GASES BREAKTHROUGH ADSORPTION ON ACTIVATED CARBON OF POROUS SYNTHESIZED RENEWABLE MATERIAL

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Specially dedicated to MY LOVELY MOM AND DAD,

Mohd Zain Bin Mahmood

and

Siti Rokiah Binti Abdullah.

"To my family, lecturers and friends, who supported me each step of the way"

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ABSTRACT

Acidic and alkaline gases are the types of pollutant which may give negative impact on human life as well as environment without proper treatment. It is important to ensure that these types of gases concentration in oil and gas industry are up to regulatory standard before it can be released to surrounding. Activated carbon is one of the most effective adsorbents used in hazardous environmental treatments and it is considered to be sustainable, environmentally friendly, economical and efficient making it a superior and the most commonly used in adsorption process compared to other adsorbents. The research is aimed to synthesize agro-based solid waste materials to activated carbon (AC) and to determine the breakthrough time adsorption isotherm of single poisoning gases on the AC. In this study, palm empty fruit bunch and palm kernel shell were selected as the raw materials to produce AC. Char was produced through carbonization process and then undergone chemical treatments [potassium hydroxide (KOH), iron (III) chloride hexahydrate ($FECI_3.6H_2O$) and zinc nitrate hexahydrate ($Zn (NO_3)_2.6H_2O$)] followed by microwave treatment in order to produce three different AC. Effect of different gases on the sorbent performance for adsorption breakthrough study were investigated. Testing of the prepared samples was accomplished using adsorption column breakthrough experiment. The breakthrough time adsorption testing was performed at 100 mL/min flowrate for 3.6 g of sample. The samples were characterized by thermo-gravimetric analysis, scanning electron microscope, Fourier transform infrared and nitrogen adsorption isotherm. Nitrogen adsorption isotherm results showed that the commercial activated carbon, palm kernel activated carbon treated with KOH-Zn (PKAC-KOH-Zn), palm kernel activated carbon treated with KOH-$FeCI_3$ (PKAC-KOH-$FeCI_3$) and palm empty fruit bunch activated carbon treated with KOH (EFBAC-KOH) have Brunauer-Emmett-Teller surface area of 1,005.87, 259.17, 122.61 and 3.48 m^2/g, respectively. For adsorption breakthrough study, the longer breakthrough time for acidic and alkaline gas adsorption implied better adsorption breakthrough performance. The end results showed that breakthrough times for three type of AC were not much different as compared to commercial AC. PKAC-KOH-$FeCI_3$ was the best material for carbon monoxide and sulfur dioxide adsorption with 6.33 s and 51 s breakthrough time, respectively. PKAC-KOH-Zn produced the longest breakthrough time for chlorine adsorption of 1002.5 s and EFBAC-KOH produced the longest breakthrough time for ammonia adsorption of 175.67 s. As a conclusion, the findings revealed the potential of AC derived from palm kernel shell and palm empty fruit bunch as poisoning gases sorbents based on the good results obtained from characterization analysis and adsorption breakthrough study.

ABSTRAK

Gas berasid dan beralkali adalah jenis bahan cemar yang boleh memberi kesan negatif kepada kehidupan manusia dan juga alam sekitar tanpa rawatan yang betul. Adalah penting untuk memastikan yang kepekatan gas-gas ini dalam industri minyak dan gas mencapai standard peraturan sebelum dilepaskan ke persekitaran. Karbon teraktif (AC) adalah salah satu bahan penjerap yang paling berkesan digunakan dalam rawatan-rawatan alam sekitar yang berbahaya dan dianggap mampan, mesra alam, menjimatkan dan berkesan yang menjadikannya lebih bagus dan yang paling biasa digunakan dalam proses penjerapan berbanding dengan penjerap-penjerap yang lain. Kajian ini adalah bertujuan untuk mensintesis bahan sisa pepejal asas tani kepada karbon teraktif dan menentukan jangka masa perintisan isoterma penjerapan gas beracun tunggal ke atas karbon teraktif. Dalam kajian ini, tandan kelapa sawit kosong dan tempurung kelapa sawit dipilih sebagai bahan-bahan mentah untuk menghasilkan AC. Arang dihasilkan melalui proses pengkarbonan dan kemudiannya melalui rawatan kimia [kalium hidroksida (KOH), besi klorida heksahidrat ($FECI_3.6H_2O$) and zink nitrat heksahidrat ($Zn\ (NO_3)_2.6H_2O$)] diikuti dengan rawatan gelombang mikro dalam menghasilkan tiga jenis AC yang berbeza. Kesan gas berbeza ke atas prestasi penjerap untuk kajian penjerapan perintisan telah dilakukan. Pengujian sampel-sampel yang telah disediakan dicapai dengan menggunakan eksperimen perintisan turus penjerapan. Ujian penjerapan masa perintisan telah dijalankan pada kadar alir 100 mL/min untuk sampel 3.6 g. Sampel telah dicirikan melalui analisis termo-gravimetri, mikroskop imbasan elektron, inframerah transformasi Fourier dan penjerapan nitrogen isoterma. Keputusan penjerapan nitrogen isoterma menunjukkan sampel karbon teraktif komersial, karbon teraktif tempurung kelapa sawit yang dirawat dengan menggunakan KOH-Zn (PKAC-KOH-Zn), karbon teraktif tempurung kelapa sawit yang dirawat dengan menggunakan KOH-$FeCI_3$ (PKAC-KOH-$FeCI_3$) dan karbon teraktif tandan kelapa sawit kosong yang dirawat dengan menggunakan KOH (EFBAC-KOH) mempunyai luas permukaan Brunauer-Emmett-Teller masing-masing 1,005.87, 259.17, 122.61 dan 3.48 m^2/g. Untuk kajian perintisan penjerapan, semakin lama masa perintisan untuk penjerapan gas berasid dan beralkali bermakna prestasi perintisan penjerapan yang lebih baik. Keputusan akhir menunjukkan bahawa masa perintisan untuk tiga jenis AC adalah tidak banyak beza berbanding dengan karbon teraktif komersial. PKAC-KOH-$FeCI_3$ adalah bahan terbaik untuk penjerapan gas karbon monoksida dan sulfur dioksida dengan masa perintisan masing-masing 6.33 s dan 51 s. PKAC-KOH-Zn menghasilkan masa perintisan yang paling lama untuk penjerapan gas klorin dengan 1002.5 s dan EFBAC-KOH menghasilkan masa perintisan yang paling lama untuk penjerapan gas ammonia dengan 175.67 s. Kesimpulannya, hasil kajian mendedahkan potensi AC yang dihasilkan daripada tempurung kelapa sawit dan tandan kelapa sawit kosong sebagai penjerap gas beracun berdasarkan keputusan yang baik diperoleh daripada analisis pencirian dan kajian perintisan penjerapan.

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LIST OF ABBREVIATIONS

AC	-	Activated Carbon
ACFs	-	Activated carbon fiber
BET	-	Brunauer, Emmett and Teller
EFB	-	Empty Fruit Bunches
FTIR	-	Fourier Transform Infra-Red Spectroscopy
IDLH	-	Immediately Dangerous to Life and Health values
IUPAC	-	International Union of Pure and Applied Chemistry
MPOB	-	Malaysia Palm Oil Berhad
PAH	-	Polycyclic Aromatic Hydrocarbon
PEL	-	Permissible Exposure Limits
PKS	-	Palm kernel shell
PKC	-	Palm Kernel Char
POME	-	Palm Oil Mill Effluent
SEM	-	Scanning Electron Microscopy
STEL	-	Short Term Exposure Limit
TGA	-	Thermo-gravimetric analysis
TLV	-	Threshold Limit Values

LIST OF SYMBOLS

A	-	Cross sectional area
C_b	-	Breakthrough concentration
C_i	-	Inlet concentration
CI_2	-	Chlorine
CO	-	Carbon Monoxide
CO_2	-	Carbon Dioxide
C	-	Concentration of adsorbate at time t
cm	-	Centimeter
[C]	-	Ceiling Limit (never exceed)
C_o	-	Feed concentration of adsorbate
^oC	-	Degree Celsius
d	-	Diameter
%	-	Percentage
$FeCI_3$	-	Iron (II) chloride
GHz	-	Gigahertz
H_2	-	Hydrogen
H_2O	-	Water
H_2S	-	Hydrogen sulfide
H_3PO_4	-	Phosphoric acid
H_2SO_4	-	Sulfuric acid
k_v	-	Adsorption rate constant
kV	-	Kilo volt
KOH	-	Potassium hydroxide
K_2CO_3	-	Potassium carbonate
M	-	Molar / Molecular weight
m_c	-	Mass of adsorbent used inside the bed

mL/min	-	Milliliter per minute
n	-	Number of filter
N_2	-	Nitrogen
NH_3	-	Ammonia
NOx	-	Nitrogen oxide
NaOH	-	Sodium hydroxide
Na_2CO_3	-	Sodium carbonate
O_2	-	Oxygen
Pa	-	Pascal
ρ_c	-	Carbon density
PEEK	-	Polyetheretherketone
ppmv	-	Part per millions volume
q	-	Adsorbent adsorption capacity
Q	-	Inlet flow rate
Q_f	-	Volumetric feed flow rate at STP
SO_2	-	Sulfur Dioxide
S	-	Second
t	-	Time
t_β	-	Breakthrough time
t_t	-	Time equivalent to the total or stoichiometric capacity
W	-	Total weight of carbon
W_e	-	Adsorption capacity
$ZnCl_2$	-	Zinc chloride

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CHAPTER 1

INTRODUCTION

1.1 Background of Study

Malaysia generates abundant amounts of agricultural residues. The main agricultural crops planted in Malaysia are oil palm, coconut, cocoa, and rubber. Agricultural residue means that the by-products which are produced from processing of agricultural crops. The examples of the residues are paddy straw, palm empty fruit bunch, coconut shell, coconut husk, paddy husk, fiber and palm oil shell. Malaysia is presently the world's main exporter of palm oil despite the fact that it is the second largest manufacturer of the oil after Indonesia. Malaysia represents 39% of the world's palm oil generation and 44% of the world's exports (Malaysian Palm Oil Council, MPOC, 2012). The Malaysian oil palm industry is one of the most highly organised sectors of any national agriculture system of the world. Today, though, the focus has shifted to how well agriculture also meets universally accepted standards of sustainability. Oil palm is the most important product from Malaysia which enhances the agriculture and economy sectors. In order to create the probable for improvement of energy in the palm oil industry, there is necessary to consider disposal of mill residues and available uses (Abdullah *et al*., 2009). Palm oil mills can produce many products such as palm kernels, fiber, and empty fruit bunches. These products are produced in huge quantities. It has the ability as a source of renewable energy and also can be converted from dried oil palm wastes into the products which contain various values. Usage of oil palm unwanted materials as a source of energy will lead another environmental advantage like decreasing in carbon dioxide that discharged (Lim, 2000). Many methods have been utilized in the removal of the gas and liquid such as membrane separation, an adsorption method, chemical separation, distillation and others which give benefits to the industry. These methods contributed to the production of high-quality products, environmental friendly, and energy saving. However, adsorption is commonly used method due to its cost effective and outstandingly efficacious.

Adsorption on activated carbon has been proven to be better than others process for some treatments based on its simple design, fast adsorption kinetics and its ability to adsorb a wide range of pollutants efficiently. However, the available commercial activated carbons are still costly because of the starting material used are non-renewable and expensive i.e. coal, which is unjustified in pollution control applications (Yin *et al.*, 2007). Therefore, nowadays, many researchers have tried to produce activated carbons from renewable and cheaper precursors which were mainly agricultural by-products like biomass wastes. They produced activated carbon for removal of various pollutants. Many studies have been reported on the application of palm kernel shell (PKS) and empty fruit bunch (EFB). One of the potential products from PKS and EFB is activated carbon, and more interest has been shown in this area.

The present research focuses on the practical application. The activated carbon produced will be applied in the safety respiratory devices like gas masks. The utilization of the products commonly used in industries like manufacturing, oil and gas and petrochemicals which involve activities in the hazardous environment. The product is necessary and is required for all the people that involved in the unhealthy environment. Consequently, this study was planned to determine the ability of AC from PKS and EFB as acidic and alkaline gases adsorbents for the gas separation application.

1.2 Problem Statement

There are abundance of waste material from the palm-oil industry especially empty fruit bunches (EFB) material and palm kernel shell (PKS). The wastes will affect the environment which they are burning illegally. Therefore, some step should be taken to overcome the crisis in managing this waste which is by using the scientific method. Among all the wastes, PKS have been proven that the materials which having highest carbon content and surface area (Noor *et al*., 2014). Moreover, previous studies (Sumathi *et al*., 2015; Hesas *et al*., 2013; Nasri *et al*., 2014; Hamza *et al*., 2015 and Lee *et al*., 2015) reported that its ability to adsorb the gas and produced the good performance in term of adsorption capacity. Therefore, PKS has the potential to be converted into activated carbon (AC). Meanwhile, EFB wastes were selected as a precursor for the production of activated carbon due to the low cost and large quantity from palm oil industry.

From the scientific method, the EFB and PKS wastes were converted into value added products like activated carbon with proper method. Furthermore, nowadays, the commercial activated carbons were derived from natural materials like coal that contribute to the high costs of the adsorbents. The production of activated carbon by using biomass waste materials like EFB and PKS will minimize the environmental impacts and lead to the low cost of the adsorbents moreover, they are renewable and sustainable materials.

Acidic gases like carbon monoxide, carbon dioxide, chlorine, chlorine dioxide, sulfur dioxide and hydrogen sulfide are the types of contamination which may give the negative effect on human life and the environment without proper treatment. It is important to ensure that these types of gases concentration on oil and gas industry are up to regulatory standard before it can be removed to surrounding. Currently, the gases are adsorbed by conventional activated carbon. Nevertheless, the commercially activated carbon which predominantly produced by coal and petroleum is very costly and become depleting subsequently, has increased the treatment cost. In this manner, production of activated carbon derived by PKS and EFB can be applied as an adsorbent for acidic and alkaline gases treatment.

Therefore, this study focused on the best working conditions in a conversion of PKS and EFB to activated carbon for the mean of gas adsorption breakthrough.

Adsorption is a process of preferential removal of gas or liquid by adsorbing it on the surface of a solid. Activated carbon is most chosen as adsorbent due to many sources can be obtained from agricultural wastes which used as the precursor for the production of AC. The agricultural wastes are converted into activated carbon using chemical or physical activation or both.

Adsorption technology especially activated carbon is one of the most popular methods used in environmental pollution treatments and it is considered to be sustainable, environmentally friendly, simple, economical and efficient which makes it a superior and the most commonly used technique in adsorption compared to other methods (Auta and Hameed, 2013; Hesas *et al*., 2013; Tan *et al*., 2008; Tan *et al*., 2009; Tham *et al*., 2011), they also possess large surface area, high porosity, and rapid adsorption capabilities (Tham *et al*., 2011; Dehdashti *et al*., 2010; Prauchner and Rodriguez-Renoso, 2012).

1.3 Objective of Research

This research is focused on synthesizing activated carbon from palm empty fruit bunch (EFB) and palm kernel shell (PKS) for capturing and separating the acid gases. The objectives include the following:

i. To synthesize agro-base solid waste materials to activated carbon and to characterize the synthesized materials.
ii. To determine the breakthrough time adsorption isotherm of single acid gases on the activated carbon.
iii. To predict the saturation time based on simulation results.

1.4 Scope of Research

Palm empty fruit bunch and palm kernel shell were used as precursors for the production of the activated carbon. The raw palm EFB and PKS were washed and dried first before undergoing carbonization process under 150 cm^3/min nitrogen gas flows at temperature 700^0C and heating rate 10°C/min. The char produced from carbonization process was treated with chemical treatment by using potassium hydroxide (KOH) solution, Iron (III) chloride hexahydrate ($FeCI_3.6H_2O$) and Zinc nitrate hexahydrate, Zn $(NO_3)_2.6H_2O$ respectively. The activation process of char was prepared by microwave treatment. The physical activation of the char through microwave heating was done at 200 cm^3/min CO_2 flow rate with 400W power level. The prepared activated carbon was then being characterized by Thermogravimetric Analysis (TGA), Scanning Electron Microscope (SEM), Fourier Transformation Infrared (FTIR), Nitrogen adsorption isotherm analysis and determination kit of density. The performance of the sample was then analyzed by poisoning gases adsorption test with 10ppm concentration at low flow rate (100mL/min) and at a high flow rate (6L/min) for mixing with the air. The activated carbon was tested by acidic gases like CO, SO_2, CI_2 and alkaline gas of NH_3.Oxygen was tested to be used as the reference in the normal human breathing rate.

1.5 Significance of Study

Adsorption is one of the most effective and economic techniques. Additionally, adsorption can occur at ambient temperatures and low pressures, so it is relatively safe moreover in air purification also activated carbon is being used in controlling potential harmful, environmentally damaging and unpleasant odours. Moreover, carbon adsorption technology is very effective for many applications and the major attribute of activated carbon treatment is its ability to remove a wide variety of toxic organic compounds to non-detectable levels. Furthermore, in Malaysia, palm empty fruit bunch generates a large part of municipal waste and produce a problem of considerable concern for the environment. The use of this cheap, viable and readily carbon source for production of activated carbon for subsequent use in the adsorption of acid gases will go a long way in promoting environmental pollution mitigation and waste management which helps in environmental sustainability and in turn curb the potential danger of health risks.

The costing for managing the uncontrollable waste from palm oil production can be reduced by using palm kernel shell and empty fruit bunch as the raw material for the production of activated carbon. The activated carbon produced will be very economical by converting low value materials (agricultural wastes) to high value products (activated carbon). Besides that, renewable material sources like biomass-waste have been recognized all around the world as a key driver to accomplish economic development while guaranteeing minimum environmental damage. At the same time, the present development of green innovation and its related strategies have improved the development of renewable material sources in the nation (Foo-Yuen *et al.*, 2011). This study is special application in gas mask respiratory system using agricultural wastes as precursors for production of activated carbon. This special product used for the gas poisoning adsorption which follows the standard requirement from NIOSH and OSHA. The application and utilisation on these products is commonplace and widespread throughout the industry activities involving contaminated air and hazardous environments .The product is essential and in fact mandatory as required by NIOSH for certain environments.

The most important is there is no research and report available for the adsorption of the selected gases (CI_2, SO_2, CO, and NH_3) on the palm empty fruit bunch activated carbon that impregnated with potassium hydroxide (KOH) especially in adsorption breakthrough study. From the adsorption breakthrough study, the time for the adsorbent material to completely saturate with the adsorbate will be obtained and how long for the adsorbent can be used before it will be replaced or regenerated will be known. This result will be applied in the gas mask respirator application and the results will be compared to the application using commercial activated carbon.

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