ash reduces the overall activity of activated carbon and it reduces the efficiency of reactivation. The metal oxides (Fe_2O_3) can leach out of activated carbon resulting in discoloration. Acid/water soluble ash content is more significant than total ash content.

8. PARTICLE SIZE DISTRIBUTION: The finer the particle size of an activated carbon, the better the access to the surface area and the faster the rate of adsorption kinetics. In vapour phase systems this needs to be considered against pressure drop, which will affect energy cost.

2.4 STRUCTURE OF ACTIVATED CARBON

A proper glance at the molecular and crystalline structure of carbon helps to understand the structure of carbon. However, activated carbon is a micro porous inert carbon with a large internal surface and this surface, organic molecules from liquids or gases can adsorb. Adsorption is the natural phenomenon in which molecules from the gas or liquid phase are attached to the surface to the solid. Carbon material are activated by series of processes which include removal of all water (dehydration), conversion of organic matter to elemental carbon, driving off the non-carbon portion (carbonization), burning off tars and enlargement of pores (activation).

The basic structural unit of activated carbon is closely approximated by the structure of pure graphite. The graphite crystal is composed of layers of fused hexagons held by weak Van-der-waal forces. Activated carbon is a disorganized form of graphite due to impurities and the method of preparation. The structure

developed is a function of carbonization and activation temperature. In terms of pores structure, the adsorbent pores can divided into three basic classes:

- Macro pores

- Transitional or meso pores

- Micro pores

The micro pores are developed primarily during carbon activation and result in the large surface area for adsorption to occur. Activated carbons contain:

a. Bulk atoms that are natural

b. Surface atoms that are real

c. Corner atoms that are very reactive and even react with metals.

2.5 APPLICATIONS OF ACTIVATED CARBON

The uses of activated carbon products are diverse as they are used in virtually every aspect of life. They are important and hence cannot be overemphasized.

Some of these applications include:

a. ANALYTICAL CHEMISTRY: Activated carbon, in 50% w/w combination with celite , is used as stationary phase in low-pressure chromatographic separation of carbohydrates (mono-, di-trisaccharides) using ethanol solutions (5–50%) as mobile phase in analytical or preparative protocols.

b. ENVIRONMENTAL APPLICATIONS: Activated carbon is usually used in water filtration systems. Carbon adsorption has numerous applications in removing pollutants from air or water streams both in the field and in industrial processes such as: Spill cleanup, Groundwater remediation, Drinking water filtration, Air purification and Volatile organic compounds capture from painting, dry cleaning, gasoline dispensing operations & other processes. Activated carbon is also used for the measurement of radon concentration in air.

c. MEDICAL APPLICATIONS: Activated carbon is used to treat poisonings and overdoses following oral ingestion. It is thought to bind the poison and prevent its absorption by the gastrointestinal tract. In cases of suspected poisoning, medical personnel administer activated carbon on the scene or at a hospital's emergency department. Dosing is usually 1 gram/kg of body mass (for adolescents or adults, give 50–100 g), usually given only once, but depending on the drug taken, it may be given more than once.

d. FUEL STORAGE: Research is being done in testing various activated carbons' ability to store natural gas and hydrogen gas. The porous material acts like a sponge for different types of gases. The gas is attracted to the carbon material via Van der Waals forces. Some carbons have been able to achieve bonding energies of 5–10 kJ per mol. The gas may then be desorbed

when subjected to higher temperatures and either combusted to do work or in the case of hydrogen gas extracted for use in a hydrogen fuel cell. Gas storage in activated carbons is an appealing gas storage method because the gas can be stored in a low pressure, low mass, low volume environment that would be much more feasible than bulky on board compression tanks in vehicles.

e. GAS PURIFICATION: Filters with activated carbon are usually used in compressed air and gas purification to remove oil vapors, odors, and other hydrocarbons from the air. Activated carbon filters are used to retain radioactive gases from a nuclear boiling water reactor turbine condenser. The air vacuumed from the condenser contains traces of radioactive gases. The large charcoal beds adsorb these gases and retain them while they rapidly decay to non-radioactive solid species. The solids are trapped in the charcoal particles, while the filtered air passes through.

f. CHEMICAL PURIFICATION: Activated carbon is commonly used to purify solutions containing un-wanted colored impurities such as during a recrystallization procedure in Organic Chemistry.

g. Distilled alcoholic beverage purification: Activated carbon filters can be used to filter vodka and whiskey of organic impurities which can affect color, taste, and odor. Passing an organically impure vodka through an

activated carbon filter at the proper flow rate will result in vodka with an identical alcohol content and significantly increased organic purity, as judged by odor and taste.

h. MERCURY SCRUBBING: Activated carbon, often impregnated with iodine or sulfur, is widely used to trap mercury emissions from coal-fired power stations, medical incinerators, and from natural gas at the wellhead.

CHAPTER THREE

MATERIAL AND METHOD

In carrying out this research work the following activities work performed:

   i.   Sourcing of corn cob.

  ii.   Activated carbon preparation from corn cob.

 iii.   Proximate analysis of activated carbon.

 iv.   Determination of the adsorption capacity of the developed activated carbon.

  v.   Comparison of the adsorption capacities of the chemically and microwave activated carbon.

3.1 SOURCING OF RAW MATERIAL

Material & Equipments used

- ➢ Hand glove
- ➢ Poly-ester bag
- ➢ Water
- ➢ Oven
- ➢ Mechanical Shaker

Procedure

Corn cobs were picked from a local market waste bin at Ihiagwa market square in owerri,Imo state, Nigeria using a hand glove and gathered into a poly-ester bag. The precursor was first washed with water to remove dirt. It was then sun dried for 6 hours after which it was it was dehydrated in an oven at a temperature of $105^{0}C$ over night to ensure that there is no after water residue in the precursor. The precursor was grounded to a particle size of -6 to $+40$mesh.

3.2 ACTIVATED CARBON PREPARATION FROM CORN COB

3.2.1 CHEMICAL ACTIVATION

Material & Equipments used

- ➢ Electronic weighing balance
- ➢ Furnace
- ➢ Beaker
- ➢ Spatula
- ➢ Mechanical mixer
- ➢ Mechanical Shaker
- ➢ Oven
- ➢ Plastic containers

Reagent

- ➢ Distilled Water
- ➢ Ammonium Sulphate (activation chemical)

Procedure

100 grams of the precursor was carbonized at a temperature of 700^0C for 2h in a stainless steel vertical tubular reactor placed in a furnace. The char produced was then added with an aqueous solution of ammonium sulphate (activation chemical) made by dissolving 50g of ammonium sulphate in 300ml of distilled water. The mixture was mixed in a mechanical mixer for 1 hrs to ensure the mixture was properly mixed. After that, the mixture was dehydrated in an oven at a temperature of 105^0C for 2hrs. The activated product was then cooled to room temperature and washed with hot distilled water to remove any undiluted residue of ammonium sulphate. Then, the precursor was dried in an oven at temperature of 105^0C for

1hrs. Finally, the dried precursor was ground and sieved to get the particle size of 125–250 μm and stored in plastic containers for further use.

3.2.2 MICROWAVE ACTIVATION

Material & Equipments used

- Electronic weighing balance
- Furnace
- Spatula
- Beaker
- Mechanical mixer
- Mechanical Shaker
- Microwave Oven
- Oven
- Plastic containers

Reagent

- Distilled Water
- Ammonium Sulphate (activation chemical)

Procedure

100 grams of the precursor was carbonized at 700^0C for 2hrs in a stainless steel vertical tubular reactor placed in a furnace. The char produced was then added with an aqueous solution of ammonium sulphate (activation chemical) made by dissolving 50g of ammonium sulphate in 300ml of distilled water. The mixture was mixed in a mechanical shaker for 1 hr to ensure the mixture was properly mixed. After mixing, the precursor was placed in a microwave oven at a temperature of 150^0C for 1 hr. The activated product was then cooled to room temperature and

washed with hot distilled water to remove any undiluted residue of ammonium sulphate. Then, the precursor was dried in an oven at temperature of 105^0C for 1hr. Finally, the dried precursor was ground and sieved to get the particle size of 125–250 μm and stored in plastic containers for further use.

3.3 PROXIMATE ANALYSIS OF ACTIVATED CARBON

The proximate analysis of a substance is a simple means of determining the distribution of products obtained when the coal sample is heated under specified conditions. As defined by ASTM D 121, proximate analysis of activated carbon was carried out to determine the percentage by mass of the moisture content, volatile matter content, ash content and fixed carbon content.

Proximate analysis is the most often used analysis for characterizing a material in connection with their utilization.

3.3.1 Moisture Content

A given amount of each sample of activated carbon was heated in a furnace at a temperature of 105^0C for an hour. The ratio of change in weight to the original weight expressed in percentage gives the moisture content. It is given by

$$\frac{W_L}{W_O} \times 100$$

Where W_L = Weight loss (Original weight – final weight)

W_O = Original weight

3.3.2 Volatile Matter Content

A given amount of each sample of moisture-free activated carbon was heated in a furnace at a temperature of 600^0C for 10 mins in the absence of air. The ratio of

change in weight to the original weight expressed in percentage gives the volatile matter content. It is given by

$$\frac{W_L \times 100}{W_O}$$

Where W_L = Weight loss (Original weight – final weight)

W_O = Original weight

3.3.3 Ash Content

A given amount of each sample of moisture-free activated carbon was heated in a furnace at a temperature of 600^0C for 30 mins in the absence of air. The ratio of change in weight expressed in percentage gives the ash content. It is given by

$$\frac{W_F \times 100}{W_O}$$

Where W_F = Final weight

W_O = Original weight

3.3.4 Fixed Carbon Content

This is the residue left after the moisture, volatile and ash is given up. It is deduced by subtracting from 100, the percentage of moisture, volatile matter and ash content. The fixed carbon content (FC) is given as

$$FC = 100 - (\%\,moisture + \%\,volatile\ matter + \%\,ash)$$

3.4 DETERMINATION OF THE ADSORPTION CAPACITY OF THE DEVELOPED ACTIVATED CARBON

Material & Equipments used

- Electronic weighing balance
- Measuring cylinder
- Spatula
- Funnel
- Beaker
- Filter Paper
- Test tube
- Colorimeter

Reagent

- Distilled Water
- Vanadomolybdophosphoric acid

Adsorbate used

- Potassium Phosphate

3.4.1 CHEMICAL ACTIVATION

Procedure

This was carried out to determine the adsorption capacity of the developed activated carbon rate of the adsorbent dose and time were varied. In this experiment, standard phosphate solutions were prepared by dissolving 1g of anhydrous potassium phosphate in distilled water (*Meites*, 1963). 600 ml of the standard phosphate solutions was transferred into 3 beakers (200 ml each). One drop of vanadomolybdophosphoric acid (indicator) was also introduced to each

beaker and stirred magnetically at moderate speed (*Hisashi*, 1982). Thereafter, a dose of 1, 2 & 3g of the chemical activated carbon process was introduced into each beaker.

At time intervals of 5, 10, 15, 20, 25 & 30 mins, 5ml portion was filtered and collected into a test tube. The initial and residual phosphate concentrations were determined by the vanadomolybdophosphoric acid colorimetric method (*APHA et al,* 1992).

3.4.2 MICROWAVE ACTIVATION

This was carried out to determine the adsorption capacity of the developed activated carbon rate of the adsorbent dose and time were varied. In this experiment, standard phosphate solutions were prepared by dissolving 1g of anhydrous potassium phosphate in distilled water (*Meites*, 1963). 600 ml of the standard phosphate solutions was transferred into 3 beakers (200 ml each). 1 drop of vanadomolybdophosphoric acid (indicator) was also introduced to each beaker and stirred magnetically at moderate speed (*Hisashi*, 1982). Thereafter, a dose of 1, 2 & 3g of the microwave activated carbon process was introduced into each beaker.

At time intervals of 5, 10, 15, 20, 25 & 30 mins, 5ml portion was filtered and collected into a test tube. The initial and residual phosphate concentrations were determined by the vanadomolybdophosphoric acid colorimetric method (*APHA et al,* 1992).

3.5 COMPARISON OF THE ADSORPTION CAPACITIES OF THE CHEMICALLY AND MICROWAVE ACTIVATED CARBON

This was carried out to compare the adsorption capacities of the chemically and microwave activated carbon in order to determine which precursor has a higher adsorption capacity. This was done by comparing the results from the adsorption capacity experiment.

CHAPTER FOUR

RESULT AND DISCUSSION

In developing activated carbon from the corn cob, three process parameters were monitored. One is the proximate analysis of the activated carbon, the second is the determination of the adsorption capacity of the activated carbon and the third is the comparison of the adsorption capacities of the activated carbon. The results are shown from table 4.1 & 4.2, Fig 4.1- 4.6.

4.1 PROXIMATE ANALYSIS

The result of the proximate analysis of the chemical and microwave synthesized activated carbon is shown in table 4.1& 4.2

TABLE 4.1 PROXIMATE ANALYSIS OF CHEMICALLY ACTIVATED CARBON

PARAMETER	VALUE (%)
Moisture content	10.46
Volatile matter	29.17
Ash content	20.32
Fixed carbon	40.05
TOTAL	**100**

TABLE 4.2 PROXIMATE ANALYSIS OF MICROWAVE SYNTHESIZED ACTIVATED CARBON

PARAMETER	VALUE (%)
Moisture content	5.44
Volatile matter	21.69
Ash content	14.80
Fixed carbon	58.07
TOTAL	**100**

The moisture content of activated carbon from chemical activation process has a higher moisture content of 10.46% while microwave-synthesized activation process has a less moisture content of 5.44%.

The volatile matter content of activated carbon from chemical activation process has a higher volatile content of 29.17% while microwave-synthesized activation process has a least moisture content of 21.69%.

The ash content of activated carbon from chemical activation process has a higher ash content of 20.32% while microwave-synthesized activation process has a less ash content of 14.80%.

The fixed carbon content data show that microwave-synthesized activation process gives a higher fixed carbon of 58.07%, while chemical activation process gives 40.05% fixed carbon. This implies that the char of carbon produced by microwave-synthesized activation process are more carbonized than those of the chemical activation process during the pyrolysis operation.

4.2 DETERMINATION OF THE ADSORPTION CAPACITY RESULT

4.2.1 FOR CHEMICALLY ACTIVATED CARBON

The result for the determination of the adsorption capacity for chemically activated carbon is shown from the Fig 4.1 – 4.3. The figures show the percentage variation of the potassium phosphate removed in a given period of time.
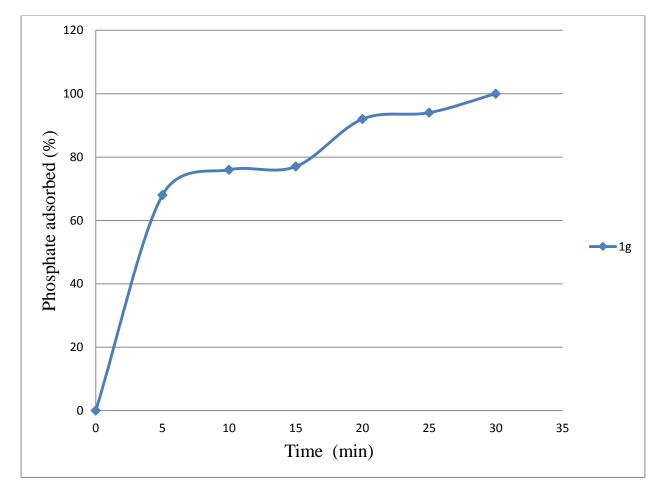

Fig 4.1: Plot of concentration of adsorbate vs time for 1g of chemically activated carbon
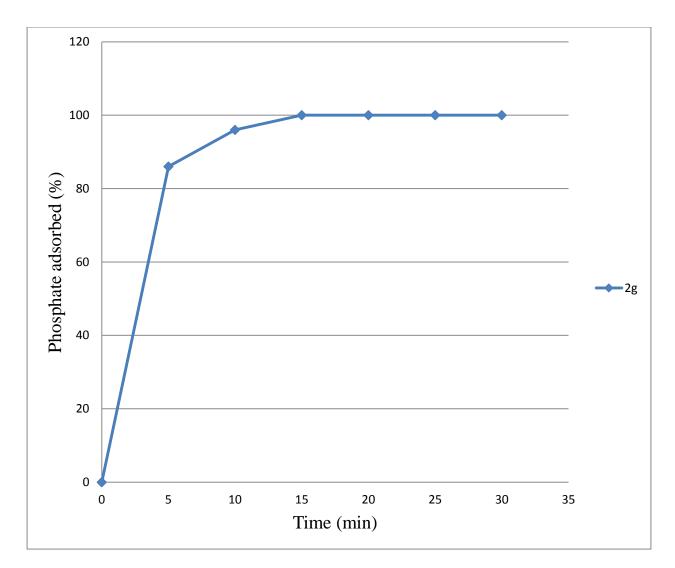
Fig 4.2: Plot of concentration of adsorbate vs time for 2g of chemically activated carbon
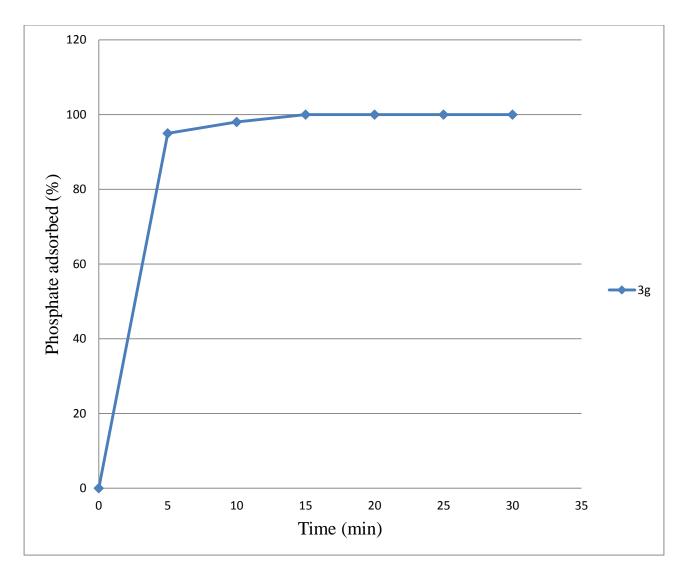
Fig 4.3: Plot of concentration of adsorbate vs time for 3g of chemically activated carbon

4.2.2 FOR MICROWAVE ACTIVATED CARBON

The result for the determination of the adsorption capacity for chemically activated carbon is shown from Fig 4.4 – 4.6. The figures show the percentage variation of the potassium phosphate removed in a given period of time.
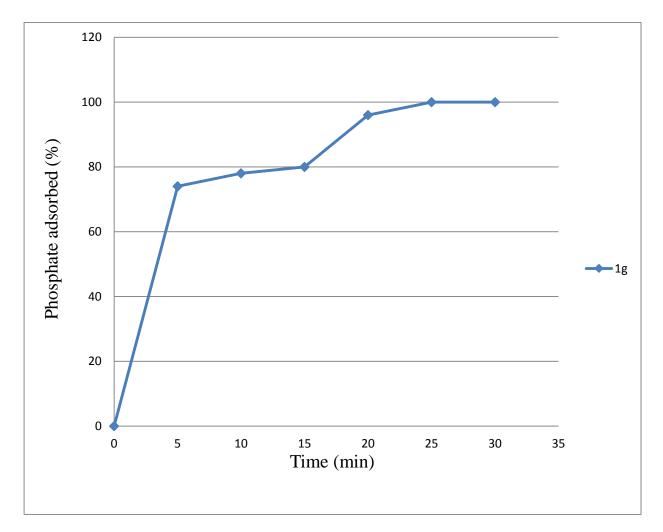

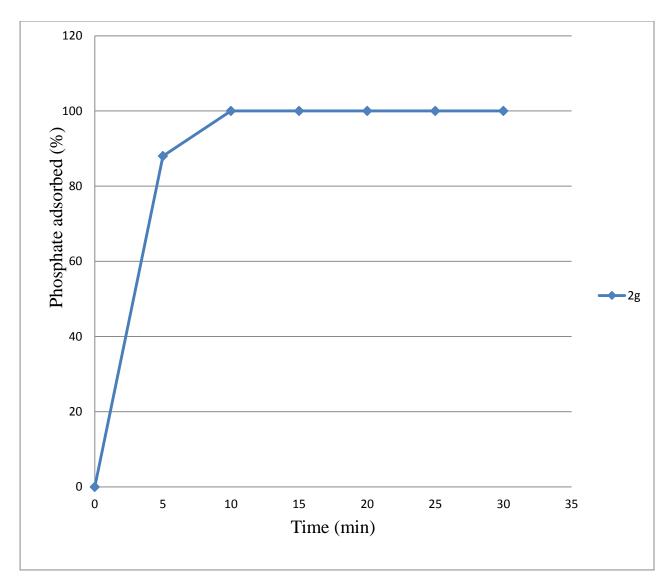Fig 4.4: Plot of concentration of adsorbate vs time for 1g of microwave-synthesized activated carbon

Fig 4.5: Plot of concentration of adsorbate vs time for 2g of microwave-synthesized activated carbon
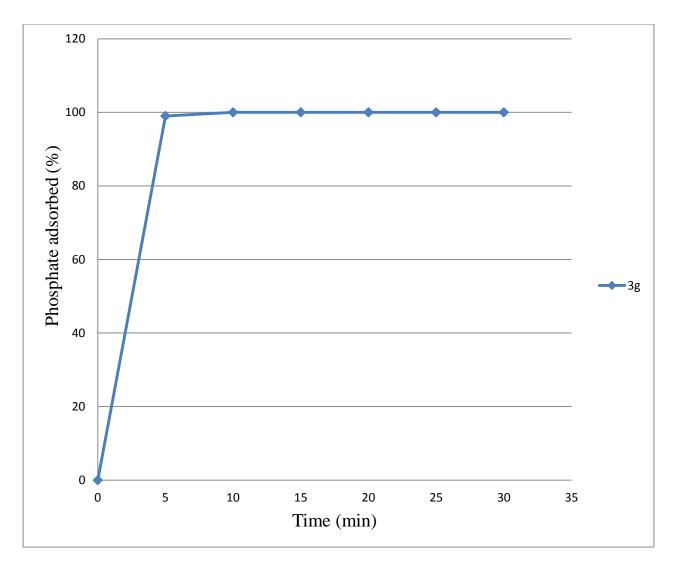
Fig 4.6: Plot of concentration of adsorbate vs time for 3g of microwave-synthesized activated carbon

From the result of the study, it was observed that the rate of phosphate removal varies with the various adsorbent doses of 1g, 2g & 3g. The adsorption capacity of the activated carbon increases as the time increases, therefore increase in time increases the adsorption capacity of the activated carbon.

4.3 COMPARISON OF THE ADSORPTION CAPACITIES OF THE CHEMICALLY AND MICROWAVE ACTIVATED CARBON

The result from the determination of the adsorption capacity of the developed activated carbon was compared as shown in fig 4.7 – 4.9 in order to know the activated carbon that has a better adsorption capacity.
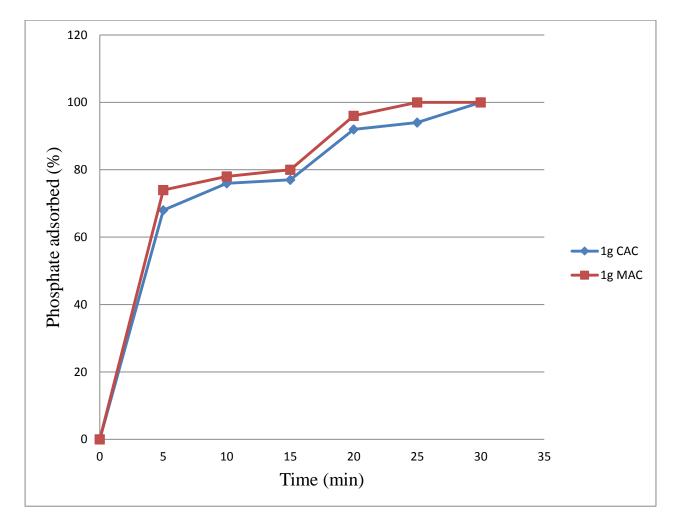

Fig 4.7: Comparison of adsorption rate of 1g of chemically activated carbon & microwave-synthesized activated carbon
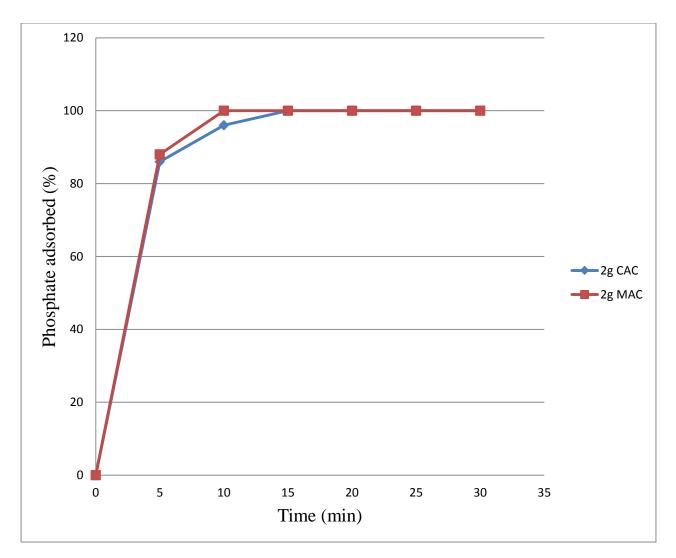
Fig 4.8: Comparison of adsorption rate of 2g of chemically activated carbon & microwave-synthesized activated carbon

Fig 4.9: Comparison of adsorption rate of 1g of chemically activated carbon & microwave-synthesized activated carbon

From the result of the study, it shows that activated carbon from microwave-synthesized activated carbon has a higher adsorption capacity than chemically activated carbon because it is assumed that microwave-synthesized activated carbon has higher porosity and large surface area compared to chemical activated carbon.

Finally the natures of the plots are indicative of the nature of formation of the layers on the adsorbent surface. The time variation curves for phosphate removal were smooth and continuous, indicating the formation of monolayer coverage on the surface of the adsorbent.

CHAPTER FIVE

CONCLUSION AND RECOMMENDATION

5.1 CONCLUSION

Having carried out this investigation on the comparative study of the performances of chemically and microwave-synthesized activated carbon from corn cob, the results of the test has shown that microwave-synthesized activated carbon has a higher adsorption capacity compared to chemical activated carbon. The best results were obtained by treating the standard phosphate solutions with 3g of microwave-synthesized activated carbon for 30mins.

This shows that method of activation can affect the properties of activated carbon produced.

5.2 RECOMMENDATIONS

Investigation made in further research in this area would earn huge foreign exchange for the country by employing the conversion of agricultural waste like banana peels, rick husks and corn cobs etc into activated carbon.

Extensive research and developmental work is needed to achieve the status of replacing the totality of activated carbons being imported into the country for diverse application in the chemical process industries, water treatment, and pharmaceutical.

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APPENDIX A

CHARACTERIZATION OF ACTIVATED CARBON

FOR CHEMICAL ACTIVATION PROCESS

MOISTURE CONTENT

Original weight of activated carbon= 9.08

Final weight of activated carbon= 8.13

Weight loss = 9.08 – 8.13 = 0.95

% Moisture = $\dfrac{W_L \times 100}{W_O}$

Where W_L = Weight loss (Original weight – final weight)

W_O = Original weight

$= \dfrac{0.95 \times 100}{9.08} = 10.46\%$

VOLATILE MATTER CONTENT

Original weight of activated carbon= 24.58

Final weight of activated carbon= 17.41

Weight loss = 24.58 –17.41= 7.17

% Volatile matter= $\dfrac{W_L \times 100}{W_O}$

Where W_L = Weight loss (Original weight – final weight)

W_O = Original weight

$= \dfrac{7.17 \times 100}{24.58} = 29.17\%$

ASH CONTENT

Original weight of activated carbon= 4.38

Final weight of activated carbon= 0.89

% Ash = $\dfrac{W_F \times 100}{W_O}$

Where W_F = Final weight

W_O = Original weight

$= \dfrac{0.89 \times 100}{4.38} = 20.32\%$

FIXED CARBON CONTENT

FC = 100 – (%moisture + %volatile matter + %ash)

= 100 – (10.46 + 29.17 + 20.32) = 40.05%

FOR MICROWAVE-SYNTHESIZED ACTIVATION PROCESS

MOISTURE CONTENT

Original weight of activated carbon= 9.01

Final weight of activated carbon= 8.52

Weight loss = 9.01 – 8.52 = 0.49

% Moisture = $\dfrac{W_L \times 100}{W_O}$

Where W_L = Weight loss (Original weight – final weight)

W_O = Original weight

$= \dfrac{0.49 \times 100}{9.01} = 5.44\%$

VOLATILE MATTER CONTENT

Original weight of activated carbon= 23.47

Final weight of activated carbon= 18.38

Weight loss = 23.47 – 18.38 = 5.09

% Volatile matter= $\dfrac{W_L \times 100}{W_O}$

Where W_L = Weight loss (Original weight – final weight)

   W_O = Original weight

= $\dfrac{5.09 \times 100}{23.47}$ = 21.69%

ASH CONTENT

Original weight of activated carbon= 4.46

Final weight of activated carbon= 0.66

% Ash = $\dfrac{W_F \times 100}{W_O}$

Where W_F = Final weight

   W_O = Original weight

= $\dfrac{0.66 \times 100}{4.46}$ = 14.80%

FIXED CARBON CONTENT

FC = 100 – (%moisture + %volatile matter + %ash)

= 100 – (5.44 + 21.69 +14.80) = 58.07%

ADSORPTION EXPERIMENT

FOR CHEMICAL ACTIVATION PROCESS

1g of activated carbon for adsorbate adsorption

Time: 5 mins

Colorimeter calibration: 100%

Adsorbate concentration: 32%

% of Adsorbate removed: $100 - 32 = 68\%$

Time: 10 mins

Colorimeter calibration: 100%

Adsorbate concentration: 24.04%

% of Adsorbate removed: $100 - 24.04 = 75.96\% \approx 76\%$

Time: 15 mins

Colorimeter calibration: 100%

Adsorbate concentration: 23.02%

% of Adsorbate removed: $100 - 23.02 = 76.98 \approx 77\%$

Time: 20 mins

Colorimeter calibration: 100%

Adsorbate concentration: 8%

% of Adsorbate removed: $100 - 8.12 = 91.88 \approx 92\%$

Time: 25 mins

Colorimeter calibration: 100%

Adsorbate concentration: 6.02%

% of Adsorbate removed: $100 - 6.02 = 93.98\% \approx 94\%$

Time: 30 mins

Colorimeter calibration: 100%

Adsorbate concentration: 0.07%

% of Adsorbate removed: $100 - 0.07 = 99.93\% \approx 100\%$

TABLE B.1: 1g of activated carbon for adsorbate adsorption

Contact time (min)	Percentage of phosphate adsorbed
	Chemical activation (%)
5	68
10	76
15	77
20	92
25	94
30	100

2g of activated carbon for adsorbate adsorption

Time: 5 mins

Colorimeter calibration: 100%

Adsorbate concentration: 13.99%

% of Adsorbate removed: 100 − 13.99 = 86.01 ≈ 86%

Time: 10 mins

Colorimeter calibration: 100%

Adsorbate concentration: 4.012%

% of Adsorbate removed: 100 − 4.012 = 95.988% ≈ 96%

Time: 15 mins

Colorimeter calibration: 100%

Adsorbate concentration: 0.02%

% of Adsorbate removed: 100 − 0.02 = 99.98% ≈ 100%

Time: 20 mins

Colorimeter calibration: 100%

Adsorbate concentration: 0.013%

% of Adsorbate removed: 100 − 0.013 = 99.99% ≈ 100%

Time: 25 mins

Colorimeter calibration: 100%

Adsorbate concentration: 0.004%

% of Adsorbate removed: 100 − 0.004 = 99.996% ≈ 100%

Time: 30 mins

Colorimeter calibration: 100%

Adsorbate concentration: 0.001%

% of Adsorbate removed: $100 - 0.001 = 99.999\% \approx 100\%$

TABLE B.2: 2g of activated carbon for adsorbate adsorption

Contact time (min)	Percentage of phosphate adsorbed
	Chemical activation (%)
5	86
10	96
15	100
20	100
25	100
30	100

3g of activated carbon for adsorbate adsorption

Time: 5 mins

Colorimeter calibration: 100%

Adsorbate concentration: 5.01%

% of Adsorbate removed: $100 - 5.01 = 94.99\% \approx 95\%$

Time: 10 mins

Colorimeter calibration: 100%

Adsorbate concentration: 2%

% of Adsorbate removed: $100 - 2 = 98\%$

Time: 15 mins

Colorimeter calibration: 100%

Adsorbate concentration: 0.08%

% of Adsorbate removed: $100 - 0.08 = 99.92\% \approx 100\%$

Time: 20 mins

Colorimeter calibration: 100%

Adsorbate concentration: 0.06%

% of Adsorbate removed: $100 - 0.06 = 99.94\% \approx 100\%$

Time: 25 mins

Colorimeter calibration: 100%

Adsorbate concentration: 0.003%

% of Adsorbate removed: $100 - 0.003 = 99.97\% \approx 100\%$

Time: 30 mins

Colorimeter calibration: 100%

Adsorbate concentration: 0%

% of Adsorbate removed: $100 - 0 = 100\%$

TABLE B.3: For 3g of activated carbon for adsorbate adsorption

Contact time (min)	Percentage of phosphate adsorbed
	Chemical activation (%)
5	95
10	98
15	100
20	100
25	100
30	100

FOR MICROWAVE-SYNTHESIZED ACTIVATION PROCESS

1g of activated carbon for adsorbate adsorption

Time: 5 mins

Colorimeter calibration: 100%

Adsorbate concentration: 26.08%

% of Adsorbate removed: $100 - 26.08 = 73.92 \approx 74\%$

Time: 10 mins

Colorimeter calibration: 100%

Adsorbate concentration: 22%

% of Adsorbate removed: $100 - 22 = 78\%$

Time: 15 mins

Colorimeter calibration: 100%

Adsorbate concentration: 20.04%

% of Adsorbate removed: $100 - 20.04 = 79.96 \approx 80\%$

Time: 20 mins

Colorimeter calibration: 100%

Adsorbate concentration: 4.05%

% of Adsorbate removed: $100 - 4.05 = 95.95\% \approx 96\%$

Time: 25 mins

Colorimeter calibration: 100%

Adsorbate concentration: 0.08%

% of Adsorbate removed: $100 - 0.08 = 99.92\% \approx 100\%$

Time: 30 mins

Colorimeter calibration: 100%

Adsorbate concentration: 0.005%

% of Adsorbate removed: $100 - 0.005 = 99.995\% \approx 100\%$

TABLE B.4: 1g of activated carbon for adsorbate adsorption

Contact time (min)	Percentage of phosphate adsorbed
	Microwave-synthesized activation (%)
5	74
10	78
15	80
20	96
25	100
30	100

2g of activated carbon for adsorbate adsorption

Time: 5 mins

Colorimeter calibration: 100%

Adsorbate concentration: 12.08%

% of Adsorbate removed: $100 - 12.08 = 87.92\% \approx 88\%$

Time: 10 mins

Colorimeter calibration: 100%

Adsorbate concentration: 0.07%

% of Adsorbate removed: $100 - 0.07 = 99.93\% \approx 100\%$

Time: 15 mins

Colorimeter calibration: 100%

Adsorbate concentration: 0.05%

% of Adsorbate removed: $100 - 0.05 = 99.95 \approx 100\%$

Time: 20 mins

Colorimeter calibration: 100%

Adsorbate concentration: 0.04%

% of Adsorbate removed: $100 - 0.04 = 99.96\% \approx 100\%$

Time: 25 mins

Colorimeter calibration: 100%

Adsorbate concentration: 0.03%

% of Adsorbate removed: $100 - 0.03 = 99.97\% \approx 100\%$

Time: 30 mins

Colorimeter calibration: 100%

Adsorbate concentration: 0.02%

% of Adsorbate removed: $100 - 0.02 = 99.98\% \approx 100\%$

TABLE B.5: 2g of activated carbon for adsorbate adsorption

Contact time (min)	Percentage of phosphate adsorbed
	Microwave-synthesized activation (%)
5	88
10	100
15	100
20	100
25	100
30	100

3g of activated carbon for adsorbate adsorption

Time: 5 mins

Colorimeter calibration: 100%

Adsorbate concentration: 1.03%

% of Adsorbate removed: 100 − 1.03 = 98.97% ≈ 99%

Time: 10 mins

Colorimeter calibration: 100%

Adsorbate concentration: 0.05%

% of Adsorbate removed: 100 − 0.05 = 99.95% ≈ 100%

Time: 15 mins

Colorimeter calibration: 100%

Adsorbate concentration: 0.03%

% of Adsorbate removed: 100 − 0.03 = 99.97 ≈ 100%

Time: 20 mins

Colorimeter calibration: 100%

Adsorbate concentration: 0.01%

% of Adsorbate removed: 100 − 0.01 = 99.99% ≈ 100%

Time: 25 mins

Colorimeter calibration: 100%

Adsorbate concentration: 0

% of Adsorbate removed: 100 − 0 = 100%

Time: 30 mins

Colorimeter calibration: 100%

Adsorbate concentration: 0

% of Adsorbate removed: $100 - 0 = 100\%$

TABLE B.6: 3g of activated carbon for adsorbate adsorption

Contact time (min)	Percentage of phosphate adsorbed
	Microwave-synthesized activation (%)
5	99
10	100
15	100
20	100
25	100
30	100