

**PREPARATION OF MW-ASSISTED N-MODIFIED PALM SHELL
ACTIVATED CARBON FOR H₂S REMOVAL FROM
SIMULATED POME BIOGAS**

by

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LIST OF ABBREVIATIONS

AC	Activated Carbon
ATDSR	Agency for Toxic Substances and Disease Registry
BE	Binding Energy
BET	Brunauer-Emmett-Teller method
BOD	Biochemical Oxygen Demand
CER	Certified Emission Reduction
CDM	Clean Development Mechanism
CNT	Carbon Nanotube
COD	Chemical Oxygen Demand
DOE	Department of Environment
DTG	Differential Thermal Gravimetry
EA	Elemental Analyzer
EFB	Empty Fruit Bunch
EPP5	Malaysia's National Biogas Implementation
FFB	Fresh Fruit Bunch
FTIR	Fourier Transform Infrared Spectroscopy
GHG	Green House Gas
GHSV	Gas Hourly Space Velocity
GM	Gelombang Mikro
N-KTTKS	N-diubahsuai Karbon Teraktif Tempurung Kelapa Sawit
MFM	Mass Flow Meter
MTZ	Mass Transfer Zone

MW	Microwave
N-PSAC	Nitrogen Modified Palm Shell Activated Carbon
N-PSAC-R1	Regenerated Nitrogen Modified Palm Shell Activated Carbon Cycle- 1 st
N-PSAC-R20	Regenerated Nitrogen Modified Palm Shell Activated Carbon Cycle- 20 th
OFAT	One Factor At a Time
PSA	Pressure Swing Adsorption
PSAC	Palm Shell Activated Carbon
PSAC-TH	Palm Shell Activated Carbon under Thermal Heating
PSAC-MW	Palm Shell Activated Carbon under Microwave Heating
POME	Palm Oil Mill Effluent
RE	Regeneration Efficiency
RH	Relative Humidity
SEM	Scanning electron microscope
SN-PSAC	Spent Nitrogen Modified Palm Shell Activated Carbon
TGA	Thermogravimetric Analyzer
XPS	X-ray Photoelectron Spectroscopy

LIST OF SYMBOLS

A	Frequency factor or collision factor (min^{-1})
C_a	Initial concentration of gas in the particular gas cylinder (ppm)
C_b	Desired concentration of gas from the particular gas cylinder (ppm)
C_e	equilibrium concentration (ppm)
C_o	Feed concentration of sorbate (ppm)
C_t	Concentration of sorbate at time t (ppm)
E_a	Activation energy for reaction (kJ/mol)
F_s	Gas flow rate set by the mass flow controller (ml/min)
F_t	Total flow rate of gas mixture (200 ml/min)
k_{ap}	Deactivation of apparent reaction rate constant
k_d	Deactivation rate constant (min^{-1})
k_o	Initial sorption rate constant ($\text{m}^3/\text{kg}\cdot\text{min}$)
K_F	Freundlich isotherm constant ($\text{mg/g}(\text{L/g})^{1/n}$) related to sorption capacity
K_L	Langmuir isotherm constant (L/g)
L_{MTZ}	Length of mass transfer zone (cm)
m_c	Mass of sorbent used inside the bed (g)
n	Sorption intensity or surface heterogeneity
Q_f	Volumetric feed flow rate at STP (m^3/min)
Q_i	Sorption capacity of the regenerated sorbent in given cycle i
Q_o	Maximum monolayer coverage capacities (mg/g)
q	Sorbent sorption capacity (mg/g)
q_e	Equilibrium sorption capacity (mg/g)

q_t	Breakthrough sorption capacity at 5% from initial concentration (mg/g)
R	Specific gas constant (J/mol.K)
t	Time (min)
T	Temperature (K)
$t_{0.05}$	Breakthrough time at $C/C_o = 0.05$ (min)
$t_{0.95}$	Exhaustion time at $C/C_o = 0.95$ (min)
t_t	Time equivalent to the total or stoichiometric capacity (min)
W	Sorbent mass (kg)
W_i	Weight of the regenerated sorbent in given cycle i (g)
W_o	Weight of the initial sorbent (g)
y_f	mole fraction of the sorbate in the feed
ΔG°	Gibb's free energy change
ΔH°	Enthalpy change
ΔS°	Entropy change
ϵ'	Dielectric constant
ϵ''	Dielectric loss
$\tan \delta$	Dielectric loss tangent

**PENYEDIAAN N-DIUBAHSUAI KARBON TERAKTIF TEMPURUNG
KELAPA SAWIT MELALUI BANTUAN KETUHAR GELOMBANG MIKRO
UNTUK PENYINGKIRAN H₂S DARI SIMULASI POME BIOGAS**

ABSTRAK

Kehadiran H₂S di dalam biogas boleh mengganggu proses pertukaran gas metana (CH₄) kepada tenaga yang boleh diperbaharui disebabkan oleh kesan pengaratan kepada peralatan dan kesan keracunan kepada kesihatan manusia. Objektif utama kajian ini adalah untuk mengubahsuai sejenis karbon teraktif tempurung kelapa sawit melalui pengubahsuaian nitrogen (N-KTTKS) dan kaedah pemanasan ketuhar gelombang mikro (GM) untuk menyingkirkan H₂S daripada POME biogas yang disimulasi. Satu kajian awal mengenai pemilihan kaedah yang terbaik untuk menghasilkan bahan penyerap yang lebih berkualiti telah dilakukan dan kaedah pemanasan GM telah dipilih berbanding dengan kaedah pemanasan secara konvensional. Bahan penyerap N-KTTKS yang berkualiti tinggi telah dihasilkan melalui pemanasan GM dan kondisi pembolehubah GM yang digunakan adalah; 900 °C suhu pemanasan GM, 400 ml/min kadar aliran N₂, 10 minit masa pemanasan GM, dan 3 g jumlah sampel. Kaedah pemanasan GM menghasilkan penyatuan kumpulan berfungsi nitrogen di dalam matriks karbon N-KTTKS dengan lebih baik dan membangunkan struktur permukaan dan poros karbon, seterusnya dipercayai menjadi faktor utama yang menyumbang kepada kapasiti penyerapan H₂S yang tinggi. Dengan menggunakan bahan penyerap N-KTTKS bagi sebelum dan selepas penyerapan H₂S, hasil butiran pencirian menyimpulkan bahawa kedua-dua penyerapan fizikal dan kimia telah berlaku semasa proses penyerapan H₂S. Satu

analisis penembusan yang lengkap untuk pelbagai keadaan operasi iaitu kelembapan, suhu operasi, jumlah bahan penyerap, kadar aliran biogas dan kepekatan suapan H₂S telah mempengaruhi kapasiti penyerapan H₂S ke atas bahan penyerap N-KTTKS. Dapat disimpulkan bahawa tetapan sedia ada (iaitu 35 °C, 45% RH dan 3000 H₂S ppm) di kilang minyak sawit Felda Besout adalah sesuai untuk penyerapan H₂S dengan menggunakan bahan penyerap N-KTTKS tanpa mengubah parameter operasi. Penjanaan semula secara kering melalui kaedah GM terbukti dapat menjana semula bahan penyerap N-KTTKS tepu H₂S sehingga lebih daripada 100% keberkesanan penjanaan semula dan telah diuji untuk 20 kitaran penjanaan semula. Ramalan keseimbangan penyerapan H₂S menunjukkan bahawa model isoterma Freundlich sesuai dengan data eksperimen. Sifat-sifat termodinamik menilai penyerapan H₂S menggunakan bahan penyerap N-KTTKS sebagai proses eksotermik dengan spontan dan secara rawak. Tenaga pengaktifan dan faktor frekuensi bagi penyerapan H₂S adalah 15.60 kJ/mol dan $3.90 \times 10^2 \text{ s}^{-1}$, setiap satu. Model kinetik pendeaktifan didapati menepati data eksperimen penyerapan H₂S.

**PREPARATION OF MW-ASSISTED N-MODIFIED PALM SHELL
ACTIVATED CARBON FOR H₂S REMOVAL FROM
SIMULATED POME BIOGAS**

ABSTRACT

The presence of H₂S in biogas could disturb the conversion of methane (CH₄) to renewable energy due to its corrosive effect to equipment and its toxicity to human health. The main objective of this study is to develop a nitrogen-modified palm shell activated carbon (N-PSAC) via microwave (MW) heating method for H₂S removal from simulated POME biogas. A preliminary study on choosing preferable method of producing high performance of sorbent was done and MW heating method was selected compared to conventional heating method. The preparation of a high quality of N-PSAC sorbent was done under MW heating variables; 900 °C of MW heating temperature, 400 ml/min of N₂ flow rate, 10 minute of MW heating time and 3 g of sample amount. MW heating method provides better incorporation of nitrogen functional groups in the N-PSAC carbon's matrix and well-developed surface and porous structures are believed to be predominant factors that contribute in high H₂S sorption capacity. The details characterizations of the N-PSAC sorbent before and after H₂S sorption concluded that both physical and chemical sorptions were taken place during the sorption process. A complete breakthrough analysis of various operating conditions namely relative humidity, operating temperature, amount of sorbent, biogas feed flow rate and H₂S feed concentration were influencing the sorption capacity of H₂S onto N-PSAC sorbent. It can be concluded that the existing settings in Felda Besout palm oil mill (i.e. 35 °C, 45% RH and 3000 ppm H₂S) are favorable for H₂S sorption by using the N-PSAC sorbent without altering the

operating parameters. The MW dry-regeneration method is proven to regenerate H₂S saturated N-PSAC sorbent up to 100% regeneration efficiency and was tested for 20 regeneration cycles. The predictions of H₂S sorption equilibrium showed that Freundlich isotherm model fitted well with the experimental data. The thermodynamic properties evaluates the H₂S sorption using N-PSAC sorbent as an exothermic process with spontaneous and randomly distributed. The activation energy and frequency factor for H₂S sorption were found to be 15.60 kJ/mole and $3.90 \times 10^2 \text{ cm}^3/\text{g}\cdot\text{min}$, respectively. The deactivation kinetic model was found to give a very good agreement with the experimental data of H₂S sorption.