

**POSSIBILITY OF PRODUCING ACTIVATED  
CARBON FROM *MORINGA OLEIFERA* SEEDS HUSKS**

**ANIS FARHANA BT ABD RAZAK**

**BACHELOR OF CHEMICAL ENGINEERING (GAS TECHNOLOGY)**

**UNIVERSITI MALAYSIA PAHANG**

©ANIS FARHANA BT ABD RAZAK (2014)

## Thesis Access Form

No \_\_\_\_\_ Location \_\_\_\_\_

Author : .....

Title : .....

Status of access OPEN / RESTRICTED / CONFIDENTIAL

Moratorium period: \_\_\_\_\_ years, ending \_\_\_\_\_ / \_\_\_\_\_ 200\_\_\_\_\_

Conditions of access proved by (CAPITALS): DR EMAN N. ALI

Supervisor (Signature).....

Faculty: .....

Author's Declaration: *I agree the following conditions:*

OPEN access work shall be made available (in the University and externally) and reproduced as necessary at the discretion of the University Librarian or Head of Department. It may also be copied by the British Library in microfilm or other form for supply to requesting libraries or individuals, subject to an indication of intended use for non-publishing purposes in the following form, placed on the copy and on any covering document or label.

*The statement itself shall apply to ALL copies:*

**This copy has been supplied on the understanding that it is copyright material and that no quotation from the thesis may be published without proper acknowledgement.**

**Restricted/confidential work:** All access and any photocopying shall be strictly subject to written permission from the University Head of Department and any external sponsor, if any.

**Author's signature**.....**Date:** .....

users declaration: for signature during any Moratorium period (Not Open work):

***I undertake to uphold the above conditions:***

Date	Name (CAPITALS)	Signature	Address

**POSSIBILITY OF PRODUCING ACTIVATED  
CARBON FROM *MORINGA OLEIFERA* SEEDS  
HUSKS**

**ANIS FARHANA BT ABD RAZAK**

Thesis submitted in partial fulfilment of the requirements  
for the award of the degree of  
Bachelor of Chemical Engineering (Gas Technology)

**Faculty of Chemical & Natural Resources Engineering  
UNIVERSITI MALAYSIA PAHANG**

JULY 2014

©ANIS FARHANA BT ABD RAZAK (2014)

## **SUPERVISOR'S DECLARATION**

We hereby declare that we have checked this thesis and in our opinion, this thesis is adequate in terms of scope and quality for the award of the degree of Bachelor of Chemical Engineering (Gas Technology).

Signature :  
Name of main supervisor : DR. EMAN N ALI  
Position :  
Date :

## **STUDENT'S DECLARATION**

I hereby declare that the work in this thesis is my own except for quotations and summaries which have been duly acknowledged. The thesis has not been accepted for any degree and is not concurrently submitted for award of other degree.

Signature :  
Name : ANIS FARHANA BT ABD RAZAK  
ID Number : KC10024  
Date : JULY 2014

## ***Dedication***

I dedicate my dissertation work to my supervisor, Dr. Eman N. Ali, my family and my friends. A special feeling of gratitude to my supervisor Dr. Eman N. Ali who encourages me and supported me throughout the process. I also dedicate this dissertation to my parents and friends for always supported me in finishing this Undergraduate Research Project.

## **ACKNOWLEDGEMENT**

I wish to thank my supervisor, Dr. Eman N. Ali for her countless hours of reflecting, reading, encouraging, and most of all patience throughout the entire process. I would like to acknowledge and thank my chemical laboratory personnel for allowing me to conduct my research and providing any assistance requested. Special thanks go to the members of staffs for their continued support. Finally, I would like to thank to Dr Cheng Chin Kui who assisted me with this project by providing me with the reactor.

## ABSTRACT

Cost of treatment process usually increased because of the activated carbon. Thus, there have been many researches that have been done by researchers to utilise cheaper raw materials for the production of activated carbon. Moreover, the commercially used activated carbon is coal which is limited and non – renewable which will be finish up. Consequently, a wide variety of agricultural by – products and wastes have been investigated such as coconut shell, wood, palm seed and *Moringa oleifera* husks. *Moringa oleifera* husks have the potential to produce activated carbon by carbonisation under nitrogen followed by steam pyrolysis. There are many advantages of using one – step steam pyrolysis which is no chemical inputs are required, the process requires less energy than traditional processes which involves two heating steps and local production reduce the transportation required for the product. The husks are milled, sieved and separated in fractions. Then, the samples are heated by an electric heater and are subjected to pyrolysis in the presence of steam at atmospheric pressure. The steam flow is 0.5 litre/hour and is introduced into the reactor when the temperature in the reactor is 150°C. The reactor will be heated at constant rate 25°C/min until selected temperature which are 600°C, 700°C and 800°C is reached. Then, the samples are kept at final temperature for one hour. The produced char are characterised. The surface area is calculated by nitrogen adsorption and desorption isotherms which uses dynamic flowing technique providing Brunauer, Emmett and Teller (BET) and Langmuir surface area to test whether it is micropores, mesopores or macropores. Iodine test are conducted to measure the adsorption capacity by determined the surface area.

**Key words:** *Moringa oleifera*, activated carbon, pyrolysis, adsorption, Langmuir surface area, Iodine test



## ABSTRAK

Kos proses rawatan untuk merawat produk yang tidak diinginkan biasanya meningkat kerana karbon yang diaktifkan. Oleh itu, terdapat banyak kajian yang telah dilakukan oleh penyelidik untuk menggunakan bahan-bahan mentah yang lebih murah untuk pengeluaran karbon diaktifkan. Selain itu, karbon diaktifkan yang digunakan secara komersial adalah arang batu yang terhad dan tidak - boleh diperbaharui yang akan habis. Oleh yang demikian, pelbagai produk pertanian dan bahan buangan telah dikaji seperti tempurung kelapa, kayu, benih sawit dan sekam *Moringa oleifera*. Sekam *Moringa oleifera* mempunyai potensi untuk menghasilkan karbon diaktifkan oleh pengkarbonan bawah nitrogen diikuti dengan pirolisis wap. Terdapat banyak kelebihan dalam menggunakan satu langkah pirolisis wap yang tidak memerlukan bahan kimia, proses itu memerlukan tenaga kurang daripada proses tradisional yang melibatkan dua langkah pemanasan dan pengeluaran tempatan mengurangkan pengangkutan yang diperlukan untuk produk. Sekam itu digiling, disaring dan dipisahkan dalam pecahan. Kemudian, sampel dipanaskan oleh pemanas elektrik dan tertakluk kepada pirolisis di hadapan stim pada tekanan atmosfera. Aliran wap adalah pelbagai yang 0.5 liter / jam dan dimasukkan ke dalam reaktor apabila suhu di dalam reaktor adalah 150 ° C. Reaktor akan dipanaskan pada kadar yang tetap 25 ° C / min sehingga suhu yang dipilih yang 600 ° C, 700 ° C dan 800 ° C. Kemudian, sampel disimpan pada suhu akhir selama satu jam. Arang yang dihasilkan akan diciri-cirikan. Luas permukaan dikira dengan penjerapan nitrogen dan isoterma desorption yang menggunakan teknik yang mengalir dinamik menyediakan Brunauer, Emmett dan Teller (BET) dan kawasan permukaan Langmuir untuk menguji sama ada ia adalah micropores, mesopores atau macropores. Ujian iodine dijalankan untuk mengukur kapasiti penjerapan oleh ditentukan luas permukaan.

Kata kunci : *Moringa oleifera*, carbon diaktifkan, pirolisis, penyerapan, luas permukaan Langmuir, ujian Iodine

## TABLE OF CONTENTS

SUPERVISOR'S DECLARATION.....	IV
STUDENT'S DECLARATION.....	V
DEDICATION.....	VI
ACKNOWLEDGEMENT.....	VII
ABSTRACT.....	VIII
ABSTRAK.....	IX
TABLE OF CONTENTS.....	X
LIST OF FIGURES.....	XI
LIST OF TABLES.....	XII
LIST OF ABBREVIATIONS.....	XIII
1 INTRODUCTION.....	1
1.1 Motivation and statement of problem .....	1
1.2 Objectives.....	1
1.3 Scope of this Research.....	2
1.4 Main Contribution of this Work.....	2
1.5 Organisation of this thesis.....	2
2 LITERATURE REVIEW.....	3
2.1 Overview.....	3
2.2 Introduction.....	3
2.3 Physical Properties of Activated Carbon.....	5
2.4 Previous Study on <i>Moringa oleifera</i> .....	6
2.5 Summary.....	7
3 MATERIALS AND METHODS.....	8
3.1 Overview.....	8
3.2 Introduction.....	8
3.3 Chemicals.....	8
3.4 Experimental Procedures and Test Conducted.....	9
4 RESULTS AND DISCUSSIONS.....	13
4.1 Overview.....	13
4.2 Results.....	13
5 CONCLUSION.....	26
5.1 Conclusion.....	26
5.2 Future Work.....	26
REFERENCES.....	27
APPENDICES.....	29

## LIST OF FIGURES

Figure 2.1: Equipment for Preparation of Activated Carbon.....	4
Figure 2.2: Activated Carbon Yield As Percentage Of Original Mass.....	6
Figure 2.3: Yield And Comparative Adsorption Properties Of Activated Carbons.	7
Figure 3.1: Correction Factor for Iodine Adsorption.....	12
Figure 4.1: Activated Carbon Surface Area at Different Temperatures.....	13
Figure 4.2: Activated Carbon Surface Area at Different Temperatures.....	14
Figure 4.3: Activated Carbon Surface Area at Different Temperatures.....	15
Figure 4.4: Langmuir Adsorption Isotherm of Manually Removed Seeds Husks .....	16
Figure 4.5: Langmuir Adsorption Isotherm of Grinded Husks (Manually Removed Seeds Husks).....	17
Figure 4.6: Langmuir Adsorption Isotherm of Mechanically Removed Seeds Husks.....	18
Figure 4.7: Iodine Test of Manually Removed Seeds Husks.....	19
Figure 4.8: Iodine Test of Grinded Husks (Manually Removed Seeds Husks).....	20
Figure 4.9: Mechanically Removed and Seeds Husks.....	20
Figure 4.10: Mass Decomposition of Manually Removed Seeds Husks.....	21
Figure 4.11: Mass Decomposition of Mechanically Removed Seeds Husks.....	22
Figure 4.12: Mass Decomposition of Grinded Husks (Manually Removed Seeds Husks) .....	23
Appendix 1: Manually Removed Seeds Husks.....	29
Appendix 2: Mechanically Removed Seeds Husks.....	30
Appendix 3: Grinded Husks (Manually Removed Seeds Husks).....	31
Appendix 4: Activated Carbon Produced from Manually Removed Seeds Husks...	32
Appendix 5: Activated Carbon from Mechanically Removed Seeds Husks.....	33
Appendix 6: Activated Carbon from Grinded Husks (Manually Removed Seeds Husks) .....	34

## LIST OF TABLES

Table 4.1: Original Size of Pods and Seeds Husks.....	14
Table 4.2: Grinded Pods and Seeds Husks Using Grinder.....	14
Table 4.3: Mechanically Grinded Pods and Seeds Husks.....	15
Table 4.4: Yield of Production of Activated Carbon from different types of Moringa oleifera Seeds Husks.....	24
Table 4.5: Comparison of Produced Activated Carbon with the Activated Carbon Available in the Market.....	25

## LIST OF ABBREVIATIONS

A	correction factor
$A_N$	cross – sectional area of nitrogen molecule in angstroms
BET	Brunauer, Emmett and Teller (BET)
$I_n$	iodine number
$M_N$	molecular weight of nitrogen
M	mass of activated carbon in g
N	Avagadro's Number, $6.025 \text{ E}+23$
$N_1$	normality of iodine solution
$N_2$	normality of sodium thiosulphate solution
S	specific surface area in $\text{m}^2/\text{g}$
V	volume of sodium thiosulphate solution
$X_m$	sorption vs lue
X	mg of iodine adsorbed by the activated carbon

# 1 INTRODUCTION

## 1.1 *Motivation and statement of problem*

Activated carbons are carbonaceous materials that can be distinguished from elemental carbon by the oxidation of the carbon atoms found on the outer and inner surfaces of carbon materials (Mattson and Mark, 1971). The characteristics of activated carbons are large surface areas, well-developed porosity and tuneable surface-containing functional groups (Baker et al., 1992, Zongxuan et al., 2003). Other than that, they have good kinetic properties and high adsorption capacities. For these reasons, activated carbons are widely used as adsorbents for the removal of organic chemicals and metal ions of environmental or economic concern from air, gases, potable water and wastewater (El-Hendawy, 2003).

Nowadays, activated carbons from many sources have been used to remove dissolved metallic salts from aqueous solutions have been reported (Goyal et al., 1999; De and De, 1994; Periasamy, 1991). Adsorption on activated carbons also can reduce COD (Das and Patnaik, 2001; Rao and Bhole, 2002). Commercially produced activated carbons are very expensive (Kadirvel et al., 2003). Thus, many substances have been screened as alternatives to the conventional raw materials for the production of activated carbons (Howlader et al., 1999). The commercially used activated carbon is coal which is a limited, non – renewable resource, and will be depleted one day. There is an alternative way to overcome this problem by using renewable sources such as coconut shells, rice husk and *Moringa oleifera* seeds and pod husks (Martinez, M.L. et al, 2003).

*Moringa oleifera* is among the 14 species of trees belong to the genus *Moringaceae*. It is the best native to North India (Ghebremichael K.A, 2004). *Moringa oleifera* is known as multipurpose tree as its part are useful for many applications (Fuglie, 1999). This tree grows fast and develops to a full tree within one year of its plantation. The dried pods and husks can produce activated carbon by single – step steam pyrolysis (Pollard et al. , 1995 and Warhurst et al. ,1997).

## 1.2 *Objectives :*

The objectives are as follows :

- To investigate the possibility of producing activated carbon from *Moringa oleifera* seeds and pods husks.
- To measure produced activated carbon properties.
- To compare produced activated carbon properties with standard or available activated carbon in the markets.

### **1.3 Scope of study :**

To investigate the possibility of producing activated carbon from *Moringa oleifera* seeds and pods husk. Then, the properties of the activated carbon will be investigated. The properties of activated carbon produced will be compared to activated carbon in the market to show the quality of activated carbon and find out if it possible to commercialize it.

### **1.4 Main Contribution of this work**

The following is the contribution:

- To find alternative for activated carbon from waste material.

### **1.5 Organisation of this thesis**

The structure of the reminder of the thesis is outlined as follow:

Chapter 2 provides an overview of the *Moringa oleifera* and activated carbon. A general description on the properties of *Moringa oleifera* and the properties of activated carbon. This chapter also provides a brief review and statistic of previous research done comparing to the activated carbon. A summary on activated carbon produced from *Moringa oleifera* compared to the activated carbon in the market.

Chapter 3 gives a review of the procedure involved in the pyrolysis process. The characteristics of the activated carbon will be compared by doing some tests which are phenol adsorption isotherm and iodine adsorption.

Chapter 4 gives a clearer understanding of the activated carbon properties where results are tabulated and graphs are drawn. Parameters differ such as temperature and heat constant.. Nevertheless, rheological tests results will show the possibility of *Moringa oleifera* producing activated carbon.

Chapter 5 draws together a summary of the thesis and outlines of the future work which might be derived from the model developed in this work.

## **2 LITERATURE REVIEW**

### **2.1 Overview**

My literature study is on possibility of producing activated carbon from *Moringa oleifera*.

### **2.2 Introduction**

#### ***Activated Carbon***

Activated carbons are carbonaceous materials with highly developed internal surface area and porosity which is sometimes called solid sponges. Large surface area results in high capacity for absorbing chemicals. One of the largest uses of activated carbon is in water treatment industry which will remove the compounds that affect the taste and odour of water (Zanzi et al, 2001). Due to necessity of environmentally friendly processes and material recovery purposes, activated carbon also used in industrial waste water and gas treatment. The food industry used activated carbon to remove the compounds that affect the colour, taste and odour of the food (Zanzi et al, 2001). In mineral industry, activated carbon is used to recover gold from leached liquors (Zanzi et al, 2001). Activated carbon also used in medical and pharmaceutical industry. Another area that used activated carbon is catalysis where the structure and features of the activated carbon are favourable. In gas applications, activated carbon is used in air filters in industrial applications as well as in general air conditioning applications. Traditionally, in order to prepare the activated carbon, two steps are used which are carbonisation of the raw material in absence of oxygen and activation with water vapour or carbon dioxide. A three – step process for producing activated carbon are carbonisation, oxidation and heating from low to high temperature. Nowadays, the sample will be carbonised by pyrolysis in a flow of steam. The processes that will involve in steam pyrolysis are thermal decomposition, multi – component steam distillation, fast escape and stabilization of the volatiles, mild oxidation, activation of the solid residues and formation of the activated carbon. The multi – component distillation which accompanies the pyrolysis in a stream of water vapour will give a mild process and efficient escape of the volatiles from the solid material. The quick removal of the volatiles and stabilization of the radicals in the presence of the steam results in increased yields of volatiles and formation of solid products with a highly developed active surface free from organic compounds (Zanzi et al, 2001).

The physical properties such as bulk density, yield, burn off and hardness and the chemical properties such as pH, conductivity and ash are analysed using standard methods. Other than that, this sample will be analysed to evaluate the effectiveness for colour removal efficiency. Activated carbon produced from bituminous or lignite coal is commercially activated carbon in the markets. Due to some factors such as long – term availability of coal, environmental impacts and potentially increasing cost have encouraged researchers to



find alternative ways in producing activated carbon. Activated carbon can be produced from any material that has carbon as its element. Any lignocellulosic material can be converted to an activated carbon (Khadija et al, 2008).

Preliminary tests on carbons produced at a variety of temperatures showed that steam pyrolysis activation produced high quality carbon (Warhurst et al., 1996). Then, it is analysed by using nitrogen adsorption which allows selection of optimum carbons based on surface area development, microporosity and product yield (Warhurst et al., 1996, 1997).

### ***Preparation of Activated Carbon***

The equipment used are cylindrical pyrolysis reactor and equipment for removal of liquid and gas meter. The reactor tube is heated by an electric heater. The sample will be placed in the removable inner cylinder with a net in the bottom. The outer cylinder is connected to a metal tube with a screw holder which will make it easier to clean. The water will be pumped to the steam generator by a peristaltic pump. The steam temperature is around 105°C. The volatiles will be cooled down in a water-cooled condenser. The gas is led through a column with cotton and phosphorus pentoxide as a drying agent, a gas meter and then collected in a bag.



Figure 2.1 : Equipment for preparation of activated carbon

## 2.3 *Physical Properties of Activated Carbon*

### **ADSORPTION MECHANISM**

A process which a surface concentrates fluid molecules by chemical or physical forces is known as adsorption whereas absorption is a process whereby fluid molecules are taken up by a liquid or solid and distributed throughout that liquid or solid.

In the physical adsorption process, molecules are held by the carbon's surface by weak forces known as Van Der Waals Forces resulting from intermolecular attraction. The carbon and the adsorbates are thus unchanged chemically. Maximum adsorption capacity is determined by the degree of liquid packing that can occur in the pores. In very high vapour pressure, multilayer adsorption can lead to capillary condensation even in mesopores. If adsorption capacity is plotted against pressure ( for gases ) at constant temperature, the curve produced is known as an isotherm. Adsorption increases with increased pressure and also with increasing molecular weight, within a series of a chemical family. Thus, methane is less easily adsorbed than propane. This is a useful fact to remember when a particular system has a number of components. Physical adsorption in the vapour phase is affected by certain external parameters such as temperature and pressure. The adsorption process is more efficient at lower temperatures and higher pressures since molecular species are less mobile under such conditions.

### **Surface Area**

The internal surface area of a carbon is usually determined by the BET method (Brunauer, Emmerr and Teller). This method utilizes the low – pressure range of the adsorption isotherm of a molecule of known dimensions ( usually nitrogen ). This region of the isotherm is generally attributed to monolayer adsorption. Thus, by assuming the species is adsorbed only one molecule deep on the carbon's surface, the surface area may be calculated using the equation :

$$S = \frac{X_m N A}{M}$$

S = specific surface in m<sup>2</sup>/g

X<sub>m</sub> = sorption value (weight of adsorbed N<sub>2</sub> divided by weight of carbon sample)

N = Avagadro's Number, 6.025 E+23

A = cross-sectional area of nitrogen molecule in angstroms

M = molecular weight of nitrogen

## 2.4 Previous Study on *Moringa oleifera*

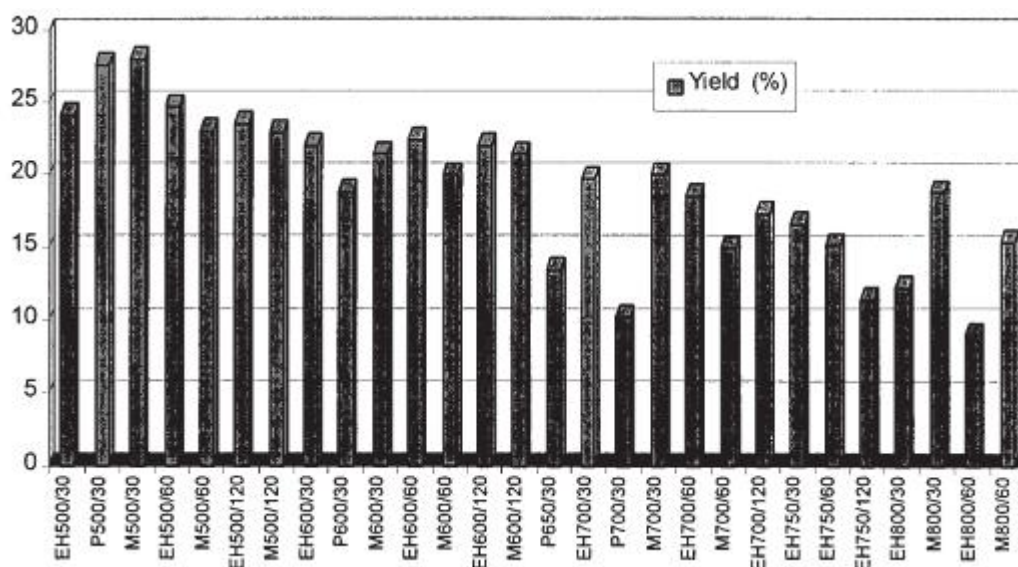


Figure 2.2 : Activated carbon yield as percentage of original mass

From Figure 2.2, EH700/30 is carbon derived from husk material used at Edinburgh and heated a 700°C for 30 minutes. M denotes carbon from husk material used at Malawi, and P is carbon from pods. The process steam pyrolysis gives reproducible yields with duplicate runs differing by less than 1 per cent. Yield tends to reduce as the heating temperature increases. The duration of heating (soak time) has little effect on yield for longer times at 500 or 600°C but tends to give reduced yield for longer times at the higher temperatures. The pods were turned to ash at temperatures >700°C whereas the husks ashed above 800°C when the time was 120 minutes.

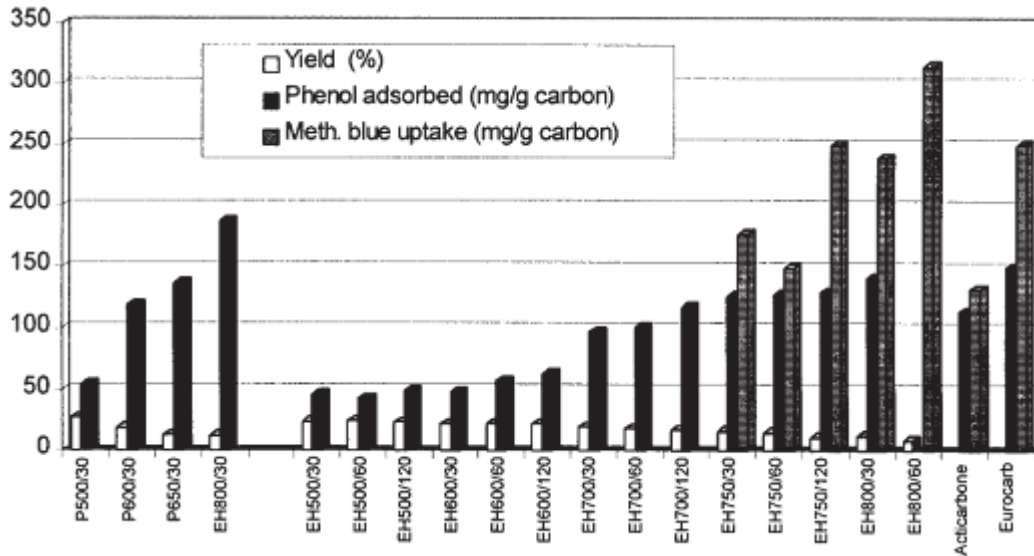


Figure 2.3 : Yield and comparative adsorption properties of activated carbons.

From Figure 2.3, the yield tends to reduce as heating temperature increases. The duration of heating has little effect in yield at 500 or 600°C but tends to give reduced yield for longer time as at the higher temperatures. Both the methylene blue and phenol results show that the capacity for a carbon to adsorb impurities will tend to improve as the temperature and the heating of production are increased.

## 2.5 Summary

Based on the previous research, it is shown that *Moringa oleifera* pods and seed husks can be commercialized into activated carbon by comparing the adsorption properties of activated carbon produced from *Moringa oleifera* and the activated in the market.

### **3. MATERIAL AND METHODS**

#### **3.1 Overview**

This paper is about to investigate the possibility of *Moringa oleifera* seeds and pods husks in producing activated carbon. First of all, the parameters of temperature and heating rate are manipulated at different range of data. It is noted that at different temperatures, the yield of activated carbon differ. Then, Langmuir adsorption isotherms and iodine adsorption will be carry out to determine the adsorption properties.

##### **3.1.1 Preparation**

The equipment used are cylindrical pyrolysis reactor and equipment for removal of liquid products and gas meter. The reactor tube are heated by an electric heater.. The sample will be placed in the removable inner cylinder with a net in the bottom. The outer cylinder is connected to a metal tube with a screw holder which will makes it easier to clean. The water will be pumped to the steam generator by a peristaltic pump. The steam temperature is around 105°C. The volatiles will be cooled down in a water – cooled condenser. The gas is led through a column with cotton and phosphorus pentoxide as drying agent, a gas meter and then collected in a bag.

##### **3.1.2 Steam pyrolysis**

The processes that will involved in steam pyrolysis are thermal decomposition, multi – component steam distillation, fast escape and stabilization of the volatiles, mild oxidation, activation of the solid residues and formation of the activated carbon. The multi – component distillation which accompanies the pyrolysis in a stream of water vapour will give a mild process and efficient escape of the volatiles from the solid material. The quick removal of the volatiles and stabilization of the radicals in the presence of the steam results in increased yields of volatiles and formation of solid products with a highly developed active surface free from organic compounds.

### **3.2 INTRODUCTION**

This paper presents the possibility of *Moringa oleifera* seeds and pods husks in producing activated carbon.

### **3.3 CHEMICALS**

Chemical will be obtained from the supplier which is ordered from Chemical Lab Technical Unit, Universiti Malaysia Pahang.

### 3.3.1 *Moringa oleifera*

*Moringa oleifera* seeds and pods husks will be collected in Kuantan, Pahang, Malaysia where they have been produced as a waste product. Then, the samples will be milled, sieved and separated in fractions. The size of the samples will be varies in order to study the influence of the particle size in the yield and properties of activated carbon produced.

## 3.4 EXPERIMENTAL PROCEDURE AND TEST CONDUCTED

There are 2 types of tests that will be conducted to determine the adsorption properties:

1. Langmuir Adsorption Isotherm
2. Iodine Adsorption

### 3.4.1 *Langmuir Adsorption Isotherm*

#### *Apparatus and Materials*

- Reaction bottles
- Burette
- Mechanical shaker
- Filter paper
- Conical flask
- 0.5 N oxalic acid
- Distilled water
- Filter paper
- Standardized  $\text{KMnO}_4$  solution

#### *Procedure*

1. 500 mL of 0.5 N oxalic acid are prepared. Then, five reaction bottles are taken and labelled.
2. 2 g of activated carbon are weighed and transferred into each of the bottles.
3. After that, by means of burette 50, 40, 30, 20 and 10 mL of 0.5 N oxalic acid are added followed by 0, 10, 20, 30 and 40 ml of distilled water so that the volume (50 mL) remains constant in each bottle.
4. These bottles are shaken thoroughly for an hour by mechanical shaker and they are set aside in a trough containing water to reach equilibrium.
5. The supernatant liquid of each bottle are filtered through a small dry filter paper and the filtrate are collected in properly labelled conical flasks.

6. The initial 5 mL or 10 mL of the filtrate will be rejected. 10 mL of the filtrate are pipette out into a clean conical flask. Then, it is titrated against standardized  $\text{KMnO}_4$  solution until a pink colour appears.
7. The titrations are repeated to get concordant values. From the titration values, the concentration of oxalic acid remaining and the amount of oxalic acid adsorbed are be calculated.
8. Lastly,  $\log(x/m)$  against  $\log C_e$  are plotted. The slopes and intercepts of the plot will give  $1/n$  and  $\log k$  respectively.

### ***3.4.2 Iodine Adsorption***

#### ***Apparatus and Materials***

- Glass stoppered flasks, pipettes, burette
- Folded filters, Whatman 2V, 18 cm  $\varnothing$
- Hot – plate
- Hydrochloric acid, 5% (w/w)
- Sodium thiosulphate solution, 0.10 N, standardized
- Iodine solution, 0.10 N, standardized.
- Starch solution

#### ***Procedure***

1. 0.7 – 2.0 g of the dried carbon are weighed and transferred to a dry, glass stoppered 250 mL Erlenmeyer flask.
2. 10 mL of 5% HCl are pipetted into the flask and swirled until the activated carbon is wetted.
3. The flasks are placed on a hot – plate and the contents will be brought to the boil and allowed to boil for 30 seconds.
4. The flask and the contents are allowed to cool to room temperature. Then, 100 mL of 0.10 N iodine solutions are added by using pipette.
5. The flask are stopper immediately and shaken vigorously for 30 seconds. Then, it is filtered by gravity through a filter paper immediately after 30 seconds shaking period.
6. The initial 20 – 30 mL of titrate are discarded and the remainder are collected in a clean beaker.
7. The filtrate are stirred in the beaker with a glass rod and 50 mL is pipetted into a 250 mL Erlenmeyer flask.
8. The 50 mL sample is titrated with 0.10 N sodium thiosulphate solution until the yellow colour almost disappeared. About 1 mL of starch solution will be added and

the titration will continue until the blue indicator colour just disappears. The volume of sodium thiosulphate solution used will be recorded.

### Result

Calculate the iodine number,  $I_n$  of the carbon using the equation :

$$I_n = X/M \times (A)$$

Where

X = mg of iodine adsorbed by the activated carbon  
 $(12.693 N_1) - 270.246 N_2 V$

Herein

$N_1$  = normality of iodine solution

$N_2$  = normality of sodium thiosulphate solution

V = volume of sodium thiosulphate solution in mL

M = mass of activated carbon in g

A = correction factor, depending on the residual normality,  $N_r$ , of the filtrate  
This factor A may be applied if  $N_r$  is between 0.008 and 0.0334 N.

$$N_r = N_2 (V/50)$$



### CORRECTION FACTOR FOR IODINE ADSORPTION

Residual filtrate Normality	0.0000	0.0001	0.0002	0.0003	0.0004	0.0005	0.0006	0.0007	0.0008	0.0009
0.0080	1.1625	1.1613	1.1600	1.1575	1.1550	1.1533	1.1513	1.1500	1.1475	1.1463
0.0090	1.1438	1.1425	1.1400	1.1375	1.1363	1.1350	1.1325	1.1300	1.1288	1.1275
0.0100	1.1250	1.1238	1.1225	1.1213	1.1200	1.1175	1.1163	1.1150	1.1138	1.1113
0.0110	1.1100	1.1088	1.1075	1.1063	1.1038	1.1025	1.1000	1.0988	1.0975	1.0963
0.0120	1.0950	1.0938	1.0925	1.0900	1.0888	1.0875	1.0863	1.0850	1.0838	1.0825
0.0130	1.0800	1.0788	1.0775	1.0763	1.0750	1.0738	1.0725	1.0713	1.0700	1.0688
0.0140	1.0675	1.0663	1.0650	1.0625	1.0613	1.0600	1.0583	1.0575	1.0563	1.0550
0.0150	1.0538	1.0525	1.0513	1.0500	1.0488	1.0475	1.0463	1.0450	1.0438	1.0425
0.0160	1.0413	1.0400	1.0388	1.0375	1.0375	1.0363	1.0350	1.0333	1.0325	1.0313
0.0170	1.0300	1.0288	1.0275	1.0263	1.0250	1.0245	1.0238	1.0225	1.0208	1.0200
0.0180	1.0200	1.0188	1.0175	1.0163	1.0150	1.0144	1.0138	1.0125	1.0125	1.0113
0.0190	1.0100	1.0088	1.0075	1.0075	1.0063	1.0050	1.0050	1.0038	1.0025	1.0025
0.0200	1.0013	1.0000	1.0000	0.9988	0.9975	0.9975	0.9963	0.9950	0.9950	0.9938
0.0210	0.9938	0.9925	0.9925	0.9913	0.9900	0.9900	0.9888	0.9875	0.9875	0.9863
0.0220	0.9863	0.9850	0.9850	0.9838	0.9825	0.9825	0.9813	0.9813	0.9800	0.9788
0.0230	0.9788	0.9775	0.9775	0.9763	0.9763	0.9750	0.9750	0.9738	0.9738	0.9725
0.0240	0.9725	0.9708	0.9700	0.9700	0.9688	0.9688	0.9675	0.9675	0.9663	0.9663
0.0250	0.9650	0.9650	0.9638	0.9638	0.9625	0.9625	0.9613	0.9613	0.9606	0.9600
0.0260	0.9600	0.9588	0.9588	0.9575	0.9575	0.9563	0.9563	0.9550	0.9550	0.9538
0.0270	0.9538	0.9525	0.9525	0.9519	0.9513	0.9513	0.9506	0.9500	0.9500	0.9488
0.0280	0.9488	0.9475	0.9475	0.9463	0.9463	0.9463	0.9450	0.9450	0.9438	0.9438
0.0290	0.9425	0.9425	0.9425	0.9413	0.9413	0.9400	0.9400	0.9394	0.9388	0.9388
0.0300	0.9375	0.9375	0.9375	0.9363	0.9363	0.9363	0.9363	0.9350	0.9350	0.9346
0.0310	0.9333	0.9333	0.9325	0.9325	0.9325	0.9319	0.9313	0.9313	0.9300	0.9300
0.0320	0.9300	0.9294	0.9288	0.9288	0.9280	0.9275	0.9275	0.9275	0.9270	0.9270
0.0330	0.9263	0.9263	0.9257	0.9250	0.9250					

Reprinted from B 600-78 — *AWWA Standard for activated carbon*, by permission.  
 Copyright © 1978, the American Water Works Association.

Figure 3.1: Correction Factor for Iodine Adsorption

## CHAPTER 4 RESULTS AND DISCUSSIONS

### 4.1 Overview

The effects of temperature and heating constant are the key factors during the preparation of this activated carbon in order to optimize the best condition in producing the activated carbons. The highest surface area is obtained when the temperature at 600°C when compared to 700°C and 800°C. This is because materials which have undergone deep carbonization accompanied by removal of large amount of volatile organic compounds are converted into solid residues with low contents of organic material and higher content of ash. From the results obtained, the mechanically grinded seeds husks give the largest surface area, hence it is the best choice to produce the activated carbon from this size.

### 4.2 Results

#### MANUALLY REMOVED SEEDS HUSKS

TEMPERATURE (°C)	SURFACE AREA (m <sup>2</sup> /g)
600	0.0020
700	0.0012
800	0.0008

Table 4.1: Manually Removed Seeds Husks

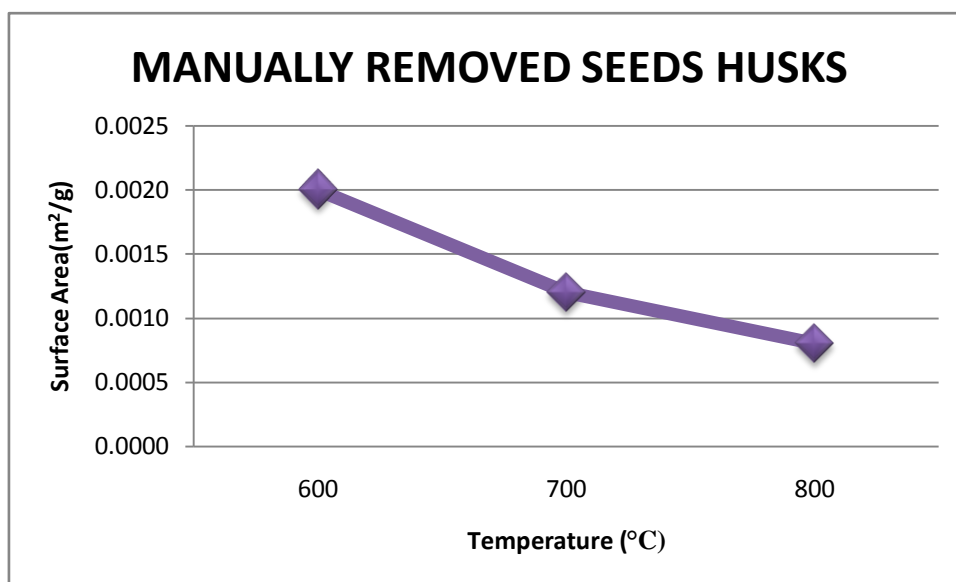


Figure 4.1: Activated Carbon Surface Area at Different Temperatures

From the above results, it showed that the optimum temperature for the production of activated carbon is 600°C as it give the largest surface area when compared with other temperatures. If the temperature beyond 800°C, then the volatile matter is minimal above this range. This is because the fixed carbon and ash content increased which may attribute to the removal of volatile matter in the husks.

### **GRINDED HUSKS (MANUALLY REMOVED SEEDS HUSKS)**

Table 4.2: Grinded Husks (Manually Removed Seeds Husks)

TEMPERATURE (°C)	SURFACE AREA (m <sup>2</sup> /g)
600	0.0026
700	0.0014
800	0.0007

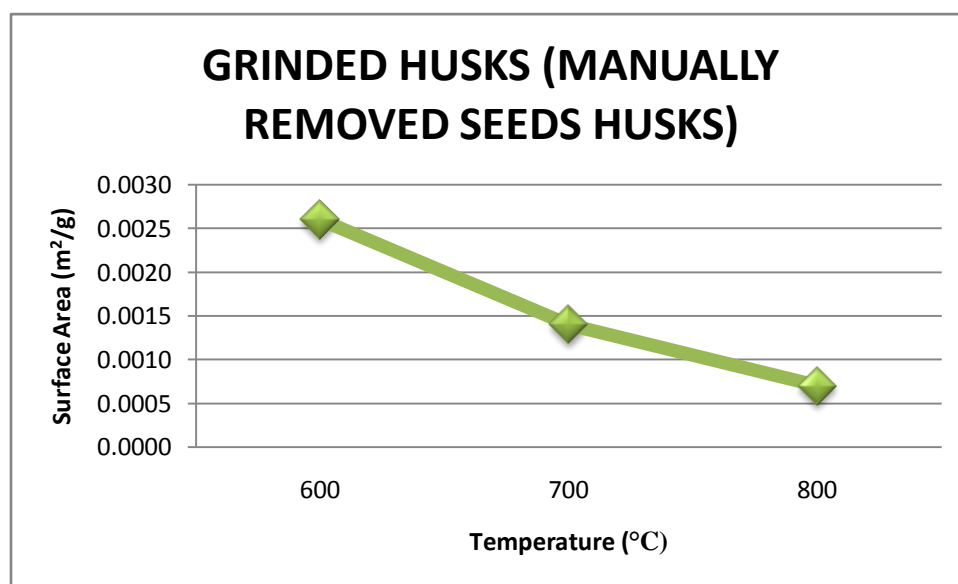


Figure 4.2: Activated Carbon Surface Area at Different Temperatures

## MECHANICALLY REMOVED SEEDS HUSKS

Table 4.3: Mechanically Removed Seeds Husks

TEMPERATURE(°C)	SURFACE AREA(m <sup>2</sup> /g)
600	0.003
700	0.0013
800	0.0005

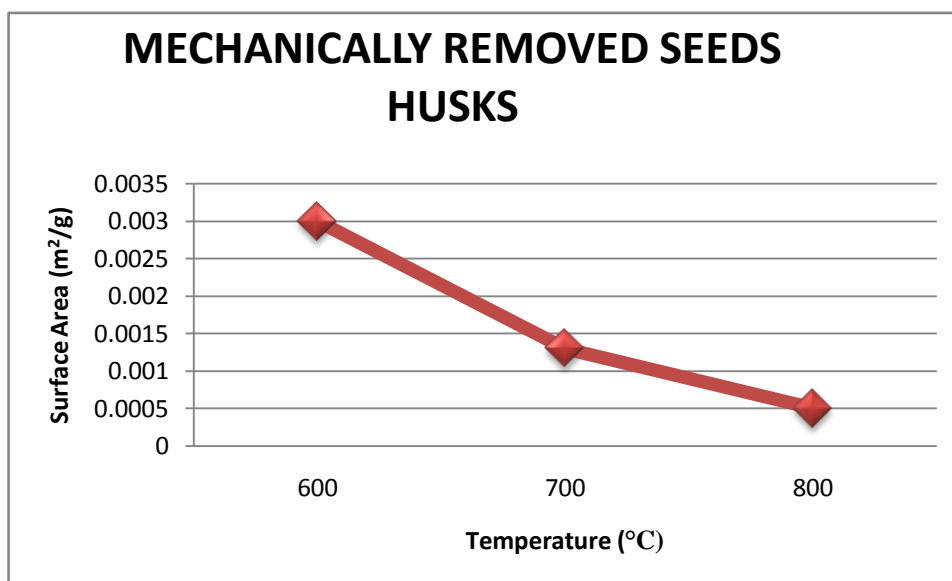


Figure 4.3: Activated Carbon Surface Area at Different Temperatures

From the above results, it can be showed that the best temperature is 600°C. This is because it give the larger surface area when compare with other temperatures. This is occurred because the percentage volatile matter decreased with an increased of temperature. If the temperature beyond 800°C, then the volatile matter is minimal above this range. This is because the fixed carbon and ash content increased which may attribute to the removal of volatile matter in the husks.

## LANGMUIR ADSORPTION ISOTHERM

The Langmuir adsorption isotherm relates the coverage or adsorption of molecules on a solid surface to concentration of a medium above the solid surface at a fixed temperature. During the test, a sample of activated carbon is contacted for some time with a test substance. The graph of  $\log x/m$  versus  $\log c$  are plotted which  $x$  is amount of impurities absorbed at equilibrium,  $m$  is mass of the activated carbon and  $c$  is the concentration of impurities remained in liquid. From the data that we gained from the experiments, it shown that the three different sizes of husks have absorbance properties. The best activated carbon with greatest absorbance properties can be gained from grinded pods and seeds husks using grinder, as it give the best  $R^2$  values when compared with others.

### MANUALLY REMOVED SEEDS HUSKS

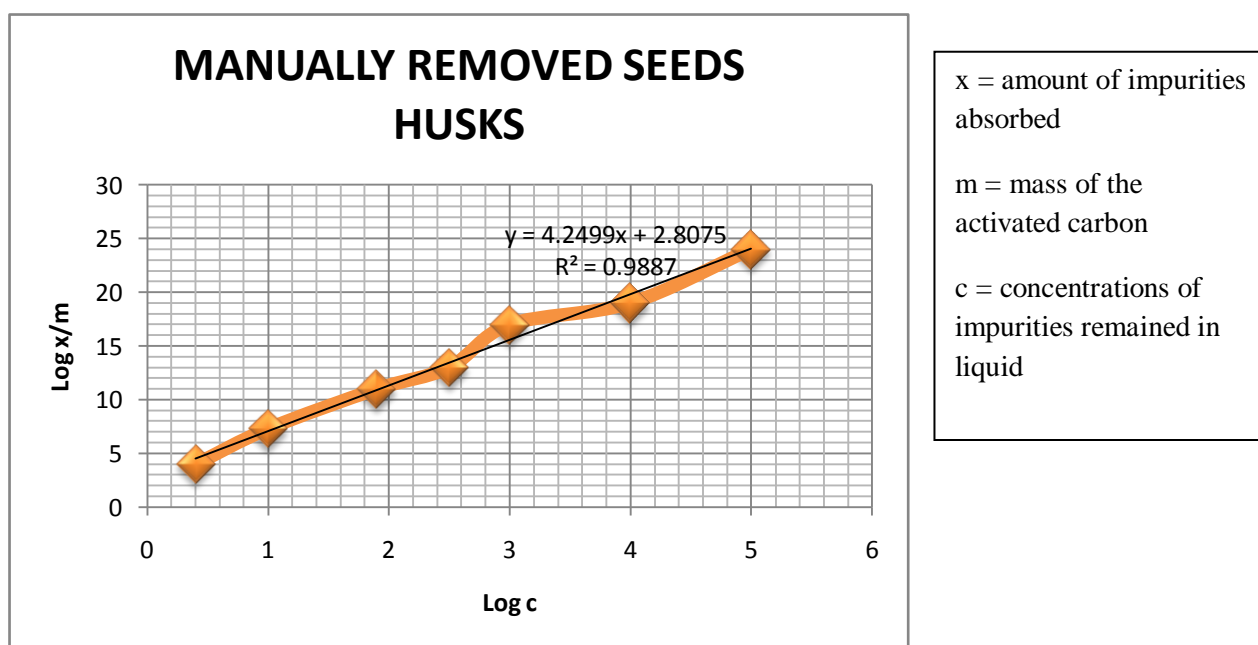


Figure 4.4: Langmuir Adsorption Isotherm of Manually Removed Seeds Husks

From this experiment, the amount of activated carbon,  $m$  is fixed which is 2g. Then, 50 ML of oxalic acid is titrated against standardized  $\text{KMnO}_4$  until the pink colour appeared. The amount of oxalic acid adsorbed,  $x$ , until the solution turned pink is recorded. After that, the concentration of the oxalic acid is recorded. Then, graph of  $\log (x/m)$  against  $\log c$  is plotted.

## GRINDED HUSKS (MANUALLY REMOVED SEED HUSKS)

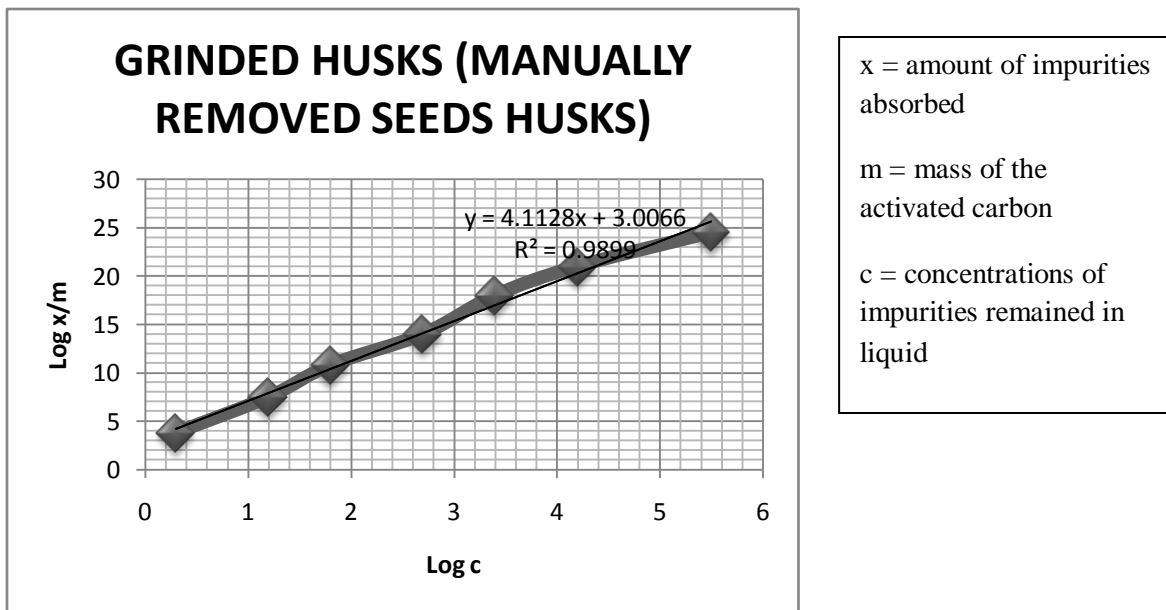


Figure 4.5: Langmuir Adsorption Isotherm of Grinded Husks (Manually Removed Seeds Husks)

From this experiment, the amount of activated carbon,  $m$  is fixed which is 2g. Then, 50 ml of oxalic acid is titrated against standardized  $\text{KMnO}_4$  until the pink colour appeared. The amount of oxalic acid adsorbed,  $x$ , until the solution turned pink is recorded. After that, the concentration of the oxalic acid is recorded. Then, graph of  $\log (x/m)$  against  $\log c$  is plotted to show that the activated carbon has adsorption properties.

## MECHANICALLY REMOVED SEEDS HUSKS

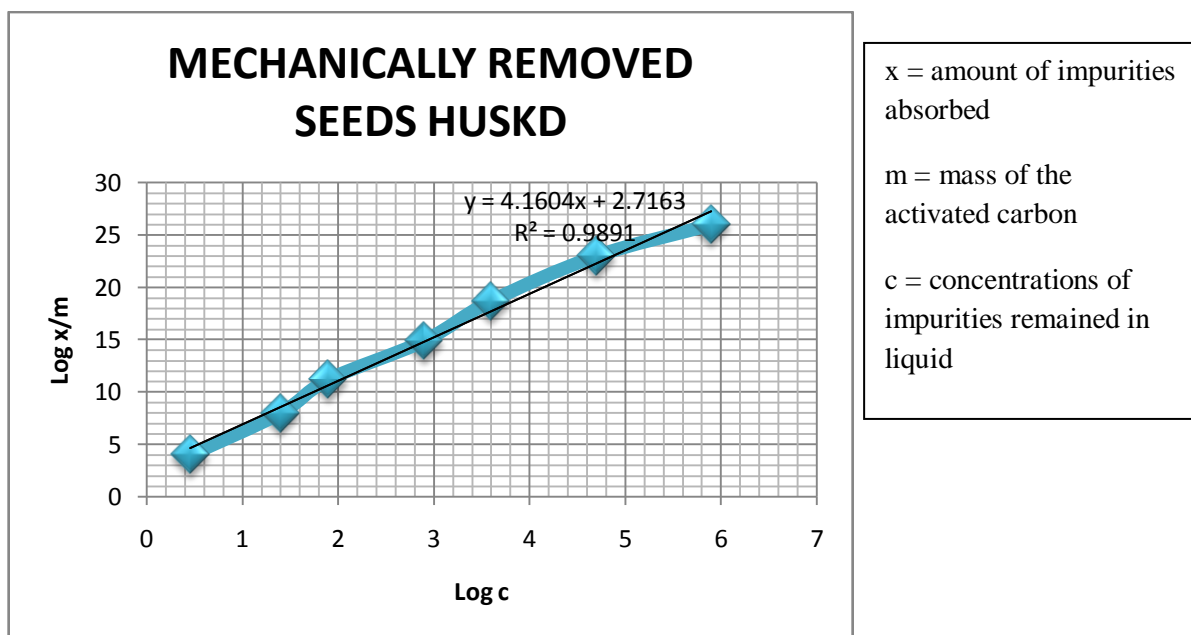


Figure 4.6: Langmuir Adsorption Isotherm of Mechanically Removed Seeds Husks

From this experiment, the amount of activated carbon, m is fixed which is 2g. Then, 50 ML of oxalic acid is titrated against standardized  $\text{KMnO}_4$  until the pink colour appeared. The amount of oxalic acid adsorbed, x, until the solution turned pink is recorded. After that, the concentration of the oxalic acid is recorded. Then, graph of  $\log (x/m)$  against  $\log c$  is plotted to show the adsorption properties of the activated carbon.

## IODINE TEST

The iodine test is carried out to determine the iodine number which gives indication of the internal surface area of activated carbon. It is defined as the number of milligrams of iodine adsorbed from an aqueous solution by 1 g of activated carbon. The higher the iodine number, the higher the surface area of the activated carbon. From the results obtained, we can conclude that mechanically grinded pods and seeds husks have the highest iodine number compare to the other sizes. The highest iodine number indicated that the activated have larger surface area. Hence, the mechanically grinded pods and seeds husks are the most recommended sizes in producing the activated carbon.

## MANUALLY REMOVED SEEDS HUSKS

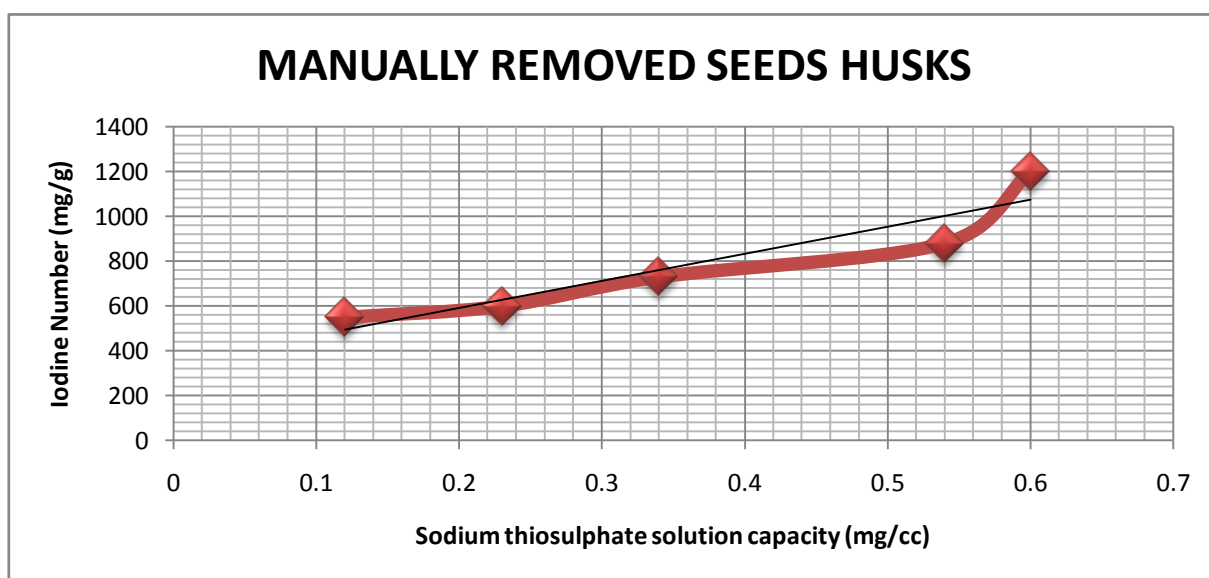


Figure 4.7: Iodine Test of Manually Removed Seeds Husks



## GRINDED HUSKS (MANUALLY REMOVED SEEDS HUSKS)

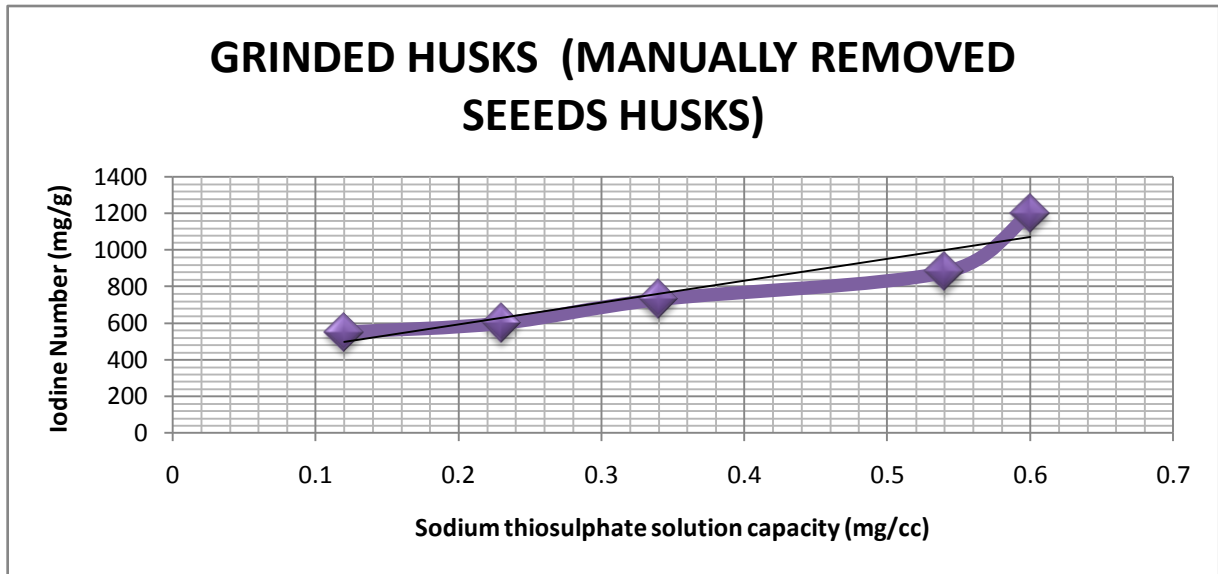


Figure 4.8: Iodine Test of Grinded Husks (Manually Removed Seeds Husks)

## MECHANICALLY REMOVED SEEDS HUSKS

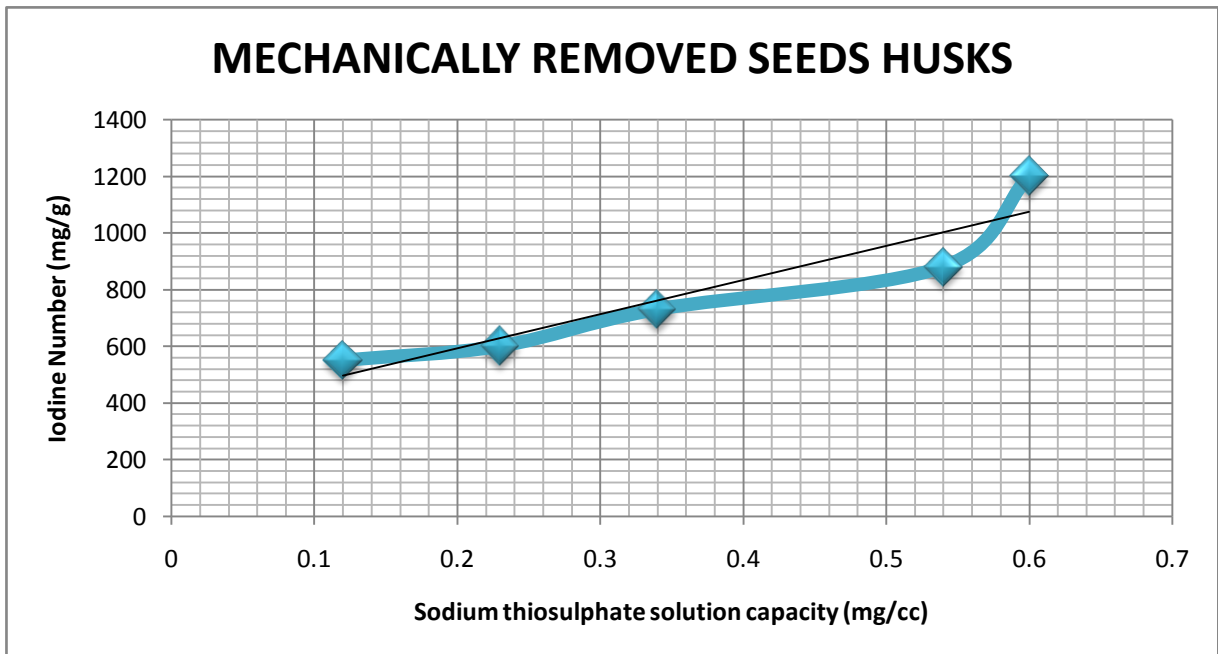


Figure 4.9: Mechanically Removed Seeds Husks

## MASS DECOMPOSITION OF SEEDS HUSKS USING TGA

From the results obtained, it can be shown that the mass decrease as the temperature increase. This is because the temperature will cause the production yield to be half of the original mass used. For example, if 10g of seeds husks are used, then after pyrolysis, we can only gained about 5g of activated carbon. This means it loss about 50% of it mass when it is heated.

## MASS DECOMPOSITION OF MANUALLY REMOVED SEEDS HUSKS

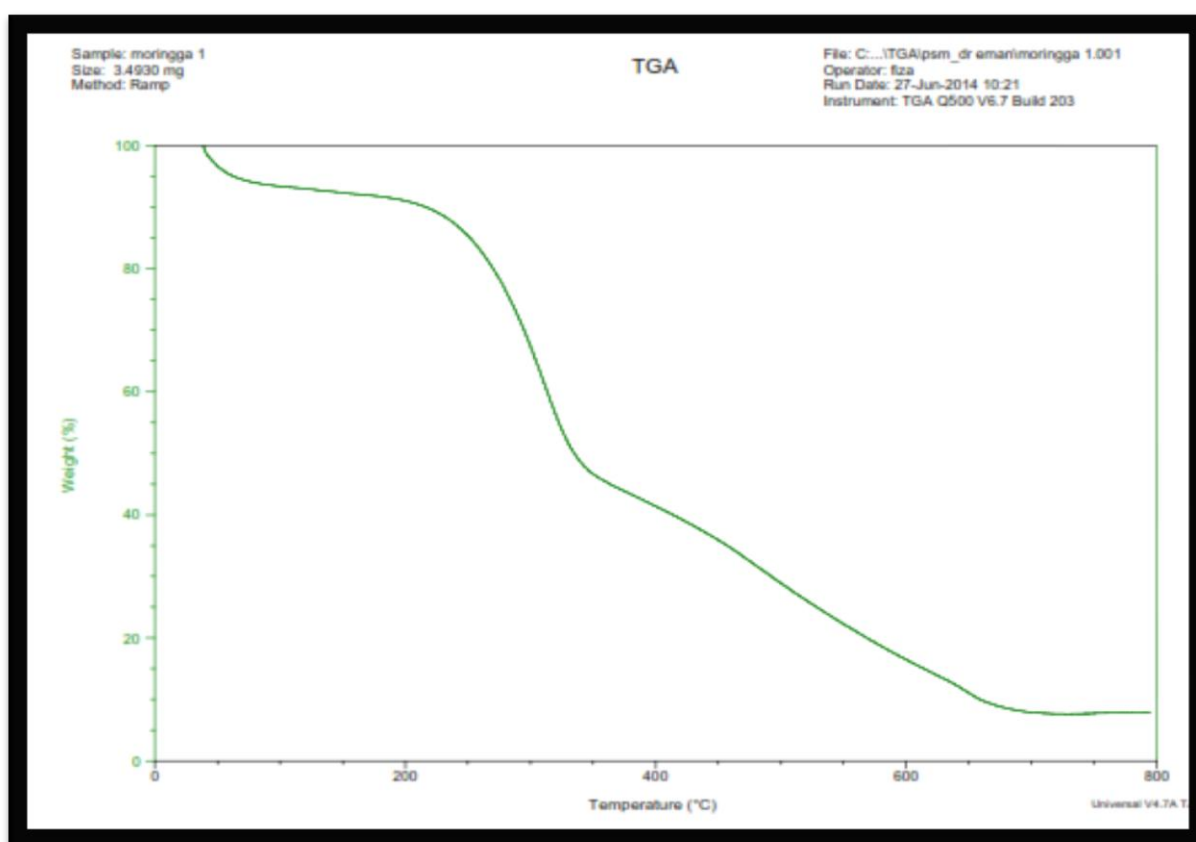


Figure 4.10: Mass Decomposition of Manually Removed Seeds Husks

## MASS DECOMPOSITION OF MECHANICALLY REMOVED SEEDS HUSKS

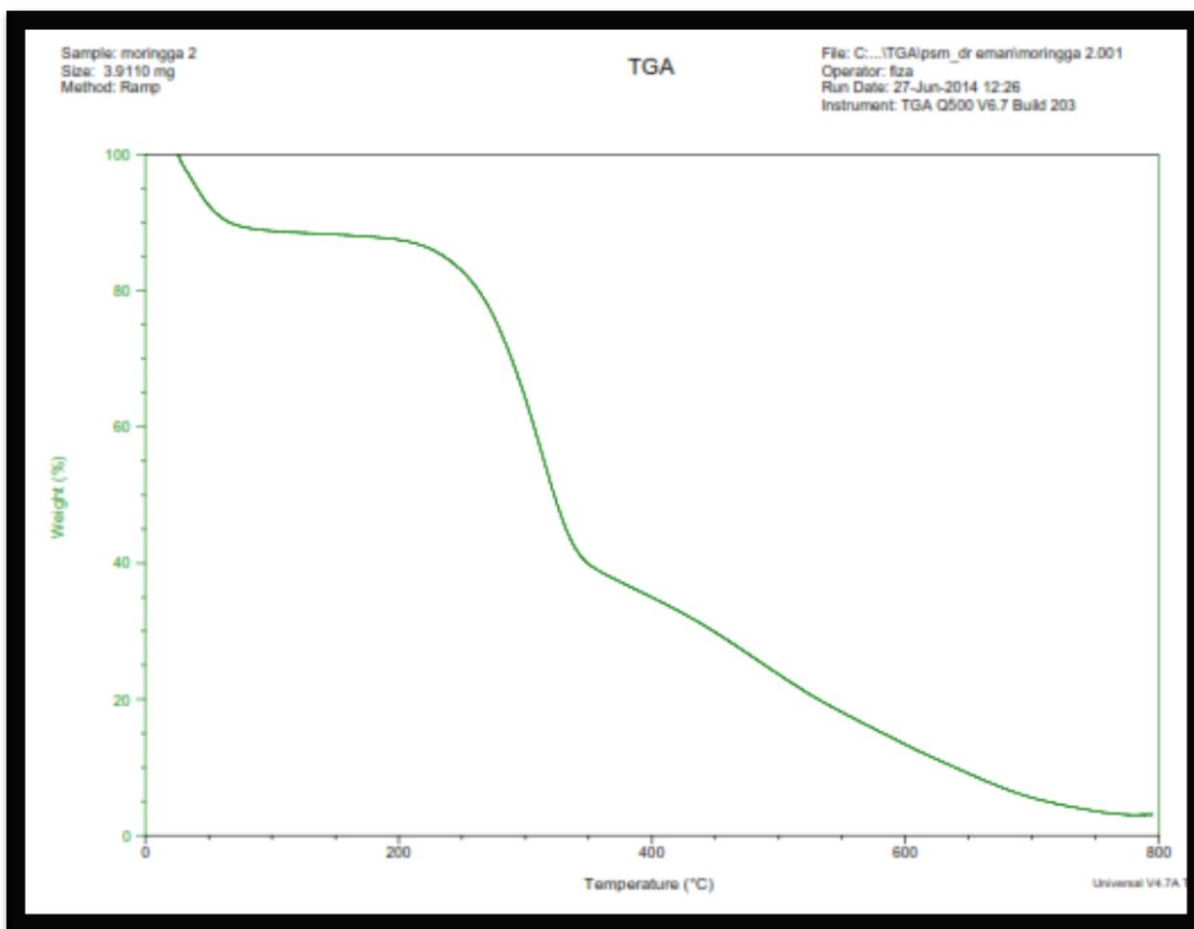


Figure 4.11: Mass Decomposition of Mechanically Removed Seeds Husks

## MASS DECOMPOSITION OF GRINDED HUSKS (MANUALLY REMOVED SEEDS HUSKS)

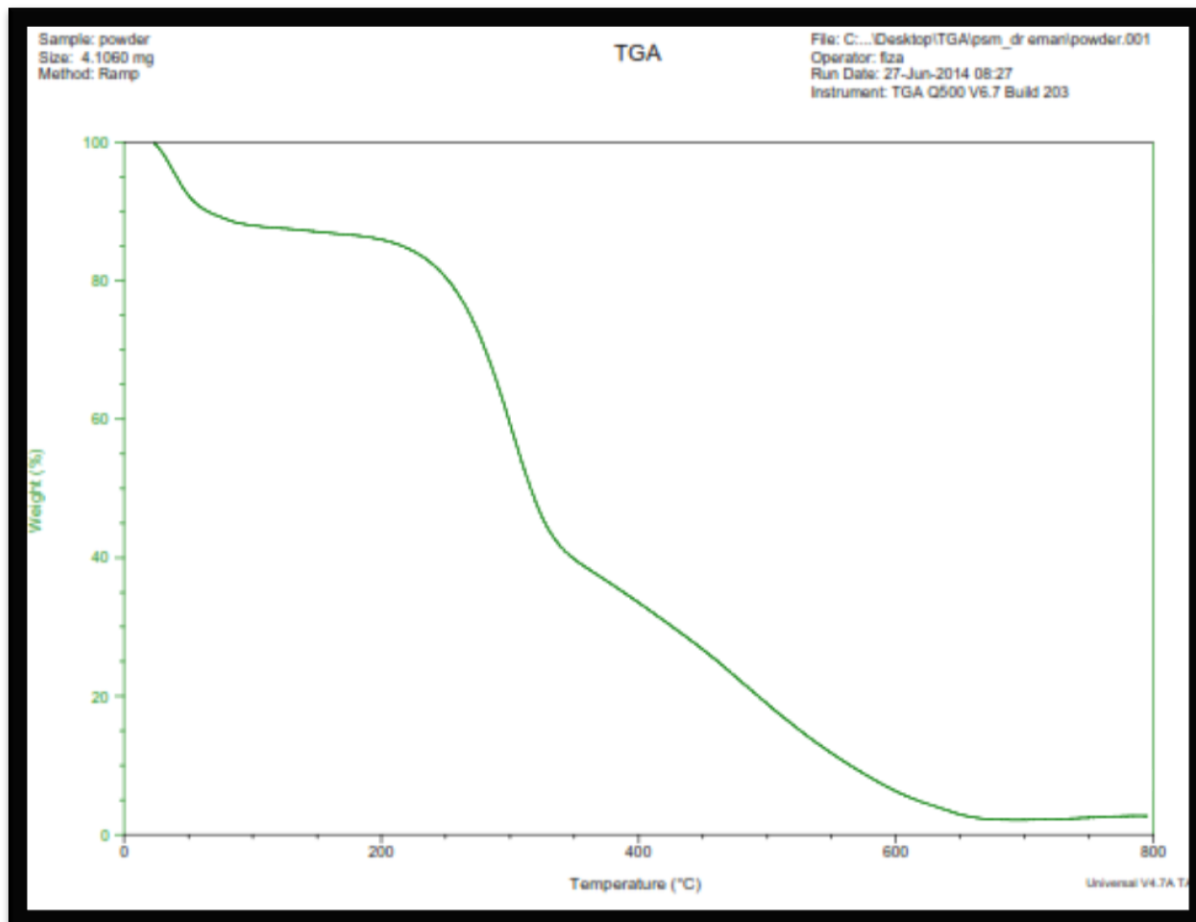


Figure 4.12: Mass Decomposition of Grinded Husks (Manually Removed Seeds Husks)

**YIELD OF PRODUCTION OF ACTIVATED CARBON FROM DIFFERENT TYPES OF *MORINGA OLEIFERA* SEEDS HUSKS WHEN UNDERGONE PYROLYSIS**

Table 4.4: Yield of Production of Activated Carbon from different types of *Moringa oleifera* Seeds Husks

Types	Mass (g)										
	1st	2nd	3rd	4th	5th	6th	7th	8th	9th	10th	Average
Manually Removed Seeds Husks	5.20	5.11	5.18	5.23	5.15	5.09	5.27	5.13	5.29	4.98	<b>5.163</b>
Grinded Husks (Manually Removed Seeds Husks)	4.72	4.93	5.02	5.16	4.33	4.79	4.65	4.87	4.69	4.53	<b>4.769</b>
Mechanically Removed Seeds Husks	5.06	5.14	5.27	4.96	5.23	5.02	4.88	5.16	5.08	5.11	<b>5.091</b>

From the data observed, it shown that the mass of *Moringa oleifera* seeds husks decomposed nearly 50% per reaction when undergone pyrolysis. The mass of the initial *Moringa oleifera* inserted in the reactor is 10g in each cycle. Since the reactor is small in size, thus several cycles need to carry out in order to get adequate amount of activated carbon.

**COMPARISON OF PRODUCED ACTIVATED CARBON WITH THE  
ACTIVATED CARBON AVAILABLE IN THE MARKET**

Table 4.5: Comparison of Produced Activated Carbon with the Activated Carbon Available in the Market

Activated Carbon	Activated Carbon from Manually Removed Seeds Husks	Activated Carbon from Grinded Husks (Manually Removed Seeds Husks)	Activated Carbon from Mechanically Removed Seeds Husks	Activated Carbon in the Market
Surface Area (m <sup>2</sup> /g)	0.0020	0.0026	0.003	0.0013
Adsorption (Value of R <sup>2</sup> )	0.9887	0.9899	0.9891	0.9881
Iodine Number (mg/g)	1250	1270	1260	1240

## 5 CONCLUSION

### 5.1 *Conclusion*

This undergraduate research project focus on the possibility of producing activated carbon from *Moringa oleifera* seeds husk. The tests are carried out give positive results as expected for producing activated carbon based on the parameters varied such as the temperature and the heating constant. Hence, the best temperature for producing the activated carbon is 600°C. The increasing of the temperature reduced the yield of the activated carbon. Hence, if the temperature beyond 800°C then the production yield will decrease and reduced the absorbance properties. From the results obtained, it can be observed that the best size of husks in producing the activated carbon is the mechanically grinded husks. As it has the properties to remove the colour of impurities, then it can be commercialize in food industry which may help in decolourisation of the food products.

### 5.2 *Future work*

As the results of this research work are positive. Then, the future generation may use *Moringa oleifera* in producing activated carbon as an alternative to replace the activated carbon available nowadays.

## REFERENCES

- AWWA Standard for activated carbon (1978). Reprinted from B 600-78 The American Water Works Association..
- Baker F. S., Miller C. E., Repic A. J. and Tolles E. D., (1992) Activated carbon. Kirk-Othmer Encyclopedia of Chemical Technology, 4, 1015-1037.
- Das C.P and Patnaik L. N (2001). 'Use of Industrial Waste for Reduction of COD from Paper Mill Effluent. Indian Journal Environmental Health. 43(1), 21-27
- De, A. K. and De, A. K (1994). Heavy Metals Removal from Waste Water Using Fly Ash and Agricultural Wastes – A Review. Journal. IAEM, 21,36-39.
- El-Hendawy A. A., (2003) Influence of HNO<sub>3</sub> Oxidation on the Structured and Adsorptive Properties of Corncob Activated Carbon, 41, 713-722.
- Fuglie, L. J., (1999). *The Miracle Tree Moringa oleifera: natural nutrition for the tropics*. Dakar.
- Ghebremichael K.A. (2004). Moringa Seed and Pumice as Alternative Natural Materials for Drinking Water Treatment.
- Goyal, M, Rattan V.K and Bansal (1999). Adsorption of Nickel from Aqueous Solutions by Activated Carbons. Indian Journal of Chemical Technology, 6, 305-312.
- Kadirvel K, Kavipriya M, Karthika C, Radhika M. Vennilamani W and Pattabhi S (2003). Utilization of Various Agricultural Wastes for Activated Carbon Preparation And Application For The Removal Of Dyes And Metal Ions From Aqueous Solution. Ioresource Technology 87, 129-132.
- Khadija Qureshi, Inamullah Bhatti, Rafique Kazi and Abdul Khaliq Ansari (2008) Physical and Chemical Analysis of Activated Carbon Prepared from Sugarcane Bagasse and Use for Sugar Decolorisation.
- Martinez, M.L. , Moiraghi, L. , Agnese, M. And Guzman, C (2003) Making and Some Properties of Activated Carbon Produces From Agricultural Industrial Residues from Argentina.
- Mattson J. S. and Mark H. B., Activated Carbon, New York: Dekker, (1971).



- Pollard, S. J. T., Thompson, F. E. and McConnachie, G. L., (1995). Microporous Carbon from *Moringa oleifera* Husks for Water Purification in Less Developed Countries. *Water. Res.*, 29(1): 337-347.
- Rao M. and Bhole A.G (2002). ‘ Removal of Organic Matter from Dairy Industry Wastewater using Low - Cost Adsorbents’ *Indian Chem.Eng Section A*, 44(1). 25-28.
- Warthust A. M., Fowler G. D., McConnachie G. L. and Pollard S. J. T. (1997) Pore Structure and Adsorption Characteristics of Steam Pyrolysis Carbons from *Moringa oleifera*. *Carbon*. In press.
- Warthust A. M., McConnachie G. L. and Pollard S. J. T. (1996) The Production of Activated Carbon for Water Treatment in Malawi from the Waste Seed Husks of *Moringa oleifera*. *Water Sci. Technol.* **34**(11), 177-184.
- Warthust A. M., McConnachie G. L. and Pollard S. J. T. (1997) Characterisation and Applications of Activated Carbon Produced from *Moringa Oleifera* Seed Husks by Single – Step Steam Pyrolysis. *Wat. Res.* **31**(4), 759-766.
- Z. Al-Qodah and R. Shawabkah. (2009) Production and Characterization of Granular Activated Carbon from Activated Sludge.
- Zanzi R., Bai X., Capdevila P., and Bjornbom E. (2001) Pyrolysis of Biomass of Activated Carbon, Liquid and Gaseous Products.
- Zongxuan Jiang, You Lin, Xiuping Sun, Fuping Tain, and Fuxia Sun, Changhai Liang, Wansheng You, Chongren Han, Can Li. (2003) Activated carbon chemically modified by concentrated H<sub>2</sub>SO<sub>4</sub> for the adsorption of pollutant from Wastewater and the dibenzothiophene from fuel oils. *Langmiur*, 19, 731-736.

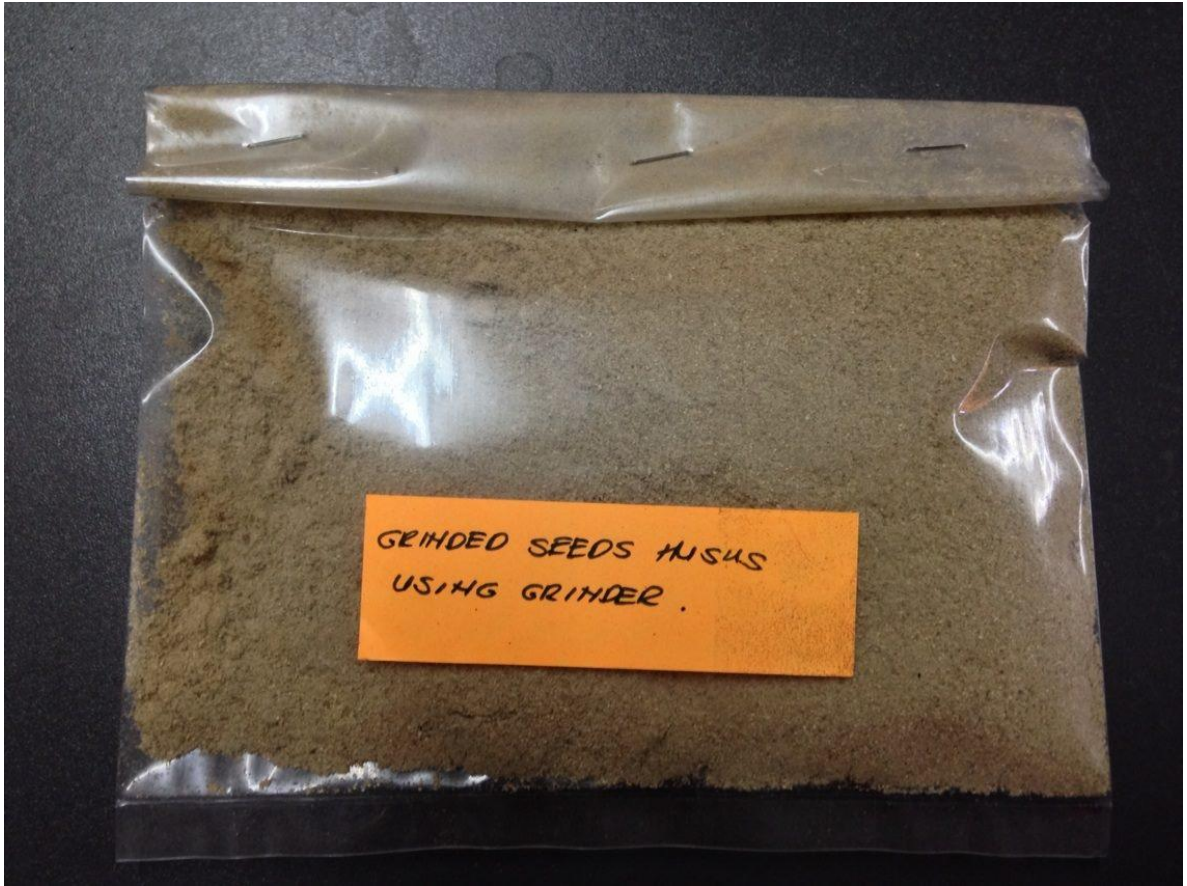
## APPENDICES



Appendix 1: Manually Removed Seeds Husks



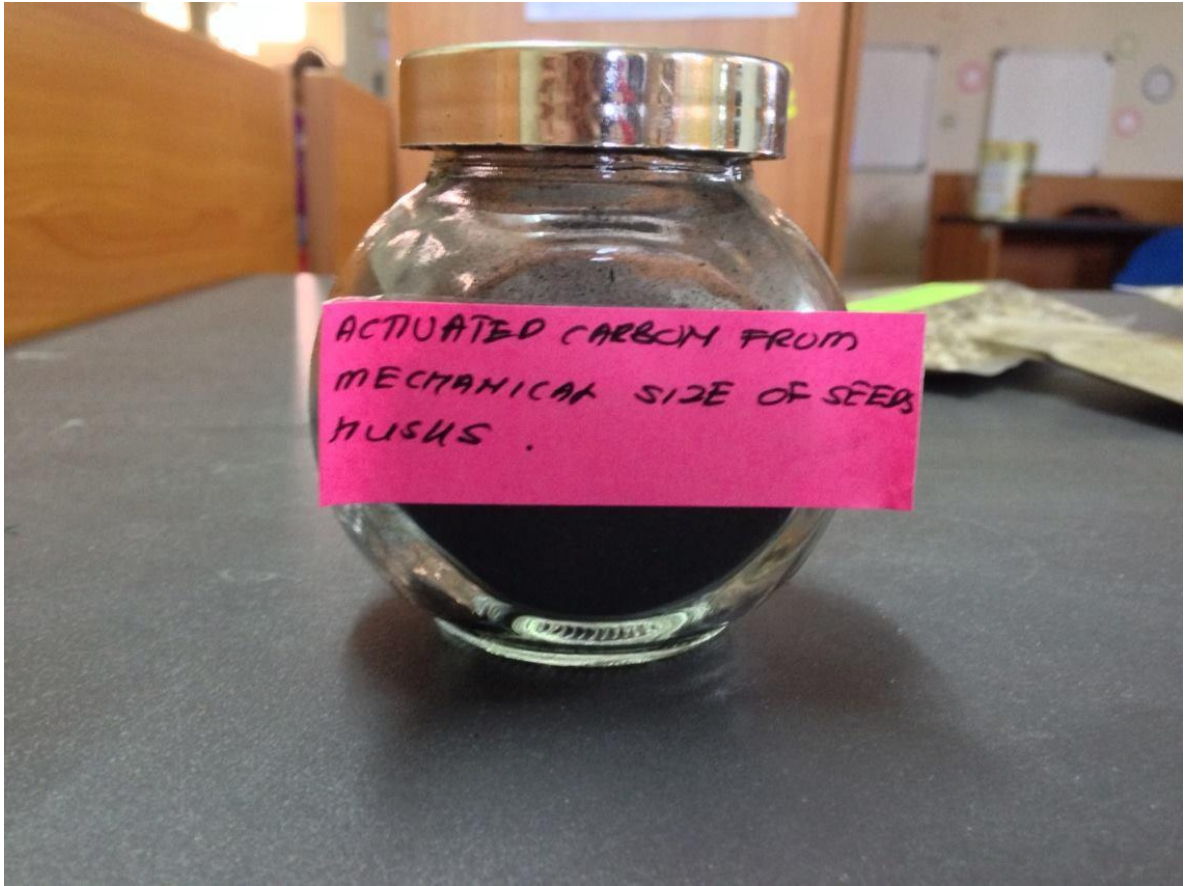
Appendix 2: Mechanically Removed Seeds Husks



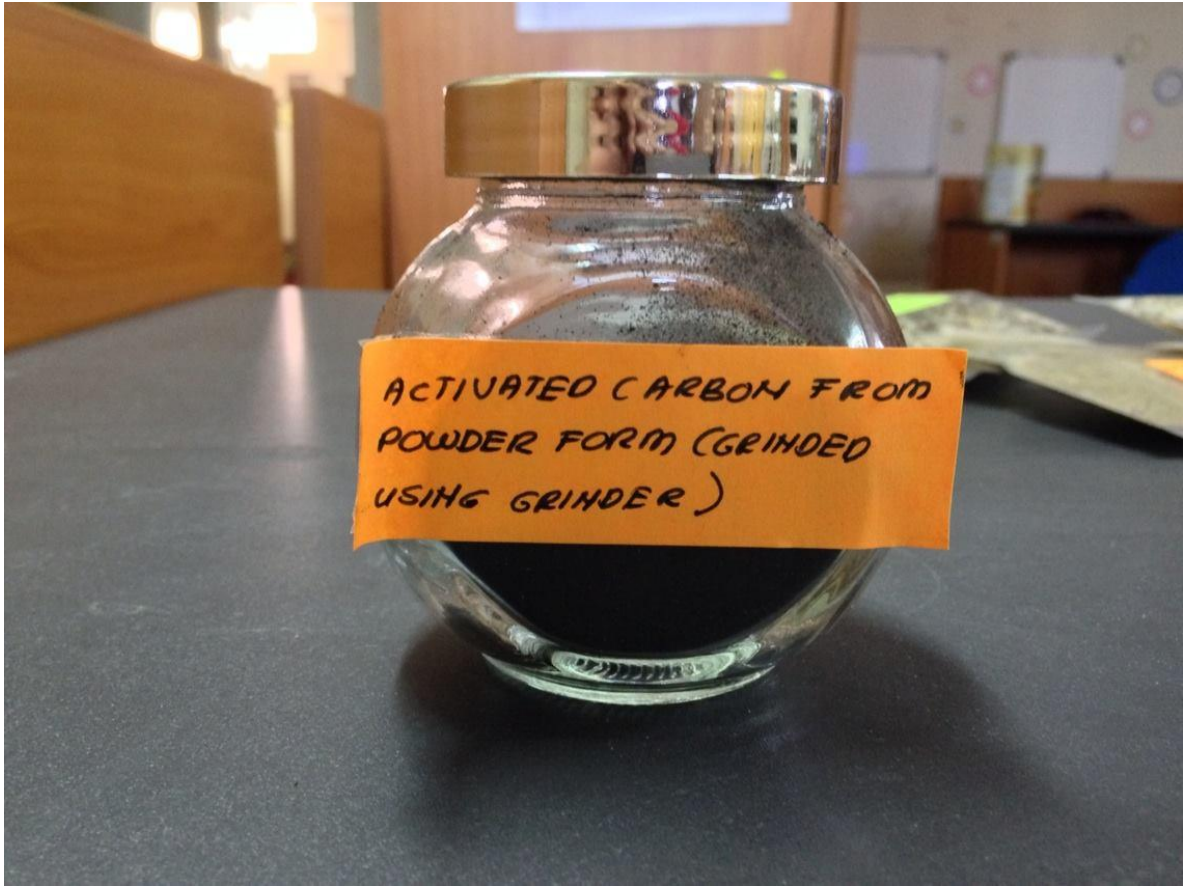
Appendix 3: Grinded Husks (Manually Removed Seeds Husks)



Appendix 4: Activated Carbon Produced from Manually Removed Seeds Husks



Appendix 5: Activated Carbon from Mechanically Removed Seeds Husks



Appendix 6: Activated Carbon from Grinded Husks (Manually Removed Seeds Husks)