



**SYNTHESIS AND PREPARATION OF
ACTIVATED CARBON FROM *COCOS
NUCIFERA L.* (COCONUT) SHELL AND
SUGARCANE BAGASSE**

by

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A report submitted in fulfillment of the requirements for the degree of
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2017

DECLARATION

I declare that this thesis entitled “Synthesis and Preparation of Activated Carbon from *Cocos nucifera L.*(coconut) shell and Sugarcane Bagasse” is the result of my own research except as cited in the references. The thesis has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.

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May ALLAH bless all of us and only HE, The Almighty could repay all my debts to all of you. I say a big thank you.

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**Synthesis and Preparation of Activated Carbon from *Cocos nucifera L*
(Coconut) Shell and Sugarcane Bagasse**

ABSTRACT

Activated carbon can be produced from carboneous materials. The aims of this study are to synthesis and characterize activated carbon from *Cocos nucifera L.* (coconut) shell and sugarcane bagasse by carbonization and activation process. The samples were carbonized and activated at 300°C in a furnace. As the concentration of activating agent increased, the percentage of weight loss also increased due to more volatiles in samples that were released. Addition of phosphoric acid (H₃PO₄) and Potassium hydroxide (KOH) gave the activated carbon more porous structure. The result indicated that the activated carbon derived from coconut shell has higher carbon content compared to sugarcane bagasse. Besides, the elements that contained in the samples were analysed using X-ray Fluorescent (XRF) and surface chemical was characterized using Fourier Transformation Infrared (FTIR). It was concluded that production of activation activated carbon from coconut shell and sugarcane bagasse can be used in a lot of applications like dye absorbent and waste water treatment.

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**Sintesis dan Penyediaan Karbon Teraktif daripada *Coconus nucifera L.*
(Tempurung Kelapa) dan Hampas Tebu**

ABSTRAK

Karbon teraktif boleh dihasilkan daripada bahan-bahan berkarbon. Kajian ini bertujuan untuk sintesis dan pencirian karbon diaktifkan dari *cocos nucifera L.* (tempurung kelapa) dan hampas tebu oleh proses karbonisasi dan proses pengaktifan. Sampel dikarbonisasi dan diaktifkan pada suhu 300 ° C di dalam relau. Apabila kepekatan peningkatan ejen bertambah, peratusan penurunan berat sampel akan meningkat kerana pemeruapan dalam sampel yang dikeluarkan. Penambahan asid fosforik (H₃PO₄) dan Kalium Hidroksida (KOH) memberi karbon diaktifkan lebih porous. Hasil kajian telah menunjukkan bahawa karbon yang diaktifkan yang berasal dari tempurung kelapa mempunyai kandungan karbon yang lebih tinggi berbanding dengan hampas tebu. Selain itu, unsur-unsur yang terkandung dalam sampel dianalisis menggunakan X-ray pendarfluor (XRF) dan permukaan kimia dicirikan menggunakan Fourier Transformasi Infrared (FTIR). Kajian ini juga bertujuan untuk menambahbaik sampel untuk menjadi lebih bermanfaat. Secara kesimpulannya, pengeluaran pengaktifan karbon dari tempurung kelapa dan hampas tebu boleh digunakan dalam banyak aplikasi seperti pewarna menyerap dan aplikasi lain.

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LIST OF ABBREVIATIONS

Abbreviation	Description
Zn	Zinc
H ₃ PO ₄	Phosphoric Acid
KOH	Potassium Hydroxide
SiO ₂	Silicon Oxide
FTIR	Fourier Transformation Infrared
XRD	X-Ray Diffraction
C	Carbon
O	Oxygen
Mg	Magnesium
Al	Aluminium
Fe	Iron
Na	Sodium
K	Potassium
He	Helium
Li	Lithium
H	Hydrogen
Al ₂ O ₃	Aluminium Oxide
MgO	Magnesium Oxide
Fe ₂ O ₃	Iron Oxide
SEM	Scanning Electron Microscope
BET	Brunauer- Emette Teller
S _{BET}	Surface Area by BET

V_T	Pore Volume
S_{mic}	Micropore Area
V_{mic}	Micropore Volume
V_{mes}	Mesopore Volume



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LIST OF SYMBOLS

Symbol	Description
$^{\circ}\text{C}$	Celcius Degree
=	Double Bond
—	Single Bond
θ	Theta
%	Percentage
h,k,l	Miller Indices
λ	Wavelength

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CHAPTER 1

INTRODUCTION

1.1 Background of Study

In recent years, activated carbon is very popular among the researchers because they are focusing on utilizing agricultural waste into beneficial usage. It is commonly derived from charcoal. The high concentration of carbon from any carboneous materials whether in plant, animal or mineral origin can produce activated carbon (Ansari 2009). Besides, activated carbon always increase in demand because the depletion of non-renewable materials sources (Guo *et al.*, 2009).

There are two continuous methods in producing activated carbon namely physical activation and chemical activation. Physical activation involves carbonization process followed by activation process of the raw material while chemical activation involves impregnation of raw materials and activated agent (Li *et al.*, 2008). Nowadays, there is abundance of agricultural byproducts as the sources of the raw material of activated carbon. The agricultural byproducts are inexpensive and the source can be easily found.

Coconut shell and sugarcane bagasse are the example of agricultural byproducts which do not have any economic value to be commercialized and may cause environment problem if they are not properly disposed. So, they can be converted into activated carbon. Coconut shells are available all year around and has advantage properties like high carbon content, low ash content, high yield,

mechanical strength and resistance to attrition (Popoola *et al.*, 2012).

From the previous study by Madakson *et al.*, (2012), coconut shells are becoming a new source of energy bio fuel. Nowadays, the waste materials are not just known as beneficial material but become worthy material because of its application and high demand in industrial applications. Besides, they are suitable in producing activated carbon because of their hardness and abrasion resistance. In addition, activated carbon can be used in a various application like in water treatment, gas cleaning, food and beverage manufacture, solvent recovery, medical and laboratory uses and minerals recovery (Mozammel *et al.*, 2002).

It also can treat the industrial wastewater for local industries and can reduce the cost of the wastewater treatment. When the powdered form of activated carbon is used, it is added to the water as a slurry and, after a suitable contact time, it is removed by clarification or filtration (F Rodriguez-Reinoso, 2016). In a study by Guo *et al.*,(2009), although activated carbon was the first recognized absorbent and is still widely used in industry, the development of appropriate methods to prepare the activated carbon and the understanding of its porous structure still continue.

1.2 Problem Statement

Activated carbon from coconut shell has larger surface area and has widely pore size distribution that can act as absorbent material (Din *et al.*, 2009). Most of the researchers who involve in this activated carbon research, only study on different activating agents that affect the morphology of activated carbon. The carbonization and activation process give effect on production of activated carbon. Hence, this study would focus in characterizing the activated carbon from coconut shell and sugarcane bagasse. The “waste to wealth” concept is applied in this study to transform the waste materials become valuable products that are widely used in industry nowadays.

1.3 Objectives

- i. To synthesis and characterize activated carbon from *Cocos nucifera L.* (coconut) shell and sugarcane bagasse by carbonization and activation process
- ii. To compare the synthesized activated carbon from *Cocos nucifera L.* (coconut) shell and sugarcane bagasse in different concentration of different activating agents

CHAPTER 2

LITERATURE REVIEW

2.1 Coconut Shell (*Cocos nucifera L.*)

Coconut plant is one of the secondary plants that the fibers come as a byproduct from some other primary utilization. This coconut plant is categorized in the species of *Areaceae* or known as palm family. Coconut plant is widely planted in Malaysia but the largest plantation is in Pacific Indonesia, Philippines, South Asia, East and West Africa and Caribbean. This species is cultivated to the all other countries (Gratuito *et al.*, 2008).

From the study by Okafor *et al.*, (2012), there are three layers of coconut shell which are exocarp, mesocarp and endocarp. For the mesocarp, it contains fibers that are known as coir. The coconut shell can be the source of activated carbon after treated with some of the treatment. Thus, it is beneficial for the country that has a lot of coconut shell because the activated carbon keeps expanding with newer applications like super-capacitors, electrodes, gas storage and so on. Popoola *et al.*, (2012) studied that the carbonaceous material like coconut shell is very easy to process to become activated carbon.

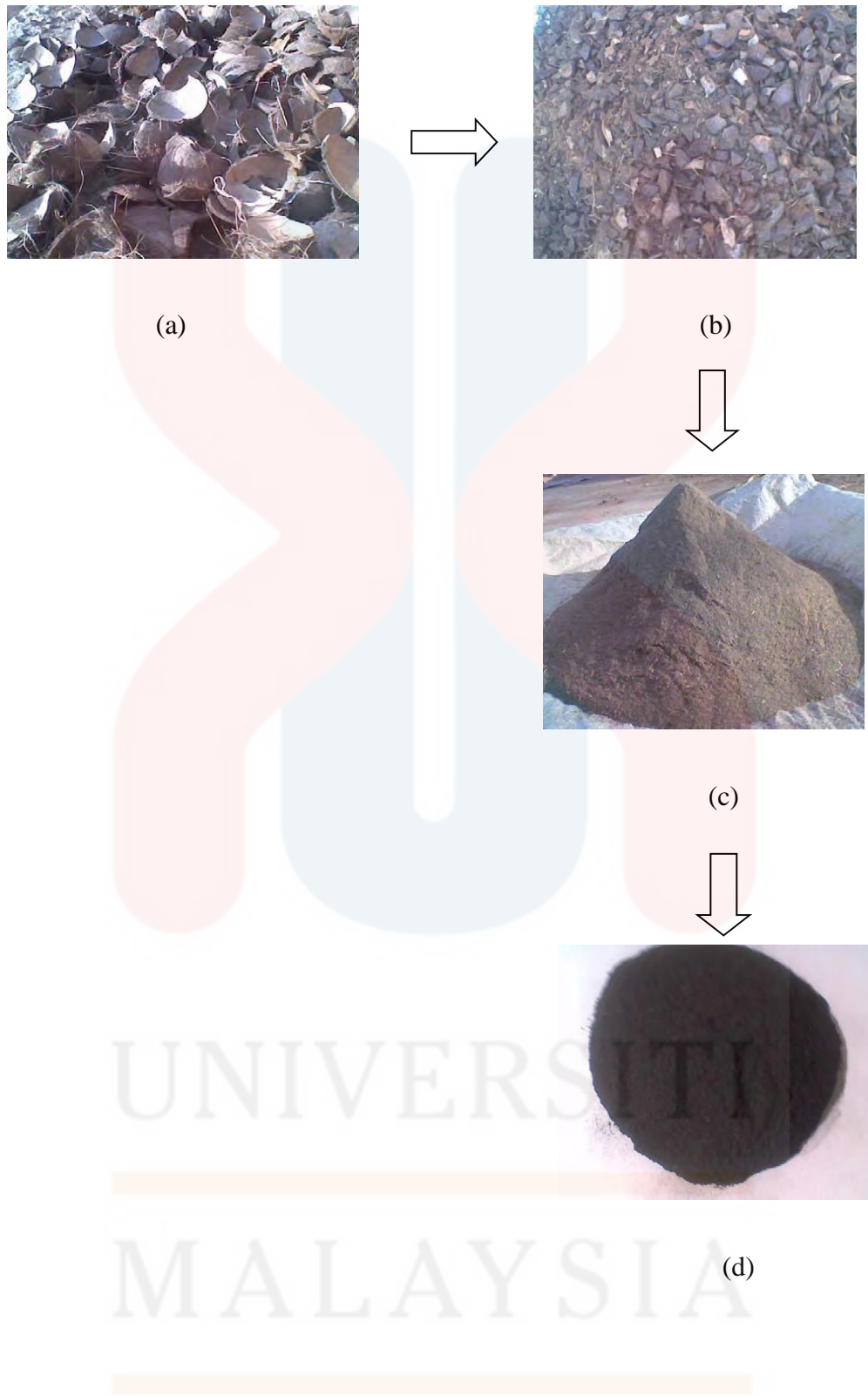


Figure 2.1: Activated carbon carbonization processing (Madakson *et al.*, 2012)

Figure 2.1 shows the activated carbon processing which is from the raw material until the final product. Figure 2.1(a) shows the raw material, coconut shells. The bigger size of the coconut shells is crashed into the smaller size shown in figure 2.1(b) before proceed with the grinded process. This grinded process is to make sure the coconut shells in powder form like in figure 2.1(c) and then placed in furnace to produce carbon. Figure 2.1(d) shows the activated carbon from the coconut shells (Madakson *et al.*, 2012b).

2.2 Sugarcane Bagasse

Sugarcane is one of the major crops grown in over 110 countries and its total production is over 1500 million tons. Hence, all economical sugar from sugarcane are extracted, leaving 8-10% ash as waste, known as sugarcane bagasse ash (Modani *et al.*, 2013). Besides, Khosravi-Darani *et al.*, (2008) stated that thousands of tons of bagasse are discarded daily by the sugarcane processing industries, leading to a big environmental problem. So, the production of activated carbon from sugarcane bagasse is one of the solution for this problem. Kalderis *et al.*, (2008) stated that the processing and transformation of agricultural residues such as sugarcane bagasse into activated carbon with good adsorption properties would alleviate problems of disposal and management of these waste by-products, while providing a high quality end product for water and wastewater treatment that could potentially expand the carbon market.

2.3 Activated Carbon

Activated carbon can be produced from any carbonaceous material. There are a lot of agricultural by products that can be produced as activated carbon like coconut shell, bagasse, rice husk and many more. Besides, activated carbon can be defined as a fine black colorless and tasteless produced from wood and other materials after exposed to the high temperature (Popoola *et al.*, 2012). Activated carbon also means as a porous with large surface area that suitable for absorbent material. In addition, activated carbon also known as the final product of an activation process of carbonaceous materials which different sources and with carbon content in the range of 70-90% (Danish *et al.*, 2013).

From Prauchner & Rodríguez-reinoso., (2012) study, it stated that the activated carbon is the collective name for a group of porous carbons, manufactured by the treatment of a char with oxidizing gases or by carbonization of carbonaceous materials impregnated with dehydrating chemicals and all these carbon exhibit a high degree of porosity and an extended internal surface area.

2.4 Characteristics of Activated Carbon

Activated carbon is a porous carbon based material which is beneficial and widely used in industrial application. The pore size grouping is based on the pore diameter. There are three group of pore which is macro pores (above 50nm), meso pores (2-50nm) and micro pore (under 2 nm). For the activated carbon from coconut

shell is categorized in micro porous and can be treated to get larger porous whether in physical or chemical treatment (Shankar, 2008).

Moreover, activated carbon from coconut shell is well known because of its high hardness, high volatility, low ash content and high specific surface area (Jain *et al.*, 2015). The high surface area is beneficial for activated carbon to act as absorbent material. Based on previous study by Din *et al.*, (2009), the common characteristics of the activated carbon are larger surface area of pore and have widely pore size distribution.

Besides, the percentage weight of activated carbon will decrease due to the releasing of volatile and gases from pyrolysis process. The weight loss is between 200°C and 300°C and burn off to 0% in weight is at 850°C in absence of chemicals (Prauchner *et al.*, 2012). Activated carbon in fact is a microcrystalline, non-graphitic form of carbon with porous structure that has been processed to develop its internal porosity.

Based on previous study by Ansari, (2009), activated carbon has a high degree of porosity, an extensive surface area, and a high degree of surface reactivity. Its large specific surface area is in fact the most important physical property of activated carbon which allows the physical adsorption of gases or vapors and dissolved or dispersed substances from liquids.

It has large number of very fine pores or known as micropore gives the activated carbon a large inner surface, which is the basis for adsorption properties. Based on previous study by Tiryaki *et al.*, (2014), the surface characteristics of the activated carbon products are strongly influenced by the chemical nature of the raw material, the activation method (chemical or physical), the activation agent and the

heat treatment process. The determinations of surface characteristics were generally based on the BET surface area, porosity and functionality of the surface.

2.5 Application of Activated Carbon

Porosity is one of the factors in selection of activated carbon for particular application. Activated carbon is a well known adsorbent material that can be used for removal of pollutants from air, soil and liquids. It means the large surface area of activated carbon allows for contaminants to adhere to the activated carbon media. The absorption application also involves chemical reaction on carbon surface which can remove chlorine from water.

Activated carbon is a broad-spectrum agent that effectively removes toxic and bio-refractive substances such as insecticides, herbicides, chlorinated hydrocarbons, heavy metal ions, and phenols (A. Mohammad-Khah, 2009). Based on previous study by Amuda *et al.*, (2007), activated carbon can be used for removal of heavy metal from industrial wastewater. For example, the activated carbon from coconut is treated with chitosan coated acid to remove Zinc (Zn) from aqueous solution.

Moreover, it provides a less expensive raw material when using coconut shell and aquatic waste like chitin in producing activated carbon. Pore size distribution is a very important property of porous absorbents because the difference in the pore size affects the adsorption capacity for molecules of different sizes and shapes. This is one of the criteria which carbon sorbents are selected for a particular application (Saygılı & Güzel, 2016).

In addition, activated carbon characterization has potential utilization in metal matrix composite for automotive applications because the density of the coconut shell ash is 2.05 g/cm^3 which means that coconut shell ash is very light material (Madakson et al., 2012a). Activated carbon from coconut shell can influence the reactivation of electrochemical performances (Geng *et al.*, 2013). It is shown when activated carbon has the highest mesopore ratio which showed superior capacitive behavior, power output and high-frequency performance in supercapacitors.

Activated carbon can be used in a lot of applications whether in gas or liquid phase (F Rodriguez-Reinoso, 2016). This applications are based on the ability to adsorb gases and vapors on the basis of molecular weight and shape, thus allowing their separation from air or other gas mixtures. The example of application in gas phase are in gas storage and gas purification. Besides, for the liquid phase applications, it requires activated carbons with a larger pore size than gas-phase uses. It is because the rapid diffusion of the liquid to the interior of the carbon particles where the adsorption pores are located is needed and because of the large size of many dissolved molecules to be retained. The examples for liquid phase application are water treatment and food and beverage processing.

2.6 Carbonization Temperature

Carbonization is one of the physical methods in producing activated carbon. Carbonization is a process to convert the coconut shell into charcoal (Shankar, 2008). According to Yusufu *et al.*, (2012) stated that carbonization is producing charcoal and the charcoal with low surface area is not an active product. Besides, the process in which to enrich the carbon content, create initial porosity and enhance the pore

structure with activation process is called carbonization process (Asma *et al.*, 2011). Carbonization involves the removal of non-carbon species and the production of a mass of fixed carbon (char) with a rudimentary porous structure (Alicante 2016).

From the Cobb *et al.*, (2012) observation, the carbonization temperature for high quality of activated carbon is about 800°C to 1000°C. The higher the carbonization temperature, the higher the micro pore volume compared to the micro pore obtained from the low carbonization temperature (Li *et al.*, 2008). The previous study from Gratuito *et al.*, (2008) showed that the optimum condition of production of activated carbon depends on its characteristics.

Mozammel *et al.*, (2002) stated that the complete carbonization process is at 400-600°C. The pore structure and adsorption properties of activated carbons were strongly affected by the nature of the precursors and the heat treatment profile of the process (Jung *et al.*, 2014).

2.7 Activation Temperature and Activation Duration

Activation enhances the adsorptive power of the product obtained from the carbonization stage (Mozammel *et al.*, 2002). Based on Gratuito *et al.*, (2008) study, the activation temperature gave effect to the development of pores, increased the surface area and the subsequent the mass loss. The type of precursor and the chemical agent used are the factors in selection of activation temperature.

Besides, the duration of activation time affected to the development of carbon's porous network. The longer the duration, the larger the pore size of

activated carbon. According to Hu *et al.*, (1999), the increasing of activation temperature would lead to increase of mesopore surface area, the micropore volume and the total pore volume of activated carbon. The optimum temperature condition of carbonization and activation is 600°C and 1000°C respectively (Popoola *et al.*, 2012).

Based on Bahri *et al.*, (2012), the most suitable activation temperature for the preparation of activated carbon is 500°C as for potential application in liquid phase adsorption due to the superior development of mesoporosity and external area. The optimum activation temperature for higher surface area was found to be at 450°C for coconut shells impregnated with phosphoric acid (Gratisito *et al.*, 2008).

In addition, the increasing of activation temperature would affect the increase in the BET surface area, the total pore volume, and the average pore diameter (Sethia & Sayari, 2016). The carbon content generally decreased at higher final temperatures and longer final activation times, with some exceptions possibly caused by the heterogeneity of the samples for analysis (Jung *et al.*, 2014).

2.8 Activating Agents

Activating agent has a significant influence on the porosity development. The use of chemical reagents in the activation process provides an increase in the amount of acid groups present in the activated carbon surface (Cazetta *et al.*, 2011). The activating agent that will be used is Phosphoric Acid (H_3PO_4) and Potassium Hydroxide (KOH). The optimum temperature for Phosphoric Acid is 450°C (Gratisito *et al.*, 2008).

Based on previous study by Yusufu *et al.*, (2012), H_3PO_4 was categorized as energy saving because the optimum activation temperature was around $850^\circ C$ to $900^\circ C$. Moreover, H_3PO_4 acted to promote the pyrolytic decomposition of the initial material and the formation of the crosslinked structure (Liu *et al.*, 2010).

From the previous study by Danish *et al.*, (2013), the phosphoric acid functions as an acid catalyst to promote bond cleavage reaction and to form of crosslink via processes such as cyclization and condensation to combine with organic species to form phosphate and phosphate bridges that connected and crosslinked the biopolymer fragments. The activation of amorphous polymers produced a mostly microporous surface, while phosphoric acid activation of crystalline cellulose produced a mixture of pores in various sizes.

The surface area for acid activated carbon is much higher because of chemical activation normally develops more porosity and increase the surface area. Besides, KOH has higher reactivity and it was selected as the activating agent due to the maximum carbon yield and adsorption uptake capacity (Foo *et al.*, 2012). Besides, KOH also able to promote lower ash content, higher surface area and much better developed porosity activated carbon (Din *et al.*, 2009).

Based on previous study by Jung *et al.*, (2014), the alkaline characteristics may have resulted from the loss of acidic functional groups (such as $-COOH$) during activation and also from the high contents of alkali and alkaline earth metals in the activated carbons obtained under severe conditions.

2.9 Weight Loss of Activated Carbon

Based on Yusufu *et al.*, (2012), as the carbonization time increased, the weight loss of activated carbon also increased. The weight loss for chemical activation was much lower than for the process of carbonization and physical activation. Most of the weight loss took place in the range of 200–300°C for simple carbonization and the presence of H_3PO_4 reduced it to below 150°C (Prauchner & Rodríguez-reinoso, 2012).

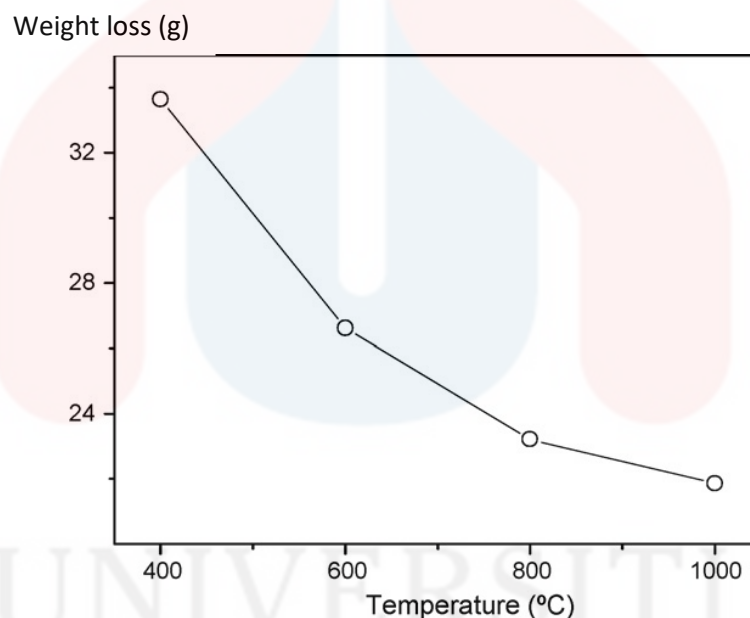


Figure 2.2: Weight loss of activated carbon (Li *et al.*, 2008)

The Figure 2.2 shows the effect of carbonization temperature on the activated carbon. It is shown as the temperature increased, the more volatiles in samples were released. It caused a lower yield of chars. Those findings of weight loss were consistent with the general concept that when the carbonization temperature increased, the amount of the unstable volatiles on the carbon samples would decreased (Li *et al.*, 2008).

2.10 Surface Chemical Characterization of Activated Carbon

The chemical characterizations of activated carbon would be characterized using Fourier Transformation InfaRed (FTIR). From the study by Pillai *et al.*, (2014), the FTIR spectrum revealed that the peaks of the raw coconut shell powder were at 3442.9, 1629.8, 1400.3 and 1066.6 cm^{-1} , which corresponded to the presence of dimeric -OH stretch, C=C stretch, tertiary alcohol bend and aromatic C-H place new bend in functional groups. Besides, the H_3PO_4 characteristics bands at 1050 cm^{-1} (w) assigned to aliphatic phosphate stretch $\nu(\text{—P—O—C—})$, 980 cm^{-1} (s) was assigned to $\nu(\text{—P—O—P—})$ stretching (Danish *et al.*, 2013).

2.11 Phase Identification of Activated Carbon

From the previous study by Madakson *et al.*, (2012), Silicon Oxide (SiO_2) has the highest percentage at element present in activated carbon that revealed by XRD. From this study, a complete Mineralogical analysis carried out by X-ray diffraction also revealed that the ash contain elements such as C, O, Mg, Al, Si, Fe, Na, K, Zn and the coconut shell ash may not contain radioactive materials such as H, He, Li and Be. The example of XRD pattern of activated carbon from coconut shell is shown in Figure 2.3.

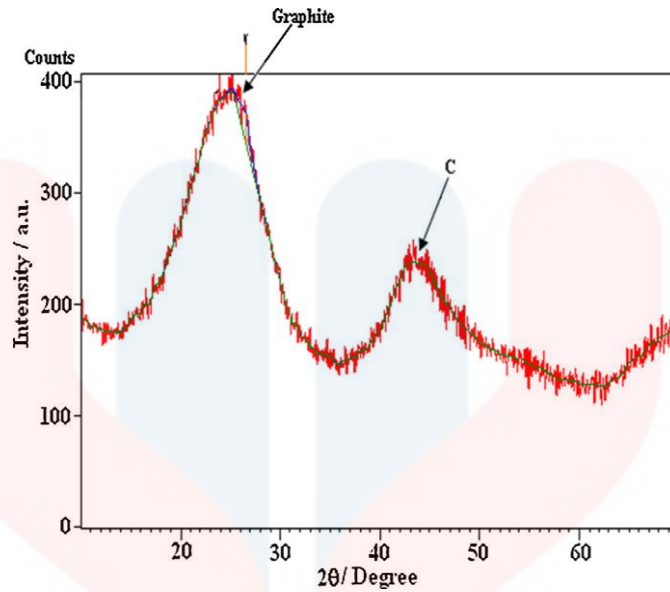


Figure 2.3: XRD pattern of coconut shell activated carbon (Amrita Jain & Tripathi, 2013)

This XRD pattern of the activated carbon was prepared from coconut shell which was activated at 600°C using KOH as an activating agent. The result above showed that the peaks were at around $2\theta = 26^\circ$ and 44° which corresponded to the peak of graphite. It can be seen that XRD signals of powder sample contains large amount of noises in it. This behavior concluded as amorphous structure of carbon (Amrita Jain & Tripathi, 2013).

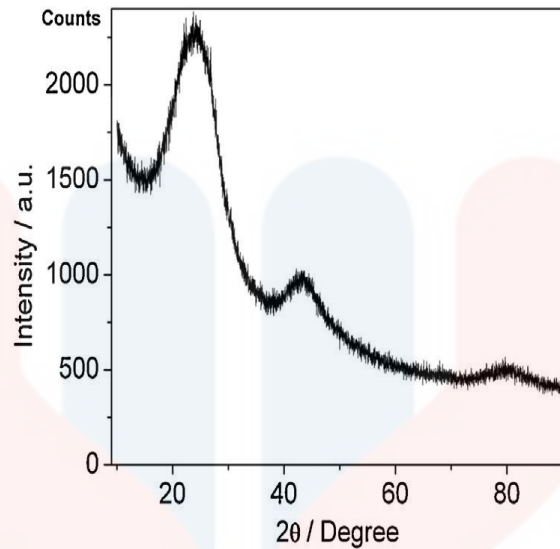


Figure 2.4: XRD pattern of bagasse based activated charcoal (Amrita Jain *et al.*, 2015)

Figure 2.4 shows the typical XRD pattern of sugarcane bagasse that consists of graphitic-like microcrystallites which are randomly oriented and distributed throughout the sample and confirms its turbostratic structure. It can be clearly seen from the figure that there exists two broad diffraction peaks at 25° and 41.6° corresponding to (0 0 2) and (10 0) structural phases respectively which confirms the formation of activated charcoal (Amrita Jain *et al.*, 2015)

Table 2.1: XRF analysis of coconut shell ash (Madakson *et al.*, 2012b)

Element	Al ₂ O ₃	CaO	Fe ₂ O ₃	K ₂ O	MgO	Na ₂ O	SiO ₂	MnO	ZnO
%	15.6	0.57	12.4	0.52	16.2	0.45	45.05	0.22	0.3

XRF analysis confirmed that SiO_2 , Al_2O_3 , MgO and Fe_2O_3 were found to be major constituents of the ash as depicted in Table 2.1. It was agreement for XRD result (Madakson *et al.*, 2012a).

2.12 Morphology of The Activated Carbon

The activation temperature and addition of activating agents have a big influence on pore development of activated carbon. The addition of phosphoric acid (H_3PO_4) caused the activated carbon to swell and opened the surface structure as H_3PO_4 was a strong dehydrating agent (Pragya *et al.*, 2013). Figure 2.4 shows the image of activated carbon from coconut shell that was characterized on Scanning Electron Microscope (SEM).

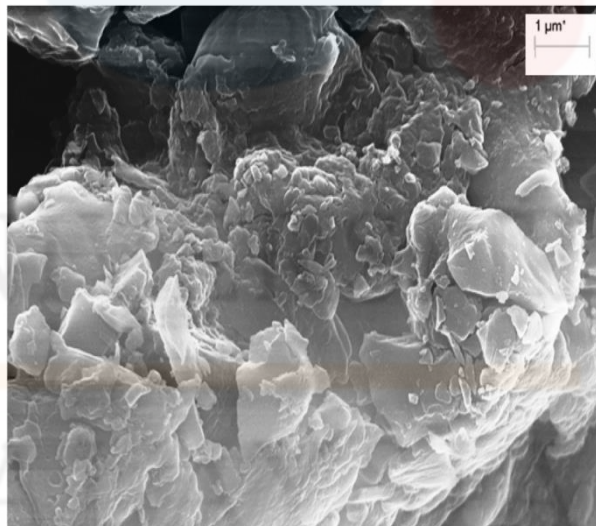


Figure 2.4: SEM image of activated carbon from coconut shell (Pillai *et al.*, 2014)

CHAPTER 3

MATERIALS AND METHODS

3.1 Introduction

This study was conducted in the laboratory of University Malaysia Kelantan, Jeli Campus which is at Agropark laboratory and Material Science Laboratory. Overall process of this research study was shown in research flowchart in figure 3.1.

3.2 Materials

The coconut shells and sugarcane bagasse were collected from Jeli Market. Other materials are Potassium Hydroxide (KOH) and Phosphoric Acid (H_3PO_4) was used as activating agent in determining the characterization of activated carbon. Distilled water also used to remove any impurities.

Table 3.1: Types of activating agent

No.	Activating agent
1	Phosphoric acid (H_3PO_4)
2	Potassium hydroxide (KOH)

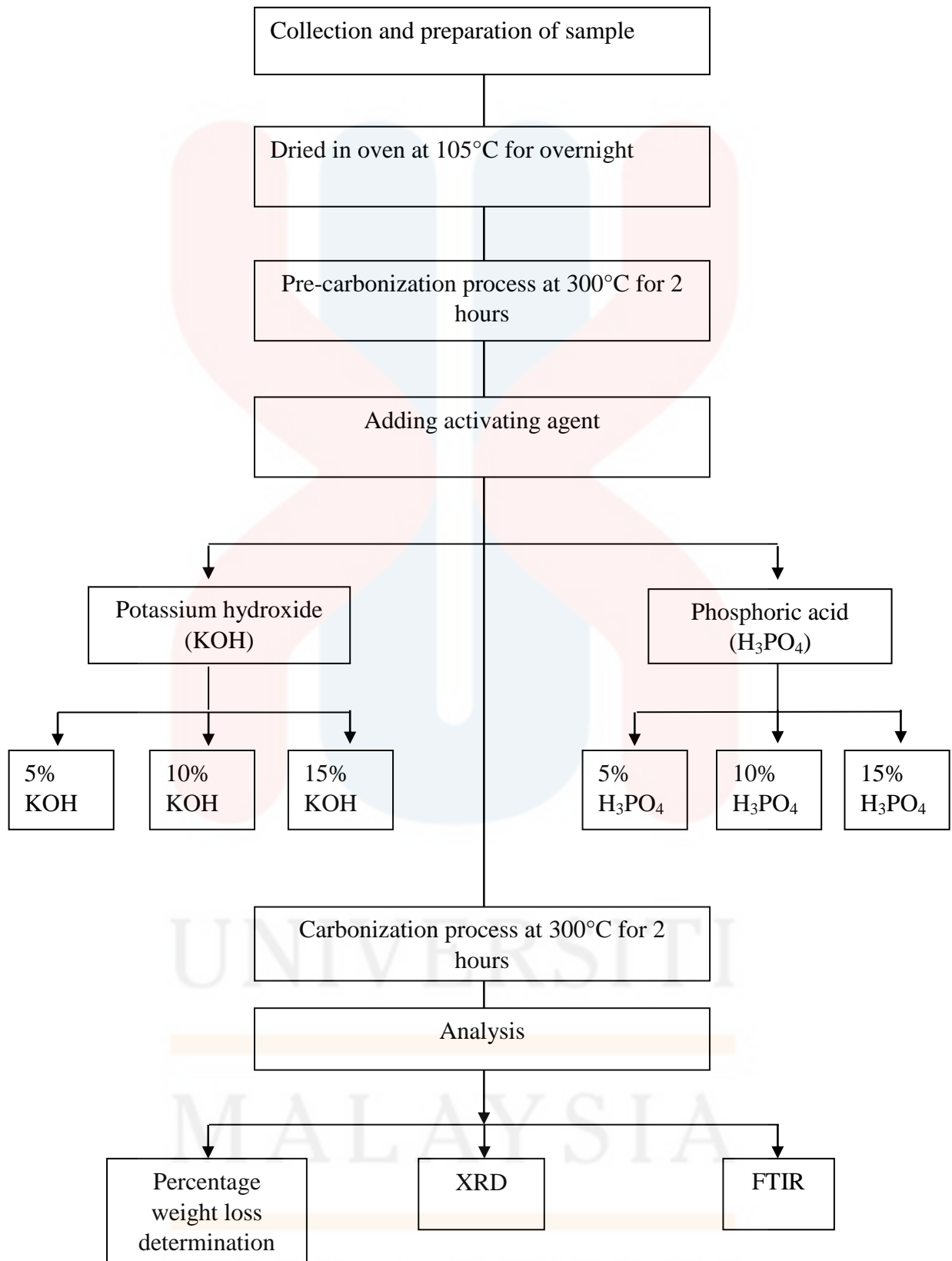


Figure 3.1: Flowchart Research

3.3 METHODOLOGY

3.3.1 Preparation of Activated Carbon by Carbonization and Activation Process

The coconut shells were crashed into smaller pieces using hammer. The smaller pieces of coconut shells then washed with distilled water to remove any impurities. Next, it was grinded using grinder before dried in an oven at 105°C for overnight to remove the moisture. Then, the grinded coconut shells were blended to get the sample in powder form. To make sure the powder in similar size, the powder samples were sieved in 250 μm .

For the sugarcane bagasse sample, firstly, it was washed with distilled water to remove any impurities. Then, the sugarcane bagasse was dried in oven at 105°C for overnight. After that, it was cut into smaller size before blending process. Figure 3.2 below show the example of sugarcane bagasse before the blending process. The blended sugarcane bagasses then were sieved in 250 μm as shown in figure 3.3.



Figure 3.2: Sugarcane bagasse before blending

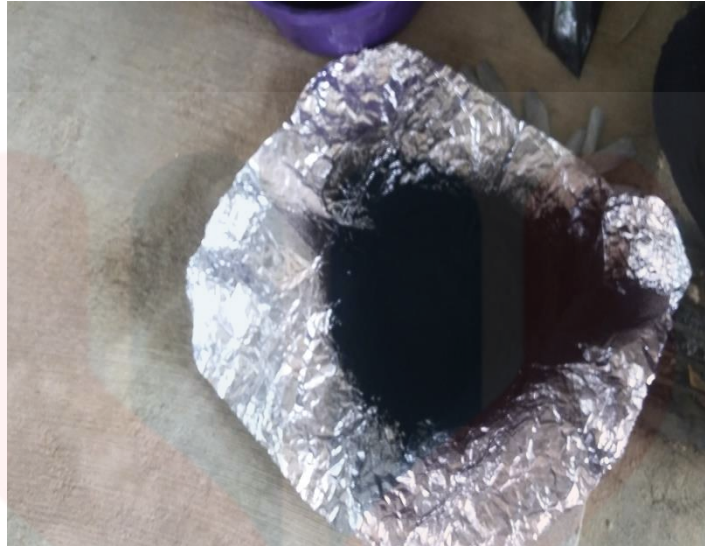


Figure 3.3: The sieved activated carbon

For the next step, the coconut shell and sugarcane bagasse samples were placed in furnace at 300°C for pre carbonization process about 2 hours. The samples were weighed, 15g each sample for coconut shell and 2g for sugarcane bagasse.

The samples then added with activating agents, H_3PO_4 and left for the room temperature for a day. The 5% concentration of H_3PO_4 was dissolved in distilled water to form 50% acid solution. The acid solution then mixed with the activated carbon using impregnation ratio which was 1:3. This chemical activation of coconut shell and sugarcane bagasse was repeated with 10% and 15% concentration of H_3PO_4 and also for KOH. For the carbonization process, the samples were placed in furnace at 300°C for 2 hours. Then, the samples were distilled with deionized water until it became neutral before proceed to analysis process as shown in figure 3.4.



Figure 3.4: Neutralize the sample

Table 3.2: Parameter of coconut shell activated carbon

Activation temperature for 2 hours (°C)	Concentration (%)	Activating agent	
		KOH	H ₃ PO ₄
300	5	Sample A	Sample D
	10	Sample B	Sample E
	15	Sample C	Sample F

Table 3.3: Parameter of sugarcane bagasse activated carbon

Activation temperature for 2 hours (°C)	Concentration (%)	Activating agent	
		KOH	H ₃ PO ₄
300	5	Sample G	Sample J
	10	Sample H	Sample K
	15	Sample I	Sample L

All the samples were activated using activation temperature at 300°C with the different concentration of activating agents. . For coconut shell activated carbon, when the samples were impregnated with 5%, 10% and 15% of KOH, they were labeled as sample A, B and C while for H₃PO₄ the samples were labeled as D, E and F were shown in table 3.2. The sugarcane bagasse samples that were impregnated with 5%, 10% and 15% of KOH namely G, H and I and for H₃PO₄ they were labeled as J, K and L were shown in table 3.3.

3.3.2 Determination of Percentage of Weight Loss

The coconut shell and sugarcane bagasse samples before carbonization and the final product (activated carbon produced after chemical activation) weighed using an electronic weighing balance. The shell samples carbonized in a furnace for durations of 1, 2 and 3 hours to measure the percentage weight decrease or weight loss of the materials during preparation from the original weight of the raw materials. The mathematically expressed as

$$\text{Percentage weight loss (\%)} = \frac{W_i - W_f}{W_i} \times 100 \quad \text{Equation 1}$$

Where W_i is the initial weight of the raw material and W_f is the final weight of the product.

3.3.3 Characterization of samples

3.3.3.1 X-ray Diffraction

XRD is a non-destructive analytical technique which reveals information about the crystallographic structure, chemical composition, and physical properties of materials. XRD also was used for determination of phase identification of activated carbon using Bruker D2 Phaser instrument. DIFFRAC.EVA software will be used for qualitative analysis. It is also used for the elemental analysis of the coconut shell ash.

It will be determined the element or compound of the activated carbon. In addition, the powder of XRD pattern will be analysed to investigate the crystallographic structures changes during activation process.

3.3.3.2 Fourier Transformation Infra-Red (Attenuated Total Reflectance)

FTIR is a nondestructive analysis that can identify chemicals either organic or inorganic. The name of FTIR that is normally used is Thermal Scientific™ iD7 ATR. It also can be used for detecting functional groups, to identify the structure of activated carbon and to observe possible structural changes after carbonization and activation process. The wavelength of light absorbed is characteristic of the chemical bond that is obtained by the spectrum. Every molecule produces a unique pattern, so if an unknown sample produces a spectrum which matches that of a known compound, the sample can be confirmed to be that particular compound.

There are a few step in using this FTIR machine. Firstly, compare and correlate spectrum peak position or x-axis. High correlation value will be achieved if

they have the same component. Then, compare and correlate spectrum both peak position and peak intensity which means more discrimination strength than normal sensitivity. Besides of ensuring sample comprise of same component, high sensitivity features able to distinguish if different formulation are present.



CHAPTER 4

RESULT AND DISCUSSION

4.1 Overview

This chapter discussed the result of the research including weight loss, phase identification and surface chemical characterization using different testing like XRD and FTIR. The results were discussed about the characterization of activated carbon before and after activation process.

4.2: Percentage Weight Loss

Table 4.1: Weight of activated carbon

Type of activated carbon	Weight (before impregnation process)
Coconut shell	15g
Sugarcane baggase	2g

Table 4.2: Weight loss of coconut shell activated carbon for H_3PO_4

Activating agent	Concentration (%)	Weight initial, W_i (before carbonization)	Weight final, W_f (after carbonization)	Percentage weight loss (%)
H_3PO_4	5	18.2	12.7	30.0
	10	28.3	22.1	22.0
	15	33.2	26.0	21.0

Table 4.3: Weight loss of coconut shell activated carbon for KOH

Activating agent	Concentration (%)	Weight initial, W_i (before carbonization)	Weight final, W_f (after carbonization)	Percentage weight loss (%)
KOH	5	18.6	9.5	49.0
	10	19.1	7.6	60.0
	15	20.5	15.1	26.0

Table 4.4: Weight loss of sugarcane bagasse activated carbon for H_3PO_4

Activating agent	Concentration (%)	Weight initial, W_i (before carbonization)	Weight final, W_f (after carbonization)	Percentage weight loss (%)
H_3PO_4	5	3.7	2.1	43.0
	10	4.7	2.9	38.0
	15	5.6	3.8	32.0

Table 4.5: weight loss of sugarcane bagasse activated carbon for KOH

Activating agent	Concentration (%)	Weight initial, W_i (before carbonization)	Weight final, W_f (after carbonization)	Percentage weight loss (%)
KOH	5	4.0	2.5	38.0
	10	3.2	1.7	47.0
	15	3.1	2.1	32.0

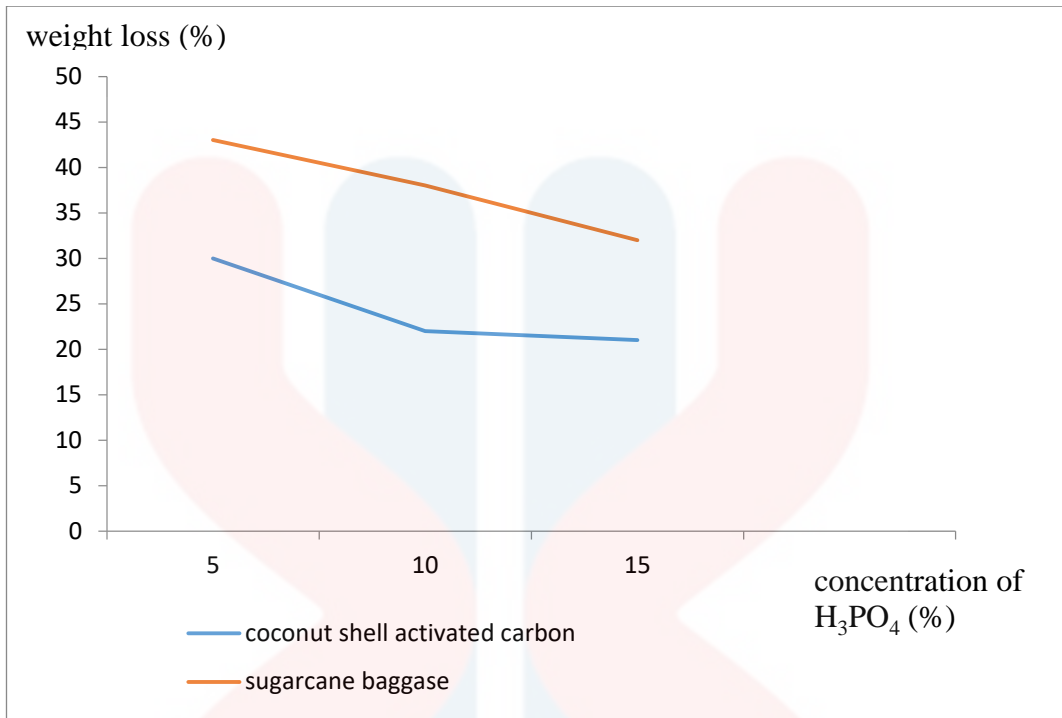


Figure 4.1: Weight loss of activated carbon in different concentration of H_3PO_4

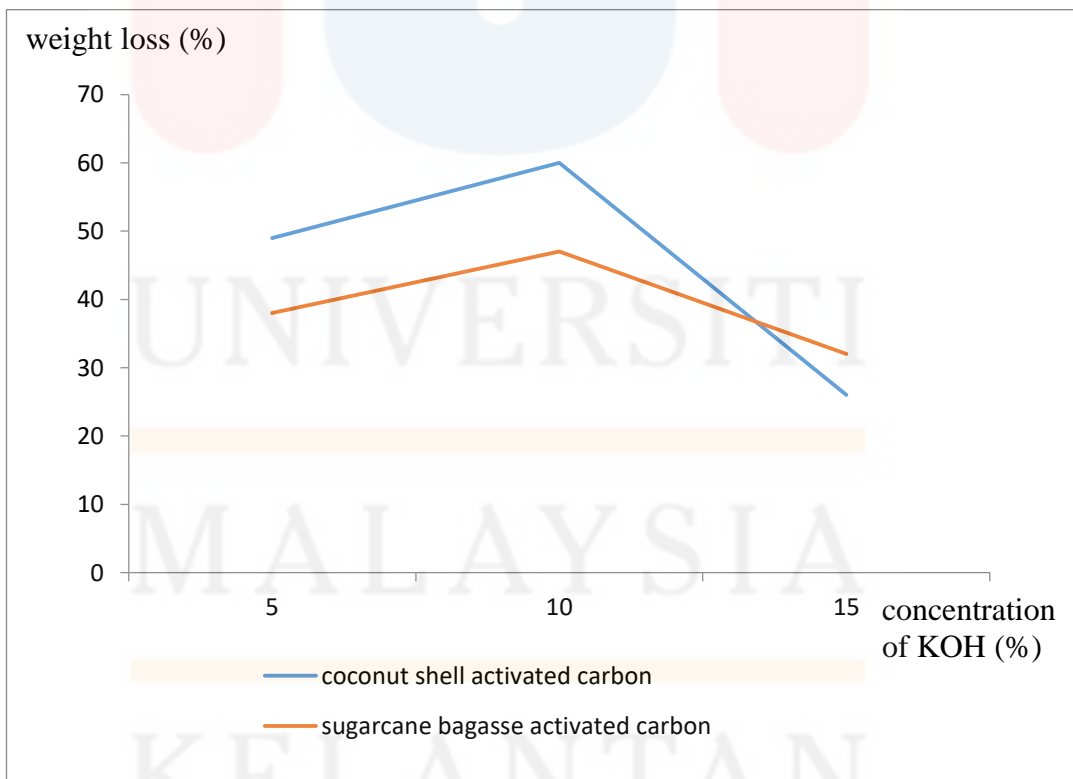


Figure 4.2: Weight loss of activated carbon in different concentration of KOH

Table 4.3, Table 4.4 and table 4.5 showed the weight before and after carbonization process of the activated carbon and the figure 4.1 and figure 4.2 showed the percentage weight loss of coconut shell and sugarcane baggase activated carbon in different concentration. These activated carbon were carbonized in a furnace at 300°C. As we seen in figure 4.1, the weight loss of activated carbon from both coconut shell and sugarcane bagasse in different concentration of H_3PO_4 were decreased. It was about 30%, 22% and 21% the weight loss of coconut shell while for sugarcane bagasse it was about 43%, 38% and 32%. As the concentration of H_3PO_4 increased, the percentage weight loss of activated carbon decreased.

As we seen in table 4.3, the weight loss of coconut shell after impregnation with KOH was about 49%, 60% and 26% while for sugarcane bagasse was about 38%, 47% and 32%. So, the higher the concentration of KOH, the higher the percentage weight loss of the activated carbon.

Besides, we also can see the increasing weight of the coconut shell and sugarcane bagasse activated carbon sample after impregnation with activating agents. This means that the residue of impregnation activating agent showed a great influence on the weight of the samples. Hence, the weight after impregnation process increase as the concentration increase (Yakout & Sharaf El-Deen, 2012a).

4.3 Phase Identification Of Activated Carbon

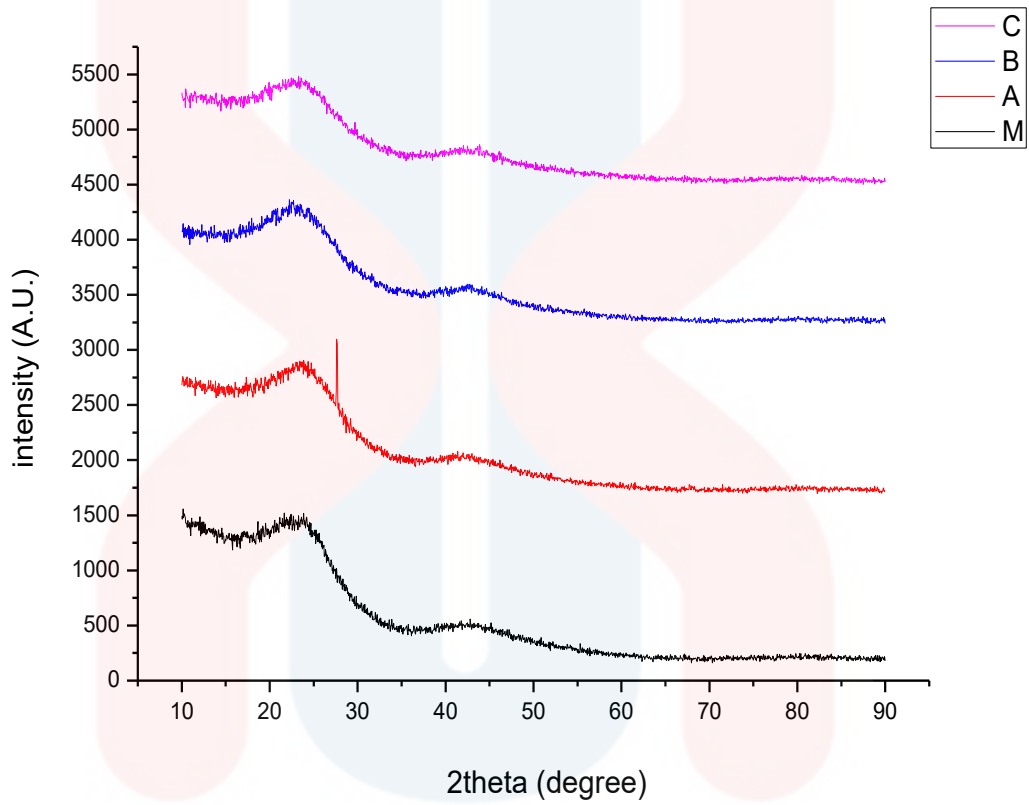


Figure 4.3(a): Untreated coconut shell (M), impregnation of 5% KOH (A), impregnation of 10% KOH (B), impregnation of 15% KOH (C)

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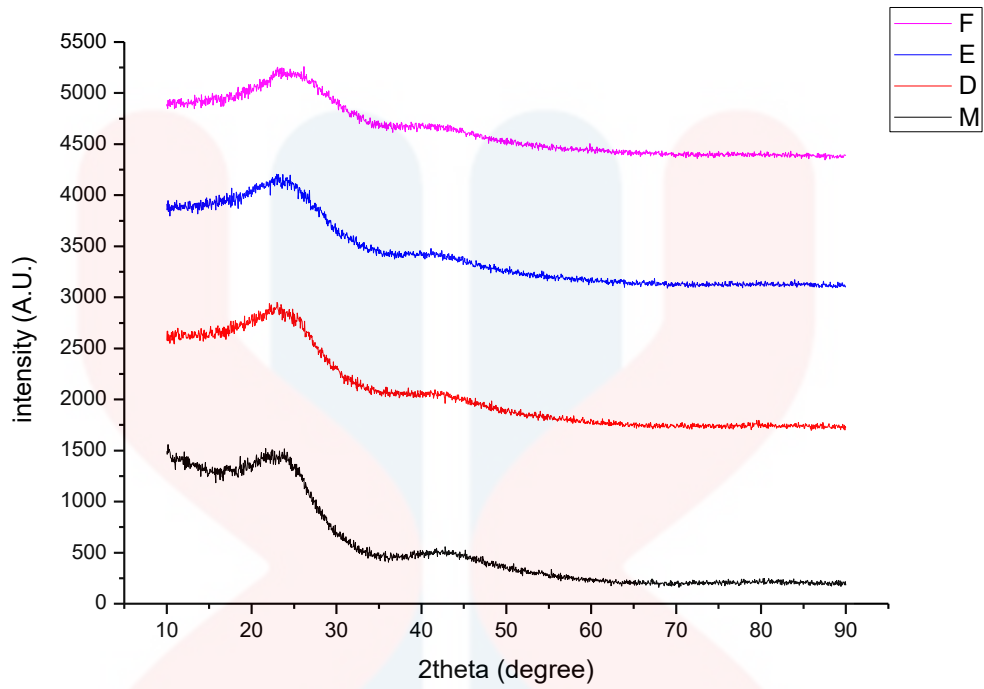


Figure 4.3 (b): Untreated coconut shell (M), impregnation of 5% H_3PO_4 (D), impregnation of 10% H_3PO_4 (E), impregnation of 15% H_3PO_4 (F)

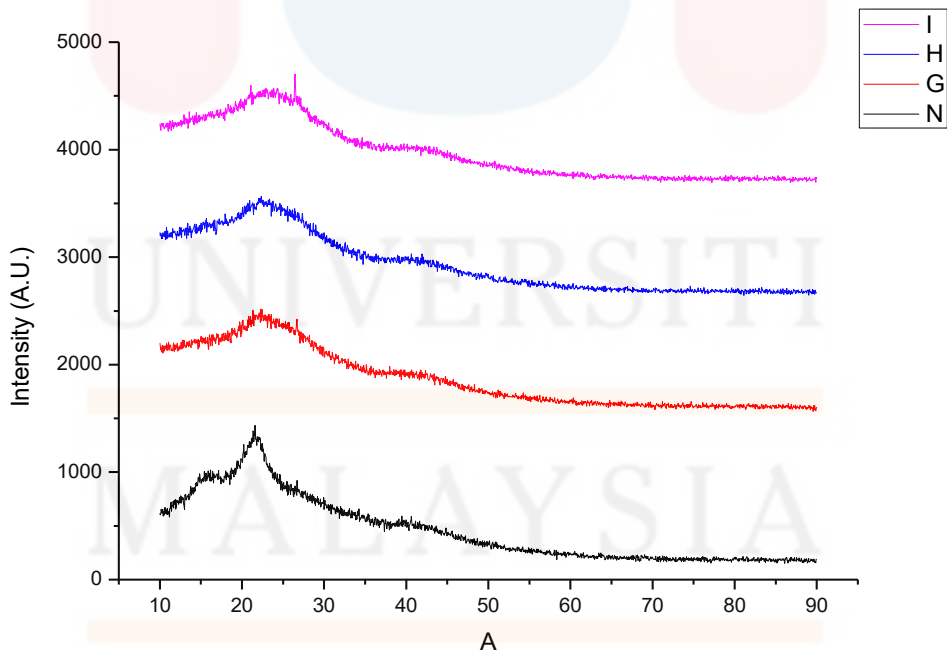


Figure 4.3 (c): Untreated sugarcane bagasse (N), impregnation of 5% KOH (G), impregnation of 10% KOH (H), impregnation of 15% KOH (I)

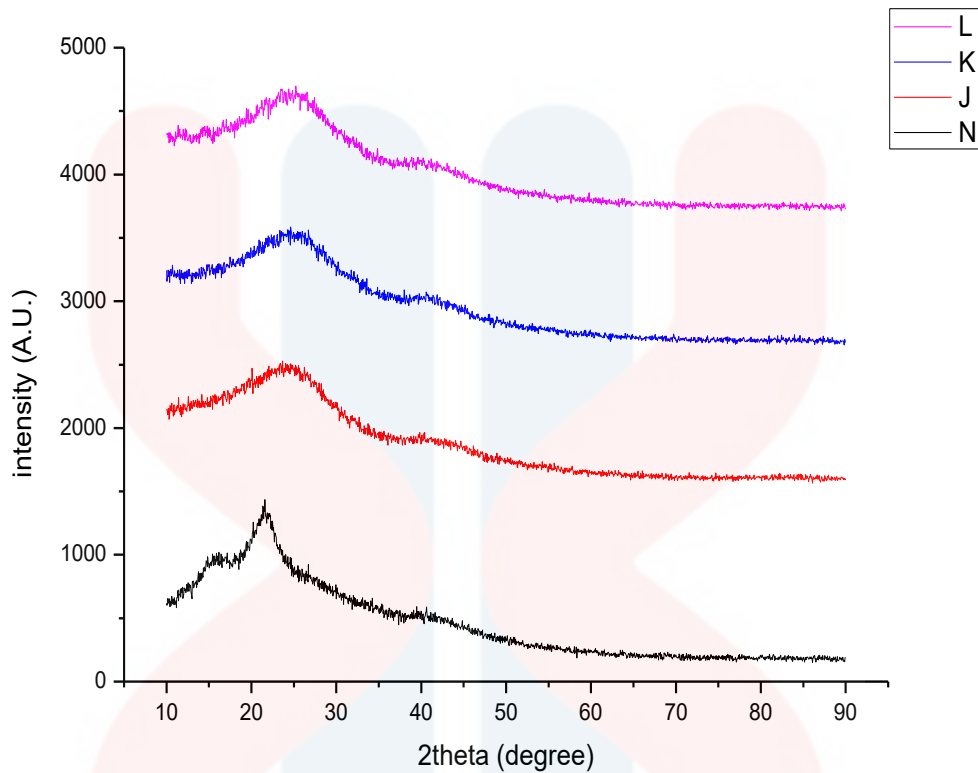


Figure 4.3 (d): Untreated sugarcane bagasse (N), impregnation of 5% H_3PO_4 (J), impregnation of 10% H_3PO_4 (K), impregnation of 15% H_3PO_4 (L)

The powder X-ray diffraction analysis of representative samples of coconut shell and sugarcane bagasse activated carbon were investigated in figure 4.3(a), figure 4.3(b), figure 4.3(c) and figure 4.3(d). As we seen, when the concentration of activating agents increase, there was more high peaks existed. From this analysis, it can be observed from the figure that irrespective of the activation agent utilized the overall pattern of the spectra are very similar. Besides, the coconut shell carbon content after impregnation of 5% KOH (A) was 68.2%, 10% KOH (B) was 68.8% and 15% KOH was 85.7%. For the sugarcane bagasse activated carbon, the carbon content after impregnation with 5% KOH (G) was 92.6%, 10% KOH (H) was 79.3% and for 15% KOH (I) was 73.5%. So, it can be concluded that the carbon content

of coconut shell and sugarcane bagasse activated carbon were definitely not same even they had the same impregnation ratio. As the concentration of potassium hydroxide increased, the carbon content of coconut shell activated carbon also increased but it was differ with the sugarcane bagasse activated carbon. The carbon content of the sugarcane bagasse decrease as the concentration of potassium hydroxide increased. Activated carbon in increasing of acid concentration resulted richer carbon and it is more porous (Yakout & Sharaf El-Deen, 2012). It can be proven when coconut shell activated carbon was in 5%, 10% and 15% of phosphoric acid impregnation, they were resulted as 85.7%, 86.8% and 89.5% of carbon content while for sugarcane bagasse, they resulted as 60.9%, 79.5% and 82.0% of carbon content.

From this analysis, the cristallinity or structure of the activated carbon also can be determined. After impregnation with Potassium Hydroxide, the sugarcane bagasse activated carbon was concluded as more amorphous than coconut shell activated carbon. It was because the sugarcane bagasse activated carbon had percentage amorphous structure in the range of 76% to 90% while for coconut shell, it contain only 67% to 70% of amorphous structure percentage. For the impregnation with the phosphoric acid, the percentage of amorphous structure of sugarcane bagasse was in the range of 78% to 82% while for coconut shell activated carbon was 70% to 74%.

4.4 Surface Chemical Characterization Of Activated carbon

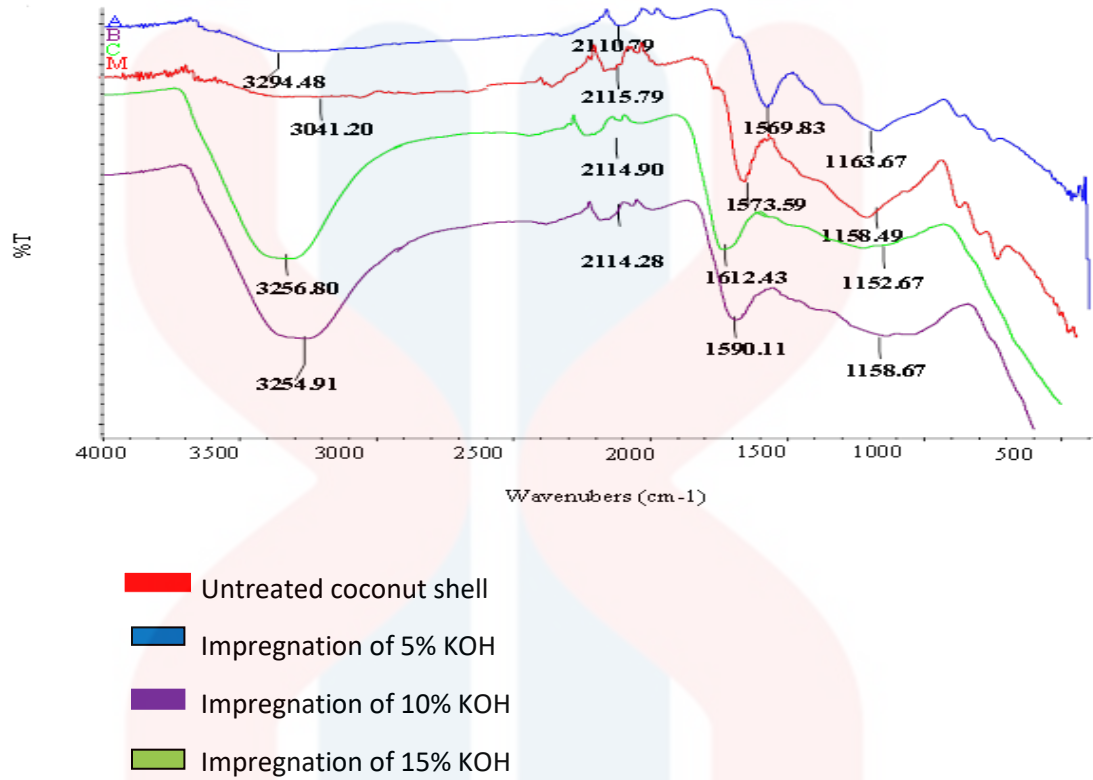
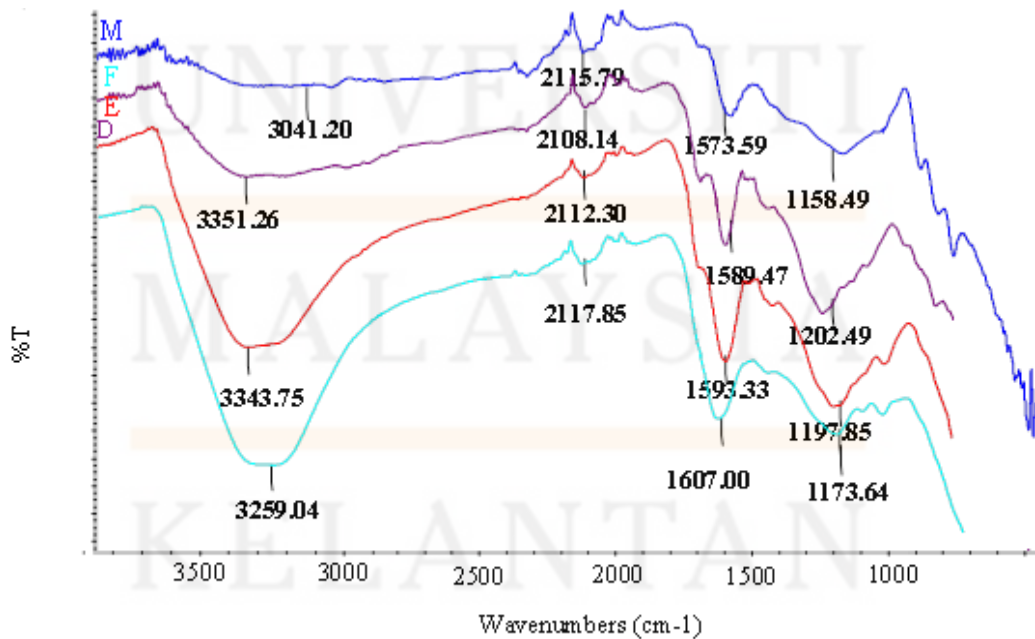
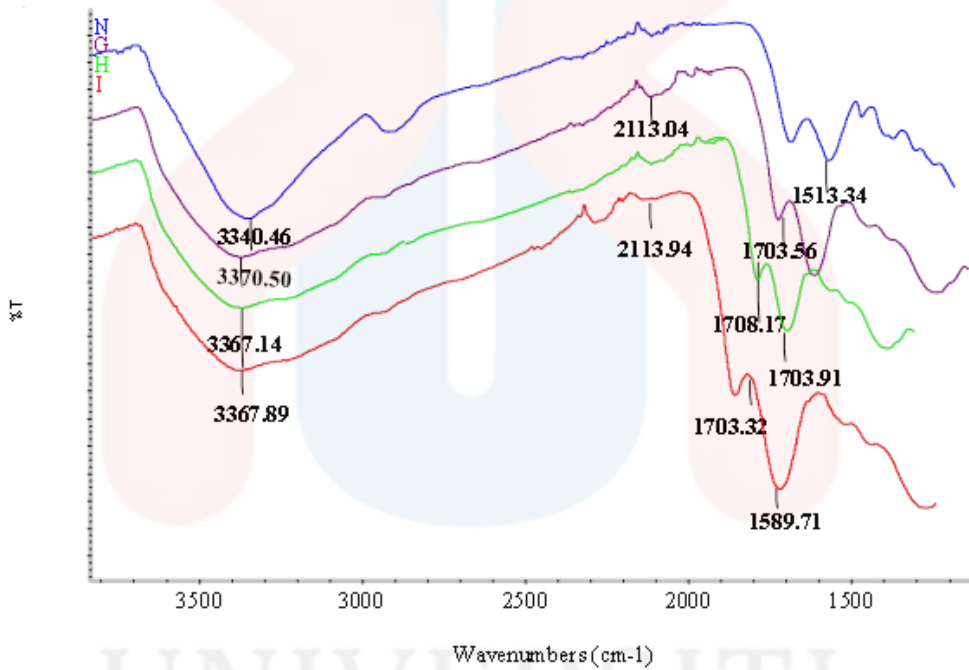


Figure 4.4 (a): coconut shell impregnation with Potassium Hydroxide (KOH)



- Untreated coconut shell
- Impregnation of 5% H₃PO₄
- Impregnation of 10% H₃PO₄
- Impregnation of 15% H₃PO₄

Figure 4.4 (b): coconut shell impregnation with Phosphoric Acid (H₃PO₄)



- Untreated sugarcane bagasse
- Impregnation of 5% KOH
- Impregnation of 10% KOH
- Impregnation of 15% KOH

Figure 4.4 (c): sugarcane bagasse impregnation with Potassium Hydroxide (KOH)

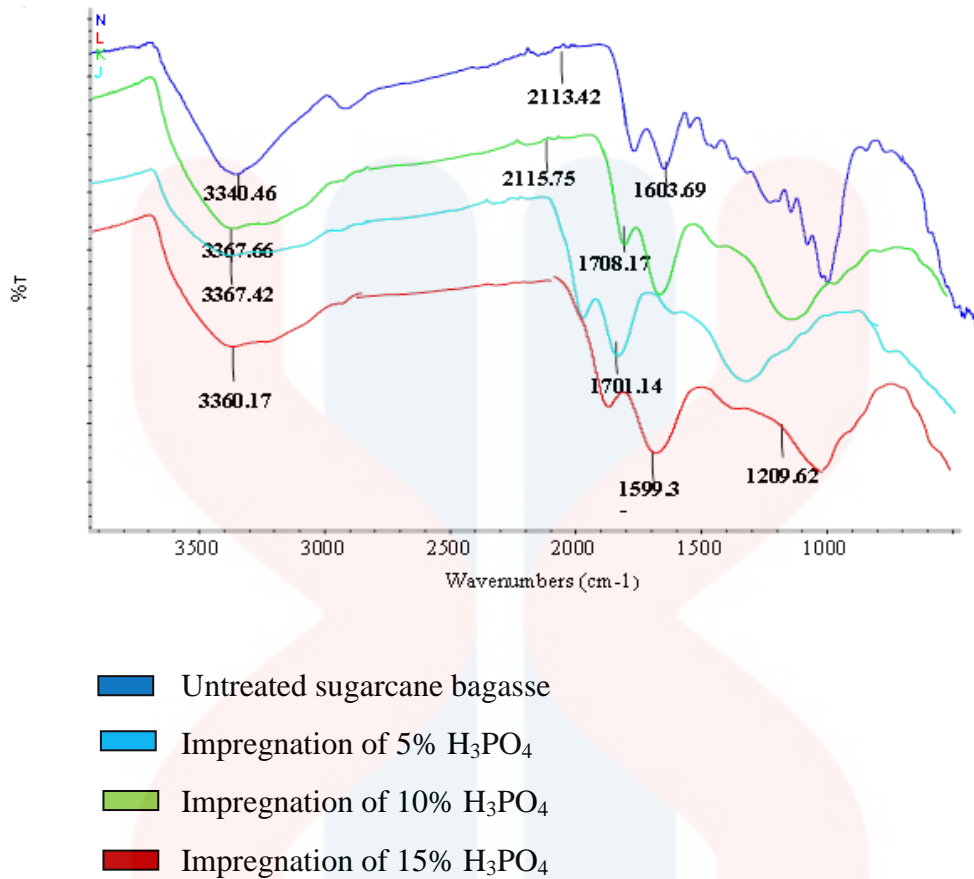


Figure 4.4 (d): sugarcane bagasse impregnation with Phosphoric Acid (H₃PO₄)

Surface chemical can be characterized using Fourier Transformation Infrared Spectroscopy (FTIR). It provided the information on the chemical structure of the activated carbon. From the result stated, the peaks were in the range of 3000-3500cm⁻¹, 1500-2000cm⁻¹ and 1000-1300cm⁻¹. For the acidic activated carbon for both coconut shell and sugarcane bagasse activated carbon, the broad bands at 1300-1000cm⁻¹ had been assigned to C-O stretching in acid, alcohols, phenols ethers and ester (Puziy *et al.*, 2002). Based on the study by Yakout & Sharaf El-Deen, (2012), it stated that the band at 3200-3600cm⁻¹ can be assigned to the O-H stretching mode of hydroxyl groups and absorbed water.

In other hand, for sugarcane bagasse activated carbon, it was resulted as aromatic phenone compound when impregnated with 5%, 10% and 15% of KOH. It was due to C=O stretching vibration that the most intense in the spectrum. The impregnation of sugarcane bagasse activated carbon with 5% H₃PO₄ was analysed as aliphatic formate ester compound because of C-O stretching . For 10% H₃PO₄ impregnation, it was showed that the activated carbon was aromatic ethers compound. Aromatic Fluorine compound was found when 15% H₃PO₄ impregnation. The sugarcane bagasse activated carbon first peak for KOH and H₃PO₄ was in the range of 3364-3398cm⁻¹ due to O-H stretching in hydroxyl functional group while the band around 1700cm⁻¹ was ascribed to C=O stretching for ketones, aldehydes or carboxyl group and this peak indicates that the porous carbons contain a small amount of carboxyl group (Adinaveen *et al.*, 2013).

Besides, as we seen coconut shell activated carbon from the figure above, when 5% and 10% concentration of Potassium Hydroxide (KOH) was impregnated, it was resulted as aromatic fluorine compound but for 15% KOH it showed the activated carbon as aromatic isothiocyanates compound. The coconut shell activated carbon impregnated with 5% Phosphoric Acid, (H₃PO₄) showed that it was aromatic fluorine compound , 10% H₃PO₄ resulted as aliphatic isothiocyanates compound and 15% H₃PO₄ showed as primary aliphatic alcohols. Primary aliphatic alcohol had one other carbon atom that attached to the oxygen bound carbon.

Based on Yakout & Sharaf El-Deen, (2012) studied, the most important changes introduced in this characterization were the development of C-H vibration and possibly because of the loss of oxygen at the surface of the carbon material.

CHAPTER 5

CONCLUSION AND RECOMMENDATIONS

5.1 Conclusion

In this study, activated carbon was produced by coconut shell and sugarcane bagasse. Nowadays, numerous economic and industrial sectors widely use activated carbon in many applications. There are two ways to modify activated carbon, which are physical and chemical methods. Chemical activation is one of the suitable methods for getting new activated carbon materials (Danish *et al.*, 2013). The activating agents that were used in this study were Phosphoric Acid (H_3PO_4) and Potassium Hydroxide (KOH) in different concentrations. Different activating agents gave different effects on activated carbon. The production of activated carbon can be used in a lot of applications such as absorbent, dyes, and many more. The activated carbon from coconut shell and sugarcane bagasse were successfully synthesized by carbonization and activation processes. The synthesized activated carbon also were characterized and compared successfully. From this study, it can be concluded that Phosphoric acid (H_3PO_4) was the best activating agent that can increase the size of the pores of activated carbon.

5.2 Recommendation

In the future, if this study is to be continued, there are several recommendations that can be considered:-

- a) The activated carbon can be further tested using Brunauer Emmett Teller (BET) to analyse the pore surface area and volume. In this study, only phase identification and surface chemical characterization of activated carbon were determined.
- b) Further studies can also tested using Scanning Electron Microscope (SEM) to observe the surface image of the activated carbon. The image of the pore structure before and after activation process can be observed.
- c) Further studies also can include the physical properties testing like different carbonization and activation temperature that can effect the characterization of activated carbon.
- d) Besides, the production of activated carbon from other material like egg shell and other waste materials can be further studied.

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