

4000006312



HADIAH

PREPARATION AND CHARACTERIZATION OF MEDICAL GRADE
ACTIVATED CARBON FROM BANANA (*Musa acuminata*) SKIN

CHOONG SU KIAT

DISSERTATION SUBMITTED AS PARTIAL FULFILLMENT FOR THE
DEGREE OF BACHELOR OF SCIENCE WITH HONORS

PERPUSTAKAAN
UNIVERSITI MALAYSIA SABAH

INDUSTRIAL CHEMISTRY PROGRAMME
SCHOOL OF SCIENCE AND TECHNOLOGY
UNIVERSITI MALAYSIA SABAH

PERPUSTAKAAN UMS MARCH 2005



1400006312



UMS
UNIVERSITI MALAYSIA SABAH

UNIVERSITI MALAYSIA SABAH

BORANG PENGESAHAN STATUS TESIS@

JUDUL: Preparation and Characterization of medical grade
activated carbon from banana (Musa acuminata) skin

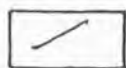
Ijazah: Ijazah Sarjana Muda

SESI PENGAJIAN: 2002/2005

Saya CHOONG SU KIAT
(HURUF BESAR)

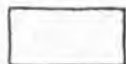
Mengaku membenarkan tesis (LPS/Sarjana/Doktor Falsafah)* ini disimpan di Perpustakaan Universiti Malaysia Sabah dengan syarat-syarat kegunaan seperti berikut:

1. Tesis adalah hakmilik Universiti Malaysia Sabah.
2. Perpustakaan Universiti Malaysia Sabah dibenarkan membuat salinan untuk tujuan pengajian sahaja.
3. Perpustakaan dibenarkan membuat salinan tesis ini sebagai bahan pertukaran antara institusi pengajian tinggi.
4. **Sila tandakan (/)



SULIT

(Mengandungi maklumat yang berdarjah keselamatan atau kepentingan Malaysia seperti yang termaktub di dalam AKTA RAHSIA RASMI 1972)



TERHAD

(Mengandungi maklumat TERHAD yang telah ditentukan oleh organisasi/badan di mana penyelidikan dijalankan)



TIDAK TERHAD

Disahkan oleh

[Signature]
(TANDATANGAN PENULIS)

(TANDATANGAN PUSTAKAWAN)

Alamat Tetap: A4-08 Chancellor Condo,
Stn-Kosas Utama 2, 68000
Ampang, Selangor D.E.

En. Collin Glen Joseph
Nama Penyelia

Tarikh: 23/3/05

Tarikh: 23/3/05

CATATAN: * Potong yang tidak berkenaan.

** Jika tesis ini SULIT atau TERHAD, sila lampirkan surat daripada pihak berkuasa/organisasi berkenaan dengan menyatakan sekali sebab dan tempoh tesis ini perlu dikelaskan sebagai SULIT dan TERHAD.

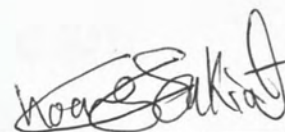
@ Tesis dimaksudkan sebagai tesis bagi Ijazah Doktor Falsafah dan Sarjana secara penyelidikan, atau disertasi bagi pengajian secara kerja kursus dan penyelidikan, atau Laporan Projek Sarjana Muda (LPSM).



DECLARATION

I admit this dissertation is all my own works, except for the translations and summaries that have been declared of its sources.

22 March 2005



CHOONG SU KIAT

HS 2002-3708




VERIFICATION

Signature

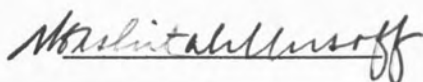
1. SUPERVISOR

(Mr. Collin Glen Joseph)



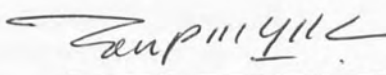
2. EXAMINER 1

(Assoc. Prof. Dr. Mashitah Yusoff)



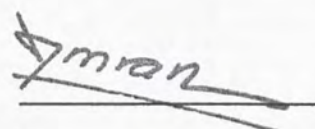
3. EXAMINER 2

(Mr. Moh Pak Yan)



4. DEAN

(Prof. Madya Dr. Amran Ahmed)


_____

ACKNOWLEDGMENTS

Firstly, I wish to express my sincere gratitude to my supervisor, Mr. Collin Glen Joseph for his guidance, advise and patient throughout this period. Also word of thanks to Pn. Zainab, En Sani, En. Khairul and En. Yusri for their generous helps on instrumentations and chemicals. I would like to thank all my friends that offer their helps when I am under difficult circumstances during the progression of this dissertation. At last, but certainly not least, I wish to thank my family too for their support, encouragement and understanding that motivated me to complete this dissertation.



ABSTRACT

Activated carbon in this study was prepared using the two steps activation method in self generated atmosphere that comprised of carbonization and activation steps. Physical activation was used and the raw material was banana skin from *Musa acuminata* species. Raw materials were activated using four different sets of carbonization and activation parameters. The proximate and ultimate physical as well as chemical analyses (percentage of yield, pH value, moisture and ash content, iodine number, methylene blue adsorption capacity and morphological studies) were performed on the prepared activated carbon for the activated carbon's adsorption capacity determination. Sample B that carbonized at 200 °C for 30 minutes followed by activation at 500 °C for 60 minutes, obtained the highest iodine number (439.1 mgg^{-1}) and methylene blue adsorption capacity (65.4 mgg^{-1}) compared with other three sets. SEM morphological studies had shown that as the carbonization and activation time and temperature increased, the pores on the activated carbon increased in number and size.



ABSTRAK

Karbon aktif dalam kajian ini disediakan dengan menggunakan kaedah pengaktifan dua langkah yang terdiri daripada langkah-langkah penkarbonan dan pengaktifan. Pengaktifan secara fizikal dan kulit pisang daripada spesies *Musa acuminata* sebagai bahan mentah digunakan. Bahan mentah diaktifkan dengan menggunakan empat set parameter penkarbonan dan pengaktifan yang berlainan. Analisis-analisis proksimat, fizikal dan kimia (peratusan hasil, nilai pH, kandungan air dan habuk, nombor iodin, kadar jerapan metilena biru dan kajian morfologikal) telah dijalankan bagi mengkaji kadar jerapan karbon aktif yang telah disediakan. Set sampel B yang dikarbonkan dengan suhu 200 °C untuk 30 minit dan diaktifkan sehingga suhu 500 °C untuk 60 minit didapati memiliki nilai iodin (439.1 mgg^{-1}) serta kadar jerapan metilena biru (65.4 mgg^{-1}) yang tertinggi berbanding set-set yang lain. Kajian morfologikal menunjukkan bahawa tempoh and suhu penkarbonan dan pengaktifan berkadar langsung dengan penambahan bilangan dan saiz liang-liang pada karbon aktif.



CONTENT

PAGE

DECLARATION	ii
VERIFICATION	iii
ACKNOWLEDGMENTS	iv
ABSTRACT	v
ABSTRAK	vi
LIST OF CONTENT	vii
LIST OF TABLES	x
LIST OF PHOTOS	xi
LIST OF FIGURES	xii
LIST OF APPENDIXES	xiii
LIST OF SYMBOLS AND ABBREVIATIONS	xiv

CHAPTER 1 INTRODUCTION

1.1 Activated Carbon	1
1.2 Objectives	2
1.3 Scopes of Study	2

CHAPTER 2 LITERATURE REVIEW

2.1 Activated Carbon	3
2.2 Preparation of Activated Carbon	4
2.2.1 Raw Material	5



2.2.2	Carbonization	6
2.2.3	Steam Activation	6
2.3	Characterization of Activated Carbon	
2.3.1	Pores	7
2.3.2	Iodine Number	8
2.3.3	pH Value	9
2.4	Adsorption Property	9
2.4.1	Factors Affecting Adsorption Capacity	10
2.4.2	Factors Affecting Adsorption Rate	12
2.5	Usages of Activated Carbon	13

CHAPTER 3 MATERIALS AND METHODS

3.1	Raw material	15
3.2	Chemicals and Instruments	16
3.3	Preparation of Activated Carbon	17
3.3.1	Predried Process	17
3.3.2	Carbonization and Activation	17
3.4	Characterization of Product	19
3.4.1	Determination of Percentage of Yield	19
3.4.2	Determination of pH Value	20
3.4.3	Determination of Moisture and Ash Content	20
3.4.4	Determination of Methylene Blue Adsorption Capacity	22
3.4.5	Determination of Iodine Number	23
3.4.6	Morphological Studies	26



CHAPTER 4 RESULTS AND DISCUSSION

4.1	Preparation of Activated Carbon	27
4.2	Characterization of Activated Carbon	
4.2.1	Percentage of Yield	28
4.2.2	pH Value	29
4.2.3	Moisture and Ash Content	30
4.2.4	Methylene Blue Adsorption Capacity	30
4.2.5	Iodine Number	33
4.2.6	Morphological Studies	34

CHAPTER 5 CONCLUSION AND SUGGESTIONS

5.1	Conclusion	38
5.2	Suggestions	39

REFERENCES	40
------------	----

APPENDIX A	43
------------	----

APPENDIX B	44
------------	----

APPENDIX C	45
------------	----

APPENDIX D	46
------------	----

APPENDIX E	47
------------	----

APPENDIX F	48
------------	----

APPENDIX G	49
------------	----



LIST OF TABLES

Table No.		Page
3.1	List of chemicals	16
3.2	List of instruments	16
3.3	The parameters for carbonization and activation processes	18



LIST OF PHOTOS

Photo No.	Page
3.1 (a) Banana tree and (b) banana fruit	15
3.2 (a) Furnace and (b) reflux set used in carbonization and activation processes	18
3.3 (a) Hotplate and (b) pH meter used in pH value determination	20
3.4 (a) UV Spectrometer and (b) orbital shaker used in methylene blue adsorption capacity determination	23
3.5 (a) Studs and (b) SEM used in sample's morphological studies	26
4.1 Predried banana skins	27
4.2 Prepared activated carbon	28



LIST OF FIGURES

Figure No.	Page
2.1 Activated carbon's structure	8
4.1 Chart for percentage of yield result	29
4.2 Chart for pH value result	30
4.3 Chart for moisture content result	31
4.4 Chart for ash content result	32
4.5 Chart for methylene blue adsorption capacity result	33
4.6 Chart for iodine number result	34
4.7 SEM micrographs of raw banana skin	35
4.8 SEM micrographs of Set A	35
4.9 SEM micrographs of Set B	36
4.10 SEM micrographs of Set C	36
4.11 SEM micrographs of Set D	37



LIST OF APPENDIXES

Appendix No.	Page
A. Percentage of yield result	43
B. Moisture content result	44
C. Ash content result	45
D. Calibration curve for methylene blue	46
E. Methylene blue analysis report	47
F. Calculations for methylene blue adsorption capacity	48
G. Iodine number result	49



LIST OF SYMBOLS AND ABBREVIATIONS

Abs	absorption
B. C	before the beginning of the Christian calendar
SEM	Scanning Electron Microscopy
UV	ultraviolet



CHAPTER 1

INTRODUCTION

1.1 ACTIVATED CARBON

Activated carbon is a synonym for form of carbon having marked adsorptive properties; that is, possess a readily accessible internal surface area (porosity) greatly in excess of the superficial surface (Davidson *et al.*, 1968). It has an extraordinarily large surface area and pore volume that gives it a unique adsorption capacity. It obtained by the process of carbonization followed by activation of the carbonaceous material such like peats, hardwoods, grain hulls, corn cobs, nutshells, coal, sawdust, scrap hides, baggasse, lignite etc as described in the patents and journal literatures. These precursors are normally exposed to a number of activation methods such as physical or chemical in order to produce the activated carbon that has high adsorption capacity (Manocha *et al.*, 2002).

The usage of activated carbon dates back to the prehistoric discovery of fire. Ancient Hindus in India filtered their drinking water with charcoal and carbonized wood was used as a medical adsorbent and purifying agent by the Egyptians as early as 1500 B.C (Jankowska *et al.*, 1991). The importance of activated carbon in daily usage, prompted more studies on the activated carbon. The consequences of these



studies contributed to a better chemically and physically of activated carbons production. However, the conditions of experiment are different for each raw material and each chemical used to activate it, in order to yield more qualitative and quantitative of activated carbons. Therefore, good sets of carbonization and activation parameters are important in order to yield the activated carbon that is high in adsorption capacity. Moreover, this can help to eliminate the wastage of raw material, save time and cost.

1.2 OBJECTIVES

Objectives of this study are:

- a. to prepare edible activated carbon from banana skins
- b. to characterize the prepared activated carbon using the proximate, physical and chemicals analyses for its adsorption capacity.

1.3 SCOPES OF STUDY

This study focuses on:

- a. using banana skin of Pisang Berangan (*Musa acuminata*) species as raw material
- b. preparation is based on two steps activation method (carbonization and activation) in self-generated atmosphere
- c. analyses on the adsorption capacity of the produced activated carbon.

CHAPTER 2

LITERATURE REVIEW

2.1 ACTIVATED CARBON

Activated carbon contains 87–97% of carbon, other elements (hydrogen, oxygen, sulphur and nitrogen), various useless mineral substances (1–20%) and various compounds either from the raw material used for production or generated during the manufacturing process (Jankowska *et al.*, 1991).

Medicinal use of activated carbon dates to ancient times. The earliest reference to the medicinal uses of activated carbon was found in the Egyptian Papyrus of 1500 B. C. The medical grade activated carbon employed during Greek and Roman times, referred to as wood charcoal, was used to treat anthrax, chlorosis, vertigo, and epilepsy. The first hint that medical grade activated carbon might have a role in the treatment of poisoning came from a series of heroic self-experiments in France during the early 19th century. In 1848, American physician B. Rand performed the first medical grade activated carbon efficacy studies in humans (Goldfrank *et al.*, 2002).

Medical grade activated carbon is a safe, odorless, tasteless and fine in texture (Wong *et al.*, 1995) antidote for drug overdoses (Tatro, 2002). It is highly effective in



preventing adsorption of many poisons (Katzung, 1987). Its non-selective absorption of many chemicals make it an excellent absorber in absorption of toxic compounds (Hamilton *et al.*, 2003), minimizing the extent of systemic absorption of the poison, enhancing the elimination of agents already absorbed (Goldfrank *et al.*, 2002) and thereby reducing or preventing systemic toxicity.

During the last 20 to 30 years there has been growing use of activated carbon as a therapeutic intervention for oral poisoning (Tatro, 2002). Clinically, activated carbon adsorbs many substances, thus it is used as an adjunct in the treatment of oral poisonings of heavy metals, phenol, mercuric chloride, atropine, strychnine, phenolphthalein, oxalic acid, poisonous mushrooms, aspirin and most drugs (McKenry *et al.*, 1992). However, it is not effective for poisoning with cyanide, ethanol, methanol, ferrous sulfate, caustic alkalis, boric acid, carbonate and organophosphates (McKenry *et al.*, 1992). Medical grade activated carbon must meet the standards for adsorption, microbial contaminants and purity.

2.2 PREPARATION OF ACTIVATED CARBON

Production methods differ according to whether a friable powder or a hard granular product is required (Davidson *et al.*, 1968). Activated carbon usually produced using the two-step process beginning with the pyrolysis (carbonization) of various carbonaceous materials such as wood, coconut or peat. Then, followed by treatment at high temperatures using variety type of activating (oxidizing) agents such as steam, carbon dioxide (Goldfrank *et al.*, 2002) or oxygen (Jankowska *et al.*, 1991). This is to



increase the adsorption capacity of the agent through the formation of an internal maze of pores with a huge surface area (Goldfrank *et al.*, 2002).

For the rate of adsorption, it depends on external surface area. More particles can be adsorbed on larger area (Wong *et al.*, 1995). The adsorption capacity is dependent on the far larger internal surface area and applied to the type of pores in the activated carbon that are macropores, mesopores and micropores (Hamilton *et al.*, 2003).

2.2.1 Raw Material

Activated carbon is produced from a wide variety of carbon-rich raw materials, including wood, coal, peat, coconut shells, nut shells, saw dust coal, lignites, bones and fruit stone (Jankowska *et al.*, 1991). New materials are currently under investigation as sources for activated carbon. The widespread use of a particular raw material as a source of activated carbon is obviously limited by the supply of that material.

The raw material from which a given activated carbon is produced often has a large effect on its porosity distribution, surface area and final quality of the activated carbon (Davidson *et al.*, 1968). As a result, activated carbons produced from different raw materials may have much different adsorbent qualities (Jankowska *et al.*, 1991).



2.2.2 Carbonization

Carbonization is the most important step in activated carbon production. It is the stage when initial porous structure of activated carbon is formed. The product of carbonization process, conducted at low temperature, in the absence of air and any chemicals, is a material virtually inactive in adsorption, with a specific surface area of several m^2g^{-1} (Jankowska *et al.*, 1991). Rate at which the final temperature is achieved is the important parameter in carbonization process. This step is to remove any volatile components and residual moisture level found in the raw materials. The weight loss is usually around 60 – 70% (Srinivasakannan and Zailani, 2003).

2.2.3 Activation

Activation is a process of heating char to a sufficiently high temperature in order to cause degradation of non-carbon impurities and producing a high surface area and porosity product (Davidson *et al.*, 1968). Activation process consists of two processes, physical activation and chemical activation (Jankowska *et al.*, 1991).

Physical activation implies the pyrolysis of the raw material followed by the activation with gases, such as carbon dioxide, air, steam or a mixture of them. The process involves elimination of a large amount of internal activated carbon mass to obtain well developed carbon structure (Jankowska *et al.*, 1991). Physical activation usually performed at temperatures between 500 °C and 900 °C (Srinivasakannan and Zailani, 2003) to yield microporous materials in which high surface area are



developed as the carbon is burnt off (Jankowska *et al.*, 1991). Physical activation is a severe reaction so the yield is less than chemical activation (Davidson *et al.*, 1968).

Chemical activation consists of the pyrolysis at relatively low temperature under the presence of an activating agent. The chemical agents such as phosphoric acid, zinc chloride and potassium sulphide are mainly used as activating reagents (Jankowska *et al.*, 1991). The chemical activation usually takes places at a temperature lower than that used in physical activation and can produce higher yield than physical activation because of the lower temperature involved in the process avoiding mass of carbon to be burn off (Davidson *et al.*, 1968).

2.3 CHARACTERIZATION OF ACTIVATED CARBON

2.3.1 Pores

Activated carbon has a large volume of very small pores that creates a large surface area. Typical activated carbons have surface areas from 600 to 1200 m²g⁻¹ with some reported as high as 3000 m²g⁻¹ (Davidson *et al.*, 1968). Three groups of pores can be distinguished in activated carbon. There are macropore, mesopore and micropore (Figure 2.1). Macropore has radii between 100 – 200 nm and specific surface area not exceeding 0.5 m²g⁻¹ (Jankowska *et al.*, 1991). It act as transport arteries in adsorption process and can be visualized using Scanning Electron Microscopy (SEM). Mesopore act as transitional pores. It is the main transport arteries for adsorbate. Having the radii in range of 1.5 – 1.6 nm to 100 – 200 nm and specific surface area in range of 20 – 100 m²g⁻¹ (Jankowska *et al.*, 1991). Macropores and mesopores are crucial for



kinetics. Micropores are the smallest pores that can found in activated carbon. It has radii less than 1.5 – 1.6 nm and generally contributes to the major part of the internal surface area (Jankowska *et al.*, 1991).

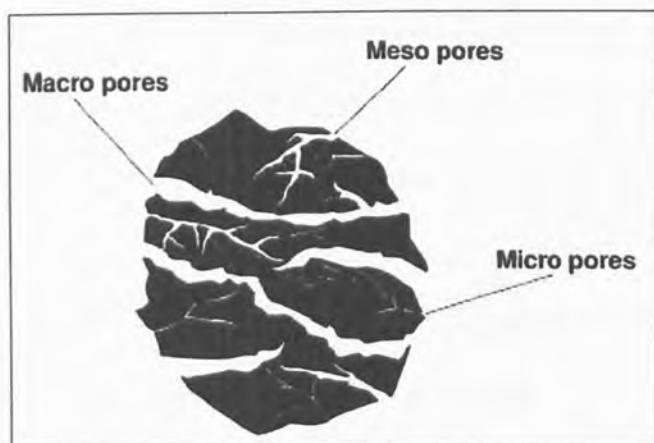


Figure 2.1 Activated carbon's structure.

2.3.2 Iodine Number

The iodine number provides an indication of the amount of small pores in carbon. It is defined as the milligrams of iodine that are adsorbed per gram of carbon when the equilibrium concentration of the bulk saturation is 0.02 N. It is also correlated with the surface area in pores with diameters less than 10 Angstroms. Because iodine is a small molecule, it provides an indication of a particular carbon's capacity to adsorb smaller molecules (Jankowska *et al.*, 1991). For compounds which do not have published isotherms, the iodine number may be used as a relative guideline (not an adsorption rate) for measuring the capacity of a carbon for larger adsorbate molecules.

2.3.3 pH Value

The pH value of activated carbon is a measure whether it is acidic or basic. Activated carbon tends to be a bit basic when coming out of the kiln (pH around 8). Too high pH indicates too much contaminants. Too low a 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 Zailani, 2003).

According to medical viewpoint for detoxification (Goldfrank *et al.*, 2002), most drugs are best adsorbed in their undissociated form (weak bases are best adsorbed at basic pHs and weak acids are best adsorbed at acid pHs).

2.4 ADSORPTION PROPERTY

Adsorption means removal of impurities from solution by activated carbon. Adsorption can be physical and chemisorption (Jankowska *et al.*, 1991). Adsorption phenomena are operative in most natural physical, biological, and chemical systems, and adsorption operations employing solids such as activated carbon and synthetic resins are used widely in industrial applications and for purification of waters and wastewaters.

The process of adsorption involves separation of a substance from one phase accompanied by its accumulation or concentration at the surface of another. The adsorbing phase is the adsorbent, and the material concentrated or adsorbed at the surface of that phase is the adsorbate (Davidson *et al.*, 1968).



REFERENCES

- Bansal, R. C., Donnet, J. B. and Stoeckli, F., 1988. *Active Carbon*. Marcel Dekker, New York.
- Biorci, G. and Pescetti, D., 1962. On the size distribution of pores in charcoal and in other porous substances. *Proceedings of the 5th Conference on Carbon*. **1**, 88-96.
- Davidson, H. W., Wiggs, P. K. C., Churchhouse, A. H., Maggs, F. A. P. and Bradley, R. S., 1968. *Manufactured Carbon*. UK Pergamon, Oxford.
- Goldfrank, L. R., Flomenbaum, N. E., Lewin, N. A., Howland, M. A., Hoffman, R. S. and Nelson, L. S., 2002. *Goldfrank's Toxicologic Emergencies*. 7th ed., McGraw-Hill, USA.
- Hamilton, G. C., Sanders, A. B., Strange, G. R. and Trott, A. T., 2003. *Emergency Medicine. An approach to Clinical Problem-Solving*. 2nd ed., Sauders, USA.
- Harvey, D., 2000. *Analytical Chemistry*. McGraw-Hill, USA.
- Jankowska, H., Swiatkowski, A., Choma, J., 1991. *Active Carbon*. Ellis Horwood Limited, England.



Katzung, B. G., 1987. *Basic and Clinical Pharmacology*. 3rd ed., Appleton&Lange, USA.

Manocha, S., Chauhan, V. B. and Manocha, L. M., 2002. Porosity development on activated of char from dry and wet babbol wood. *Carbon Science* 3 (3), 133-141.

McKenry, L. M. and Salerno, E., 1992. *Mosby's Pharmacology in Nursing*. 18th ed., Mosby-Year Book Inc., USA.

Puri and Bansal, 1965. Iodine adsorption method for measuring surface area of carbon blacks. *Carbon* 3, 227.

Ryu, S. K., Shim, J. W., Yang, K. S. and Mochida, I., 2000. Activated carbon fibers from chemically modified coal tar pitches. *Carbon Science* 1 (1), 6-11.

Sekolah Sains dan Teknologi, 2002. *Panduan Penulisan Disertasi*. Universiti Malaysia Sabah, Kota Kinabalu.

Srinivasakannan, C. and Zailani, M., 2003. Production of activated carbon from rubber wood sawdust. *Biomass and Bioenergy* 27, 89-96.

Standard and Industrial Research Institute Malaysia, 1984. *Specification of Powdered Activated Carbon MS873:1984*, SIRIM.



Tatro, D. S., 2002. *Drug Interaction Facts*. Facts and Comparisons, USA.

Wong, D. L. and Wilson, D., 1995. *Nursing Care of Infants and Children*. 5th ed.,
Mosby-Year Book Inc., USA.

