

UTILIZATION OF OIL PALM SHELLS AS A SOURCE OF  
ACTIVATED CARBON: PREPARATION AND  
CHARACTERIZATION

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ACTIVATED CARBON: PREPARATION AND CHARACTERIZATION

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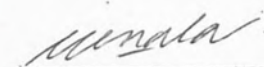
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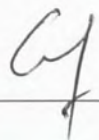


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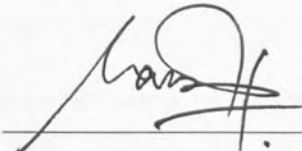
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## ABSTRACT

Activated carbons can be produced from the wastes of agricultural product. In this study oil palm shells were been use as the source to produce activated carbon using phosphoric acid. These wastes can be used by the industries to manufacture activated carbon. Carbonization and activation were done in the furnace using different temperatures which were 200°C for 15mnutes and 500°C for 45 minutes. Oil palm shells were impregnated with different concentration of phosphoric acid. The concentration of phosphoric acid that was used was 0M, 2M, 4M, 7M, 10M, and 12M. Several physical and chemical characterizations (moisture and ash, pH, iodine number, SEM, AAS, and methylene blue) of the prepared activated carbons were performed. An increase in the micropores with the increasing chemical shows that the development of porosity was also accompanied by a widening of the porosity as the amount of  $H_3PO_4$  increased. The amount of iodine number and the amount of methylene blue adsorbed was increasing as the concentration of  $H_3PO_4$  increased. The increase in adsorption process is due to the nature of pores on the surface of the activated carbons and throughout the surface of the carbons.



## ABSTRAK

Karbon aktif boleh dihasilkan daripada pemprosesan sisa hasil pertanian. Dalam kajian ini tempurung kelapa sawit digunakan untuk menghasilkan karbon aktif menggunakan asid fosforik. Sisa ini digunakan oleh industri untuk memproses karbon aktif. Proses pengkarbonan dan pengaktifan telah dilakukan di dalam 'furnace' dengan menggunakan dua suhu berlainan iaitu, 200°C untuk 15 minit dan 500°C untuk 45 minit. Tempurung kelapa sawit telah dicampurkan dengan asid fosforik dengan kepekatan berlainan. Kepekatan asid fosforik yang telah digunakan ialah 0M, 2M, 4M, 7M, 10M, dan 12M. Beberapa ciri fizikal dan kimia (kelembapan dan abu, pH, nombor iodine, SEM, AAS, dan metilena biru) dilakukan ke atas karbon aktif yang telah disediakan. Peningkatan dalam liang dengan penambahan bahan kimia menunjukkan bahawa perkembangan dalam liang dapat diperhatikan dengan adanya perluasan pada liang akibat daripada peningkatan pada kepekatan  $H_3PO_4$ . Jumlah nombor iodine dan jumlah metilena biru yang dijerap telah meningkat akibat daripada perluasan pada liang di permukaan karbon aktif.



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**LIST OF SYMBOL / ABBREVIATION**

%	Percentage
Å	Angstroms
AlCl <sub>3</sub>	Alluminium chlorite
°C	Celcius
C	Carbon
CO	Carbon monoxide
g	gram
hr	hour
H <sub>2</sub>	hydrogen gas
H <sub>2</sub> O	water molecule
H <sub>3</sub> PO <sub>4</sub>	phosphoric acid
HHO <sub>3</sub>	nitric acid
K	Kelvin
kg	kilogram
kJ	kilojoule
KBr	Potassium bromate
KOH	Potassium hydroxide
K <sub>2</sub> CO <sub>3</sub>	Potassium carbonate
KI	Potassium Bromide
L	Liter
m <sup>2</sup>	metre squared





ml	milliliter
min	minute
MgCl <sub>2</sub>	Magnesium chlorite
NaOH	Sodium hydroxide
Na <sub>2</sub> CO <sub>3</sub>	Sodium carbonate
NH <sub>3</sub>	Ammonia
NaS <sub>2</sub> O <sub>3</sub>	Sodium thiosulphate
ppm	Concentration
P	Pressure
s	second
wt%	amount of weight need to impregnate the sample
ZnCl <sub>2</sub>	zinc chlorite
0M (S1)	physically activated carbon
2M (S2)	chemically activated carbon prepared by impregnation with 2M H <sub>3</sub> PO <sub>4</sub>
4M (S3)	chemically activated carbon prepared by impregnation with 4M H <sub>3</sub> PO <sub>4</sub>
7M (S4)	chemically activated carbon prepared by impregnation with 7M H <sub>3</sub> PO <sub>4</sub>
10M (S5)	chemically activated carbon prepared by impregnation with 10M H <sub>3</sub> PO <sub>4</sub>
12M (S6)	chemically activated carbon prepared by impregnation with 12M H <sub>3</sub> PO <sub>4</sub>



## CHAPTER 1

### INTRODUCTION

#### 1.1 INTRODUCTION

Activated carbons are high porosity, high surface area materials manufactured by carbonization and activation of carbonaceous materials, which find extensive use in the industrial sector for adsorption of pollutants from gaseous and liquid streams. One can design activated carbon for adsorption of specific adsorbate, using appropriate precursor and by optimizing the activation process conditions. Either physical or chemical activation method is used for manufacture from a wide variety of precursor materials (Srinivasakannan and Zailani, 2004).

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 or air at temperatures between 800°C 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°C–800°C to simultaneously form and activate the carbon (Diao *et al.*, 2002).



Characterization of the activated carbons has been mainly focused on determinations of surface area and pore size distribution. Additional information on their pore size distribution was obtained by comparing the adsorption characteristics from different adsorbates such as methylene blue and iodine. The choice of these molecules is justified by their properties. Thus, the mesopores of carbon are often studied by methylene blue adsorption and this also serves as a model compound for adsorption of organic contaminants from aqueous solution (Lussier *et al.*, 1994).

Activated carbons prepared with phosphoric acid are useful in the adsorption of hydrocarbons, evaporative emission control from vehicles fuel system, and solvent recovery (Derbyshire *et al.*, 1995). According to Jagtoyen, the porosity is generated with phosphoric acid remaining intercalated in the internal structure of lignocellulosic materials, hindering their shrinkage. As the amount of  $H_3PO_4$  used in the impregnation increases, the volume filled by it and the various polyphosphate structures will obviously increase, resulting a larger pore volume and pore size (Suárez *et al.*, 2001).

Palm oil is an economically important crop in several humid and tropical countries. Malaysia is in the zone of warm humid climate (Spence and Cook, 1983). At present, Malaysia has produced more than half of the world's total output of palm oil (PORIM, 1998). Oil palm wastes are in the forms of leaves, fronds, and old trunks from the palm plantations. The palm mill wastes include empty fruit bunches, shells, pericarp, and effluent. Limited efforts had been carried out in the past to gain economical benefits from these wastes as construction material in producing activated carbons.



The total area planted with oil palm in Malaysia covers about 2.6 million hectares (Spence and Cook, 1983), with capability of oil palm shell (OPS) production of over 3.13 million tones annually (Mohd Noor *et al.*, 1990). The vast availability of this discarded OPS resource is yet unutilized commercially. To generate economic utilization for this cheap and abundant solid waste, it is proposed to use it 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 (Lua and Guo, 1998).

The main uses of palm oil are as a replacement in formulation of blends of hydrogenated liquid oils, marine oils and other oils and fats. As a frying fat, because of its low foam and longer keeping qualities stand out. It is also been used in the manufacture of biscuits, ice-creams, soap detergent and shampoos and in formulation of margarine and cooking fats. Palm oil can also be used to yield protein and anti-biotic by fermentation process using selected microorganisms (PORIM, 1998).



## 1.2 HISTORY OF ACTIVATED CARBON

Activated carbon was first used to treat water over 2000 years ago. 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 1 (WWI) for gas masks and has been used subsequently for water treatment, solvent recovery and air purification (American Water Works Association, 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 (using heat, pressure, etc.) (American Water Works Association, 1997).



### 1.3 RESEARCH OBJECTIVES

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

### 1.4 SCOPE OF STUDY

The scope of this study is to prepare activated carbon from oil palm shell activated with  $H_3PO_4$  using two-stage activation process.

## CHAPTER 2

### LITERATURE REVIEW

#### 2.1 Literature Review

Activated carbon is used extensively in many fields and most carbon materials can be used to make activated carbon. The academic literature contains many references to activated carbon derived from many agricultural and industrial high-carbon waste products. Commercial activated carbon however is manufactured from only a few carbon sources such as wood and sawdust, oil palm shells, peat, coal, coke, and some other material used (Anuar *et al.*, 2003). Activated carbon has a great surface area due to their network capillaries, and internal surface area. Activated carbon also has the ability to adsorb various substances from both gas and liquid phases (Blanco *et al.*, 2000).

#### 2.2 Properties 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 *et al.*, 1994).

Characteristics of importance in choosing carbon types include pore structure, particle size, total surface area and void space between particles (Clark, 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 *et al.*, 1994).

Activated carbon contain a wide range of micropore, the walls of which have surface area that range from 400 to over 1800m<sup>2</sup>/g in various commercial brands. Figure 2.1 below shows the schematic of the activated carbon model. 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 (Anuar *et al.*, 2003).





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