UNIVERSITI PUTRA MALAYSIA

DEVELOPMENT OF BIOCHAR FROM OIL PALM FROND FOR PALM OIL MILL EFFLUENT TREATMENT

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By

LAWAL ABUBAKAR ABDULLAHI

Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfilment of the Requirements for the Degree of Doctor of Philosophy

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DEDICATION

This thesis is dedicated to His Excellency the Executive Governor of Borno State Professor Dr. Babagana Umara Zulum for the moral, financial support and encouragement throughout this program

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the degree of Doctor of Philosophy

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February 2021

Chair : Professor Mohd Ali Hassan, PhD
Faculty : Engineering

In order to meet the growing demand for adsorbents to treat wastewater effectively, there has been increased interest in producing biochar from sustainable biomass feedstocks at minimum energy input. Although physical and chemical activations have been commonly employed to produce activated biochar with superior textural properties capable of treating wastewater effectively, little attention has been given to the effect of biomass nature and pyrolysis agents on biochar structure and adsorption performance. Therefore, the purpose of this research is to evaluate the effect of biomass cellulosic content and steam pyrolysis on evolution of pyrogenic nanopores and molecular structures of biochar, and evaluates the performance of biochar from oil palm frond (OPF) with respect to treating POME final discharge. Commercial cellulose, OPF, and palm kernel shell were pyrolyzed at 630 °C, and their biochar structures were analyzed. Evaluation of biochar nanotexture based on cellulosic content revealed that commercial cellulose decomposed rapidly into non-graphitizing large size crystallites (65 nm) with substantial defects within their graphene sheets forming mesopores resulting in the external surface area (SA_{ext}) of 95.4 m^2/g and micropore surface area (SA_{mi}) of 231.2 m^2/g. Amorphous chars derived from lignin were thermally stable, slowing down the rapid formation of crystallites in biochar from palm kernel shell, which resulted to forming microporous structure with SA_{mi} of 377.0 m^2/g and SA_{ext} of 58.6 m^2/g. Biochar from OPF had SA_{mi} of 293.4 m^2/g and SA_{ext} of 73.3 m^2/g. The iodine number of the biochars from commercial cellulose, OPF and palm kernel shell correlated with the SA_{ext} demonstrating the suitability of mesoporous biochar for wastewater treatment. Increasing the pyrolysis temperature from 400 to 600 °C in presence of superheated steam increased the BET surface area (SA_{BET}) of biochar from OPF from 1.63 to 461.3 m^2/g, while increasing retention time from 2 to 10 h at 600 °C increased the SA_{BET} from 461.3 to 530.1 m^2/g. Comparatively, steam pyrolysis of OPF at 600 °C produced biochar with higher SA_{BET} of 461.3 m^2/g and SA_{ext} of 189.4 m^2/g compared to nitrogen pyrolysis with lower SA_{BET} of 368.4 m^2/g and SA_{ex} of 77.4 m^2/g demonstrating pore broadening activity of steam. Steam pyrolyzed biochar from OPF achieved a maximum adsorption capacity of 24.6 mg/g COD, 49 mg/g Pt-Co, 58.1 mg/g phenol, and 63.6 mg/g tannic acid by interacting with the contaminants through van der Waals force, π-π interactions,

H bonding, and water-bridged H bonding. Using 30 g/L dosage, the biochar from OPF exhibited an effective reduction of COD from 224 to 41.6 mg/g and color from 344 to 15 Pt-Co. Findings of this work revealed the tendency of cellulose to yield a mesoporous biochar and demonstrated the effectiveness of steam pyrolysis in terms yielding biochar with superior textural properties through its greater pore deepening and broadening activities. Biochar from OPF was capable of engaging into multiple adsorption mechanisms signifying its high affinity for a variety of organic contaminants and suitability for wastewater treatment.

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

PEMBANGUNAN BIO-ARANG DARI PELEPAH KELAPA SAWIT UNTUK RAWATAN SISA AIR DARI KILANG SAWIT

Oleh

LAWAL ABUBAKAR ABDULLAHI

Februari 2021

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Untuk memenuhi permintaan penjerap untuk merawat sisa air yang semakin meningkat dengan efektif, minat untuk menghasilkan bio-arang dari bahan biojisim yang lestari dengan input tenaga minimum telah meningkat. Walaupun kaedah pengaktifan secara fizikal dan kimia digunakan sebagai kaedah utama untuk menghasilkan bio-arang teraktif yang mempunyai liang nano yang unggul dan struktur molekul yang sesuai untuk rawatan sisa air, sedikit perhatian diberikan kepada kesan sifat biojisim dan ejen pirolisis kepada struktur bio-arang dan prestasi penjerapan. Oleh itu, tujuan penyelidikan ini adalah untuk mengkaji dan menerangkan kesan kandungan selulosa biojisim dan pirolisis stim terhadap perubahan liang nano pirogenik dan struktur molekul bio-arang serta menilai prestasi bio-arang dari pelepah kelapa sawit untuk rawatan pelepasan terakhir POME. Selulosa komersil, pelepah kelapa sawit, tempurung isirong kelapa sawit dipirolisis pada suhu 630 °C, dan struktur bio-arangnya telah dianalisis. Penilaian tekstur nano berdasarkan komposisi bio-polimer menunjukkan bahawa selulosa komersil terurai dengan cepat menjadi hablur bersaiz besar tanpa grafit (65 nm) dengan penghakisan yang besar di dalam kepingan grafin membentuk struktur liang meso megakibatkan luas permukaan luaran (SA_{ext}) sebanyak 95.4 m^2/g dan luas permukaan liang mikro (SA_{mi}) sebanyak 231.2 m^2/g. Arang amorfus terhasil dari lignin adalah stabil secara terma membataskan pembentukan hablur dalam bio-arang yang cepat dari tempurung isirong kelapa sawit dan mempelopori struktur liang mikro dengan bacaan SA_{mi} 377.0 m^2/g dan SA_{ext} of 58.6 m^2/g. Bio-arang dari pelepah kelapa sawit mempunyai bacaan SA_{mi} 293.4 m^2/g dan SA_{ext} 73.3 m^2/g. Bacaan nombor iodin bio-arang dari selulosa komersil, pelepah kelapa sawit dan tempurung isirong kelapa sawit saling berkait dengan SA_{ext} menunjukkan kesesuaian bio-arang dengan liang meso untuk rawatan sisa air. Peningkatan susu pirolisis dari 400 ke 600 °C dengan kehadiran stim superpanas meningkatkan luas permukaan (SA_{BET}) bio-arang dari pelepah kelapa sawit dari 1.63 kepada 461.3 m^2/g, manakala peningkatan masa penahanan dari 2 ke 10 jam pada suhu 600 °C meningkatkan SA_{BET} dari 461.3 kepada 530.1 m^2/g. Sebagai perbandingan, pirolisis stim dari pelepah kelapa sawit pada suhu 600 °C menghasilkan bio-arang dengan luas permukaan lebih tinggi iaitu 461.3 m^2/g dan SA_{ext} 189.4 m^2/g berbanding pirolisis nitrogen dengan luas permukaan lebih rendah iaitu SA_{BET} 368.4 m^2/g dan SA_{ex}

of 77.4 m^2/g menunjukkan aktiviti perluasan liang oleh stim. Bio-arang pelepah kelapa sawit dari stim pirolisis mencapai kapasiti penjerapan maksimum iaitu 24.6 mg/g COD, 49 mg/g Pt-Co, 58.1 mg/g fenol, and 63.6 mg/g asid tanik dengan berinteraksi bersama bahan cemar melalui daya van der Waals, interaksi π-π, ikatan hidrogen, dan ikatan hidrogen air. Dengan menggunakan dos 30 g/L, bio-arang dari pelepah kelapa sawit menunjukkan pengurangan COD dengan efektif dari 224 ke 41.6 mg/g dan warna dari 344 ke 15 Pt-Co. Penemuan penyelidikan ini mendedahkan kecenderungan selulosa untuk menghasilkan bio-arang liang meso dan menunjukkan keberkesanan pirolisis stim dari aspek penghasilan bio-arang dengan sifat tekstur yang unggul melalui aktiviti memperdalam dan memperluas liang yang lebih besar. Bio-arang dari pelepah kelapa sawit mampu menggunakan beberapa mekanisma penjerapan keafinan tinggi untuk pelbagai bahan cemar organic dan kesesuaiannya untuk rawatan sisa air.

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This thesis was submitted to the Senate of University Putra Malaysia and has been accepted as fulfilment of the requirement for the degree of Doctor of Philosophy. The members of the Supervisory Committee were as follows:

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Nomenclature and units

A	Specific surface area of biochar (m^2/g)
A_{Te}	Temkin isotherm equilibrium binding constant (L/g)
a_R	Redlich–Peterson isotherm constant (1/mg)
a_T	Tóth isotherm constant (L/mg)
b_{Te}	Temkin isotherm constant
C_e	equilibrium concentration (mg/L)
C_o	adsorbate initial concentration (mg/L)
C_s	adsorbate monolayer saturation concentration(mg/L)
E	Energy of adsorption (kJ/mole)
ΔG^0	Gibb's free energy (kJ/mole)
g	Redlich–Peterson isotherm exponent (heterogeneity factor)
ΔH^0	Change in enthalpy (kJ/mole)
K_F	Freundlich isotherm constant (mg/g) $(L/g)^n$, related to adsorption capacity
K_L	Langmuir isotherm constant (L/mg)
K_{RP}	Redlich–Peterson isotherm constant (L/g)
K_T	Toth isotherm constant (mg/g)
k_1	pseudo-first-order rate constant (1/min)
k_2	pseudo-second-order rate constant (g/mg·min)
k_{pi}	intraparticle diffusion rate constant (mg/g min)
q_e	amount of adsorbate in the adsorbent at equilibrium (mg/g)
$q_{e,cal.}$	calculated adsorbate concentration at equilibrium (mg/g)
$q_{max.}$	monolayer maximum adsorption per unit gram of biochar (mg/g)
q_t	amount of adsorbate adsorbed at time (mg/g)
R	universal gas constant (8.314 J/mol K)
ΔS^0	Change in entropy (kJ/mole K)
T	Temperature (K)
t	Time (min.)
t_T	Toth isotherm constant

Greek letters

α	Initial adsorption rate (mg/g· min)
β	desorption rate constant (g/mg)
β_{DR}	Dubinin–Radushkevich constant related to adsorption energy
π	two-dimensional spreading pressure

Subscripts

$_1$	Adsorbate 1 of binary component
$_2$	Adsorbate 2 of binary component
$_i$	i adsorbate of multiple component
$_j$	j adsorbate of multiple component
$_{max.}$	maximum

LIST OF ABBREVIATIONS

BC	Biochar
BET	Brunauer-Emmett-Teller
CC	Commercial cellulose
COD	Chemical oxygen demand
D	Biochar dosage
d_{002}	Interlayer spacing
FC	Fixed carbon
FWHM	Full width at half maximum
L_a	Crystallite diameter
L_c	Crystallite size
OPF	Oil palm frond
PD	Average nanopore diameter
PKS	Palm kernel shell
POME	Palm oil mill effluent
POMEF	Palm oil mill effluent final discharge
fPOMEF	Filtered palm oil mill effluent final discharge
SA_{BET}	Brunauer-Emmett-Teller surface area
SA_{mi}	Micropore surface area
SA_{ext}	External surface area
SEM	Scanned Electron Microscopy
VM	Volatile matter
V_{me+ma}	Mesopores and macropores volume
V_{mi}	Micropores volume
V_T	Total nanopore volume
XRD	X-ray diffraction

CHAPTER 1

INTRODUCTION

1.1 Background of study

In the past decades, there has been growing interest and attention in the scientific community on biochar, culminating into multidisciplinary areas for researches related to agricultural, engineering, and environmental applications (Tan et al., 2015). Biochar is a porous carbon-rich product obtained after thermal decomposition of biomass or organic waste at moderate pyrolysis temperature in an ambient condition of limited or unavailable air. Because of its porous structure, biochar has the potential to serve as an adsorbent for wastewater treatment as its surface area contains a wide range of functional groups capable of interacting with different types of contaminants (Dawood et al., 2017; Kan et al., 2016; Moreira et al., 2017). In addition to its adsorption affinity, sources for biochar feedstocks are abundant and renewable; accordingly, it is considered a more sustainable and cheaper alternative adsorbent in comparison to most conventional adsorbents. Other advantages are biochar can be produced without adding chemicals and consumed less energy, making its production environmentally friendly and cost-effective. Though biochar has less surface area per unit weight compared with activated carbon, the potential benefits of using it as an adsorbent for treating wastewater cannot be overlooked.

When biochar is the desired product, slow pyrolysis is the thermal treatment of choice to maximized yield (Ahmad et al., 2014) and to produce adsorbent for treating contaminated water (Tan et al., 2015). Some pyrolysis reactors that can achieve these goals include a fixed bed, fluidized bed, screw, rotating cone, and vacuum pyrolyzers. The basic operating parameters for these pyrolyzers are temperature and reaction environment. Feedstock condition is also an important parameter that limits the choice of a type of pyrolyzer. The extent of the surface area of biochars produced from lignocellulosic-based feedstocks using fixed bed reactor operated at 400 to 600 °C are between 13.6 and 316 m^2/g (Azuara et al., 2017; Lee et al., 2013; Mohanty et al., 2013), and between 18 and 170 m^2/g for biochars produced from non-lignocellulosic-based feedstocks (Agrafioti et al., 2013; Atienza-Martínez et al., 2020; Touray et al., 2014). A similar magnitude of surface area between 92 and 259 m^2/g was also reported for biochars produced from lignocellulosic feedstocks in vacuum pyrolyzer at 475 °C (Uras-Postma et al., 2014). Significantly low surface area (1.2 – 1.6 m^2/g) was reported for biochar produced from corrugated cardboard using a screw reactor (Sotoudehnia et al., 2020). Although the surface area of biochar is low compared with activated carbons, selection of suitable feedstocks, and optimum setting of pyrolysis parameters can ensure production of biochar with a high surface area.

The nature of feedstock used for producing biochar is an influential factor that contributes to surface area formation during pyrolysis. In order words, the proportion of feedstock components (i.e., biopolymers) and their conformation could have a considerable effect on the extent of the evolved surface area. Structurally altered lignocellulosic-based (e.g. corrugated cardboard) and digested biomass feedstocks produce biochars with a relatively low surface area that range from 1.2 to 93 m^2/g (Agrafioti et al., 2013; Sotoudehnia et al., 2020; Touray et al., 2014; Tsai et al., 2012) compared with biochars having a relatively higher surface area between 13.6 and 316 m^2/g produced from lignocellulosic-based biomass feedstocks with their structure preserved (Lee et al., 2013). During thermal decomposition of biomass, it is expected that restructuring and re-organization processes of the decomposing biopolymers would lead to the evolution of nanopores, identified as pyrogenic nanopores (Gray et al., 2014), within the biochar nanotexture. Keiluweit et al. (2010) conducted a molecular-level assessment study of the physical organization of biomass-derived biochar and observed a transition of the biomass, as it thermally decomposed, from transition chars to turbostratic char and also noticed a gradual increase in surface area with increasing proportions of turbostratic crystallites embedded in amorphous char. Graphene layers that are irregularly stacked in bilayer are known as turbostratic crystallites (Inagaki and Kang, 2014a; Rouzaud et al., 2015). Consequently, knowledge of the proportion of feedstock components and their conformation, along with their thermal decomposition behaviour, will assist in the selection of feedstock that can produce high surface area biochar.

Although the components of feedstock and their conformation can contribute to developing nanoporous biochar, the pyrolysis conditions that determine the degree of decomposition, dictate the extent of the surface area developed. Treatment temperature is the main pyrolysis condition that positively correlates with the thermal decomposition of biomass into successive phases of carbonization (Keiluweit et al., 2010). Components of lignocellulosic feedstock containing high volatile fraction such as extractives, cellulose and hemicellulose decomposed at < 400 °C. The remaining volatiles of intermediate products are released at heat treatment temperature between 400 °C and 500 °C due to the transformation process of amorphous chars into turbostratic crystallites. At this temperature range, continuous release of volatiles and structural reorganization of the charring carbons results in increased surface area (Chen et al., 2017). The heating rate during biomass pyrolysis is also an important parameter that enhances surface area formation as the proportion of the pyrolysis products highly depends on the rate of heating feedstock. High heating rate enhances the release of volatiles and encourages the formation of porous char products (Rangabhashiyam and Balasubramanian, 2019). In contrast, Bouchelta et al. (2012) observed surface area increased with increasing heating rate from 1 to 10 °C and declined at higher heating rates. They attributed the former to enhanced heat and mass transfer, which encouraged the release of volatiles, and the latter to partial graphitization of the carbonizing biomass components. Duration of pyrolysis was reported to be significantly longer at lower treatment temperature for releasing of volatile matter, indicating the decreasing significance of residence time at high pyrolysis temperatures (Rutherford et al., 2012). In general, pyrolysis temperature, retention time and heating rate are crucial production parameters that when optimally applied to a suitable feedstock can yield a high surface area biochar.

The nanopore structure of biochar determines the extent of a surface area developed. As a result of thermal decomposition of biomass, different types and sizes of pores evolved in the final solid product. Pyrogenic nanopores – usually small-sized voids < 100 nm – evolved within the carbonized cell fibers due to release of volatiles, restructuring and reorganization of charring carbons. The large-sized pores (1 to 100 μm) known as the residual macropores are inherited from plant cellular structure. These pores determine the total surface area of the biochar, and the majority of the surface area is located within the pyrogenic nanopores (Gray et al., 2014). The pyrogenic nanopores are identified as micropores (< 2 nm), mesopores (2 to 5 nm), and macropores (> 50 nm) based on the classification of International Union of Pure and Applied Chemistry (IUPAC). Bourke et al. (2007) proposed a nanotextural model to estimate the contribution of pyrogenic nanopores to the surface area. This model assumes the presence of voids within carbon graphene sheets forming turbostratic crystallite nanostructure. They attributed the reduction of surface area to a high proportion of amorphous carbon, which lacks voids and blocked pores within graphene sheets. Keiluweit et al. (2010) corroborated this assumption by studying the char forming mechanism of lignocellulosic-based biomass. Other nanotextural models simply assume the presence of pores within three-dimensional randomly distributed turbostratic crystallites, in which porosity is observed to decrease with degree of graphitization (Inagaki and Kang, 2014b). These nanotextural models clearly demonstrate the relative contribution of pyrogenic nanopores on the extent of the surface area developed particularly for non-graphitizing biochar.

1.2 Problem Statement

Recently, the oil palm industry in Malaysia has growing interest on converting the huge amounts of oil palm biomass residues and palm oil mill effluent (POME) it generated into value-added products aiming at achieving zero-waste emission in the mills, and a more sustainable and profitable palm oil industry (Ali et al., 2015). Accordingly, there is increasing interest and attention on producing biochar from the residues for treating POME, particularly its final discharge. A variety of oil palm biomass residues have been considered for biochar production: palm kernel shell, mesocarp fibre, empty fruit bunch, palm kernel cake, palm oil mill sludge, frond and trunk (Kong et al., 2014). However, despite the abundance of the residues, selecting biomass precursor capable of yielding high surface area biochar with appropriate nanopore structure using simple environmentally friendly production method to effectively treat POME final discharge has always been challenging. This limitation leads to further activation step associated with high energy input or chemical use, putting the sustainability of the industry into question. Although a lot of research have been conducted on converting oil palm biomass into a high surface area biochar by physical activation (Abd Waft et al., 2017; Jia and Lua, 2008; Zainal et al., 2018), and on their performance for removing residual contaminants in POME final discharge (Amosa et al., 2014; Parthasarathy et al., 2016; Rugayah et al., 2014), little is known about the contribution of lignocellulosic components and steam pyrolysis, on the textural properties of biochar and how these properties interact with the residual organic contaminants to improve adsorption performance. Correlation between the components of feedstock and textural properties will guide in the production of engineered biochar suitable for treating a specific wastewater.

Lignocellulosic biomass encompasses a multiscale complexity of an uneven mixture of cellulose, hemicellulose, and lignin with small fractions of nonstructural components such as extractives and ash (Wang et al., 2017). These structural components thermally decompose through different mechanisms following a series of reaction pathways to form a composite char having different nanopore structure (Kumar et al., 2020; Liu et al., 2015). Cellulose is known to produce a higher fraction of non-aromatic chars before conversion into condensed aromatic chars with a higher surface area than lignin-derived char (Deng et al., 2016; Sharma et al., 2004, 2001). Li et al. (2014) compared the textural properties of biochars from cellulose, lignin, and *Pinus radiata* wood pyrolyzed at 400 °C and 600 °C. Their findings showed that cellulose consistently produced biochar with higher surface area and total pore volume with a wider average pore size than biochar from lignin. Corroborating this claim, Deng et al. (2016) confirmed the presence of a lower percentage of micropores in cellulose-derived biochar – 80% for cellulose biochar and 87% for lignin biochar – suggesting that lignin predominantly produces microporous biochar. This finding is significant as the information on biochar nanopore structure can be partly deduced from the relative proportion of biomass structural components. Li et al. (2020) recommended that it would be more beneficial to investigate the control of biochar structure to produce biochar for targeted application. The outcome can be extended by broadening the scope into relating biomass components to biochar adsorption capacity.

In addition, the thermal stability of lignocellulosic biomass decrease in flowing super-heated steam atmosphere (Pütün et al., 2006; Pütün et al., 2008). Steam pyrolysis depressed the deposition of condensable volatile matter and preferentially removed amorphous carbons that could block pyrogenic nanopores in biochar (Antal and Grønli, 2003; Bourke et al., 2007). Corroborating this claim, Önal et al. (2011) pyrolyzed potato skin waste at 550 °C under either nitrogen or steam flow and examined bio-oil yield of 27.11 % and 41.09 % respectively. This means that steam activities during biomass pyrolysis encourage synthesis and removal of condensable volatiles. By this means promoting the evolution of pyrogenic nanopores in biochar nanotexture when compared to the conventional nitrogen pyrolysis. However, little information is available on the effect of steam as a pyrolyzing agent on biochar nanotexture.

Effective treatment of POME final discharge requires high surface area biochar with the adequate nanopore structure and functional groups to interact with its contaminants. For the contaminants to have access to the adsorption sites, nanopore sizes of adsorbent must be wide enough to avoid pore blockage by larger contaminants. Most highly lignified biomass feedstocks produced microporous biochars that are often suitable for adsorbing microcontaminants (Daud and Ali, 2004). Therefore, the production of biochar with adequate nanopore sizes for a specific task will require selection of the right feedstock capable of yielding the required porosity at low energy input.

Going by these assertions, it means that high cellulosic biomass feedstocks can produce high surface area biochar with wider nanopores using steam pyrolysis, which could be suitable for treating POME final discharge. Among the oil palm residues generated, oil palm frond best fulfils this criterion. In addition, it is the most abundant and renewable biomass in the oil palm industry, making it a cheap feedstock for biochar production. This research aims to fill the knowledge gaps mentioned by investigating the following hypothesis: (1) evolution of pyrogenic nanopores could be explained by composition and distribution of cellulose in biomass, (2) steam pyrolysis can improve the surface area of high cellulosic biomass at lower temperature and time, and (3) adsorption capacity of organic contaminants in POME final discharge and the aqueous solution is a function of biochar nanopores structure and surface area.

1.3 Research Objectives

The purpose of this research is to examine and describe the evolution of nanopores and molecular structure of biochar targeting at producing high surface area adsorbent from oil palm frond using steam pyrolysis and evaluate the effect of the structural properties and operational conditions on adsorption of organic contaminants. The specific objectives of this research are as follows:

1. To assess the effect of cellulosic content on the evolution of pyrogenic nanopores and molecular structure of biochar.
2. To evaluate steam and nitrogen pyrolysis methods and evaluate the effects of the steam pyrolysis process conditions on the textural and chemical properties of oil palm frond-derived biochar.
3. To investigate the effects of the biochar structural properties, particle size and adsorption conditions on adsorption capacity of organic contaminants in aqueous solution and palm oil mill effluent final discharge.
4. To identify the adsorption mechanisms during the interaction between the biochar from oil palm frond and the organic contaminants.

1.4 Scope and Limitation of the Study

The research activities carried out to achieve each objective is illustrated in Figure 1.1. The first objective involved characterization of cellulose, oil palm frond and palm kernel shell and production of their biochar at 630 °C followed by their analysis using thermogravimetric analysis, proximate and ultimate analysis, X-ray diffraction, nitrogen adsorption-desorption isotherm, FTIR and point of zero charge pH aimed at correlating biomass cellulosic composition and pyrolysis with the evolution of nanopores and molecular structures and developing biochar structural nanopore model. The activity of the second objective included a comparison between steam and nitrogen pyrolysis methods and producing series of biochars from oil palm frond using one-step steam pyrolysis by varying pyrolysis temperature and retention time so that the one-step pyrolysis process performance could be evaluated based on the evolution of pyrogenic nanopores, surface area and surface functional groups. The third objective encompassed conducting a series of adsorption experiments using simple batch adsorption method for systematic evaluation of the effect of structural properties and particle size of the biochar from oil palm frond and adsorption conditions on the adsorption of organic contaminants in aqueous solution and POME final discharge. The final activity involved adsorption isotherm experiments and simulation of the isotherm data using appropriate equilibrium and kinetic isotherm expressions aimed at revealing the interaction mechanism between the biochar and organic contaminants and determined biochar condition suitable for adsorption.

Evaluation of cellulosic content on evolution of pyrogenic nanopores in this study did not take into consideration the effect of biomass mineral content. Presence of some minerals results to catalytic effect during biomass pyrolysis and mineral such as silicon are known to suppress the formation of pyrogenic nanopores. Using steam as pyrolyzing agent requires additional energy for heating water to produce steam. This additional requirement necessitate for utilizing corrosive resistant boilers and piping materials.

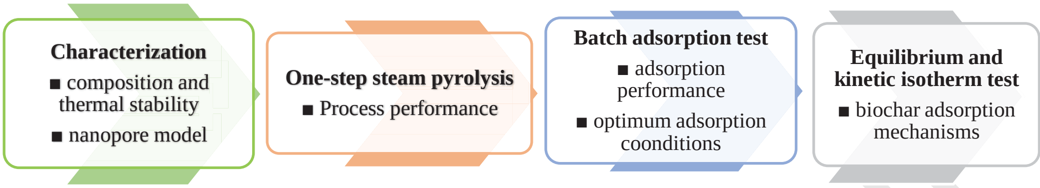

Figure 1.1: Graphical representation of the flow of research activities

1.5 Significance of the Study

The findings of this study will contribute significantly to the sustainability oil palm industry, considering that biochar has become an essential multifunctional material, particularly in environmental management. The greater demand of cost-effective biochar for treating POME final discharge in oil palm mills justifies the need for utilizing a high surface area biochar with adequate nanopores structure produce from oil palm biomass using a greener one-step steam pyrolysis approach. Therefore, mills that apply the biochar production protocols established in this study, which is easy and less time consuming, will be able to produce high surface area biochar with adequate nanopore structure to effectively treat their POME final discharge. The findings of this study will guide producers as to the most suitable biomass capable of producing high surface area biochar using one-step steam pyrolysis technique. For the research community, the study will point to a critical area for the evolution of pyrogenic nanopore in lignocellulosic-based biochar that was not revealed before. Thus, a new model on biochar nanopore structure could be developed.

1.6 Thesis Organization

This thesis is structured into five chapters that begin with the introduction, followed by a literature review, then materials and methods before results and discussions and ends with conclusions and recommendations. References and appendices come after the fifth chapter. The basic background and problem that necessitates research on biochar production from oil palm frond are covered in the first chapter. Chapter 1 also covers the aim, objectives, scope and significance of the research. Chapter 2 deals with a comprehensive review of biochar structure and major production factors influencing its evolution. It also includes factors influencing biochar adsorption and its interaction mechanisms with organic contaminants, and previous studies on characteristics of POME final discharge. Chapter 3 covers a description of materials and equipment used, the methods employed for biochar production and characterization, adsorption experiments, and data analysis. The fourth chapter presents the results and its thorough discussion and findings of the research. The final chapter provides the overall summary of the research findings and suggestions for further modifications and improvement.

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APPENDICES

Appendix A

Raw data of FTIR analysis for raw CC

PE IR SPECTRUM ASCII PEDS 1.60
-1
~SP.SP
20/09/07
12:01:59.00
20/09/07
12:02:28.00
Administrator

650.000000
4

SPECTRUM 100, CPU32 MAIN 00.02.8200 12-DECEMBER-2006 10:00:00
MIR TGS
MIR

77353
0.200000

4.000000
STRONG
RATIO
SPECTRUM
MAGNITUDE
MIRACLE ATR
DOUBLE

COMBINED

0

#HDR
-1
-1
#GR
CM-1
%T
0.00002384185791015625
0.0
4000.000000
-1.000000

3351
8
100.000000
3.162277
#DATA

4000.000000	86.869097
3999.000000	86.617851
3998.000000	86.446428
3997.000000	86.395616
3996.000000	86.460673
3995.000000	86.604268
3994.000000	86.757122
3993.000000	86.835145
3992.000000	86.797837
3991.000000	86.673601
3990.000000	86.529692
3989.000000	86.442215
3988.000000	86.466302
3987.000000	86.602027
3986.000000	86.784916
3985.000000	86.911377
3984.000000	86.890169
3983.000000	86.689488
3982.000000	86.354481
3981.000000	85.985068
3980.000000	85.680711
3979.000000	85.484469
3978.000000	85.368728
3977.000000	85.276049
3976.000000	85.173496
3975.000000	85.065549
3974.000000	84.968385
3973.000000	84.892304
3972.000000	84.853606
3971.000000	84.885769
3970.000000	85.019087
3969.000000	85.245256
3968.000000	85.509693
3967.000000	85.742700
3966.000000	85.895900
3965.000000	85.955484
3964.000000	85.926977
3963.000000	85.814492
3962.000000	85.623828
3961.000000	85.375060
3960.000000	85.098840
3959.000000	84.807175
3958.000000	84.468905
3957.000000	84.032615
3956.000000	83.480178
3955.000000	82.860490

3954.000000	82.274320
3953.000000	81.825942
3952.000000	81.576383
3951.000000	81.515883
3950.000000	81.570858
3949.000000	81.643122
3948.000000	81.673681
3947.000000	81.679430
3946.000000	81.719894
3945.000000	81.838558
3944.000000	82.037483
3943.000000	82.295937
3942.000000	82.593139
3941.000000	82.896850
3940.000000	83.150216
3939.000000	83.292211
3938.000000	83.308487
3937.000000	83.256260
3936.000000	83.221525
3935.000000	83.259786
3934.000000	83.378220
3933.000000	83.566296
3932.000000	83.816242
3931.000000	84.098475
3930.000000	84.340451
3929.000000	84.456455
3928.000000	84.399386
3927.000000	84.188658
3926.000000	83.901033

Spectroscopic assignments for raw CC, OPF, PKS and their respective biochar

	Cellulose	Cellulose char	OPF	OPF char	PKS	PKS char
Hydroxyl (-OH) stretch	3353	3466	3363	3456	3370	3463
Aromatic C-H stretch	-	3028	-	3024	3020	3023
Symmetric stretch of aliphatic CH_3	-	2950	-	2950	2973	2950
Asymmetric stretch of aliphatic CH_3	-		-		-	
Symmetric stretch of aliphatic CH_2						
Asymmetric stretch of aliphatic CH_2	2885	2891	-	2883	-	2884
Ester, carboxyl C=O stretching	1731	1740	1724	1739	1740	1738
Aromatic -C=C-, -C=O- stretching	1642	1634	1628	1636	1642	1635
CH, CH_2 and CH_3 deformation	1428	1430	1418	1431	1425	1429
-C-H or O-H bending vibration	1369	1369	1358	1370	1369	1370
C-O-C stretching	1057	1061	1043	1058	1051	1064
Aromatic C-H out-of plane deformation	-	888	-	888	-	895
Aromatic C-H out-of plane deformation	757	760	754	771	776	768

Appendix B

Summary of nitrogen adsorption -desorption isotherms of biochar from commercial cellulose

Full Report Set

MicroActive for TriStar II Plus 2.03 | MicroActive for TriStar II Plus Version 2.03 | Page 1
Serial # 384 Unit 1 Port 2

Sample: 000-484
Operator: NAZRUL
Submitter: ABU BAKAR
File: C:\Users\BET\Documents\ABUBAKAR AB...\CELLLOSE 600.SMP

Started:	9/4/2020 5:26:06 PM	Analysis Adsorptive:	N2
Completed:	9/5/2020 5:58:48 AM	Analysis Bath Temp.:	-195.800 °C
Report Time:	9/7/2020 10:12:41 AM	Thermal Correction:	No
Sample Mass:	0.0579 g	Warm Free Space:	16.1154 cm³ Measured
Cold Free Space:	49.0598 cm³	Equilibration Interval:	5 s
Low Pressure Dose:	None	Sample Density:	1.000 g/cm³
Automatic Degas:	No		

Summary Report

Surface Area

Single point surface area at p/p° = 0.299866699: 276.6043 m²/g

BET Surface Area: 326.6191 m²/g

Langmuir Surface Area: 468.1260 m²/g

t-Plot Micropore Area: 231.2176 m²/g

t-Plot External Surface Area: 95.4015 m²/g

Pore Volume

Single point adsorption total pore volume of pores less than 1.1614 nm width at p/p° = 0.011457285: 0.112257 cm³/g

Single point desorption total pore volume of pores less than 20.6503 nm width at p/p° = 0.900000000: 0.202001 cm³/g

Pore Size

Adsorption average pore diameter (4V/A by BET): 1.37477 nm

Desorption average pore diameter (4V/A by BET): 2.47384 nm

BJH Adsorption average pore width (4V/A): 4.0540 nm

Appendix C

XRD profile of biochar from commercial cellulose

```
//////////////////////////////////////////////////////////////////////////////
/// Profile Data Ascii Dump (XRD)                                          ///
//////////////////////////////////////////////////////////////////////////////

Group     : Syam
Data      : A_Bakar_Cell600(1)
File Name : A_Bakar_Cell600(1).RAW

# Profile Datafile
Sample Name        = A_Bakar_Cell600(1)
comment            = start from 5 degree
date & time        = 20-08-26 16:24:37

# Measurement Condition
X-ray tube
target             = Cu
voltage            = 30.0 (kV)
current            = 30.0 (mA)
Slits
divergence slit    = 1.00000 (deg)
scatter slit       = 1.00000 (deg)
receiving slit     = 0.30000 (mm)
Scanning
drive axis         = Theta-2Theta
scan range         =    5.000 -   50.000
scan mode          = Continuous Scan
scan speed         =   2.0000 (deg/min)
sampling pitch     =   0.0200 (deg)
preset time        =   0.60 (sec)

# Data      [ Total No. = 2251 ]
<2Theta>   <   I   >
5.0000      184
5.0200      194
5.0400      194
5.0600      188
5.0800      190
5.1000      184
5.1200      186
5.1400      178
5.1600      194
5.1800      196
5.2000      176
5.2200      174
5.2400      178
5.2600      168
5.2800      204
5.3000      186
```

5.3200	176
5.3400	186
5.3600	164
5.3800	180
5.4000	188
5.4200	146
5.4400	182
5.4600	214
5.4800	224
5.5000	162
5.5200	162
5.5400	202
5.5600	164

Appendix D

TGA profile of commercial cellulose

Filename: C:\Program Files\PerkinElmer\Pyris\data\Abu Bakar\VM Data\CELLULOSE CHAR VM.thd
Operator ID: Abubakar
Sample ID: CELLULOSE CHAR620
Comment:
Serial Number: 005031906
Data Collected: 6/8/2020 3:20:09 PM
Sample Weight: 16.078 mg
Display Weight: 16.078
Validation
Validated: No
By:
Date:
Calibration Information
Filename: C:\Program Files\PerkinElmer\Pyris\Calibrations\Brian\Cal 21-05-2015\Cal 18-03-2019.t6c
Date/Time: 18/3/2019 10:06:59 AM
Initial Conditions
Temperature: 50.00 °C
Baseline Filename:
End Condition: Go To: 45.00°C
Total Points in Run: 2328
Method Steps:
Pre-Run Actions
Start the Run
Action occurs Immediately
Switch the Gas to Nitrogen at 80.0 ml/min
Action occurs Immediately

1) Heat from 50.00°C to 110.00°C at 50.00°C/min

2) Hold for 15.0 min at 110.00°C

3) Heat from 110.00°C to 950.00°C at 100.00°C/min
Step Detail: 500

4) Hold for 7.0 min at 950.00°C

5) Cool from 950.00°C to 110.00°C at 200.00°C/min

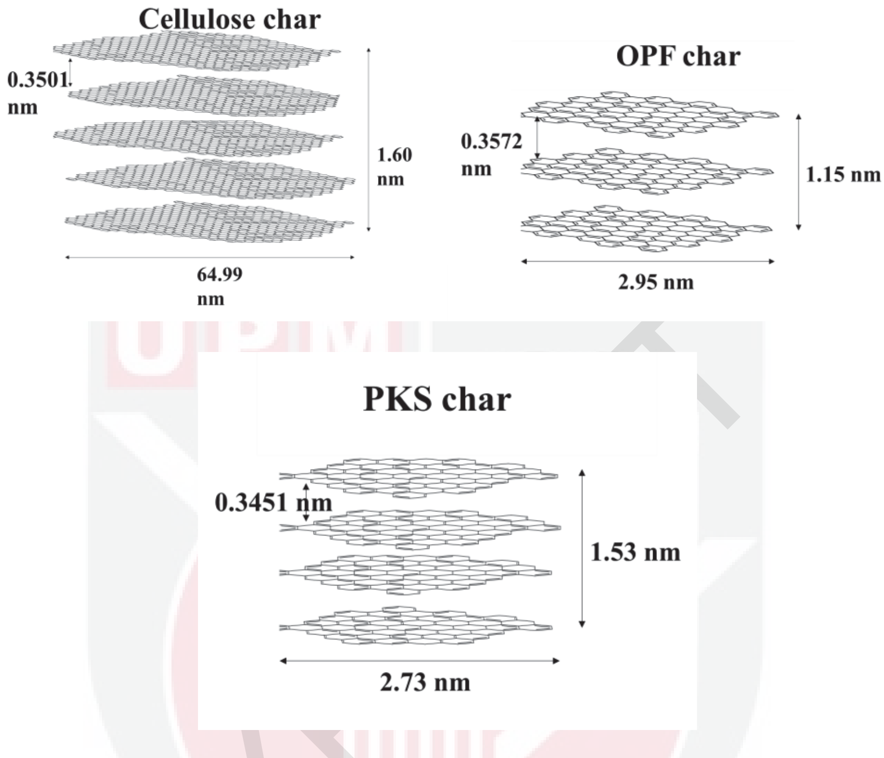
6) Hold for 3.0 min at 110.00°C

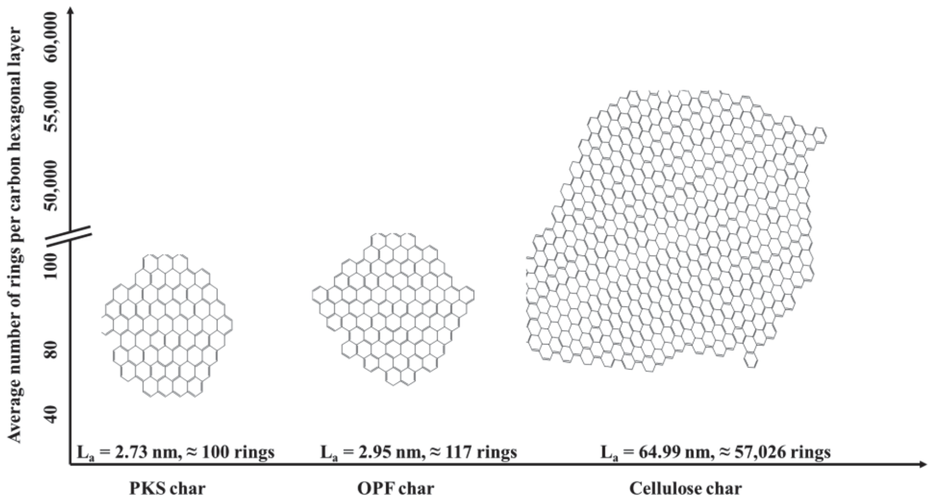
1) TGA Temperature Scan

Time	Unsubtracted Weight	Baseline Weight	Program Temperature	Sample Temperature	Approx. Gas Flow	R25 Diagnostic Temperature
0.000000	15.699768	0.000000	50.833333	50.240000	79.800000	0.000000
0.016667	15.697618	0.000000	50.833333	50.260000	79.800000	0.000000
0.033333	15.696328	0.000000	51.666667	50.280000	79.800000	0.000000
0.050000	15.690952	0.000000	52.500000	50.300000	79.800000	0.000000
0.066667	15.689662	0.000000	53.333333	50.320000	79.800000	0.000000
0.083333	15.686222	0.000000	54.166667	50.340000	79.800000	0.000000
0.100000	15.683857	0.000000	55.000000	50.360000	79.800000	0.000000
0.116667	15.681492	0.000000	55.833333	50.380000	79.800000	0.000000
0.133333	15.678697	0.000000	56.666667	50.400000	79.800000	0.000000
0.150000	15.675901	0.000000	57.500000	50.420000	79.800000	0.000000

Appendix E

Structure of CC-BC, OPF-BC and PKS-BC





BIODATA OF STUDENT

Abubakar Abdullahi Lawal was born in Gwoza, Borno State of Nigeria on 23rd June 1979. He attended University of Maiduguri in Borno state, Nigeria and graduated with Bachelor's degree in Agricultural Engineering in 2006. He later joined University of Maiduguri for his master's degree in Food Process and Storage Engineering and graduated in July 2013. He joined the Faculty of Engineering, Universiti Putra Malaysia in February 2017 to pursue a Doctor of Philosophy (PhD) in Agricultural Waste Engineering. His research focused on Biochar production and application from oil palm biomass materials. He is presently a lecturer in the Department of Agricultural and Environmental Resources Engineering, University of Maiduguri, Borno state, Nigeria. He is fully registered as member of the Council for the Regulation of Engineering in Nigeria (COREN) and Nigerian Society of Engineers (NSE).

LIST OF PUBLICATIONS

Lawal, A.A., Hassan, M.A., Farid, M.A.A., Yasim-Anuar, T.A.T., Yusoff, M.Z.M., Zakaria, M.R., Roslan, A.M., Mokhtar, M.N., Shirai, Y., 2020. Production of biochar from oil palm frond by steam pyrolysis for removal of residual contaminants in palm oil mill effluent final discharge. *Journal of Cleaner Production*, 265, 121643. 10.1016/j.jclepro.2020.121643

Lawal, A.A., Hassan, M.A., Ahmad Farid, M.A., Yasim-Anuar, T.A.T., Mohd Yusoff, M.Z., Zakaria, M.R., Roslan, A.M., Mokhtar, M.N., Shirai, Y., 2020. One-step steam pyrolysis for the production of mesoporous biochar from oil palm frond to effectively remove phenol in facultatively treated palm oil mill effluent. *Environmental Technology Innovation*, 18. 10.1016/j.eti.2020.100730

Lawal, A.A., Hassan, M.A., Ahmad Farid, M.A., Tengku Yasim-Anuar, T.A., Samsudin, M.H., Mohd Yusoff, M.Z., Zakaria, M.R., Mokhtar, M.N., Shirai, Y., 2021. Adsorption mechanism and effectiveness of phenol and tannic acid removal by biochar produced from oil palm frond using steam pyrolysis. *Environmental Pollution* 269, 116197.

Lawal, A.A., Hassan, M.A., Farid, M.A.A., Yusoff, M.Z.M., Zakaria, M.R., Mokhtar, M.N., Shirai, Y., 2021. Effect of oil palm biomass cellulosic content on nanopore structure and adsorption capacity of biochar. *Bioresource Technology*. 332, 125070. 10.1016/j.biortech.2021.125070