MICROWAVE-ASSISTED PRODUCTION OF PYROLIGNEOUS ACID FROM PALM KERNEL SHELL

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ABSTRACT

Oil palm plantation is one of the biggest contributors to Malaysian economy. However, the processing of oil palm fruit into palm oil resulted in the generation of huge volumes of biomass. Traditionally, oil palm biomass such as oil palm frond, oil palm trunk, palm kernel shell and empty fruit bunch was left at the plantation to decompose naturally, which with time, would ultimately lead to soil and water pollutions. Amongst the various solutions available to manage the abundance of oil palm biomass, pyrolysis offers an interesting solution where oil palm biomass can be converted into bio-oil, pyroligneous acid (PA) and biochar. The objectives of this study were to determine the optimum pyrolysis condition to produce PA with the highest total phenolic contents, to evaluate the potential of optimized microwave-assisted PA as antioxidant and antifungal agents and to investigate the adsorptive properties of the biochar produced from pyrolysis of palm kernel shell to remove dye from wastewater. The dye removal capability was evaluated using methylene blue (MB) based on three parameters namely initial concentration of MB, amount of adsorbent dosage and batch equilibrium studies. The adsorption equilibrium data was best fitted with the Freundlich isotherm model with R^2 of 0.95 and q_{max} of 27.23 mg/g. PA containing highest total phenolic contents (optimized PA) was produced at the following condition: final temperature of 480°C, microwave power of 580 W and nitrogen flow rate of 2.4 LPM (liter per minute). The ethyl-acetate extracted optimized PA showed an antifungal capacity with inhibition zone between 12.3 to 56.1 mm. Microwave-assisted concentrated pyroligneous acid (CPA) showed 2,2-Diphenyl-1-picrylhydrazyl (DPPH) free radical scavenging activity of 83 ± 2.11 µg/mL and trolox equivalent antioxidant activity (TEAC) of 35.75 ± 0.63 µg/mL while for industrial CPA, the value was 1096.24 ± 3.56 µg/mg (DPPH) and 1287.82 ± 2.89 µg/mg (TEAC). From the findings of this study, it can be concluded that biochar and PA produced from the pyrolysis of palm kernel shell showed good potential to be used as dye removal, antifungal and antioxidative agents.

ABSTRAK

Perladangan kelapa sawit merupakan salah satu penyumbang terbesar kepada ekonomi Malaysia. Walau bagaimanapun, pemprosesan buah kelapa sawit untuk dijadikan minyak kelapa sawit mengakibatkan terhasilnya sisa biojisim yang sangat banyak. Secara tradisinya, sisa biojisim kelapa sawit seperti pelepah kelapa sawit, batang kelapa sawit, tempurung isirung sawit dan tandan kelapa sawit dibiarkan mereput secara semula jadi di kawasan ladang, yang dengan masa, akhirnya akan menyebabkan pencemaran terhadap tanah dan air. Di antara pelbagai penyelesaian yang ada untuk mengurus masalah lambakan sisa biojisim kelapa sawit ini, pirolisis menawarkan penyelesaian menarik dimana sisa biojisim kelapa sawit boleh ditukar menjadi minyak-bio, asid piroligneus (PA) dan bioarang. Objektif kajian ini adalah untuk menentukan keadaan optimum pirolisis bagi menghasilkan PA dengan kandungan fenolik tertinggi, menilai potensi PA yang terhasil dengan berbantu gelombang mikro yang dioptimumkan sebagai agen antioksida dan anti-kulat dan menentukan sifat-sifat penjerapan bioarang yang dihasilkan daripada pirolisis tempurung isirung sawit untuk menyingkir pencelup dari air sisa. Keupayaan penyingkiran pencelup ditentukan menggunakan metilena biru (MB) berdasarkan tiga parameter iaitu kepekatan awal MB, jumlah dos penjerap dan kajian keseimbangan. Data keseimbangan penjerapan paling sesuai dengan model isoterma Freundlich dengan R^2 bersamaan 0.95 dan q_{max} bersamaan 27.23 mg/g. PA dengan jumlah kandungan fenolik tertinggi (PA optimum) telah dihasilkan pada keadaan pirolisis berikut: suhu akhir 480^0C, kuasa gelombang mikro 580 W dan kadar aliran nitrogen 2.4 LPM (liter per minit). PA optimum yang diekstrak menggunakan etil asetat menunjukkan sifat anti-kulat yang baik dengan luas zon perencatan 12.3-56.1 mm. *Concentrated pyroligneous acid* (CPA) berbantu gelombang-mikro menunjukkan aktiviti pengumpulan radikal bebas 2,2-Difenil-1-pikrilhidrazil (DPPH) sebanyak 83 ± 2.11 μg/mL dan *trolox equivalent antioxidant activity* (TEAC) sebesar 35.75 ± 0.63 μg/mL. Manakala CPA industri pula, nilainya adalah 1096.24 ± 3.56 μg/mg (DPPH) dan 1287.82 ± 2.89 μg/mg (TEAC). Berdasarkan kajian ini, dapat disimpulkan bahawa bioarang dan PA yang dihasilkan daripada pirolisis tempurung isirung sawit ini berpotensi untuk digunakan sebagai agen penyingkiran pencelup, anti-kulat dan antioksida.

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LIST OF ABBREVIATIONS

AIL	-	Acid-insoluble lignin
ANOVA	-	ANOVA
ASTM	-	American society for testing material
ASL	-	Acid soluble lignin
BET	-	Brunauer–Emmett–Teller
$C_6H_8O_6$	-	L(+)-Ascorbic acid
CO_2	-	Carbon dioxide
CCD	-	Central composite design
CPAE	-	Concentrated pyroligneous acid extract
DPPH	-	1,1-diphenyl-2-picrylhydrazyl
DTG	-	Derivative curve
EA	-	Ethyl acetate
EFB	-	Empty fruit bunch
FTIR	-	Fourier transform infrared analysis
FFB	-	Fresh fruit bunches
GAE	-	Gallic acid equivalent
HPLC	-	High performance liquid chromatography
H_2SO_4	-	Sulphuric acid
IUPAC	-	International Union of Applied Chemistry
LPM	-	Liter per minute
MB	-	Methylene blue
MF	-	Mesocarp fibre
MPOB	-	Malaysian palm oil board
Na_2CO_3	-	Sodium carbonate
NO_2	-	Nitrogen dioxide
NOCs	-	Neutral organic compounds
NREL	-	National renewable energy laboratory

O_2	-	Oxygen gas
OPT	-	Oil palm trunk
OPF	-	Oil palm fronds
PA	-	Pyroligneous acid
P/Po	-	Relative pressure
PKS	-	Palm kernel shell
POME	-	Palm oil mill effluent
R^2	-	Regression coefficient
RSM	-	Response surface methodology
SEM	-	Scanning Electron Microscopy
SRS	-	Sugars recovery standard
SO_2	-	Sulphuric acid
TEAC	-	Trolox Equivalent Antioxidant Capacity
TGA	-	Thermogravimetric analysis
TPC	-	Total phenolic content
WO_3	-	Tungsten trioxide

CHAPTER 1

INTRODUCTION

1.1 Research Background

The oil palm industry is one of the pillars of the Malaysian economy and plays a pivotal role in feeding and fueling the growing global population. Currently, Malaysia is one of the largest producers and exporters of palm oil in the world, accounting for 11% of production and 27% of the export trade of all oils & fats (MPOC, 2015). As the most versatile and productive oil crop, palm oil in Malaysia has made impressive and sustained growth in the global market over the past four decades, and it has reached 5.81 million hectares of plantation in 2017 (MPOB, 2017). Today, cultivation of oil palm has yielded 17.73 million tons of palm oil and 2.13 tons of palm kernel oil (MPOC, 2015). The replanting activity has been accelerated every year to provide high yield and Malaysian oil palm industry has been working efficiently to improve oil extraction rate and to expand the mill capacity in order to fulfil the demand in oil palm industry (Loh, 2017). Consequently, through the palm oil extracting process, a vast amount of oil palm biomass wastes generated because of the fast-growing palm oil industry. These abundant biomass wastes include empty fruit bunch (EFB), palm kernel shell (PKS), mesocarp fibre (MF), oil palm trunk (OPT), oil palm fronds (OPF) and palm oil mill effluent (POME).

Current management practice for oil palm wastes was involving landfill and open burning that will contribute to the release of greenhouse gases and global warming (Hassan *et al.*, 2011). In addition, open burning of oil palm biomass residues can lead to air pollutants as well as an emission of toxic chemicals (Abdullah and Sulaiman, 2013). Disposal of biomass waste into oil palm plantation (mulching activity) without recovering the remnant oil in the EFB contributes to environmental effects such as soil contamination, generation of methane from composting and housing of pest and diseases (Mathew and Zakaria, 2015). It was estimated that from every tonne of fresh fruit bunches (FFB), around 6% of PKS will be produced (Mohammad Dit, 2011). The palm kernel shell waste produced will require a large space of landfill to decompose naturally. Currently, Palm oil mill effluent is also hardly used and creates an accumulating problem at the oil production mills. Therefore, there are strong regulations on anti-dumping policy to be implemented in order to restraint palm oil mills from dumping biomass waste in the plantation and nearby mills. However, oil palm biomass residue has a huge potential to be utilized for applications in industries such as renewable energy and as feedstock for the production of various biochemicals.

Thermo-chemical conversion is a process commonly used to convert biomass waste into bio-fuels in the forms of solid (e.g., charcoal), liquid (e.g., bio-oils, methanol and ethanol), and gas (e.g., methane and hydrogen), which can be used further for heat and power generation (Zhang et al., 2010). This process can be divided into four major conversion routes such as combustion, gasification, liquefaction and pyrolysis process. Combustion is a spontaneously continued heat generation phenomenon of exothermic chemical reaction occurred at temperature range from 800 to 1200°C which is inexpensive, well understood and commercially available technology (Zhang et al., 2010). Next, gasification process occurred in a partial oxygen usually with above 350°C and above 10MPa to obtain fuel gases such as H_2, CO, CO_2, and CH_4 (Zhang et al., 2010). As for liquefaction, this process take place in low temperature and high pressure thermos-chemical process to broke down biomass into fragmanets of small molecules in water or other suitable solvent (Dermibas, 2000, Zhong and Wei, 2004). Amongst these available techniques, pyrolysis has received special attention as it can convert biomass directly into gaseous, oil-like liquid (bio-oil and pyroligneous acid) and a carbon rich solid residue (biochar) products in the absence of oxygen (Goyal *et al.,* 2008; Balat *et al.,* 2009). In addition, pyrolysis is an environmentally friendly method where it produces less hazardous gas due to lower process temperature (Czajczynska et al., 2017). There are two main categories of pyrolysis process namely conventional and microwave pyrolysis. Conventional pyrolysis has slow reaction time, heat transfer resistance, inefficient heating, lack of rapid heating, and non-selective heating (Salema and Ani, 2011). Thus, microwave pyrolysis has widely used as an alternative where its offer many advantages such as internal heating, shoter processing time, low relative energy consumption, environmental friendliness, quick start-up, automated and volumetric heating (Domingues et al., 2006 and Menendez et al., 2010).

Pyroligneous acid (PA), also known as wood vinegar, is an acidic reddish-brown aqueous liquid obtained from the condensation of gaseous parts generated during the pyrolysis process. PA consists of two main fractions namely liquid and semi-solid. The liquid fraction of PA is highly acidic and includes several families of volatile organic compounds such as organic acids, phenols, aldehydes, phenols, alcohols, ketones, pyranfuran derivatives, and polyphenolic compounds (Mathew and Zakaria, 2015). Phenol can be used as a wood preservative to discourage the growth of fungal and molds (Hamidah *et al*., 2015). The semi-solid fraction consists mostly of tar. Researches have indicated that PA has high potential to be used as antibacterial, antioxidant, termicidal and strong antifungal agent (Ma *et al*., 2013, Loo et al., 2008, Yatagai et al., 2004, Chalermsan and Peerapan, 2009 and Oramahi and Yoshimura, 2013). It is believed that phenolic derivatives are one of the groups of compounds responsible for antimicrobial activity of PA (Cowan, 1999). In addition, PA is easier to detoxify, considered low hazard and easier to dispose without adverse environmental effects.

For decades, fungi have been a major concern in agricultural, environment, human and animal health since its presence resulted in great economical loss due to plant diseases and low quality of production yield (Clark and Tilman, 2017)). In agricultural practice, fungi attack has been a lethal disease towards basal and steam oil palm trees (Kinge and Mih, 2011). Fungi are well known for their special ability to degrade lignin in woody material. They metabolize wood polysaccharides and produced an array of cellulolytic and hemicellulolytic activities which can contribute to the degradation of plant cell wall material (Copping and Menn, 2000). A wood preservative such as formaldehyde is used to treat the fungi to prevent the attack. However, these methods involve the use of chemical inputs that are harmful to humans, animals and the environment. In view of this, the application of PA offers a useful alternative to address this problem in a more environmental friendly way. Based on the previous study (Lee *et al.*, 2010), protection characteristics against fungi came from phenolic compounds such as terpenes, flavonoids, quinoids, alkaloids, stilbenes and tannins in PA. Mohan et al., (2008) stated that PA from pine wood, pine bark, oak wood and oak bark has inhibitory effects against sapstaining fungi. In addition, phenolic compounds have been claimed to be most likely responsible for fungicidal activities.

Dye materials are widely used in various industrial activities such as paper, plastic, silk, cotton, fabrics and ink manufacturing. As the demand grows in the industries, the use of dyes as colourant continuously increases, which will cause significant hazardous impacts such as visible pollution, limiting penetration of light and limiting the photosynthetic zone in the water body (Kumar *et al.*, 2011, Ong *et al.*, 2012. Nowadays, biochar from agricultural biomass were being consider as adsorbent due to its adsorption potential for wastewater pollution such as phenol (Tan *et al.*, 2009), dye (Cheng *et al.,* 2013) and heavy metals (Kolodyn´ ska *et al.*, 2016; Mohan *et al.*, 2007).

In recent times, biochar without the activation process seem to have potential as adsorbent for neutral organic compounds (NOCs) too (Yang and Sheng, 2003). The copiousness of polar functional groups on biochar surface enhanced NOC adsorption by biochar compared to activated carbon (Yang *et al.,* 2004). In addition, biochar offers a good potential for strong adsorption effect because it contains a large surface area and highly porous structure, which are important characteristics in an adsorbent.

1.2 Problem Statement

Various researchs have been investigated on the production of pyroligneous acid (PA) and activated carbon (AC) from oil palm waste produced every year from palm industries. Oil palm waste is increasing yearly due to high demand of palm oil. PKS waste was approximately five million tonnes generated through palm milling operation and required an enormous landfill space for decomposition process to take place naturally. Thus, PKS waste is converted into PA and charcoal products by using conventional and microwave pyrolysis. Previous studies showed that the production of PA via conventional heating required to produce at high temperature with uneven of heat distribution which is highly produced highly carcinogenic compounds and takes long time and energy comsumption. Therefore, microware heating can be an alternative to produce high quality of PA at lower temperature, shorter pyrolysis time and better heat distribution during pyrolysis process. Previous studies on the production of PA from PKS via microwave-assisted heating would focus on optimization of final temperature, sample weight, AC loading and nitrogen follow rate for the production of PA (Jamaluddin et al., 2013 and Chan et al., 2017). Hence, it is important to determine optimum pyrolysis condition to obtain highest total phenolic content (TPC) of PA for antifungal properties.

Fungal phyto pathogens effects on crop growth rate in the agricultural industry has raised major concerns on food supplies. Chemical pesticides agent has been used for decades to deal with these fungal attacks. However, chemical based pesticides are reported to be acutely and chronically hazardous to humans, animals and ecosystems. Therefore, there is a need to have efficient pesticides which is more friendly to the environment and ultimately human health. Currently, PA produced from lignocellulosic biomass is used as a natural fungicide to prevent fungi attack (Darah *et al.,* 2013). Further researches need to be conducted to compete with chemical pesticide as well as to raise the commercial value of these natural biological

Dyes are manufactured to meet the requirements of various types of industries such as textile, printing and paper industries. Normally these industries used synthetic dyes which is contributed to potential source of solution that ends up in waste water. Moreover, improper handling of dyes can result in adverse health and environmental impacts such as visible pollution, limiting penetration of light and limiting the photosynthetic zone in water body (Kumar *et al*., 2011, Ong *et al*., 2012) as well as potential mutagenic and carcinogenicity (Ratna and Padli, 2012). One of the most commonly used dyes is the Methylene Blue (MB) with known application in medical, chemical, pharmaceutical and aquaculture industries (Oz et al., 2011; Schirmer et al., 2011). Activated carbon has been used to remove MB due to its high surface area, microporous structure, pore volume, great adsorption capacity, effective regeneration, and high reactivity (Attia et al., 2008, Karagoz et al., 2008, Momcilovic et al., 2011, Tham et al., 2011). However, the corrosive nature of the chemical activating agents has negative environmental impacts and often limits its application (Awoyemi, 2011).

1.3 Research Objectives

The main objectives of this research study are:

1. To optimize the operating conditions of microwave-asissted pyrolysis for the production of pyroligneous acid from palm kernel shell containing highest total phenolic contents.
2. To evaluate the potential of optimized microwave-assisted pyroligneous acid as antioxidant and antifungal agents .
3. To investigate the dye removal properties of microwave-assisted production of biochar from palm kernel shell with highest surface area.

1.4 Research Scopes

The scopes of work are:

1. The laboratory scale microwave pyrolysis parameters include final temperature (300-600°C), microwave power (350-800 W) and nitrogen flowrate (1.0-5.0 L/min).
2. Use of Response Surface Method - Central Composite Design (CCD) for the production of optimised PA.
3. For comparing purpose, industrial biochar and PA produced in MPOB-UKM station in Bangi, Selangor was used.

4. Biochar and PA were analyzed using Fourier-Transformed Infrared Spectroscopy (FT-IR), Gas Chromatography-Mass Spectrometry (GC-MS), Brunauer Emmet Teller (BET), Scanning Electron Microscopy (SEM), proximate and ultimate analyses.
5. Optimized PA was evaluated for its antioxidative properties i.e. DPPH radical scavenging, and Trolox equivalent antioxidant capacity (TEAC).
6. *Aspergillus niger*, *Fusarium oxysporum*, *Saccharomyces cerevisiae* and *Candida albicans* were used for the evaluation of antifungal properties of optimized PA.
7. MB was used for dye removal studies using biochars where the effects of initial concentration (25-400 mg/L) and contact time, and adsorbent dosage (0.5-5.0 g) were evaluated.

1.5 Significant of the Study

Utilization of palm kernel shell is very important because it is one of the rich biomass resources in oil palm industries. The utilization of palm kernel shell biomass wastes can reduce the space area for decomposition at the oil palm mill as well as reducing the environmental problem. In addition, it can be converted into more beneficial substances, particularly in biochemical activities. Pyrolysis products of palm kernel shell can be turned into real potential sources in agricultural applications where biochar can be used for its adsorption properties and pyroligneous acid as a natural antioxidant micronutrient content and a natural antifungal agent.

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