PREPARATION AND CHARACTERIZATION OF ACTIVATED CARBON PREPARED FROM OIL PALM SHELL BY PHYSICAL AND THERMAL ACTIVATION PROCESS

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UNIVERSITI TEKNIKAL MALAYSIA MELAKA

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Submitted in accordance with requirement of the Universiti Teknikal Malaysia Melaka (UTeM) for Bachelor Degree of Manufacturing Engineering (Hons.)

by

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FACULTY OF MANUFACTURING ENGINEERING

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DECLARATION

I hereby, declared this report entitled "Preparation and Characterization of Activated Carbon Prepared from Oil Palm Shell By Physical And Thermal Activation Process" is the result of my own research except as cited in references.

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APPROVAL

This report is submitted to the Faculty of Manufacturing Engineering of Universiti Teknikal Malaysia Melaka as a partial fulfilment of the requirement for Degree of Manufacturing Engineering (Hons). The member of the supervisory committee is as follow:

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ABSTRAK

Satu dekad yang lalu, bahan pencemar air menjadi perhatian besar kerana terdapat banyak kesan terhadap kesihatan manusia yang boleh menyumbang kepada sebarang masalah kesihatan. Akibatnya, bahan cemar dapat diatur dengan adanya karbon aktif. Dalam cecair dan gas, karbon aktif membolehkan racun, racun serangga, bau busuk, rasa, logam berat dan unsur-unsur yang tidak diingini terperangkap. Di samping itu, beberapa eksperimen telah dilakukan pada kulit kelapa sawit dalam berbagai aplikasi karbon aktif untuk mengawal pelepasan. Untuk mencapai penyerapan pelepasan udara dan air tertinggi, masa pemprosesan yang lebih pendek, biaya rendah dan pelbagai aplikasi yang dapat digunakan, kaedah pengeluaran karbon aktif digunakan melalui proses kimia, termal atau fizikal tetapi pada suhu yang dapat diterima oleh yang dihasilkan bahan kimia. Fokus penyelidikan ini adalah untuk mengkaji karbon aktif dengan pengaktifan terma dan fizikal melalui penggunaan tempurung kelapa sawit untuk menghasilkan karbon aktif penyerapan tinggi yang disediakan oleh kawasan permukaan spesifik yang lebih tinggi dan untuk mencirikan pencirian kulit inti kelapa sawit. Penyelidikan sebelumnya, karbon aktif berdasarkan kayu biasa dihasilkan dari jenis kayu dan habuk papan terpilih. Beberapa masalah yang dihadapi oleh proses penguraian kayu yang berlaku kerana pemanasan dalam proses penyulingan . Dengan menggunakan tempurung kelapa sawit sebagai bahan mentah baru untuk menghasilkan karbon aktif yang lebih berkesan, kelemahan bahan kayu dapat diperkuat dan mempunyai sifat yang lebih baik daripada diaktifkan secara komersial. Walaupun begitu, oleh kerana banyak penyelidik menumpukan perhatian pada pengaktifan kimia, sehingga kini terdapat kajian yang terhad mengenai kaedah pengaktifan termal dengan menggunakan tempurung kelapa sawit. Permukaan morfologi dan pengaruh pH karbon aktif telah ditetapkan sebagai parameter untuk pengumpulan dan analisis karbon aktif. Permukaan morfologi sebelum dan selepas proses pengaktifan, struktur dan kesan pelebaran liang disebabkan suhu dijangka wujud .karbon aktif pada suhu 600 ℃ dipilih sebagai sample yang terbaik dengan hasil darjah bakar yang tinggi iaitu 74.71 wt%, liang pori lebih luas seperti yang ditunjukkan oleh struktur mikrograh dan penyaringan air yang terbaik pada pH 7. Oleh itu, berbanding dengan bahan bukan organik lain, karbon aktif dari tempurung kelapa sawit dapat menjadi pasaran global yang baik untuk kecekapan penggunaan air dan bahan berpotensi tinggi.

ABSTRACT

Since decade ago, water pollutants became a big concern as there are many impacts on human health that may contribute to any health issue. Activated carbon can be used to regulate water contaminants. In both liquid and gas, activated carbon allows poison, insecticides, bad odor, taste, heavy metal, and undesirable elements to be trapped. In addition, several experiments have been carried out on oil palm shells in various applications of activated carbon to control emissions. In order to achieve the highest absorption of air and water emissions, shorter processing time, low cost and a wide variety of usable applications, the method of activated carbon production is used through chemical, thermal or physical processes but at an acceptable temperature to the produced chemical. The focus of this research is to study activated carbon prepared using oil palm shells by thermal and physical activation through the use of to generate high absorption activated carbon provided by a higher specific surface area and to characterize the oil palm shell derived activated carbon. In previous researches, activated carbon were prepared using common wood. Some problems faced by the wood decomposition process that occurs due to warming in the dry distillation process. By using the oil palm shell as the new raw material to generate more effective activated carbon, the weakness face by wood material can be strengthened and has better properties than commercially available activated carbon. Nevertheless, as many researchers concentrating on chemical activation, there has been limited research to date on the method of thermal activation by using oil palm shell. The surface morphology and effect of activated carbon pH has been set as a parameter for the collection and analysis of activated carbon. The surface morphology before and after the activation process of carbon structure and the pore widening impact due to the temperature expected to be present. Activated carbon prepared of 600 °C of activation process was selected as the best sample sample as it is yield high burn-off degree of 74.71 wt%, wider pore as shown by micrograph structure and performed the best water filtration of pH 7. Thus, compared to other inorganic materials, activated carbon from oil palm shell could be the favourable global market for its water application efficiency and low potential cost material.

DEDICATION

Only

my beloved father, Yusof bin Abd Kadir

my appreciated mother, Siti Noraizah binti Mohd Sharif

my adored sister and brother, Nur Afiqah and Muhammad Wafiq

for giving me moral support, money, cooperation, encouragement and also understandings

Thank You So Much & Love You All Forever

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LIST OF ABBREVIATIONS

AC	-	Activated Carbon
FTIR	-	Fourier Transform Infrared Spectroscopy (FTIR)
FESEM	-	Field Emission Scanning Electron Microscopy
PAC	-	Powdered Activated Carbon
GAC	-	Granular Activated Carbon
EAC	-	Extruder Activated Carbon
FELDA	-	Federal Land Development Authority
FELCRA	-	Federal Land Cosolidation and Rehabilitation Authority
RISDA	-	Industry smallholders Development Authority
ASAP	-	Accelerated Surface Area and Porosimetry

LIST OF SYMBOLS

%	-	Percent
wt. %	-	Weight percent
mm	-	Millimetre
°C	-	Degree Celsius

CHAPTER 1

INTRODUCTION

The background of study, problem statement, goals, research scope and significance of this research are mentioned in this chapter.

1.1Background of Study

The oil palm tree or the other name (*Elaeis guineensis*) is a native African tropical palm plant. The cultivation of oil palm trees has grown in less than 100 years from being a latively small-scale crop in Africa to one of the most lucrative agricultural commodities in the world. In Malaysia's climate, oil palm trees will grow well (Awalludin et al., 2015). Malaysia is one of the countries that is effectively harnessing the oil palm industry's beneficiaries and is responsible for the global uprising of this industry through contributing significantly and continuous commitment. As one of the world's most successful palm oil producers, Malaysia has kept the tittle for decades (Sayer et al., 2012).

Using agricultural waste as a supply for activated carbon synthesis might lower production costs. Agricultural waste has a high carbon proportion. Agricultural waste, as a renewable resource is also suitable in large quantities. The use of simple methods for the processing of activated carbon would also substantially reduce the cost of production. An easy, low-cost system for producing carbon from agricultural waste would greatly improve living standards in rural and remote areas (Aravind & Amalanathan, 2020). Moreover, many raw material from waste agricultural such as corn straw, wheat straw, rice straw, sawdust, corncob, bagasse, cotton stalk, coconut husk, rice husk, tobacco stem, nut shells, soybean oil cake and oil palm fiber have been prepared from farming for a large variety of activated carbon (Adegoke & Bello, 2015).

An attractive technique for extracting toxins from water is activated carbon adsorption. Research is being conducted worldwide for the production of low-cost activated carbon (Adegoke & Bello, 2015). Other than that, water treatment is a method of reducing to appropriate levels the number of pollutants present in water that are not harmful to living organisms and their environment. Water treatment utilities use traditionally to reduce the number of pollutants present in water that is safe for potable use and disposal to the environment (Rahman et al., 2019). The activated carbon method is commonly used and is well known to be effective in the treatment of water and waste water (Thamilselvi & Radha, 2017).

The objective of this current work is to prepare and characterize activated carbon that derived from oil palm shell using physical activation and thermal activation method followed by temperature as parameter. This is due to the lack of previous studies that using thermal activation process and the parameter of pH effect that shows the efficiency activated carbon to apply in water treatment. Using selected methods such as Fourier Transform Infrared spectroscopy (FTIR), the activated carbon produced will be further examined and Scanning Electron Microscopy (SEM), a well define activated carbon structure can be evaluated due to the preparation the activated carbon.

1.2 Problem Statement

The world production of activated carbon demand is estimated at 375,000 tons in 1990, except for Eastern Europe and China (Mozammel et. al, 2002). Order activated reached up to 200,000 tons per year in the United States in activated carbon 2002, and demand is increasing. The growth of the market for these materials to a various application is estimated at 4.6% per year (Mozammel et. al, 2002).

The activated carbon produced from waste material such as rubber, wood, sawdust and coconut oil. Activated carbon mainly used as adsorbents to remove organic compound and contaminants from the flow of liquid and gas. Common wood based activated carbon is produced from selected types of wood and sawdust. However, there are materials such as woods have listed some of the problems faced by wood decomposition process that occurs due to warming in the dry distillation process. The weakness that faced on wood material can be improved by using the oil palm shell as the new raw material to produce more efficient activated carbon and has better properties than commercial activated.

The series of pre-treatment washing procedures should be carry out in the physical and thermal activation phase. Scanning Electron Microscope used to the characterization of surface morphology. The study concentrated on the growth of pore size and their activated carbon behavior.

The most common type of water filter, particularly for household use, is activated carbon based on charcoal, which is significantly determined by factors such as molecular weight, pH, particle size, surface area, and flow rate. Sand filtration and ceramic filtration are also types of filtrations. The purpose of this research is to determine the potential of oil palm shells to replace the current filter absorbent as a cost-effective activated carbon. The figure 1.1 shows the summarize of problem statement and research gap.

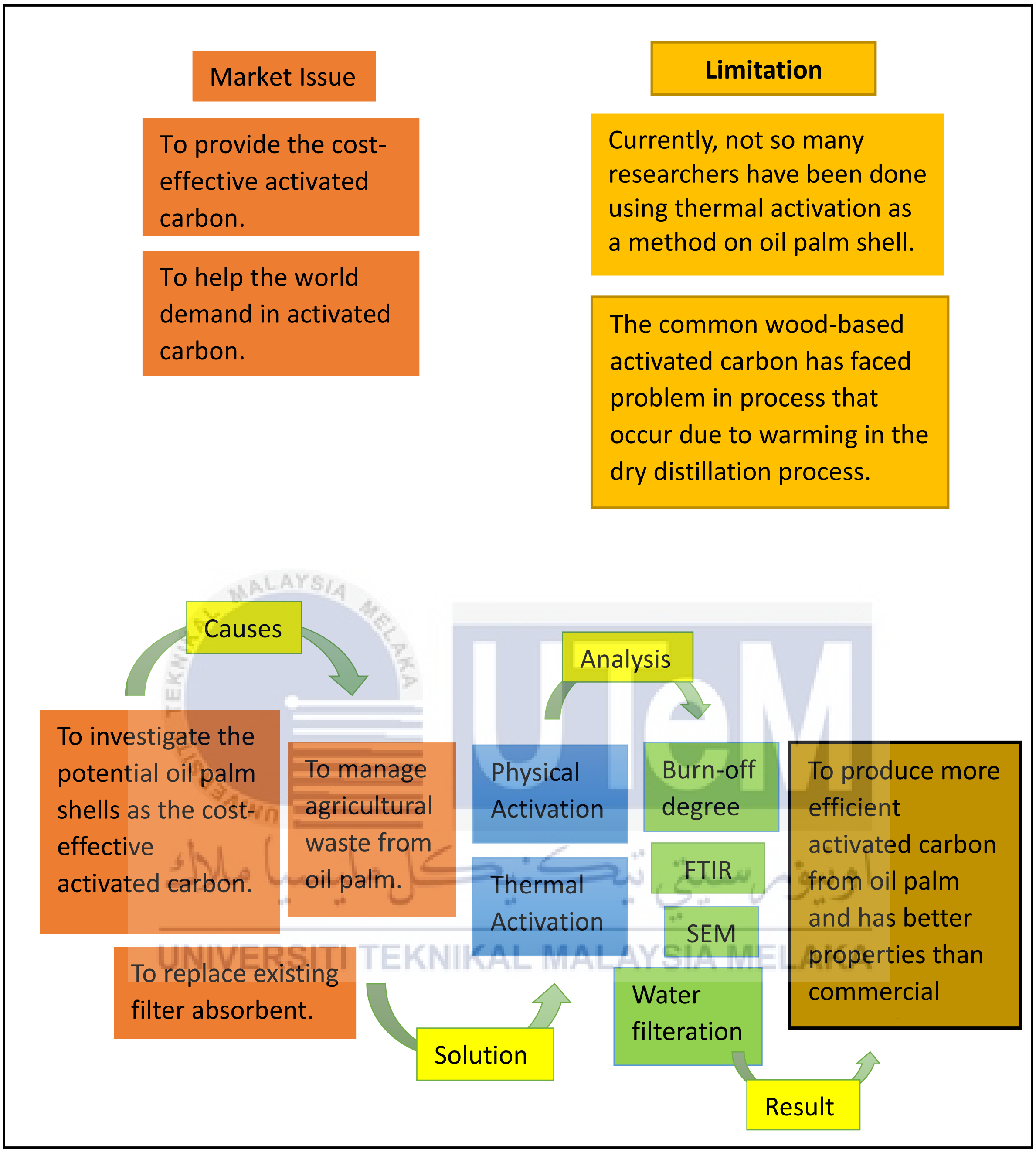

Figure 1.1: The summarize of problem statement and research gap

1.3 Objective

The objective are as follows:

1. To produce activated carbons by physical and thermal activation process by utilizing oil palm shell.

2. To characterize the surface morphology of the oil palm shell based activated carbon.

3. To evaluate of activated carbon for water filtration application by water filtration analysis.

1.4 Research Scopes

The research concentrates on the preparation and characterization activated carbon based on oil palm shell via physical activation and thermal activation method. In order to achieve the purpose of the study, the experiments are carried out in different conditions based upon various scopes in order to produce productive activated carbon in order to provide cost-effective active carbon.

In this research, the first objective is to produce activated carbons by physical activation and thermal activation process by utilizing oil palm shell. The method used in this study was to activate the oil palm shell, resulting in activated carbon. The parameter that will be vary in these two methods are the temperature reading and the preparation of the activated carbon. Physical activation boiled the oil palm shell in 150℃ distilled water. The thermal activation burns the oil palm shell at 400 ℃,600 ℃ and 900 ℃. However, the time is fixed for the thermal activation.

This tools used to characterize the pores growth, the structure and the efficiency of water filtration were used to achieve objective numbers two and three. The Fourier Transform Infrared Spectroscopy (FTIR) and Scanning Electron Microscopy (SEM) were two machines for this research. The FTIR analysis is used to assess the elemental raw oil palm shell and the whole sample of activated carbon, and the SEM is used to describe the morphology of the surface and the growth of the pore scale. Furthermore, the burn-off degree and will be use in this study. Figure 1.2 shows the relation objective and scope.

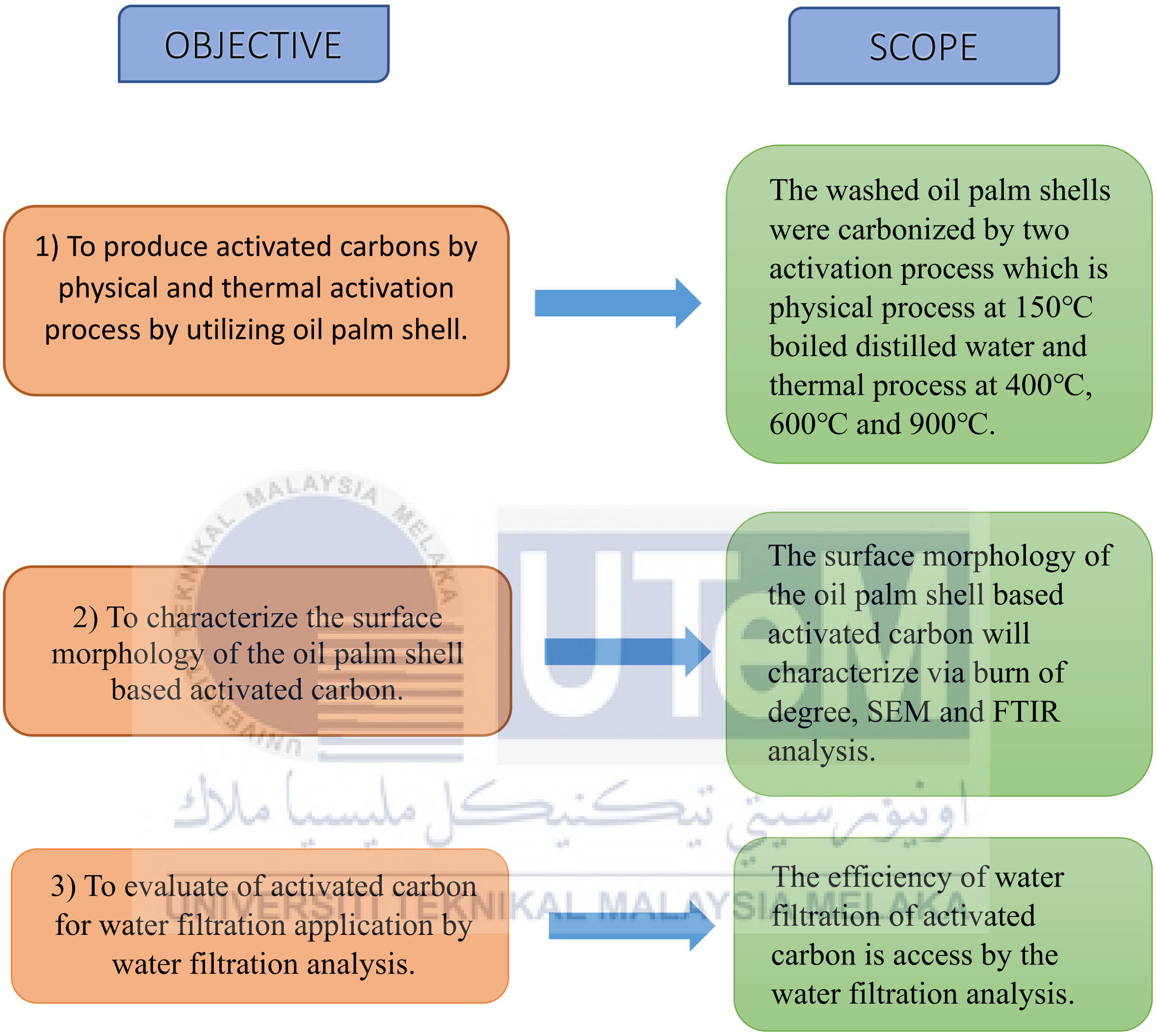

Figure 1.2: The relation objective and scope.

1.5 Significant of The Study

The research of preparation and characterization of activated carbon by physical activation and thermal activation process is done to provide the cost-effective activated carbon and to overcome the waste of agricultural. Beside that also to help the world demand in commercial activated carbon. The oil palm shell provides another alternative raw material for activated carbon. It also helps save the problems of the ecosystem caused by agricultural waste since the oil palm shell is a waste of agriculture, it can be served as a sustainable source for activated carbon. The study also avoids the chemical usage or chemical method would be hazardous in order to obtain excellent activated carbon that can be used in many applications for example at industry of filters such as in air conditioning air purifier and water filter..

CHAPTER 2

LITERATURE REVIEW

Literature reviews of previous analyzes are covered in this chapter. The raw materials for activated carbon were investigated and discussed in depth in various agricultural waste extractions. The morphological structure of activated carbon was illustrated in particular. Different raw materials addressed the preparation methods and the future application of activated carbon. Late development in the characterization of activated carbon and associated work has been regularly reviewed using different instruments and procedures. In the entire study, activated carbon was used for water filtration because it was managed effectively.

2.1 Activated Carbon

In the kitchen, charcoal is commonly used. It is used in systems of water treatment, vacuum cleaners, art creation and barbecue preparation. Most of them know about and use charcoal at some stage. The charcoal is flexible and is used in water extraction from chlorine, odor reduction, drawing, and cooking.

The version in which oxygen is upgraded and applied to it, thereby adding to its surface area, is activated with charcoal or activated carbon (AC). Normally, it is used for the removal of chemicals, contaminants, and gases. Consequently, in water purification systems and fish tank filtering systems, AC has replaced charcoal. Activated carbon has proved to be more effective if toxins are to be removed.

Activated carbon can be derived from carbon aceous materials as shown Figure 2.1 and Figure 2.2 such as coconut shell, oil palm shell, scrubber dust, agricultural residues. It was noted that effectiveness in the removal of impurity is a feature of the material and the production method. Oil Palm Shell is typically regarded as waste in oil palm production, although it is widely used as fuel for cooking and boiler firing during the processing of palm oil.

Figure 2.1: Activated carbon from coconut shell (Activated carbon coconut shell charcoal, 2017)

Figure 2.2: Activated carbon from oil palm shell (Palm Kernel Shell Charcoal Machine for Sale - Beston Group, 2021)

2.1.1 Types of Activated Carbon

Activated carbons are complex products which are difficult to define based on their behavior, surface characteristics and preparation methods. The kinds of activated carbon available are granular and pellet in the current market, which is powder. It is categorized according to its particle sizes and form, and each type is given its application. However, some broad classifications are made for general purposes based on their surface features, behavior and preparation methods.

2.1.1.1 Powdered Activated Carbon (PAC)

Enabled Powdered Carbon consists as shown in Figure 2.3 of fine granules smaller than 1.00 mm in size. On average, their pore diameter is between 0.15 and 0.25 mm. They are usually prepared with raw materials which are finely ground. In general, because of its high loss of pressure in applications, PAC is not used. The existence of a high surface area to volume ratio, however, is their main property. Water treatment, sugar decoloration, grease discoloration, food additives, purification, deodorisation, surgical injection, plating and waste incineration are the main applications for the PAC.

Figure 2.3: Powdered Activated Carbon (PAC) (khy, 2021)

2.1.1.2 Granular Activated Carbon (GAC)

Granular Activated Carbon (GAC) as shown in Figure 2.4 is much larger than PAC in size. A lower surface area PAC exhibits this type of activated carbon. Usually, the main application GAC is used for water treatment, caramel decolorizing, air purification, swimming pools and aquariums. GAC also be used for gas or vapor phase application.

Figure 2.4: Granular Activated Carbon (GAC) (khy, 2021)

2.1.1.3 Extruder Activated Carbon (EAC)

Extruder Activated Carbon (EAC) as shown in Figure 2.5 is a mixture of powdered activated carbon and a binder that is merged and extruded into a 0.8 to 130 mm diameter cylindrical activated carbon block. These are primarily used for gas-phase applications because of their low pressure drop, high mechanical strength and low dust content.

Figure 2.5: Extruder Activated Carbon (EAC) (khy, 2021)

2.1.1.4 Impregnated Carbon

Specific applications related to air pollution control, inorganic metals such as aluminum, manganese, zinc iron, lithium and calcium are impregnated with types of antimicrobial or antiseptic agents, this type of activated carbon also finds use in water purification processes.

2.1.2 Commercial Raw Material of Activated Carbon

2.1.2.1 Palm Oil

The oil palm is native to West Africa, where it is found in the Angola-Gambia region. When it was planted in 1848 at the Bogor Botanical Garden, Indonesia, it was introduced to South-east Asia. In the 1870s, from the Royal Botanic Gardens in Kew, England, where it was planted at the Singapore Botanic Garden, Malaysia collected its first batch of oil palm. Its appearance has made it suitable as an ornamental plant for use. Soon, along major roads, in front of government buildings and in public parks, oil palm was a common sight. The industrial Revolution of the 19th century in Europe prompted many young entrepreneurs, including a young Frenchman, Henri Fauconnier, to make their fortunes in East Asia. Fauconnier arrived in Malaya in 1905 and, months later, established a coffee plantation in Rantau Panjang, Selangor, with his friends. In 1917, he planted oil palm at Tennamaram Estate in Batang Berjuntai, Selangor. When rubber and coffee prices begin to lose value. This first commercial oil palm estate formed the basis for the development of the palm oil industry in Malaysia.(Nambiappan et al., 2018)

Following independence in 1957, the Malaysian government faced a major challenge for the redistribution of economic resources between the people. While the standard of life in urban areas was high, in rural areas there was rampant poverty. In bridging the gap and improving the livelihoods of rural poor, expansion of agriculture has been seen as a key goal. In order to take up this formidable task, the government formed the Federal Land Development Agency (FELDA) to provide 'land for landless and employment for unemployed' (Nambiappan et al. 2018). The oil palm planted area expanded remarkably to 5.74 million hectares in 2016 from a mere 55 000 ah in 1960. With the expansion of the field, palm oil production dramatically increased from less than 100 000 tons in 1960 to around 17,32 million tons in 2016.

The environmental consequences of the production of oil palm are highly controversial. Oil palm shells (OPS) are agricultural goods from the solid end of oil palm processes. Palm trees flourish in areas with heat and plenty of rainfall, such as Malaysia, Indonesia and Nigeria. (Yew et al., 2014).

In the past year, The Malaysian palm oil factory has been the world's leading oil palm oil exporter (Hamada et al., 2020). The number of plantations of palm oil increased from 400 ha (ha) in 1920 to around 4.5 million ha in 2008 from (Sulaiman et al., 2011). Furthermore, during 2008, the 410 palm oil mills in Malaysia processed almost 17.7 million tons of crude palm oil, which accounts for 41 percent of the world's total production (Hamada et al., 2020)

The area planted with oil palm trees in Malaysia grew dramatically from a mere 55 000 ha in 1960 to 193 000 ha in 1970. With the planted area reaching 1.02 million hectares in 1980 and subsequently expanding to 2.03 million hectares in 1990 and 5.74 million hectares in 2016, the development was remarkable as shown in Figure 2.6 and Table 2.1 (Nambiappan et al 2018).

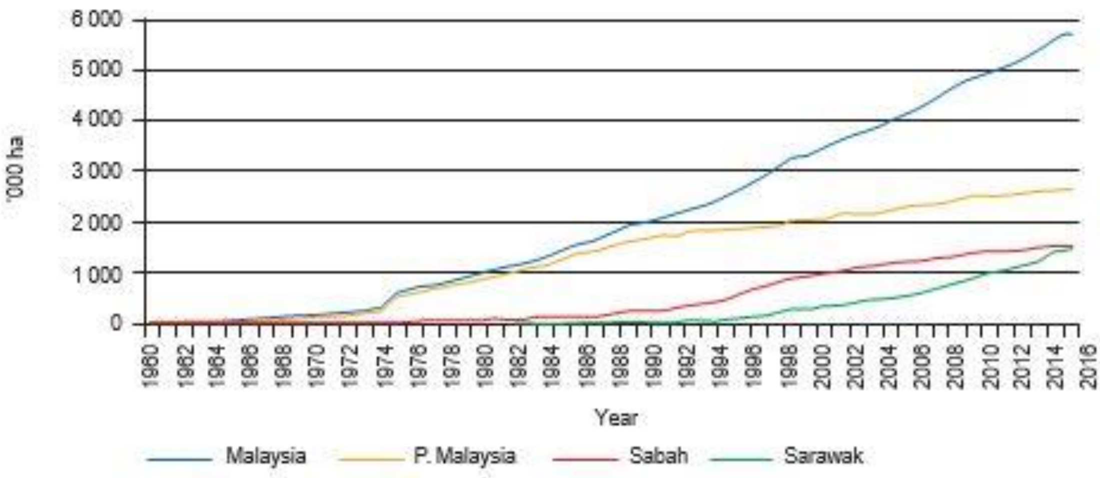

Figure 2.6: Oil palm planted area in Malaysia (Nambiappan et al., 2018).

Table 2.1: Oil palm planted area according to category, 2016 (ha) (Nambiappan et al., 2018).

Category	**ha**	**%**
Private Estates	3 508 553	61.2
Government schemes:		
FELDA	706 588	12.3
FELCRA	173 032	3.0
RISDA	71 549	1.2
State schemes	344 314	6.0
Independent smallholders	933 948	16.3
Malaysia	5 737 985	100

Other than that from (Awalludin et al., 2015), palm oil production has increased remarkably, as shown in Figure 2.7.

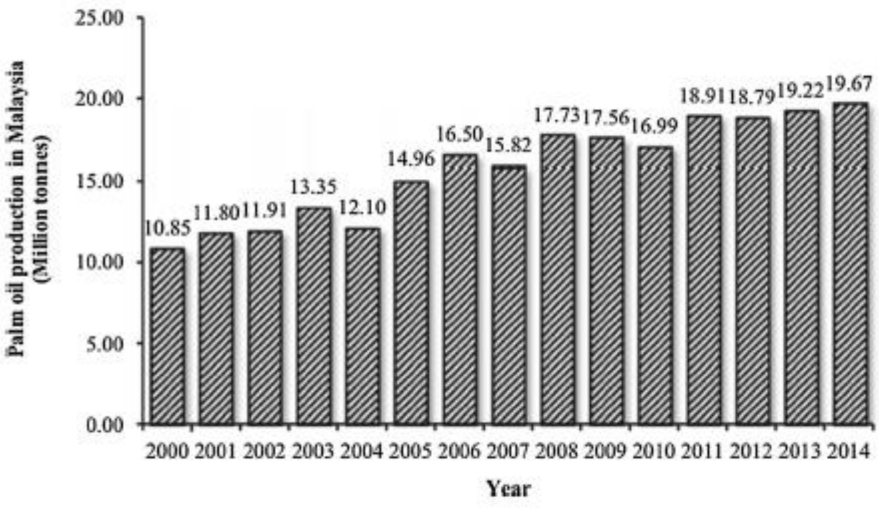

Figure 2.7: Developing the production of palm oil in Malaysia (Awalludin et al., 2015).

Two species of the *Arcaceae* or palm family, primarily *Elaeis guineensis* and *Elaeis oleifera* (less cultivated species), compose the oil palm tree. *Elaeis guineensis* is a *amonecious* species belonging to the palm subfamily *Arecoideae* that is especially planted in oil palm plantations in Malaysia. The variety, also known as the *tenera Ivariety,* is primarily a hybrid of *dura* and *pisifera* (Awalludin et al., 2015).

2.1.2.2 Oil Palm Shell

The oil palm shell is a waste agro substance that is collected after the palm oil is extracted by grinding the nut at the oil mill. (Ogundipe et al., 2021). In addition, there was most hydrogen in the oil palm shell. Palm oil is the most popular vegetable edible oil in the world. Indonesia and Malaysia are the main countries in the palm oil industry, contributing around 85% of global production. (Ocampo Batlle et al., 2020). Any ton of palm oil that is processed would produce about five tons of wastes. Palm waste consists of palm leaves, fresh palm fruit bunches, oil palm shell shell and palm trunks, and millions of tons of palm kernel shells have been made each year. (Chen et al., 2021).

In Osogho, Nigeria the oil palm shell was obtained from a local market. To extract unnecessary materials such as stone, wood, and so on, the oil palm was sorted. The Fig shows before being crushed, reveals the different sizes and shapes of the raw oil palm shell (Ikubanni et al., 2020). In rural community environments, when cooking, oil palm kernel shell is added into the fire in their raw form. While this helps to speed up the cooking process, due to the organic material property, which is unsafe for health, this causes unnecessary smoke (Osei Bonsu et al., 2020). Typically, after introducing the shells into the flames, much of the energy content is not absorbed as incompletely burnt palm kernel shells are a common sight at ash dumps (Osei Bonsu et al., 2020).

Figure 2.8: Oil Palm Shell ("Buy Palm Kernel Shells No.1 seller | Asian Biomass Resource," 2020)

Although oil palm shell waste products have now been recognized as a useful source of energy, the waste oil palm shell generated by oil producers far outweighs industry use (Bediako et al., 2016). These oil palm shell wastes are dumped in large tonnages on sites and across many of the country's palm oil producing areas (Bediako et al., 2016; Osei Bonsu et al., 2020). As one of the items accruing from oil palm processing, oil palm shell can be adequately converted to renewable energy to meet the de-mand of the ever-growing population of fuelwood and charcoal (Agyei et al., 2018). Table 2.2 displays the physical properties of the oil palm shell sample in this analysis. The collected oil palm shell moisture content was 6.56 % while the ash content was 8.86%. After achievement of equilibrium with the laboratory environment, The moisture content value is within the usual level (Edmund et al., 2014)

Table 2,2: Physical properties of oil palm shell (Edmund et al., 2014).

Property	Value	Okoroigwe et al.
Moisture content (%)	6.56	6.11
Ash content 9%)	8.86	8.68
Bulk density (kg/m^3)	745	740

2.1.3 Summary of Literature Review

From the reviewed, it can be summarized that oil palm shells are one of the sources raw material of the natural plants that produce or planted in huge amount in Malaysia which making for source for economy Malaysia. Thus, the waste product has now been recognized as energy and potential to be used as the alternative for the non-renewable resource. The summary of the main findings in oil palm and oil palm shell is shown in Table 2.3.

Table 2.3: A summary literature review of oil palm and oil palm shell

References	Subtopic	Findings
(Nambiappan et al., 2018)	Palm oil	The first commercial palm oil property established the base for the growth of the Malaysian palm oil industry
(Yew et al., 2014)		Issues the environmental effects of oil palm production
(Hamada et al., 2020)		Malaysia becoming the world's largest exporter of oil palm products.
(Sulaiman et al., 2011)		The number of plantations of palm oil increased from 400 ha (ha) in 1920 to around 4.5 million ha in 2008.
(Awalludin et al., 2015)		Two species of the *Arcaceae* or palm family, primarily *Elaeis guineensis* and *Elaeis oleifera*

		(less cultivated species), compose the oil palm tree
(Ogundipe et al., 2021)	Oil palm shell	The oil palm shell is a waste agro substance that is collected after the palm oil is extracted by grinding the nut at the oil mill.
(Ocampo Batlle et al., 2020)		The oil palm shell had the largest amount of hydrogen.
(Chen et al., 2021)		Manufacture millions of tons of palm kernel coats
(Ikubanni et al., 2020)		Nigeria the oil palm shell was obtained from a local market
(Osei Bonsu et al., 2020)		Speed up the cooking Process, due to the organic material property, which is unsafe for health, this causes unnecessary smoke
(Agyei et al., 2018)		Population of fuelwood and charcoal
(Edmund et al., 2014)		The collected oil palm shell moisture content was 6.56 % while the ash content was 8.86

2.2 Application of Activated Carbon

2.2.1 Water Treatment

In water treatment, activated carbon is commonly used to extract water contaminants from drinking water and pool water. Activated carbon is used in the home water filter system due to its excellent adsorption capacity). Water is important for humans; it accounts for about 60% of human weight and a sufficient intake must be balanced by losses by various metabolic and excretory processes. Water can contain pollutants that can affect the quality of life and health. Water intended for human consumption must be free from organisms and must contain quantities of chemical compounds that are likely to pose a health hazard (Atabaki, 2014)

2.2.2 Supercapacitor

The pass researcher from (Ukkakimapan et al., 2020), the supercapacitor can bridge the gap between batteries (high energy capacity and low power density) and ordinary capacitors, as a high energy density, high charging rate and long life cycles renewable energy storage system (low energy density and high power density).

The preparation of activated carbons for supercapacitor application through acidic dehydration of durian husk, has been performed. A wide range of applications are provided by Supercapacitor, from handheld electronic devices to electric vehicles. Most of the output of the supercapacitor the electrode and compatible electrolyte material are determined. In recent decades, activated carbons have been widely used as supercapacitor electrode materials, using their advantages in terms of a high specific surface area, a tunable pore structure, adjustable carbon network functional groups and low cost. Powdered activated carbon made from coconut shells are used for industrial supercapacitors (Ukkakimapan et al., 2020).

2.3 Preparation of Activated Carbon

Based on the recent study, only a few studies are reporting on the preparation of activated carbon. Based on the research (Lv et al., 2020), by using rice husk as the precursor, the rice husk-based activated carbon was prepared. First, the carbon of the rice husk was obtained by carbonizing the pretreated rice husk by using nitrogen as a protective gas in a tubular furnace at the specified temperature. And the activated carbon was then obtained, KOH and EDTA-4na in a tubular furnace at 750°C by copyrolysis of rice husk carbon in Figure 2.9 show the procedures and devices used to prepare the activated carbon process.

Figure 2.9: Rice husk pyrolysis diagram and alteration of activated carbon (Lv et al., 2020)

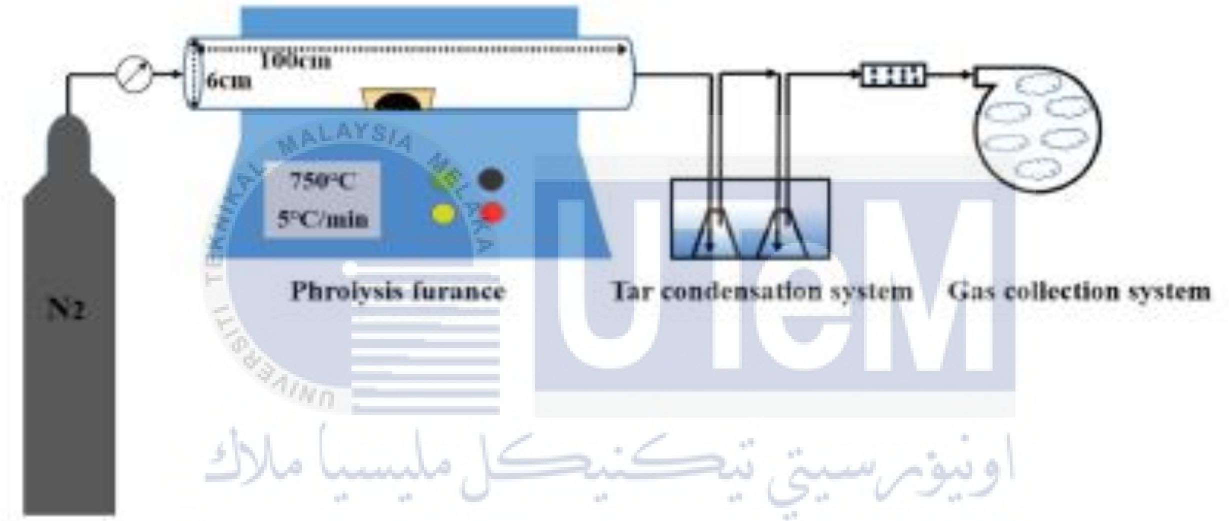

Figure 2.10: The process and preparation device used to produce the activated carbon (Lv et al., 2020)

Moreover from (Ukkakimapan et al., 2020), At a ratio of 1:2.5, the hydrochar is impregnated with NaOH. The activation was carried out under an argon (99.999 %) atmosphere at a flow rate of 500 mL/min at 720 °C at a heating rate of 5 °C/ min for 1 h. The sample was washed with DI water again after activation, until it reached a normal pH. The schematic diagram shows in Figure 2.11.

The durian husk derived activated carbon were also prepared through a traditional process of carbonization and activation, as stated elsewhere. In short, the carbonization of durian husk powder under the argon atmosphere was performed at 600 °C for 2 hours at a heating rate of 5 °C/min at a flow rate of 500 mL/min. The carbonized sample was then washed at 100 °C for 3 h with 1 M of H_2SO_4 to eliminate inorganic impurities and was repeatedly washed up to neutral pH with DI water.

Figure 2.11: Schematic diagram of preparation activated carbon via acidis dehydration of durian husk (Ukkakimapan et al., 2020).

From the passed studies, the summarization of preparation of activated carbon in Table 2.4.

Table 2.4: The summarization of preparation of activated carbon.

References	Subtopic	Findings
(Lv et al., 2020)		The active carbon dependent on rice husk was made The pretreated rice husk by using nitrogen as a protective gas in a tubular furnace at the specified temperature The activated carbon was then obtained, KOH and EDTA-4na in a tubular furnace at 750°C by copyrolysis

(Ukkakimapan et al., 2020)	Preparation of Activated carbon	A ratio of 1:2.5, the hydrochar is impregnated with NaOH The activation was carried out under an argon (99.999 %) atmosphere at a flow rate of 500 ml/min at 720 °C at a heating rate of 5 °C/ min for 1 h Durian husk derived activated carbon were also prepared through a traditional process of carbonization and activation The carbonized sample was then washed at 100 °C for 3 h with 1 M of H_2SO_4

2.4 Activation Process

Rice husk is the rice processing by producing the advantages of large-scale, rich resources and renewable, collectable and affordable cultivation(Wang et al., 2020) Many passed researcher use chemical activation for activated carbon based on rice husk. Referring to the (Muniandy et al., 2014; Prauchner et al., 2016) the chemical activation is normally achieved by KOH, K_2CO_3, $ZnCl_2$ or H_3PO_4 as activator in the inert gas at high temperatures. In general, chemical activation, particularly with KOH as an activator, can be achieved within a short period of time, with low energy consumption that used for activation process based on rice husk (Singh et al., 2017; Yang et al., 2018).

Based on the pass researcher (Lv et al., 2020). The as-prepared carbon of the rice husk was blended with KOH with a 1:3 mass ratio. The mixture was then placed into the tubular furnace, into which nitrogen was continuously flowed for 1 h to extract air with a flow rate of 250 ml·min-1. When the tubular furnace temperature reached 750 ° C, the flow rate of nitrogen was set to 150 ml·min-1, and the temperature remained for 1 h. The as-made carbon content was washed until the pH value was stable with distilled water, and then dried for 4 h at 80 °C to get the activated carbon.

Table 2.5 highlights the previous research on the single-step activation mechanism, which indicates that less scientific attention has been given to single-step CO^2 activation compared to single-step steam activation from (Shoaib & Al-Swaidan, 2015)

Table 2.5: Compilation single step-activation (Rashidi & Yusup, 2017)

Year	Precursors	Activation agent	Temperature (°C)	Dwell time (min)	Flow rate (cm^3/min)	Heating rate (°C/min)	Application
Activation agent was flown directly from beginning of heating process							
1997	Cornelian cherry, Oleaster Stones	Steam	700–800	240	n/a	n/a	n/a
2000	Oil palm stone	CO_2	650–950	30–180	25–200	5–20	NO_2 adsorption
2002	Coconut husk	Steam	600	60	3	10	Phenol adsorption
2006	Apricot stone	Steam	650–850	60–240	30 g/min	n/a	n/a
2010	Coconut shell	CO_2	750–950	60–160	60–600	10, 30, 50	n/a
2011	Pine cone, maple seeds, peach stone	Steam	400–1000	30	20	n/a	n/a
2014	Olive stone,	O_2-N_2	400–650	30–115	100	n/a	CO_2 uptake
2014	Almond shell	O_2-N_2	400–650	26–108	100	n/a	CO_2 uptake
2015	Cherry stones	CO_2, steam	750–950	10–20	100	n/a	CO_2 uptake
Activation agent was flown once reaching the desired activation temperature (Ramping under N_2 atmosphere)							
2013	Pinang frond	CO_2	800	180	150–600	20	MB adsorption
2015	Pecan nutshell	Steam	600–857	80	0.7	10	Dyes removal
2014	Pinang frond	CO_2	800	60–420	300	20	RBBR removal
2017	Sawdust	Steam	800	120	n/a	10	In liquid phase catalysis

In Table 2.6, Some of the agricultural waste materials used for activated carbon preparation in the past are listed. In the preliminary studies of agriculture waste biomass, activated carbon has been used primarily for water treatment, adsorption of heavy ion metals, dyes and surfactants on account of its high surface area. Activated carbon preparation can be carried out with two processes, either by two-stage physical activation or chemical activation, a single-stage process (Om Prakash et al., 2020).

Table 2.6: The various materials used for the preparation of activated carbon from agricultural waste (Om Prakash et al., 2020).

S.No	Raw Material	Activation method
1	Cocunut shell	Chemical activation
2	Rice hulls	Physical activation
3	Nutshell	Chemical activation
4	Sugarcane molasses	Chemical activation
5	Rice husk	Chemical activation
6	Corn cob	Physical activation
7	Coir pith	Chemical activation

From the past studies, the summarize of activation process shows in Table 2.7.

Table 2.7: The summarization of activation process

References	Subtopic	Findings
(Wang et al., 2020)		Raw material rice husk
(Muniandy et al., 2014; Prauchner et al., 2016)		The chemical activation is normally achieved by KOH, K_2CO_3, zncl$_2$ or H_3PO_4 as activator
(Singh et al., 2017; Yang et al., 2018)		Chemical activation, particularly with KOH as an activator
(Shoaib & Al-Swaidan, 2015)	Activation process	Single-step CO2 activation compared to single-step steam activation
(Rashidi & Yusup, 2017)		Various activation from past researcher such as steam CO2 and O2-N2
(Om Prakash et al., 2020)		Chemical activation, which is a one-step procedure, or physical activation, which requires two phases.

2.5 Characterization of Activated Carbon

Generally, the elemental of pore development or morphology structure attributes of activated that can be evaluated and determined by using various microscopy and physical analysis tools. However, Fourier Transform Infrared Spectrometry (FTIR) analysis and Field Emission Scanning Electron Microscopy (FESEM) were emphasized and discussed in detail for characterize of activated carbon obtained in this study.

2.5.1 Burn-off Degree

Based on passed research, the porous structure was characterized by N^2 adsorption of the various activated carbons obtained. Using a Micrometers ASAP 2010 Accelerated Surface Area and Porosimetry System, experiment was conducted at 77 K. Using Micromeritics and Quantachrome tools, by analysis of the isotherm adsorption via the BET equation and DFT models, the basic surface was determined. The results of BET and DFT were compared using the equations of Kaneko and Dubinin (Lobato et al., 2017). The degree of activation, also known as burn-off, defined as the percentage after thermal treatment of the carbonaceous material burnt mass) could be calculated from the equation.

$$burn - off(\%) = \frac{x_1 - x_2}{x_1} \cdot 100 \qquad \text{Equation 2.1}$$

Where x1 and x2 are the carbonaceous mass before and after activation. It is important to remember that, according to the burn-off rate, temperatures have been chosen and higher temperatures have not usually been applied because of excessive burn-off. To achieve X-ray diffraction patterns, the X'Pert Philips PMD diffractometer was used with a Panalytical X'celerator sensor using graphite-monochromised Cu-Ka (k = 1.54046 Å). An i-Raman spectrometer by B&W Tek with a laser beam of excitation 532 nm was used for the collection of Raman spectra. The spectral lines were obtained with a buying time of 6 seconds, 2 seconds and a 6-time multiplier. Without any special procedure, the powder samples were directly placed on the sample holder. (Lobato et al., 2017).

2.5.2 Fourier Transform Infrared Spectroscopy (FTIR)

In order to find out the various chemical bonds, chemical compounds and functional groups present in the materials, FTIR spectroscopy analysis is conducted. For activated carbon study, PerkinElmer FTIR spectroscopy with a spectral range of 4000-400 cm^{-1} was used (Om Prakash et al., 2020).

An activated carbon FTIR analysis was conducted to study the surface functional groups. Figure 2.12 demonstrates the FTIR spectroscopy of activated carbon as well as of the parent material. As compared to arhar powder AC, the peak intensities of AC materials are lower. The 3443 cm^{-1} peak is due to the hydroxyl group's -OH stretching, which decreased significantly in activated carbon (Sreńscek-Nazzal et al., 2013). Reduced strength due to decomposition during the process of carbonization in activated carbon FTIR spectroscopy. The band with 2922 cm-1 corresponds to the C–H vibration of a methoxylic group, symmetrical and asymmetric. In activated carbon content, the sharp peak at 1047 cm-1 due to C-O vibrations in the parent material has almost disappeared. C=C stretching of aromatic carbon groups is due to the peak at 1635 cm^{-1} (Om Prakash et al., 2020).

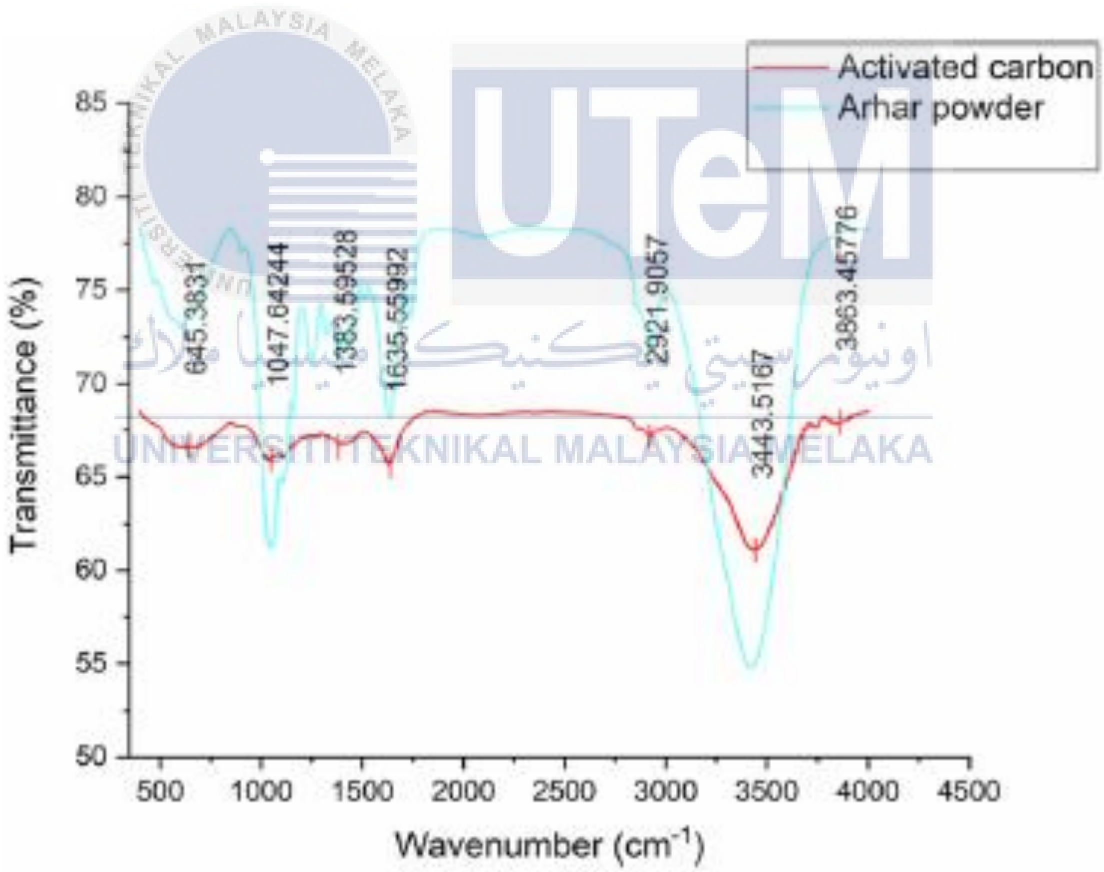

Figure 2.12: Arhar Stalk powder and activated carbon FTIR spectroscopy (Om Prakash et al., 2020).

Moreover according to (Muniandy et al., 2014), the identification of the basic functional groups, all activated carbon samples were analyzed by FT-IR Spectroscopy (Perkin Elmer Spectroscopy 2000 FT-IR Spectrophotometer, 4000-400 cm-1) using sample pellets formed by mixing with anhydrous KBr. Prior to study, the samples and the KBr salt were oven-dried at 110 _C for 24 h.

FT-IR spectra were extracted from the activated carbon samples shown in Figure 2.13, which were prepared from RH under different conditions. The wide peaks in the spectrum about 3300-3400 cm^{-1} were due to the stretching vibrations of water molecules' O-H bonds that were adsorbed to the sample matrices. The occurrence of peaks between 1500 and 1600 cm^{-1} in the area suggests the presence of aromatic compounds in C=C bonds. As a consequence of washing the activated carbon samples with 1.0 M nitric acid, the sharp band at 1384 cm^{-1} is due to residual nitrate ions (NO_3) (Muniandy et al., 2014).From the pass studies, the characterize of activated carbon show in Table 2.8

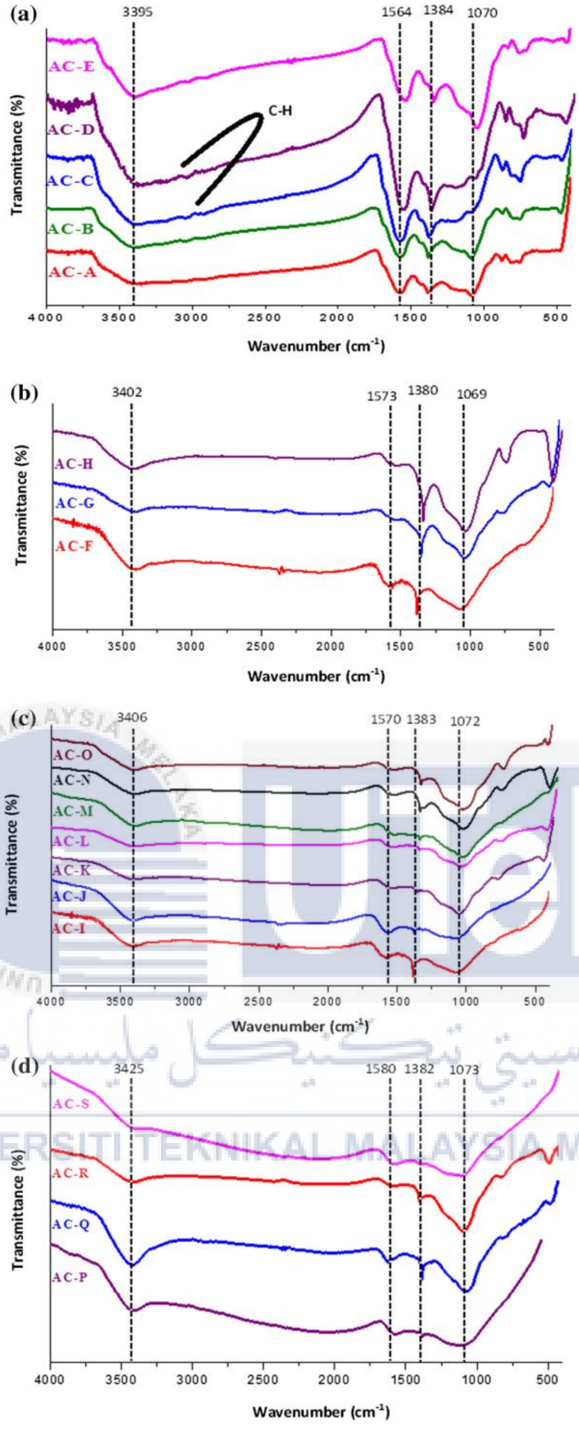

Figure 2.13: Activated carbon samples were prepared under different activation temperatures, period and triggering agent FT-IR spectra. .For the activation conditions of samples in (a), (b), (c) and (d) (Muniandy et al., 2014).

2.5.3 Field Emission Scanning Electron Microscopy (FESEM)

FESEM model: FEI NOVANANO SEM450 and high-resolution transmission electron microscopy (HRTEM,300 kV, FEG: make and model-FEI, Tecnai G2 TF30-ST) were subjected to activated carbon samples of Calotropis gigantea stem. The surface physical morphology of the samples is obtained at various active temperatures and at different impregnation ratios. (Sahu et al., 2020)

According to (Kumar & Jena, 2016), The pores on the external carbon surfaces vary in size and shape depending on the impregnation ratio and activation temperatures. The micrographs show that the activated carbons' external surfaces are riddled with pores that have become very irregular as a result of activation. The pores appear to be the product of a chemical evaporation, leaving the space filled previously by chemicals, according to these micrographs. More advanced porous structures are found on the activated surface of the carbon, where small pores form. The creation of pores is thus assisted by FESEM research following activation on the surface of carbon materials. Such highly porous activated carbon structures may provide numerous adsorption locations.. (Pfaffmann et al., 2016; Yek et al., 2019; Yu et al., 2018) External surfaces of activated carbons in large holes also have cracks, crevices, and some grains of different sizes The external surfaces of the activated carbons have also cracks, and certain grains in wide, different-sized holes. (Köseoılu & Akmil-Başar, 2015). The Figure 2.14 show the FESEM image at various reading of temperature.

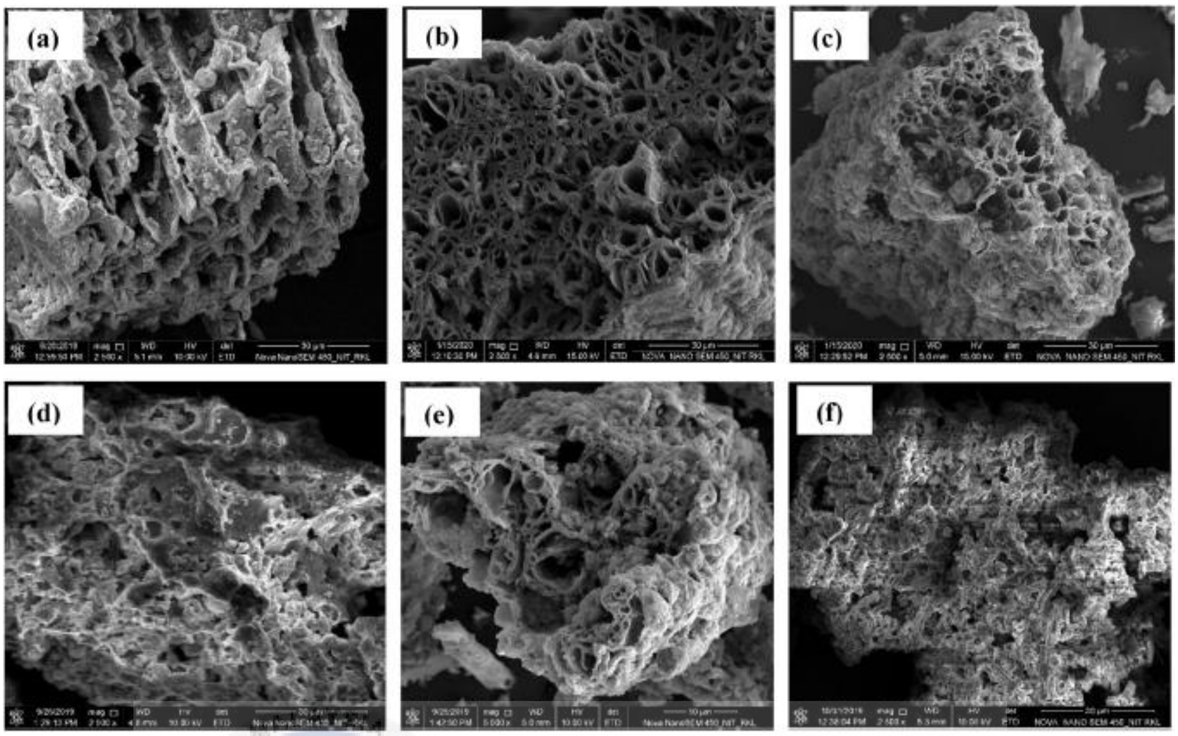

Figure 2.14 : FESEM pictures of activated carbon at different temperatures (a) (b) (c) 400 °C, 600 °C, 900 °C for impregnation ratios of 0.5:1 and (d) (e) (f) 400 °C, 600 °C and 900 °C for impregnation ratios of 1:1 (Sahu et al., 2020)

2.5.2 Scanning Electron Microscopy (SEM)

Scanning Electron Microscopy is used to study the effectiveness of activated carbon that evaluated through surface morphology and structure of the activated carbon. According to (Daouda et al., 2021), Scanning Electron Microscopy use to look at how the physical structure of the activated carbon. According (Widiyastuti et al., 2020), the Figure 2.15 (a) show indicates that when CSC 50 wt% without iodine treatment is used, the fibre structure collapses, leaving the rupture fibre behind. The fibre structure, on the other hand, is maintained with CSC 50 wt% with iodine treatment, as illustrated in Figure 2.15 (b). CSC 60 wt% without iodine treatment is recommended for use.

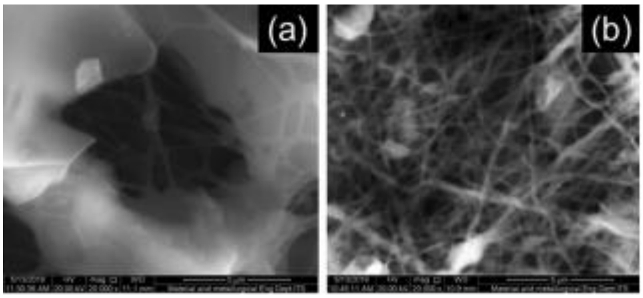

Figure 2.15 : SEM images activated carbon fiber synthesized by (a) 50 wt% CSC without iodine treatment, (b) 50 wt% CSC with iodine treatment (Widiyastuti et al., 2020)

From the past studies, the summarize of characteristic of activated carbon show in Table 2.8.

Table 2.8: The characterize of activated carbon

References	Subtopic	Findings
(Lobato et al., 2017)	Burn-off Degree	The activation degree, also called burn-off, is defined as the rate of the carbonate burnt material after thermal processing.
(Om Prakash et al., 2020)	FTIR	FTIR spectroscopy with a spectral range of 4000-400 cm-1 was used. activated carbon content, the sharp peak at 1047 cm-1 due to C-O vibrations in the parent material has almost disappeared. C=C stretching of aromatic carbon groups is due to the peak at 1635 cm-1
(Sreńscek-Nazzal et al., 2013)		Activated carbon FTIR analysis was conducted to study the surface functional groups.
(Muniandy et al., 2014)		activated carbon samples were analyzed by FT-IR Spectroscopy (Perkin Elmer Spectroscopy 2000 FT-IR Spectrophotometer, 4000-400 cm-1) The wide peaks in the spectrum about 3300-3400 cm-1 were due to the stretching vibrations of water molecules' O-H bonds that were adsorbed to the sample matrices.

CHAPTER 3

METHODOLOGY

The step by step recommended procedure for carrying out the experiment for this research is explained in Chapter 3. The discussion consists of the flow chart of the experimental materials, and also procedures that are a literature review depend on the previous observation in the previous chapter.

3.1 Introduction

The main aim of this study was the physical and thermal activation method for the production of activated carbon with the use of oil palm. First of all, the oil palm shell was cleaned by a variety of pretreatment procedures via physical and thermal activation processes. As a guide to comparison, an untreated oil palm shell has been created.

Scanning Electron Microscopy (SEM) use to exam the microscopic structure and the surface morphology of the activated carbon. Moreover, the FTIR analysis also use in this research to analyze the activated carbon. The focus of the study was focused on the growth of surface morphology and their activated carbon behavior. To determine their burn-off degree, the treated oil palm shells were weighted, and the pH value of different filtering levels was recorded.

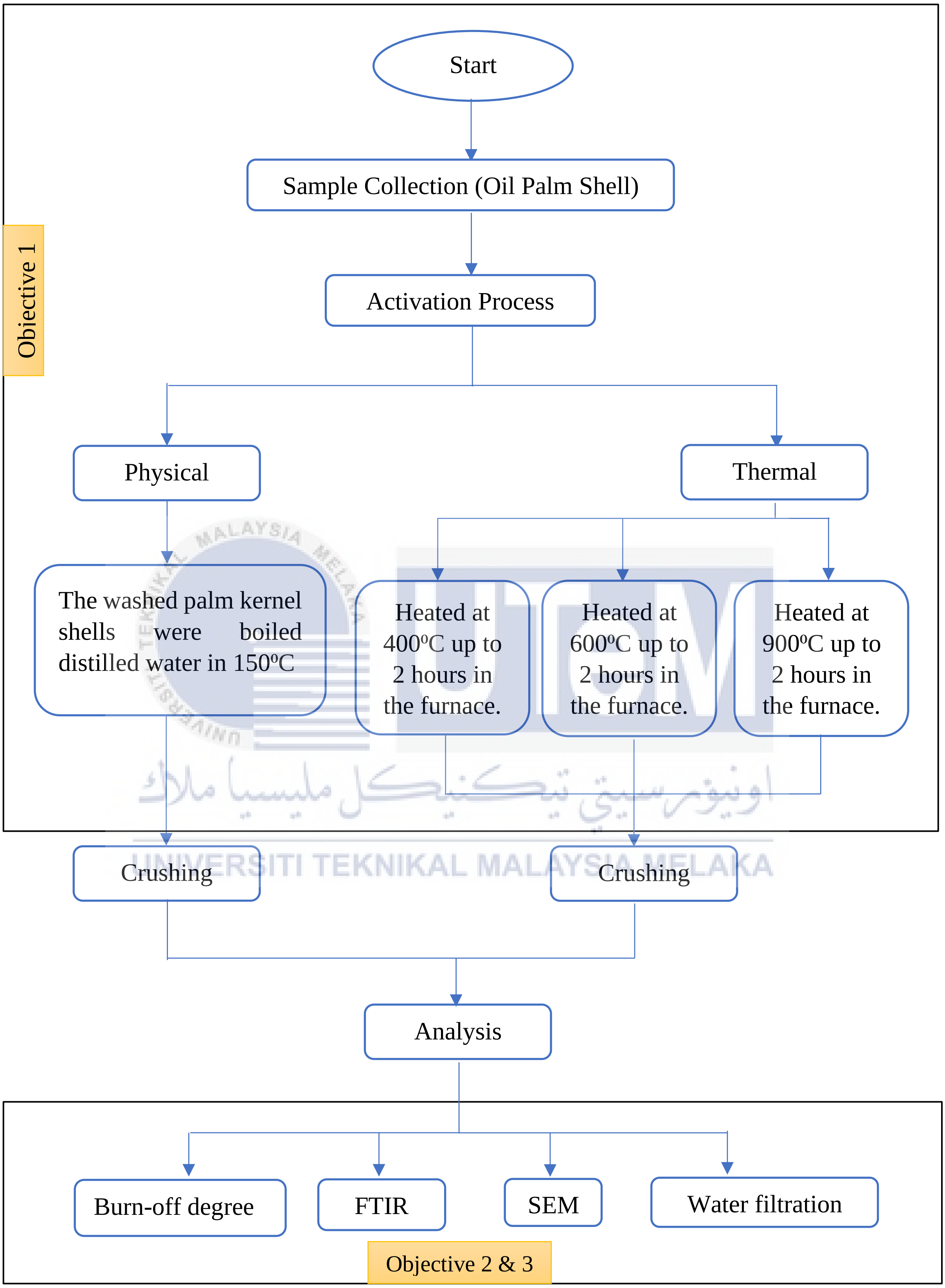

Figure 3.1: The flowchart of experiment study

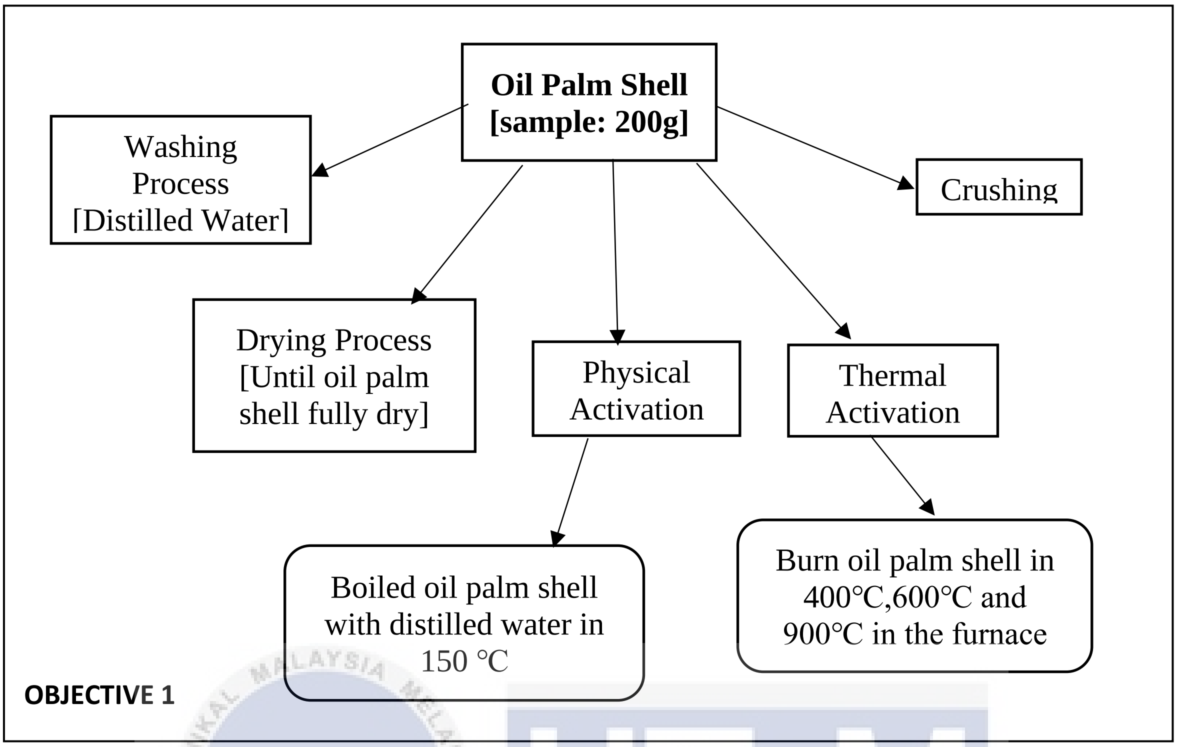

Figure 3.2: Experimental works in Objective 1

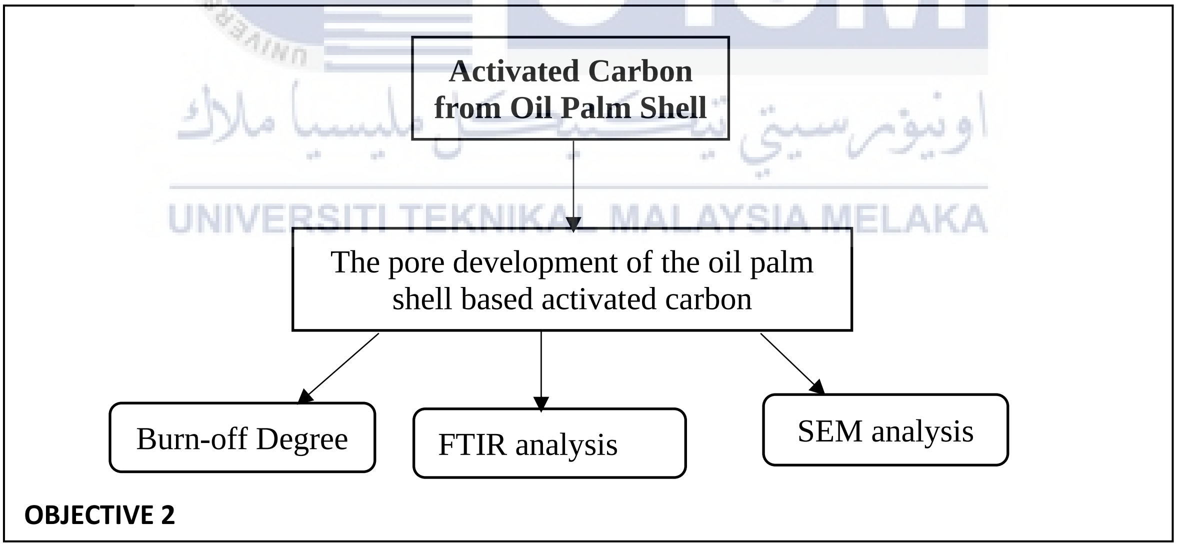

Figure 3.3: experimental works in Objective 2

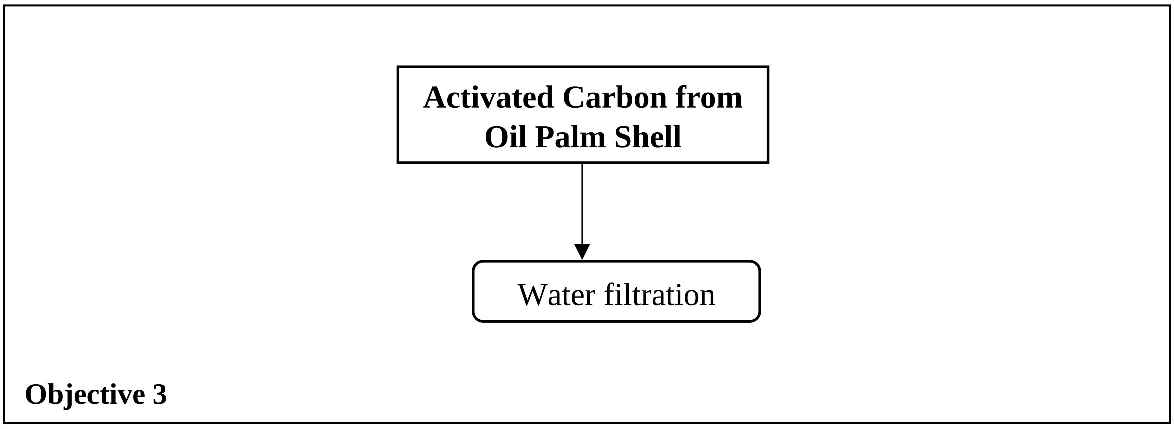

Figure 3.4: Experimental work on Objective 3

3.2 Experimental Work Regarding Objective 1

This section describes in depth the experimental work involved in Objective 1, from the preparation of the raw material to the formation of activated carbon.

3.2.1 Experimental Materials

The main experimental materials used in Objective 1, including raw materials, gloves, furnace and various functional item.

3.2.1.1 Raw Materials

In this analysis the oil palm shell is used to determine the efficiency and characterization as the raw material of activated carbon from the oil palm shell. In particular, the oil palm shell will be generated in the form of activated carbon. The oil palm shell was chosen because it is simple to obtain.. In addition, the oil palm shell is one of Malaysia's major agricultural wastes. In this research report, to investigate the potential of oil palm shell as the cost-effective activated carbon to replace the existing filter absorbent.

3.2.1.2 Other Substances

In this study research, chemical substance does not require in the process to converting activated carbon from raw material. Distilled water is being used to wash the oil palm shell to remove particulate or contaminate material from their surface material from their surface to prevent any constituent in the experiment.

3.2.1.3 Experimental Equipment

Throughout the completion of the experiment with regard to Objective 1, a few equipment used in the experiment which is bowl, gloves, furnace and weigh scale in gram as shown in Figure 3.5, Figure 2.6 and Figure 3.7.

Figure 3.5: Furnace

Figure 3.6: weigh scale

Figure 3.7: Bowl

3.2.2 Experimental Method

There were numerous experimental methods to be uses for producing the activated carbon which are consist of sample preparation, physical activation, and thermal activation method. The method had been carried out to accomplish the first objective of this research

3.2.2.1 Sample Preparation

This method was to prepare the raw material which was oil palm shell. First and foremost, wash the oil palm shell to remove any contaminants using distilled water. After the oil palm cleaned, dried under the sun for couple of degree until it dried completely. An estimated 300g of activated carbon from oil palm was prepared in every specimen. The sample of oil palm shell had been boiled at 150℃ and other three sample oil palm shell were heated at 400℃, 600℃ and 900℃ for 2 hours in the furnace. Then, all the specimen oil palm shell turns to activated carbon and the color turns to dark black.

3.2.2.2 Physical Activation Process

For the physical activation, prepared the sample oil palm shell in quantity of 300g after washed and dry oil palm shell. The sample oil palm shell with 300g were poured and mixed with distilled water. The sample were boiled in distilled water at 150℃ for approximate 2 hours in furnace. Next, the boiled oil palm shell was dry at 150℃ until fully dry. The final step for this process, crush oil palm shell and sieve in size fraction of 500um.

3.2.2.3 Thermal Activation Process

For the thermal activation, prepared three sample oil palm shell in quantity 300g after washed and dry oil palm shell. The oil palm shell separated three sample according to the temperature. All three sample burn in the furnace with temperature 400℃,600℃ and 900℃ in approximately 2 hours. For the final step after the burn process was done, record the weight every sample for the next step process.

3.2.3 Design of Parameter

Study on the process of preparation activated carbon from oil palm from the different method preparation was done. The temperature and the duration time of burn oil palm shell in the furnace. Weight of oil palm shell for every specimen was determined as the fixed. The temperature and boiled distilled water are the significant parameter for this study. Thus, four samples with different method and temperature were designed. The design was shows in the Table 3.1 and the illustration preparation of activated carbon in Figure 3.8.

Table 3.1: Design of Parameter

Physical Activation		
No of run	Temperature (℃)	Time (hour)
1	150	2
Thermal Activation		
No of run	Temperature (℃)	Time (hour)
1	400	2
2	600	2
3	900	2

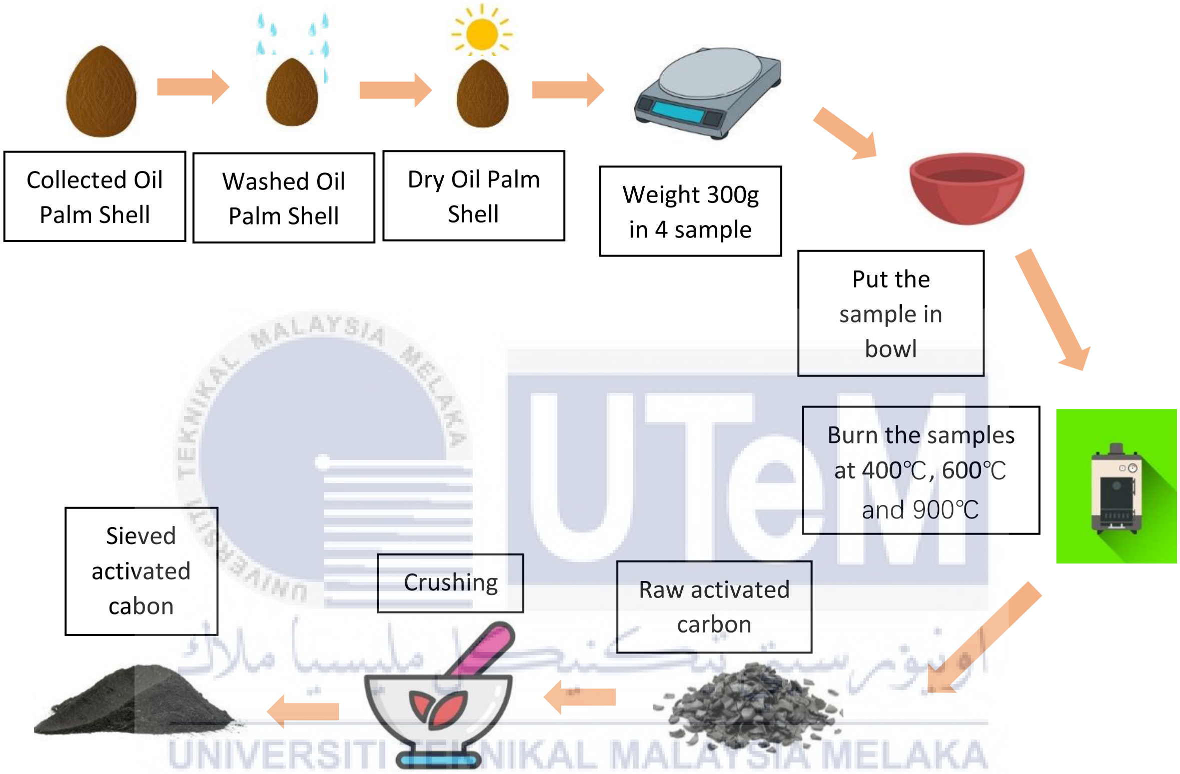

Figure 3.8: Illustration preparation of activated carbon

Based on past research, the activation process demonstrated a suitable approach for the processing of activated carbon. Moreover, the physical activation was a simple process that could be carried out using a different type of temperature and use the same time. Thus, a set of experiments with a different parameter was designed in this study.

3.3 Experimental Work Regarding Objective 2

This section describes in depth the experimental work involved in Objective 2, the pore development of the oil palm shell based activated carbon will characterize via Burn-off Degree, FTIR and SEM analysis.

3.3.1 Burn-off Degree

The degree of activated carbon burn-off, Ɵ (wt%) was calculated through equation as follow:

$$\Theta = \frac{W_i - W_f}{W_i} \times 100\% \qquad \text{Equation 3.1}$$

where Wi is the initial mass of the kernel shell sample (g) and Wf is the mass of sample after subjected to activation (g). The objective of indicating the degree of activated carbon burn-off is to define the information of micro-porous activated carbon when the extent of burn-off is less than 50 wf%. From the past research, proposed that a macro-porous active carbon was formed when the burn-off degree is greater than 75 wt% and the sample has a mixed porous structure when the degree burn-off is between 50wt% to 75wt%. The later research of pore structures in activated carbon confirmed the linearity of pores size is ascended to the degree of burn off..

3.3.2 Scanning Electron Microscopy (SEM)

A scanning electron microscopy (SEM) is widely used to examine the microscopic structure by scanning the fractured surface materials of the oil palm shell based activated carbon. In this study, SEM had been used to determine the surface morphology activated carbon before and after treatment were performed. From pass researcher, scanning electron microscopy (SEM) model Zeiss EVO 50 was used. On the surface of activated carbon after activation, new structure of surface morphology is seen along with the widening of pore diameters. On the activated carbon surface, where the smooth and roughly are formed, a greater number of temperatures the surface changes due to activation. Thus, the formation of structure after activation on the surface of carbon materials is supported by SEM research. Such different surface activated carbon structures may provide different sites of adsorption.

Figure 3.9: Zeiss SEM type Evo 50 Series. (Zeiss, Germany)

3.3.3 Fourier Transform Infrared Spectroscopy (FTIR)

In this study to analyze the functional group of the activated carbon, Fourier Transform Infrared Spectroscopy (FTIR) had been used in this study. The FTIR analysis was performed by using the FT-IR-6100 Spectrum GX-FT-IR spectrophotometer (Perkin Elmer, Germany) machine as shown in the Figure 3.10. In Addition, four samples were analyzed from different method and temperature that had been examined, which are the physical activation sample that boiled in 150°Cdistilled water and the thermal activation sample with three different temperature 400, 600°C and 900°C.

Prior to this analysis, all samples have been crushed with a diameter of about 500 µm. From the pass research, the increased activation temperature decreases the number of functional acid groups and raises the carbon's basic surface groups. The increase in activation temperature induces the decomposition of many functional groups to form CO and CO_2. This phenomenon is due to the high temperature instability of acidic groups. In the other side, when the temperature is raised, simple classes are raised. After the heating process, these groups may be formed during the cooling of activated carbon. This method of cooling allows oxygen to be fixed in the active site.

Figure 3.10: FT-IR-6100 machine (Perkin Elmer, Germany)

3.4 Experimental Work Regarding Objective 3

This section explained the experimental work in Objective 3, which water filtration analysis

3.4.1 Water Filtration

The water filtration to show the efficiency activated carbon in water filtration. The weighted activated carbon was thoroughly washed with 30 ml of distilled water for 5 m and filtered with a filter paper, and the pH of the filtrate was determined with a pH meter by dipping the pH meter into the filtrate sample. The pH influences the adsorbent's surface charge and the shape of heavy metals in the environment. Most organics at a lower pH are less soluble and more readily adsorbed. The removal decreases as the pH increases. For each sample of the activated carbon, this process was repeated. Recent research has shown that the efficiency of Cu removal by precipitation is around 10% when the pH is 6, and when the pH exceeds 8, the removal of Cu depends fully on precipitation.

CHAPTER 4
RESULT AND DISCUSSION

This chapter discussed the experimental results collected from various analyses that have been performed during the period of study. There are several subchapters that explain all the stage of preparation of the activated carbon. Burn-off degree also known as degree of activation, defined as the percentage after thermal treatment of the carbonaceous material burnt mass. The analysis is supported with the Fourier Transform Infrared Spectrometry (FTIR) analysis which is used to reveal the presence of carbon and hydrogen. Scanning Electron Microscopy (SEM) is used to study the surface morphology of oil palm shell and activated carbon. Water filtration analysis is used to specify the acidity or basicity of water filtered by activated carbon. Further elaboration of the results that were obtained for this experiment was explained in detailed in this chapter.

4.1 Activated Carbon by Physical and Thermal Activation Process

Physical activation and thermal activation are the stages in order to prepare the activated carbon. The main purpose of activation process is to provide the activated carbon from oil palm shell. The physical activation process was done by boiled oil palm shell in distilled water with duration 2 hour while the temperature as the parameter at 150 ℃ and the thermal activation process also was done by burned in furnace with duration 2 hour while the temperature varies in parameters of 400 ℃, 600 ℃ and 900 ℃. Through the experiment result, the physical activation resulted slightly change the colour of the oil palm shell. While for the thermal activation at temperature 400 ℃, 600 ℃ and 900 ℃, the oil palm shell turn to black dark colour and the weight reduces after burning process. As supported by Kumar & Jena, (2016) the textural and chemical properties of activated carbons are affected by the activation process conditions such as parameters.

4.2 Burn-off Degree

The total mass of the activated carbons after the physical activation process is 230.46 g and for thermal activation process after burned at 400 ℃,600 ℃ and 900 ℃ is 90.3 g, 55.72 g and 41.76 g. Respectively, the degree of activated carbon burn-off, ϴ (wt%) was calculated through equation below as stated by Mohd Samdin *et al.*, (2015) :

$$\Theta = \frac{Wi - Wf}{Wi} \times 100 \qquad \text{Equation 4.1}$$

Where:

Wi the initial mass of the oil palm shell

Wt is the mass of the sample after activation process

Table 4.1 : Burn-off degree physical activation

Temperature	Burn-off degree
150℃	$\frac{250-230.46}{250} \times 100 = 7.8wt\%$

Table 4.2 : Burn-off degree thermal activation

Temperature	Burn-off degree
400℃	$\frac{250-90.30}{250} \times 100 = 63.88wt\%$
600℃	$\frac{250-61.72}{250} \times 100 = 74.71wt\%$
900℃	$\frac{250-41.755}{250} \times 100 = 83.29wt\%$

As shown Table 4.1 and Table 4.2 the burn-off degree of physical activation at 150 ℃ yield as 7.8 wt%. Meanwhile, thermal activation at temperature 400 ℃, 600 ℃ and 900 ℃ resulted the yield of 63.88 wt%, 74.71 wt% and 83.29 wt%. Respectively, the higher the temperature activation, the higher burn-off degree calculated. This is supported by Chang *et al*. (2000) mentioned the higher the temperature, the more it overcomes the disadvantages of a longer activation period necessary to achieve a bigger surface area and has the ability to generate activated carbon with higher adsorption capacity. The mass of activated carbon loss, respectively burn-off degree defines the weight ratio of the activated carbon obtained to that of the calcined due to carbonization. This is supported by Tagne *et al*. (2021) the high efficiency is due to the loss of volatiles other than carbon as a result of temperature.

4.4 Fourier Transmission Infrared (FTIR) Spectroscopy Analysis

FTIR spectra of untreated oil palm shell, physical activation and thermal activation was shown in the Figure 4.3. This analysis used to study the functional group for different activation stages in order to shows the hemicellulose react before and after activation process.

Table 4.3 summarized the typical functional group that presence on the FTIR spectra in a sample untreated, 150 ℃ using physical activation and 400 ℃, 600 ℃ and 900 ℃ using thermal activation.

Table 4.3: Functional group of untreated and activation samples

Wavelength (cm^{-1})	Functional group	References
3400	Hydroxyl group (-OH)	(Hoseinzadeh Hesas *et al*., 2013)
2920	C-H stretching and deformation vibration from alkanes/aliphatic	(Wilk *et al*., 2015)
1620	C=H, aldehydes, esters, ketones, carboxylic	(Wilk *et al*., 2015)
1600-1030	C=C Aromatic hydrocarbon	(Pang *et al*., 2014)
790	C-H alkynes bends	(Wilk *et al*., 2015)

The FTIR spectra of untreated oil palm shell and activated carbon in physical and thermal activation are shown in Figure 4.1. Based on the FTIR spectrum, it was found that the activated carbon in physical and thermal activation has slightly changes of spectra as compared to untreated oil palm shell. At peak 3300-3400 cm^{-1} the strength of -OH peak was noticeably reduced; it is because with increasing pretreatment temperature. This -OH group assigned to alcohols and phenols. The peaks at 2920 cm^{-1} and 2840 cm^{-1} are associated with aliphatic methylene groups. The peak intensity of activated carbon at higher temperature activation of 150 ℃, 400 ℃, 600 ℃, and 900 ℃ had a lower peak intensity compared to untreated oil palm shells. At 1620 cm^{-1}, the carbonyl functional group (C=O) was observed, corresponding to various acids, aldehydes, and ketones formed by the decomposition of cellulose and hemicellulose. The peak intensity was reduced at higher temperatures, which could be attributed to hemicellulose breakdown.

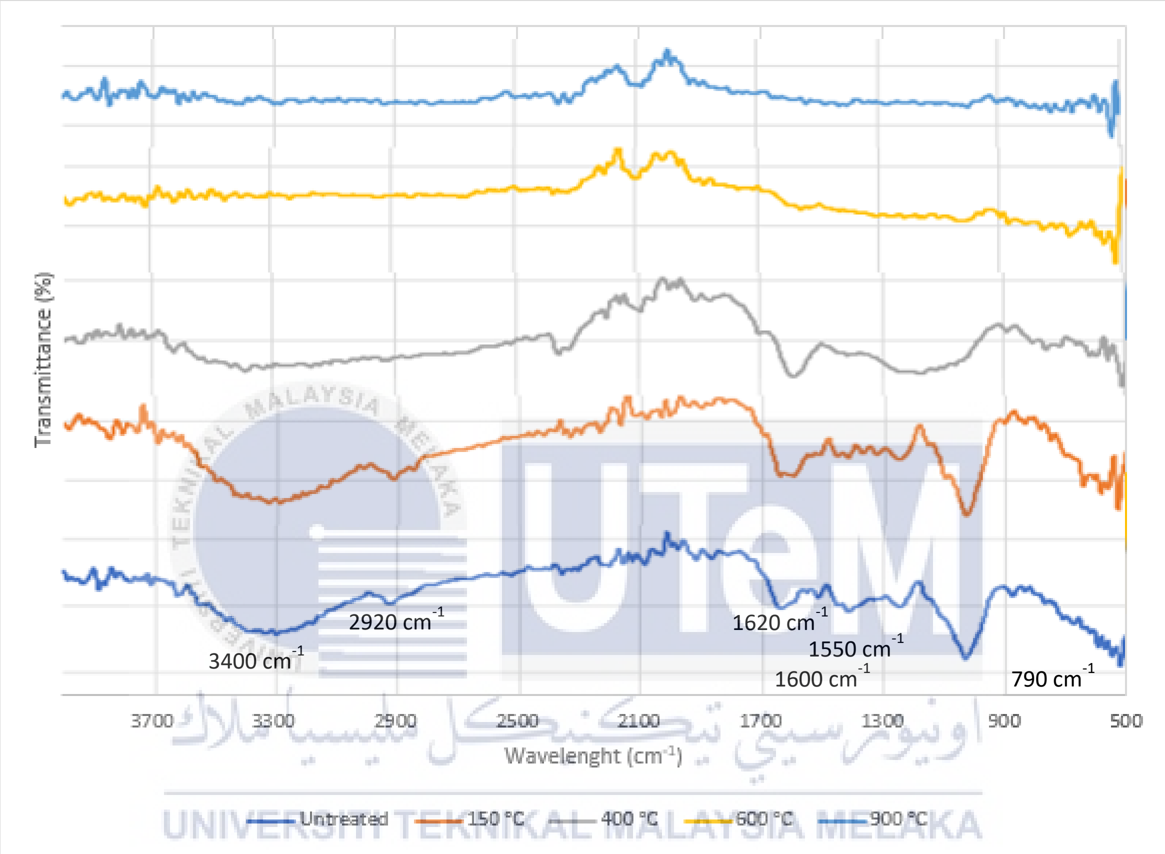

Figure 4.1: FTIR spectra of untreated and activation samples

Peaks at 1550 cm^{-1} are caused by C=C stretching alkenes. The most concentrated peaks were found in the 1600-1030 cm^{-1} range and were attributed to C=O stretching and O-H deformation in organic compounds containing oxygen (alcohols, phenols, and others). Aromatic functional groups are represented by peak at 790 cm-1 for oil palm shell. Finally, the decrease in intensity of oxygenated peak as observed in the FTIR spectra implied that the temperature of activated carbon could break the oxygen and hydrogen bonding. All the functional groups were observed on the surface of the activated carbons due to their narrow variation temperature of activation. Which is an agreement similar supported by Andas *et al*. (2017). According to Hidayu & Muda, (2016), when heat was applied to the sample during the carbonization and activation process, the functional groups from the raw material were released as volatile materials.

4.3 Scanning Electron Microscopy (SEM)

The result of the surface morphology for untreated oil palm shell and activated carbon by physical and thermal were studied after completed the process of activation. The result was analyzed by using the Carl Zeiss Model 1450 VP of variable pressure scanning electron microscope (SEM).

The raw material oil palm shell were washed with distilled water and dried before further activation process proceed to avoid other contaminant on material. The color and texture of the sample were change after the activation process. The sample then continued analyzed by SEM in order to compare their surface morphology before activation and after activation process.

Figure 4.2, the raw material surface appears to be rough and battered, with no apparent pores. Raw oil palm shell is non-porous, with a surface that is relatively flat and compact, with no obvious cracks or crevices. This is supported by Rashidi & Yusup, (2017), an aggregation is visible on its external surface, which could be caused by the presence of inorganic compounds in the carbon structure, resulting in pore blockage.

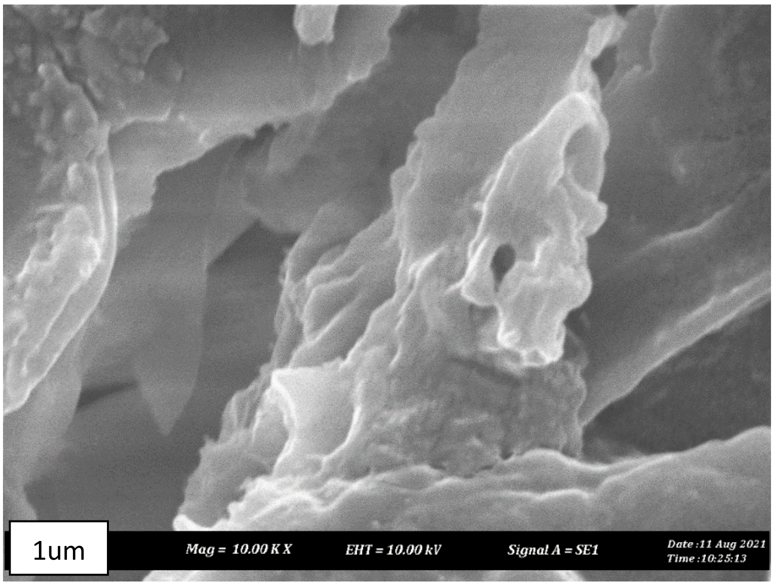

Figure 4.2: SEM micrograph of untreated oil palm shell

Besides, the surface morphology of activated carbon shown the difference structure and shape. This is due to the varies temperature of activation method. It can be seen that the activated carbon forms the porous on the surface, as formed in Figure 4.3 (a). In contrast, at an optimized temperature with duration 2 hour the activated carbon would expand and smoother as in Figure 4.3 (b). The pore widening impact is greatly increased when the activation temperature is raised which can see the Figure 4.3 (b) and Figure 4.3 (c). Meanwhile, in the Figure 4.3 (c) the structure wider and a few pores shape on the surface. Besides, the structure of Figure 4.3 (d) more roughly and the pore was observed shrinking.

The result was supported by Wang *et al,* (2014) mention that the micrograph it reveals a well-developed pore system but still contain some residues. This also proven by Nabais *et al*. (2010), whereas the residues are burned off and disappear from the SEM micrographs as the burn-off rate increases. The Figure 4.3 (b) at low temperature were resulting that the pore not fully open. This is proven by Gao *et al*., (2021) mention the pore not exist due to not provide enough energy activation reaction.. There were visible cracks and pore structure collapses when the activation temperature was raised to 900 ℃ as shown at Figure 4.3 (d). This is supported by Gao *et al*. (2021) mention due largely to the excessive etching reaction caused by the high activation temperature.

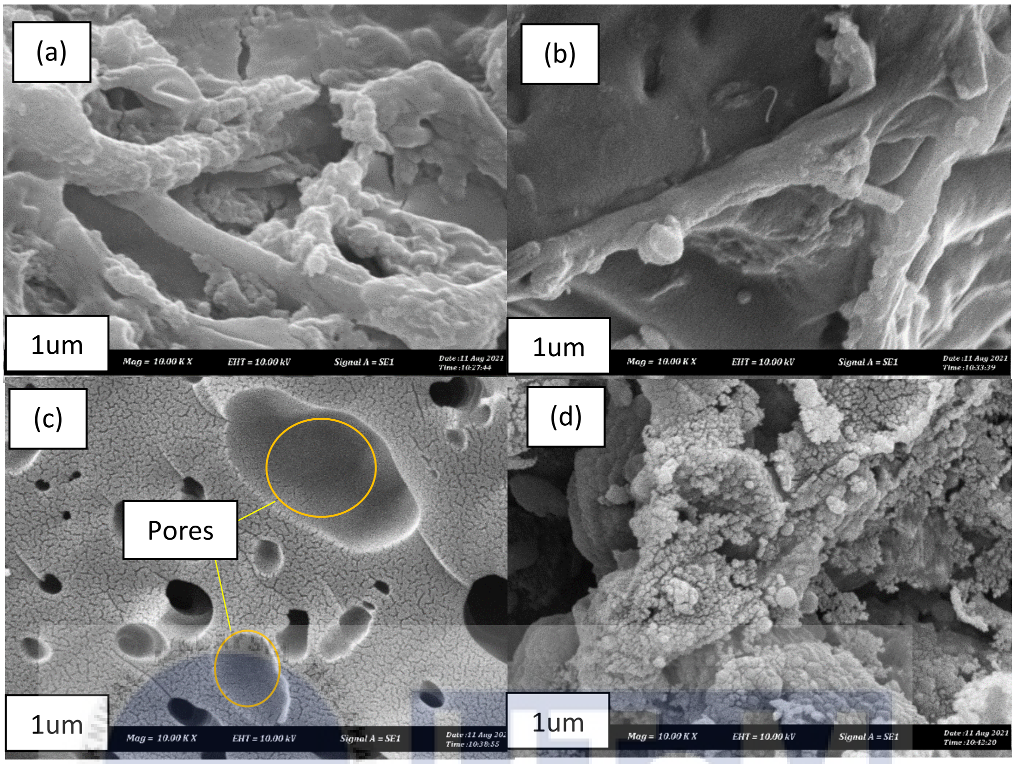

Figure 4.3: SEM micrograph of sample (a) 150 °C for physical activation and (b) 400 °C, (c) 600 °C, (d) 900 °C for thermal activation

4.5 Water Filtration Analysis

At a lower pH, most organics become less soluble and more easily absorbed. Contaminant concentration can define as the higher the contaminant concentration, the greater the capacity of activated carbon to remove it. The contaminant molecule is more likely to diffuse in a pore and become adsorbed. The upper limit for contaminants is a few hundred parts per million, if higher concentrations of the contaminate may necessarily require more contact time with activated carbon. The presence of hardness in the water improves organics removal, so place activated carbon units upstream of iron removal units whenever possible. Usually, the activated carbon is used frequently to remove chlorine upstream of ion exchange or membranes.

The use of chlorine and fluoride as disinfectants in water treatment is regulated by the Malaysian Ministry of Health (MOH) under the National Standard for Drinking Water Quality (NSDWQ). The amount of chlorine and fluoride used to disinfect the water is sufficient to kill the bacteria. However, it is safe to drink. Although drinking water treatment meets permitted criteria, residual iron, manganese, and aluminum levels in the domestic water distribution system are excessive, creating precipitation and stagnation in the system.

The pH value of direct pipe water without using an oil palm shell activated carbons filtration system was measured to be an acidic solution at pH 5.5 in the present of study. The resulted was supported by Mohd Samdin *et al*. (2015) mentioned these residues may not have been swept away by self-cleaning velocity, resulting in rust in the pipe. This is proven by Foo & Hameed, (2012) mentioned pH regulates the surface chemistry of dye molecules and activated carbons in aqueous solutions, which is crucial.

Figure 4.4 shows the graph pH values of water filtered by the activated carbons in average at five times reading with 0.5 litre water. The result 600℃ selected has the best result at pH 7. The resulted supported by Mohd Samdin *et al*. (2015), which stated the pH value was indicated to increase the reading pH value of water filtering which has achieved a constant value of the pH.

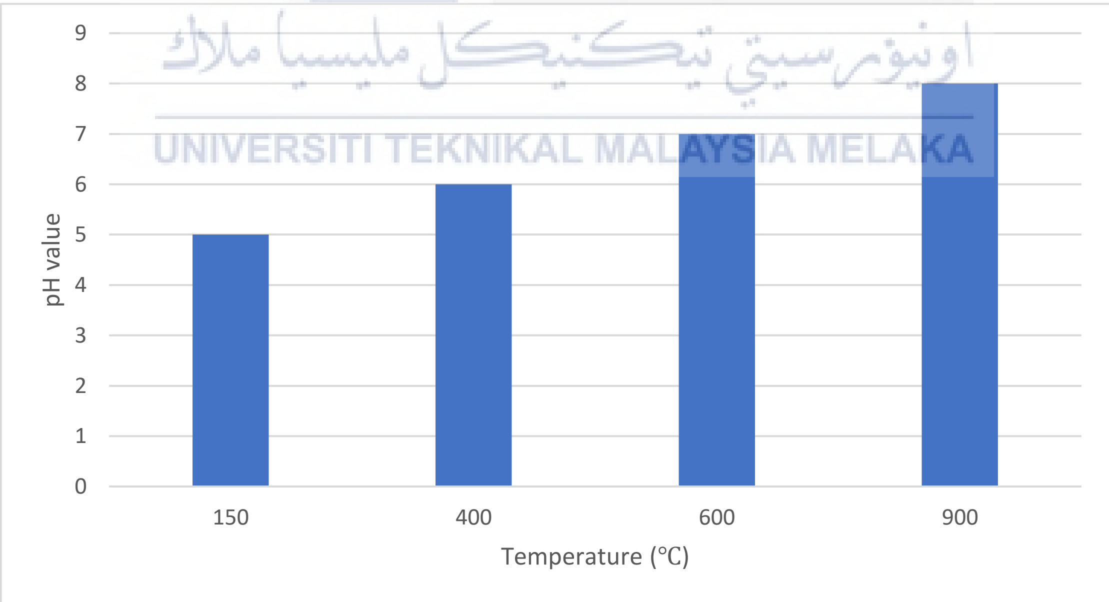

Figure 4.4 : The pH value reading.

CHAPTER 5
CONCLUSION AND RECOMMENDATIONS

5.1 Conclusion

As the conclusion, the activated carbon from oil palm shell has successfully prepared asto address the first objective of this study. The activated carbon obtained obtained was using physical activation and thermal activation method. Physical activation was performed by boiled in distilled water at 150°C for 2 hours and thermal activation was carried out by heating in the furnace for 2 hours different temperature 400℃, 600℃ and 900℃. The obtained activated carbon from oil palm shell gives another alternative of activated carbon sources to provide the cost-effective activated carbon and to manage agricultural waste.

The second objective of the study is to determine the pore development of the oil palm shell based activated carbon. The burn-off degree successfully proved the presence of pores in activated carbon. Scanning Electron Microscopy (SEM) was used to study the surface morpholgy before and after the activation process of carbon structure and the pore widening impact due to the temperature. Meanwhile, from the Fourier Transform Infrared (FTIR) analysis, it was confirmed that the hemicellulose was breakdown by activation process.

The third objective of this study is to observe the best pH value of water filtration analysis, where the best temperature of activation is 600 ℃ due to the neutral pH value reading at 7. The water filtration analysis prove that the activated carbon from oil palm shell may be applied as a effiecient filtration materials and considerable potential as a cost-effective absorbent material.

5.2 Recommendations

In this study, it is recommended for the future reasearch about the pore distribution analysis on the micrograph SEM which is to study the pore size which the surface morpholgy of activated carbon. Due to the pandemic Covid-19 and the movement, the student and Assistant Engineer is limited to go inside UTeM where most of them need to work from home so the pore distribution can't be done. Futhermore, future research can used the physical and thermal activation method to perform to the other raw material. In addition, the study must be considered the sustainability and overcome the waste from other agricultural sources as well.

5.3 Sustainable Design and Development

Recent advances towards sustainable design and development rely upon positive attention to the natural. The implemantation of oil palm shell as a alternative material for activated carbon. This is due to the waste that can be applied as the raw materials in order to produce others product or material. The attention had been concentrate towards the use of green materals as raw materials. Without coincidentally, the waste oil palm shell can be activated carbon as alternative to commercial activated carbon. Thus, this study will help reducing the potential of the enviromental problems besides can be applied the waste as a product. The concept of sustainable design was varified to reduce the detrimental effects of enviromental issues.

5.4 Complexity

Many unanticipated limitations are involved in the final moments of preparation for this investigation. Initially, this study was planned to be carried out in a laboratory environment. The Malaysian government has issued a Movement Control Order (MCO) by level to citizens in response to the growing COVID-19 pandemic around the world. Due to the Movement Control Order (MCO) the experimental works finished at a longer time from the exact schedule. The final year students should dicuss with the supervisor to perform the project in the COVID-19 pandemic season.

Finally, the experimental that use the machine laboratory can proceed followed the schedule from coordinater but with the lack time of schedule and waiting list student to use the machine, the analysis not fully done. Other analysis that can be tested outside can be perfomed in the lack of tools. In this study, complex solution and the knowledge profiles have been applied through this research for example design a water filtration used to define the pH value that filtered with activated carbon.

5.5 Life Long Learning and Basic Entreprenuership

The oil palm shell which has its potential application in different fields can be a global commercial market size due to the demand of activated carbon. This is because oil palm shell can provide the cost-effective activated carbon. The wasted agricultural can be used as a raw material converted into the product such as activated carbon. Therefore, these result have studied the use of wasted agricultural give a sustainable production and have been useful in entreneurship. Meanwhile, in the recommendations section, the knowledge of lifelong learning is assessed and ideas for further growth are made.

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