# PREPARATION AND CHARACTERIZATION OF ACTIVATED CARBON FROM OIL PALM SHELLS USING H<sub>3</sub>PO<sub>4</sub> AS DEHYDRATING AGENT

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PERPUSTAKAAN UNIVERSITI MALAYSIA SABAH

DISSERTATION SUBMITTED AS PARTIAL FULLFILLMENT FOR THE DEGREE OF BACHELOR OF SCIENCE WITH HONOURS

> INDUSTRIAL CHEMISTRY PROGRAMME SCHOOL OF SCIENCE AND TECHNOLOGY UNIVERSITI MALAYSIA SABAH

> > 2006



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## ACKNOWLEDGEMENT

I would like to take this opportunity to appreciate everyone that involved directly as well as indirectly on helping hand on my project paper until I finally finished this thesis writing. A special thanks to my supervisor, Mr. Collin Joseph for helping me a lot on giving an advice, guidance and encouragement along I am doing this project. I also want to thanks to all lecturers, my program coordinator and nor forget to Dean's of Sekolah Sains dan Teknologi (SST) for giving an encouragement and make my experiments and analyzed going smoothly with their support.

Special thanks also to all my friends that helping a lot during I am doing my experiment and during the writing process happen. A gratitude to all the lab assistants of SST, SKTM, and IBTP that helping me a lot while I am doing analysis and chemical preparation. I also want to give a special thanks to all my family members, start with my parents on giving me encouragement and advice, and to my siblings that support me a lot during I am doing my final year project.



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### ABSTRACT

The porosity and adsorption capacity in an activated carbon depended on the chemical activation, activation time and temperature as well as the surrounding condition. Porous carbons have been prepared from oil palm shells in different amount of chemical activator, activation time and temperature. The oil palm shells have undergone two stages activation, which is chemical activation before time and temperature activation process occurs. Results show that yield percentages as well as moisture and ash content will increase as impregnation ratio of phosphoric acid increase and activation time and temperature decrease. The SEM micrographs taken, proved the porosity of activated carbons generated as the activation temperature blue adsorption gives the results for the comparison of different concentration and shaking time. Langmuir model indicates that 4.5 M H<sub>3</sub>PO<sub>4</sub> and 450 °C are the optimum conditions for preparing good quality activated carbons and pseudo-second-order equation produces has the better result.



## ABSTRAK

Keliangan dan kapasiti jerapan bagi sesuatu karbon aktif bergantung kepada kimia aktivasi, masa dan suhu aktivasi serta keadaan di sekeliling. Karbon aktif ini telah disediakan daripada kulit kelapa sawit yang dijalankan dengan pengaktif kimia, masa dan suhu aktivasi yang berbeza. Kulit kalapa sawit telah dijalankan melalui proses dua peringkat, iaitu proses kimia aktivasi dijalankan terlebih dahulu daripada proses masa dan suhu aktivasi dijalankan. Peratusan air hilang, kelembapan dan debu telah memberikan peratusan yang tinggi apabila pengaktif kimia tinggi serta masa dan suhu aktivasi rendah. Daripada mikrograf yang diambil, adalah terbukti wujudnya liang-liang pada sample-sampel terbabit apabila suhu aktivasi meningkat. FTIR spectra memberi kumpulan berfungsi untuk karbon aktif. Proses jerapan metalene biru memberikan keputusan untuk perbandingan kepekatan dan masa goncang. Langmuir Isotherm menunjukkan bahawa 4.5 M H<sub>3</sub>PO<sub>4</sub> dan 400 °C suhu aktivatisi adalah keadaan yang bersesuaian untuk meyediakan karbon aktif dan begitu juga persamaan 'pseudo-second-order'adalah sesuai untuk mendapat keputusan yang bagus.



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#### **CHAPTER 1**

#### INTRODUCTION

### 1.1 Introduction

Activated carbon is a processed carbon material with a highly developed porous structure and a large internal specific surface area (Jankowska *et al.*, 1991). It is among the most promising adsorbents when adsorption of traces of gases or vapors is considered (El-Sayed and Bandos, 2001).

Activated carbon is produced from organic based materials such as coconut shells, palm-kernel shells, wood chips, sawdust, corn cobs, seeds etc. The raw material is carbonized to obtain the char or carbonaceous material, which is activated to yield the highly porous final product. Typically, surface areas ranging from 500-1400 m<sup>2</sup>/g is obtained for the activated material (Dubinin, Plavnik and Zevarina, 1964). The adsorption properties, specially regarding to polar compounds, are controlled by their surface chemistry (Rodriguez-Reinoso & Molina-Sabio, 1998).



Although activated carbons are mainly built of graphite layers with slit-shaped porous structure, the carbonaceous matrix also contains heteroatoms such as oxygen, nitrogen, hydrogen, sulfur or phosphorous. Those heteroatoms are present in functional groups analogous to well-known groups studied in organic chemistry. They have a key role on the chemical character of carbon surface, acidic or basic, and on its hydrophobicity (Diaz *et al.*, 2005).

According to Kipling (1965), activated carbon has the ability to adsorb various substances from both gas and liquid phases. The ability of fluid or gas molecules that acts as adsorbate which are able to remain on the surface of the adsorbent is a phenomenon known as adsorption process.



### 1.2 BACKGROUND OF ACTIVATED CARBON

In 1794, charcoal was first applied to the refinement of sugar. Natural forms of activated carbon such as charred animal bones (bone black) were used to refine sugar. Inventors patented a number of methods to improve the clarification, decolorization, and purification power of the bone char. These included improvements in the control of the heat of carbonization, differential oxidation, mixing of bone with, or bitumenous coal, addition of calcium phosphate to carbonized sugar, the packing of various clays upon the bone char in the retorts, complexing with various binders, acidulation. By 1901, scientists had developed ways to synthesize activated carbon from coal that had equivalent or superior adsorptive and decolorizing capacity to bone black. These methods were soon introduced to the US, used for removal of taste and odor from municipal water supplies in the US began about 1929 (Jankowska *et al.*, 1991).

During World War I, Nikolai Zelinski, a professor of Moscow University, was the first to suggest the use of active carbon as the adsorption medium in gas masks. These carbons have found commercial application in the adsorption of gases and vapours. (Jankowska *et al.*, 1991).

Active carbons are widely used as adsorbents of waste gases and vapours. Removing CS<sub>2</sub> from air, the purification of sugar syrup, treatment of potable water and waste waters, air conditioning devices and vacuum technology, sorption pumps, adsorption gas chromatography, purification of blood and adsorption of toxins from systematic fluids, are the examples of applications activated carbons used as. Active



carbon are also finding increasing application as catalyst supports as well as materials for electrodes in chemical sources of electricity. The ever-increasing hazards of environmental pollution open new important prospects for active carbon (Jankowska *et al.*, 1991).

## 1.3 OBJECTIVES OF STUDY

The objectives of this study are:

- To prepare activated carbon from oil palm shell using H<sub>3</sub>PO<sub>4</sub> as chemical activator.
- 2. To study the physical and chemical character of activated carbon prepared.

### 1.4 SCOPE OF STUDY

This study is focus on the use of oil palm shell as a raw material to prepare activated carbon, chemically activated with phosphoric acid using two-stage activation process.



#### **CHAPTER 2**

### LITERATURE REVIEW

Activated carbon is a highly porous material which has various applications in adsorption of both gases and solutes from aqueous solution. It has been wellestablished the fact that activated carbons have over many years been widely used for the separation of gases, recovery of solvents, removal of organic pollutants from drinking water and as well as catalyst support. Coals and lingo-cellulose materials are commonly used as the starting raw material for preparing activated carbons. Recently numerous attempts to prepare activated carbons from solid wastes have also been undertaken (Garcia-Garcia *et al.*, 1994).

### 2.1 PROPERTIES OF ACTIVATED CARBON

#### 2.1.1 Physical Properties

Both the porous structure (texture) and the chemical nature of the surface of active carbon are significantly related to its crystalline constitution. The graphite-like microcrystalline structure is the basic structural unit of active carbon, as in the case of carbon black. The ordering of carbon atoms in an elementary microcrystallite indicates considerable similarity to the structure of pure graphite, the crystals of which consist of parallel layer of condensed regular hexagonal rings spaced 0.335 nm apart. Such interlayer spacing is diagnostic of interaction by means of van der Waals forces. The scheme of arrangement of the carbon atoms in a crystal of graphite is presented in Figure 2.1 (Jankowska *et al.*, 1991).

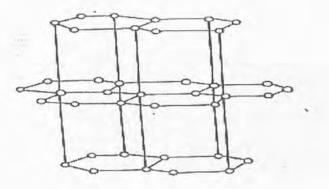


Figure 2.1 Carbon atom arrangements in a graphite crystal (Jankowska et al., 1991).

The formation of the crystalline structure of active carbon begins early during the carbonization process of the starting material. Thus sets of condensed aromatic rings of various numbers, which are the nascent centers of graphite-like microcrystallites, are formed. Although their structure resembles that of graphite, there exist some deviations from that structure. Thus, among other things, the interlayer distances are unequal in crystals of active carbon and range from 0.34 to 0.35 nm. Again, the orientations of the respective layers generally display deviations. Such deviations from the ordering characteristic of graphite, called a turbostratic structure, are illustrated in Figure 2.2. Disordering of the crystal lattice may be caused to a considerable degree both by its defects (e.g. vacant lattice sites) and the presence of built-in heteroatom. It results from the kind of the raw material used, and the nature and quantity of its impurities as well as the methods and conditions of the production processes of the active carbon. In addition to disordering within the internal structure of the crystallites, the second significant difference between the structure of graphite and that of active carbon lies in the quantity and mutual orientation of the crystallites. The range of order of the crystal structure, which is very high in the case of graphite, is limited for active carbons (Jankowska *et al.*, 1991).

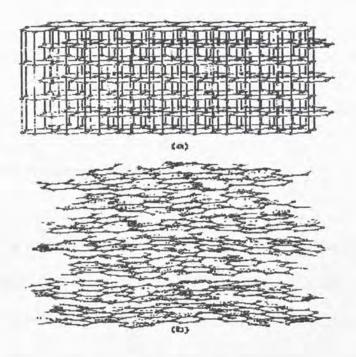


Figure 2.2 Schematic diagrams comparing a) Three-dimensional lattice, with b) A turbostratic structure (Bokros, 1969).

Besides that, activated carbons also have a very wide range of pore sizes, from the Ångstrom scale of micropores to the micrometer scale of macropores. They are used in various applications depending on their porous properties. For example, activated carbons with many micropores are used for gas adsorption, mesopores are necessary for the adsorption of large molecules, etc (Nakagawa *et al.*, 2003).



Pore development by activation reactions is governed by the diffusivity and reactivity of the activation agents. Mesopores develop under the conditions where micropore development is restricted, and vice versa. These conditions are determined by complex combinations of the activation temperature, activation agents, concentrations of activation agents, and the pore structures of the activated materials, which influence the diffusivity and/or reactivity of the activation agents. This knowledge is very important not only to decide on the activation conditions, but also to deduce methods for developing many pores in activated carbons (Yoshizawa *et al.*, 1997).

#### 2.1.2 Chemical Properties

Active carbons usually contain apart from carbon itself, which constitutes generally over 90 per cent of their mass, various types of non-carbon additives differing from each other both in composition and their mode of combination, particularly with the surface. Two basic types of additive can be distinguished: mineral substances occluded in the pores of active carbon (after its combustion) and denoted as ash and heteroatoms chemically combined with carbon atoms mainly at the edges and corners of elementary planes of graphite crystallites.

Ash consists mainly of oxides and, in smaller amounts, of sulphates, carbonaceous, and other compounds of iron, aluminium, calcium, sodium, potassium, magnesium and many other metals. Depending upon the type of raw material, it may comprise different and often fairly large quantities of silicon.



The surface chemistry of the activated carbon is strongly influenced by the activation process and subsequent chemical treatment (Pradhan and Sandle, 1999). The surface contains abundant oxygen and hydrogen groups which can decompose to  $CO_2$  and water (Puri, 1966). Other surface oxide complexes that have been found include phenols, carbonyl, lactones, carboxylic acid, and quinines (McCreey, 1991; Pradhan and Sandle, 1999). The abundance of surface complexes causes activated carbon to be a good absorber of many gases and aqueous chemicals. The non-selective absorption of many chemicals makes activated carbon an excellent absorber in poisoning or environmental contamination (Cooney, 1980). Non-selectivity is less desirable when a specific chemical is to be removed from a process stream. Activated charcoal is impregnated with potassium carbonate for efficient catalytic reduction of  $CO_2$  gas, and debittering of citrus peels is mostly accomplished through the use of ion exchange resin (Grohmann *et al.*, 1999).

Oxygen surface compounds are usually divided into two main types: functional groups of acidic nature (undergoing neutralization by bases) and basic groups which may be neutralized by acids. The acidic group is exemplified schematically in Figure 2.4 (Jankowska *et al.*, 1991).

### 2.2 ELEMENTARY ASPECTS OF ADSORPTION

Adsorption phenomenon happens when the carbon is surrounded by atoms and is held in position by the equilibrium between the attractive and repulsive forces exerted by the surrounding atoms. The atoms in the surface, however, are uniquely situated in that only on one side there are atoms and severe distortion of the interatomic forces and spacing occur, with the result that attractive forces are developed when a molecule approaches the surface. The approaching molecule is held for a finite time before its kinetic energy causes it to leave (Davidson *et al.*, 1968).

According to Cheremisinoff (1986), physisorption occurs when there is an energy differences and/or electrical attractive forces (weak van der wall's forces), the adsorbate molecules become physically fastened to the adsorbent molecules. This phenomenon is multilayered which is each molecular layer forms on top of the previous layer with the number of layers being proportional to the contaminant concentration. Physisorption process is reversible and the reversibility of physisorption is dependent on the strength of attractive forces between adsorbate and adsorbent. If these forces are weak, desorption is occurred.

When a chemical compound is produced by the reaction between an adsorbed molecule and an adsorbent, chemisorption is occurred. This process is differ from physisorption process whereby this process is one molecule thick or a single layer of chemically bound adsorbate at the surface and it is irreversible, because energy is required to form the new chemical compound at the surface of the adsorbent, and energy would be necessary to reverse the process.

### 2.2.1 Factors which influence adsorption at carbon/liquid interface

Adsorption capacity of activated carbons depends on:

- a) Physical and chemical characteristics of the adsorbent (carbon);
- b) Physical and chemical characteristics of the adsorbate (the food or beverage);



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