DEVELOPMENT AND CHARACTERIZATION OF ACTIVATED CARBON PREPARED FROM PAPAYA SEED BY H_3PO_4 ACTIVATION

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ABSTRACT

Preparation of activated carbons from papaya seed, a carbonaceous agricultural waste product, with different impregnation ratio of H_3PO_4 by using two-stage self-generated atmosphere method were studied. The papaya seeds were carbonized in a muffle furnace at a constant temperature of 200°C for 15 min followed by activation at a desire temperature and time. Several physical and chemical characteristics (moisture, ash, pH, functional groups, surface structure and surface area) of prepared activated carbons were studied. Semi-carbonized at 200°C for 15 min followed by activation at 500°C for 45 min with impregnation ratio of 2.0 yielded a product with the highest BET surface area which was 895.7 m^2g^{-1}. While the sample which prepared by semi-carbonization at 200°C for 15 min followed by activation at 500°C for 30 min with impregnation ratio of 1.5 yielded the highest micropores surface area and iodine number which were 822.8 m^2 g^{-1} and 870 m^2 g^{-1} respectively. Besides that, the methylene blue adsorption showed that all activated carbons fitted well in Langmuir model which yielded the q_m between 53 mgg^{-1} and 97 mgg^{-1}.

ABSTRAK

Penyediaan karbon teraktif daripada biji betik, sesuatu hasil buangan pertanian, dan diolah oleh nisbah impregnasi H_3PO_4 yang berlainan dengan menggunakan kaedah dua-pentas penjanaan atmosfera sendiri telah dikaji. Biji betik telah dikarbonkan separa dalam relau pada suhu malar 200°C dalam masa 15 min diikuti dengan proses pengaktifan pada suhu dan masa tertentu. Beberapa ciri fizikal dan kimia (kelembapan, abu, pH, kumpulan berfungsi, struktur permukaan dan luas permukaan) bagi karbon teraktif telah dikaji. Proses pengkarbonan separa pada suhu 200°C dalam masa 15 min diikuti dengan proses pengaktifan pada suhu 500°C dalam masa 45 min dengan menggunakan nisbah impregnasi 2.0 telah menghasilkan sesuatu produk yang mempunyai luas permukaan BET terbesar iaitu 895.7 m^2 g^{-1}. Manakala sampel yang disediakan melalui proses pengkarbonan separa pada suhu 200°C dalam masa 15 min diikuti dengan proses pengaktifan pada suhu 500°C dalam masa 30 min dengan menggunakan nisbah impregnasi 1.5 menghasilkan luas permukaan micropor terbeasr dan iodin nombor yang tertinggi iaitu 822.8 m^2 g-1 dan 870 m^2 g^{-1} masing-masing.

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%	percentage
Å	angstroms
$AlCl_3$	alluminium chloride
BET	brunauer, emmett & teller
CH	hydrocarbon
cm	centre meter
CO	carbon monoxide
CO_2	carbon dioxide
°C	degree Celsius
FTIR	fourier transform infrared spectrometer
g	gram
hr	hour
HCl	hydrochloride acid
H_20	hydrogen
H_2SO_4	sulphuric acid
H_3PO_4	Phosphoric acid
Kg	kilogram
KOH	potassium hydroxide
K_2CO_3	potassium carbonate
L	Liter
m^2	meter squared
ml	milliliter
min	minute
$MgCl_2$	magnesium chloride
M	molarity
nm	nanometer
NaOH	sodium hydroxide
Na_2CO_3	sodium carbonate
SEM	Scanning Electron Microscopy
x	initial mass of impregnated sample
y	mass of the prepared activated carbon

CHAPTER 1

INTRODUCTION

1.1 Background

The solution of ecological problems has recently gained a global significance on account of the increasingly growing pollution of the air and water resources. The need for purification of waste from various pollutants requires the development of new methods for the production of highly efficient adsorbents (Girgis and Ishak, 1999). In this respect, carbon adsorbents have attracted special attention.

Activated carbon, an amorphous carbon-based material, is a unique and versatile adsorbent because of its high porosity, extended surface area, high degree of surface reactivity and favorable pore size distribution (Alfonso *et al.*, 1996). It has a very complex structure, with pore sizes ranging from micropores (< 20 Å slit width) to macropores (> 500 Å slit width), and has a variety of surface groups, impurities, and irregularities (Ismadji *et al.*, 2004). Therefore, it is one of the most important microporous adsorbents from an industrial view of point.

The rational use of active carbon for industrial purposes was started at the end of the 18 century. In the year 1773, Karl Wilhelm Scheele, an apothecary by profession, was the first to discover the phenomenon of adsorption of gases on charcoal. In the years 1900-1901 R.V. Ostrejko was granted two patents. The essence of the invention described in the first patent consisted in heating a vegetable material with metal chlorides, and that of the second patent in activating charcoal with carbon dioxide and stream at red heat (Helena *et al.*, 1991). The first plant of activated carbon name Chemische Werke was built in 1909 which manufactures a commercial scale, powered active carbon with the trade name Epoint. It was primarily intended as decolorizer in manufacture of sugar. (Smisek and Cerny, 1970).

Activated carbon is manufactured by the carbonization and activation of carbonaceous solid precursor: both naturally occurring and synthetic. In order to save cost, agricultural by-products have been recommended for the preparation of activated carbons such as olive seeds, guava seeds, acorn, palm tree cobs, rubber wood, coffee ground, sugarcane bagasse, rice hulls, coconut shells, and so on. They are exists in large amount and represents a solid pollutant to the environment. The preparation of activated carbon from agricultural by-product could increase economic return and reduce pollution (Youssef *et al.*, 2005).

In order to yield more qualitative and quantitative sample various concentration were used to activate the papaya seed. Optimum carbonization and activation parameters are important to yield the activated carbon that is high in adsorption capacity.

1.2 Research Objective

The objectives of the study were:

a. To prepare activated carbon from papaya seeds using two stage self-generated atmosphere method using H_3PO_4 as the activating agent.
b. To physically and chemically characterize the prepared activated carbon.
c. To study the methylene blue adsorption isotherm of activated carbon in aqueous solution.

1.3 Scope of Study

This project was focus on the study of preparation of activated carbon from papaya seeds which impregnated with various impregnation ratio, activation temperature and time. The process parameters were analyzed based on product yield, iodine number, the methylene blue adsorption capacity and the surface area by using BET method.

CHAPTER 2

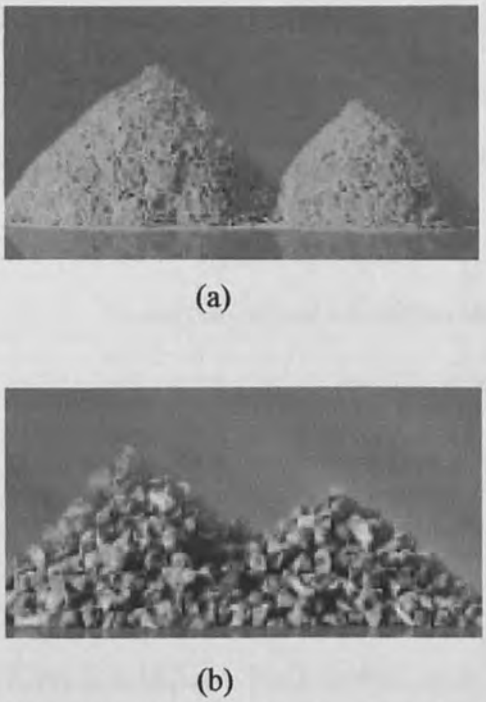
LITERATURE REVIEW

2.1 Activated Carbon

Activated carbons are carbons of highly microporous form with both high internal surface area and porosity (Tsai *et al.*, 1997). It is a versatile adsorbent because of its good adsorption properties which can be produced from a variety of raw materials. It is manufactured by the carbonization and activation of carbonaceous materials.

Activated carbons can be produced from a number of precursor materials including coal, wood, polymer and agricultural by-product. Recently, specials emphasis on the preparation of activated carbons from several agricultural by-products has been given due to the growing interest in low cost activated carbons from renewable, safe, copious supplies (Castro *et al.*, 2000). Many activated carbon researches had been using from agricultural waste, such as olive seed (Stavropoulos and Zabaniotou, 2005), coffee grounds (Namane *et al.*, 2005), apricot stones (Youssef *et al.*, 2005), guava seeds (Rahman and Saad, 2003), peanut hulls (Girgis *et al.*, 2002), apple pulp (Suarez-Garcia *et al.*, 2001), cotton stalks (Girgis and Ishak, 1999), nut shells (Toles *et al.*, 1997), rice husk (Alfanso *et al.*, 1996). Papaya seed is a kind of agricultural by-product which currently no economic value. It is black in color and round seeds encased in a gelatinous-like substance.

Active carbon can be divided into two groups which are powdered and granular as shown in Figure 2.1. Powdered carbons are used for adsorption from solution. Disintegration into fine particles enhances the rate of establishment of adsorption equilibrium. This group of carbons includes decolorizing, removal of coloring matter from solutions, and medicinal carbon (Smisek and Cerny, 1970). On the other hand, granulated active carbons are used mainly for adsorption of gases and vapors, therefore known as gas-adsorption carbons. Granular active carbons (GACs) are generally considered more versatile than the powdered carbons due to their regenerability.


(a)

(b)

Figure 2.1 (a) Powdered activated carbon and (b) Granular activated carbon.
(Source: Norit Activated Carbon, 1918)

2.2 Properties of Activated Carbon

Activated carbon contains wide range of micropore and mesopore (Wang *et al.*, 2005). It can meet many diverse needs because of the adsorptive properties which are the porous nature can be developed in various forms by appropriate changes in the manufacturing process.

2.2.1 Physical Structure of Activated Carbon

The structure of activated carbon is complex and it is basically composed of an amorphous structure and a graphite-like microcrystalline structure. The graphitic structure is important from the capacity point of view as it provides "space" in the form of slit-shaped channel to accommodate molecules. Because of the slit shape the micropore size for activated carbon is reported as the micropore half-width (Do, 1998). However, the formation of the crystalline structure of activated carbon begins early during the carbonization process of the starting material (Helena *et al.*, 1991).

The arrangement of carbon atoms in the graphitic structure is similar to that of pure graphite. The layers are composed of condensed regular hexagonal rings and two parallels layers are separated with a spacing of 0.335 nm which is shown as the Figure 2.2 and Figure 2.3 (Do, 1998). Such interlayer spacing is diagnostic of interaction by means of van der Waals forces. The distance between two adjacent carbon atoms on a layer is 0.142 nm. Each carbon atom bonds with the three adjoining ones by means of covalent bonds, and the fourth delocalized π-electron may more freely in a system of conjugated double bonds of condensed aromatic rings (Helena *et al.*, 1991).

The graphitic unit in activated carbon usually is composed of about 6-7 layers and the average diameter of each unit is about 10 nm. The size of the unit can increase under the action of graphitization and this is usually done at very high temperature (>1000 °C) and in an inert atmosphere. Although the basic configuration of the graphitic layer in activated carbon is similar to that of pure graphite, there are some deviations, for example the interlayer spacing ranges from 0.34 nm to 0.35 nm. The orientation of the layers in activated carbon is such that the turbostratic structure is resulted. Furthermore, there are crystal lattice defect and the presence of built-in heteroatoms. The linkage between graphite units is possible with strong cross linking. The interspace between those graphite units will form pore network and its size is usually in the range of mesopore and macropore (Do, 1998).

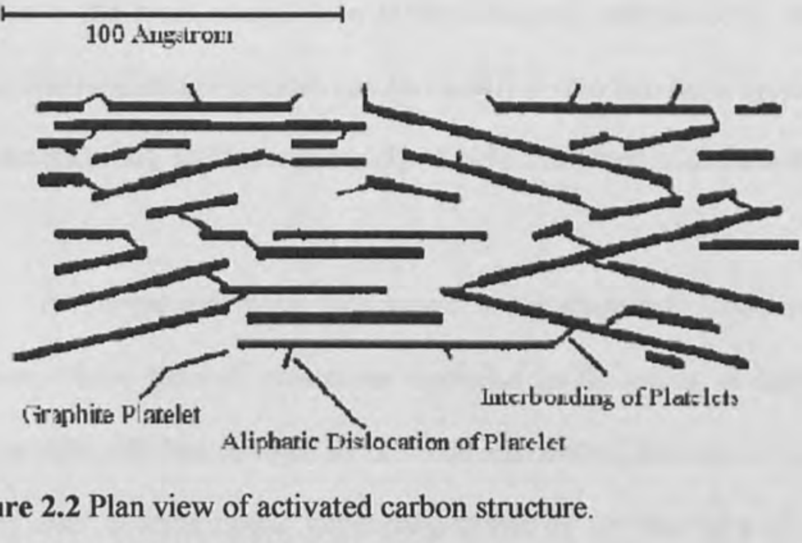


Figure 2.2 Plan view of activated carbon structure.
(Source: Norit Activated Carbon, 1918)

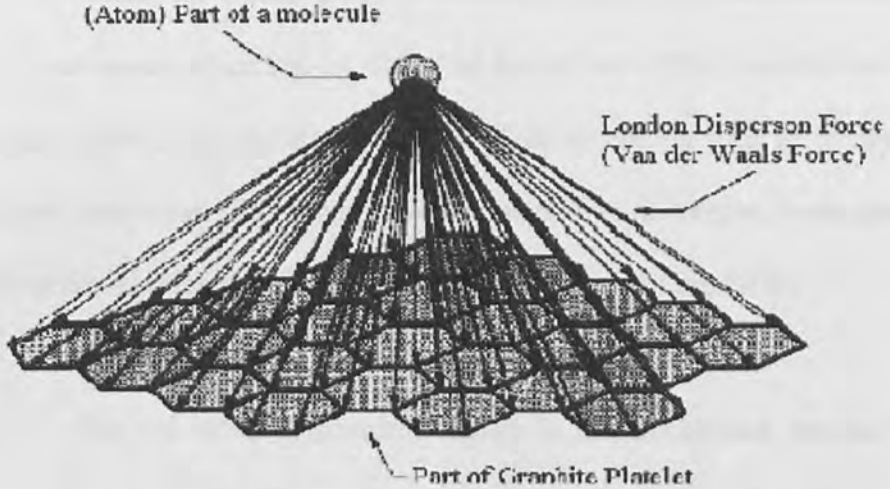


Figure 2.3 Hexagonal graphic crystallites structure of activated carbon.
(Source: Norit Activated Carbon, 1918)

2.2.2 Chemical Properties of Activated Carbon

Carbon is the main composition in the activated carbon which composed about 87-97%. The remaining elements can be classified into two basic types which are mineral substances or ash and heteratoms chemically combined with the carbon skeleton.

A mineral substance such as ash is not chemically combined with the carbon surface. These mineral substances occluded in the pores of active carbon after its combustion and then denoted as ash. The ash content in various types of active carbon varies over a wide range, depending primarily on the type of raw material. Ash consists mainly of oxides and in smaller amounts of sulphates, carbonates, and other compounds of iron, aluminium, calcium, sodium, potassium, magnesium, and many other metals. However, it can remove by leaching with acids such as hydrochloric or hydrofluoric acids (Helena *et al.*, 1991).

While, the second types of remaining elements in active carbon are combined with the atoms of carbon by chemical bonds and differ from the ash in forming an organic part of the chemical structure of active carbon such as carbonyl, carboxylic, phenol, and ester. This type of elements consists of oxygen, hydrogen, sulphur, and halogens of which the important is oxygen (Guo and Lua, 1998).

The pH value of activated carbon is a measurement whether it is acidic or basic. Too high pH indicates too much contaminant. Too low pH, especially for acid washed activated carbon, means that the acid has not been properly rinsed away. Most activated carbon is specified for a pH of 6-8 (Srinivasakannan and Mohamad, 2004).

2.2.3 Physical Properties of Activated Carbon

Surface area is the most important physical property of activated carbon. Typically active carbon have surface areas from 600-1200 m^2g^{-1} with some reported as high as 3000 m^2g^{-1} (Davidson *et al.*, 1968). It has a large volume of very small pores that creates a large surface area. For specific applications, the surface area available for adsorption also depends on the molecular size of the adsorbate and the pore diameter of the active carbon (Guo and Lua, 1998). In general, the finer the particle sizes of an active carbon, the better the access to the surface area and the faster the rate of adsorption kinetics. Srinivasakannan and Mohamad (2004) used the iodine number to estimate the rough surface area of activated carbon. Iodine number is defined as the milligrams of iodine adsorbed per grams of carbon. It indicates the porosity of the activated carbon. Thus, iodine number represents the surface area which is contributed by the pores.

2.3 Porosity

Activated carbon has the ability to adsorb well because of its porosity structure. The porosity structure of active carbon is formed during the process of activation. During activation, the spaces between the elementary crystallites becomes cleared of various carbonaceous compounds and non-organized carbon, and the carbon is also removed partially from the graphitic layers of the elementary crystallites. The resulting voids are termed pores. A suitable activation process causes an enormous number of pores to be formed so that the total surface area of their walls such as internal surface of activated carbon is very large and this is the main reason for its large adsorptive capacity (Smisek and Cerny, 1970). Figure 2.4 shows the structure of activated carbon.

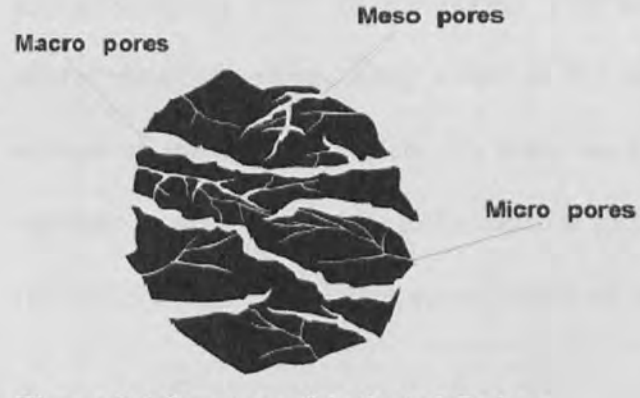


Figure 2.4 Sturcture of Activated Carbon.
(Source: Smisek and Cerny, 1970)

According to the IUPAC Manual of Symbols and Terminology (1972), the porosity of activated carbon is classified by the size of the diameter of the pores, varies from micropores(less than 2 nm), to mesopores (2-50 nm), to macropores (greater than 50 nm) as shown above in Figure 2.4. These values represent the width

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