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UTILIZATION OF WOOD CHIPS AS A SOURCE OF ACTIVE CARBON FOR
REMOVAL OF METHYLENE BLUE FROM AQUEOUS SOLUTION (H₃PO₄)

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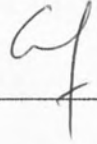


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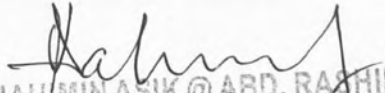
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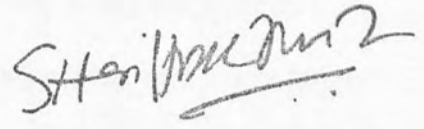
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ABSTRACT

The porosity and adsorption capacity of active carbons depended on the nature of the raw materials. Wood chips have been used as a raw material to produce active carbon. Active carbons have been prepared by chemical activation of wood chips with H_3PO_4 in order to show the effect of variables such as concentration, time and temperature of the resulting active carbons. The adsorption capacity was demonstrated by the isotherms of methylene blue from aqueous solution. Different concentrations of phosphoric acid were applied to all active carbons samples. Phosphoric acid activation was used to produce high porosity and high surface area active carbons. Experiments were conducted in lab scale muffle furnace in a two stage self-generated method covering process parameters such as concentration, activation time and temperature. The resulting active carbons were characterized by the removal of methylene blue from aqueous solution. Adsorption isotherms of methylene blue from aqueous solution onto active carbons were investigated. The Freundlich model is suited to fit the adsorption data according to correlation coefficient. From all active carbon samples, AK8 shows good correlation coefficient with $R^2 = 0.91$. The kinetic adsorption of methylene blue on all carbons follows a pseudo-second-order equation. Surface morphology carried out on the wood chips based active carbons were observed by scanning electron microscope. Infrared spectroscopy showed the chemical structure of the adsorbent materials. As a result, the adsorption capacity of active carbons was differentiate with concentration, time and temperature.



ABSTRAK

Keliangan dan muatan jerapan karbon aktif bergantung kepada kandungan bahan semulajadi karbon tersebut. Serpihan kayu telah digunakan sebagai bahan mentah untuk menghasilkan karbon aktif. Karbon aktif yang disediakan daripada serpihan kayu secara pengaktifan kimia oleh asid fosforik adalah untuk menunjukkan kesan pembolehubah seperti nisbah kepekatan, masa dan suhu terhadap karbon aktif yang terhasil. Muatan jerapan ditentukan dengan menggunakan larutan akueus metilena biru. Semua sampel karbon aktif disediakan dengan kepekatan asid fosforik yang berbeza. Asid fosforik digunakan sebagai agen pengaktifan dalam menghasilkan keliangan dan jumlah luas permukaan bahan penyerap yang tinggi. Eksperimen dijalankan secara pemanasan dua peringkat dalam ketuhar merangkumi parameter seperti kepekatan, masa dan suhu pengaktifan. Karbon aktif yang terhasil dikaji dengan penyerapan larutan metilena biru. Isoterma penyerapan larutan metilena biru terhadap karbon aktif dikaji. Didapati ia sesuai dengan model Freundlich kerana pekali kolerasinya. AK 8 menunjukkan pekali kolerasi yang baik iaitu $R^2 = 0.91$ daripada semua sample. Kinetik penyerapan larutan metilena biru bagi semua sampel karbon aktif adalah pseudo-second-order. Morfologi permukaan karbon dikaji dengan mikroskop sinaran elektron. Spektroskopi inframerah menunjukkan kumpulan berfungsi bahan penyerap. Kesimpulannya, muatan penyerapan karbon aktif adalah berbeza mengikut kepekatan, masa dan suhu.



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LIST OF ABBREVIATIONS

H_3PO_4	Phosphoric acid
H_2O	Water
KBr	Kalium Bromide



LIST OF SYMBOL

cm	Centimeter
m	Meter
nm	Nanometer
kg	Kilogram
g	Gram
mg	Milligram
L	Liter
mL	Milliliter
mg ^l ⁻¹	Miligram per liter
°C	Degree Celsius
%	Percentage
ppm	Parts per million
min	minutes
sec	second



CHAPTER 1

INTRODUCTION

1.1 INTRODUCTION

Active carbon is a porous carbonaceous material, prepared by carbonizing and activating organic substances of mainly biological origin. Its most important property is a very large adsorptive power, which is primarily due to a highly developed porous structure (Smisek and Cerny, 1970) and a large internal specific surface area. It consists of course, principally of carbon 87 to 97%, but it also contains such elements as hydrogen, oxygen, sulphur and nitrogen, as well as various compounds either originating from the raw material used in its production or generated during its manufacture (Jankowska *et al.*, 1991).

Carbon can exist in two forms with crystalline carbon included graphite and diamonds, the other form known as amorphous carbon. Therefore, active carbons were disordered and amorphous forms of carbon (Smisek and Cerny, 1970). The arrangements of carbon atom in a graphite crystal as shown in figure 1.1.



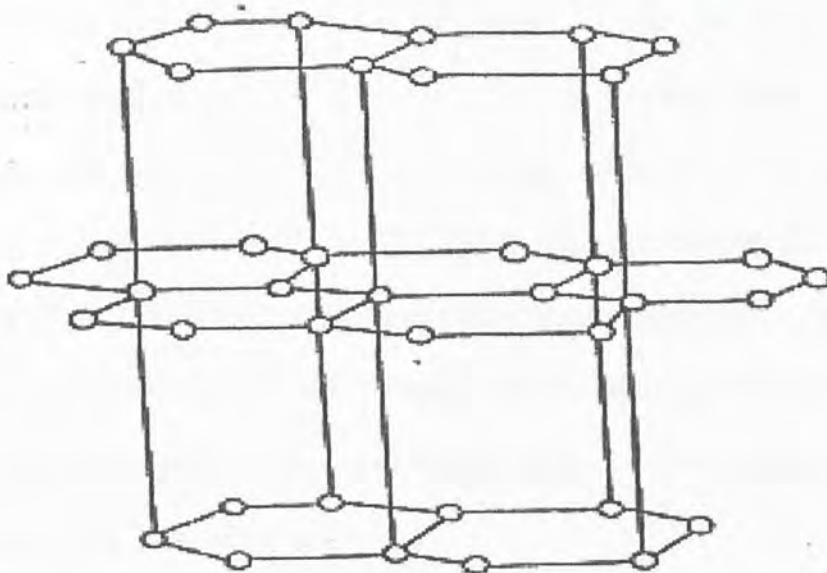


Figure 1.1 Carbon atom arrangements in a graphite crystal (Jankowska *et al.*, 1991).

Active carbon has the availability to adsorb various substances both from the gas and liquid phases (Jankowska *et al.*, 1991). It is this ability to arrest different molecules at the inner surface of active carbon that justified calling it an adsorbent, and a very powerful adsorbent indeed it is. The pore volume of active carbon usually exceeds $0.2 \text{ cm}^3 \text{ g}^{-1}$ but in many instances, it is greater than $400 \text{ m}^2 \text{ g}^{-1}$ but often exceeds this value reaching $1000 \text{ m}^2 \text{ g}^{-1}$ (Jankowska *et al.*, 1991).

1.2 HISTORY

Large quantities of charcoal were produced in the 17th and 18th centuries to feed the furnaces of the new smelting industries and complete deforestation of Europe was only halted by the adoption of coke as a fuel (Davidson *et al.*, 1968). The rational use of

active carbon for industrial purposes was started at the end of the 18th century. According to Jankowska *et al.*, (1991), in 1773, the Swedish Chemist Karl Wilhelm Scheele, an apothecary profession, was the first discover of the phenomenon of adsorption of gases on charcoal. In 1785, the Russian academician Lovits from Saints Petersburg found that charcoal, when immersed in tartaric acid solution, decolorized it by adsorbing the organic contaminants present. This discovery led to the first industrial application of charcoal in the sugar industry in England in 1794, where it was used as a decolorizing agent for sugar syrup.

In the years 1900-1901 R. V. Ostrejko was granted two patents (Jankowska *et al.*, 1991), which were 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 steam at red heat.

The production was undertaken in this plant of new kinds of active carbon in 1911, known as norit and purit, obtained from peat by activation with steam. About the same time as the Raciborz factory was founded, a wood distillation plant was built in Hajnowka (East Poland), initially manufacturing active carbon solely from wood. The processes of the chemical activation of sawdust with zinc chloride were carried out for the first time in 1914 in the Austrian plant in Aussig, and in 1915 in the dyestuff plant of Bayer. The powdered carbons were used at the time chiefly for decolorizing solutions in the chemical and food industries (Jankowska *et al.*, 1991).



The requirement for a gas mask during World War I stimulated interest and research into granular forms of active carbon and durable granular charcoal of high adsorptive capacity soon become available (Davidson *et al.*, 1968). Nikolai Zelinski, is a professor of Moscow University, was the first to suggest the use of active carbon as the adsorption medium in gas masks for the protecting of respiratory tracts. This was prepared mainly from coconut shell, although many other nuts and shells. These have led to the development of new technologies for obtaining granulated active carbons of suporsorbon and benzosorbon type (Jankowska *et al.*, 1991).

Active carbon has become one of the technically most important and most widely used adsorbents because of its large adsorptive capacity, universal adsorption effect, favorable pore size (Smisek and Cerny, 1970). The properties of active carbon are used for purifying gases, separating mixtures, purifying solutions in the food industry, treatment of potable water and wastewater. Active carbons are also finding increasing application as catalyst support materials for electrodes in chemical sources of electricity. The ever-increasing hazards of environmental pollution open new important prospects for active carbons (Jankowska *et al.*, 1991). Its disadvantages is its relatively high costs, because of which adsorbents of inferior properties, but less costly, compete with it successfully in some cases.



1.3 OBJECTIVES

1. To prepare active carbons from wood chips using phosphoric acid as the dehydrating agent.
2. To study the physical and chemical properties of the active carbons.
3. To study the adsorption isotherm of methylene blue in aqueous solution.

1.4 SCOPE OF THE STUDY

This study is focus on the use of wood chips as a source of active carbon production via chemical activation process by using phosphoric acid (H_3PO_4) as activating agent. Experiments were conducted in a muffle furnace in a self-generated atmosphere covering process parameters such as concentration, time and temperature.

CHAPTER 2

LITERATURE REVIEW

The terms charcoal, wood charcoal and active carbon are synonyms for forms of carbon all having marked adsorptive properties; that is, all possess a readily accessible internal surface area (porosity) greatly in excess of the superficial surface (Davidson *et al.*, 1968). It is a high porosity and high surface area material manufactured by the carbonization and activation of coal and lignocellulose materials such as wood chips (Evans *et al.*, 1999), oil palm shell, sawdust, coal, peat, fruit stones and shells (Smisek and Cerny, 1970). Active carbons are useful in adsorption of both gases and solutes from aqueous solution. It has widely applied to environmental applications such as air and gas purification, solvent recovery, the removal of organic pollutants from wastewaters and drinking water (Laszlo *et al.*, 1997) and as a catalyst support. Over the past ten years, the consumption quantity of active carbon used for water treatment and air purification in the more industrialized countries has been increasing dramatically due to environmental issues.



2.1 PROPERTIES OF ACTIVE CARBON

Active carbon is a porous carbonaceous materials which contains pores with surface area that ranges from 600 to over 2000 m²/g. Due to its highly developed porous structure and huge surface area, activated carbon is used as an excellent absorbent primarily to selectively absorb gases, vapors or colloidal solids from liquid or vapor stream. The properties of activated carbon could be categorized as physical properties, chemical properties and adsorptive properties, all of which are very important for the selection and application of activated carbon. The physical properties of active carbon, such as surface area and pore volume, have a little effect on dye adsorption, while the pore size distribution and the surface chemical characteristics play important role (Shaobin *et al.*, 2005).

2.1.2 Physical structure of Active Carbon

Active carbon, together with other types of chars and carbon blacks, form a group of carbonaceous materials of which the structure, and the properties depending on it, are more or less similar to the structure and properties of graphite (Smisek and Cerny, 1970). There are two types of carbon structure which are graphite and turbostratic carbon.

The first type of structure consists of elementary crystalline; these are in two dimensions analogous to graphite (Smisek and Cerny, 1970). The ordering of carbon atoms in an elementary microcrystallite indicates considerably similarity to the structure



of pure graphite, the crystals of which consists of parallel layers of condensed regular hexagonal rings spaced 0.335 nm apart. Such interlayer spacing is diagnostic of interaction by means of van de Waals forces. The length of the carbon-carbon bond in individual layers is 0.142 nm (Jankwoska *et al.*, 1991). The structure of active carbon is less perfectly ordered than that of graphite. However, in that, the parallel planes are not perfectly oriented with respect to their common perpendicular axis, the angular displacement of one layer with respect to another is random and the layers overlap one another irregularly (Smisek and Cerny, 1970).

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 centres of graphite-like microcrystallites are formed. Although their structure resembles that of a crystal 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. The orientation of the respective layers generally displays deviations. Such deviations from the ordering characteristic of graphite are called turbostratic carbon structure are illustrated in figure 2.1 (Jankwoska *et al.*, 1991).

The second type of structure, Riley describes as a disordered, cross-linked space lattice of the carbon hexagons, which results from their deflection from the planes of graphitic layers. This structure is probably stabilized by heteroatom, in the first place by



oxygen, as it has been found in chars prepared from materials of high oxygen content (Smisek and Cerny, 1970).

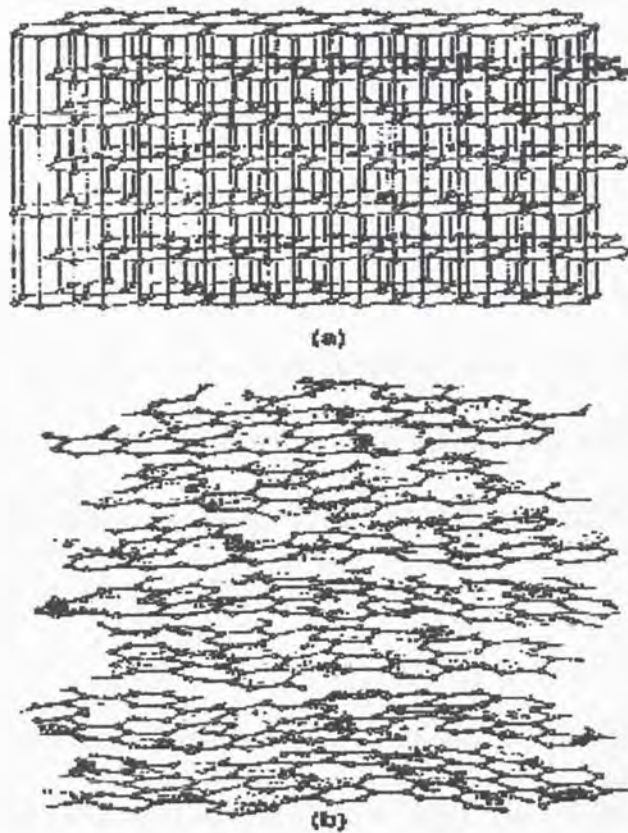


Figure 2.1 Schematic diagrams comparing a) three-dimensional crystal lattice of graphite, with b) a turbostratic structure (Jankwoska *et al.*, 1991).

Active carbon can be divided into two groups: powdered and granular (Smisek and Cerny, 1970) and each group are available in many sizes.

Powdered active carbon (PAC) are black pulverized tiny powder and mainly used for liquid phase application and flue gas treatment. It is usually produced by activating lump material, or chips of wood charcoal, or lumps of paste prepared by mixing sawdust

with a solution of zinc chloride (Smisek and Cerny, 1970) or any chemical activators, then grinding the activated product. The typical size of PAC is in the range 15 to 25 μm (Gregg, 1961). Powdered active carbons are used for adsorption from solution. PAC is basically used to correct taste and odor problems which are primarily an aesthetic quality of the water (Smisek and Cerny, 1970).

Granular active carbons (GAC) are black irregular shaped particles with sizes ranging from 4 to 80 mesh. GAC can be prepared either in the form of crushed (coal or shell) or pressed form (produced as uniform cylindrical shapes). Granular active carbons are used mainly for adsorption of gases and vapours and are therefore known as gas-adsorption carbons (Smisek and Cerny, 1970).

The starting material considerably influences the quality of the resulting active carbon. For special carbons, it is necessary to use a uniform source that imparts to the product characteristic properties that are difficult to simulate. This concerns especially the active carbons from which selective effects are required, as, for example, in the treatment of wine, or in the separation of components in preparative chemistry (Davidson *et al.*, 1968).

Any cheap substance with a high carbon and low ash content can be used as a starting material for the production of active carbon. In older production procedures preference was given to recent and younger fossil materials- wood, peat, and wastes of vegetable origin (fruit stones, nutshells, sawdust)- which can be activated easily and give

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