UNIVERSITI PUTRA MALAYSIA

CHARACTERIZATION AND STABILITY OF SELECTED BIOCHARS AND EFFECT OF THEIR APPLICATION ON SOIL PHYSICAL PROPERTIES AND CORN GROWTH

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FP 2015 72

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By

HUDA ABULRAZZAQ MOHIALDEEN

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

April 2015

DEDICATION

This thesis is dedicated to:

The sake of Allah the Almighty, my Master and my Creator,
My beloved homeland Iraq, the warmest womb,
My dearest late Husband, may Allah have mercy on him,
My beloved kids, Ibrahim and Abdullatif, who make me happy and empowered me to live the life with purpose,
My family, who did not stop supporting me all the way,
My friends who never let me alone,
To all of warm people in my life who touched my deep heart,

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

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April 2015

Chairman: Hamdan Jol, PhD
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The intentional amendment of soil with biochar is offering a new strategy for improving soil fertility and sequestering atmospheric CO_2. Nonetheless, the characteristics of biochars vary with their different conditions and pyrolysis techniques to improve the physical properties and carbon sequestration of soil as to enhance plants growth. This research is to prove that biochar considers as a stable product that can be produced from feedstock pyrolysis by one of the available bioenergy production techniques. The study aimed to improve the understanding of how adding biochar applications and the pyrolysis of native feedstock to soil can be utilized to improve carbon stability, soil physical properties, and soil fertility in Malaysia. Adding biochar to soils has established a variety of advantages that vary according to the type of the feedstock as the types of the utilized pyrolysis conditions in biochar production. An emphasis was placed on understanding how both of biomass material and production conditions of the pyrolysis process can influence biochar characteristics, and what effects can result from adding different rate of the amendment biochar on soil and plant growth. Three kinds of primary biochar were used, namely, empty fruit bunch biochar (EFB), wood biochar (WB), and rice husk biochar (RHB). EFB and WB were produced by slow pyrolysis method, whereas RHB was produced by the gasification. Four sets of experiments were conducted. In the first set of experiments, the influences of slow pyrolysis and gasification on biochars were examined independently. The physicochemical structure of the produced biochars was characterized with various analytical techniques including FTIR, XRD, SEM, HPLC, and BET surface area analysis. In the second and third sets of experiments, a field study had been carried out to evaluate the effect of the biochar incorporation on soil in regards to the carbon stability and soil physical properties, the final set was carried out in a shade house to evaluate the effect of adding biochars treatment on plants' heights and dry shoot growth. The results revealed the presence of a very labile C-fraction in RHB with a very small decay constant K_3. Fourier transform infrared spectroscopy and X-ray diffraction showed three phases of the biochar, from the microcrystalline C of the labile fraction to the largely amorphous intermediate C of the unstable fraction, and finally the formation of turbostratic crystallite C in the recalcitrant fraction. Furthermore, biochar incorporation into soil has effect on soil water content and

hydraulic conductivity, contributes to improve soil structure, and improved moisture characteristics, regarding to the extensive pore structures, surface characteristics, and high porosity of RHB as compared to EFB and WB. EFB30 in pot trials resulted in a highly positive effect on sweet corn growth with 50% of fertilizers; On the other hand, RHB30 did not have positive impact on the growth. Due to its labile fraction might cause microbial immobilization of soil N.

It has been concluded that, RHB had a higher degree of aromaticity, greater stability, and therefore should be more recalcitrant to biological and chemical degradation. Applying biochar substances by using local materials is assessing promises of being an environmentally sound in enhancing the physical characteristics of soil and crop productivity in Malaysia.

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

PENCIRIAN, KESTABILAN DAN KESAN BIOARANG TERHADAP SIFAT FIZIKAL TANAH DAN PERTUMBUHAN JAGUNG

Oleh

HUDA ABULRAZZAQ MOHIALDEEN

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Pindaan yang disengajakan terhadap tanah menggunakan bioarang (*biochar*) menawarkan strategi baharu untuk meningkatkan kesuburan tanah dan pemencilan CO_2 dalam atmosfera.. Walau bagaimanapun, ciri-ciri bioarang berbeza mengikut keadaan dan teknik pirolisis untuk menambah baik sifat-sifat fizikal dan pemencilan karbon bagi tanah untuk merangsang pertumbuhan tanaman. Kajian ini dijalankan untuk membuktikan bahawa bioarang boleh dianggap sebagai produk yang stabil yang boleh dihasilkan daripada pirolisis bahan mentah melalui salah satu teknik penghasilan biotenaga sedia ada. Kajian ini bertujuan meningkatkan pemahaman mengenai kesan penambahan aplikasi-aplikasi bioarang dan pirolisis bahan mentah setempat kepada tanah boleh digunakan untuk meningkatkan kesuburan tanah, sifat fizikal tanah dan kestabilan karbon di Malaysia.Penambahan bioarang kepada tanah telah memantapkan pelbagai kelebihan yang berbeza mengikut jenis bahan mentah dan juga keadaan pirolisis yang digunakan dalam penghasilan bioarang. Penekanan diberikan ke atas pemahaman cara kedua-dua bahan biojisim dan keadaan penghasilan bagi proses pirolisis boleh mempengaruhi ciri-ciri bioarang, dan kesan-kesan yang diperoleh dengan mencampurkan bioarang yang diubah suai dengan kadar yang berbeza terhadap tanah dan pertumbuhan tanaman. Terdapat tiga jenis bioarang utama yang digunakan iaitu bioarang hampas buah (EFB), bioarang kayu (WB), dan bioarang sekam padi (RHB). EFB dan WB dihasilkan daripada kaedah pirolisis perlahan, manakala RHB terhasil melalui proses pengegasan. Empat set eksperimen telah dijalankan. Dalam eksperimen set pertama, pengaruh pirolisis perlahan dan pengegasan terhadap bioarang diperiksa secara berasingan. Struktur fizikokimia bioarang yang dihasilkan dicirikan dengan pelbagai teknik analisis termasuk analisis kawasan permukaan FTIR, XRD, SEM, HPLC, dan BET. Dalam eksperimen bagi set kedua dan ketiga, kajian lapangan dijalankan untuk menilai kesan penggabungan bioarang dengan tanah terhadap kestabilan karbon dan sifat-sifat fizikal tanah. Perawatan melalui tiga kadar bioarang yang berbeza 0 t ha^{-1}, 15 t ha^{-1} dan 30 t ha^{-1} secara berturutan mengikut reka bentuk Blok Lengkap Terawak. Bioarang ditanam pada 10 cm kedalaman tanah. Selepas 194 hari, pengukuran lapangan yang terdiri daripada empat replikasi bagi kekonduksian hidraulik dan pensampelan teras dilaksanakan untuk mengukur ketumpatan pukal, keliangan tanah, dan menganalisis ciri-ciri kelembapan. Set terakhir dilakukan di sebuah rumah lindung untuk menilai kesan perawatan bioarang campuran terhadap

ketinggian tanaman dan pertumbuhan pucuk kering melalui ujian reka bentuk yang telah diatur sebagai satu reka bentuk yang rawak sepenuhnya dengan empat replikasi. Aplikasi bahan bioarang melalui penggunaan bahan-bahan tempatan menjanjikan teknologi mesra alam bagi merangsang ciri-ciri fizikal tanah dan produktiviti tanaman di Malaysia.

ACKNOWLEDGEMENTS

IN THE NAME OF ALLAH, THE MOST GRACIOUS, THE MOST MERCIFUL.

All praise belongs to Allah, glorified is He and exalted. I thank Allah for giving me the strength and wisdom for successfully completing my thesis work.

This thesis could not be accomplished without the support and the guidance as the help of many people who I'd like to gratefully acknowledge here.

First, I am greatly honoured to express my highest thankfulness to my supervisor Dr. Hamdan Jol for their support and guidance as fruitful effort all the way in accomplishing this work. Thank you for making my life easier.

I also would like to thank all of my friends and family members for their support and encouragement to finalize my postgraduate study. This dissertation could not be accomplished without their love and warm feelings.

Thank you all.

I certify that a Thesis Examination Committee has met on 28th April 2015 to conduct the final examination of Huda Abd Alrazzaq Al-Gailanion her thesis entitled "Characterization and stability of selected biochars and effects of their application on soil physical properties and corn growth" in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U. (A) 106] 15 March 1998. The Committee recommends that the student be awarded the Doctor of Philosophy.

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This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfillment of the requirement for the degree of Master of Science. The members of the supervisory committee were as follows:

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LIST OF ABBREVIATIONS

ANOVA	Analysis Of Variance
ASTM	American Society for Testing and Materials
AWC	Available Water Content
BET	Brunauer-Emmer-Teller
C_1	Amount of labile pool
C_2	Amount of unstable pool
C_3	Amount of recalcitrance pool
C_t	C mineralized (mg C g^{-1})
CEC	Cation Exchange Capacity
CRD	Completely Random Design
EFB	Empty Fruit Bunch Biochar
EFB15	Empty Fruit Bunch Biochar, applied to the soil at a rate 15 t/ha
EFB30	Empty Fruit Bunch Biochar, applied to the soil at a rate 30 t/ha
FC	Field Capacity
FTIR	Fourier Transform Infrared Spectroscopy
GHG	Greenhouse Gases
HPLC	High Performance Liquid Chromatography
IBI	International Biochar Initiative
K_1	Decay rate constant of labile C_1pool
K_2	Decay rate constant of unstable C_2 pool
K_3	Decay rate constant of recalcitrance C_3 pool
K_s	Hydraulic conductivity
LCA	Life Cycle Assessment
MRT	Mean Residence Time
MWD	Mean Weight Diameter
OM	Organic Matter
ρb	Soil bulk density
PWP	Permanent wilting point
RCBD	Randomized Complete Block Design
RHB	Rice Husk Biochar
RHB15	Rice Husk Biochar, applied to the soil at rate 15 t/ha
RHB30	Rice Husk Biochar, applied to the soil at rate 30 t/ha
SEM	Scanning Electron Microscope
SOM	Soil Organic Matter
Syngas	Synthesis Gas
t	Time
$t_{1/2}$	Half-life of carbon
t/ha	Ton/ hectare
WB	Wood Biochar
WB15	Wood Biochar, applied to the soil at rate 15 t/ha
WB30	Wood Biochar, applied to the soil at rate 30 t/ha
WHC	Water holding capacity
XRD	X-Ray Diffraction

CHAPTER 1

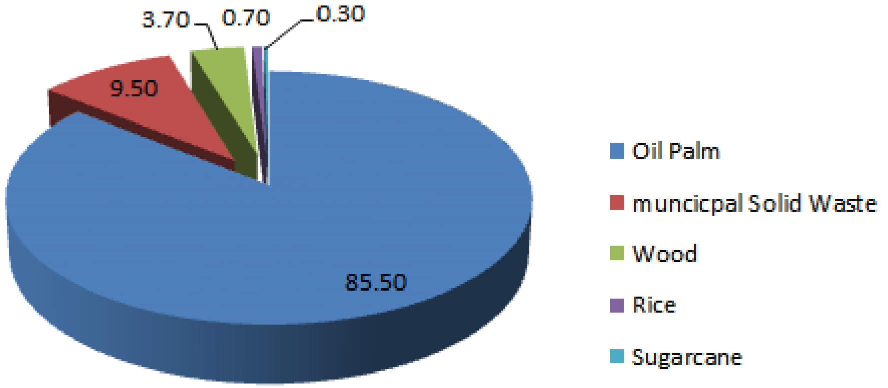
INTRODUCTION

1.1 Background

More than two million tons of agricultural wastes are produced in Malaysia annually. The presence of this waste and residues has generated several disposal problems to the country (Sopian et al., 2005), the main problems are: uncontrolled released of greenhouse gases/global warming, groundwater contamination from leachate, environmental pollution for example emission of smoke and haze hazard and emission of toxic chemicals such as dioxins (Hassan et al., 2005). Therefore, it is important to find beneficial way to utilize for these resources.

The main sources for agricultural wastes in Malaysia are generally produced from oil palm, paddy straw, sawn logs, and tropical fruits (Azlina et al., 2010). The process of milling palm to extract oil produces solid wastes of fibber, shell, and empty fruit bunches, where refining one ton of oil palm is producing nearly 0.15 tons of palm fiber, 0.07 tons of palm shell, and 0.2 tons of empty fruit bunches (Azlina et al.,2010). In contrast, paddy straw and rice husks can consequently produce wastes of 78% and 22% its weight that can be reused for generating biomass-based power (Umamaheswaran & Batra, 2008), Figure 1.1.

Figure 1.1 Biomass Residues Produced From Various Industries in Malaysia
(Source: Hassan & Shirai, 2003)



Thermo-chemical conversion of waste via pyrolysis demonstrates an excellent potential technique that produces biochar as being a co-product. That, when applied to soil, it probably enhances the soil structure, sequesters carbon, and absorbs toxins (Liu et al., 2012; Ahmad et al., 2014; Ennis et al., 2012).

The term 'biochar' is a solid material obtained from thermo-chemical conversion of biomass in an oxygen-limited environment that is used for the safe and long-term storage of carbon in the environment and for soil improvement. Additionally, it is a relatively recent development, emerging in conjunction with soil management and carbon sequestration issues (Lehmann et al., 2006). Another definition is referring 'biochar' term simply as the meaning of 'Charcoal application to soils' that could be enhanced its physicochemical features of soil (Brown, 2009).

Realizing the heterogeneity of Biochar' material can possess a range of chemical structures and elemental composition. This variability is based on the pyrolysis temperatures, heating rates, and biomass, which given this large variability is to be expected in the physicochemical properties of the biochars, and ultimately, in their performance as a soil amendment in the soil (McHenry, 2011).

The three main techniques employed for producing biochar from various biomass sources are pyrolysis, gasification and combustion. Pyrolysis is actually a thermo-chemical process where biomass is converted to a carbon- rich solid (biochar) and volatile matter (liquids and gases) by heating in the absence of oxygen (Maschio et al., 1992). Furthermore, traditional charcoal producing commonly utilizes slow pyrolysis conditions: slow heating rates (1-250 °C/min) in the absence of oxygen, and long residence times (hours to days), while gasification means incomplete combustion of biomass resulting in production of combustible gases consisting of Carbon monoxide, Hydrogen and traces of Methane (Bridgwater, 1995).

Malaysia produces large quantities of palm oil empty fruit bunches approximately 18 million tons annually. Universiti of Putra Malaysia worked together with Nasmech Technology Sdn Bhd to process empty fruit bunches biochar (EFB) through slow pyrolysis of biomass, with a capacity of 20 tons/day (Kong et al., 2014). The plant consisted of three pairs of ovens and a rotating drum. For the start-up, the oven heated by using a produced hot air by a diesel burner. When fully operational, the heat supplied through generated hot gas by a recycle gas burner. Whereas biochar from Malaysian mangrove wood, that is regarded as a widespread tree located in swamps especially at river mouths, is produced by kilning the logs over a lengthy eight to ten days at 220 °C followed by baking process for another two weeks at 83 °C, it is appeared as a strong and high density structure (Sivakumarana, 2010).

Rice husk and paddy straw tend also to be a significant potential for biomass-based power generation following wood and palm oil residues. (Hashim, 2005) recorded that the rice husk generated in year 2000 up to 471000 tonnes and also the potential power generated from the utilization of rice husk was approximately 72.07 MW during the gasification process. This trend is predicted while continue rising every year (Yusof et al., 2008).

The stability of biochar in soil is an important whether it is to be used for carbon sequestration in addition to long-term enhancement of soil characteristics. It is widely recognized that a substantial fraction of biochar is quite stable in soil;but CO_2 gas is released immediately after application (Bruun et al., 2014).

Although, biochars are produced in Malaysia; few research studies have been conducted on the decomposition of biochars in soil. Thus, an investigation on the biochar decay rate is important prior to the biochar application. This thesis correlates pyrolysis technologies and the resulting biochar characteristics to its stability. In addition, the carbon mineralization was assessed based on the characteristics of the biochar. The half-life was determined for the biochars produced by both pyrolysis and gasification.

This thesis results have potential consequences related to the concerns about global warming; as decreasing the emission of CO_2 gas from the soil might increase the value of the applied biochar, which in its turn, can contribute in producing more economically productive bioenergy. These facts are valuable in an attempt to substitute feedstock sources for soil amendments, reducing greenhouse gas emissions, in addition to consider it as a sustainable source to obtain energy.

1.2 Objectives

The overall objective of this thesis was to study the short-term effects of soil application of slow pyrolysis and gasification biochar on carbon mineralization. It also correlates the pyrolysis technologies and the described characteristics of biochar to its stability. The half-life and mean residences time were determined.

This main objective was attained by achieving the following objectives:

1) To study the characteristics of three kinds of produced biochar from the slow pyrolysis and gasification.
2) To measure the stability of biochars in soil, and to correlate between the defined stability and the biochar structure.
3) To determine the effect of adding biochars to soil on the physical properties of the treated soil.
4) To determine the effect of adding biochars to soil on growth of the sweet corn

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