

**MODIFICATION OF SURFACE, PHYSICAL AND  
CHEMICAL PROPERTIES OF ACTIVATED CARBONS FOR  
WATER PURIFICATION**

**BY**

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## APPROVAL PAGE

This is to certify that this project was carried out by DURUAKU JACINTA NKIRUKA with registration number CHE/2007/063 for the award of bachelor of engineering degree (B.Eng.) in Chemical Engineering Caritas University Amorji-Nike Enugu.

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## **DEDICATION**

This work is dedicated to God almighty, the author and finisher of my being.

## **ACKNOWLEDGEMENT**

Success is a collective effort. In this regard, I wish to express my unending gratefulness to my lovely parents Mr. and Mrs. D. DURUAKU and my siblings for their relentless effort financially, spiritually, morally and otherwise.

My profound gratitude goes to my able supervisor, Engr. (Prof) J.I Ume for his fatherly advice, corrections and constructive criticisms.

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I stand for a sincere salute to my lecturers Dr. Gracinee Tejano, Dr. G. O Mbah, Engr. Ken Eze, Engr. Boniface Ugwu, Engr. Mrs. Odinlinye, Engr. Mrs Otegbulu and other staffs of Caritas University for their tutelage. God bless you all.

Finally, I bow in reverence with humility to thank the unchangeable and all-loving God for His mercy, favors, understanding and blessings upon my life as well as His guidance and direction throughout my stay in Caritas University.

## **ABSTRACT**

The purpose of this work was to study the effect of chemical surface properties of activated carbons for their use in water purification. To achieve this objective, activated carbon in both granular and powdered forms namely: as received and devolatilized carbons were prepared. Proximate analysis of the carbon materials was performed. Mass titration experiments were carried out to determine the point of zero charge of the activated carbon materials. Again, using an ultraviolet spectrophotometer, the adsorption of phenol on as-received and devolatilized activated carbon was investigated. Adsorption isotherms were acquired from which the monolayer adsorption capacities were calculated.

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## **CHAPTER ONE**

### **INTRODUCTION**

#### **1.1 ORIGIN AND NATURE OF ACTIVATED CARBON**

Carbon is the fifteenth most abundant element in the earth's crust and the fourth most abundant element in the universe by mass after hydrogen, helium and oxygen. Scientists, industries, and consumers use different forms of carbon and carbon containing compounds in many ways such as activated carbon or carbon in its active form which can be used to purify water, among others.

Activated carbon is a form of carbon that has been produced to make it extremely porous and thus have a very large surface area available for adsorption or chemical reactions.

It can be defined as a microcrystalline non-graphitic amorphous form of carbon which has been processed to develop a high internal porosity due to its network of inter-connecting pores.

The history of activated carbon is dated since the fifteenth century, during the time of Columbus when sailors used to blacken the inside of wooden water barrels with fire, since they observed that the water would stay fresh much longer. It is likely that people at that time proceeded by intuition only without having any

insight into the mechanism of the effect. The mechanism was recognized beginning from the eighteenth century.

In 1862, Lipscombe prepared a carbon material for purifying portable water. This development paved the way for the commercial application of activated carbon first for portable water and then in waste water sector.

## **1.2 METHODS OF MANUFACTURE OF ACTIVATED CARBON**

The methods employed in the industrial manufacture of activated carbons are numerous but consist of three main methods namely; Chemical activation, Steam activation and thermal processing techniques.

The raw materials or precursors used in the manufacture of activated carbon are as follows; Softwood, coconut shell, lignite, hardwood, grain and agro products, bituminous coal, anthracite, etc.

Chemical activation is generally used for the production of activated carbon from sawdust, wood or peat and uses chemicals for activation. Chemical activation technique involves mixing an inorganic chemical compound with the carbonaceous raw materials and the most widely used activating agents are Phosphoric acid and Zinc Chloride.

Steam activation technique is generally used for coal-based, coconut shell and grain-based activated carbons and uses gases, vapors or a mixture of both for its activation.

Thermal processing technique is a separation process that removes unwanted materials from the carbonaceous precursor used under varying heat applications.

This technique is at a lower cost compared to the two techniques above and meets all environmental standards, while others need expensive solutions to achieve the same results.

### **1.3 NEED FOR PRESENT INVESTIGATION**

The need for present investigation of this material cannot be over emphasized. This is as a result of the pressing need for treatment of waste water emanating from domestic and industrial concerns.

Activated carbon plays an important role in the purification of fluids (water), including vegetable oils used in domestic cooking and as a precursor in industrial manufacture of food products. The slow pace of technological development in the country has resulted to the expenditure of the nation's resources on importation of activated carbons to meet the demand for local chemical and process industries, as well as the demand for municipal and industrial water treatment plants.

Rapid industrialization, together with the increase in modern methods of agriculture and the increase in population, has contributed to the pollution of the ecosystem. Most of the pollutants are toxic to living organisms. It is therefore imperative that waste water has to be treated to remove the toxic materials before disposal to the environment. Most methods of treating water have some inherent shortfalls. Activated carbon treatment was therefore developed because of its effectiveness in pollutants removal, especially in water purification.

#### **1.4 OBJECTIVES AND SCOPE OF THE STUDY**

The primary objectives and scope of the present investigation include the following:

- i. Acquisition of the different types of activated carbons available to the nation's chemical industry.
- ii. Modification of the surface physical and chemical properties of the carbon material, for their use in liquid phase applications.
- iii. Determination of the physical properties of the as-received and modified activated carbon materials.
- iv. Testing the adsorption capacity of the carbon materials in adsorption processes.
- v. Evaluation of the fractional surface coverage for each carbon material.

vi. Proposal of a scheme for the optimal modification of activated carbon material for optimal application in liquid phase adsorption.

## **CHAPTER TWO**

### **LITERATURE REVIEW**

#### **2.1 PREAMBLE**

An activated carbon is a highly adsorbent carbon material obtained by heat treatment of a carbon precursor resulting in a highly porous structure with a very large surface area. It can also be define as a micro crystalline, non-graphitic amorphous form of carbon which has been processed to develop high internal porosity due to its network of inter-connecting pores. It is therefore used as an adsorbent.

#### **2.2 THE ADSORPTION PROCESS**

Adsorption is a term commonly used for several different processes involving physical as well as chemical interactions between the solid surface of a substance and dissolved specie. Adsorption occurs when molecules diffusing in the fluid phase are held for a period of time by forces emanating from adjacent surface.

Adsorption is employed in many natural, physical, biological and chemical systems and is widely used in such industrial applications as chemical synthetic water purification. The huge surface area of activated carbon gives it countless bonding sites which help the chemicals which are passed through activated carbon

surface to be attached and trapped to the surface. Activated carbon is good at trapping other carbon-based impurities (organic chemicals) as well as some inorganic chemicals like Chlorine while other chemical like Sodium, Nitrates etc that pass right through and not attracted to carbon at all. These mean that an activated charcoal or carbon filter will remove certain impurities while ignoring the others.

### **2.3 HISTORICAL BACKGROUND OF ACTIVATED CARBON**

Activated carbon was first produced on an industrial scale at the beginning of the twentieth century and major development took place in Europe.

In 1900 and 1901, a Swedish chemist by name Von Ostrejko obtained two patents covering the basic concepts of chemical and thermal of physical activation of carbon with metal chloride and with carbon dioxide and steam respectively.

Also in 1911, the Norit company, a manufacturer in Holland produced an activate carbon known as Norit and Purit in a chemische werke plant by the activation of peat with steam and the company became widely known in the sugar industry at that time, a powdered activated carbon were used mainly for decolorizing solution in a chemical and food industries.

On an industrial scale, the process of chemical activation of sawdust with Zinc chloride was carried out for the first time in a Austrian plant at Ausing in 1914 and also in the dye plant of Bayer in 1915.

In 1913, parallel to the development in Europe, in the United States of America, the first activated carbon was produced from blash ash, a waste product of soda production, after it was accidentally discovered that the ash was effective in decolorizing liquid. The use of poisonous gases in the First World War paved a way for the development and large scale production of granular activated carbons. These carbons were use in gas masks for the adsorption of poisonous gases.

After the First World War, considerable progress was made in Europe in the manufacture of activated carbons using new raw carbonaceous material such as coconut and almond shells. The treatment with Zinc chloride yielded activated carbon with high mechanical strength and high adsorptive capacity for gases and vapors.

Later in 1935 to 1940, pelletize carbons were produced from sawdust by Zinc chloride activated for the recovery of volatile solvents and the removal of Benzene from town gas.

Nowadays, the Zinc chloride of chemical activation has been largely superseded by the use of phosphoric acid.

## **2.4 RAW MATERIALS FOR THE PRODUCTION OF ACTIVATED CARBON**

The raw materials used for activated carbon production include reagents such as Zinc chloride, phosphoric acid and precursors such as charred coconut shell, palm kernel shell, bamboo, bone, rice husks etc.

## **2.5 ACTIVATED CARBON MANUFACTURING PROCESS**

Activated carbons in the form of granules, powders, fibers or beads can be produced from suitable thermosetting precursor by either thermal or chemical routes. The thermal and most common route involves a two-step process of carbonization at about 600 to 650 °C, followed by partial gasification (activation) in steam or carbon dioxide at 800 to 900 °C to develop the desired pore structure.

The sequence is intended to preserve and further the blue print for pore structure which is inherent in the raw material (and which differs with the raw material type). For the most part, the reactions are thermally driven. The second commercial route involves the reaction of a precursor, usually cellulosic, with chemical reagents such as Zinc chloride or phosphoric acid in a single heat treatment stage. In the absence of any formally agreed terminology, these two process routes will be referred as thermal and chemical activation respectively. The general features of both processes including steam activation are described below. The precise details

of methods used by different manufacturer are not publicly available for proprietary reasons. The similarities in the processes are basically the use of elevated temperature in the manufacture.

### **2.5.1 THERMAL PROCESSING TECHNIQUE**

The fixed carbon content of various raw materials used for the production of activated is shown in table 2.1.

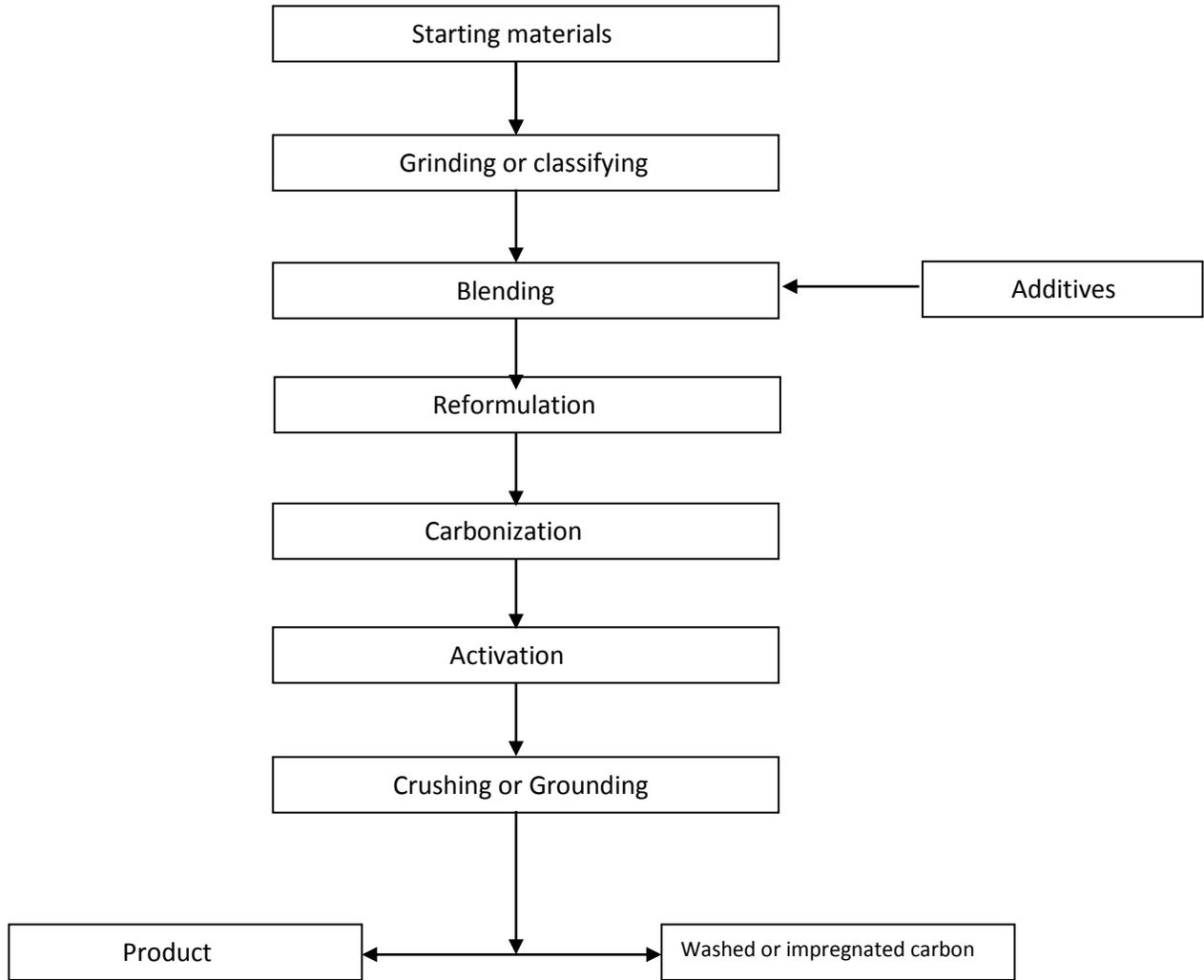
**TABLE 2.1 FIXED CARBON CONTENTS OF PRECURSORS FOR THE PRODUCTION OF ACTIVATED CARBONS**

PRECURSORS	FIXED CARBON CONTENT (%)
WOOD	35
COAL	28
LIGNITE	14
COCONUT SHELL	10
PEAT	10
GRAIN AND AGRO PRODUCTS	40
ANTHRACITE	90
OTHERS	3

The selection of the precursor essentially determines the range of adsorptive and physical properties that can be attained in the activated carbon products. Important considerations to be made in selecting a source include, Cost availability, consistency of quality and particularly for coals, peat and lignite, the mineral matter and Sulphur content. The thermal process involves two stages:

- a) Carbonization stage: This is conducted at 600 to 650<sup>0</sup>C in the essentially inert atmosphere. Between 400 and 600<sup>0</sup>C, most organic solids undergo reactions, leading to the loss of hydrogen and formation of free radical which condense to form a rigid cross-linked solid char. This process causes some increase in porosity, although this is generally insufficient for practical use, and serves to modify the pore structure inherent to the precursor as opposed to creating it. The resulting char from this carbonation stage is milled before the next step.
- b) High temperature stage: Here, the milled char is heated to an elevated temperature say between 700 and 1200<sup>0</sup>C in the presence of an oxidizing gas stream such as CO<sub>2</sub>, steam or Nitrogen. The temperature is increased slowly during this stage to allow time for each step of activation before the next stage. During the high temperature activation, the carbon particles blocking the pore are preferentially burnt off.

A generalized schematic diagram of the thermal processing route is shown in figure 2.1



**FIGURE 2.1 THERMAL PROCESSING ROUTES FOR PRODUCTION FOR ACTIVATION OF ACTIVATED CARBON**

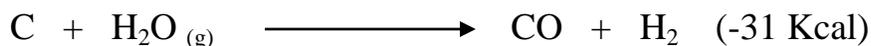
## **2.5.2 CHEMICAL ACTIVATION TECHNIQUE**

The raw materials used in chemical activation are usually sawdust and the most popular activating agent is phosphoric acid, although Zinc chloride and Sulfuric acid are well documented. Others used in the past included calcium hydroxide, calcium chloride, manganese chloride and sodium hydroxide, all of which are dehydrating agents. The raw material and reagent are mixed into a paste, dried and carbonized in a rotary furnace at 600<sup>0</sup>C. When phosphoric acid is the activating agent, the carbonized product is further heated at 800 to 1000<sup>0</sup>C during which stage the carbon is oxidized by the acid. The acid is largely recovered after the activation stage and converted back to the correct strength for reuse. The activated product is washed with water and dried. Activity can be controlled by altering the proportion of raw material to activating agent between the limit 1:5 to 1:4. By increasing the concentration of the activating agent, the activity increases although control of furnace temperature and resident time can achieve the same objective.

## **2.5.3 STEAM ACTIVATION TECHNIQUE**

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 to 600<sup>0</sup>C followed by activation with steam at 800 to 1100<sup>0</sup>C. Since the overall reaction (converting carbon to carbon dioxide) is

exothermic, it is possible to utilize these energy and have a self-sustaining process. Initially, gasification of the carbonized material with steam occurs and is shown in the following reaction known as water-gas reaction.



This reaction maintains temperature by partial burning of the CO and H<sub>2</sub>



A number of different types of kilns and furnaces can be used for carbonization/activation and include rotary (fired directly or indirectly), vertical multi-hearth furnaces, fluidized bed reactors and vertical single throat retorts.

## 2.6 PROPERTIES OF ACTIVATED CARBON

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

## **2.6.1 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 a concern.

## **2.6.2 CHEMICAL PROPERTIES**

1. IODINE NUMBER: Iodine number is defined as the milligrams of iodine adsorbed by 1gram of carbon when the iodine concentration in the residual filtrate is 0.02 normal. Iodine number is the most fundamental parameter used to characterized activated carbon performance. It is a measure of activity level often reported in milligram per gram (mg/g). It is equivalent to surface area of carbon

between  $900\text{m}^2/\text{g}$  and  $1100\text{m}^2/\text{g}$ . It is the standard measure for liquid phase applications.

2. **MOLASSES NUMBER:** Some carbons are more adept at adsorbing larger 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 indicate a high adsorption by big molecule (range 95 -600). Molasses efficiency is reported as a percentage (range 40% - 185%). The European molasses number (range 525 - 1100) 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 micro pores and mesopores adsorb tannins. The ability of a carbon to adsorb tannin is reported in parts per million concentrations (range 200ppm -362ppm).

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

5. **DECHLORINATION:** Some carbons are evaluated based on the dechlorination half-value 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 5ppm to 3.55ppm

6. **ASH CONTENT:** Ash reduces the overall activity of activated carbon. The metal oxides especially iron oxide ( $\text{Fe}_2\text{O}_3$ ) can leach out of activated carbon resulting in discoloration. Acid or water soluble ash content is more significant than total ash content.

7. **HARDNESS/ ABRASSION NUMBER:** This is a measure of the ability of the activated carbon to maintain its physical integrity and withstand frictional forces imposed by backwashing etc. There are large differences in the hardness of activated carbon depending on the raw material for its production and activity level.

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 the adsorption kinetics. In vapor phase systems, this needs to be considered pressure drop, which will affect energy cost.

## **2.7 STRUCTURE OF ACTIVATED CARBON**

The molecular and crystalline structures of carbon facilitate the understanding of the surface chemistry of carbon. A structure of carbon black makes this achievable, although carbon black has smaller internal surface area than the activated carbon.

The development of the structure of activated carbon is a function of the carbonization and activation temperatures.

During carbonization, several aromatic, nuclear structure are transformed to micro crystalline consisting of fused hexagonal rings of carbon atoms. The preparation of impurities and methods of preparation affects the formation of micro crystalline. Further, during activation, the regular array of carbon-bonds in the surface of the micro crystallites is disrupted yielding free valences. The structure of the pores developed in the micro crystallites of the activated carbon has been grouped into three namely:

- Macropores
- Transitional pores
- Micropores

Macropores are formed mainly by the burn-off of the edge groups of the crystallites while micropores are formed mainly by the burn-off of the micro crystallite planes as observed during activation by oxidation by Dubiet as two stages of oxidation.

The structure of activated carbon is therefore tridisperse: containing macro, transitional and micropores. The macropores open out directly to the outer surface of the particles, the transitional branch off from the micropores. The chemical composition of active carbon plays an important role in determining the adsorptive property of carbon. The arrangement of the electron clouds in the carbon skeleton

maybe changed by the presence of imperfect (partial burnt-off) graphite layers resulting in the appearance of the unpaired electrons. This affects the adsorptive power of carbon especially, for polarized substances.

## **2.8 APPLICATIONS OF ACTIVATED CARBONS**

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 APPLICATIONS:** Activated carbon in 50% w/w combination with celite is used as stationary phase in low-pressure chromatographic separation of carbohydrates (mono-di-tri saccharides) using ethanol solutions (5 – 50%) as mobile phase in analytical or preparative protocols.
- b. **FUEL STORAGE:** Research work is on-going on testing activated carbon's 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 energy of 5 - 10 kilojoules per mole (kJ/m). The gas may then be desorbed when subjected to high temperature and either combusted or used in a hydrogen fuel cell.

- c. **GAS PURIFICATION:** Filters with activated carbons are usually used in compressed air and gas purification to remove oil vapors, odor and other hydrocarbons from 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.
- d. **MEDICAL APPLICATIONS:** Activated carbon is used to treat poisoning and overdoses following oral ingestion. It is thought to bind the poison and prevent its adsorption 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 empirical at 1g/kg of body mass (for adolescent or adult, given 50 – 100g).
- e. **CHEMICAL PURIFICATION:** Activated carbon is commonly used to purify solutions containing unwanted colored impurities such as during a recrystallization procedure in organic chemistry.
- f. **WATER PURIFICATION:** Activated carbon usage in water purification is the most accepted method of portable water purification. It removes toxic substances such as insecticides, herbicides, chlorinated hydrocarbons and

phenols in water supply. It is also used in removing impurities from electroplating baths, reaction catalysis and aquarium water filters.

- g. **MECURY SCRUBBING:** Activated carbon often impregnated with iodine or sulphur is widely used to trap mercury emission from coal fire power station, medical incinerators and from natural gas at the well head.

## **CHAPTER THREE**

### **EXPERIMENTAL**

#### **3.1 SELECTION OF MATERIALS**

##### **3.1.1 SELECTION OF ACTIVATED CARBON MATERIAL**

Two types of coconut shell based activated carbon material were employed. They include powdered activated carbon and granular activated carbons.

##### **3.1.2 SELECTION OF ADSORBATE**

Phenol has well-defined parameters as molecular area and a conjugated structure with its characteristic wavelength for UV (ultraviolet) spectrophotometer maximum at 270 nm. Hence, phenol was used for the investigation of the adsorption capacity.

#### **3.2 APPPARATUS USED**

The apparatus used include the following:

- ❖ Electronic weighing balance
- ❖ Ultraviolet spectrophotometer
- ❖ pH meter
- ❖ Vortex mixer

- ❖ Measuring cylinder
- ❖ Spatula
- ❖ Funnel
- ❖ Oven
- ❖ Furnace
- ❖ Beakers
- ❖ Conical flask with stoppers
- ❖ Burette
- ❖ Retort stand
- ❖ Filter paper
- ❖ Pipette

### **3.3 REAGENTS USED**

The reagents used include the following:

- ❖ Nitric acid
- ❖ Distilled water
- ❖ Phenol

## **3.4 MODIFICATION OF THE ACTIVATED CARBON**

### **3.4.1 DEVOLATILIZATION OF THE COCONUT SHELL BASED ACTIVATED CARBON**

The coconut shell based activated carbon was placed in an air tight oven and heated at 600<sup>0</sup>C for 6 hours. The sample was left to cool in the oven after which it was placed in a desiccator to preserve its integrity.

### **3.4.2 TREATMENT OF THE ACTIVATED CARBON WITH NITRIC ACID**

The treatment with nitric acid was done in two phases thus:

#### **❖ TREATMENT WITH CONCENTRATED NITRIC ACID**

Both the granular and the powdered coconut shell based activated carbon were treated with concentrated nitric acid. 5g of the activated carbon was mixed with 50ml of concentrated nitric acid and heated in a fume cupboard for 2 hours. After the treatment, the activated carbon was filtered out, rinsed with distilled water repeatedly to remove all traces of acid and dried at 110<sup>0</sup> C in an oven.

#### **❖ TREATMENT WITH DILUTE NITRIC ACID**

A molar solution of nitric acid was prepared with distilled water. 5g of the activated carbon was mixed with 50ml of dilute nitric acid heated in a fume cupboard for 2hours. After the treatment, the activated carbon was filtered out,

rinsed with distilled water repeatedly to remove all traces of acid and dried at 110<sup>0</sup>C in an oven.

### **3.5 MASS TITRATION**

This has to do with point of zero charge of the adsorbent. Point of zero charge of an adsorbent is the value of the pH required to give zero net surface charge using American Society for Testing and Materials (ASTM) method.

2g, 4g, 6g, 8g, and 10g of as-received carbon sample were mixed with 50ml of distilled water, allowed to equilibrate and the pH of the mixture was measured and recorded. The same procedure was repeated for devolatilized activated carbon, activated carbon treated with concentrated nitric acid and activated carbon treated with dilute nitric acid and reported as well for both granular and powdered activated carbon.

### **3.6 PROXIMATE ANALYSIS OF THE ACTIVATED CARBON**

#### **MATERIAL**

The proximate analysis of the coconut shell based activated carbon material was carried out to determine the percentage by mass of moisture, volatile matter, ash and fixed carbon. This was done at varying temperature.

### **3.6.1 MOISTURE CONTENT**

This was determined by heating at a temperature of 105<sup>0</sup>C for an hour. The difference in weight was recorded accordingly on percentage basis.

### **3. 6.2 VOLATILE MATTER CONTENT**

Volatile matter content was determined by heating the moisture-free activated carbon at a temperature of 600<sup>0</sup>C for ten minutes in the absence of air. The corresponding weight difference was reported on percentage basis.

### **3.6.3 ASH CONTENT**

The sample is further heated for thirty minutes at a temperature of 600<sup>0</sup>C in the presence of air. The difference in weight was reported as ash content in percentage basis.

### **3.6.4 FIXED CARBON**

This is the residue left after the moisture, volatile matter and ash is given up. It is deduced by subtracting from 100, the percentage of moisture, volatile matter and ash content. Thus,

$$\text{Fixed Carbon (FC)} = 100 - (\% \text{moisture} + \% \text{volatile matter} + \% \text{ash})$$

### **3.7 ADSORPTION EXPERIMENT**

The effect of such parameters as adsorbance and phenol concentration was put into consideration. The procedure taken is as follows:

Various amount of the adsorbent (0 – 5.0g) was mixed with a fixed weight of the adsorbate (25m) in stoppered flasks and the mixture was shaken intermittently for six hours. The adsorbate was subsequently separated by filtration and the filtrate analyzed by UV spectrophotometer to determine the concentration of residual adsorbate and recorded accordingly.

## CHAPTER FOUR

### RESULTS AND DISCUSSION

#### 4.1 MASS TITRATION

The mass titration results of the four grades of coconut shell based activated carbon are shown in Tables 4.1 – 4.4.

**TABLE 4.1: MASS TITRATION RESULT FOR AS-RECEIVED  
ACTIVATED CARBON**

SOLID FRACTION (Wt %)	EQUILIBRIUM pH	
	GRANULAR ACTIVATED CARBON	POWDERED ACTIVATED CARBON
2	4.25	3.50
4	4.27	3.46
6	4.30	3.41
8	4.33	3.38
10	4.35	3.35

**TABLE 4.2: MASS TITRATION RESULT FOR DEVOLATILIZED ACTIVATED CARBON**

SOLID FRACTION (Wt %)	EQUILIBRIUM pH	
	GRANULAR ACTIVATED CARBON	POWDERED ACTIVATED CARBON
2	4.70	3.95
4	4.75	3.92
6	4.78	3.88
8	4.81	3.85
10	4.85	3.82

**TABLE 4.3: MASS TITRATION RESULT FOR CONCENTRATED  
NITRIC ACID TREATED ACTIVATED CARBON**

SOLID FRACTION (Wt %)	EQUILIBRIUM pH	
	GRANULAR ACTIVATED CARBON	POWDERED ACTIVATED CARBON
2	4.52	3.80
4	4.60	3.78
6	4.66	3.74
8	4.72	3.71
10	4.80	3.68

**TABLE 4.4: MASS TITRATION RESULT FOR DILUTE NITRIC ACID  
TREATED ACTIVATED CARBON**

SOLID FRACTION (Wt %)	EQUILIBRIUM pH	
	GRANULAR ACTIVATED CARBON	POWDERED ACTIVATED CARBON
2	4.50	3.70
4	4.58	3.67
6	4.60	3.62
8	4.67	3.58
10	4.70	3.50

Figures 4.1 – 4.4 shows mass titration plot for as-received activated carbons, devolatilized activated carbons, concentrated nitric acid treated activated carbons and dilute nitric acid treated activated carbons.

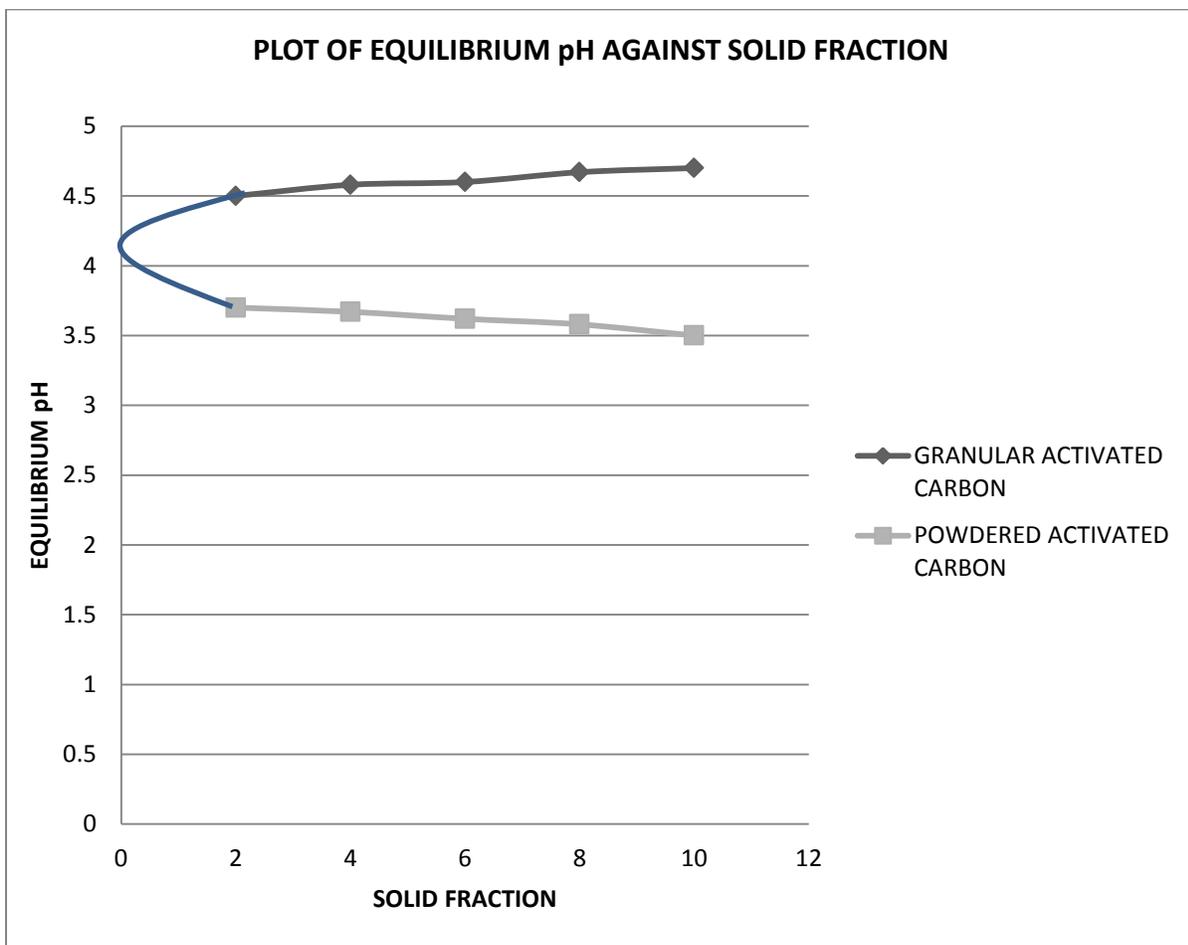


Figure 4.1 Mass titration plots for as-received activated carbons

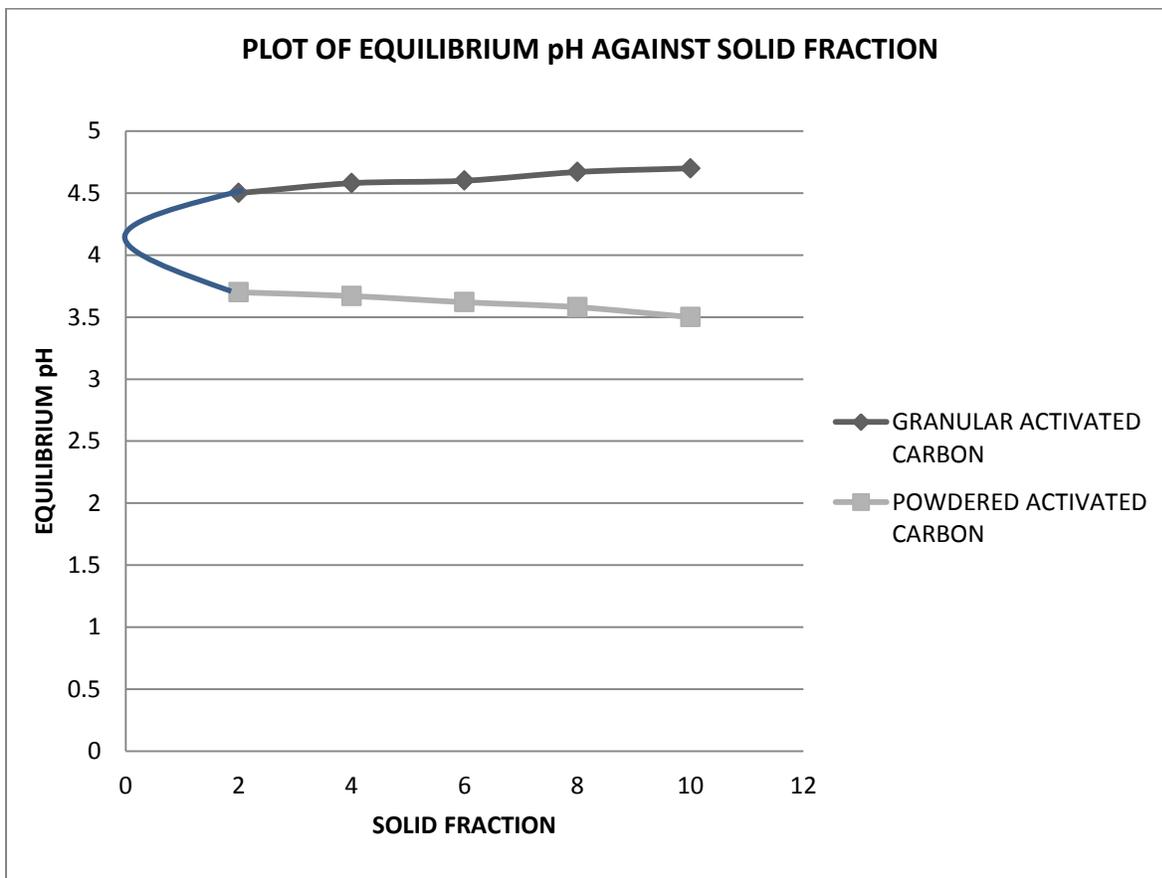


Figure 4.2: Mass titration plot for devolatilized activated carbons.

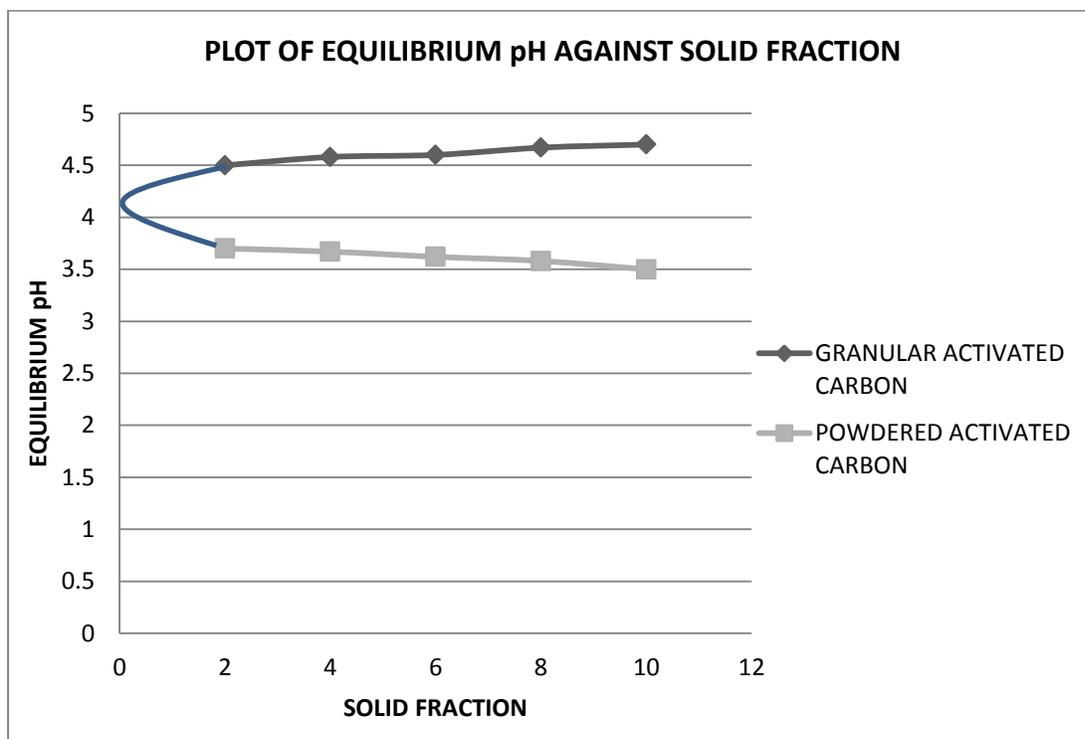


Figure 4.3: Mass titration plot for activated carbon treated with concentrated nitric acid.

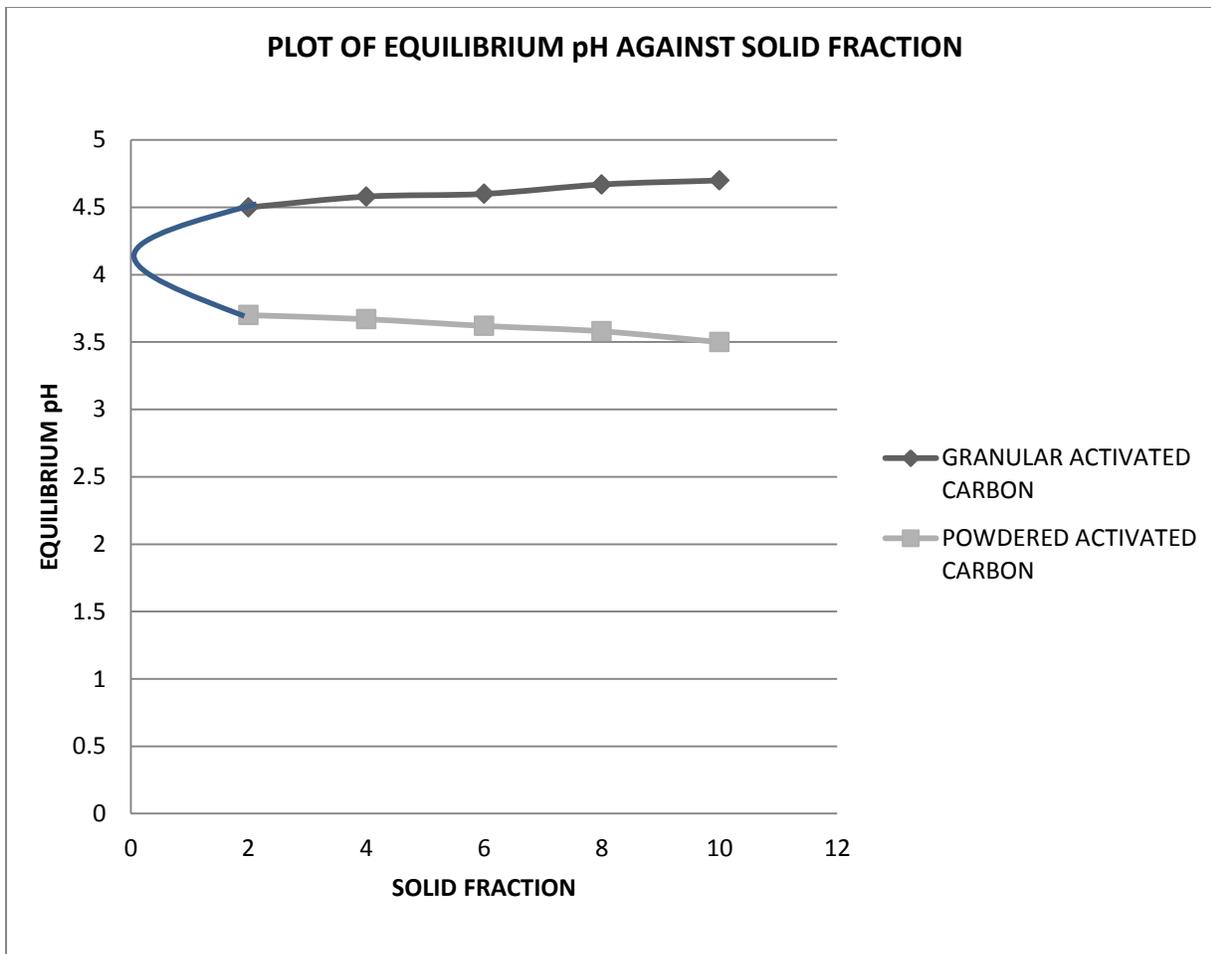


Figure 4.4: Mass titration plot for activated carbon treated with dilute nitric acid.

## 4.2 PROXIMATE ANALYSIS

The proximate analysis of the coconut shell based activated carbon is shown in Table 4.5.

**TABLE 4.5 PROXIMATE ANALYSIS OF GRANULAR AND POWDERED COCONUT SHELL BASED ACTIVATED CARBON.**

PARAMETER	GRANULAR ACTIVATED CARBON (%)	POWDERED ACTIVATED CARBON (%)
Moisture Content	10.80	2.10
Volatile Matter	25.80	52.10
Ash Content	13.40	18.00
Fixed Carbon	50.00	27.80
TOTAL	100.00	100.00

### 4.3 ADSORPTION ISOTHERMS

The calibration of phenol is shown in Table 4.6 while the adsorption isotherms for phenol on the four grades of activated carbon produced from coconut shell based activated carbon are shown in Table 4.7 – 4.9.

**TABLE 4.6: CALIBRATION OF PHENOL**

CONCENTRATION (mmolar)	ADSORBANCE
0.05	0.04
0.5	0.30
1.25	0.70
2.5	1.30
5.0	2.60

**TABLE 4.7 DATA FOR THE ADSORPTION OF PHENOL SOLUTION ON AS-RECEIVED ACTIVATED CARBON.**

WEIGHT OF ADSORBENT X g/25ml OF ADSORBATE (W)	X 40x g/l	EQUILIBRIUM CONCENTRATION mmol/l	CHANGE IN CONCENTRATION mmol/l ( $\Delta C$ )	ADSORBATE UPTAKE $\frac{(\Delta C)}{(X)}$ mmol/g
0	0	1.050	0	$\infty$
0.2	8.0	0.802	0.248	0.031
0.4	16.0	0.592	0.458	0.029
0.8	32.0	0.346	0.704	0.022
1.0	40.0	0.255	0.805	0.020
1.5	60.0	0.159	0.891	0.015
2.0	80.0	0.120	0.830	0.012

**TABLE 4.8 DATA FOR THE ADSORPTION OF PHENOL SOLUTION ON  
THE DEVOLATILIZED ACTIVATED CARBON**

WEIGHT OF ADSORBENT X g/25ml OF ADSORBATE (W)	X 40x g/l	EQUILIBRIUM CONCENTRATION mmol/l	CHANGE IN CONCENTRATION mmol/l ( $\Delta C$ )	ADSORBATE UPTAKE $\frac{(\Delta C)}{(X)}$ mmol/g
0	0	1.050	0	$\infty$
0.2	8.0	0.752	0.298	0.0372
0.4	16.0	0.460	0.590	0.0368
0.6	24.0	0.294	0.756	0.0315
0.8	32.0	0.172	0.878	0.0274
1.0	40.0	0.092	0.958	0.0240
1.2	48.0	0.064	0.986	0.0205
2.0	80	0.045	1.005	0.0126

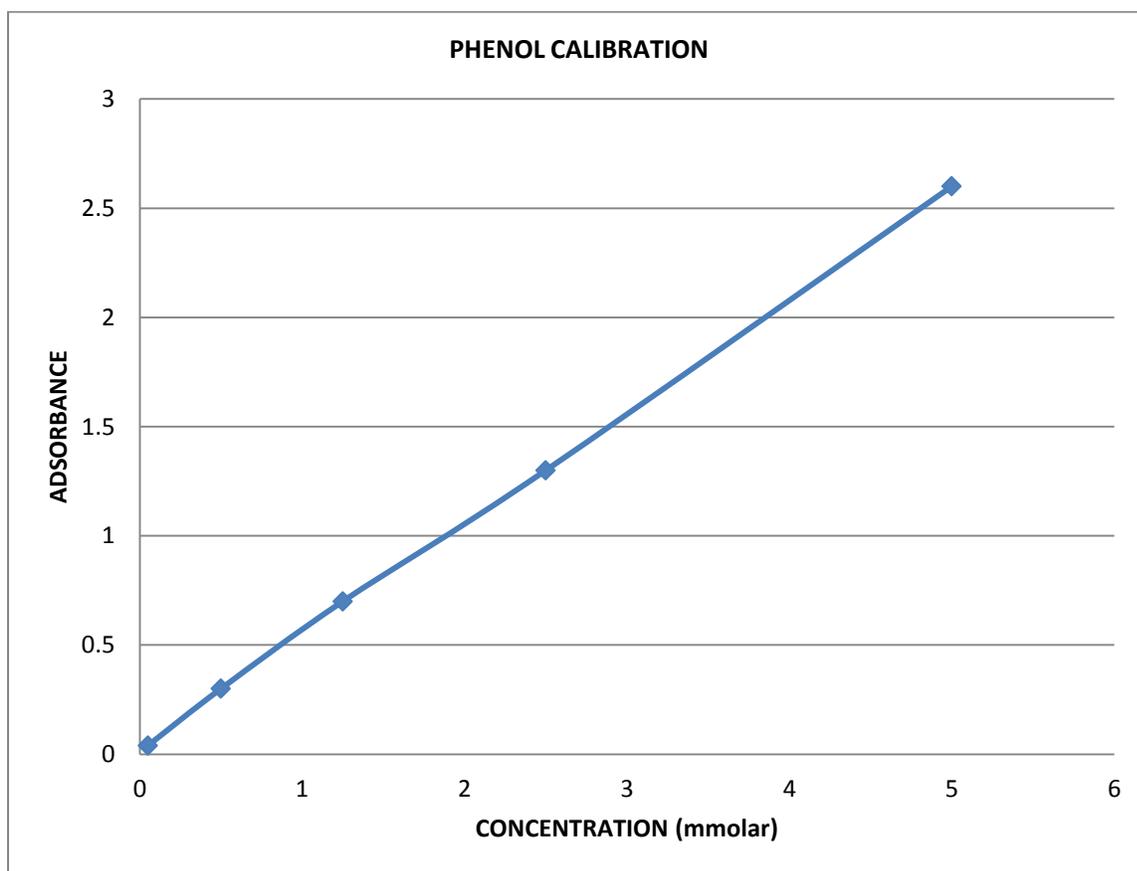
**TABLE 4.9: DATA FOR THE ADSORPTION OF PHENOL SOLUTION ON THE ACTIVATED CARBON TREATED WITH CONCENTRATED NITRIC ACID.**

WEIGHT OF ADSORBENT X g/25ml OF ADSORBATE (W)	X 40x g/l	EQUILIBRIUM CONCENTRATION mmol/l	CHANGE IN CONCENTRATION mmol/l ( $\hat{C}$ )	ADSORBATE UPTAKE $\frac{(\hat{C})}{(X)}$ mmol/g
0	0	1.050	0	$\infty$
0.4	16	0.752	0.298	0.019
0.8	32	0.640	0.410	0.013
1.2	48	0.451	0.599	0.012
1.6	64	0.327	0.723	0.011
2.0	80	0.256	0.794	0.009
4.0	160	0.142	0.908	0.006
5.0	200	0.114	0.936	0.005

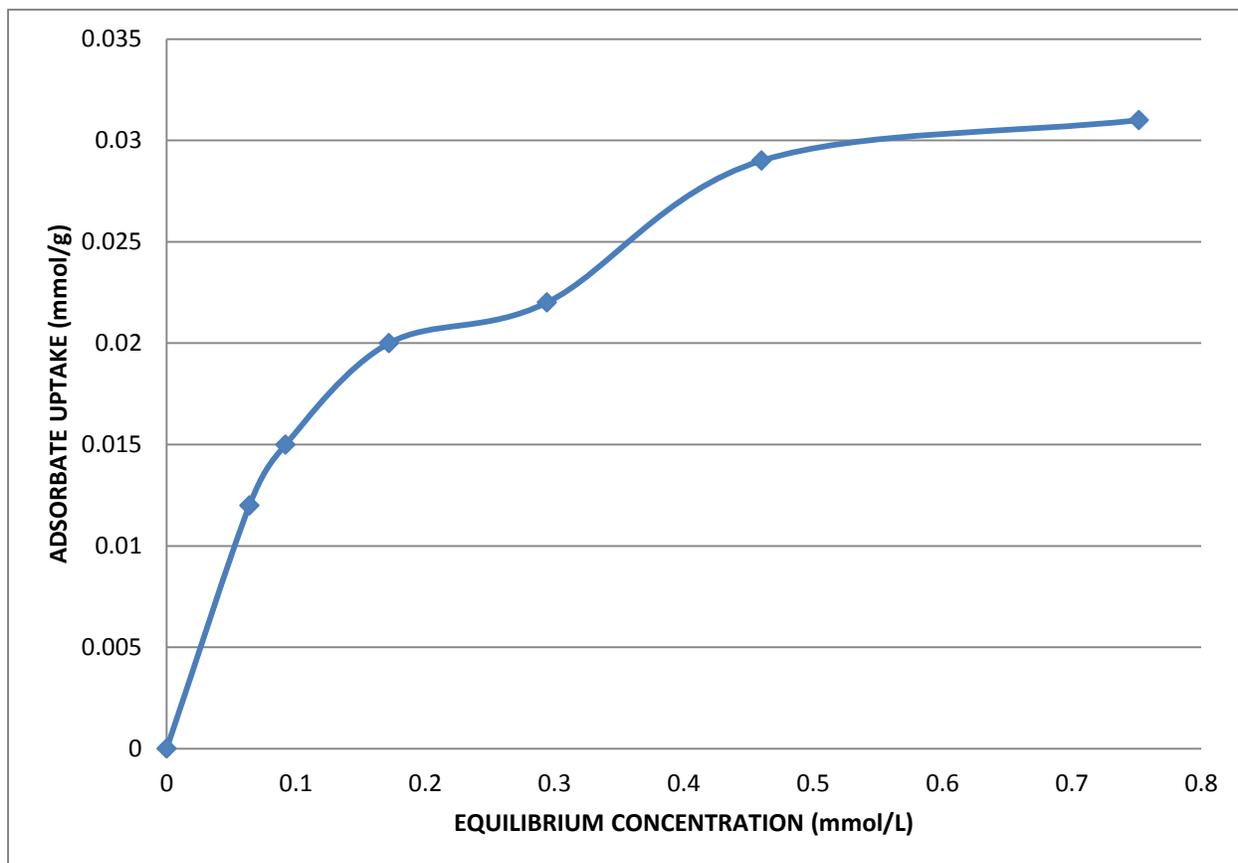
**TABLE 4.10: DATA FOR THE ADSORPTION OF PHENOL SOLUTION ON THE ACTIVATED CARBON TREATED WITH DILUTE NITRIC ACID.**

WEIGHT OF ADSORBENT X g/25ml OF ADSORBATE (W)	X 40x g/l	EQUILIBRIUM CONCENTRATION mmol/l	CHANGE IN CONCENTRATION mmol/l ( $\hat{C}$ )	ADSORBATE UPTAKE $\frac{(\hat{C})}{(X)}$ mmol/g
0	0	1.050	0	$\infty$
0.4	16	0.750	0.300	0.019
0.8	32	0.638	0.412	0.013
1.2	48	0.448	0.602	0.012
1.6	64	0.324	0.726	0.011
2.0	80	0.254	0.796	0.010
2.4	96	0.140	0.910	0.009
2.8	112	0.111	0.939	0.008

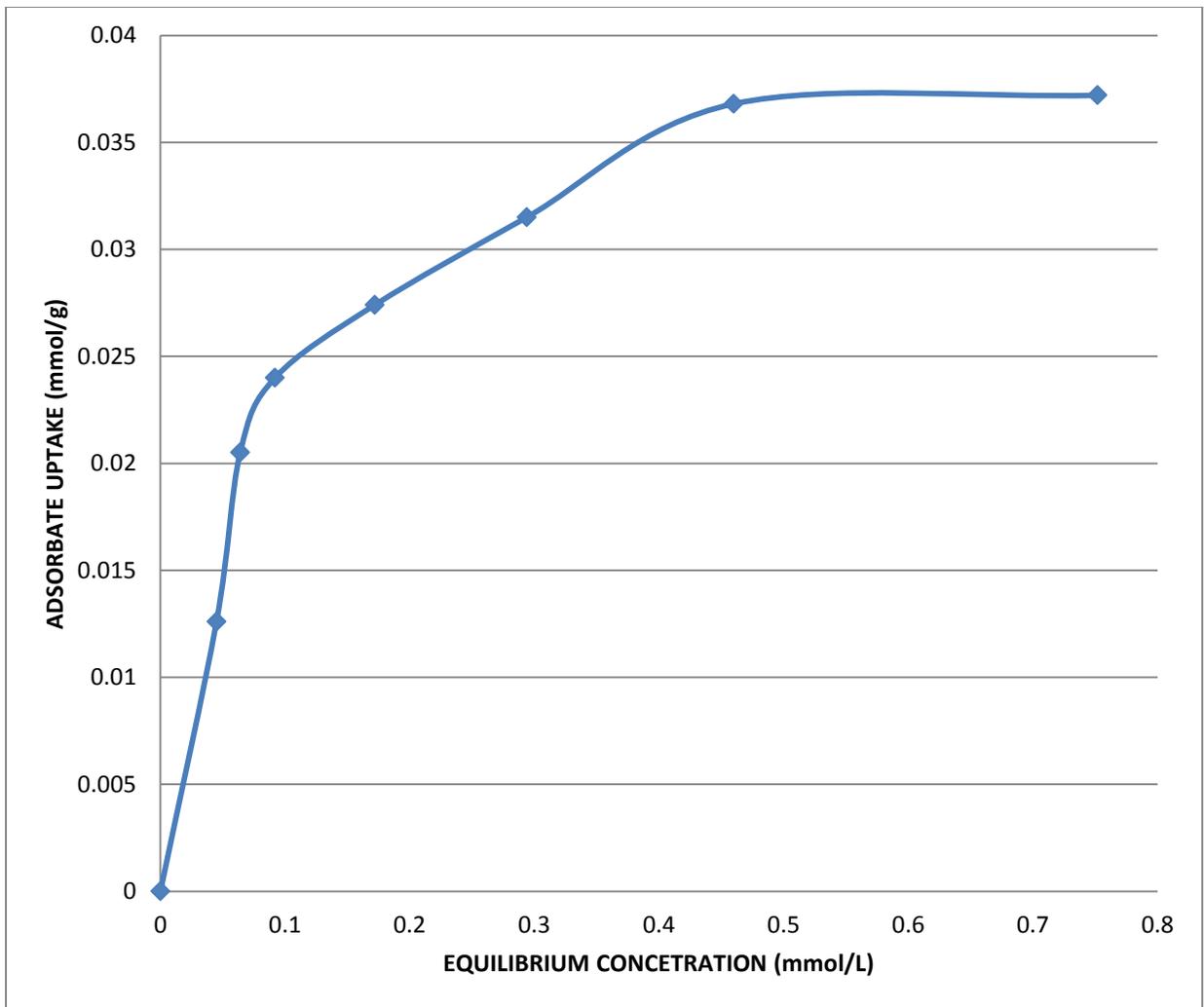
Figure 4.5 shows the calibration of phenol while Figures 4.6 - 4.8 show plots of adsorption isotherm of phenol on as-received activated carbons, devolatilized activated carbons, concentrated nitric acid treated activated carbons, dilute nitric acid treated activated carbons for coconut based activated carbon.



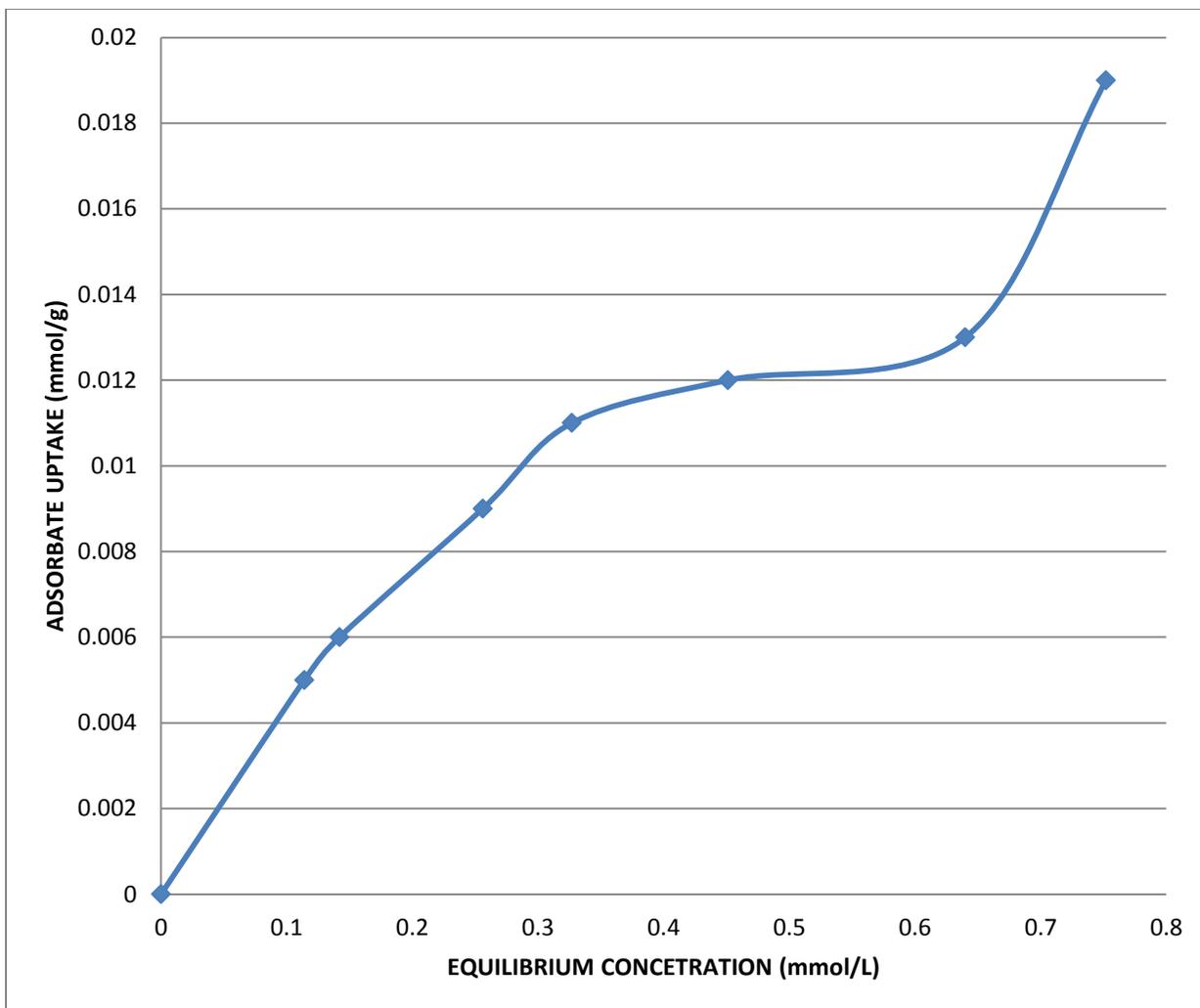
**FIGURE 4.5 CALIBRATION OF PHENOL**



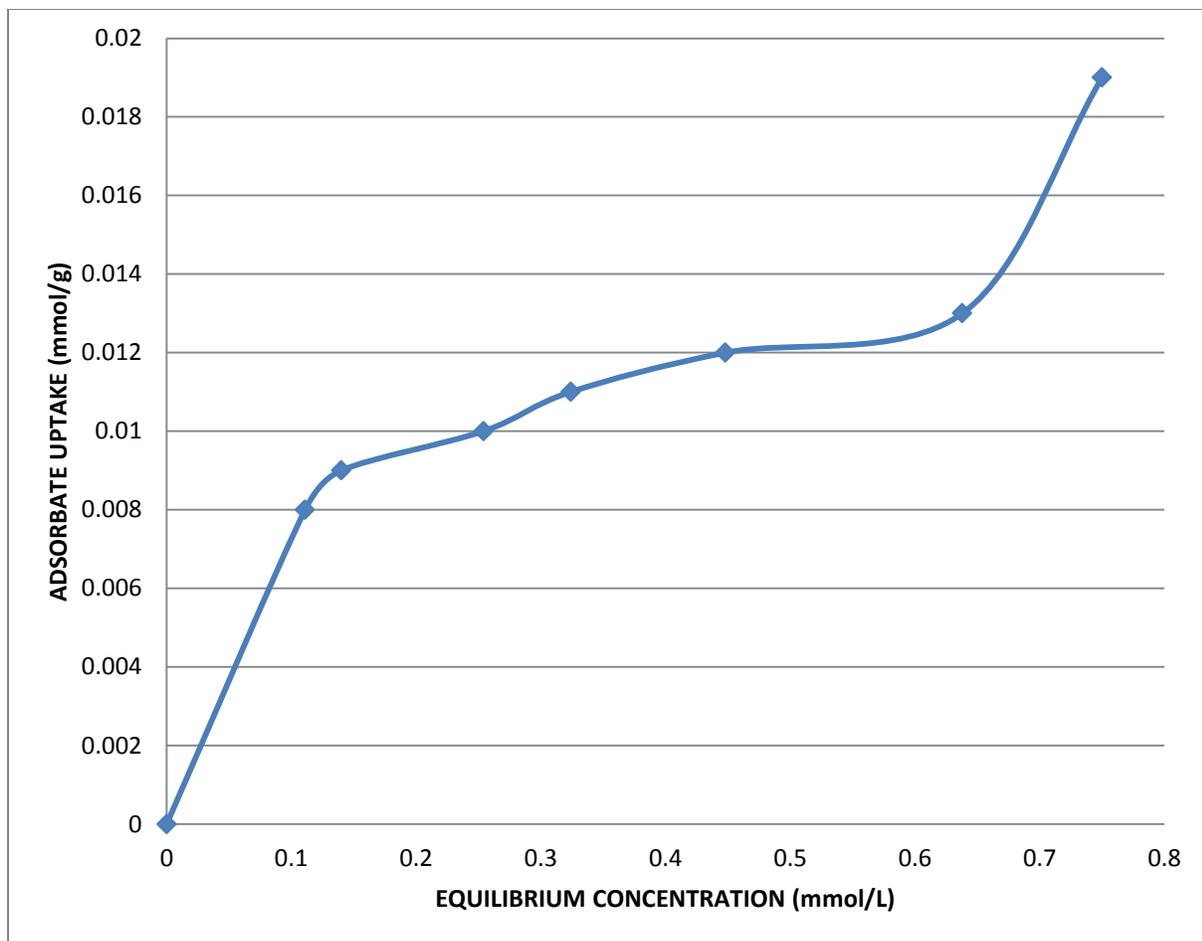
**Figure 4.6: Adsorption isotherms of phenol on as-received activated carbon.**



**Figure 4.7: Adsorption isotherms of phenol on devolatilized activated carbon**



**Figure 4.8: Adsorption isotherms of phenol on concentrated nitric acid treated activated carbon.**



**Figure 4.9: Adsorption isotherms of phenol on dilute nitric acid treated activated carbon.**

#### 4.4 SURFACE COVERAGES

The monolayer capacity and surface coverage for as-received, devolatilized and nitric acid treated activated carbons is shown in Table 4.8.

**TABLE 4.8 MONOLAYER CAPACITIES AND SURFACE COVERAGE FOR THREE GRADES OF COCONUT SHELL BASED ACTIVATED CARBON.**

TYPES OF ACTIVATED CARBON	MONOLAYER CAPACITY(mmol/g)	SURFACE FRACTIONAL COVERAGE
As-received activated carbon	0.031	0.010
Devolatilized activated carbon	0.036	0.012
Concentrated nitric acid treated activated carbon	0.012	0.004
Dilute nitric acid treated activated carbon	0.011	0.003

## 4.5 DISCUSSION OF RESULTS

### 4.5.1 MASS TITRATION

Mass titration was carried out to determine the point of zero charge using ASTM standard by plotting the value of equilibrium pH against the solid fraction. This was done for four grades of activated carbon thus: as-received activated carbon, devolatilized activated carbon, concentrated nitric acid treated activated carbon and dilute nitric acid treated activated carbon. The point of zero charges of the carbon materials were observed from the plots to be as in Table 4.9.

Table 4.9: POINT OF ZERO CHARGE FOR THE ACTIVATED CARBON MATERIAL

ACTIVATED CARBON TYPE	POINT OF ZERO CHARGE	
	GRANULAR ACTIVATED CARBON	POWDERED ACTIVATED CARBON
Devolatilized	4.85	3.82
Concentrated nitric acid	4.76	3.68
Dilute nitric acid	4.70	3.50
As-received	4.35	3.35

From the table, the devolatilized carbon material had the highest point of zero charge. This means that the devolatilized carbon is the most basic and this explains the optimal adsorption of phenol. This result is consistent with the calculated surface coverages.

#### **4.5.2 PROXIMATE ANALYSIS**

The proximate analysis of the coconut shell based activated carbon sample was carried out and the moisture content was found to be 10.80% and 2.10% for granular and powdered activated carbon respectively. Moisture impairs the adsorption capacity of any activated carbon. Also the Ash content was found to be 13.40% and 18.00% for granular and powdered activated carbon respectively. The volatile matter 25.80% and 52.10% and the fixed carbon also 50% and 27.80% respectively.

#### **4.5.3 ADSORPTION ISOTHERMS AND SURFACE COVERAGES**

In determining the monolayer capacity for the adsorption of phenol solution on the grades of activated carbon, four isotherms were plotted:

- i. Graph of adsorbate uptake (mmol/g) against equilibrium concentration (mmol/L) for the as-received activated carbon.

- ii. Graph of adsorbate uptake (mmol/g) against equilibrium concentration (mmol/L) for devolatilized activated carbon.
- iii. Graph of adsorbate uptake (mmol/g) against equilibrium concentration (mmol/L) for concentrated nitric acid treated activated carbon.
- iv. Graph of adsorbate uptake (mmol/g) against equilibrium concentration (mmol/L) for dilute nitric acid treated activated carbon.

From the results of the surface coverage shows, the devolatilized activated carbon had the highest surface coverage while the nitric acid treated activated carbon had the least surface coverage.

The results can be attributed to the fact that devolatilization at extreme temperature, removes all surface functional groups of the activated carbon. The prevalence of  $\pi$ - $\pi$  interactions between phenol molecules and the carbon material would therefore enhance the adsorption capacity of the adsorbent. On the other extreme, the treatment with nitric acid incorporates oxygen functional groups of the adsorbent. Repulsive interaction between phenolic groups of adsorbate and oxygen functional group of carbon would retard adsorption on the carbon surface.

## CHAPTER FIVE

### CONCLUSION AND RECOMMENDATIONS

#### 5.1 CONCLUSION

As a result of their internal and external surface areas as well as developed pore structure, activated carbons are good adsorbents and are therefore used to remove a broad spectrum of dissolved organic and inorganic from the gas and aqueous phases.

Coconut shell based activated carbon at its initial stage has little internal surface area and hence need further modification to enhance their surface area and pore structures.

Devolatilization of the coconut shell based activated carbon was shown to enhance the monolayer capacity and surface coverage of the adsorbent.

On the other hand, treatment with nitric acid attaches oxygen functional groups and adsorbent and hence lowers the adsorption capacity, monolayer capacity as well as surface coverage of the carbon material.

Agricultural based activated carbon as coconut shell provides room for productive use of agricultural waste.

## **5.2 RECOMMENDATIONS**

Developmental work and extensive research is needed to achieve the status of replacing the totality of activated carbons presently been imported into the country for diverse application in the chemical process industries, water treatment, pharmaceuticals etc with agricultural based activated carbon.

It is therefore recommended that research facilities and more laboratory equipment be provided in chemical engineering laboratory in Caritas University.

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## APPENDIX A

### PREPARATION OF DILUTE NITRIC ACID SOLUTION

Molecular weight of nitric acid ( $\text{HNO}_3$ ) = 63g

Specific gravity of nitric acid = 1.40

Percentage purity = 70

$$\begin{aligned} \text{1 molar solution of nitric acid} &= \frac{\text{molecular weight}}{\text{Specific gravity}} \times \frac{100}{\% \text{ purity}} \\ &= \frac{63}{1.40} \times \frac{100}{70} \\ &= \underline{64.27\text{ml}} \end{aligned}$$

### PREPARATION OF PHENOL SOLUTION

Molecular weight of phenol ( $\text{C}_6\text{H}_5\text{OH}$ ) = 94g

1molar solution of phenol = 94g = 1L

1mmol solution of phenol =  $\frac{94\text{g}}{1000\text{ml}}$   
= 0.09g/ml

1.05mmol solution of phenol =  $\frac{94}{1000} (1.05)$   
= 0.099g/ml

## APPENDIX B

### PROXIMATE ANALYSIS OF GRANULAR AND POWDERED

### COCONUT SHELL BASED ACTIVATED CARBON

#### MOISTURE CONTENT AT 105<sup>0</sup>C

##### Moisture content of granular activated carbon

Weight of sample before heating = 9.150g

Weight of sample after heating = 8.161g

Weight loss = 9.150 – 8.161  
= 0.989g

$$\% \text{ Moisture} = \frac{\text{Weight loss}}{\text{Weight of sample before heating}} \times \frac{100}{1}$$

$$= \frac{0.989}{9.150} \times \frac{100}{1}$$

$$= 10.80\%$$

##### Moisture content of powdered activated carbon

Weight of sample before heating = 4.563g

Weight of sample after heating = 4.469g

Weight loss = 4.563 – 4.469

$$= 0.094\text{g}$$

$$\% \text{ Moisture} = \frac{\text{Weight loss}}{\text{Weight of sample before heating}} \times \frac{100}{1}$$

$$= \frac{0.094}{4.469} \times \frac{100}{1}$$

$$= 2.10\%$$

### **VOLATILE MATTER CONTENT AT 600<sup>0</sup>C**

#### Volatile matter content of granular activated carbon

$$\text{Weight of sample before heating} = 20.095\text{g}$$

$$\text{Weight of sample after heating} = 14.910\text{g}$$

$$\text{Weight loss} = 20.095 - 14.910$$

$$= 5.185\text{g}$$

$$\% \text{ Volatile matter} = \frac{\text{Weight loss}}{\text{Weight of sample before heating}} \times \frac{100}{1}$$

$$= \frac{5.185}{20.095} \times \frac{100}{1}$$

$$= 25.80\%$$

Volatile matter content of powdered activated carbon

Weight of sample before heating = 6.796g

Weight of sample after heating = 3.255g

Weight loss = 6.796 – 3.255

= 3.541g

$$\begin{aligned} \% \text{ Volatile matter} &= \frac{\text{Weight loss}}{\text{Weight of sample before heating}} \times \frac{100}{1} \\ &= \frac{3.541}{6.796} \times \frac{100}{1} \\ &= 52.10\% \end{aligned}$$

**ASH CONTENT AT 600<sup>0</sup>C**

Ash content of granular activated carbon

Weight of sample before heating = 3.901g

Weight of sample after heating = 0.523g

$$\begin{aligned} \% \text{ Ash content} &= \frac{\text{Weight of ash formed}}{\text{Weight of sample before heating}} \times \frac{100}{1} \\ &= \frac{0.523}{3.901} \times \frac{100}{1} \end{aligned}$$

$$= 13.40\%$$

Ash content of powdered activated carbon

$$\text{Weight of sample before heating} = 4.001\text{g}$$

$$\text{Weight of sample after heating} = 0.721\text{g}$$

$$\% \text{ Ash content} = \frac{\text{Weight of ash formed}}{\text{Weight of sample before heating}} \times \frac{100}{1}$$

$$= \frac{0.721}{4.001} \times \frac{100}{1}$$

$$= 18.00\%$$

**FIXED CARBON CONTENT**

Fixed carbon content of granular activated carbon

$$\text{Fixed carbon (FC)} = 100 - (\% \text{ moisture content} + \% \text{ volatile matter} + \% \text{ Ash})$$

$$= 100 - (10.80 + 25.80 + 13.40)$$

$$= 100 - 50$$

$$\text{Therefore, Fixed carbon} = 50.00\%$$

Fixed carbon content of powdered activated carbon

$$\text{Fixed carbon (FC)} = 100 - (\% \text{ moisture content} + \% \text{ volatile matter} + \% \text{ Ash})$$

$$= 100 - (2.10 + 52.10 + 18.00)$$

$$= 100 - 72.20$$

Therefore, Fixed carbon = 27.80%

## APPENDIX C

Evaluation of the monolayer values for 1.0mmol/g of phenol solution using [15]:

- a. BET surface area of 1000 m<sup>2</sup>/g
- b. Molecular area of phenol = 52.2 A<sup>2</sup>

A complete surface coverage of phenol on coconut shell based activated carbon requires:

$$\left( \frac{1000 \text{ m}^2}{\text{g carbon}} \right) \left( \frac{1 \text{ mol phenol}}{52.2 \times 10^{-20} \text{ m}^2} \right) \left( \frac{1 \text{ gmol phenol}}{6.023 \times 10^{23} \text{ molecules}} \right)$$

$$= \frac{3 \times 10^{-3} \text{ gmol of phenol}}{\text{g activated carbon}}$$

$$= \frac{3 \text{ mmol of phenol}}{\text{g activated carbon}}$$

For the as-received activated carbon:

$$\text{Monolayer capacity} = \frac{0.031 \text{ mmol phenol}}{\text{g carbon}}$$

$$\begin{aligned}\text{Surface coverage} &= \frac{0.031}{3} \\ &= 0.010\end{aligned}$$

For the devolatilized activated carbon:

$$\text{Monolayer capacity} = \frac{0.036\text{mmol phenol}}{\text{g carbon}}$$

$$\begin{aligned}\text{Surface coverage} &= \frac{0.036}{3} \\ &= 0.012\end{aligned}$$

For the concentrated nitric acid treated activated carbon:

$$\text{Monolayer capacity} = \frac{0.012\text{mmol phenol}}{\text{g carbon}}$$

$$\begin{aligned}\text{Surface coverage} &= \frac{0.012}{3} \\ &= 0.004\end{aligned}$$

For the dilute nitric acid treated activated carbon:

$$\text{Monolayer capacity} = \frac{0.011 \text{ mmol phenol}}{\text{g carbon}}$$

$$\begin{aligned} \text{Surface coverage} &= \frac{0.011}{3} \\ &= 0.003 \end{aligned}$$