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DEVELOPMENT OF CARBONISATION-ACTIVATION SYSTEM FOR PRODUCTION OF BIOCHAR AND ACTIVATED CARBON FROM OIL PALM KERNEL SHELL

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Chairman : Professor Suraini Abd Aziz, PhD
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Activated carbon is commonly produced through carbonisation process followed by activation process in two separate reactors. These two-steps or separate processes of carbonisation and activation contribute to energy and time consumption, the cost of materials and apparatus and low yield of activated carbon produced. Therefore, the carbonisation-activation system was designed to enable the activation stage to be continuously conducted after carbonisation without termination of the operation. This process could reduce the production cost, energy and time without compromising on the yield and quality of the activated carbon produced, while at the same time avoiding the involvement of chemical agent in the process.

Currently, the industry produce biochar by purchasing it from local or from backyard industry, which is producing it through conventional drum kiln or pit kiln methods, where this conventional heating methods will causes to environmental issue such as gaseous emission and particulate matter emission. Therefore, a study of the production of biochar with high higher heating value (HHV) and low gaseous emission from oil palm kernel shell using a microwave-assisted pre-carbonisation system was conducted. Microwave function is to provide heat to biomass materials that has the ability to reduce the treatment time and energy consumption during the pre-carbonisation process. Biochar with high HHV of 27.63 MJ/kg was obtained, whereby the emissions of the particulate matter <10 μm (PM_{10}) was 35 μg/m^3 below the standard limits set by the Malaysian Ambient Air Quality Standards (2014). Therefore, the biochar produced using microwave-assisted pre-carbonisation technology proposed in this study can be used as co-combustion for renewable energy generation.

The small-scale carbonisation-activation system using electric vertical reactor was developed to produce activated carbon from oil palm kernel shell as a preliminary study. The process from this preliminary study had resulted a high activated carbon yield of 32%, high fixed carbon content of 88.6% with surface area of 305.67 m^2/g. The activated carbon was further tested as bio-adsorbent on the removal of methylene blue. About 99.7% of methylene blue has been adsorbed using minimum dosage of bio-adsorbent 0.6 g/L for 24 hours of treatment time. The results have been correlated in the Freundlich isotherm which was well fitted to the experimental data over the methylene blue experimental concentration range with correlation coefficients of R^2=0.992.

In order to improve the surface area and yield of oil palm kernel shell activate carbon, a double insulated carbonisation-activation reactor was developed. This reactor was double insulated using low cement castable and covered around the internal space of the reactor with stainless steel plated and fibre glass jacketed heat insulation layer, which allow efficient heat transfer into the bed of material in the reactor. The optimisation carbonisation conditions achieved from carbonisation process using microwave-assisted pre-carbonisation system was applied to the new development double insulated carbonisation-activation system. The temperature of carbonisation process was 400°C for three hours carbonisation time. The activated carbon produced gave the surface area of 935 ± 36.7 m^2/g and 30% yield within only seven hours retention time which is the highest compare to the preliminary systems (small-scale carbonisation-activation system).

The concentration of carbon monoxide was 8.98 mg/m^3 lower than permitted level set by the Malaysian Ambient Air Quality Standards 2015. The activated carbon produced was then used as a bio-adsorbent to treat palm oil mill effluent (POME) final discharge at various dosages using 40 g/L bio-adsorbent, the total suspended solid (TSS), chemical oxygen demand (COD), colour and biological oxygen demand (BOD) were reduced from 240 mg/L, 604 mg/L, 3170 ADMI unit and 100 mg/L to 18 mg/L, 189 mg/L, 80 ADMI unit and 5.1 mg/L, respectively. The concentrations meet the river water quality standard set by Department of Environment Malaysia (DOE), suitable to be applied for wastewater treatment in the palm oil mill. Therefore, the high quality of activated carbon produced using the carbonisation-activation system developed in this study shown it potential for the various application in the palm oil industry.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Doktor Falsafah

PEMBANGUNAN SISTEM KARBON-AKTIF BAGI PENGHASILAN ARANG DAN KARBON TERAKTIF DARIPADA TEMPURUNG KELAPA SAWIT

Oleh

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Karbon teraktif biasanya dihasilkan melalui proses karbonisasi diikuti oleh proses pengaktifan dalam dua reaktor berasingan. Kedua-dua langkah atau proses karbonasi dan pengaktifan yang berasingan menyumbang kepada penggunaan tenaga dan masa, kos bahan dan peralatan dan hasil karbon teraktif yang dihasilkan rendah. Oleh itu, sistem pengaktifan karbonisasi telah direka untuk membolehkan peringkat pengaktifan dijalankan secara berterusan selepas karbonasi tanpa menghentikan operasi. Proses ini dapat mengurangkan kos pengeluaran, tenaga dan masa tanpa menjejaskan hasil dan kualiti karbon teraktif yang dihasilkan, sementara pada masa yang sama mengelakkan penglibatan agen kimia dalam proses tersebut.

Satu kajian mengenai pengeluaran arang menghasilkan nilai pemanasan tinggi (HHV) yang lebih tinggi dan pelepasan gas rendah dari tempurung kelapa sawit menggunakan sistem pra-karbonisasi dibantu gelombang mikro. Fungsi gelombang mikro adalah untuk memberikan haba kepada bahan biojisim yang mempunyai keupayaan mengurangkan masa rawatan dan penggunaan tenaga semasa proses pra-karbonisasi. Arang dengan HHV yang tinggi sebanyak 27.63 MJ/kg diperolehi dengan pelepasan bahan partikel <10 µm (PM_{10}), CO, NO_2, SO_2 dan HCl adalah di bawah had standard yang ditetapkan oleh Standard Kualiti Udara Ambien Malaysia (2015). Oleh itu, arang yang dihasilkan menggunakan teknologi pra-karbonisasi dibantu gelombang mikro yang dicadangkan dalam kajian ini boleh digunakan sebagai pembakaran bersama untuk penjanaan tenaga boleh diperbaharui.

Sistem pengaktifan karbonisasi berskala kecil telah dibangunkan untuk menghasilkan karbon teraktif dari tempurung kelapa sawit. Proses ini menghasilkan jumlah karbon teraktif yang tinggi sebanyak 32%, kandungan karbon tetap tinggi sebanyak 88.6% dengan luas permukaan 305.67 m^2/g. Karbon teraktif telah diuji dengan menggunakan metilena biru sebagai bahan penjerap untuk penyingkiran metilena biru. Sebanyak 99.7% metilena biru telah diserap menggunakan hanya minimum dos 0.6 g/L karbon teraktif bagi masa rawatan 24 jam. Hasilnya menunjukkan korelasi isotherm Freundlich yang sangat sesuai untuk karbon teraktif tempurung kelapa sawit dengan koefisien korelasi R^2=0.992.

Untuk meningkatkan luas permukaan karbon teraktif dari tempurung kelapa sawit, sistem reaktor karbon-aktif dual penebat telah dibangunkan. Sistem reaktor dual penebat ini menggunakan tuangan simen rendah dan diliputi dengan permukaan dalaman oleh besi tahan karat dan lapisan penebat haba kaca gentian seterusnya meningkatkan pemindahan haba yang efisien kepada bahan. Sistem ini beroperasi berdasarkan kepada suhu optimum yang dicapai semasa pembakaran arang dengan menggunakan sistem pra-karbonisasi dibantu gelombang mikro. Sistem ini menghasilkan karbon teraktif dengan luas permukaan 935 ± 36.7 m^2/g dan hasil yang tinggi sebanyak 30% diperoleh dalam masa pengekalan 7 jam sahaja. Kepekatan karbon monoksida 8.98 mg/m^3 lebih rendah daripada paras yang dibenarkan oleh Standard Kualiti Air Ambient Malaysia 2015. Karbon teraktif yang dihasilkan digunakan sebagai bio-penjerap untuk merawat pelepasan kumbahan minyak kelapa sawit (POME) pada pelbagai dos. Pada dos 40 g/L bio-penjerap, jumlah pepejal terampai (TSS), keperluan oksigen kimia (COD), warna, dan keperluan oksigen biologi (BOD) dikurangkan masing-masing kepada 18 mg/L, 189 mg/L, 80 unit ADMI dan 5.1 mg/L. Kepekatan tersebut memenuhi piawaian kualiti air sungai yang ditetapkan oleh Jabatan Alam Sekitar Malaysia (DOE), menjadikannya sesuai untuk digunakan untuk rawatan air kumbahan di kilang minyak sawit. Oleh itu, kualiti karbon teraktif yang dihasilkan dengan menggunakan sistem karbon-aktif berpotensi untuk mewujudkan pelbagai peluang dalam industri kelapa sawit.

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I certify that a Thesis Examination Committee has met on 17 January 2018 to conduct the final examination of Nahrul Hayawin binti Zainal on her thesis entitled "Development of Carbonisation-Activation System for Production of Biochar and Activated Carbon from Oil Palm Kernel Shell" in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The Committee recommends that the student be awarded the Doctor of Philosophy.

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TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF ABBREVIATIONS

HHV	Higher Heating Value
°C	Degree celcius
MJ/kg	Mega Joule per kilogram
mm	Milimeter
nm	Nanometer
PM_{10}	Particulate matter below 10 micrometer
CO	Carbon monoxide
CO_2	Carbon dioxide
O_2	Oxygen
C_2H_4	Ethylene
C_2H_6	Ethane
NO_2	Nitrogen dioxide
NO_x	Nitrogen oxides
Mg	Megagram
CH_4	Methane
H_2	Hydrogen
H_2O	Water
SO_2	Sulphur dioxide
HCl	Hydrochloric
L/min	Liter per minute
m^2/g	Meter square per gram
%	Percentage

BTU	British Thermal Unit
L	Liter
mg	Miligram
mg/L	Miligram per litre
s	Second
wt%	Weight percent
HC	Hydrocarbon
$ZnCl_2$	Zinc chloride
H_3PO_4	Phosporic acid
KOH	Potassium hydroxide
°C/min	Degree celcius per minute
C	Carbon
H	Hydrogen
N	Nitrogen
S	Sulphur
NDF	Neutral detergent fibre
ADF	Acid detergent fibre
ADL	Acid detergent lignin
rpm	Revolution per minute
C_e	Equilibrium concentration
q_e	Amount adsorbed
C_o	Initial concentration
psi	Pound-force per square
W	Mass of adsorbent

V	Volume
R_L	Equilibrium parameter
EFB	Empty fruit bunch
OPF	Oil palm frond
FFB	Fresh fruit bunch
OPT	Oil palm trunk
PKS	Palm kernel shell
PKC	Palm kernel cake
BOD	Biochemical oxygen demand
COD	Chemical oxygen demand
TSS	Total suspended solids
EJ	Exajoule
db	Dry basis
daf	Dry-ash free basis
NA	Not available
MW	Megawatt
POME	Palm oil mill effluent
K	Kelvin
K/s	Kelvin per second
mmol/g	Milimol per gram
mg/g	Miligram per gram
kW	Kilowatt
min	Minute
DOE	Department of Environment

BET	Brunauer-Emmett-Teller
TGA	Thermogravimetric analysis
FTIR	Fourier Transform Infrared
SEM-EDX	Scanning Electron Microscopy with Energy-Dispersive X-ray
keV	Kilo electronvolt
KBr	Potassium Bromide
W	Watt
Hz	Hertz
g/L	Gram per litre
PLC	Programmable logic controller
µm	Mikrometer
$\mu g/m^3$	Mikrogram per meter cubic
MB	Methylene blue

CHAPTER 1

INTRODUCTION

1.1 Research Background

Production of activated carbon from lignocellulosic biomass is a field of research that has gained increased interest in recent years because of its potential for the disposal of ligno-residues and agricultural waste solution and management. Activated carbon from lignocellulosic biomass is less expensive, is renewable and could effectively transform waste into wealth (Ioannidou and Zabaniotou, 2007). In addition, inexpensive lingo-residues with a low inorganic content and high carbon content, such as coconut shells, oil palm kernel shell, empty fruit bunch and wood are suitable for use as an activated carbon precursor (Ioannidou and Zabaniotou, 2007; Largitte et al., 2016; Tzvetkov et al., 2016).

Malaysia is the main palm oil producer in the world after Indonesia and produced massive volumes of oil palm biomass such as empty fruit bunch (EFB) and in the plantations there is a large quantity of oil palm frond (OPF), oil palm trunk (OPT) during replantation, mesocarp fibre, oil palm kernel shell (OPKS), oil palm kernel cake (OPKC) and palm oil mill effluent (POME) generated from the palm oil mills (Nahrul Hayawin et al., 2014). A statistical report has stated that about 454 mills remained process in 2016, with an overall of 100,416 million tonnes of FFBs being processed (MPOB, 2016). Therefore, it might be expected that about 4.44 million tonnes (dry weight) of oil palm kernel shells are produced annually. Currently, the palm oil mills use particularly empty fruit bunch and oil palm kernel shell as a boiler fuel for the generation of power (Bazargan et al., 2015; Lai and Goh, 2015; Suheri and Kuprianov, 2015), and the residual of the biomass will be cast-off via burning. However, the biomass excess can be transformed into beneficial and valued products, such as activated carbon production that will provide benefits and advantages to the oil palm industry.

Biochar is a carbonaceous product produced from thermal degradation of organic materials during carbonisation process at low temperature below than 800°C. Biochar can be used as a fertiliser (Zheng et al., 2017), carbon sequestration (Sheng et al., 2016), soil conditioner and soil improvement (Sandhu et al., 2017; Yue et al., 2017), as well as a biofuel (Idris et al., 2015a, 2015b; Nahrul Hayawin et al., 2017). However, there are also studies have been conducted by other researchers on biochar to its application as bioadsorbent by converting it to activated carbon.

In particular, activated carbon is the most significant and direct applications for use in environmentally friendly applications, and can sufficiently adsorb organic compounds and heavy metals in water and wastewater systems. For various applications, activated carbon needed sufficient pore size distribution, high mechanical strength, great adsorption ability and high absorptions of active sites, and the novel uses of activated carbon, such as electrodes, gas storage and super-capacitors keep expanding as a result of technological development (Lee et al., 2016; Schaefer et al., 2016; Wang et al., 2015; Wei et al., 2016; Yuan and Zhang, 2006). Activated carbons are commonly applied for numerous uses and applications, for examples in air cleaning, biogas enrichment, drinking water treatment, waste water treatment, food and beverages, industrialised processes and health applications (Daud and Houshamnd, 2010; Dalai et al., 2015; Nor Faizah et al., 2016; Seo et al., 2016; Wang et al., 2014).

1.2 Problem Statement

In the production steps of activated carbon, few challenges faced in two primary stepwise processes: firstly, carbonisation with the use of oxygen with a temperature below 700-800°C to produce biochar, and secondly the activation of the pyrolysis products uses chemical or physical activation to produce activated carbon. For the production of biochar, it has been found that self-sustained combustion methods using the partial presence of oxygen, without the use of an electric heater, were desirable to industry, owing to the simplicity of the process and low energy requirements (Idris et al., 2015a). However, long retention times required during carbonisation process under self-sustained carbonisation system resulted in low yields of biochar.

Microwave-assisted pre-carbonisation technology showed high ability and potential to be used during pre-carbonisation step before proceed with the carbonisation step (Nahrul Hayawin et al., 2017). In recent years, microwave-assisted pre-carbonisation has developed as an effective technology for the production of bio-oil and biochar (Alslaibi et al., 2013). Microwave provides heat to dielectric materials as a microwave absorber during carbonisation process. Microwave technology has many advantages and benefits over conventional heating technology; such as easy to operate, internal heating into the biomass materials, reduce processing time and energy, and uniform heating throughout the biomass materials (Alslaibi et al., 2013). It has been successfully studied that microwave technology reduces the treatment time of carbonisation process and produce high yield as compared with the conventional heating process (Pianroj et al., 2016).

Moderate microwave-assisted pre-carbonisation temperatures between 200°C and 300°C are sufficient to produce high-quality biochar with a higher heating value (HHV) of 18-27 MJ/kg using oil palm pressed fruit fibres (Hooi et al., 2009), which can then be used for fuel production or power generation. Specifically, this new approach could potentially produce an acceptable HHV with high yields for oil palm kernel shell derived biochar with low gaseous emission. The gaseous emission produced during the process can be treated in a gaseous treatment system connected to the microwave reactor to meet the national environmental standards before being discharged into the environment. Therefore, in this study, we conducted pilot-scale tests to assess the feasibility of using a microwave-assisted pre-carbonisation system for the production of biochar from oil palm kernel shell with high HHV and low gaseous emission. Therefore, a new, more efficient and innovative technology should be developed for the production of high-quality activated carbon using optimisation temperature and carbonisation time obtained from microwave-assisted pre-carbonisation system.

Carbonisation and activation processes are normally carried out separately. Carbonisation and activation processes contribute to the total cost of materials and devices, as well as to time and energy depletion. Hence, a single step that combines the carbonisation and activation process, carrying two-in-one, can decrease costs and energy depletion compared to the conventional two separate processes. Production of activated carbon via a carbonisation-activation system remains still limited; however, this strategy has been applied for the production of activated carbon by physical activation (Ngernyen et al., 2006; Ould-Idriss et al., 2011; Olivares-Marín et al., 2012; Vargas et al., 2010), and via the use of the microwave assisted-heating process (Ania et al., 2005; Foo and Hameed, 2011, 2012; Nabais et al., 2004; Yang et al., 2010), tubular electric furnaces (Hussein et al., 2001; Ould-Idriss et al., 2011), vacuum ovens (Rashidi et al., 2013) and thermal degradation equipment (Su et al., 2006; Osovsky et al., 2011; Velo-Gala et al., 2014).

The carbonisation-activation system enables a staged activation to be continuously conducted without stopping the operation. Carbonisation-activation to produce activated carbon using biomass feedstock is unique due to its simplicity of the design and the ease of feeding and uploading the reactor. Combinations of the carbonisation-activation step can reduce the production cost, energy and time without compromising on the yield and quality of activated carbon produced. In this study, the improvement of carbonisation-activation has been developed properly to allow efficient heat transfer into the bed of material in it, to produce high surface area and maintain a high yield and at low retention time. The optimisation temperature and carbonisation time during carbonisation of oil palm kernel shell using microwave-assisted pre-carbonisation system has been applied during carbonisation process using an improvement of carbonisation-activation system.

1.3 Objectives

The overall objective of this study is to develop a carbonisation-activation system for the production of biochar and activated carbon from oil palm kernel shell. Specific objectives are listed below:

1. To produce biochar with a high Higher Heating Value (HHV) and low gaseous emission from oil palm kernel shell using a microwave-assisted pre-carbonisation system;
2. To evaluate and characterise activated carbon of oil palm kernel shell at optimum carbonisation and activation temperature under carbonisation-activation system; and
3. To characterise and analyse palm oil mill effluent (POME) final discharge on BOD, COD, colour and TSS reduction after treated using activated carbon.

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