



**THE DEVELOPMENT OF OIL PALM PETIOLAR
FELT-SHEATH BASED BIOSORBENT FOR
HEAVY METAL REMOVAL IN AQUACULTURE
WASTEWATER.**

by

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degree of Bachelor of Applied Science (Sustainable Science) with
Honour

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THESIS DECLARATION

I hereby declare that the work embodied in this Report is the result of the original research and has not been submitted for a higher degree to any universities or institutions.

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I certify that the Report of this final year project entitled “ The Development of Oil Palm Petiolar Felt-sheath based Biosorbent for Heavy Metal Removal in Aquaculture Wastewater” by Sangkeeta Periyasamy, matric number E15A0257 has been examined and all correction recommended by examiners have been done for the degree of Bachelor of Applied Science (Sustainable) with Honours Faculty of Earth Science, University Malaysia Kelantan.

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THE DEVELOPMENT OF OIL PALM PETIOLAR FELT-SHEATH BASED BIOSORBENT FOR HEAVY METAL REMOVAL IN AQUACULTURE WASTEWATER

ABSTRACT

Recently, aquaculture industries are showing positive growth in our country, which led to a severe retrogression in the water environment. Copper and manganese can cause bioaccumulation in the aquatic organism and creates potential danger for people who eat the contaminated fish and many environmental degradations. This research is done to utilize the waste from oil palm industry to develop into activated carbon that can used for heavy metals removal. Data involved the investigation on different dosage of activated carbon (0.0.25g and 1.0g) on varies concentration of metals (0, 1ppm and 5ppm). The research has proved that copper and manganese that can be found in aquaculture wastewater can be removed by the activated carbon that is developed from Oil Palm Petiole Felt-Sheath. The research shows that larger amount of activated carbon can remove higher concentration of metal concentration. Copper also have higher ability to be adsorbed by activated carbon compare to manganese because the copper has higher electronegativity and lower ionic radius, hence it has greater ability to be adsorbed by activated carbon compare to manganese. The findings of this paper will be useful for application in the industries of aquaculture farm to treat their waste water.

Keywords: Activated carbon; aquaculture industry; heavy metals; oil palm waste; wastewater treatment

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ABSTRAK

Baru-baru ini, industri akuakultur menunjukkan pertumbuhan positif di negara kita, yang menyebabkan kemerosotan teruk dalam persekitaran air. Tembaga dan mangan boleh menyebabkan bioakumulasi dalam organisma akuatik dan mencipta bahaya yang berpotensi bagi orang yang memakan ikan tercemar dan banyak kerosakan alam sekitar. Kajian ini dilakukan untuk menggunakan sisa dari industri kelapa sawit untuk berkembang menjadi karbon diaktifkan yang boleh digunakan untuk penyingkiran logam berat. Data melibatkan penyiasatan terhadap dos karbon yang berbeza pada pelbagai kepekatan logam. Penyelidikan telah membuktikan bahawa tembaga dan mangan yang boleh didapati dalam air kumbahan akuakultur boleh dikeluarkan oleh karbon diaktifkan yang dibangunkan dari Palm Oil Petiole Felt-Sheath. Penyelidikan menunjukkan bahawa jumlah karbon yang lebih besar dapat menghilangkan konsentrasi kepekatan logam yang lebih tinggi. Tembaga juga mempunyai keupayaan yang lebih tinggi untuk diserap oleh karbon diaktifkan berbanding dengan mangan kerana tembaga mempunyai elektronegativiti yang lebih tinggi dan radius ionik yang lebih rendah, oleh itu ia mempunyai keupayaan yang lebih besar untuk diserap oleh karbon diaktifkan berbanding dengan mangan. Penemuan kertas ini akan berguna untuk digunakan dalam industri ladang akuakultur untuk merawat air sisa mereka.

Kata kunci: Karbon diaktifkan; industri akuakultur; logam berat; sisa kelapa sawit; rawatan air kumbahan

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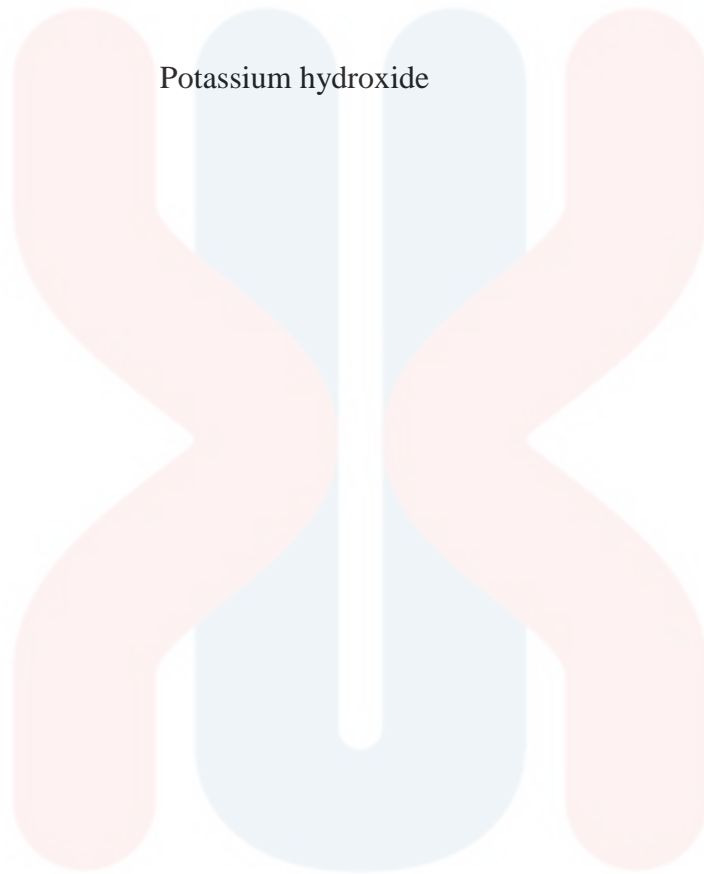
LIST OF ABBREVIATIONS

Cu	Copper
Mn	Manganese
SEM	Scanning electron microscope
OPT	Oil palm tree
XRF	X-ray fluorescence
FTIR	Fourier transform infrared spectroscopy
AC	Activated carbon
FFB	Fresh fruit bunch
OPF	Oil palm frond
EFB	Empty fruit bunch
OPW	Oil palm wood
MDF	Medium density fibre board
ICP-OES	Inductively Coupled Plasma Optical Emission Spectroscopy
HCl	Hydrochloric acid
HNO ₃	Nitric acid
H ₂ O ₂	Hydrogen peroxide
ZnCl ₂	Zinc chloride

KMnO_4 Potassium permanganate

$(\text{NH}_4)_2 \text{S}_2\text{O}_8$ Ammonium persulphate

KOH Potassium hydroxide



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LIST OF SYMBOLS

mg	milligram
ha	hectare
nm	nanometer
°C	temperature
kg	kilogram
mm	millimeter
μL	microliter
g	gram
ml	milliliter
mA	milliampere

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CHAPTER 1

1.0 INTRODUCTION

1.1 Background of Study

Aquaculture industries are recently show accelerating development in our country has led to a severe retrogression in the water environment (Farmaki et al., 2014). Many types of heavy metals usually can be found in aquaculture wastewater, such as copper, silver, manganese and cadmium. Copper is highly used in aquaculture field to exterminate algae, mold and shellfish shows there is very high harmfulness to aquatic organisms. Copper is one of the most harmful metals to aquatic organisms and ecosystem. This is why pollution free excavation performances are very vital. Sediments and organic matter easily binds with copper as it is fairly dissolvable in water and binds easily to sediments and organic matter. Bio concentration, defines the amount of copper in living bodies is higher than in the habitat as aquatic bodies or sediments. The animals found in the sediments at the bottom of a water body specifically have larger amount of copper as in mollusc, such as oysters, that can percolate foreign particles from tremendous mass of water (F Solomon, 2009). Heavy

metals which exist in a small amount of concentration (1–100 mg) in land and wastewater is verified to be feeble or overpriced to be removed by conventional stated by (Brinza et al., 2009). Even at very low concentrations, it's able to be harmful to the living organism including human and to ecological well-being for their dangerous effects, prolongation and accretion tendency (Yang et al., 2015).

There is some elimination method to remove this harmful heavy metals from water bodies. Activated carbons are broadly utilized as adsorbents for the evacuation of natural synthetics and metal particles of ecological or financial concern from the air, gases, drinking water and sewage (Qodah and Shawabkah, 2009). There have been quite a number of researchers that has been done to utilize agricultural waste as activated carbon by physical and chemical activation technique (Tham et al., 2010). Few researchers' expertise in this came up with trials of oil palm wood pyrolyzed into charcoal but there are difficulties while ruling the transformation adequacy together with pyrolysis rate as the crumbly character of the wood causes a low calorific value to obtain. Hence, oil palm wood charcoal is inappropriate to innovate as charcoal fuel (Ahmad et al., 2007). In Malaysia, palm trees are used widely for industrial and economic purposes. There are abundance of palm petiolar felt-sheath (PPF) as a waste material from palm trees, while it also contains the capability of removing heavy metals as copper and manganese from waste water. This entwined network of palm petiolar felt-sheath (PPF) as a new biosorbent is investigated.

Oil palm tree (*Elaeis guineensis* jacq.) is developed as an agricultural crop, it is usually can be found in the wild. In Malaysia, there are land under oil palm development about 4.49 million now where 17.73 million tons of palm oil and 2.13 tons of palm part oil are still developing. One of the largest originator and merchant of palm oil on the world is Malaysia which is 11% of the world's oils and fats production and 27% of the fare entrepreneur of oils and fats. It provides employment for superior part of a citizens to an anticipated one million people (Alriols et al., 2009).

In terms of start-up cost, secondary harmful matter and ease of performance, the advantages of adsorbent are outstanding. Optimization problems is from biodegradation while other method as ion exchange and chemical oxidation are considered as expensive technique. Electrochemical procedure has three leading issues. Primarily, exorbitant and inconsistent anodic materials. Secondly, the presence of high chloride concentration causes the effluent cannot be discharged in and thirdly low columbic efficiency. Additionally, floatation coagulation techniques have a low efficiency. There are further complex procedures that are high cost or unable to bear with large amount of contaminants as solvent extraction, bio-sorption, and ultra-filtration. There are significance cons from these techniques such as unfinished ion dismissal, large energy needs, and production of toxic sludge or other waste products that require further disposal. Activated carbon is being used for a long period for this purpose compare to other adsorbents application. Highly developed porosity, large

surface area, variable characteristics of surface chemistry, and high degree of surface reactivity makes activated carbons known as very effective adsorbents. The treatment of wastewater and polluted groundwater utilizing AC is expanding all through the world because of the constrained wellsprings of water supply (Eapen, S.J. et al., 2016). Part of environmental problems can be reduced instantaneously by converting the by-product into a capital for another industry is possible is concluded. Oil Palm Trees trunk are residues from shaved fronds in the plantation area are eliminated during reaping while the trunk and stipules are eliminated at reseeded operation.

Characterization of activated carbon in which very important in order to classify active carbon for specific uses. Normally, active carbon is characterized by physical properties and chemical properties. The characteristics of activated carbon depend on the physical and chemical properties of the raw materials as well as activation method used for physical property consist of ash content and moisture content. For the chemical property of active carbon, it involves the surface area of the activated carbon and also elemental analysis (Jabit, 2007). Characterization of active carbon can be done straight by measuring the pores by using images from the scanning electron microscope (SEM).

1.2 Problem Statement

Freshwater fish are widely available in Malaysia and there are many people enjoy eating this type of fish without considering the source of the aquatic fish. However, the fish they consume daily may contain harmful heavy metals such as manganese and copper.

Aquaculture wastewater contains these heavy metals from the food compounds the owner of aquaculture feed for the fishes. This heavy metals can cause bioaccumulation in the aquatic organism and creates potential danger for people who eat the contaminated fish.

While other heavy metal as copper and manganese can cause negative impacts to human who consume it, aquatic life and the environment around it. This heavy metal also can cause many environmental degradations. Thus, active carbon development can add value to PPF as it will be used to remove those harmful copper and manganese. Furthermore, it can be done in low cost as PPF can be found in abundance as agricultural waste annually.

1.3 Objective

The aim of this research is to develop and to study the performance of Palm Petiolar Felt-Sheath (PPF) as activated carbon. The study is carried out with the following objectives:

- To develop active carbon from Oil Palm Petiole Felt-Sheath
- To determine the characteristics of PPF
- To remove heavy metals from aquaculture wastewater.

1.4 Scope of Study

There are few parameters to take in count in this research, firstly it will be the development of activated carbon from Oil Palm Petiole Felt-Sheath. The challenge is the limited time given. PPF as active carbon test on removing heavy metal from aquaculture wastewater with different concentration and dosage. There are only a few studies done on PPF active carbon which limits the research.

Next, the removal of heavy metal from aquaculture water can be limited by its optimum condition as time range, temperature, pH, the volume and dosage used. At optimum condition maybe the adsorption can be 100%

The characterization of PPF active carbon will be done by using Scanning Electron Microscope.

1.5 Significant of Study

Only a few types of research have been done to develop PPF as active carbon to remove heavy metals. Therefore, this study is conducted to develop active carbon from PPF and to remove the heavy metal cadmium from aquaculture wastewater by using AC. Thus, the result obtains will be used for further study and to reduce the abundance of waste produced in agricultural activity. By that the impact of copper and manganese on aquaculture environment also can be reduced. Not only that, if the performance gives out best result it can be implemented in real aquaculture and agriculture industries with large volumes. There should be right application method of activated carbon in wastewater treatment field.

CHAPTER 2

2.0 LITERATURE REVIEW

2.1 Aquaculture Waste Water

Aquaculture defines to the aged performance of fish breeding and the growing of aquatic crops in ponds fertilized with wastewater and excreta is a common practice, particularly in Asia. There are more than 130 wastewater fertilized fish pond systems in India, covering an area of some 12 000 ha; most are located in West Bengal (World Health Organization,1989).

Aquaculture systems needs treatment and disposition as it generates expansive amounts of organic matter and nutrients example as nitrogen, phosphorus and other elements. As metabolic wastes of 1 ton of live channel catfish production, discharges 1190 kg of dry matter, 60 kg of nitrogen and 12 kg of phosphorus to the culture water. Aquaculture wastewaters apply variety of environmental effects especially when the outflow from these systems was released to water bodies. These organic matter play roles in formation of sediments as dissolved oxygen levels reduces at the bottom. While water quality will be damaged by stimulating excessive phytoplankton production with high nutrient loadings (Ghaly, Kamal, & Mahmoud, 2004).

2.2 HEAVY METALS IN AQUACULTURE WASTEWATER

2.2.1 Copper (Cu) and its Effect

Every living organism need different levels (5-20 micrograms for each gram ($\mu\text{g/g}$)) of copper by humans, different vertebrates, fish and shellfish for sugar digestion and there are more than 30 compounds working together. Copper is fundamental for haemoglobin and haemocyanin development, it is independently the oxygen-transporting shades in the blood of vertebrates and shellfish. Copper can be dangerous if more than surpass 20 micrograms for every gram ($\mu\text{g/g}$) as stated in a article by Solomon, F. (2009). Anaemia, renal and intestinal irritations, coma, death, and Wilson's disease can be caused by high level of copper accumulation in liver and kidneys (Alomary et al., 2007).

Saw dust also can be utilized in removing copper metal ions, whereas other agriculture by products materials have proved can be used for sequestering copper metal in high efficiency like wheat shells and carbonized coir pith. Other substantiate as possible biosorbent are mustard oil cakes and modified bark of pinus radiate (Sud, D et al., 2008).

2.2.2 Manganese (Mn) and its Effect

Manganese made up of many rock types, especially those of metamorphic and sedimentary origin, about 0.085% to 0.095% of the earth's crust. Predominant ores of manganese include pyrosulite (MnO_2), manganite ($\text{Mn}_2\text{O}_3 \cdot \text{H}_2\text{O}$), hausmannite (Mn_3O_4), psilomelane and rhodochrosite (MnCO_3). Manganese is affiliated with iron ores of sub marginal concentration (Samuel, 1999).

Manganese is a product of momentous natural sources soils, sediments, metamorphic and sedimentary rocks. In the pedogenesis process, weathering of rock results in soil containing manganese. On the sea bed, huge quantity of manganese and manganese-rich nodules have been found along with cobalt, nickel and copper. Ferromanganese minerals for instance biotite mica and amphiboles contain this properties (Samuel, 1999).

Manganese may dissolve in ground and surface water. It happens because manganese presence naturally in some component of environment. For abolition it might corrode and precipitate as sediment, with the subsequent ability. In plant material it usually allows a source for annihilation as accumulation in the course of decompose. The ability to dissolve of manganese will get high at a low pH, as 2^+ and 4^+ oxidation states in aquatic systems. While under reducing conditions, manganese solubility increases as chlorides, nitrates and sulphates concentrations goes up. It rises both aqueous agility and intake by plants.

Manganese precipitates out residue and re-solubilizes in the water segment successively as Mn^{4+} and Mn^{2+} . Dissolved concentrations of manganese in characteristic waters that are basically free of anthropogenic sources 10 mg/L. In native surface waters, manganese concentrations sometimes can range 1.0 mg/L and less than 0.2 mg/L, while nearly 2 μ g/L of manganese in seawater (Samuel, 1999).

Table 2.1: Shows epitome of research for heavy metal removal by various researchers utilizing variety of agricultural waste materials (Sud,D ,Mahajan.G , MP Kaur. ,2008).

Agricultural waste	Metal Ion	Results (%)	Reference
Papaya Wood	Cd(II), Cu(II), Zn (II)	98, 95, 67	Saeed et al. (2005a)
Hazelnut shell, orange peel, maize cob, peanut hulls, soybean hulls treated with NaOH & jack fruits	Cd (II), Cr (VI), Cu (II), Ni (II), Zn (II)	High metal adsorption	Kurniawan et al. (2006)
Rice bran	Pb (II), Cd(II), Cu(II), Zn (II)	>80	Montanher et al. (2005)
Maple saw dust	Pb (II), Cu (II)	80–90	Yu et al. (2001)

		60– 90	
Water hyacinth	Pb (II), Cu(II), Co(II), Zn (II)	70– 80	Kambl e and Patil (2001)
Activated carbon from coir pith	Cd(II), Cu (II)	Cd- 100 Cu- 73	Kadirv elu et al. (2001)
Rice straw, soybean hulls, sugarcane bagasse, peanut shells, pecan and walnut shells	Pb(II), Cu(II), Cd(II), Zn(II), Ni (II)	Pb >Cu >Cd >Zn > Ni	Johns et al. (1998)
PFP (petiolar felt sheath palm)- peelings from trunk of palm tree	Pb(II), Cd(II), Cu(II), Zn(II), Ni(II), Cr(VI)	>70 Pb >Cd >Cu >Zn > Ni > Cr	Iqbal et al. (2002)

Activated carbon of peanut shells	Pb (II), Cd(II), Cu(II),	>75%	Wilson et al. (2006)
Bark of Abies sachalinensis & Pecia glehnii	Cd(II), Cu(II), Zn(II), Ag(II), Mn(II) Ni (II)	>63%	Seki et al. (1997)
Mustard oil cake	Ni (II), Cu(II), Zn(II), Cd(II), Mn(II)	>94%	Ajmal et al. (2005)

2.3 Oil Palm Tree

In tropical country as Malaysia and Indonesia, Species of *Elaeis guineensis* under the family *Palmaceae* initiated in the tropical forests of West Africa. During British ruled Malaysia in early 1870s they introduced it for decorative purposes. By 1917 at Tennamaran Estate in Selangor this plantation developed for commercialization. A programme by Government as agricultural diversification increased the refinement of oil palm at a high speed have helped country's economic dependence on rubber and tin (Godswill et al., 2016).

Fresh Fruit Bunch (FFB) is the each ripen bunch. There are mainly palm oil trees variety of tenera in Malaysia, it is a hybrid. The hybrid been done betwixt the dura and pisifera. Every year, each hectare produces about 4 to 5 tonnes of crude palm oil (CPO) while 1 tonne of palm kernels produced the tenera variety. In the world the most efficient oil-bearing crop is oil palm. It only requires 0.26 hectares of land to produce one tonne of oil while soybean, sunflower and rapeseed require 2.22, 2 and 1.52 hectares, respectively, to produce the same (Cochard et al., 2009).

Oil palm can be both gender in same tree so it is known as a monoecious crop. Per bunch there are almost 1000 to 3000 fruitlets and weighs between 10 and 25 kilograms by each tree. The shape of each fruitlet was mostly spherical or elongated in shape. Usually the fruitlet will be dark purple, almost black and the colour turns to orange red when ripe. The fruitlet contains of a hard kernel (seed) surrounded in a shell (endocarp) which is enveloped by a fleshy mesocarp (Godswill et al., 2016)

Palm trees height can be up to sixty feet and more. The trunks usually give a rough appearance as young and mature trees are wrapped in fronds. The fronds which have withered and fallen off left scars in the older tree as the trunk usually smoother.

Palm oil is produced the most in the world as the production of palm oil is 39 million tonne per year in 2007. In universal, by 2.5 years after planted the palm will begin bearing oil-contained fruits, while its productivity will reduce small after 20 to 25 years. So it is necessary to cut the old palm trees and to replant new seedlings at plantation sites. In consequence, in our country Malaysia about 120,000 ha of oil palm tree is predicted to be replanted yearly from 2006 to 2010 for continuity of the oil

productivity as. To maintain oil productivity in our country, about 120,000 ha of oil palm is estimated to be replanted annually from 2006 to 2010 (Godswill et al., 2016)

Waste production in high amount inclining in oil palm plantation in Malaysia especially at replanting process oil palm petiole felt-sheath (PPF) and oil palm trunk (OPT), as PPF consist of 70% of overall oil palm waste. Empty fruit bunch (EFB) will be acquired after the of oil seeds are removed from fruit bunch to extract the oil. Recently, the waste from oil palm by-product were not effectively managed, this leads to gigantic quantity of vegetable waste, creating difficulties in replanting operations as it drives to tremendous environmental concerns. Only Malaysia have produces around 30 million tons annually of oil palm biomass, including trunks, fronds, and empty fruit bunches. Consequently, it will be advantageous for economic utilization of these fibers (Abdul Khalil et al., 2008).

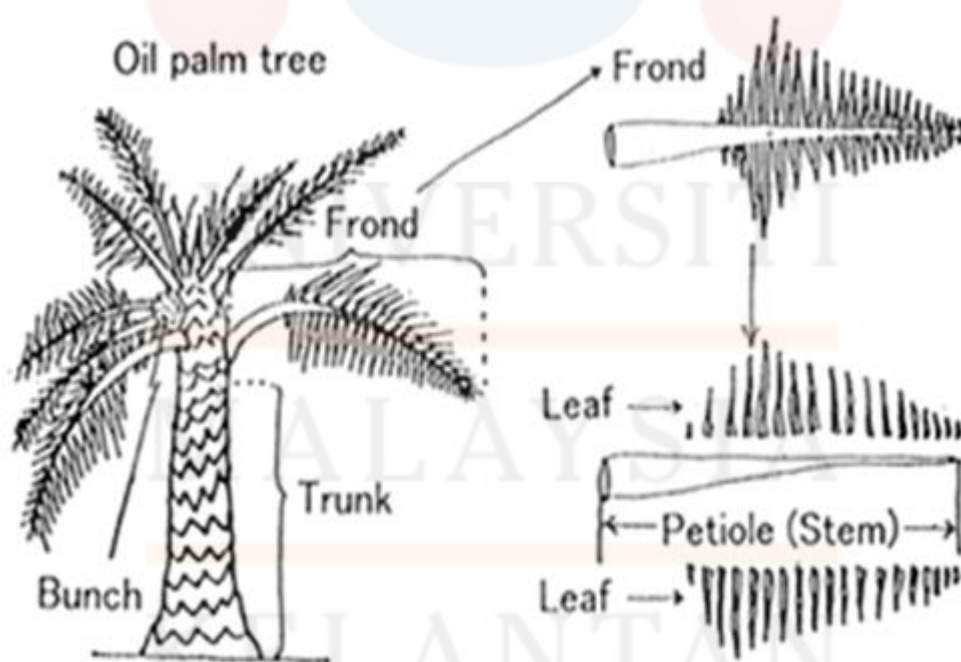
Usually the waste generated from oil palm trunks were left to rot, burnt off in stacks or used as mulch. Since the waste in high scale are produced from PPF, there are novitiate of few investigations to convert oil palm frond (PPF) to value added products. Therefore, economical approach to take advantage of oil palm felt-sheath for ideal oil palm plantation and sustainable palm oil industry. (Yamada et al.,2010).

While it also contains the capability of removing heavy metals from aquaculture waste water. Those waste materials can give beneficial outcome to the environment with proper modification as it also helps to reduce the waste production in agro based industry. The foliage of the palm tree is the source of these fibers, in particular from the leaf sheath. The leaf is prorated into certain components: the blade and the leaf

axis, the latter is itself branched into a sheath surround the stem, leafstalk and rachis bearing leaflets (Abdullah, Madon, Low, Ithnin, & Singh, 2012).

OPT also can be used as production of a variety of composite materials, such as cement-bonded particle boards, moulded table tops, medium density fibre boards (MDF), glucose production, as ruminant or animal feed and activated carbons as it contains lignocellulosic-rich materials (Hussein et al.,2001). A number of agricultural waste materials are being studied for the removal of different heavy metals from aquaculture waste water at different operating conditions.

Figure 2.1: Anatomy of an oil palm tree and oil palm frond. (Source: Ishida and Abu Hasan, 1997).



2.4 Activated carbon

Activated Carbon (AC) is the usual term practiced for carbon with many large pores of different sizes as micro, macro and mesoporous at the internal of carbon that adsorb substances. It also in crystalline form that make the carbon more adsorbent. The term of activated carbon is discovered from the word “carbon” and” active” which carbon mean raw material undergoes a carbonization process. Carbonization process undergoes activation process to enlarge a pore surface area as maximum to increase the adsorption rate of activated carbon by burning the material in in high temperature flame to active a material in carbon condition (Jamaludin, 2010).

Activated carbon(AC) was prominent as a solid, porous black carbonaceous material and tasteless (Jamaludin, 2010). Activated carbon has the benefits of providing an effective low cost substitute for non-renewable coal by acquired from agricultural by-products based granular activated carbon (GACs) provided that they have similar or better adsorption efficiency (Sugumaran et al., 2012).

ACs is carbons of extremely tiny penetrable form with both large surface area and porousness (Loannidou and Zabaniotou, 2006). The large surface infers a high limit with respect to adsorbing synthetic substances gases and liquids (Dinesh, 2010). The most generally utilized commercial active carbons have a particular surface area shifting from 800 to 1500 m²/g, as determined typically by nitrogen gas adsorption (BET surface area) (Loannidou and Zabaniotou, 2006). The adsorption capability for molecules of different shapes and sizes, as the distinction in pore measure influences the adsorption limit and consequently is one of the criteria by which carbons are chosen

for a particular application. Porosity is classified by IUPAC into three different groups of pore sizes (Dinesh, 2010).

Table 2.2: Shows pore size of Activated Carbon

Types of pores	Description
Macropores	Pores with diameters larger than 50 nm
Mesopores	Pores with diameters between 2 nm and 50 nm
Microspores	Pores with diameters less than 2 nm

2.5 Preparation of active carbon

Carbon produced from carbonaceous source materials called activated carbon for an instance nutshell, peat, wood, coir, lignite, coal and petroleum pitch. Physical activation or chemical activation method is being used to produce it (Pradhan, 2011).

The development of porosity is diverse in both methods, in terms of realistic procedures and mechanism (Baseri et al, 2012). Carbonizing and activating the carbonaceous precursor material is the general process to generate activated carbon is founded on (EkPete and Horsfall, 2011).

2.5.1 Physical Activation

By physical activation gases are applied in the process precursor to develop activated carbons. Carbonization and activation generally combined to done the following processes. First in the absence of oxygen usually inert atmosphere as argon or nitrogen at temperature ranging between 600-900°C, the material with carbon content is pyrolyzed. Then followed by activation/oxidation where in this process raw material or carbonizes matters are exposed with oxidizing atmospheres as carbon monoxide, oxygen or steam. This temperature usually exceeds 250°C, in the temperature range of 600-1200°C (Pradhan,2011). The activation gas is usually CO₂, since it is clean, easy to handle and it facilitates control of the activation process due to the moderate process rate at temperatures around 800°C (Zhang *et al.*,2004).

2.5.2. Chemical activation

The carbon is reacted with a dehydrating agent at high temperatures that removes the majority of hydrogen and oxygen from it by chemical activation. The carbonization and activation step often combines to form chemical activation, but usually this way happens separately based on the process (Leimkuehler,2010). Chemical activation strategies include the response between the carbon surface and arrangements of oxidizing agents. There are few oxidizing agents such as phosphoric acid H₃PO₄, nitric acid HNO₃, hydrogen peroxide H₂O₂, zinc chloride ZnCl₂, potassium permanganate KMnO₄, ammonium persulphate (NH₄)₂ S₂O₈, potassium hydroxide KOH, etc. (Al-Qodah and Shabawabkah,2009). Usually for lignocellulosic materials activation,

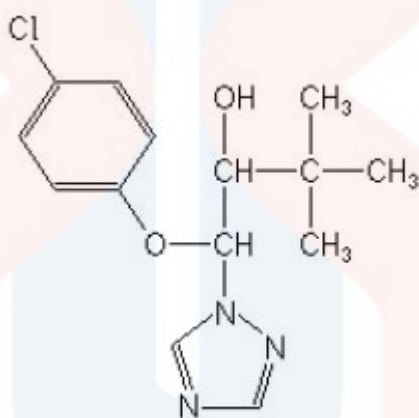
phosphoric acid and zinc chloride used as oxidizing agents, (Al-Qodah and Shabawabkah,2009). The effectiveness of such oxidizing agent is in the following order: $\text{H}_3\text{PO}_4 > \text{H}_2\text{SO}_4 > \text{HCl} > \text{HNO}_3$.

There are also finding that shows that microporous activated carbons prepared at low temperatures while by-product of larger surface areas are from higher temperatures, will result to mesoporosities (Guo, J., and Lua, A. C., 2000). In a single operation, a carbonized product with very good sorption properties can be produced by chemical activation. In physical activation, the temperature required for pyrolysis is likewise lower than that required for activation with gaseous agents and this advances the improvement of a porous structure (Yagsi, 2004).

2.6 Types of Activated carbon

Classification is done based upon their surface characteristics, behavior and preparation methods.

Figure 2.2: Image shows chemical structure of active carbon (Al-Qodah & Shawabkah, 2009)



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Table 2.3: The types of activated carbon that can be produced with descriptions.

TYPES OF ACTIVATED CARBON	EXPLANATION
Powdered Activated Carbon (PAC)	Fine granules whose measure is under 1.00 mm. Their normal pore width is between 0.15 to 0.25 mm. It is readied by finely ground crude material. In any case, their principle property is the nearness of a high surface region to volume ratio (Dinesh,2010).
Granular Activated Carbon (GAC)	Larger molecule estimate contrasted with powdered activated carbon and thus, shows minimum external surface. Dispersion of the adsorbates is consequently a critical factor. These carbons are in this manner favored for all adsorption of gases and vapors as their rate of dispersion are faster (Pradhan,2011).
Extruded Activated Carbon (EAC)	Powdered activated carbon with a fastener, which are bounded and expelled into a barrel shaped formed activated carbon bar with diameter from 0.8 to 130 mm. These are basically utilized for gas stage applications as a result of their low pressure drop, high mechanical quality and low residue content (Pradhan,2011).
Impregnated Carbons	Inorganic metals are impregnated upon them for explicit applications identified with air contamination control. This sort of activated carbon additionally discovers use in water cleansing procedures when impregnated with explicit sorts of antimicrobial/sterile agents (Dinesh, 2010)
Polymer Coated Carbon	This is a procedure by which a permeable carbon can be covered with a biocompatible polymer to give a smooth and porous coat without obstructing the pores. The subsequent carbon can be utilizing for hemoperfusion. Hemoperfusion is a treatment system in which substantial volumes of the patient's blood are disregarded an adsorbent substance so as to expel poisonous substances from the blood (Pradhan,2011).
Pellet Activated Carbon	High pressure tasks and high volumetric adsorption is needed. Pellet is like based on these activated structures are conservative, hard and have a high surface region for adsorption. Generally, pellet activated carbons have a length of 20 mm to 40 mm and a width of 5 to 10 mm (Dinesh, 2010).

2.6 Characterization and Properties

To classify and determine the specific purposes of activated carbon, characterization is very important. The activation method plays crucial role in determining the characteristics of activated carbon based on the physical and chemical properties of the raw materials while physical property consist of ash content and moisture content. For chemical property of active carbon involves surface area of activated carbon (Jabit, 2007).

2.6.1 Moisture Content

Activated carbon is for the most part evaluated on a dampness free basis, albeit infrequently some dampness content is stipulate. Except if stuffed in water and air proof holders, some activated carbons when put away under muggy conditions will retain significant dampness over a time of month. Activated carbon can still appear dry even after adsorbing 25 to 30% dampness. There are some reasons why this dampness content does not influence the adsorptive power, but it clearly weakens the carbon. Along these lines, an extra weight of wet carbon is expected to provide the required dry weight. (Jabit, 2007).

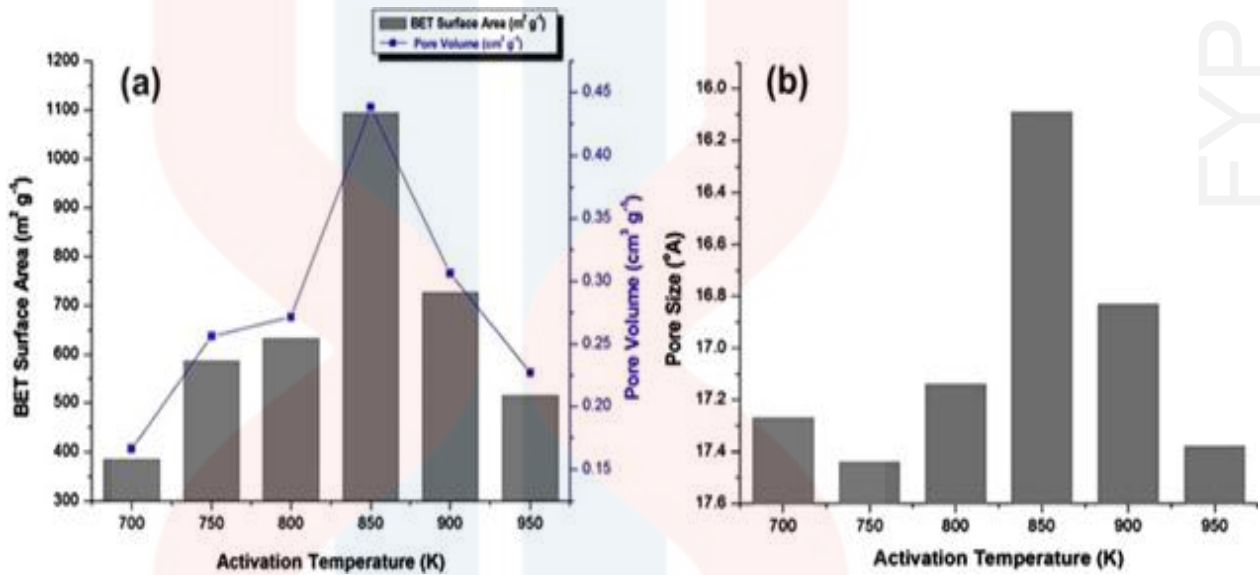
2.6.2 Ash Content

The ash content of a carbon is the remaining parts when the carbonaceous materials is scorched off. There will be difference from carbon grade by the total sum of inorganic constituents. It happens as activated carbon contain inorganic constituents originated from the source materials and from activating agents included while in the production (Jabit, 2007).

2.6.3 Surface Area

The BET surface area of active carbon is important because, like other physical-chemical characteristics, it may strongly affect the reactivity and combustion behavior of the active carbon from pyrolysis above 400°C had a surface area and a high surface area formed (Putun et al.,2005)

Figure 2.3: Effects of activation temperature on (a) surface area and pore volume and (b) pore size.
(Shoaib .M et al., 2015)



2.6.4 Pore Size and Volume

Both the size and dispersion of microspores, mesospores and macrospores fix the adsorptive characteristics of activated carbons. For example, little pore size will not be able to trap large particles while extensive pores will be unable to hold little adsorbates, regardless of whether they are charged, polar atoms or uncharged, non-polar compounds (Ahmedna et al.,2000).

A greater content of lignin material can develop ACs with macro porous structure, where else microporous structure primarily can be yield in ACs from raw materials with a higher content of cellulose (Savova et al.,2001). The surface and pore contexture will be noticed with scanning electron microscope (SEM) (Zhang et al.,2007).

The SEM can relinquish information about the topography as the surface features of an object, morphology is the shape and size of the particles form the surface of an object, the elements that the object is composed and the relative amounts of composition and crystallographic information like the arrangement of atoms are in the object(Dinesh,2010).

SEM it has been utilized in numerous territories of science and industry, especially in materials designing, organic and therapeutic sciences for the examination and portrayal of the smaller scale structure of substances since it has enormous amplifications and amazing achievable resolutions.

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Figure 2.4: Scanning electron micrographs (SEM) of the activated carbons prepared

from

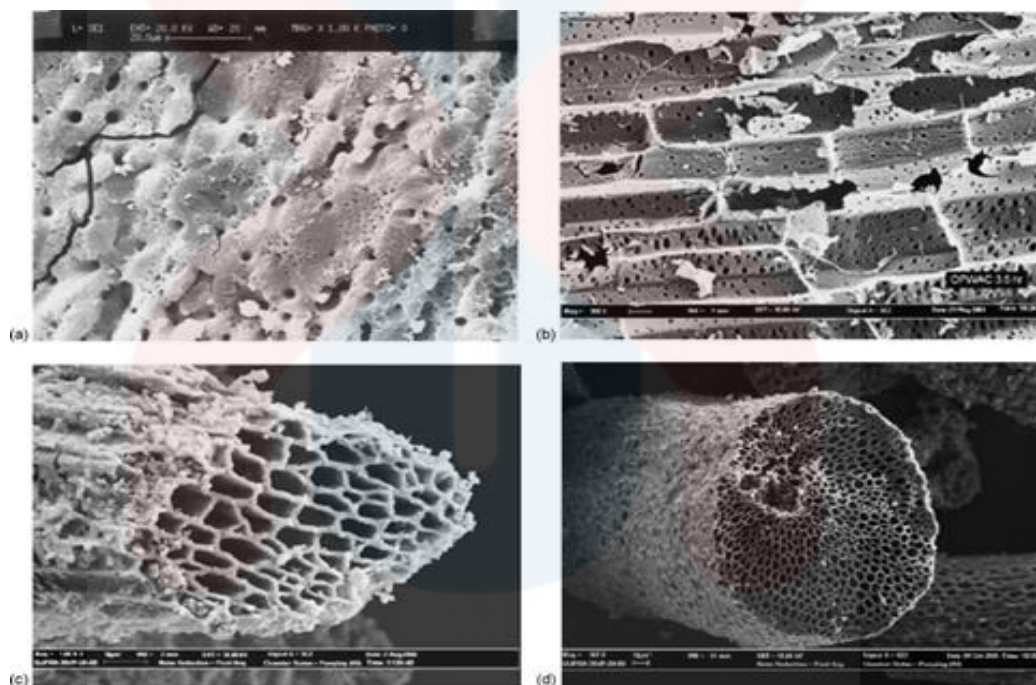
a) raw palm stone (1000×)

c) oil palm shell (1000×)

b) oil palm wood (500×)

d) oil palm fiber (362×)

(Foo K et al., 2009)



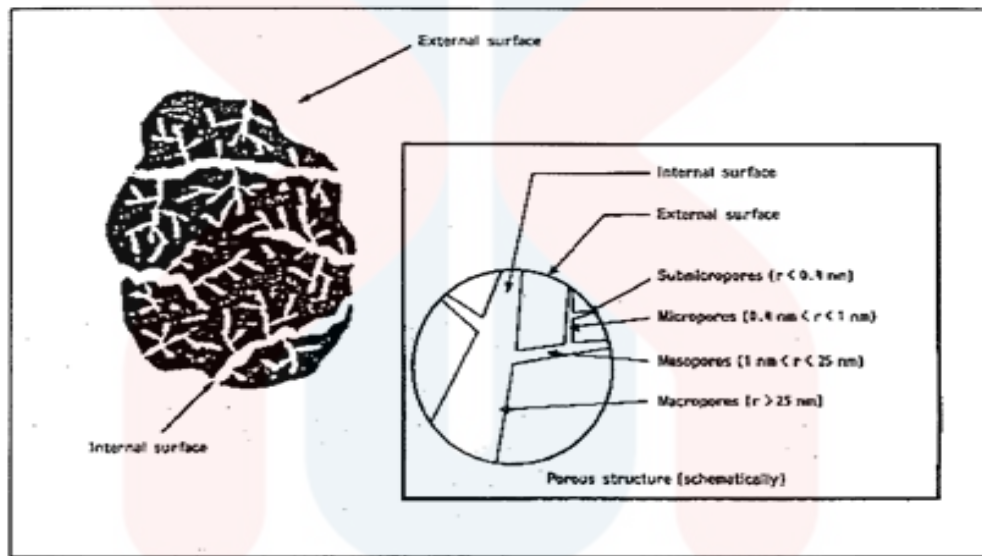
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SEM image of palm oil fronds activated carbon prepared under optimum conditions

(magnification = 1000×)

(Salman, 2014).

Figure 2.5: Shows the schematic of activated carbon model (Jamaludin, S. R., 2010)



2.7 Activated Carbon in Heavy Metal Adsorption

2.7.1 Oil Palm Trunk as AC

There are few studies have done based palm oil tree trunk and oil palm shell have shown it has great potential as a raw material for activated carbon production. The surface properties of the resulting activated carbons can be tailor-made or enhanced, by controlling the activation process. The surface area of the resulting activated carbon strongly depended on the amount of the activator, zinc chloride. The preparation of activated carbon from chips of oil palm trunk (COPT) was activated by using phosphoric acid coupled with carbon dioxide as the chemical and physical activation agent, respectively. Synergetic effect of both activators produced high surface area activated carbon. This property can enhance the absorption properties of the resulting materials (Hussein et al., 2001).

Another study done based oil palm biomass as precursor as cellulose is insoluble in most solvents and has a low accessibility to acid and enzymatic hydrolysis. Oil palm biomass is now considered to be one of the most promising non wood lignocellulosic raw materials as precursor for the production of activated carbon. Chemical modification of cellulose is a promising technique for modifying its physical and chemical properties to improve the adsorption property toward removal of various pollutants (Rafatullah, 2013).

2.7.2 Palm Petiole Felt-Sheath(PPF) as AC

PPF was tested for its biosorption level with heavy metals such as Pb(II), Ni(II), Cd(II), Cr(III) and Zn(II). The efficient removal of all the heavy metals ions with selectivity order of $Pb > Cd > Cu > Zn > Ni > Cr$. There are few factors that influence the capability of PPF adsorption such as pH, metal ion concentration and dosage of biosorbent.(Shoaib & Al-Swaidan, 2015).

PPF remained efficient even after the metal ion were recovered after the adsorption-desorption cycles. Current investigations propose that PPF contain the ability to be operated in the removal elimination/retrieval of heavy metal ions from aqueous solutions. Other than that, physiochemical activation method also can have used to develop activated carbon by potassium hydroxide (KOH) treatment and carbon dioxide (CO₂) gasification. The impact of activated carbon on pesticides are tested by variable parameters. Those parameters were activation temperature, activation time and chemical impregnation ratios (KOH: char by weight) also on the preparation of the activated carbon(Salman, 2014).

CHAPTER 3

3.0 MATERIALS AND APPARATUS

All chemicals and reagents were analytically graded. There are list of chemicals and reagents used in this experiment. Firstly, concentrated hydrochloric acid is used for destroying all the foreign particles. This acid also used to clean the pile of impurities from the exterior of activated carbon. Other than that, it introduces the hydrophilic functional group as acid and base that enhances the presence of surface chemistry. This acid also enlarged the entrance of the pore which will increase the surface area. Lastly, distilled water were used to clean the prepared activated carbon a few times until it reaches to neutral state, the reading of pH is taken by using pH meter.

The apparatus that used in the laboratory in this present study is handled and measured carefully to obtain the actual and precise results. Hot plate is function to heat up the distilled water that used to wash the activated carbon until the neutral ph.

Next is muffle furnace is an extremely heated chamber, walls of which can radiantly heat the content which is kept inside so that material cannot have direct contact with the flame. These furnaces are often utilized in the testing laboratories as a means to create extremely high-temperature inside the chamber. These instruments are utilized to measure the characteristics of the materials at extremely accurate and high temperatures. These furnaces are also known as retort furnaces. It is capable of creating high-temperature as it is equipped with a high-temperature heating coil in a glass wool insulated material. This material acts as muffle and prevents the heat from escaping.

Beaker (500 mL) to transfer acid and function as temporary storage container. Crucible is a pot that is utilized to keep metals for liquefying in a furnace. Crucibles are intended to withstand the most astounding temperatures experienced in the metal casting works. The crucible materials ought to likewise have great quality notwithstanding when to a great degree hot. Variety of metal constructions used for furnace crucibles. These materials can oppose the outrageous temperatures in run of the factory foundry activities.

Desiccator have desiccants function to preserve moisture-sensitive, it is presence with sealable enclosures. They are used to store safely the chemicals which are hygroscopic or which react with water from humidity. Weighing balance to weigh each sample before and after a process is done. Aluminium arranged in thin metal leaves with a thickness under 0.2 mm. The foil is flexible, and can be promptly bowed or folded over items.

Spatula (metal) will be used to transfer particular amount of solid around the place near to it. Filter paper will be used during washing process of the prepared activated carbon. Funnel is used to channel liquid or small-grained substances into containers with a small opening, used to avoid spillage.

Vacuum pump will be used together with büchner filtration apparatus. And büchner filtration apparatus will remove foreign unwanted compound from liquid. The mixture of the liquid and solid is passed through the filter, with a piece of filter paper in the funnel role as filtering agent.

Before using büchner funnel, make sure the filter paper ought to be soaked with a dissolvable preceding use. Initial leakage can be prevented by this way. Furthermore, the pouring liquid which is to be shifted into another holder is constrained through a

suction of vacuum. Make sure that the solvent does not surpass the important condition, else, the additional also would get sucked into the vacuum suction.

The concentration level of heavy metals can be determined by the instrument Inductively Coupled Plasma-Optical Emission Spectroscopy (ICP-OES) is function in atomic spectroscopy. The sample is decomposed by intense heat into a cloud of hot gases during analysis. These hot gases contain free atoms and ions of the elements of interest.

The high temperatures cause significant amounts of collisional excitation and ionization of the sample atoms. At the point when the particles achieved their energized state, they can rot to bring down states through warm or radiative (outflow) energy transitions.

During ICP-OES analysis the power of the light radiated at explicit wavelengths is measured and utilize to study the concentration of the element(s) of interest. The thermal excitation sources able to populate a large number of different energy levels for several different elements at definite time. All of the excited atoms and ions able to explicit their characteristic radiation at the same time. This phenomenon will result in the flexibility to select from few different emissions concurrently and allows determination of multiple elements concurrently

Finally, characterization done by scanning electron microscope (SEM). It is a type of electron microscope that images a sample by scanning it with a high-energy beam of electrons in a raster scan pattern. The electrons cooperate with the atoms that build up the sample releasing signals that have information about the sample's surface topography, composition, and other properties such as electrical conductivity (Dinesh,2010).

3.1 METHODOLOGY

3.1.1 SAMPLE COLLECTION AND PREPARATION OF ACTIVE CARBON

FROM PPF

Materials and Apparatus

Oil palm frond chips, blender, oven(binder), sieve (1mm)

The oil palm frond is obtained from the palm oil trees near PSU campus Suratthani. The reticulated fibrous network of petiole felt-sheath of palm was obtained as peelings from the trunk of palm tree *Livistona chinensis* (Iqbal and Zafar, 1997). The felt-sheaths were washed extensively with distilled deionized water to remove dirt and other particulate matter.

The sample was first air-dry for several days, followed by oven drying at 100°C for a day (24 hours) to remove excess moisture. The sample was burned for 450°C (8 hours) for pre-carbonization. Pre-carbonization is intended to develop micropores. About 800 g of the charcoal was weighed and placed in a large container for further process.

3.1.2 DIGESTION OF PPF

Materials and Apparatus

The first step of the activated carbon generation process is by digestion of the 800g of charcoal with 2.5L hydrochloric acid to destroy the foreign particles under a vacuum condition. The sample were left for overnight in the vacuum chamber. The following days, the sample were left to dry in hood until it is completely dried. Record the weight of the sample again for every 6 hours until the weight does not change to make sure there is no moisture left.

3.2 Pyrolysis

Materials and Apparatus

Aluminium foil, spatula, muffle furnace

After that, crucibles were half filled with active carbon and placed into a claypot and occupied with dried sand, to make sure there is no air pass through. The prepared sample was located in furnace for 800⁰C (8 hours) for carbonization. During the process primary pyrolysis gases are produced, which separate into permanent gases and oils (tars) if they are cooled to ambient temperatures.

The residue of the pyrolysis process is the primary carbon, which serves as base material for the activation step. There are significant effects of pyrolysis temperature on densities and porosities of the chars. At a low pyrolysis temperature of 450⁰C, pore development was poor due to insufficient energy to release the volatile matter.

However, at a high pyrolysis temperature of 950⁰C, a sintering effect and shrinkage of the char reduced the pore volumes and pore areas, particularly micro pores (Mohd Rafatullah et al.,2012).

3.3 Neutralization of pH

Materials and Apparatus

oven (binder), Buchner vacuum pump filtration

The activated carbon washed again with distilled water using Buchner vacuum pump filtration until the pH becomes 7. Finally, the activated carbon dried under 100⁰C, and the weight was recorded again for every 6 hours (less than 2 days) until it shows same readings. The activated carbon was in granules form, sieve was used to separate the powdered and pieces of AC. The sample is weighed again and transferred into a zip lock bag and kept in a desiccator for further test on heavy metal and characterizations.

3.4 Activated Carbon Test on Heavy Metal

Materials and Apparatus

Conical flask, orbital shaker, syringe membrane

Stock solution of copper and manganese heavy metals are prepared for 100ml in volumetric flask separately. Batch adsorption test was carried out by shaking 10 mL of different concentration heavy metals solutions with different dosages of activated carbon in a stoppered test tubes placed in an orbital shaker at 150 rpm at 30°C. After a predetermined contact time, the bottle was removed from the shaker and the supernatant was filtered filter paper and analyzed for copper and manganese concentration by an ICP-OES. The effect of contact time (1-3 h), metal concentration 0, 1 and 5 mg/L and carbon dose (0, 0.25 and 1.0 g) on adsorption were evaluated by batch adsorption test (Chaudhuri, et al.,2010).

3.5 Physical Characterization of OPT Activated Carbon

The pore structures of the resulting carbons will be analyzed using scanning electron microscope (SEM)

Apparatus

3.5.1 SEM

SEM images recorded by using JEOL JSM-6300F field emission SEM. A thin layer of platinum is sputter-coated on the samples for charge dissipation during FESEM imaging. The sputter coater (Eiko IB-5 Sputter Coater) will be operated in an argon atmosphere using a current of 6 mA for 3 min. The coated samples then shifted to the SEM specimen chamber and observed at an accelerating voltage of 5 kV, eight spot size, four apertures and 15 mm working distance. (Prahas, D., et al., 2008)

3.5.2 ImageJ Software

ImageJ is a public domain Java image processing and analysis program inspired by NIH Image for the Macintosh. It can display, edit, analyze, process, save and print 8-bit, 16-bit and 32-bit images. Other than that, imagej also able to read many image formats including TIFF, GIF, JPEG, BMP, DICOM, FITS and 'raw' while supporting 'stacks' (and hyperstacks), a series of images that share a single window.

ImageJ made as multithreaded, so operations that take long time such as image file reading can be done together with other operations. It is also open source picture preparing program intended for logical multidimensional pictures. It is exceedingly extendible, with a large number of modules and contents for playing out a wide assortment of errands, and a vast client network.

This software is used in my research to measure the micropores size of the activated carbon from the SEM image. The results were accurate and easy to analyze as it is arranged according to the position and angle. This can describe the type of pore by the diameter of each pores.

3.6 Sample analysis for determination of heavy metal concentrations by Inductively Coupled Plasma-Optical Emission Spectroscopy (ICP-OES)

3.6.1 Chemical analysis

Copper and Manganese were determined by a Perkin-Elmer DV 4300 inductively coupled plasma-optical emission spectrometry (ICP-OES). In the ICP-OES analysis, the following wavelength lines were used; Cu 324.8 nm and Mn 232 nm.

ICP-OES runs will include an initial calibration verification solution and a blank solution at the beginning and end of every run. A calibration verification solution will be analyzed after every 10 samples throughout the run. The verification measurements must be within 10 percent of the expected value. If they are not, the problem should be corrected before continuing with analyses. During the analysis, every element will be analyzed in triplicate for every sample and used by the instrument to calculate relative standard deviation (RSD) automatically. Analyses above the method detection limit with a RSD greater than 5 % should be discontinued and the problem corrected before continuing with the analyses.

3.7 Data Analysis Method

Convex Integrated Design (CID) method is a new simultaneous mechanical structure and control system design method. This CID method can be used to solve the simultaneous mechanical structure and control system design problems with multiple simultaneous conflicting closed-loop design specifications.

To use this method, all design specifications must be convex with respect to the closed-loop transfer function. By taking advantage of this convex property, the CID design problem can be solved with a simple two-stage algorithm (Lu Ren., et al., 2008).

There are two factors that have been studied in the research, firstly factor A dependent variable. It was the metal concentrations. The metal that used is copper and manganese with following concentration (0,1 and 5 ppm). Whereas factor B was independent variable which is dosage of activated carbon. The dosage that have been tested were 0,0.25g and 1.0g.

This design is used to compare mean by using factorial design. The mean that is compared is the efficiency of activated carbon in adsorbing copper and manganese from the concentration prepared.

CHAPTER 4

RESULTS AND DISCUSSION

4.1 ACTIVATED CARBON PRODUCTION

Activated PPF has been prepared by chemical activation method using HCL as activating agent. The amount of AC synthesized from 800g of pre-carbonized PPF is 404.60 g.

4.2 RAW MATERIAL COLLECTION

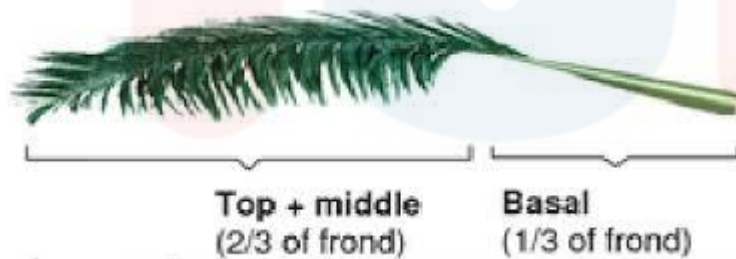
Figure 4.1: Photograph of oil palm tree frond: a) palm oil trees, b) raw palm petiole felt sheath and c) the part of frond basal is used for AC production. (Aliyu., et al., 2015)



a)



b)



c)

4.3 CHARACTERIZATION OF PPF

4.3.1 SURFACE MORPHOLOGY IDENTIFICATION

The surface morphology of the activated carbon samples can be determined by Scanning Electron Microscopy (SEM), JEOL at 150KV acceleration voltage and with 15mm working distance. There are few SEM images of PPF activated carbon before and after treatment with hydrochloric acid. The pore characteristics that formed in the

activated carbons can be observed clearly in SEM. These pores are the ascertain factors of the rate and the ability to adsorb foreign particles. Other than that,

Lignin, hemicellulose and cellulose are main component that made up of oil palm frond. Mostly lignin develops into macropores, which is more than 50 nm that can be seen in the diagram. There are studies shows that lignin have the ability to form macropore by fusing on pyrolysis (Md Som, Wang, & Al-Tabbaa, 2012).

Hydrochloric acid role play that inhibits tar development, as a dehydrating agent. It particularly blocks the mesopores and micropores. After these substances emancipated through chemical activation, there are more determined pore structure that can be visible via scanning electron microscopy (SEM) while the percentage yield becomes lesser.

By the comparison it can be clearly visible that the pores of activated carbon after treated with hydrochloric acid is larger than the activated carbon before treated. The magnification used are 500,1000 and 3000 respectively in SEM to capture the image of specific area of AC before and after treated.

Other than that, the activated carbon after treated with hydrochloric acid have no space between each pore (refer to 3000 magnification) while activated carbon before treated have more space between each pores.

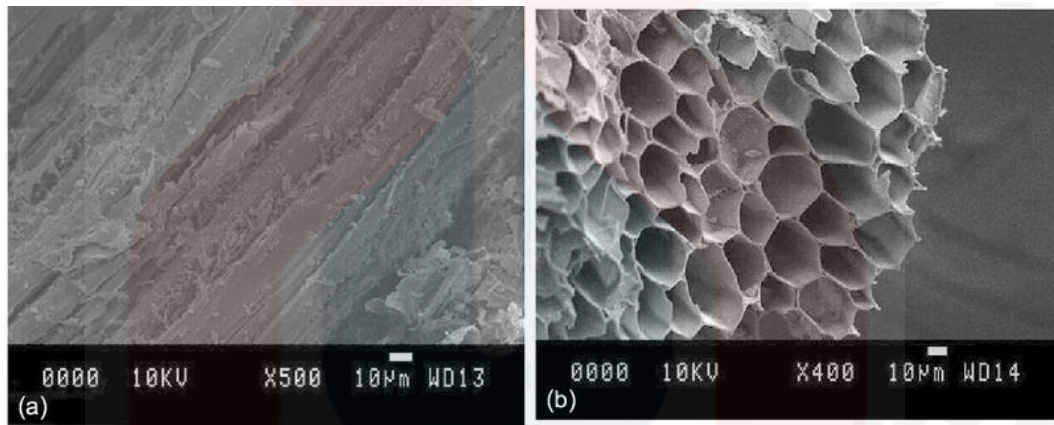
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There are some visible micropores in the macro and mesopores of AC after treated image while there are none can be much visible in the AC before treated image. ImageJ software is used to measure the diameter of the pores of after AC magnification at 3000x which is less than 2nm.

Figure 4.2: SEM image from carbonization studies of palm frond comparison with PPF.

(Md Som., et al., 2012)



(a) Before palm frond carbonization (b) After palm frond carbonization.

Table 4.1: The SEM image of PPF according to description.

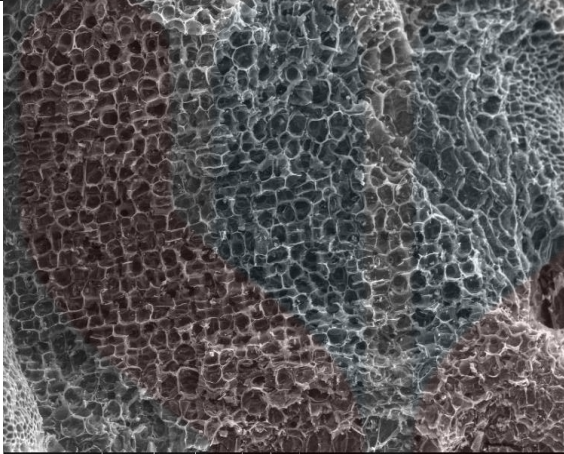
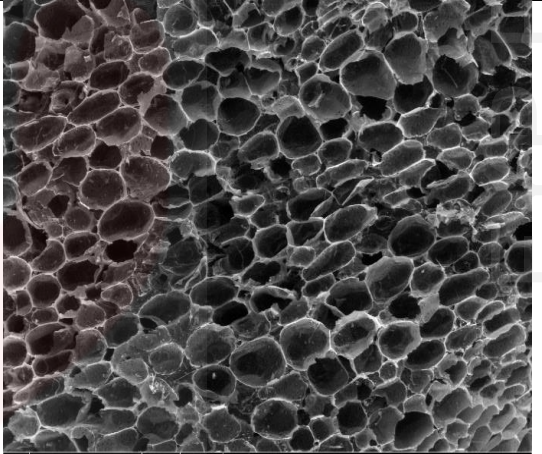
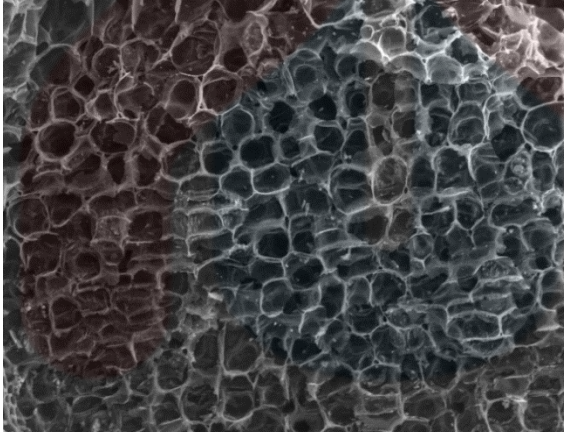
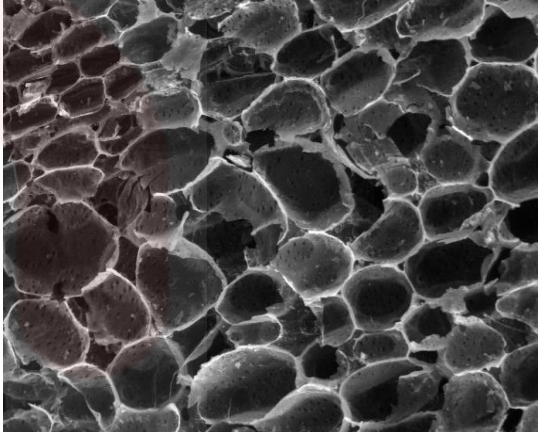
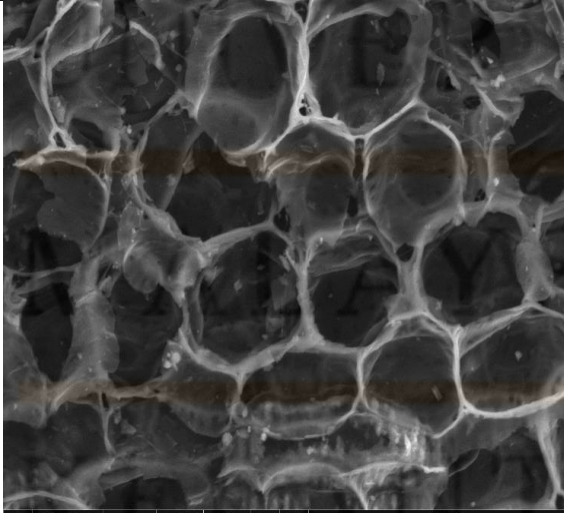
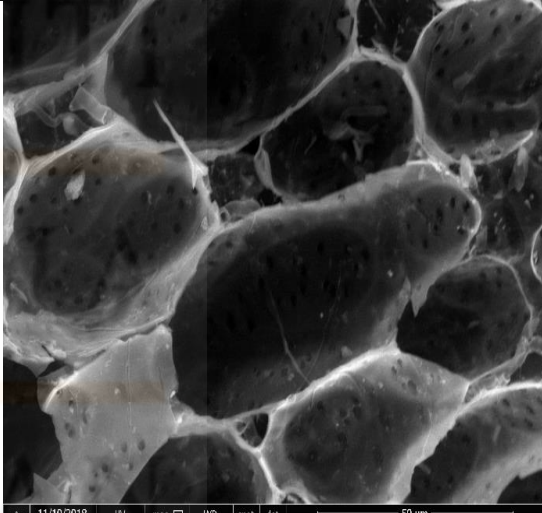
Magnification	Before Activated	After Activated
500 ×	 <p>11/19/2018 9:25:41 AM HV 15.00 kV mag 500 x WD 9.2 mm spot 4.5 det LFD 300 μm HiRes Gold on Carbon</p>	 <p>11/19/2018 9:40:50 AM HV 15.00 kV mag 500 x WD 9.0 mm spot 4.5 det LFD 300 μm HiRes Gold on Carbon</p>
1000 ×	 <p>11/19/2018 9:25:41 AM HV 15.00 kV mag 1 000 x WD 9.3 mm spot 4.5 det LFD 100 μm HiRes Gold on Carbon</p>	 <p>11/19/2018 9:40:43 AM HV 15.00 kV mag 1 000 x WD 9.1 mm spot 4.5 det LFD 100 μm HiRes Gold on Carbon</p>
3000 ×	 <p>11/19/2018 9:32:02 AM HV 15.00 kV mag 3 000 x WD 9.2 mm spot 4.5 det LFD 50 μm HiRes Gold on Carbon</p>	 <p>11/19/2018 9:52:30 AM HV 15.00 kV mag 3 000 x WD 8.9 mm spot 4.5 det LFD 50 μm HiRes Gold on Carbon</p>

Table 4.2: The size of micropores in the image after AC with 3000x magnification.

Number pores	Diameter(μm)			
	Micropores		Macropores	
	Before	After	Before	After
1	-	0.286	42.749	160.458
2	-	0.217	53.916	101.222
3	-	0.286	43.191	120.816
4	-	0.214	31.032	77.940
5	-	0.179	39.733	73.098
6	-	0.214	41.484	63.393
7	-	0.25	50.307	82.671
8	-	0.258	47.816	70.680

4.4 ADSORPTION PERFORMANCE

In this research, adsorption process has been carried by using Activated Palm Petiole Felt Sheath (PPF) to reduce two types of heavy metals which are copper and manganese. The activated PPF added and left for 3 hours in stock solution of heavy metals which varies in weight of AC (0,0.25g and 1.0g) and concentration of heavy metal (0, 1ppm and 5ppm). The temperature used during the research is constant at 30°C. The result of the research has been transferred in the form of graph to be analyzed further.

4.4.1 ADSORPTION PERFORMANCE OF PPF ON COPPER

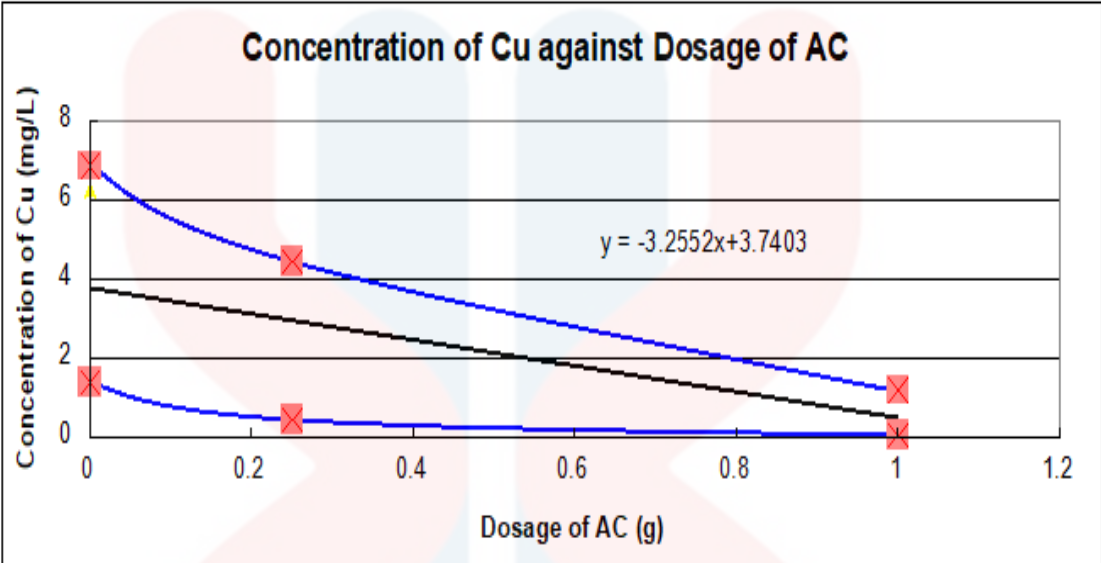
Adsorption performance of activated PPF has been tested at vary dosage of AC and concentration. The differences of copper concentration before and after shaken with AC for 3 hours is the amount of reduced by the AC sample.

Lowest dosage of AC used was 0.25g and the highest is 1.0g while for the concentration is 1ppm and 5ppm for 3 hours. The experiment started with 1ppm with no AC, the initial reading was **1.37 mg/L**. When 0.25g was used to treat 1 ppm concentration of copper for 3 hours, the result shows reduction about **0.95 mg/L** of copper concentration but when 1.0g of AC is used it displays a great varies as **1.33 mg/L**. Whereas when 5ppm is used with the absence of AC, the initial reading was **6.86 mg/L**. While it reduced about **2.42 mg/L** and **5.69 mg/L** when 0.25 g and 1.0 g is used consequently.

This is due to the limitation of the amount of pore structure which cannot adsorb 5ppm of copper concentration completely but it able to adsorb 1ppm almost completely. The different dosage has different amount of surface area that varies the ability of AC adsorption.

The graph shows that the higher adsorption of copper taken place when larger dosage of activated carbon is used. The equation in the graph can be used to estimate the value of adsorption with different dosage of activated carbon and concentration of manganese.

Figure 4.3: The graph of Copper adsorption by prepared AC



4.4.2 ADSORPTION PERFORMANCE OF PPF ON MANGANESE

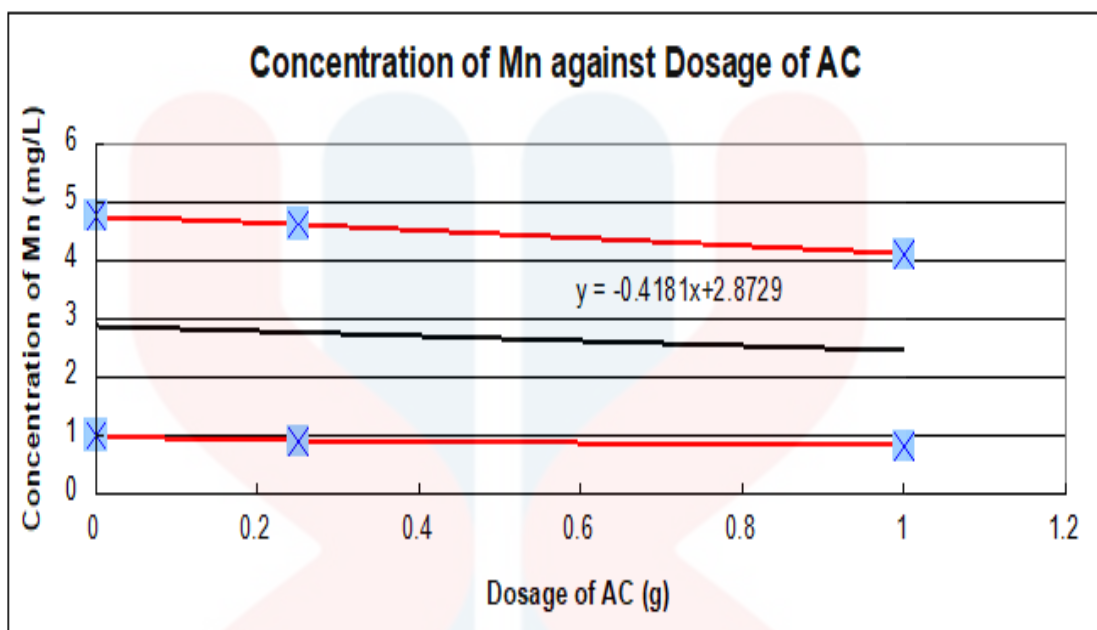
Adsorption performance of activated PPF has been tested at vary dosage of AC and concentration. The optimum temperature for adsorption is 30°C, the differences of manganese concentration before and after shaken with AC for 3 hours is the amount of reduced by the AC sample.

For manganese test there are also lowest dosage of AC used as 0.25g and the highest was 1.0g while for the concentration is 1ppm and 5ppm for 3 hours. The experiment started with 1ppm with no AC, the initial reading was **0.99 mg/L**. When 0.25g was used to treat 1 ppm concentration of manganese for 3 hours, the result shows reduction about **0.1 mg/L** of manganese concentration but when 1.0g of AC is used it displays a great varies as **0.19 mg/L**. Whereas when 5ppm is used with the absence of AC, the initial reading was **4.77 mg/L**. While it reduced about **0.14 mg/L** and **0.66 mg/L** when 0.25 g and 1.0 g is used consequently.

This results shows that the amount of pore structure in 1.0g of AC cannot adsorb 5ppm of manganese concentration completely but it able to adsorb 1ppm almost completely. The different dosage has different amount of surface area that varies the ability of AC adsorption. The higher concentration of metal needs larger amount of AC for efficient adsorption.

The equation in the graph can be used to estimate the value of adsorption with different dosage of activated carbon and concentration of manganese.

Figure 4.4: The graph of Manganese adsorption by prepared AC



4.5 METAL COMPETITION FOR ADSORPTION

There are two metals used to test adsorption capacities of the Granular Activated Carbon (GAC) prepared as manganese and copper. The research result shows that the copper has been highly adsorbed by the GAC compared to manganese. There are few factors that caused the difference in the ability of the metals to be adsorbed by the GAC.

Factor that greatly influenced the GAC performance were electronegativity trend and ionic radius. Pauling scale is used to read the electronegativity trend, it was invented by a chemist, Linus Pauling. Electronegativity is chemical property that used to measure of atom's ability to attract and form bonding with electrons.

Electronegativity measures an atom's tendency to attract and form bonds with electrons. This property exists due to the electronic configuration of atoms. The electronegativity of Cu is higher than the Mn which are 1.9 and 1.5 respectively.

From the studies of the research done electronegativity here is the ability to measure the strength of Cu and Mn attach to negative charge at activated carbon surface. The GAC ability is corresponded with the high electronegativity to higher adsorption strength of metal ions(Ahmad Jusoh, et.al, 2005)

Ionic radius is another reason that influence the adsorption of GAC. Cu has moderately lower ionic radius than that of the Mn. Juxtapose to Mn, Cu has the higher attractive charge in nucleus on the electron orbital. The smaller ionic radius of Cu makes it less demanding to enter into the micropores of the GAC.

There were four major functional groups on the surface of activated carbon which are carboxyl, carbonyl, hydroxyl, and lactonized carboxyl. They were encouraged to attract cation so that ion exchange can take place.

Furthermore, positively charged copper and manganese have high possibilities to be bonded chemically with functional group that found onto GAC surface's. However, the actual chemical reaction between the metal ion and functional groups on the activated carbon surface was complex.

Table 4.3: The percentage of adsorption competition by metals

<i>Concentration</i>	<i>Reduction % with 1.0 g</i>	
	5 ppm	1 ppm
Metals		
Copper	<p>–</p> <p>$\frac{5.69}{6.86} \times 100\% = 82.94 \%$</p>	<p>$\frac{1.33}{1.37} \times 100\% = 97.08 \%$</p>
Manganese	<p>$\frac{0.66}{4.77} \times 100\% = 13.84 \%$</p>	<p>$\frac{0.19}{0.99} \times 100\% = 19.19 \%$</p>

Table 4.4: The table shows the reading of copper and manganese concentration.

Metals	Activated Carbon	Initial Reading	Reading
Copper	0	1.303	1.365
	0.25		0.419
	1		0.044
	0	6.259	6.859
Manganese	0.25		4.443
	1		1.174
	0	0.928	0.994
	0.25		0.894
	1		0.804
	0	4.849	4.765
	0.25		4.625
	1		4.11



CHAPTER 5

5.0 CONCLUSION AND RECOMMENDATION

CONCLUSION

In the nutshell, the objectives of the research have been achieved. The activated carbon has been developed from Oil Palm Petiole Felt-Sheath by using a modified methodology. The characterization of activated carbon and non-activated carbon is done by Scanning Electron Microscopy (SEM). The properties found in the activated carbon can be useful in determining the effectiveness of activated carbon in heavy metal adsorption.

Other than that, the research has proved that copper and manganese that can be found in aquaculture wastewater can be removed by the activated carbon that is developed from Oil Palm Petiole Felt-Sheath. The overall result shows that larger amount of activated carbon can remove higher concentration of metal concentration.

Not only that but copper have higher ability to be adsorbed by activated carbon compare to manganese. These happens because the copper with higher electronegativity, lower ionic radius has greater ability to be adsorbed by activated carbon compare to manganese.

RECOMMENDATION

The activated carbon that been developed can be used to further research into adsorption of ammonium that also one of the large amount found in the aquaculture wastewater. The special characteristics of activated carbon with large pore structure can be applied in the industries of aquaculture farm to treat their waste water.

These research need to be studied further to determine the different activating agent, activation time during pyrolysis to produce high quality of activated carbon which have larger amount and size of pore structure. Thus it the activated carbon prepared from agriculture waste which is low cost can be used to reduce heavy metal compare to commercial adsorbent.

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APPENDIX



Figure 3.1: Charcoal before soaked with hydrochloric acid



Figure 3.2: Concentrated hydrochloric acid (37%) is added into pre-carbonized charcoal.

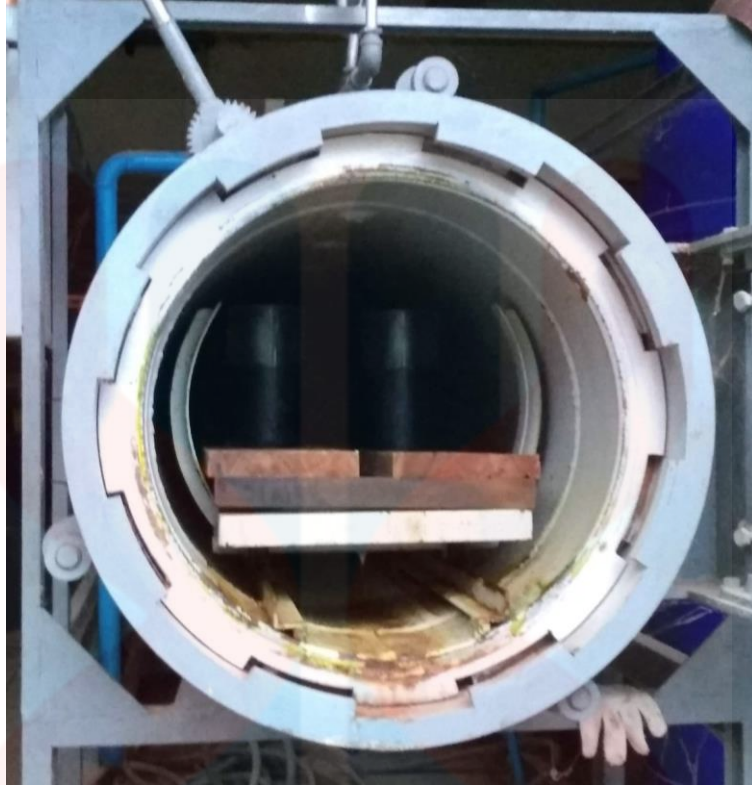


Figure 3.3: The charcoal soaked with hydrochloric acid is left overnight in the vacuum chamber.



Figure 3.4: The vacuum chamber used to store acid soaked charcoal for overnight.



Figure 3.5: Well soaked active carbon is filtered and left in the hood for few days to dry completely.



Figure 3.6: The weight of activated carbon after dried under the hood.



Figure 3.7: The weight of AC in 5 crucibles 67.29g before furnace.



Figure 3.8: Half filled crucibles placed into a claypot and occupied with dried sand to avoid air entry during furnace.

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Figure 3.9: The prepared claypot is placed into furnace without touching the side surface and set to 800°C for 6 hours.



Figure 3.10: The weight of AC in 5 crucibles after furnace 37.01g.



Figure 3.11: The weight of activated carbon after furnace under 800°C for 6 hours.



Figure 3.12: The vacuum pump is used with filter paper to wash the activated carbon.



Figure 3.13: Prepared activated carbon for adsorption test.



Figure 3.14: Measuring cylinder used to measure 10ml of each heavy metal concentration.

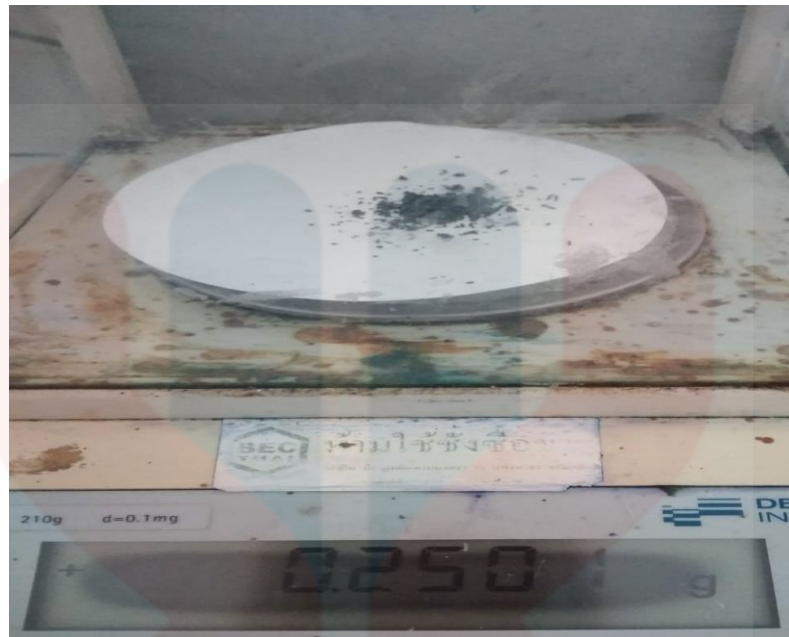


Figure 3.15: The weight of 0.25 g of activated carbon used for adsorption test.



Figure 3.16: The weight of 0.50 g of activated carbon used for adsorption test.

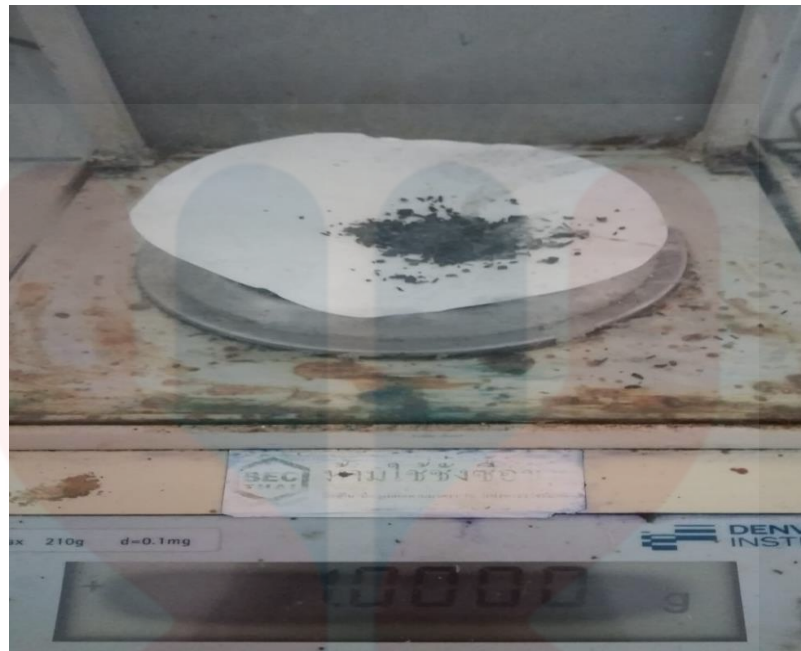


Figure 3.17: The weight of 1.0 g of activated carbon used for adsorption test.

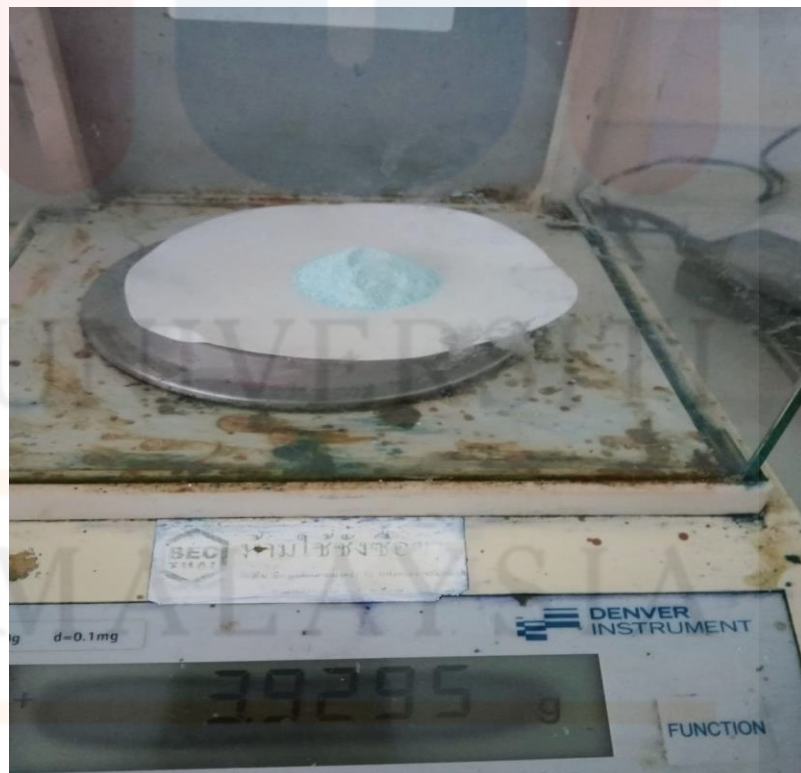


Figure 3.18: The weight of 3.93 g of copper powder used to make stock solution.



Figure 3.19: The weight of 3.08 g of manganese powder used to make stock solution.

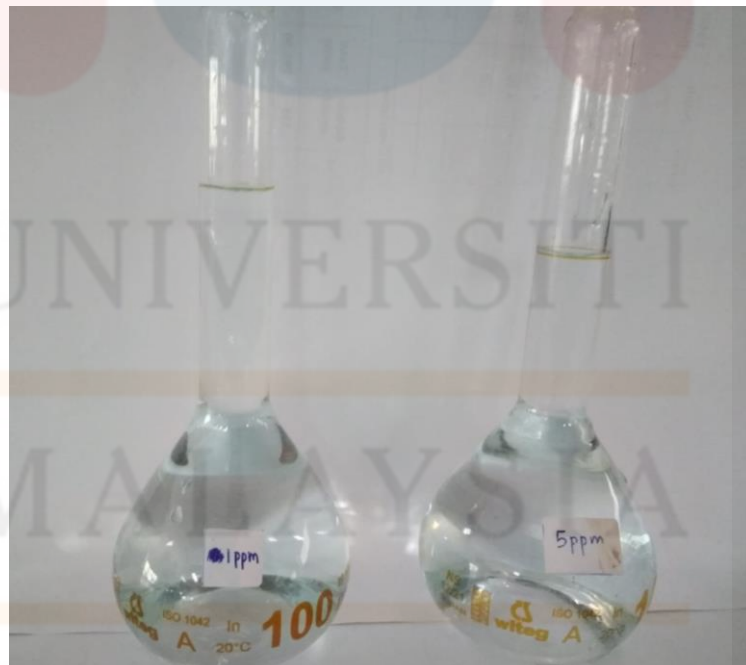
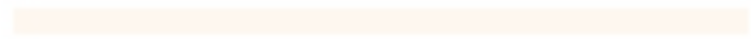


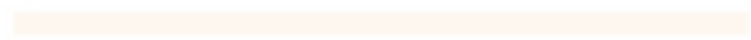
Figure 3.20: The mix stock solution of copper and manganese in different concentration.



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