



**Faculty of Mechanical Engineering**

**SYNTHESIS AND CHARACTERIZATION OF MICROPOROUS  
CARBON ADSORBENTS FROM OIL PALM SHELL**

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**MSc. in Mechanical Engineering**

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**SYNTHESIS AND CHARACTERIZATION OF MICROPOROUS CARBON  
ADSORBENTS FROM OIL PALM SHELL**

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in fulfillment of the requirements for the degree of Master of Science  
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## ABSTRACT

Carbon adsorbents are microporous in structure that can be produced from any raw materials that contain high carbon contents such as wood and coal. The increasing research in carbon adsorbents because of its high adsorption capacities. This research is conducted to study the performance of carbon adsorbents produced from oil palm shell. The preparation of carbon adsorbents consists of carbonization and activation process. This study revealed that the parameters of carbonization and activation process such as temperature affect the carbon adsorbents properties such as pore size, pore volume and surface area. Previous research has proved that the initial properties of carbon adsorbents produced will affect the effectiveness of adsorption and desorption analysis. The results also revealed that the optimum carbonization and activation temperature of 500°C in the preparation of carbon adsorbents significantly enlargement of pores size and efficient for adsorption-desorption applications. This study showed that oil palm shell carbon adsorbents produced from activation temperature of 700°C possesses the largest surface area and micropore volume among the adsorbents tested. Adsorption and desorption of gaseous was observed by the percentage adsorption and desorption of nitrogen and LPG in experimental rig and two types of test bed model. Study shown that gas adsorption and desorption analysis is appropriate to determine the surface area and pore size distribution of carbon adsorbents. In addition, the oil palm shell carbon adsorbents are compared with carbon adsorbents produced from coconut shell and commercial activated carbon and the used of LDPE plastic bags as additive to existing carbon adsorbents in terms of nitrogen and LPG adsorption-desorption analysis. It was examined that the LDPE plastic bag might block or shrink the pore structure of the char. Other parameters and adsorption-desorption capability of each carbon adsorbents samples have been defined and discussed. Lastly, the impact of carbon adsorbents in the application of LPG and methane storage tank was studied. The application of oil palm shell carbon adsorbents increased the amount of LPG and methane in storage tank.

## ABSTRAK

Karbon penjerap adalah dalam struktur berliang mikro yang boleh dihasilkan dari sebarang bahan mentah yang mengandungi kandungan karbon yang tinggi seperti kayu dan arang batu. Peningkatan penyelidikan dalam karbon penjerap kerana kapasiti penjerapannya yang tinggi. Penyelidikan ini dijalankan untuk mengkaji keupayaan karbon penjerap yang dihasilkan dari tempurung kelapa sawit. Penyediaan karbon penjerap terdiri dari proses karbonisasi dan pengaktifan. Kajian ini menunjukkan bahawa parameter bagi proses karbonisasi dan pengaktifan seperti suhu mempengaruhi ciri-ciri karbon penjerap seperti saiz liang, isipadu liang dan luas permukaan. Penyelidikan terdahulu telah membuktikan bahawa ciri-ciri awal penghasilan karbon penjerap akan mempengaruhi keberkesanan proses penjerapan dan pembebasan. Keputusan kajian juga menunjukkan suhu optimum untuk proses karbonisasi dan pengaktifan  $500^{\circ}\text{C}$  dalam penyediaan karbon penjerap sangat berkesan membesarkan saiz liang dan sesuai untuk aplikasi penjerapan-pembebasan. Kajian ini menunjukkan karbon penjerap tempurung kelapa sawit yang dihasilkan pada suhu pengaktifan  $700^{\circ}\text{C}$  mempunyai keluasan permukaan dan isipadu liang mikro yang paling tinggi dikalangan penjerap yang dikaji. Penjerapan dan pembebasan gas dikenalpasti melalui peratus penjerapan dan pembebasan nitrogen dan LPG di dalam kelengkapan eksperimen dan dua jenis model kajian. Kajian menunjukkan bahawa analisis penjerapan dan pembebasan gas diperlukan untuk mengenalpasti keluasan permukaan dan taburan saiz liang bagi karbon penjerap. Sebagai tambahan, karbon penjerap tempurung kelapa sawit dibandingkan dengan karbon penjerap yang dihasilkan dari tempurung kelapa dan karbon penjerap komersil dan penggunaan plastik beg LDPE sebagai bahan tambah kepada karbon penjerap sedia ada dari segi analisis penjerapan-pembebasan nitrogen dan LPG. Kajian mendapati plastik beg LDPE mungkin akan menghalang atau membesarkan struktur liang arang. Lain-lain parameter dan keupayaan penjerapan-pembebasan bagi setiap sampel karbon penjerap telah dikenalpasti dan dibincangkan. Akhir sekali, kesan karbon penjerap dalam aplikasi tangki penyimpanan LPG dan metana telah dikaji. Aplikasi karbon penjerap tempurung kelapa sawit telah meningkatkan kandungan LPG dan metana dalam tangki penyimpanan.



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I declare that this thesis entitle “Synthesis and Characterization of Microporous Carbon Adsorbents from Oil Palm Shell” is the result of my own research except as cited in the references. The thesis has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.

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## LIST OF SYMBOLS

$A$	Adsorption potential
$\text{\AA}$	Angstrom
$C$	BET constant that can be obtained from a plot of straight line, or regression line through the points on plotted graph.
$^{\circ}\text{C}$	Degree Celsius
$E_o$	Characteristics energy of adsorption for the reference vapor
$\Delta G$	change in the Gibbs free energy
$K$	Kelvin
$N_A$	Avogadro constant
$\theta$	Degrees of burn-off
$P$	absolute pressure of bulk gas above sample
$P_o$	saturation pressure of the adsorptive
$P/P_o$	Relative pressure
$R$	universal gas constant
$T$	Temperature
$V_a$	Volume of gas adsorbed by sample
$V_m$	Volume of gas adsorbed when the entire surface is covered with a monomolecular layer
$W_o$	Initial mass
$W_t$	Mass after pyrolysis
$b$	empirical constant
$d$	Average pore diameter
$\sigma$	area of surface
$\beta$	Affinity coefficient
$\%$	Percentage

## LIST OF ABBREVIATIONS

A700	Activated Carbon 700
AC	Activated carbon
$\text{AlCl}_3$ ,	aluminium chloride
ANG	Adsorbed natural gas
ASAP	Accelerated Surface Area and Porosimetry Analyzer
ASCMs	adsorption-selective carbon membranes
BET	Brunauer-Emmett-Teller equations
C	carbon
C400-10-60	Char 400 mixed Plastic Bag LDPE
C700	Char 700
C700 (D)	Char 700 Direct
C400-CO <sub>2</sub> -60	Activated Carbon 400
C700-CO <sub>2</sub> -10-60	Activated Carbon 700 mixed Plastic Bag LDPE
CA	Carbon adsorbents
CAC	Commercial activated carbon
CFCMS	carbon fiber composite molecular sieves
CH <sub>4</sub>	methane
CMSs	Carbon Molecular Sieves
CNG	compressed natural gas
CO <sub>2</sub>	carbon dioxide
CVCC	constant volume combustion chamber
DA	Dubinin-Astakhov equations
DFT	density functional theory
DME	dimethyl ether
DR	Dubinin- Radushkechich equations
FT	methanol or Fischer-Tropsch
GHG	greenhouse gas

H	hydrogen
H <sub>3</sub> PO <sub>4</sub>	sulfuric acid
IUPAC	International Union of Pure and Applied Chemistry
KOH	potassium hydroxide
LDPE	light disposal plastic ethylene
LNG	liquefied natural gas
LPG	liquefied petroleum
MPa	megapascal
N	nitrogen
Na <sub>2</sub> CO <sub>3</sub>	sodium chloride
NaOH	sodium hydroxide
NG	natural gas
NGV	natural gas vehicle
NLDFT	Non-Local Density Functional Theory
NO <sub>2</sub>	nitrogen dioxide
NO <sub>x</sub>	monoxide
O	oxygen
PAC	pitch-based activation carbons
PSA	Pressure swing adsorption
PSD's	Pore-size distributions
S <sub>BET</sub>	total surface area
SnO <sub>2</sub>	stannum oxide
Sox	sulfate
TCA	1,1,1-trichloroethane
TCE	trichloroethylene
TSM	Tank Storage Model
TVFM	theory of volume filling of micropores
VOC	Volatile organic compounds
cm <sup>3</sup> /min	centimeter cube per minute
h	hour
kg/L	kilogram per liter
kg/m <sup>3</sup>	kilogram per meter cube
m <sup>2</sup> /g	meter square per gram
min	minutes

mm	millimeter
nm	nanometer
s	second
wt %	percentage of weight



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## CHAPTER 1

### INTRODUCTION

#### 1.1 Overview

Carbon adsorbents in terms of activated carbon or carbon molecular sieves have been used as medium in adsorption and desorption process to reduce or eliminate objectionable gaseous components. Carbon adsorbents can be prepared from any raw materials that contain high carbon contents, relative cheap, availability, high stability, high adsorptive capacity, and have a regenerative characteristic due to the finer pore structure. Therefore, raw materials such as coconut shell (Noah, 1991), oil palm shell (Herawan, 2000; Soon, 2001) and guava seeds (Rahman & Saad, 2003) have been analyzed on their physical and chemical properties and excelled in areas of practical applications such as purification, separation and adsorption.

Previous research also interested to investigate the characteristics of carbon adsorbents with different textures such as pore volume and pore size distribution (Chiang et. al., 2002; Tang & Huang, 2005). In addition, plastic bags as solid wastes materials mixed with existing carbon adsorbents have been found as a promising tool to increase the adsorption and desorption capacity of gas (Pierella et. al., 2005). For that reason, the main assessment of this study is to examine the used of carbon adsorbent prepared from oil palm shells, coconut shell and low-density polyethylene (LDPE) plastic bags.

## 1.2 Research Background

The oil palm shells are selected as the main raw carbon material for present study. The carbon adsorbents produced from an oil palm shell is then compared with potential carbon adsorbents from coconut shells. The synthesis methods, carbonization and activation are used in the preparation of those materials. Careful investigations have been made from the handling and preparation step, to carbonization and activation process, especially in terms of temperature, pressure and time controlled. These carbon materials are used because of its prominent characteristics, such as high surface area, relatively uniform pore size, ordered pore structure and mechanical stabilities. The production of these materials is then defined based physical characteristic such as surface area and pore size distribution. Then, the carbon adsorbents are used to evaluate their potential in gas adsorption and desorption of nitrogen, LPG and methane.

## 1.3 Research Objectives

The objectives of this study are;

- i. To produce carbon adsorbents from oil palm shells;
- ii. To investigate the effect of temperature, pressure and time controlled on the carbon adsorbents characteristics such as surface area, pore size and pore volume;
- iii. To design and develop tank storage models in laboratory scale for hydrocarbon gas storage;
- iv. To evaluate the applicability of carbon adsorbents for hydrocarbon gas such as LPG and methane for adsorption and desorption applications;
- v. To assess the performance of the carbon adsorbents for the applications in hydrocarbon gas storage tank models.

## 1.4 Limitation of Study

Some limitations due to time factor and experimental equipments constraints should be considered such as;

- i. Small amounts (about 5 grams) of raw oil palm shell or carbon samples have been used for all the laboratory carbon processing, characterization and application analysis. Therefore, the results were based on the laboratory scale and not tested for pilot or industrial scale. The form of particles applied was in granular or powder form and not in the pelletized or extrudated particles;
- ii. In the preparation of microporous carbon adsorbents, operating parameters such as temperature, process hold time, flow rate gases, whereas the heating rate, particle size, etc. have been fixed for different processes;
- iii. To obtain surface area, mesopore and micropore volume and pore size distribution information of the carbons using Micrometric ASAP 2010 surface analysis. Various essential physical surface characteristics of the products were analyzed by physical gas adsorption method without involving the analysis of the surface chemistry although it may affect the adsorption behaviors.

## 1.5 The Importance of Study

Carbon adsorbents hold a strong market position due to their unique properties and low cost for adsorption and desorption applications. In addition, the results of this study provide deeper understanding of the mechanisms of adsorption and desorption through porous materials. This research is currently important for hydrocarbon gas adsorption and desorption systems especially for LPG and methane gas as alternative fuel in vehicles.