PREPARATION OF ACTIVATED CARBON FROM OIL PALM EMPTY FRUIT BUNCH FOR ADSORPTION OF PHENOL AND HYDROGEN

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To my dearest;

Husband,

Mak Abah, Family,

Supervisors,

and

Best friend

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ABSTRACT

This study was carried out to investigate the potential of empty fruit bunch (EFB) based activated carbon (AC) as a phenol and hydrogen adsorbent. The precursor was prepared in laboratory tube furnace by carbonization in nitrogen flow followed by carbon dioxide activation at 900°C, 10°C/min under 15 minutes residence time and treatment with potassium hydroxide (KOH) solvent at different concentrations (i.e. 0.5M, 1.0M and 2.0M). The optimization study on carbonization parameters; temperature, heating rate, and residence time on the phenol removal was investigated by using response surface methodology (RSM) with box-benken design. The optimal process conditions obtained was; 500°C carbonization temperature, 10°C/min of heating rate, and 80 min of residence time; which given 7.57% of phenol removal. The best condition EFB fibre size was the one with greater than 2 mm mesh size; gave 73% of phenol removal. The kinetics of adsorption was well described by pseudo-second order model whilst the adsorption equilibrium was best represented by Langmuir isotherm model. Hydrogen adsorption study was analysed at ambient pressure by Accelerated Surface Area and Porosimetry analyzer (ASAP 2020) and at high pressure by high pressure volumetric analyzer (HPVA). The AC that was activated by physical and followed by impregnated with 2M KOH adsorbed a maximum hydrogen adsorption capacity value of 2.14 wt% at 19 bars and -196.15 °C. Regeneration study was carried out by sodium hydroxide (NaOH) treatment and the regeneration efficiency (RE) of the AC has been reduced to 42 % after the third treatment. The Breuner, Emmer and Teller (BET) study showed that the ACs produced have surface area between 489 to 687 m^2/g. This study has identified EFB has a potential to be used as a precursor in the preparation of AC for phenol and hydrogen adsorbent.

ABSTRAK

Kajian ini dijalankan bagi mengkaji potensi karbon teraktif (AC) daripada tandan kosong kelapa sawit (EFB) sebagai penjerap fenol dan hidrogen. Bahan mentah diproses melalui proses pembakaran dengan aliran gas nitrogen menggunakan tiub relau pembakaran berskala makmal diikuti dengan pengaktifan karbon dioksida pada 900°C, 10°C/min selama 15 minit dan rawatan menggunakan larutan kalium hidroksida (KOH) pada kepekatan yang berbeza (iaitu: 0.5M, 1.0M and 2.0M). Kajian bagi mengoptimum parameter-parameter proses pembakaran; suhu, kadar pemanasan, dan masa terhadap penyingkiran fenol telah dijalankan menggunakan *response surface methodology* (RSM) dengan *box-benken design*. Proses kondisi yang optimum telah diperoleh pada keadaan; 500 °C bagi suhu pembakaran, 10 °C/min bagi kadar pemanasan, and 80 minit bagi masa pembakaran; iaitu berjaya menjerap sebanyak 7.57% penyingkiran fenol manakala AC dengan saiz jaringan fiber EFB lebih besar daripada 2 mm memberikan keputusan terbaik dengan sebanyak 73% penyingkiran fenol. Kajian terhadap kinetik penjerapan boleh diterangkan dengan lebih baik melalui model *pseudo-second order* manakala keseimbangan penjerapan boleh diwakili oleh model isoterma Langmuir. Kajian menjerapan hydrogen dijalankan pada tekanan sekitar menggunakan alatan *Accelerated Surface Area and Porosimetry* (ASAP 2020) dan pada tekanan tinggi menggunakan penganalisa isipadu bertekanan tinggi (HPVA). AC yang diaktifkan melalui proses pengaktifan fizikal diikuti rawatan dengan 2M KOH menjerap jumlah hidrogen yang tertinggi iaitu 2.14 wt% pada 19 bars dan -196.15 °C. Kajian penggunaan semula AC telah dijalankan dengan menggunakan natrium hidroksida (NaOH) dan kebolehan penggunaan semula (RE). AC menunjukkan bahawa kapasiti kebolehan penggunaan semula berkurang kepada 42 % selepas tiga kali rawatan. Kajian Breuner, Emmer dan Teller (BET) menunjukkan bahawa kesemua AC yang dihasilkan mempunyai luas permukaan di antara 489 ke 687 m^2/g. Kajian ini mendapati bahawa EFB mempunyai potensi untuk digunakan sebagai bahan pelopor dalam menghasilkan AC untuk penjerap fenol dan hidrogen.

CHAPTER 1

INTRODUCTION

1.1 Background of the Problem

The Malaysian palm oil industry generates a total of 15 million tonnes of empty fruit bunches (EFB) annually (as reported in 2008), representing one of the most abundant agricultural residues in the country. About 9.69 million and 6.36 million tonnes of EFB per year was produced in Peninsular Malaysia and in Sabah and Sarawak, respectively (Anis *et al.*, 2008). Numerous studies have been reported on the application of EFB including feedstock of activated carbon (AC) (Alam *et al.*,2007), production of cellulose (Astimar *et al.*, 2002), advanced carbon products (Astimar *et al.*, 2008), derivation of bio-oil (Abdullah and Gerhauser, 2008), liquefaction of EFB (Akhtar *et al*., 2009), and bio-oil production (Misson *et al*., 2009). One of the potential products from EFB is activated carbon, and more interest has been shown in this area. Thus, this research was aimed to study the ability of AC from EFB as phenol and hydrogen gas adsorbent.

1.2 Statement of the Problem

Malaysia is among the biggest exporter and producer of palm oil. About 5 million hectare of oil palm planted area was recorded in Malaysia by 2011 (MPOB, 2011). Meanwhile, about 21.88 million tonnes (wet weight basis) of empty fruit bunch (EFB) have been produced from the 423 palm oil mills in 2011. The carbon content on EFB has been reported as in the range of 42-43% (wet basis). The abundance of EFB has attracted many researches from several areas in pursuing the research and development (R&D) in preparing of many value added products such as activated carbon (AC) (Astimar *et al.,* 2011). ACs have wide range of application in industry, especially to cater the water and air pollution. It also has potential as hydrogen storage medium.

Phenolic is a type of pollutant which may give negative impact on human life as well as flora and fauna without proper treatment. High concentrations of phenolic compound (>1000 mg/L) can be found in palm oil mill effluent (POME) from oil palm industry. It is important to ensure that the phenol concentration on wastewater is up to regulatory standard before it can be removed into waterways. Currently phenol is treated by extraction process and by using conventional AC. However, the commercial AC which mainly produced by coal and petroleum is quite expensive and known as depleting thus, has increased the treatment cost. Therefore, conversion of EFB to AC can be used as an adsorbent for phenolic compounds treatment.

Hydrogen is one of renewable resources which may help in addressing the growing on energy demand and thus lower down the global climate change (Viktor, 2007). Four types of storage systems can be anticipated such as the compression of hydrogen gaseous, liquefaction of liquid hydrogen, metallic hydrides, chemical storage and physisorption process onto an adsorbent. However liquefaction of hydrogen needs high amount of energy (Züttel, 2003). Meanwhile compression requires very high pressure operations, which contribute to a high cost and rises on safety issues (Zhou, 2005). Besides, metallic hydrides are suspect to be poisoning, incompletely reversibility and low kinetics behaviour (Fierro *et al.*, 2010a). Above all, physisorption storage system on carbon materials is an interesting alternative for lower pressure needs and thus safer processes, complete reversible, fast kinetics (Fierro *et al.*, 2011a) environment friendly, economically feasible (Agarwal *et al.*, 1980 and Texie-Mandoki *et al.*, 2004) and offers relatively high hydrogen storage capacity.

Thus, this research studied the best operating conditions in conversion of EFB to activated carbon for the mean of liquid (phenol) and gas (hydrogen) adsorption.

1.3 Objectives of the Study

This research aims are:

i. To produce and optimize the production of oil palm EFB based AC for the removal of phenol.
ii. To characterize the EFB AC prepared by both physical (carbon dioxide) and chemical treatment (potassium hydroxide).
iii. To study the sorption capacity of AC for phenol and hydrogen adsorption.
iv. To study the regeneration capacity of the activated carbon by NaOH treatment.

1.4 Scope of Research

Empty fruit bunch which was supplied by Malaysia Palm Oil Board (MPOB) was used as precursor for the preparation of the AC. Response Surface Methodology (RSM) statistical software with Box-Benken application was applied to get the optimum conditions of experiment parameter (i.e. carbonization temperature, heating rate and residence time). This raw material will undergo carbonization process under N_2 flow at different range of fibre size (i.e. > 2 mm, 0.355-1.0 mm, and < 0.15 mm), carbonization temperature (i.e. 500 - 800 °C), heating rate (i.e. 6 °C /min -10 °C /min), and residence time (i.e. 40 - 120 minutes). RSM was applied only on carbonization process because it will be further activated under the optimum activation process parameters obtained by Alam *et al.*, (2009). The activation of the optimum char was done at CO_2 gas flow under 900 °C with 0.1 L/min CO_2 flow rate and activation time of 15 min (Alam *et al.*,2009). Different concentration of potassium hydroxide solutions (i.e. 0.5, 1.5 and 2.0M) were used under chemical treatment process. The AC produced was then characterized by Scanning Electron Microscope (SEM), Fourier Transformation Infrared (FTIR), Thermo Gravimetric Analysis (TGA), Carbon, Hydrogen, Nitrogen, Oxygen (CHNO) analysis, and Nitrogen adsorption isotherm analysis. The prepared sample's performance was then analysed by phenol adsorption test at different temperatures (i.e. 25, 35, 45 and 55 °C) and hydrogen adsorption at different pressures (i.e. ambient pressure - 100 bars). Finally the regeneration efficiency of ACs as phenol adsorbent was studied by chemical treatment with NaOH until three cycles of treatment processes were completed. The flowchart of the experimental design is to be illustrated in Figure 3.1.

1.5 Research Hypothesis

The hypotheses of this study are:

i. EFB is biomass by product which can be converted into high value added product such as the AC with significant values of phenol adsorption and hydrogen storage capacity (Fierro *et al.*, 2011a).

ii. KOH is a good alkaline activating agent for enhancing the micropore structure of the AC (Kunowsky *et al.*, 2008).

iii. The performance of EFB AC as phenol adsorbent can be regenerated for three treatment cycles after being treated with NaOH solutions.

iv. Smaller EFB fibre size, long carbonization time low temperature and higher heating rate are necessary to produce AC with large BET surface area (Demirbas, 2004).

v. Microporous material with high micropore content is essential for the adsorption of phenol (Liu *et al.*, 2010) and hydrogen (Sun and Paul, 2010; Zhao *et al.*, 2011a).

The AC was synthesised by the preparation of char under different carbonization parameters, and followed by CO_2 activation and KOH treatment which give an alternative as renewable adsorption material for phenol removal and storage of hydrogen.

1.6 Organization of Thesis

This thesis consists of five chapters. Chapter 1 presents problem background of the study, statement of problem, research's objectives, and of the research. Literature review on phenol in environment, hydrogen as future renewable energy, activated carbon, activated carbon adsorption, and optimization study are represented in Chapter 2. Research Methodology is presented in Chapter 3 which covers synthesis of AC, sample characterization, phenol adsorption study, hydrogen adsorption study, and regeneration study. The results and discussion of this study are presented in Chapter 4 at which the performance of AC in phenol and hydrogen adsorption, regeneration, and optimization study are discussed in detail. Summary of the research findings and some practical recommendations for future works are included in Chapter 5.

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EFB	-	Empty Fruit Bunches
AC	-	Activated carbon
CAC	-	Commercial activated carbon
GAC	-	Granular activated carbon
PAC	-	Powder activated carbon
R&D	-	Research and Development
MPOB	-	Malaysia Palm Oil Board
POME	-	Palm Oil Mill Effluent
US	-	United States
USEPA	-	United States Environmental Protection Agency
SEM	-	Scanning Electron Microscope
TGA	-	Thermo Gravimetric Analysis
FTIR	-	Fourier Transform Infrared Spectroscopy
CHNO	-	Carbon, Hydrogen, Nitrogen, Oxygen
RSM	-	Response Surface Methodology
TPR	-	Temperature Programmed Reduction
NKRA	-	National Key Result Area
GNI	-	Gross National Income
IUPAC	-	International of Pure and Applied Chemistry
BWD	-	Back washed and Drained
BET	-	Breuner, Emmer and Teller
ASTM	-	American Society for Testing and Materials
UK	-	United Kingdom

NaOH	-	Sodium Hydroxide
Mg/L	-	Milligram per litter
Min	-	Minutes
L/min	-	Litter per minutes
DIG	-	Digital Imaging Microscopy
ASAP	-	Accelerated Surface Area and Porosimetry Analyzer
HPVA	-	High Pressure Volumetric Analyzer
KH_2PO_4	-	Potassium dihydrogen phosphate
K_2CO_3	-	Potassium carbonate
NaOH	-	Sodium hydroxide
C_6H_5OH	-	Phenol
CO_2	-	Carbon dioxide gas
N_2	-	Nitrogen gas
H_2	-	Hydrogen gas
H_2O	-	Water
$-CH_2-$	-	Hydrocarbon chains
KOH	-	Potassium hydroxide
UV	-	Ultraviolet
BC	-	Char
BCF	-	Char fibre
DOE	-	Design of experiment
RE	-	Regeneration efficiency
ANOVA	-	Analysis of variance
DF	-	Degree of freedom
S	-	Sulphur
NAD	-	Nitrogen adsorption-desorption

%	-	Percent
K	-	Kelvin
q_e	-	Amount of adsorbent at equilibrium
q_t	-	Equilibrium rate constant
C_o	-	Initial concentration
C_e	-	Equilibrium concentration
V	-	Volume of solution
M	-	Absorber weight
$\frac{x}{m}$	-	Mass of adsorbate over mass of adsorbent
C_e	-	Equilibrium adsorbate concentration after adsorption
N	-	Freundlich intensity parameter
A	-	Amount of adsorbate to form a complete monolayer
B	-	Emphirical formula
R_L	-	Langmuir constant
M	-	Monolayer capacity
c	-	BET constant
i.e	-	It is
°C	-	Degree Celsius
C /min	-	Degree Celsius per minutes
G	-	gram
M_1	-	Amount of raw EFB before pyrolysis
M_2	-	Amount of char produced
Mm	-	Milimolar
Ml	-	Mililiter
C_o	-	Initial phenol concentration
C_e	-	Final (equilibrium) phenol concentration
V	-	Volume of solution
W	-	Mass of adsorbent

>	-	Greater than
<	-	Less than
Rpm	-	Revolutions per minute
T	-	Time
K	-	Equilibrium rate constant
K_F	-	Freundlich coefficient factor
K_L	-	Langmuir coefficient factor
k_1	-	Pseudo-first order equilibrium rate constant
K	-	Pseudo-second order equilibrium rate constant
Ki	-	Intraparticle diffusion constant
K_{DW}	-	Rate constant of adsorption
X_1	-	Carbonization temperature
X_2	-	Carbonization heating rate
X_3	-	Carbonization residence time
Y	-	Phenol adsorption uptake
Y_1	-	RSM second order polynomial function's predicted response
B	-	RSM second order polynomial function's coefficient
Ø	-	Pore diameter
Mpa	-	Mega Pascal
H	-	Hour
Å	-	Angstrom (10^{-9})
R^2	-	Correlation coefficient
Hg	-	Mercury
Cu	-	Copper
Pb	-	Lead
Ppm	-	Part per millions
Vol	-	Volume
R_p	-	Total particle radius
q_s	-	Average value of q (adsorption quantity) in a spherical particle at any particular time
q(r)	-	Local value of solid phase concentration
q_{if}	-	Average concentration in the solid at infinite time

D_s	-	Intraparticle diffusion coefficient
R	-	Radial position
Qt	-	Adsorption capacity at time t

LIST OF APPENDICES