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JUDUL: ~~CHARACTERIZATION AND PREPARATION ACTIVATED CARBON~~
PREPARATION AND CHARACTERIZATION OF ACTIVATED CARBON FROM OIL PALM SHELLS USING $ZnCl_2$ AS DEHYDRATING AGENT

IJAZAH: Sarjana Muda Sains

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PREPARATION AND CHARACTERIZATION OF ACTIVATED CARBON FROM OIL PALM SHELLS USING $ZnCl_2$ AS DEHYDRATING AGENT

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2006

DECLARATION

I hereby declare that this is my own work with the exception for quotations and references, which have been properly acknowledged.

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ABSTRACT

In this study, activated carbon was prepared from oil palm shell through the preparation process consisted of semi-carbonization followed by zinc chloride impregnation and activation. The semi-carbonization and activation temperature ranged from 400°C to 600°C. The activated carbons were characterized by its percentage of yield, moisture content, ash content, pH value, porosity, adsorption of methylene blue and organic functional group. Moisture content increased as $ZnCl_2$ concentration and activation temperature increased. However, ash content increased as $ZnCl_2$ concentration increased, but activation temperature decreased. It was found that AC 4 which was prepared at the semi-carbonization 400°C in 1h followed by impregnated with 5M $ZnCl_2$ and activation temperature 400°C in 45 minute was the best activated carbon among the entire sample because of its highly methylene blue sorption ability. It was found that acid treatment had little effect on adsorption capacity. The analysis of Langmuir adsorption isotherms obtained proved that the adsorption involved a single layer adsorption at the acidic solution. The methylene blue adsorption on all carbons follows a pseudo-second-order kinetics.

ABSTRAK

Dalam kajian ini, karbon aktif telah disediakan daripada tempurung kelapa sawit melalui proses penyediaan yang terdiri daripada proses setengah karbonisasi diikuti dengan impregnasi zink kloride dan pengaktifan. Suhu untuk proses setengah karbonisasi dan pengaktifan adalah dalam lingkungan 400°C hingga 600°C. Karbon aktif akan dicirikan dengan penentuan peratus hasil, kelembapan, kandungan abu, pH, keadaan berliang, jerapan metilena biru dan kumpulan organik berfungsi. Kandungan kelembapan memberikan peratusan yang tinggi apabila kepekatan zink kloride dan suhu pengaktifan meningkat. Manakala, kandungan abu bertambah apabila kepekatan zink kloride meningkat dan sebaliknya untuk suhu pengaktifan. AC 4 yang disediakan pada suhu setengah karbonisasi 400°C dalam 1h diikuti dengan 5M zink kloride dan suhu pengaktifan 400°C dalam 45 minit adalah karbon aktif yang terbaik antara semua sample. Kajian ini mendapati bahawa rawatan asid membawa sedikit kesan ke atas kapasiti jerapan. Analisis untuk isoterma jerapan Langmuir yang diperolehi membuktikan bahawa jerapan itulah melibatkan satu lapisan pada larutan berasid. Jerapan metilena biru ke atas semua karbon adalah dicontohi dengan susunan palsu kedua kinetik.

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LIST OF SYMBOLS / ABBREVIATION

M	Molarity (in unit $molL^{-1}$)
mg	Milligram
g	Gram
mm	Millimeter
µm	Micrometer
%	Percentage
°C	Celsius degree
ml	Milliliter
L	Liter
ppm	Part per million
nm	Nanometer
$ZnCl_2$	Zinc Chloride
PAN	Polyacrylonitrile
KBr	Potassium Bromide
HCl	Hydrochloride Acid
SO_2	Sulphur Dioxide

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CHAPTER 1

INTRODUCTION

1.1 INTRODUCTION

Carbon has been one of the most magnificent elements which have revolutionized materials science field. Carbon provides excellent properties of the materials for a large spectrum of industrial applications (Pierson, 1993).

Activated carbon consists mainly of carbon and is produced from every carbonaceous material. Carbonaceous materials used to be classified into two groups. They were known as "amorphous carbon" and "crystalline carbon" (i.e. graphite and diamond). Activated carbon was included in the group known as "amorphous carbon" (without a regular shape) structure (Smisek and Cerny, 1970). Besides that, activated carbon also consist other elements such as hydrogen, oxygen, sulphur and nitrogen (Jankowska *et al.*, 1991).

According to the conceptions of Bernal and confirmed by many authors, graphite is one of the two crystalline modifications of pure carbon (Smisek and Cerny, 1970). The

significant different between the structure of graphite activated carbon was lies in the quantity and mutual orientation of the crystallites. In addition, the second significant was the kind of the raw material used, the nature and quantity of its impurities as well as the methods and conditions of the production processes of the activated carbon (Jankowska *et al.*, 1991). Therefore, their dimensions depended on the type of activated carbon and influenced by the conditions of production.

1.2 HISTORY

Porous carbons, especially activated porous carbons, constitute one of the most important types of industrial carbons and have been in use for thousands of years. Activated carbon was first known to treat water over 2000 years ago. However, the rational use for the commercial use of active carbon began at the end of the 18^{th} century. The Swedish chemist, Karl Wilhelm Scheele was the first discovers the phenomenon of adsorbing of gases on charcoal in 1773. Then activated carbon was used to decolorized sugar and this lead to the first industrial application of charcoal as a decolorizing agent in the sugar industry in England in 1794 (Jankowska *et al.*, 1991). Bohemia. J. Wunsch found that the decolorizing power of the reactivated material was much higher by using zinc chloride as an activation agent (Smisek and Cerny, 1970).

During the World War I, granular activated carbon was first developed and gas masks were started produced by activating woodchips with zinc chloride. This kind of mask used to protect the soldiers' respiratory tracts throughout the world to the present

day. At the same time, coconut shell is the famous raw materials for production of activated carbon. Coconut shells which activated with zinc chloride yielded an activated carbon of high mechanical strength and adsorptive power for the adsorption of gases and vapours (Smisek and Cerny, 1970). The zinc chloride process is still one of the most widely used for the production of activated carbon.

Nowadays, activated carbons are highly useful in overcoming both the pollution and waste problems simultaneously. Activated carbons are generally used for such applications because of the excellent properties for the adsorption, separation, and decomposition of harmful gases and solutions in the environment. Beside that, activated carbon has several important uses including removal of tastes and odours from domestic and industrial water supplies, pharmaceuticals, catalyst support and in the waste water treatment (Manocha, 2003).

1.3 RESEARCH OBJECTIVES

The objectives of this study are:

a. To prepare activated carbon from oil palm shells using $ZnCl_2$ as dehydrating agent.
b. To study the physical and chemical properties of the activated carbon prepared.

1.4 SCOPE OF STUDY

This project focuses only on the preparation of activated carbon by oil palm shells as a raw material using $ZnCl_2$ as dehydrating agent. Several properties will be analyzed; which include ash, moisture, pH and sorption ability. Instruments such as FTIR and SEM will be used to study the product.

CHAPTER 2

LITERATURE REVIEW

Activated carbons have over many years been widely used for multipurpose in many fields. Carbon's materials are commonly used as the starting raw material for preparing activated carbons. Recently, numerous attempts to prepared activated carbons from solid wastes have also been undertaken. There is a clear need for a cheaper, more cost effective, product which could possibly be used on waste materials. So, the development of methods to re-use waste materials is greatly desired and the production of activated carbons from wastes offers a promising future (Namasivayam and Kardivelu, 1997).

2.1 ACTIVATED CARBON

Activated carbons are carbons of highly microporous form with both high internal surface area and porosity (Figure 2.1) (Anuar *et al.*, 2004). The presence of micropore substantially influences its sorption properties because amount adsorbed on the macropore surface is negligible in comparison to that for the micropores. Therefore, characterization of the activated carbons has become one of the most important problems in adsorption technology (Avom *et al.*, 1997). The volume of pores of the activated

carbon usually exceeds 0.2ml g^{-1} and the internal specific surface area is generally greater than 400m^2 g^{-1}. The width of the pores ranges from 0.3 to several thousand nanometers (Jankowska *et al.*, 1991).

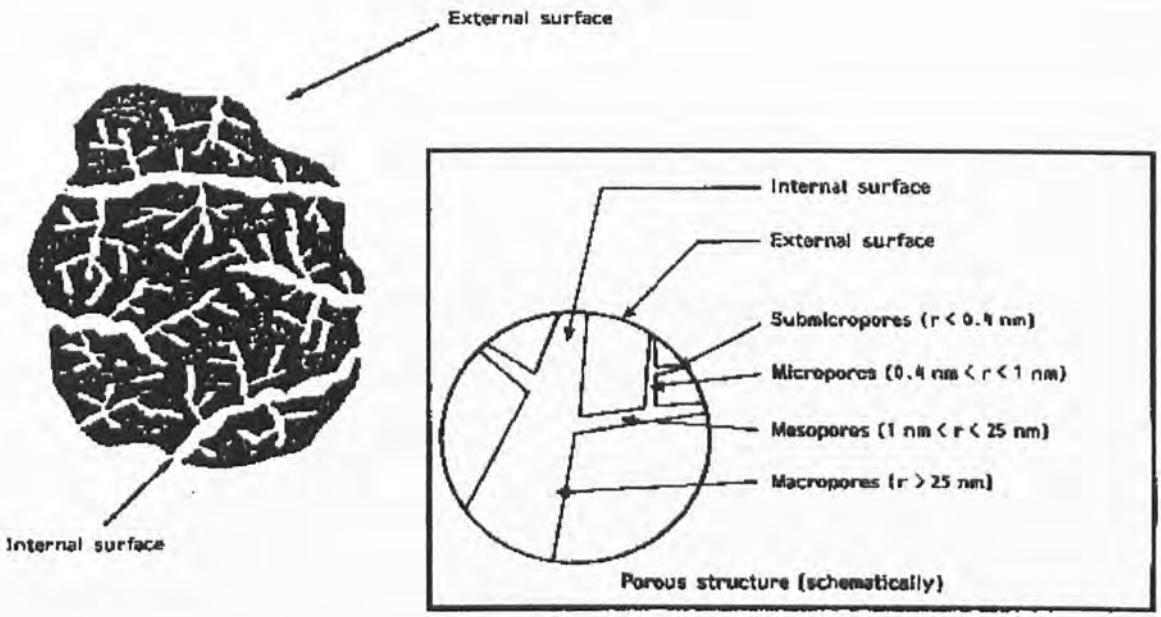

Figure 2.1 Schematic activated carbon model

In the activated carbon, the macropores provide a passageway to the interior of the particle into the micropores but do not contribute substantially to the particle surface area. The mesopores for transportation and the micropores are responsible for the large surface area of activated carbon particles created during the activation process. It is the micropores where adsorption largely takes place.

Adsorptive capacity is related to the total surface area which does not affect the adsorption rate. The increase in porosity is normally accompanied by an increase in

specific surface area and this by an increase in the magnitude of the selective adsorption (Alexander, 1968). Adsorption in these pores is due to the effect of the dispersion component of the van der Waals forces because its ability to separate individual entities from a mixture of adsorbates of different chemical nature. Therefore, activated carbon is known as a universal adsorbent of high adsorptive power (Smisek and Cerny, 1970).

2.2 CLASSIFICATION OF ACTIVATED CARBON

Basically, activated carbon comes in two variations: Powder Activated Carbon (PAC) and Granular Activated Carbon (GAC). Specific processes have been developed to produce these two types of activated carbon.

2.2.1 Physical structure of Activated Carbon

a. Powdered activated carbon

Traditionally, activated carbons are made in particular form as powders which is less than 100mm in size with an average diameter between 15-25μm (Manocha, 2003). Thus they present a large internal surface with a small diffusion distance. Powdered activated carbons (PAC) are used for adsorption from solution and it is usually produced by chips of wood charcoal, or activating lump material, or wood in the form of saw dust with a solution of zinc chloride, and then grinds to be activated product. Besides that, this group

of carbon is also used for removal of colouring matter from solutions and medicinal carbon (Smisek and Cerny, 1970).

b. Granulated activated carbon

Granular activated carbons are larger than the powdered activated carbons and present a smaller external surface. This group of carbons are preferably used for adsorption of gases (Samuel, 1970) and therefore known as gas-adsorption carbons (Smisek and Cerny, 1970). It can be prepared in the form of crushed granules (coal or shell) and usually used for water treatment, deodorization and separation of components of flow system.

2.2.2 Chemical Structure of Active Carbon

Activated carbon contains two types of admixtures. One of them is chemically bonded elements, such as oxygen and hydrogen. These elements are derived from the starting material and remain as a result of imperfect carbonization or otherwise chemically bonded to the surface during activation. These elements are chemically bonded to form functional groups such as carbonyl, carboxylic, phenol, lactone, quinine and ester (Figure 2.2) (Faust and Aly, 1986).

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