SYNTHESIS OF ACTIVATED CARBON FROM WASTE RAW MATERIAL USING “BULUH LEMANG“”, *SCHIZOSTSCHYUM BRACHYCLADUM*.

SHAFARUL BIN MUSTAFA

UNIVERSITI TEKNIKAL MALAYSIA MELAKA

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Signature : ....
Supervisor I : En. Imran Syakir bin Mohamad
Date : ....

Signature : ....
Supervisor II : En. Hairul bin Bakri
Date : ....

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Faculty of Mechanical Engineering
Universiti Teknikal Malaysia Melaka

MAY 2011

DECLARATION

"I hereby declare that the work in this report is my own except for summaries and quotations which have been duly acknowledged."

Signature	: ……………………………………
Name of Candidate	: SHAFARUL BIN MUSTAFA
Date	:

For my beloved mother,
dearest family members and next of kin,
lecturers and friends.

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ABSTRACT

The main objective of this work was to synthesis waste of bamboo as the activated carbon and characterized the activated carbon. The unique of activated carbon commonly known by the principle of adsorptivity which be applied in removing contaminants and odor in the water and air. This has been adopted in many industrial field associated with waste treatment to reduce pollution. The potential of activated carbon has been acknowledge in many publications to efficiently provide adsorption site for this purpose. Hence, in this particular study, the application of activated carbon manufactured from the waste of bamboo (*Schizostachyum brachycladum*) or better known as *buluh lemang* amongs the Malay community was investigated. The effect on process variables such as activating agents zinc chloride ($ZnCl_2$) activation temperature, impregnation ratio and carbonization temperature were studied to optimize these parameters. The resulting activated carbon were characterized in terms of iodine number (I_2 number) for its adsorptive capacity , Scanning Electron Microscope (SEM) and Brunauer, Emmett, Teller (BET) method were used to provide information on surface area, pore size, types of pore (micropores, mesopores, macropores) and total pore volumes. Result iodine number shows, sample activated carbon, chemical activation(temperature 500ºC, impregnation ratio 1:1) has higher value which is 295 mg/g among others samples. In BET analysis, sample activated carbon, physical activation (500ºC) shows good surface area, 339.1 m^2/g and total volume pore, 1.042 cc/g. Lastly, for SEM-EDX data analysis, sample activated carbon, physical activation (600ºC) has higher carbon content, 90.99% (atomic percentage).

ABSTRAK

Objektif utama projek ini adalah untuk mensintesis sisa buangan daripada buluh lemang untuk menjadi karbon teraktif dan seterusnya menganalisa sifat-sifat karbon teraktif tersebut. Keunikan karbon teraktif umumnya diketahui berdasarkan prinsip jerapan yang dapat diaplikasikan bagi menghilangkan kotoran dan bau dalam air dan udara. Karbon teraktif telah diterima pakai di dalam pelbagai bidang industri yang berkaitan dengan pengurusan sisa bagi mengurangkan pencemaran. Potensi karbon teraktif telah diakui melalui pelbagai penerbitan sebelum ini dalam menyediakan kajian jerapan secara cekap. Oleh itu, dalam kajian yang dijalankan, aplikasi karbon teraktif dihasilkan dari sisa buangan daripada buluh (*Brachycladum Schizostachyum*) atau lebih dikenali sebagai 'buluh lemang' dalam komuniti Melayu. Selain itu, kajian ini mengkaji karbon teraktif apabila pembolehubah yang digunakan di dalam pembuatan karbon teraktif seperti agen pengaktifan yang digunakan, zink klorida ($ZnCl_2$), suhu pengaktifan, nisbah impregnasi dan suhu karbonisasi bagi mengoptimumkan penghasilan karbon teraktif tersebut. Karbon teraktif yang dihasilkan dikenalpastikan sifatnya dengan menggunakan analisis nombor iodin (nombor I_2) untuk memperoleh daya serap, selain itu, Mikroskop Imbasan Elektron (SEM) dan kaedah Brunauer, Emmett, Teller (BET) digunakan untuk memperolehi maklumat mengenai luas permukaan, saiz liang, jenis liang (liang mikro, liang meso, liang makro) dan jumlah liang yang terdapat pada karbon teraktif yang dihasilkan. Keputusan nombor iodine menunjukkan sampel karbon teraktif daripada pengaktifan kimia (suhu 500ºC , nisbah impregnasi 1:1) mempunyai nilai yang paling tiggi daripada sampel karbon teraktif yang lain dengan nilai 295 mg/g. Dalam analisis BET, sampel pengaktifan fizikal (500ºC) mempunyai keluasan permukaan yang baik 339.1 m^2/g dan jumlah isipadu liang, 1.042 cc/g. Akhir sekali, untuk data analisis SEM-EDX , sampel karbon teraktif daripda pengaktifan fizikal (600ºC) mengandungi kandungan karbon yang tinggi 90.99%.

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LIST OF SYMBOLS

%	percent
g	gram
ml	milliliter
cm	centimeter
mm	milimeter
N	number of adsorbed molecular layers
n	dependence of the amount of adsorption
ε	adsorption potential
K	Kelvin
ºC	degree Celsius
η	electron coefficient
δ	secondary electron coefficient
H_3PO_4	phosphoric acid
$ZnCl_2$	zinc chloride
I_2	iodine
NaOH	sodium hydroxide
HCl	hydrochloric acid
CO_2	carbon dioxide
H_2SO_4	sulfuric acid
KOH	potassium hydroxide
Na_3PO_4	sodium phosphate
NaCl	sodium chloride
$KMnO_4$	potassium permanganate
N_2	nitrogen
$Na_2S_2O_3.5H_2O$	sodium thiosulfate
C	filtrate normalities
Å	angstrom

LIST OF ABBREVIATION

ASTM	American Society for Testing Material
AWWA	American Water Works Association.
BET	Brunauer–Emmett–Teller
SEM	Scanning Electron Microscope
IUPAC	International Union of Pure and Applied Chemistry.
AC	Activated Carbon
GAC	Granular Activated Carbon
PAC	Powder Activated Carbon
CRT	Cathode Ray Tube
BC	Before century
U.S	United State

CHAPTER 1

INTRODUCTION

1.0 INTRODUCTION

Increasing solid waste has become a serious issue that leads to water pollution. The impact of solid waste was significantly severe when it comes to festive seasons in Malaysia. Thus, one of the solutions for water or air pollution is to use the ability of activated carbon (AC) to filter the chemical associated with the waste. Currently, the bamboo culm, or stem, has been made into an extended diversity of products ranging from domestic household products to industrial applications (McClure ,1967). Examples of bamboo products are food containers, skewers, chopsticks, handicrafts, toys, furniture, flooring, pulp and paper, boats, charcoal, musical instruments and weapons. In Asia, bamboo is quite common for bridges, scaffolding and housing, but it is usually a temporary exterior structural material. In many overly populated regions of the tropics, certain bamboos supply the one suitable material that is sufficiently cheap and plentiful to meet the extensive need for economical housing.

Bamboo is a natural resource in Malaysia as it takes only several months to grow and be ready for harvesting. Primary processing of bamboo for conversion into specified products generates a large amount of residues is generated. This residue could be effectively converted into value-added products such as activated carbon and charcoal. Bamboo is also a good source of carbon and there is possibility for it to be made AC for the purpose of pollution control. Waste of bamboo from lemang also can produce the activated carbon. Thats means, from bamboo waste , we can recycle and create other applications for industrial.

According to Gadkaree (1998), activated carbon is a very important material industrially, with applications in a variety of areas such as adsorbers in air and water pollution control, catalysts in the chemical and petrochemical industries. Typically activated carbon is made from naturally occurring materials such as wood, coal and nutshell flour etc. via high temperature, inert atmosphere processing followed by activation to create porosity in the nanometer size range. This porosity imparts special adsorption characteristics to carbon and makes it useful in the variety of applications mentioned above.

1.1 OBJECTIVE

The main objective of this project is to synthesis * activated carbon (AC) from easily available raw material (bamboo). This is done by aiming on the specific objectives as follow:

1. To synthesis activated carbon from waste materials.(waste of bamboo)
2. To investigate the adsorption performance of activated carbon using iodine number analysis
3. To characterize the properties of activated carbon prepared using analytical spectroscopy.(SEM-EDX, N_2 adsorption analysis)

1.2 PROBLEM STATEMENT

In this project, the material for the manufacturing of activated carbon was investigated. This was done to reduce waste especially during festive season in our country such as Hari Raya. Additionally, to investigate the suitability of „buluh lemang‟ (Schizostachyum brachycladum) as the activated carbon and its applicability in various industrial applications. This project also aims to the investigate adsorption performance of those materials for the use in the manufacturing activated carbon especially in waste management and pollution control.

1.3 SCOPE

1. To find and prepared the waste of bamboo . Type of bamboo was selected is (*Schizostachyum brachycladum*) , among Malay call buluh lemang. Washed with water and dried the bamboo under normal sun light. The bamboo was cut into part about 10cm- 15cm and crush using industry crusher (3-4 mm) to make its easier during synthesis process.
2. This studied about activated carbon had been carried out only on laboratory scale. It is started from synthesis waste of bamboo by activated manufacturing system carbonization and activation. Activation that used for synthesis raw material are Physical (Gas Carbon Reactor) and Chemical activation , use zinc chloride as activating agents ($ZnCl_2$)
3. Characterize activated carbon after finished the synthesis process and find properties of activated carbon by using a few method such as Scanning Electron Microscopy (SEM) to find structural properties and morphology of activated carbon . Others method used are Brunauer Emmet Teller Method (BET), N_2 adsorption and Iodine Number, $I_{2,}$ analysis.

1.4 EXPECTED RESULT

To be a guideline in producing activated carbon from bamboo (*Schizostachyum brachycladum*) waste. Low cost alternative substitute raw material and increase efficiency of utilization, performance of activated carbon. Value-addition of agricultural residues and on other side helps solving problem, which otherwise disposal cause.

CHAPTER TWO

LITERATURE REVIEW

2.1 HISTORY OF ACTIVATED CARBON

Activated carbon was first generated industrially at the first part of the 20th century, when carbon activated from vegetable material was produced for use in sugar refining. Powered activated carbon was first produced commercially in Europe in the early 19th century, using wood as a raw material. This carbon found widespread use in the sugar industry. In the United States, the first production of activated carbon used black ash as the source, after it was accidentally discovered that the ash was very effective in decolorizing liquids. Activated carbon has since been used extensively for this purpose in many industries. In particular, it has been commonly used for the removal of organic dyes from textile wastewater. The first documented use of activated carbon in a large scale water treatment application was in 19th century England, where it was used to remove undesirable odors and tastes from drinking waters. Use in the United States for similar purposes closely followed. In recent years, the use of activated carbon for the removal of priority organic pollutants has become very common. The use of carbon extends back far in time. Charcoal was used for drinking water filtration by the ancient Hindus in India, and carbonized wood was used as a medical adsorbent and purifying agent by the Egyptians as early as 1500 BC.

2.2 ACTIVATED CARBON

Activated carbon is porosity (space) enclosed by carbon atoms. Activated carbon is a microcrystalline, nongraphitic form of carbon that has been processed to develop internal porosity. This porosity yield the surface area that provides ability to adsorb gases and vapors from gases and to adsorb dissolved or dispersed substances from liquid.activated carbon is characterized by a vast system of pore of molecular size within the carbon particles resulting in the formation of a material with extensive surface area. However, commercial grade of activated carbons generally have a surface areas ranging from 600 to 1200 m^2/g.(Chilton Ng et al.2002)

Activated carbon is a very important material industrially, with applications in a variety of areas such as adsorbents in air and water pollution control, catalysts in the chemical and petrochemical industries. Electrodes in batteries and supercapacitors and also purifiers in the food and pharmaceutical industries. Further, as an environmental pollution is increasingly becoming a serious problem the demand for activated carbon is growing.(K.P Gadkaree.1998)

2.2.1 Properties of Activated carbon (AC):

- Solid, porous, black carbonaceous material
- Extraordinary large surface area (1200m^2/g) and pores volume that give unique
- Complex action mode
- Having both chemical and physical effect on substances

Figure 2.1: Application of activated carbon.

(Source: http://www.cataler.co.jp)

2.2.2 Structure of Activated carbon

Activated carbon has a disordered microstructure, which is termed amorphous carbon. X-rays studied have demonstrated that many so-called amorphous substances have crystalline characteristics, even they many not show certain features such as crystal angles and faces, usually associated with the crystalline state. The kind of carbon from which activated carbon is derived is non-graphitizing, meaning that it cannot be transformed into crystalline graphite even at temperatures of 3000ºC and above. A simple model of non-graphitizing carbon based on small graphitic crystallites joined together by cross-links, but did not explain the nature of these cross-links.

A later idea was that the cross-links might consist of domains containing sp3-bonded atoms, but this had to be discounted when neutron diffraction studies showed that non-graphitizing carbons consist entirely of sp2 atoms .A much more recent suggestion is that non-graphitizing carbon has a structure related to that of the fullerenes, in other words that it consists of curved fragments containing pentagons and other non-hexagonal rings in addition to hexagons. Such a structure would explain the micro porosity of the carbon, and many of its other properties. However, obtaining direct experimental support for this hypothesis is extraordinarily difficult. Both x-ray and neutron diffraction have been extensively applied to non-graphitizing carbons, and in some studies the diffraction data has been interpreted in terms of a structure containing non-hexagonal rings (Peter J F Harris et al. .2008).

The IUPAC classification of pores by sizes defines the following three class of pore (IUPAC .1972):

- Micropores, less than 2 nm (less than 20 Å)
- Mesopores between 2 and 50 nm, (20 to 500 Å)
- Macropores, 50 nm, (more than 500 Å).

According to T. Otowa et al. (1997), Tsutomu Suzuki et al. (2007) and A. Linares-Solano et al. (2000), usually more than 95% micropores content in the total area surface of activated carbon. The volume of micropores range from 0.15 up to 0.6 cm^3/g. Conventional activated carbon were tridisperse, having all three type of pores present within their structure. Absorbance molecules penetrate through the wider pore to micropores. Activated carbon prepared with high surface area, low volume resistivity or low ash content has been reported largely. The composition and structure of an activated carbon depends on the raw material, the activating agent and the preparation method. These parameters condition the properties of the activated carbon and hence its applications.

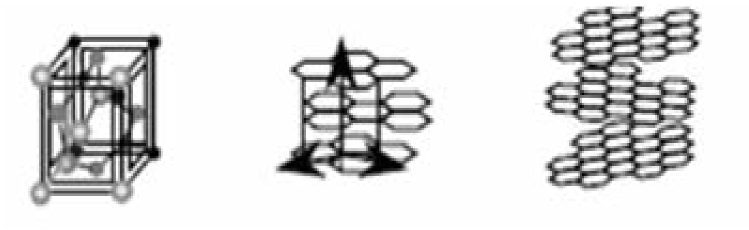

Figure 2.2: Carbon Black "Quasi-Graphitic" Microstructure compared to the Two Regular Crystalline Forms of Carbon (Diamond and Graphite).

2.2.3 Activated Carbon adsorption.

According to Khadija Qureshi et al. (2008), they are most effective adsorbents in treating drinking water and industrial wastewater. The food industry is also a major consumer of activated carbon, where it is used to remove compounds that adversely affect color, taste and odor. In the mineral industry activated carbon are used to recover gold from leached liquors. Medicinal uses and pharmaceutical industry is also another wide area for the utilization of activated carbon. In gas cleaning applications activated carbon are extensively used in air filters at industrial level as well as in general air conditioning application.

According to K.P Gadkaree, (1998), adsorption is a process in which matter adheres to the surfaces of adsorbents (activated carbon). In the adsorption process, molecules of a contaminated are attracted to and accumulate on the surface of the activated carbon. Carbon is a commonly used adsorbent due to its very large surface area. Physical adsorption is dependent on the characteristics of the contaminant to be adsorbed, the temperature of the gas stream to be processed, and the concentration of the contaminant in the gas stream. The adsorption capacity for a particular contaminant represents the amount of the contaminant that can be adsorbed on a unit weight of activated carbon consumed at the conditions present in the application.
Typical adsorption capacities for moderately adsorbed compounds range from 5 to 30 percent of the weight of the carbon.

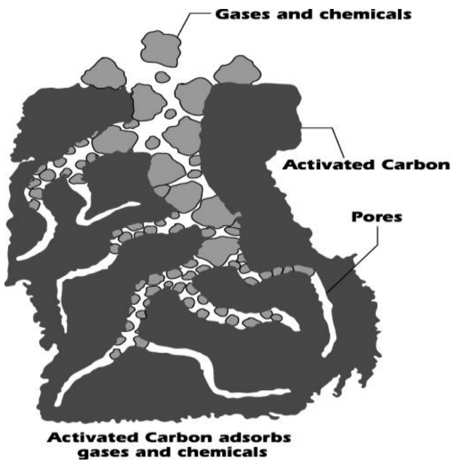

Figure 2.3**:** Cross section activated carbon adsorption for gases and chemicals.
Source: (home-air-purifier-guide.com)

2.2.4 Type and selection of activated carbon.

Refer to C. Srinivasakannan (2003), there are three different types of activated carbon. Based on the application a specific type of activated carbon is used:

a. Powdered Activated Carbon (PAC) (particle size predominantly smaller than 80 mesh) - used for taste and odor removal
b. Granular Activated Carbon (GAC) (particle size predominantly more than 80 mesh)- Used for liquid and gas phase application
c. Pellet Activated Carbon- Used mainly for gas phase application

Moreover, activated carbon is able to offer a range of different types of activated carbon media such as carbon briquettes, chemical impregnated granular activated carbon and fibrous activated carbon.

(a)

(b)

(c)

Figure 2.4 : Types of activated carbon. (a) PAC, (b) GAC, (c) Pellet A.C .

Table 2.1: Commercially Available Carbons.

Source: (William D.F. et al. 1987)

FORM	MEAN PARTICLE SIZE (mm)	SIZE FRACTION U.S.SIEVE NO.	RAW MATERIAL
Granular	2.5 2.1 1.7	6x12 6x16 8x16	Coconut, Bituminous Coal
Pellet	2.6	2 mm diameter	Coal, Peat
Powder	0.04	65%-325	Coal Peat, Wood