PREPARATION AND CHARACTERISITION OF ACTIVATED CARBON FROM RICE HUSK

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THIS THESIS IS PRESENTED TO FULFILL THE REQUIREMENT TO OBTAIN A BACHELOR OF SCIENCE WITH HONOURS DEGREE

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DECLARATION

I hereby declare that this is my original work except the quotation that I have stated the sources.

28 March 2005

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ABSTRACT

Activated Carbon is a key element in a number of control technologies as it is an extremely effective adsorbent. It possesses a high surface area per unit weight, and intricate pore structure. The rice husk *(oryza sativa)* mostly found in the tropical region is the by-product produced from rice production industries. The rice husk used for the research purposes not having high commercial sources have been used, selected as a suitable raw material. In this study, an activated carbon was obtained from rice husks by impregnation with different amount of phosphoric acid and in a self-generated atmosphere two step method which involve physical activation in muffle furnace. Experiments were carried out by chemical activation and the process parameter is concentration of impregnating reagent. Phosphoric acid is a very common method for obtaining activated carbons with very high surface area. The resulting carbons were characterized by removal of methylence blue and iodine from aqueous solution. Surface morphology carried out on the samples of rice husk based activated carbon after activation and using scanning electron microscope. Physically activated carbons yield a poor adsorbing capacity (256.68 mg/g of iodine absorbed and 220.39 mg/g of methylene blue absorbed) due to the blockage of pores by decomposition products of lignocellulosic materials. Optimum absorption capacity was obtained when the samples were subjected to chemical activation. The absorption capacity increases as the amount of impregnation ratio increase.

ABSTRAK

Karbon teraktif semakin memainkan peranan penting dalam beberapa teknologi kawalan sebagai bahan penjerap yang sangat efektif. Ini adalah kerana sifat liang mikro dan jumlah luas permukaan per berat yang tinggi pada karbon teraktif. Sekum padi (*oryza sativa*) yang boleh didapati dengan mudah kawasan tropika merupakan sisa buangan daripada industri penghasilan beras. Kos yang rendah dan mudah didapati adalah faktor utama sekum padi dipilih sebagai bahan mentah dalam kajian ini. Karbon teraktif diperolehi dengan pengaktifan kimia oleh asid fosforus dalam kepekatan yang berbeza dan diikuti pemanasan dua peringkat dalam ketuhar. Pengaktifan kimia dengan kepekatan yang berbeza semasa penyediaan karbon teraktif adalah parameter utama dalam kajian ini. Pangaktifan kimia dengan asid fosforus iaitu agen pengaktif kuat, amat berkesan dalam menghasilkan jumlah luas permukaan bahan penjerap yang tinggi. Muatan penjerapan dikaji dengan penjerapan larutan metilena biru dan larutan iodin. Morfologi permukaan karbon dikaji dengan mikroskop sinaran electron. Pengaktifan fizikal hanya menghasilkan muatan penjerapan yang rendah (256.68 mg/g iodin dijerap dan 220.39 mg/g metilena biru dijerap) kerana penutupan liang oleh hasil-hasil penguraian bahan lignoselulosa. Muatan penjerapan optimum diperoleh apabila pengaktifan kimia dilakukan kepada sample. Muatan penjerapan meningkat apabila kepekatan asid bertambah.

CONTENTS

LIST OF TABLE

Table No. Page

LIST OF FIGURES

LIST OF PHOTO

ABBREVIATION

CO_2	Carbon dioxide
KOH	Kalium hydroxide
H_3PO_4	Phosphoric acid
P_2O_5	Diphospate Pentoxide
$Na_2S_2O_3$	Sodium thiosulphate
NH_3	Ammonia

NOMENCLATURE

cm	Centimeter
g	Gram
m	Meter
mg	Milligram
ml	Milliliter
nm	Nanometer
μm	Micrometer
^0C	Degree Celsius
%	Percectage
m^2/g	Meter per gram
kcal/mole	kilocalori per mole
ppm	Parts per million
kg	Kilogram
M	Molarity

CHAPTER 1

INTRODUCTION

1.1 BACKGROUND

Carbon material is used in various fields especially in modern industrial practice and everyday life. Activated carbon are generally obtained by heating or burning under controlled conditions carbonaceous material such as coal, oil, rice husk, oil palm shell, peat, nutshells, peat, and ignite. The conditions of experiment are different for each raw material and each chemical used to activate it. The chemically processes of carbon activation are essential to be elucidated in order to yield more qualitative and quantitative of activated carbons.

The process includes first carbonizing the raw material at low temperatures, and then activating the carbon in a high temperature steam process. Activated carbons have high porosity and large surface area manufactured by carbonization and activation of carbonaceous materials, which find extensive use in the industrial sector for adsorption of pollutants from gaseous and liquid stream. Activated carbon is a highly porous carbonaceous substance with a wide range of applications in gas, vapor, and liquid treatment. The use of activated carbon dates back to 1500 BC where its use was discovered in an Egyptians papyrus for medicinal purposes.

Activated carbon is a highly porous carbonaceous substance with a wide range of applications in gas, vapor, and liquid treatment. The use of activated carbon dates back to 1500 BC where its use was discovered in an Egyptians papyrus for medicinal purposes. In the 18 century, Sheele recognized the adsorptive powers of carbons in experiments with gases. During World War I, activated carbon use increased when the Allies used it in gas masks to filter out chlorine gas. Activated carbon is used successfully today, especially in water treatment to remove organic compounds that impart color, taste and odor to the water. Contaminant removal is achieved through a process called adsorption by which contaminants adhere to the surface of the carbon and are thus removed from the water (Helena *et al*, 1991).

Activated carbon is a processed carbon material with a highly developed porous structure and a large internal specific surface area (Helena *et al*, 1991). Activated carbons are non-hazardous, processed, carbonaceous products, having a porous structure and a large internal surface area. These materials can adsorb a wide variety of substances. They are able to attract molecules to their internal surface, and are therefore called adsorbents (Henning *et al*, 1998). It is an efficacious adsorbent possesses a high surface area per unit weight, extensively developed pore structure, a high crystalline form and mechanical strength. Activated carbon is a porous carbonaceous material with surface area of around 300-3000 m^2/g (Henning *et al*, 1998).

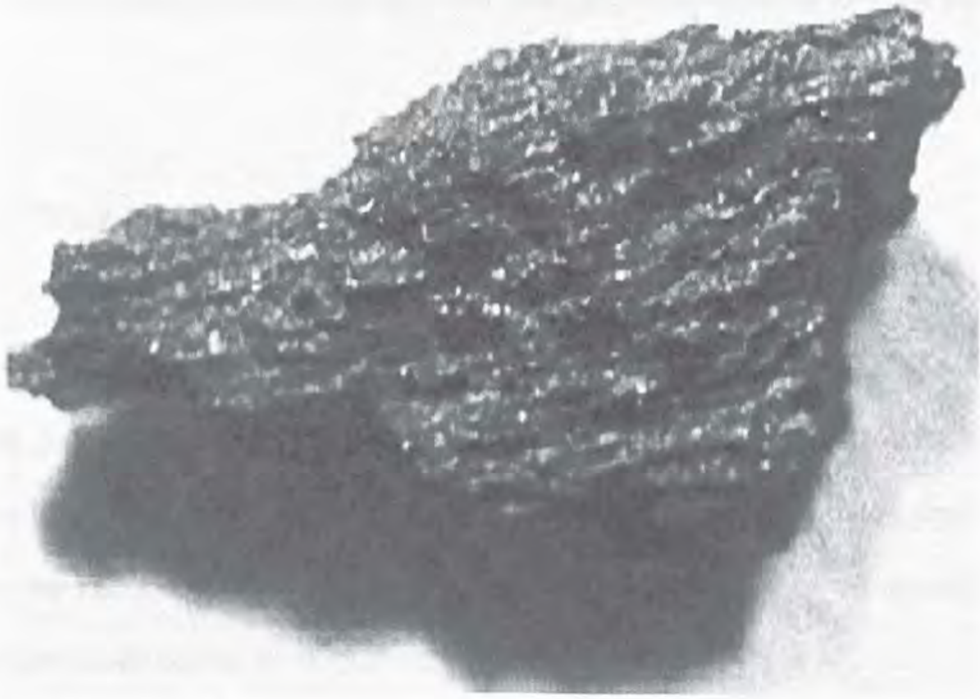

Photo 1.1 Close up of sample activated carbon particles.

Rice husk is composed of complex carbon rich compound. A Rice husk consists of a thin but abrasive skin covering the edible rice kernel, and is mainly comprised of hemicelluloses, lignin, cellulose and silica. Appropriately treated one can make a valuable product out of these complex organic molecules.

Rice husk, a major by-product of the rice milling process, can be a significant resource in activated carbon producing, because of its high carbon content. Rice husk has so far been considered as a cheap, abundantly available renewable waste. At present the rice husk is considered as an agricultural waste. Burning has been the primary means of disposal. Not only burning creates pollution problems but the extremely fine silica ash is also toxic and thus constitutes a health hazard (Singh *et al*, 2002). With the knowledge of producing activated carbon, rice husk can changed into value-added product. A product that could be used by medical industries throughout the world, or used by environmental waste management companies to clean polluted water.

1.2 OBJECTIVES OF STUDY

The research on activated carbon is relatively recent. A few study have been carried out by previous researchers however the use of rice husk as a source of activated carbon for removal of methylence blue from aqueous solution using method self-generated atmosphere two step method and chemical activated by phosphoric acid never been done before. The objectives of this study are:

i) Prepare activated carbon from rice husk.

ii) Physically and chemically characterize the activated carbon that has been prepared.

1.3 SCOPE OF STUDY

The research is based on rice husks. The rice husks (*oryza sativa*) were obtained from Kampung Rukom, Pitas in Kota Marudu, Sabah. It is located about 5 kilometers from Kota Marudu small town. The crushed and grinded rice husks were dried in an oven at 110 °C for 24 hour. The samples for activation are prepared by impregnating phosphoric acid at various concentration. The next procedure is called two step activation which carried out in a muffle furnace. The first activation involves heating the material at 200 °C and the followed by 500 °C for second activation. After the two step activation, the sample rice husks are carried out for further analyst to determine the chemical and physical characteristic of the activated carbon that has been prepared.

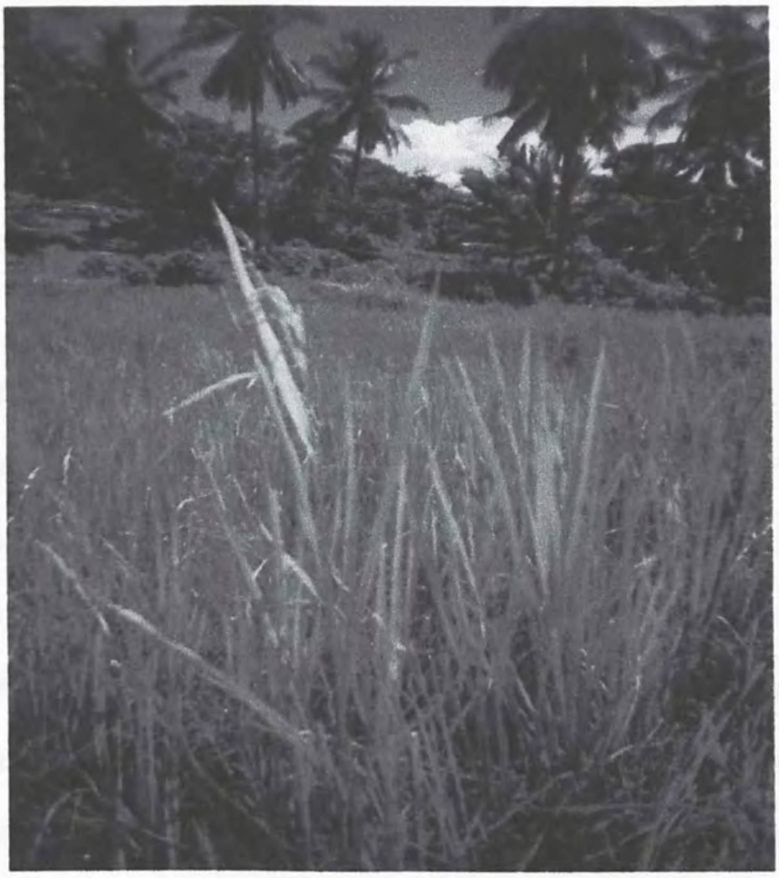

Photo 1.2 The picture of Paddy field in Kampung Rukom, Pitas, Kota Marudu.

CHAPTER 2

LITERATURE REVIEW

2.1 CHARACTERISTICS OF ACTIVATED CARBON

Activated carbon can exist in form of powder, pallets, and granules. Activated carbon consists of three pores and has been classified of according to their width. Micropores has width less than 2 nm, mesopores has width between 2 to 50 nm, and macropores has width more than 50 nm. Micropores generally contribute to the major part of the internal surface area. Macropores and mesopores can generally be regarded as the highways into the carbon particle, and are crucial for kinetics. Macropores can be visualized using Scanning Electron Microscopy (SEM). The pore size distribution is highly important for the practical application; the best fit depends on the compounds of interest, the matrix and treatment conditions. The desired pore structure of an activated carbon product is attained by combining the right raw material and activation conditions (Hoehn *et al*, 1996). Liquid phase carbon is light fluffy powder that has large percentage of transitional or macropores. Gas phase carbon is in form of granules or pallet that has large percentage of micropores. The pore structure and porosity are the most important characteristics of activated carbon. Macropores acts as large access ways for diffusion of particles. Microspore contributes towards large surface areas which are responsible for absorption action (Hoehn *et al*, 1996).

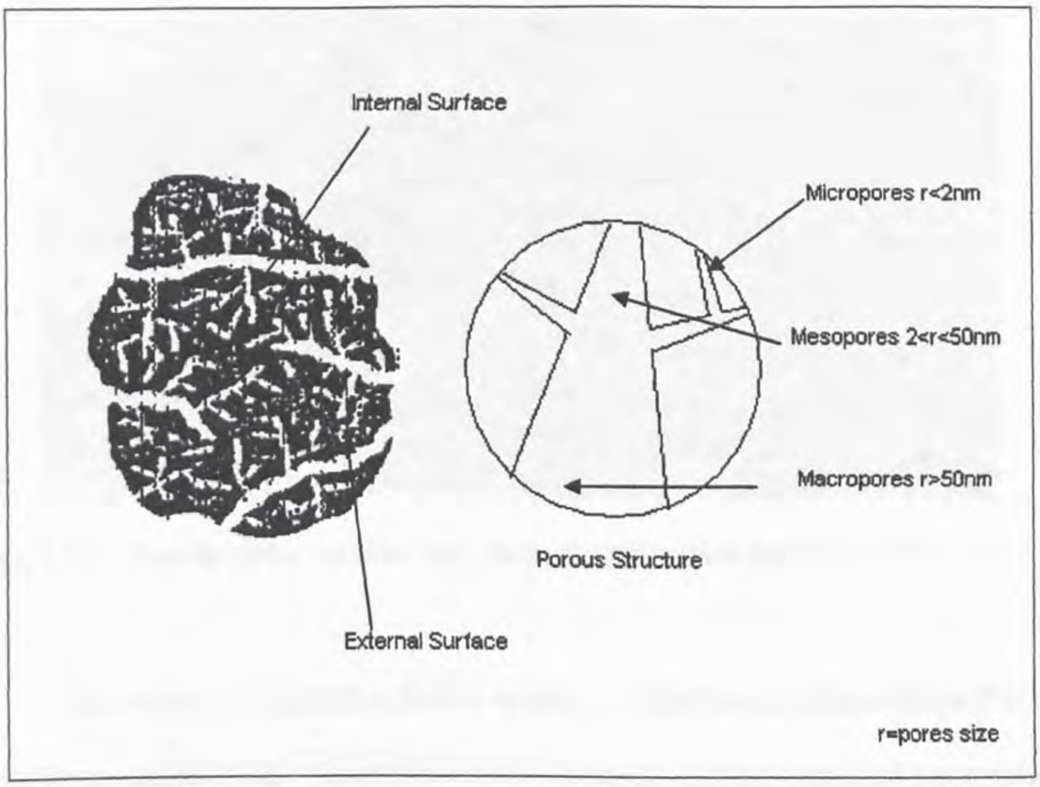

Figure 2.1 Schematic of activated carbon.

2.1.1 Adsorption Process

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. Adsorption is often confused with absorption, where the substance being collected or removed actually penetrates into the other solid (Reynold *et al*, 1996). Activated carbon is useful because it acts as an adsorbent and effectively removes particles and organics from aqueous solution. It is a removal process where certain particles are bound to an adsorbent particle surface by either chemical or physical attraction.

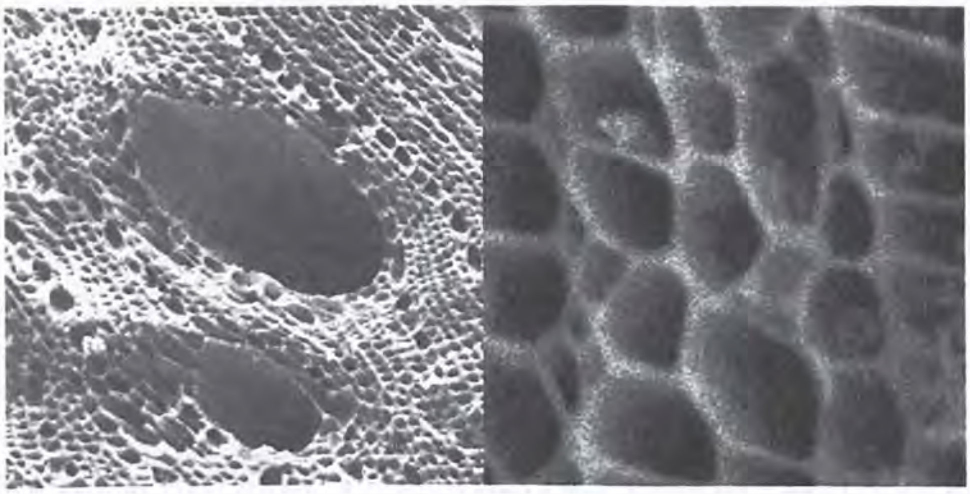

Figure 2.2 Close up carbon surface and pores magnification increases left to right.

The reason that activated carbon is such an effective adsorbent material is due to its large number of cavernous pores as in Figure 2.2. These provide a large surface area relative to the size of the actual carbon particles and its visible exterior surface. An approximate ratio is 1 g is 100 m^2 of surface area (Hoehn *et al*, 1996). If the surface areas increase, the adsorption property also increases. The adsorption behaviors depending on the process of activation, steam, as or chemically activated, the activated carbon differs in their adsorption behaviors (Hoehn *et al*, 1996).

a. Physical Adsorption

Physical adsorption or physisorption also known as Van der Waals adsorption, is an adsorption in which the forces involved are intermolecular forces or Van der Waals forces and which do not influence a significant change in the electronic orbital patterns of the species involved. Physisortion occurs when London-Van der Waals forces bind the adsorbing molecule to the solid substrates where this intermolecular force are the same as that bond molecules to the surface of a liquid. The molecules adsorbed into adsorbate surface are in form of multilayer. Physisorption is a weak binding and consist of enthalpy between 0 to 5 kcal/mole. Almost all adsorption processes pertinent to air pollution control involve physical adsorption. The internal surface area of the activated carbon exhibits weak van der Waals forces which lock the impurities into the pore structure (Helena *et al*, 1991).

b. Chemical Adsorption

Chemical adsorption or Chemisorption is an adsorption in which the forces involved are valence forces of the same kind as those operating in the formation of chemical compounds. It occurs when electrons are exchanged or shared between activated carbon and the liquid gas molecules. This bonding reacts to a change in the chemical form of the adsorbed compounds and is therefore not reversible. Chemisorption is strong binding and consists of enthalpy between 20 to 100 kcal/mole. Thus, more heat is liberated. For many applications, the adsorbent is chemically impregnated with a substance that encourages chemical reactions with particular adsorbates with chemical adsorption, higher temperature can improve performance of the adsorption. Carbon surfaces have both negative (anionic) or positive (cationic) charges to attract free ions in solution or suspensions, depending on how they treated. Treatment of carbon with a base increases the capacity of carbon to exchange anions, acidulation of the surface makes carbon a powerful cation exchanger (Helena *et al*, 1991). Surface oxidation involves the chemisorption of atmospheric oxygen to the carbon and the further reaction of the surface oxides that chemically react with other substances that are oxidized. The surface of activated carbon has an electrical double layer. The molecules adsorbed into adsorbate surface are in form of monolayer.

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