THE INFLUENCE OF PRODUCTION TEMPERATURE ON PHYSICOCHEMICAL PROPERTIES OF BIOCHARS

CHINEDUM CHUKWUJEKWU ANYIKA

UNIVERSITI TEKNOLOGI MALAYSIA

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CHINEDUM CHUKWUJEKWU ANYIKA

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To my Family.

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ABSTRACT

In the past, polycyclic aromatic hydrocarbons (PAHs) remediation strategies in soils relying on the use of biochar studied sorption or biodegradation of PAHs separately. However, those studies did not acknowledge that sorption and biodegradation of PAHs in soils can occur simultaneously. As a result, biochar productions were conducted using different temperatures and pyrolysis mechanisms, which resulted into different physicochemical properties. In particular, previous studies were not able to sufficiently resolve the scientific mechanisms behind the use of biochar for sorption or stimulation of biodegradation of PAHs by microbes. For example, they relied on production temperatures of 700-800^{o}C, at which organic nitrogen needed for microbial growth and metabolism volatilizes. Similarly, they relied on fast pyrolysis, which yields biochar that have no soil carbon storage value and low aromaticity, necessary for the stronger binding of PAHs. Consequently, biochar production, characterization and application for PAH remediation have been conducted separately and continuously using different production temperatures and pyrolysis mechanisms, without identifying a unique production temperature or pyrolysis mechanism at which these two processes occur simultaneously based on the physicochemical properties of the resulting biochar. The objective of this work was to gain knowledge of the characteristics of biochars at high (650^{o}C) and low (450^{o}C and 350^{o}C) production temperatures, out of which a unique production temperature for the production of biochar for its impact on large-scale petrogenic PAH remediation was identified. The purpose was to contribute to the use of the resulting biochar for PAH remediation in soils. This was achieved by focusing on two topics of concern. Firstly, the characteristics of biochar of slow pyrolysis relevant to petrogenic PAHs remediation in soils were studied. Secondly, the toxicants present in biochar that may hinder microbial activity and lead to soil quality deterioration were quantified and certified. Towards these ends, a novel hypothesis on how biochar production temperature can impact on PAH fate processes in soils simultaneously was formulated. Biochar properties were evaluated by physicochemical, structural and stability characterizations. Characterization of the sample biochar produced at 650^{o}C displayed a greater surface area of 245 m^{2}/g, had a greater organic carbon content of 83%, with greater aromaticity and the most stable with 12% of labile carbon. Differentiation between the carbon storage values and its novel mechanism was achieved, which was in a descending order of sBC+$_{100}$=587 g kg^{-1}, sBC+$_{100}$=532 g kg^{-1} and sBC+$_{100}$=407 g kg^{-1}, for 650 > 450 > 350 to qualify for C_{org} (organic carbon) storage classes 4, 4 and 3, respectively. This mechanism is the online IBI classification-CPMAS ^{13}C NMR Spectra-van Krevelen diagram model. Toxicant assessment/enrichment behaviour of heavy metals in biochar was investigated. The results indicated that the lowest metal concentration of 15709 µg/Kg was at the CS650, indicating minimum enrichment. The distribution and stability of heavy metals in biochar was determined. The results indicated a minimum distribution of 620 mg/kg at 650^{o}C, implying greater stability. In conclusion, the study found that the 650^{o}C biochar resulted in better characteristics for the rapid sorption of PAH in soils due to its greater proportion of the nanopores and aromaticity, both of which are responsible for stronger binding. Simultaneously, the greater aromaticity will result in greater biodegradation since the microbes will concentrate on the PAHs alone due to the absence of appreciable labile C substrate, thereby implying that biodegradation proceed at a faster rate.

ABSTRAK

Pada masa lalu, strategi pemulihan hidrokarbon aromatik polisiklik (PAH) dalam tanah adalah bergantung kepada penggunaan bio-arang yang dikaji melalui penyerapan atau biodegradasi PAHs secara berasingan. Walau bagaimanapun kajian tersebut tidak memperakui bahawa penyerapan dan biodegradasi PAH dalam tanah boleh berlaku serentak. Hasilnya, penghasilan bio-arang telah dijalankan menggunakan suhu yang berbeza dan mekanisme pirolisis, yang menghasilkan sifat fizikokimia yang berbeza. Kajian terdahulu tidak mampu menyelesaikan mekanisme saintifik di sebalik penggunaan bio-arang untuk penyerapan atau stimulasi biodegradasi PAH oleh mikrob. Sebagai contoh, mereka bergantung pada suhu penghasilan 700-800^{o}C, yang mana nitrogen organik yang diperlukan untuk pertumbuhan dan metabolisme mikrob telah meruap. Begitu juga, mereka bergantung kepada pirolisis pantas, yang menghasilkan bio-arang tanpa nilai penyimpanan karbon organik bagi tanah dan nilai struktur aromatik yang perlu bagi ikatan PAH yang lebih kuat. Akibatnya, penghasilan, pencirian, dan aplikasi bio-arang untuk pemulihan PAH telah dijalankan secara berasingan dan secara berterusan menggunakan suhu penghasilan dan mekanisme pirolisis yang berbeza, tanpa mengenal pasti suhu penghasilan unik atau mekanisme pirolisis yang mana kedua-dua proses boleh berlaku secara serentak berdasarkan sifat-sifat fizikokimia bio-arang yang dihasilkan. Objektif kajian ini adalah untuk mendapatkan pengetahuan tentang ciri-ciri bio-arang pada suhu penghasilan yang tinggi (650^{o}C) dan rendah (450^{o}C dan 350^{o}C), yang mana satu suhu penghasilan yang unik untuk penghasilan bio-arang telah dapat dikenalpasti untuk memberi kesan yang besar terhadap pemulihan petrogenik PAH. Tujuannya adalah untuk menyumbang kepada penggunaan bio-arang yang dihasilkan untuk pemulihan PAH dalam tanah. Ini telah dicapai dengan memberi tumpuan kepada dua topik utama. Pertama, ciri-ciri yang berkaitan dengan bio-arang daripada pirolisis lambat relevan dengan pemulihan PAH petrogenik dalam tanah yang telah dikaji. Kedua, bahan toksik di dalam bio-arang yang boleh menghalang aktiviti mikrob dan membawa kepada kemerosotan kualiti tanah telah diukur dan disahkan. Akhirnya, hipotesis novel tentang bagaimana suhu pengeluaran bio-arang boleh memberi kesan terhadap proses PAH dalam tanah secara serentak telah diformulasi. Sifat-sifat bio-arang telah dinilai oleh pencirian fizikokimia, struktur dan kestabilan. Pencirian sampel bio-arang yang dihasilkan pada 650^{o}C menunjukkan luas permukaan yang lebih besar laitu 245 m^2/g, mempunyai kandungan karbon organik yang lebih tinggi laitu 83%, tahap aromatik yang lebih tinggi dan paling stabil dengan 12% karbon labil. Perbezaan antara nilai penyimpanan karbon dan mekanisme novel telah dicapai, yang mana dalam susunan menurun sBC + $_{100}$ = 587 g kg^{-1}, sBC + 100 = 532 g kg-1 dan sBC + $_{100}$ = 407 g kg^{-1}, untuk 650> 450> 350 untuk layak bagi kelas penyimpanan C_{org} masing-masing 4, 4 dan 3. Mekanisme ini merupakan model diagram klasifikasi-CPMAS IBI ^{13}C NMR Spectra-van Krevelen atas talian. Penilaian bahan toksik/pengkayaan ciri logam berat dalam bio-arang telah dikaji. Hasilnya menunjukkan bahawa kepekatan logam berat yang paling rendah ialah 15709 µg/Kg pada 650^{o}C, merupakan pengkayaan minimum. Pengagihan dan kestabilan logam berat dalam bio-arang dapat ditentukan. Hasilnya menunjukkan pengagihan minimum sebanyak 620 mg/Kg pada 650^{o}C, merujuk kepada kestabilan yang tinggi. Kesimpulannya, kajian ini menemukan bio-arang pada 650^{o}C menghasilkan ciri yang lebih baik bagi penyerapan pantas PAH di dalam tanah berdasarkan bahagian liang-liang nano dan aromatik yang besar, kedua-duanya bertanggungjawab bagi ikatan yang lebih kuat. Secara serentaknya, tahap aromatik yang lebih besar, menghasilkan keupayaan biodegradasi yang lebih tinggi, kerana mikrob akan bertumpu ke atas PAHs sahaja, oleh sebab ketidakhadiran substrat, C labil yang ketara, dengan demikian bermaksud biodegradasi boleh berlaku pada kadar yang lebih cepat.

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LIST OF ABBREVIATIONS

AC	-	Activated Carbon
ADS-CSA	-	Adsorption-Cumulative Surface Area
AEC	-	Anion Exchange Capacity
ARISA	-	Automated ribosomal intergenic spacer analysis
ASTM	-	American Society for Testing and Materials
ATR	-	Attenuated Total Reflectance Spectroscopy
BCM	-	Biocarbon Mineral
BET	-	Brunauer Emmet Teller
BJH	-	Barret Joyner Halenda
BTEX	-	Benzene toluene ethylbenzene xylene
C	-	Constant
CA	-	Cluster Analysis
CCME	-	Canadian Council of Ministers of Environment
CD	-	Contamination Degree
CDFC	-	Canonical Discriminant Function Coefficient
CEC	-	Cation Exchange Capacity
CF		Contamination Factor
C/N Ratio	-	Carbon/Nitrogen Ratio
CNS	-	Carbon Nitrogen Sulphur
Corg	-	Organic Carbon
CRM	-	Certified Reference Material
CSB	-	Coconut Shell Biochar
CS	-	Coconut Shell
CVAAS	-	Cold Vapour Atomic Absorption Spectroscopy
CPMAS	-	Cross Polarization Magic Angle Spinning
DA	-	Discriminant Analysis

Df	-	Degree of Freedom
DF	-	Discriminant Function
DFT	-	Density Functional Theory
DLD	-	Delay Line Detector
DNA	-	Deoxyribonucleic Acid
DSC	-	Differential Scanning Calorimeter
DTG	-	Derivative Thermo Gravimetric
EC	-	Electrical Conductivity
EDXRF	-	Energy Dispersive X-ray Fluorescence Spectroscopy
EF	-	Enrichment Factor
EP	-	Extractable Phosphorus
EPA	-	Environmental Protection agency
ESA	-	External Surface Area
F	-	Function
FESEM	-	Field Emission Scanning Electron Microscopy
FL	-	Feed Lot
FTIR	-	Fourier Transform Infra-Red
GBC	-	Granular Biochar
GCMS	-	Gas Chromatography Mass Spectrometer
HACA	-	Hierarchical Agglomerative Cluster
HCA	-	Hierarchical Cluster Analysis
HBP	-	High Boiling Point
HPLC	-	High-Performance Liquid Chromatography
HMW	-	High Molecular Weight
HPT	-	High Production Temperature
HRPP	-	High Range Powdered Pillow
IBI	-	International Biochar Initiative
IC	-	Inorganic Carbon
ICP-MS	-	Inductively Coupled Plasma Mass Spectroscopy
ICP-OES	-	Inductively Coupled Plasma Optical Emission Spectroscopy
IUPAC	-	International union of pure and applied chemistry
Kv	-	Kilo Volts
KMO	-	Kaiser Meyer Olkin

MA	-	Mercury Analyser
MDA	-	Multiple Discriminant Analysis
MPA	-	Micropore Area
MPV	-	Micropore Volume
n	-	Number of Replicates
Nd	-	Not Determined
NIC	-	Nippon Instruments Corporation
NMR	-	Nuclear Magnetic Resonance
NTE	-	Non Toxic Elements
OC	-	Organic Carbon
PACS2	-	Protective Action criteria for Chemicals
PAHs	-	Polycyclic Aromatic Hydrocarbons
PBC	-	Powdered biochar
PCA	-	Principal Component Analysis
PCR	-	Polymerase Chain Reaction
PHs	-	Petroleum Hydrocarbons
PL	-	Poultry Litter
PLFA	-	Phospholipid Fatty Acid
PM	-	Poultry Manure
ppb	-	Parts Per Billion
ppm	-	Parts Per Million
PT	-	Production Temperature
qPCR	-	Quantitative Polymerase Chain Reaction
NTE	-	Nontoxic Elements
R_{CSB}	-	Biochar Recovery
R_C	-	Carbon Recovery
RIM	-	Resident Inorganic Matter
ROM	-	Resident Organic Matter
rRNA	-	Ribosomal Ribonucleic Acid
RT	-	Room Temperature
S	-	Step
SA	-	Surface Area
SD	-	Standard Deviation

SE	-	Standard Error
SOC	-	Soil Organic Carbon
SSA	-	Specific Surface Area
SS	-	Swine Solid
SPA	-	Single Point Adsorption
SPSA	-	Single Point Surface Area
SPSS	-	Statistical Package for Social Science
SW	-	Solid Waste
SW	-	Shapiro-Wilks
TA	-	Thermal Analysis
TE	-	Toxic Elements
TG	-	Thermo Gravimetric
TGA	-	Thermo Gravimetric Analysis
TC	-	Total Carbon
TOC	-	Total Organic Carbon
TL	-	Turkey Litter
TM	-	Transmission Electron Microscope
TN	-	Total Nitrogen
TPH	-	Total Petroleum Hydrocarbons
TPAH	-	Total Polycyclic Aromatic Hydrocarbons
TRFLP	-	Terminal Restriction Fragment Length Polymorphism
UP	-	Ultra-Pure
USDA	-	United States Department of Agriculture
USEPA	-	United States Environmental Protection Agency
VF	-	Varimax Factor
VOC	-	Volatile Organic Carbon
VP	-	Vapour Pressure
VP	-	Variable Pressure
VPH	-	Volatile Petroleum Hydrocarbons
Vs	-	Versus
W_{cs}	-	Weight of Un Charred Coconut Shell
W_{CSB}	-	Weight of Charred Coconut Shell Biochar
WHC	-	Water Holding Capacity

XPS	-	X-ray photo Electron Spectroscopy
ZP	-	Zeta Potential

LIST OF SYMBOLS

$\Delta H_{(hyd)}$	-	Enthalpy of Hydration
d_{10}	-	Deuterated
D10	-	10% of sample mass have diameter < 10 µm
D50	-	50% of sample mass have diameter < 50 µm
D90		90% of sample mass have diameter < 90 µm
µ	-	Micron
MHz	-	Mega Hertz
$SA_{\mu P}$	-	Micropore surface area
$V_{\mu P}$	-	Micropore volume
Π	-	Pi
sBC_{+100}	-	Stock Biochar $C_{+100\ years}$
Σ	-	Summation
Hz	-	Unit of frequency
Λ	-	Wilks' Lambda

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CHAPTER 1

INTRODUCTION

1.1 Research Background

The exploration and exploitation of oil resources, due to the continuous demand for energy, have led to an increase in soil contamination (Leung *et al.*, 2007). Additionally, spills and leaks of Petroleum Hydrocarbons originating from storage tanks and delivery facilities result in soil pollution and this may percolate into ground water (Urum *et al.*, 2006). Petroleum hydrocarbons such as the polycyclic aromatic hydrocarbons (PAHs) are known to be inert but they can be activated by physical and chemical reactions to become toxic; resulting in DNA damage, mutations, cancer and tumours (Fu *et al.*, 2012, Harvey, 1991). Wilson and Jones (1993) had reported that PAHs are teratogenic, therefore, they can hinder the development of the foetus, resulting in deformities in newly born children. As a result, their presence in soils is handled using compound specific approach or risk evaluation (Edwards *et al.*, 1997).

The soil is the major medium of uptake of PAH by plants and animals that feed on the plants (Phillips, 1999, Diggs *et al.*, 2011), both of which are in turn consumed by humans. For example, the authors reported that benzo[a]pyrene can be found in concentrations of up to 2-500 ng/day in human diets. PAHs are usually the first contaminants of investigation following a spill because their presence in soils regardless of concentration will warrant remediation due to their toxicity Edwards *et al.* (1997) and the risk posed by PAH is mostly due to the dissolved concentrations (Oleszczuk *et al.*, 2012).

The use of either activated carbon (AC) or biochar reduces the transport of PAH in soils (Oleszczuk *et al.*, 2012). In particular, Oleszczuk *et al.* (2012) found that amendment with 5% AC was able to bind 90% of PAHs in sewage sludge, but biochar resulted into a longer time period to bind the PAHs. Nonetheless, the use of AC has been reported to have a negative impact on the environment over time Sparrevik *et al.* (2011b) and AC may not be able to impact beyond sorption of PAHs, unlike biochar that can give simultaneuous impact on sorption and biodegradation. Additionally, biochar may be more beneficial to the environment and cheaper (Oleszczuk *et al.*, 2012, Cornelissen *et al.*, 2005b) than AC for soil remediation (Sparrevik *et al.*, 2011b). Also, the use of manures and composts may lead to pollution of streams and groundwater (Barrow, 2012). Pathogens, heavy metals and pharmaceutical products may reside in manures and composts, in addition to the evolution of ammonia and methane which increases the effect of greenhouse gases (Barrow, 2012).

Beesley *et al.* (2011b), defined biochars as biological residues, pyrolyzed under anoxic conditions, to yield porous, less compact, carbon-rich material. Due to their large surface areas and cation exchange capacities, they can sorb organic compounds as much as 10-1000 times greater than organic matter, thereby decreasing their transport in contaminated soils (Cornelissen *et al.*, 2005b). Biochar may also serve to get rid of wastes such as agricultural, industrial, sewage, refuse, livestock and manure (Barrow, 2012).

In this study, biochar is defined as biological materials produced in the absence of oxygen, at temperatures below 700^{o}C, Lehannes and Joseph (2009), to generate, permeable, less dense, carbon-rich product. As a result, of their surface areas, aromatic and aliphatic structures; which are mostly a function of production temperature, they can retain PAHs 10-1000 times greater than organic matter. Also due to their recalcitrance, pore structures and nutrient properties, they can influence microbial degradation of PAHs in soils. As a result, there is a need to understand how biochar production temperature influences hydrocarbon fate in soils.

Further to the above, the rapid release of oil at accidental spills can cause environmental damage if not immediately contained. Conventional methods such as natural dispersion or oil spill chemicals are usually, used, to reduce the impact of oil contamination in seawater. However, the action results in some of the oil been deposited on the soil by the dispersion process. Further, in developing countries, these oil-polluted soils are close to farmlands, surface water sources and have the potential to leach into groundwater. For example, in a preliminary survey by the author in the oil rich Niger delta area of Nigeria, it was clear that this could pose a threat to local farmers and inhabitants who rely on these soils for agricultural production and nearby surface and ground water source as a means of livelihood.

As a result, normal bioremediation strategies such as the use of fertilizers and commercial bioremediation agents are not appropriate for these kinds of areas. Moreover, for effective bioremediation by bacteria, the PAH contaminants should not be present at toxic concentrations to the microbes (Leung *et al.*, 2007). An opposing strategy, involving amendment with biochar may be a better strategy for bioremediation by bacteria. The amendment with biochar will lead to a rapid sorption of spilt oil. Subsequently, the oil will form bound residues within the biochar, leading to a rapid risk reduction against uptake of PAHs into crops and livestock. After the rapid sorption of PAHs, a subsequent desorption process will lead to a long-term bioremediation process.

1.2 Problem statement

Studies by Hamer *et al.* (2004) found that addition of biochar may support rather than inhibit microbial degradation of organic compounds in soils. Previous work with biochar to study PAHs fate processes have relied only on information on biochar interaction with microbes without any reference to the influence of production temperature, which can give simultaneous impact on sorption and biodegradation. In order words these studies either relied on one or several different production temperatures without acknowledging the mechanisms behind the use of biochar at those production temperatures. For example, in the paper by Bushnaf *et al.*

(2011b) using fast pyrolysis, they investigated the influence of biochar application rate i.e. 2% biochar addition in soils to stimulate biodegradation of PAHs and compared with those of non-amended soils. The outcome was that the degradation of the low molecular weight alkanes influenced the degradation of the PAHs; no differences were found between 2% biochar additions and the non-amended soil control. In the paper by Qin *et al.* (2013b), a slow pyrolysis biochar at 500^{o}C, was applied after 80 days of soil contamination, to test the effect of time of application on PAHs degradation. The outcome was that the PAHs degraded faster from the 80th day than those applied at day zero, due to reduction in toxic components of the oil i.e. PAHs at day 80. In the paper by Chen *et al.* (2012a) the influence of a mixture of biochar and plant residues as carriers to stimulate biodegradation of PAHs by microbes was investigated at 100, 300, 400 and 700^{o}C, respectively using slow pyrolysis biochar. The outcome was that the 400^{o}C biochar-plant residue carrier mixture stimulated the degradation of the PAHs. In another set up, they also used the same carrier to study sorption-biodegradation of PAHs with and or without out biochar. The outcome was that the biochar carrier stimulated the degradation of more of the PAHs relative to the plant residue-bacteria carrier.

In order to understand the contribution of the current thesis, the demerits of the studies mentioned above are highlighted. In the paper by Bushnaf *et al.* (2011b), no reason was given for adopting the 2% biochar application rate, i.e. no mechanism was pointed out on how biochar of 800^{o}C at the rate of 2% can stimulate the degradation of PAHs. In the past, application rates were mainly for fertility trials using inorganic fertilizers to determine the rate at which the plant will grow faster. In PAH remediation, nutrients are needed in a stoichiometric ratio of C:N: P; 100:10:1 for microbial growth and metabolism but the study was not referring to nutrient nitrogen or this ratio since only one production temperature (800^{o}C) and one application rate (2%) were used hence no hypothesis was tested. Unfortunately, at 700- 800^{o}C, nutrient nitrogen is lost by volatilization, therefore, there was no scientific idea or bases behind the use of the 800^{o}C biochar for degradation when nutrient nitrogen needed for microbial growth and metabolism is absent. In the paper by Qin *et al.* (2013b), the reason for adopting the time point of 80 days for application was to reduce PAHs toxicity, before adding the biochar. With respect to time, just like 2% application rate, time is not scientific; also no hypothesis can be

tested at one time point. Again the advantage of adding biochar for rapid sorption like in the case of an accidental spill has been defeated if an investigator has to wait for 80 days before applying the biochar to achieve results. In the paper by (Chen *et al.*, 2012a), although several production temperatures were used and hence hypothesis tested, the fact that they used biochar-plant residue carrier mixture for sorption-biodegradation, implies that biochar cannot independently provide habitat and nutrients for microbes except in the presence of a plant residue part-carrier. In order words biochar cannot increase microbial presence except when mixed with plant residue as a substrate. Again, they used 700^{o}C production temperatures at which nutrient nitrogen is volatilized. Similarly, at 400^{o}C and below, aromaticity is not yet, attained since carbonization, which is responsible for aromaticity and nanopore formation that results in the stronger binding of PAHs are achieved only at high production temperatures. In conclusion, the studies mentioned above did not carry out any advanced characterization to unlock these characteristics.

Conversely, the studies mentioned above cannot adequately explain the scientific mechanisms that may underpin the use of biochar for soil amendment to stimulate biodegradation of PAHs in soils. This is because those studies are not different from normal biostimulation studies where manures and fertilizers have, been, used to enrich microbial presences in soils. In this study, to understand the scientific mechanisms behind the use of biochar, which, will in turn, unlock the mechanisms on how biochar can give simultaneous impact on sorption and biodegradation. The idea of Thies and Rillig (2009a) on the behaviour of biochar of fast pyrolysis at different production temperatures was relied upon and extrapolated to PAH using biochar of slow pyrolysis. It is worth noting that Thies and Rillig (2009a) made no mention of PAHs while stating this behaviour of biochar but they were rather referring to the general behaviour of biochar between production temperatures and how this can influence their activities in soil towards microbes. Again, no mention was made of sorption either. In this study, this idea was now extrapolated to PAHs by way of advancing or moving the knowledge forward by building on the idea of Thies and Rillig (2009a). Since the studies mentioned above have continued to use biochar as fertilizers for normal biostimulation or bioaugmentation, which was the common knowledge in the past.

Consequently, the probable influence of biochar production temperature has been highlighted by Thies and Rillig (2009a), who reported that brief carbonization and low production temperature (LPT) generates residual bio-oils and other residues on biochar surfaces. It has also been reported that these materials may be made up of substrates that can aid microbial growth and metabolism (Ogawa, 1994). Further, these bio-oils, which are mostly aliphatic, can be biodegraded in a short time when incorporated into the soil. In contrast, it was found that high production temperature (HPT) biochars have greater aromatic structures and were abiotically degraded at a faster rate than biochars produced at LPTs with mainly aliphatic structures (Lehmann *et al.*, 2009a).

In this study, it was therefore, hypothesized that using slow pyrolysis, which is not a brief carbonization, soil amendment with HPT biochar is expected to stimulate biodegradation and oxidation of PAH, contaminated, soils at a faster rate compared to amendment with LPT biochar. This is because at HPT, it is assumed that the recalcitrant aromatic structures and lack of bio-oils will result in the petrogenic PAHs alone to be the major substrate for the microbes to act on, while at LPT, the microbes will act on both the petrogenic PAH and degradable aliphatic compounds (bio-oils), thereby decreasing the rate of biodegradation at LPT. To date, no attempt has been made to investigate the influence that biochar production temperature may have to stimulate the biodegradation of PAHs in soils by way of the stated mechanism. Again, due to the presence of the aromatic structures, biochar can bind PAHs and simultaneously, the aromatic recalcitrance will result in the PAHs been the only available substrate for the microbes to act on thereby the biodegradation will proceed at a faster rate.

In this study, we investigated the effect of production temperature on the physicochemical characteristics of biochars that can influence the chemical and biological degradation of PAHs in soils simultaneously and no study has investigated the physicochemical characteristics of biochar and linked it to providing a solution to sorption and biodegradation simultaneously. This was also pointed out by Saikia *et al.* (2015) who reported that previous studies focused on generic characterization of

biochars most of which were not directed towards a solution to environmental problems.

1.3 Research Objectives

The objective of this research is to investigate the influence of biochar production temperature on the physicochemical properties of biochar that can result in a rapid chemical and biological treatment for hydrocarbon clean up with long-term impact to achieve permissible soil quality limits. In this way, an effective strategy is developed that ensures cleanup levels able to maintain soil reuse for farming activities and clean water quality in the vicinity of a spill. Therefore, the following are the objectives of this research:

i. To determine the effect of production temperature on the physicochemical properties of coconut shell biochar.
ii. To determine the effect of production temperature on the quality assessment of coconut shell biochar based on its advanced chemical properties.
iii. To determine the effect of production temperature on the surface characterization of coconut shell biochar based on its advanced physical properties.

This study addresses the following research questions: What is the effect of production temperature on the physicochemical parameters of biochar that can influence PAHs sorption and biodegradation simultaneously? What is the effect of production temperature on the concentration of toxic and nontoxic elements in biochar? What is the effect of production temperature on the heavy metal concentrations in biochar? What is the effect of production temperature on the surface characterization of biochar?

1.4 Scope

The project addresses the following issues: (1) Biochar characteristics at 2 levels; high (650^{o}C) and low (450 and 350^{o}C) production temperatures for relevant characteristics to enhance rapid sorption and simultaneously subsequent biodegradation of PAHs in soil. (2) Toxicant assessment for biochar quality to be used as a soil application (3) Surface characterization of biochar for its surface area and pore structures for rapid sorption of PAHs.

1.5 Research significance

Previous fate studies/processes using biochar, had always, been carried out separately. For example, most sorption studies had always made provision to inhibit biodegradation during sorption; thus implying that sorption and biodegradation occur simultaneously. Similarly, biodegradation had always, been studied separately but little or no attempt had been made to both study biodegradation and sorption simultaneously. This has led to the critical problem that no study has linked biochar characterization to its ability to give simultaneous impact on sorption and biodegradation. As a result, this has resulted in the independent characterization of biochars separately for sorption and most recently for biodegradation studies. Again, most of the characterizations for sorption were based on coconut shell AC rather than coconut shell biochar, due to lack of knowledge of the fact that biochars can give simultaneous impact on sorption and biodegradation.

Secondly, the bioremediation processes are relatively slow and may not be able to compete with the rapid release of petrogenic PAHs into the soil at accidental spills. Rapid sorption of such PAHs and subsequent slow bioremediation might be a good concept to overcome the problem of accidental oil spills. As a result, the relevant physicochemical characteristics of biochar that will ensure rapid sorption and biodegradation simultaneously needs to be better understood. In this study, a triphasic concept is proposed on how biochar can influence rapid PAH sorption, also, reduce volatilization and subsequent biodegradation by bacteria in soils: (i) The

biochar will sorb the spilt PAH thereby reducing transport and volatilization (risks) (ii) The biochar will positively modify the soil environment; pH, nutrients, moisture, oxygen, microbial community and habitation. (iii) The sorbed PAH will be desorbed over time and subsequently degraded by microbes to release CO_2 and H_2O, leading to long-term controlled bioremediation.

Thirdly, most studies using biochar for sorption and biodegradation had always ignored the toxicant assessment thereby disregarding the risks that may be posed by the application of the biochar to the soil. The matter, is, made, worst by the fact that such assessments are only limited in scope by comparison of the outcomes with established quality assessment guidelines. Whereas, ignoring the processes and mechanisms, responsible for the quality.

Finally, the optimum production temperature for biochar to ensure rapid sorption and stimulation of bioremediation by bacteria simultaneously will be classified.

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