

**PREPARATION AND CHARACTERIZATION OF ACTIVATED CARBON
FROM OIL PALM TRUNK**

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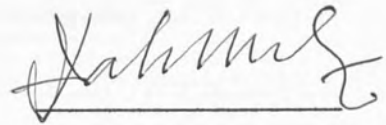
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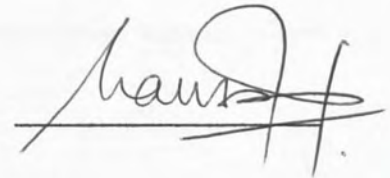
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ABSTRACT

A series of activated carbons were prepared from oil palm trunk impregnated with varying concentration of phosphoric acid using a two-steps activation procedure: carbonization at 200°C for 15 minutes followed by activation at 500°C for 45 minutes. Physical and chemical characterizations such as iodine number, ash content, moisture content, pH, percent yield and methylene blue adsorption determination were performed. Percent yield of activated carbon ranged from 28.3% to 55.6%, depending on the concentration of phosphoric acid. The activated carbons have pH values ranging from 6.32 to 5.14, ash content ranging from 0.4% to 3.8%, moisture content in the range of 2.4% to 7.33%, iodine number ranging from 187.96mg/g to 492.76mg/g and methylene blue adsorption capacity ranging from and 80mg/g to 855mg/g.



ABSTRAK

Satu siri karbon teraktif telah disediakan daripada batang kelapa sawit menerusi percampuran dengan asid fosforik pada kepekatan berlainan menggunakan proses pengaktifan dwilangkah: pengkarbonan sehingga 200°C untuk 15 minit diikuti oleh pengaktifan pada 500°C untuk 45 minit. Pencirian secara kimia dan fizikal seperti penentuan nombor iodin, kandungan abu dan kelembapan, pH, peratus hasil dan kapasiti penyerapan larutan metilina biru telah dijalankan. Peratus hasil karbon teraktif yang berjulat antara 28.3% dan 55.6%, adalah bergantung kepada kepekatan asid fosforik. Karbon teraktif mempunyai nilai pH berjulat dari 6.32 kepada 5.14, kandungan abu berjulat dari 0.4% kepada 3.8%, kelembapan berjulat 2.4% hingga 7.33%, nombor iodin dengan julat 187.96mg/g kepada 492.76mg/g dan kapasiti penyerapan metilina biru dalam julat dan 80 mg/g kepada 855mg/g.



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LIST OF SYMBOL, UNITS AND ABBREVIATION

%	Percentage
Å	Angstroms
AlCl ₃	Alluminium chlorite
°C	Celcius
C	Carbon
CO	Carbon monoxide
g	gram
hr	hour
H ₂	hydrogen gas
H ₂ O	water molecule
H ₃ PO ₄	phosphoric acid
K	Kelvin
kg	kilogram
kJ	kilojoule
KBr	Potassium bromate
KOH	Potassium hydroxide
K ₂ CO ₃	Potassium carbonate
KI	Potassium Bromide
L	Liter
m ²	metre squared
ml	milliliter
min	minute
MgCl ₂	Magnesium chlorite
NaOH	Sodium hydroxide



Na_2CO_3	Sodium carbonate
NH_3	Ammonia
NaS_2O_3	Sodium thiosulphate
ppm	Concentration
P	Pressure
s	second
wt%	amount of weight need to impregnate the sample
0M (S1)	physically activated carbon
2M (S2)	chemically activated carbon prepared by impregnation with 2M H_3PO_4
4M (S3)	chemically activated carbon prepared by impregnation with 4M H_3PO_4
7M (S4)	chemically activated carbon prepared by impregnation with 7M H_3PO_4
10M (S5)	chemically activated carbon prepared by impregnation with 10M H_3PO_4
12M (S6)	chemically activated carbon prepared by impregnation with 12M H_3PO_4



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CHAPTER 1

INTRODUCTION

1.1 Introduction

Activated carbons are still probably the most widely used adsorbents in industry. They are recommended for the general removal of organic molecules from water, where special hydraulic requirements must be considered. Elsewhere, granular activated carbon adsorption is a recognized technology in waste water and drinking water treatment (AWWA, 1997).

Most practical applications of activated carbons require sorbents having a large volume of very fine pores. The presence of micro pores substantially influences its sorption properties because the amount adsorbed on the macro pore surface is negligible in comparison to that for the micro pores. Therefore, Characterization of the activated carbons has become one of the most important problems in adsorption technology.

Physical or chemical activation method can be used for manufacture from a wide variety of precursor materials. Physical activation is usually carried out in two steps: the first is carbonization of the raw material in an inert atmosphere at a temperature below 700 °C, and the second is activation in the presence of steam, carbon dioxide, and air at temperatures between 800 and 1000 °C. Chemical activation is generally carried out in



one step, which consists of impregnation of the raw material with a strong dehydrating agent, typically phosphoric acid, and then heating the mixture to temperatures of 400-800 °C to simultaneously form and activate the carbon (Diao, Walawender and Fan, 2002).

The process of activated carbon generation begins with the selection of a raw carbon source. These sources are selected based on design specifications since different raw source will produce activated carbon with different properties (AWWA, 1971).

Several activating agents have been reported for chemical activation process; however the most important and commonly used activating agents are phosphoric acid, zinc chloride and alkaline metal compounds. Phosphoric acid and zinc chloride are used for the activation of lignocellulose materials, which have not been carbonized previously; whereas metal compounds such KOH, are used for activation of coal precursors or chars. When compared to zinc chloride, phosphoric acid is the most preferred because of the environmental disadvantage associated with zinc chloride. Moreover, the carbon obtained using zinc chloride cannot be used in pharmaceutical and food industries as it may contaminate the product (Srinivasakannan and Mohamad Zailani Abu Bakar, 2004).

Palm oil industry produces a large amount of solid wastes, such as palm fibres, palm shells, empty fruit bunches and old palm tree trunk. For example, in Malaysia, which is one of the largest palm oil producers in the world, more than two million tons (dry weight) of extracted oil palm fibre estimated to be generated annually (PORIM, 1998). The extracted oil palm fibre, are also known as palm cake fibre, which contains minute impurities of palm shells and palm stones particles. At present, it is usually used as fuel or building material.



The use of oil palm trunk to prepare activated carbon also helps to generate economic utilization for cheap and abundant solid waste, where it is proposed to be use as a prospective starting material for the preparation of activated carbon because of its relatively high fixed-carbon content (about 18 wt%), low ash content (less than 1.0 wt%) and the presence of inherent porous structures. In fact, a large number of naturally occurring carbonaceous solid wastes, such as coconut shell, almond shell and grape seed, walnut shell, sawdust, extracted rockrose, eucalyptus kraft lignin and various fruit stones have been successfully converted into activated carbons (Jia and Aik, 2003). Differences between raw materials used to prepare activated carbon are shown in Table 1.1.

Table 1.1 Differences between raw materials used to prepare activated carbon.

Raw Material	Density (kg/L)	Texture of AC	Applications
Soft wood	0.4 - 0.5	Soft, large pore volume	Aqueous phase adsorption
Nutshells	1.4	Hard, large micropore volume	Vapor phase adsorption
Hard coal	1.5 - 1.8	Hard, large pore volume	Gas vapor adsorption
Lignite	1.00 - 1.35	Hard, small pore volume	Wastewater treatment

(Source: Carlos J. Dura'n-Valle *et al*; 2005)

Activated carbon is used in water and wastewater treatment primarily as an adsorbent for the removal of relatively low levels of organic and inorganic contaminants via transfer from the dissolved phase to the solid carbon surface. While its adsorbent nature also makes it useful for the filtration of contaminated air, only its use in water treatment will be considered in this document (AWWA, 1997).



Most forms of activated carbon are non-polar in nature, so they have the greatest affinity for other non-polar substances. As a result, they are most effective in the removal of a variety of organic contaminants, including trihalomethanes, pesticides and herbicides, and polyaromatic hydrocarbons. However, activated carbon may also be used for the removal of trace metals such as cadmium and lead, and it has also been effective in removing some polar organics as well. On the other hand, activated carbons do not effectively remove contaminants of high solubility or inorganic salts like nitrates (AWWA, 1997).

In remediation systems, activated carbon is almost always used as a component in some form of pump-and-treat system. Commonly, it is used as a sorptive filter medium through which contaminated water is passed before it is re-injected or routed to a stream or sewer. In these applications, activated carbon is most commonly used as a tertiary step, in which low concentrations of contaminants are removed from partially treated water.

Dyes are usually present in trace quantities in the treated effluents of many industries. The effectiveness of adsorption for dye removal from wastewater have made activated carbon an ideal alternative to other expensive treatment methods. These studies investigate the potential use of oil palm trunk, for the removal of methylene blue from an aqueous solution where higher adsorption percentages are observed at lower concentrations of methylene blue (Avom *et al.*, 1997).



1.2 Objective of Study

The objectives of this study are:

- a. To prepare activated carbon from oil palm trunk using phosphoric acid as the activating agent.
- b. To physically and chemically characterize the activated carbon that has been prepared.

1.3 Scope of study

In this study, oil palm trunk will be used as the raw material for producing activated carbon. The activation process will consist of two steps namely activation and carbonization and phosphoric acid will be used as the activating agent. The activated carbon obtain will then be analyzed for percentage of yield, pH, ash content, moisture content, iodine number and methylene blue adsorption.

CHAPTER 2

LITERATURE REVIEW

2.1 What Is Activated Carbon

Activated carbon includes a wide range of amorphous carbon-based materials prepared to exhibit a high degree of porosity and an extended interparticulate surface area. These qualities impart activated carbon with excellent adsorbent characteristics that make carbon very useful for a wide variety of processes, including filtration, purification, deodorization, decolorization, purification and separation with this definition, little else is left to be said. Activated carbon is defined by its properties and source and it can be produced from almost any organic substance with high carbon content such as coconut, coal, lignite and wood (Powder) (Table 2.1). The activation of the carbon provides it with many of its useful properties, and the type and degree of activation affect its physical and chemical properties (Bansal, Donnet, and Stoeckli, 1988).



Table 2.1 Types of activated carbon

Property	Coconut Activated Carbon	Coal Activated Carbon	Lignite Activated Carbon	Wood Activated Carbon (Powder)
Micropore	High	High	medium	Low
Macropore	Low	Medium	high	High
Hardness	High	High	low	n/a
Ash	5%	10%	20%	5%
Water Soluble Ash	High	Low	high	Medium
Dust	Low	Medium	high	n/a
Reactivation	Good	Good	poor	None
Apparent Density	0.48 g/cc	0.48 g/cc	0.4 g/cc	0.35 g/cc
Iodine No.	1100	1000	600	1000

(Source: Bansal, Donnet, and Stoeckli, 1988)

In practice, activated carbon is found in two general forms: granular activated carbon (GAC) and powdered activated carbon (PAC). GAC is defined as being retained on a 50-mesh sieve (0.297 mm) and PAC material as finer material. Particle sizes of GAC is classified corresponding to an 80-mesh sieve (0.177 mm) and smaller as PAC. Exact specifications vary, but GAC is generally said to have a larger internal surface area and smaller internal pores, while PAC is thought to have larger pores and a smaller internal surface area. PAC is made up of crushed or ground carbon particles, 95–100% of which will pass through a designated mesh sieve or sieves. PAC is not commonly used in



a dedicated vessel, owing to the high heat loss that would occur. PAC is generally added directly to other process units, such as raw water intakes, rapid mix basins, clarifiers, and gravity filters (AWWA, 1998).

2.2 History

The use of carbon extends so far back into history that its origin is impossible to document. Activated carbon was first used to treat water over 2000 years ago. Ancient Hindus in India used charcoal for drinking water filtration, and carbonized wood was used as a medical adsorbent and purifying agent by the Egyptians as early as 1500 B.C (Bansal, Donnet, and Stoeckli, 1988). However, it was first commercially produced at the beginning of the 20th century and was only available in powder form. Initially activated carbon was mainly used to decolorize sugar and then from 1930 for water treatment to remove taste and odor. Granular activated carbon was first developed as a consequence of World War I (WWI) for gas masks and has been used subsequently for water treatment, solvent recovery and air purification (AWWA, 1997).

Activated carbon can be produced from a variety of carbonaceous raw material, the primary ones being coal, oil palm shell, coconut shells, wood and lignite (Anuar *et al.*, 2003). The intrinsic properties of the activated carbon are dependent on the type of precursor and method of preparation used. The activated carbon surface is non-polar which results in an affinity for non-polar adsorbates such as organics. Adsorption is a surface phenomenon in which an adsorbate is held onto the surface of the activated carbon by Van der Waal's forces and saturation is represented by an equilibrium point. These forces are physical in nature, which means that the process is reversible by using heat and pressure (AWWA, 1997).



2.3 Properties of Activated Carbon

Activated carbon contains a wide range of micropore, the walls of which have surface area that range from 400 to over 1800 m²/g in various commercial brands. To be effective, activated carbon must have more than just a large surface, it must be selective, that is, to take up and hold molecules of the substance to be removed without disturbing other substances in the system. Activated carbon can meet many of the diverse needs because the adsorptive properties can be developed in various forms by appropriate changes in the manufacturing process (Jia and Aik, 2003).

Activated carbon is similar to crude graphite - the material used in pencils. Along with diamonds, activated carbon and graphite are both forms of carbon and contain almost no nitrogen, hydrogen, halogens, sulfur or oxygen. From a chemist's perspective, activated carbon is an imperfect form of graphite (Clark and Roberts, 1989).

Intermolecular attractions in the smallest pores result in adsorption forces. Carbon adsorption forces work like gravity, but on a molecular scale. They cause a reaction similar to precipitation, in which adsorbates are removed from solution or vapor stream. To develop a strong adsorption force, either the distance between the carbon platelets and the adsorbate must be decreased (by reducing its pore size), or the number of carbon atoms in the structure must be increased (by raising the density of the carbon). Physical adsorption enables activated carbon to remove taste and odor-causing organic compounds like Trihalomethanes (THMs) and other halocarbons from process water and vapor streams (Silva, 1998).



2.3.1 Characteristics of Activated Carbon

The process of activated carbon generation begins with the selection of a raw carbon source. These sources are selected based on design specifications since different raw sources will produce activated carbon with different properties. Some of the more common raw sources include wood, sawdust, lignite, peat, coal, coconut shells, and petroleum residues (Lussier, Shull and Miller, 1994).

Characteristics of importance in choosing carbon types include pore structure, particle size, total surface area and void space between particles (Clark and Roberts, 1989). After selection of a source, preparations for use are made. These preparations most often include dehydration, carbonization, and activation. Dehydration and carbonization involve slow heating of the source in anaerobic conditions. Chemicals such as zinc chloride or phosphoric acid can be used to enhance these processes. The stage of activation requires exposure to additional chemicals or other oxidizing agents such as a mixture of gases. Depending upon the specifics of the process and the source carbon, the newly activated carbon can be classified according to density, hardness, and other characteristics (Lussier, Shull and Miller, 1994). Activated carbon can be classified in these factors:

- Surface area (BET N₂) - Activated carbon is often characterized by its extremely large surface area. In fact, the surface area per gram of material can range from 500 to 1400 square meters, and values as high as 2500 m²/g have been reported (Díaz-Díez *et al.*, 2004). Is measured by the adsorption of nitrogen gas onto the carbon and is expressed in square meters of surface area per gram of carbon. Because the gas molecules used to measure adsorption are very small, it should be

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