EFFECT OF TEMPERATURE AND CONCENTRATION OF UREA SOLUTION IN UREA IMPREGNATION PROCESS INTO BIOCHAR

Submitted in accordance with the requirement of the University Teknikal Malaysia Melaka (UTeM) for the Bachelor Degree of Manufacturing Engineering (Engineering Materials) (Hons.)

by

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I hereby declare this report entitled "Effect of Temperature and Urea Concentration Solution in Urea Impregnation Process into Biochar" is the results of my own research except as cited in reference.

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APPROVAL

This report is submitted to the Faculty of Manufacturing Engineering of Universiti Teknikal Malaysia Melaka as a partial fulfilment of the requirements for the degree of Bachelor of Manufacturing Engineering (Engineering Materials) (Hons.).
The members of supervisory committee are as follows:

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(Principal Supervisor)

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(Co-Supervisor)

ABSTRAK

Biochar adalah produk akhir yang dihasilkan daripada pembakaran serbuk kayu getah dan telah dirawat bersama urea untuk digunakan sebagai baja dalam tanah. Kajian ini akan mengkaji kesan suhu dan kepekatan larutan urea semasa proses impregnasi urea. Pembakaran serbuk kayu getah dikawal dengan teliti menggunakan penganalisis gravimetrik termal (TGA) di bawah atmosfera nitrogen. Berdasarkan data daripada TGA, 8 mg serbuk kayu getah telah menjalani proses pirolisis pada suhu 350 ° C dan 600 ° C di bawah aliran gas nitrogen, pada kadar 30 ml / min dan kadar pemanasan 5 ° C / min. Pada suhu ini, pirolisis akan menghasilkan struktur liang yang berbeza dijalankan di bawah Mikroskop Pengimbasan Elektron (SEM). Larutan urea disediakan dengan mencampurkan urea bersama air pada dua kepekatan, 4% dan 8%. Biochar telah diregregasikan dengan larutan urea pada suhu bilik iaitu 30 ° C dan dipanaskan sehingga 50 ° C. Tahap kristal dan fasa bahan struktur selulosa dijalankan menggunakan Difraksi sinar-X (XRD). Kumpulan hidroksil fungsi permukaan yang diimpregnasikan dengan larutan urea telah dilakukan menggunakan Inframerah Spektroskopi Fourier Transform (FT-IR). Spektrum XRD menunjukkan bahawa pada puncak bahan kristalografi yang tinggi adalah pada suhu pirolisis yang rendah. Mikrograf SEM telah menunjukkan struktur liang menghilang pada suhu lebih tinggi 600 ° C. Oleh itu, biochar yang telah dipirolisiskan pada suhu 350 ° C sesuai untuk diimpregnasikan dengan urea pada kepekatan 4% dan 30 ° C.

ABSTRACT

Biochar is the final product produced from the combustion of sawdust and was impregnated with urea to be used in soil. This study will examine the effect of temperature and concentration of urea solution during urea impregnation process. The combustion of sawdust was closely monitored using thermal gravimetric analyse (TGA) under nitrogen atmospheres. Based on data from TGA, 8 mg of sawdust was undergo pyrolysis process at 350 ° C and 600 ° C under nitrogen gas flow at the rate of 30 ml / min and heating rate of 5 ° C / min. Pyrolysis at this temperature will create pore structure differently examined by Scanning Electron Microscope (SEM). Urea solution was prepared by mixing with water at two concentrations, 4% and 8%. Biochar was impregnated with urea solution at room temperature of 30 ° C and slightly heated up to 50 ° C. The degree of crystallinity and phase of the material were examined out using X-ray Diffraction (XRD). The surface functional hydroxyl group of biochar impregnated with urea solution was conducted using Fourier Transform Infrared-Spectroscopy (FT-IR). The XRD spectrum showed that at the peaks of high crystallinity material at lower pyrolysis temperature. The SEM micrographs have showed the pore structure disappeared at higher temperature of 600°C. Hence, the biochar that have been pyrolised at 350°C is suitable to be impregnated with urea at concentration of 4% and mixing temperature of 30°C.

DEDICATION

Only

My beloved father, Jamalludin Bin Abdul Hazis

My appreciated mother, Jaslina Binti Mohd Nor

My adored brother and sister, Aisam, Izzudin, Atikah and Nizam

For giving me moral support, money, co-operation, encouragement and also understandings

Thank You So Much & Love You All Forever

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LIST OF ABBREVIATIONS

AEC	-	Anion Exchange Capacity
CEC	-	Cation Exchange Capacity
HTT	-	High Treatment Temperature
FT-IRs	-	Fourier Transform Infrared– Spectroscopy
XRD	-	X-Ray Diffraction
SEM	-	Scanning Electron Microscope
EDS	-	Energy Dispersive X ray Spectroscopy
BC	-	Biochar
UBC	-	Urea Biochar
TGA	-	Thermal Gravity Analysis
T1	-	Temperature 1
T2	-	Temperature 2
C1	-	Concentration 1
C2	-	Concentration 2

LIST OF SYMBOLS

C	-	Carbon
°C	-	Degree Celsius
nM	-	Nanometre
N	-	Nitrogen
H	-	Hydrogen
O	-	Oxygen
N	-	Nitrogen
P	-	Potassium
S	-	Sulphur
CL	-	Chloride
Si	-	Silicon
kg^{-1}	-	Kilogram
cm^{-1}	-	Centimetre
min^{-1}	-	Minute
sec^{-1}	-	Second
μm	-	Micrometre
mM	-	Millimetre
C: N	-	Carbon to Nitrogen
KJ mol^{-1}	-	Kilojoule mole
H°f	-	Henry Degree Fahrenheit

CHAPTER 1

INTRODUCTION

1.0 Background of Biochar

Biochar is a stable form of organic carbon that improve the quality of soil and a carbon-rich material obtained from the process of pyrolysis. It was produced from difference biomass sources such as waste straw, woody leftovers (sawdust), animal manure and other waste products. Lehman and Joseph (2009) stated that application of biochar can potentially increase carbon sequestration, improves soil condition and lead to sustainable management of organic waste due to its structure and composition.

Sawdust is a by-product from wood sawing process. It is composed of three main substances: cellulose, hemicellulose and lignin, which is polymers. Polymers are a long-chained molecules of repeating units with high molecular weight, where each molecule has tens of thousands of atoms. The main chemical components of sawdust are carbon (60.8%), hydrogen (5.2%), oxygen (33.8%) and nitrogen (0.9%) (Horisawa et al., 1999). Apart from cellulose, hemicellulose and lignin, a variety of additional materials are present in low abundance, including nitrogenous materials, such as pectin, starch, low molecular weight sugars and minerals (iron, magnesium and manganese). In addition, due to the greater amount of lignin and cellulose present in the sawdust, it will have high aromatic carbon, thus, gives the output product (biochar) high stability and resistance to microbial decomposition.

Besides, the sawdust made from biomass will undergo thermal pyrolysis process by using a pyrolysis reactor with little or absence of oxygen. Then, with little or absence of oxygen, the sawdust does not combust but the chemical compound that found in sawdust (e.g. cellulose, hemicellulose and lignin) that make up the sawdust thermally decompose into three outputs; solid (biochar), liquid (bio-oil) and gas (syngas). Figure 1.1 shows the structure of wood cells that composed of cellulose, hemicellulose and lignin.

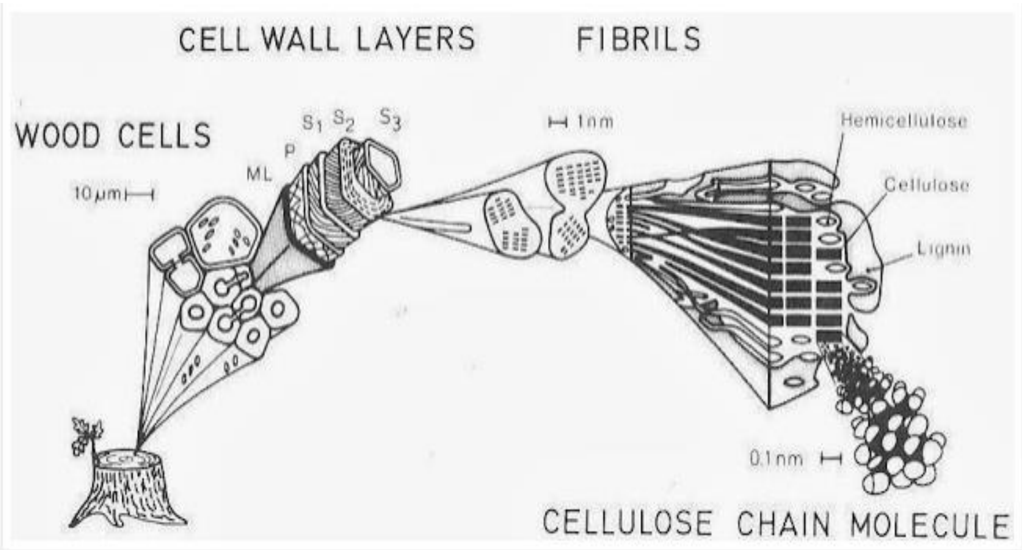


Figure 1.1: Structure of wood cells (Hoffman, P. and Jones, M. A, 2011).

The three outputs are depending on the thermal environment and its temperature. The pyrolysis processes can be classified into three types which is fast pyrolysis (600°C), intermediate pyrolysis (400°C) and slow pyrolysis (350°C). At temperature 600°C, the pyrolysis will yield mainly gas (syngas) meanwhile at temperature 400°C and 350°C the main products are liquid (bio-oil) and solid (biochar) respectively. Thus, the slow pyrolysis which is 350°C being the most suitable process to obtain higher biochar yields. Lehmann and Joseph (2009) present that when the char is applied for environmental management and productivity benefits to soil it is described as biochar. Figure 1.2 shows that the output of pyrolysis processes of sawdust.

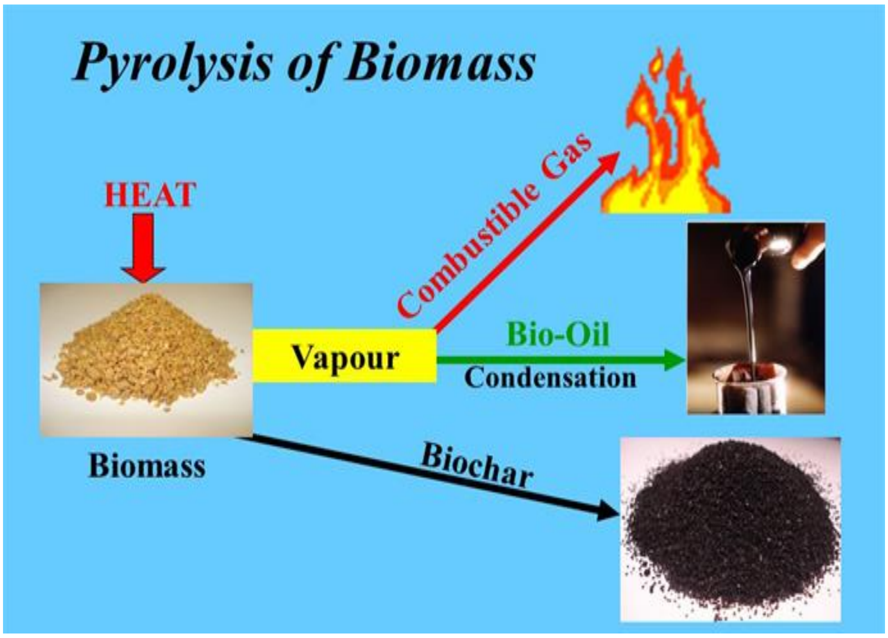
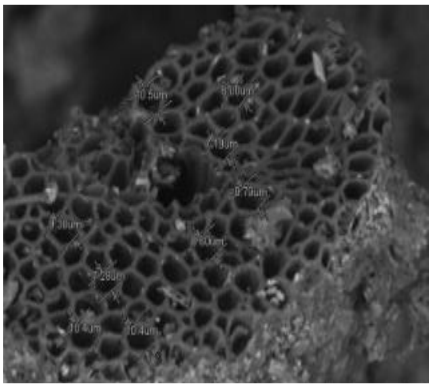
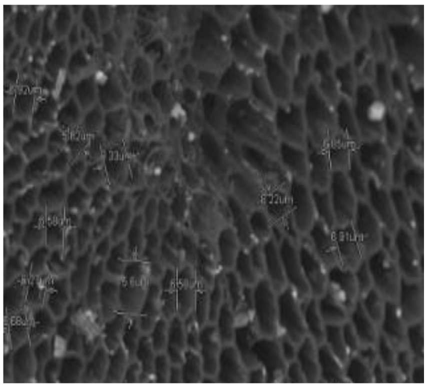


Figure 1.2: Pyrolysis of biomass (Qambrani et al., 2017).

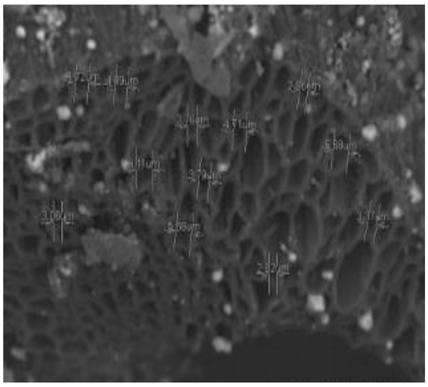
During pyrolysis process as shown in Figure 1.2, the porosity surface will be created and it is an important physical feature of biochar in soil processes and its behaviour. The reaction conditions of the pyrolysis production process can be engineered to change the product ratios and properties (Bridgwater, 2007; Di Blasi, 2008). There are three types of pores according to its internal diameter, which is micropores (< 2 nm), mesopores (between 2 nm and 50 nm) and macropores (> 50 nm) as shown in Figure 1.3. Micropores are tiny pores that hold air while macropores are the large space found in soil that hold water. At temperature 350°C, macropores will be formed while at temperature 400°C and 600°C, mesopores and micropores is mainly yields respectively.



(i) Micropores



(ii) Mesopores



(iii) Macropores

Figure 1.3: Type of Pores (Downie et al. 2009).

Macropores that have large surface area will be preferred habitat for microbial communities in utilizing the soil. Besides, macropores present on biochar may provide suitable area for group of microorganism to grow and reproduce. In addition, micropores and mesopores of biochar may also allow it to maintain more moisture in soil and increase the water holding capacity.

Besides, Lee Y. et al., (2013) investigated that the pH value is an important property of the soil, which effect the types of plants and microorganism to grow and the opportunity of nutrients to be absorbed. At temperature 400°C, the biochar obtain higher pH value which is 7.67. As the biochar had the highest pH value, it can be assumed that the biochar is in highly alkaline condition. Udoetok I.A., (2012) stated that the high pH value of the biochar makes it suitable to reduce the acidity of soil and also increase the availability of soil nutrients.

The presence of functional groups such as phenolic, carboxylic acid, lactone, carbonyls, quinones and pyranose on biochar surface affect surface negative charge (e.g. cation exchange capacity, CEC) and surface positive charge (e.g. anion exchange capacity, AEC). AEC and CEC help to maintain ionic nutrient compound in soil. At temperature 600°C, the biochar acquire the lowest CEC value compared to the temperature 400°C and 350°C. This is because of the aromatization of carbon in the ash of biochar produced might reduce the content of CEC in the biochar.

Moreover, the highest CEC value of biochar will increase the retention ability of nutrients in soil which is good for growing plants (Uddin M.A. et al.).

Last but not least, due the transforming biomass into biochar, it causes rearrangement of the original sugars to aromatic, formation of porous, reactive carbon surface and N use efficiency. Therefore, the biochar will be mixed with urea solution by undergoing impregnation process to produce the fertilizer. Thus, this research is carried out to investigate the effect of temperature and urea solution concentration in urea impregnation process into biochar. Furthermore, as the biomass gives benefit to human and environment as well, it is importantly that the biomass to be fully utilized for future.

2.0 Problem Statements

Nowadays, to reduce greenhouse gas emission and global warming crisis, the use of alternative energy sources and renewable fuels were implemented. These implementation can help to reduce the use on fossil fuel resources and also CO_2 emission. Thus, an idea in recent years that has attracted the worldwide interest by introducing biochar which produced from carbonization of biomass through pyrolysis as soil amendment and also C sequestration in the soil. The biochar act as a new approach to establish, in long term a significant sink CO_2 in terrestrial ecosystem.

Biochar, the biomass waste obtained from the process of pyrolysis in the absence of oxygen is a potential as a soil amendment, N use efficiency and C sequerestation medium. As the biochar is used as soil amendment, the applied of biochar to the soil will increase soil fertility and improve soil quality. Moreover, the other soil benefits include raising soil pH, increasing moisture holding capacity, improving cation exchange capacity (CEC) and maintain nutrients. Then, in this case, to improve the regulation of N use efficiency, the biochar will be combined with urea solution at varying temperature and concentration throughout the impregnation process to produce the fertilizer.

3.0 Objectives

The experiments were carried out with the following specific objectives:

1. To examine the effect of temperature and concentration of urea solution in the biochar- urea impregnation process.

2. To analyse the characteristic of biochar.

4.0 Scope of Project

This study is focused on the conversion of rubber wood sawdust into biochar and the urea impregnation process into biochar at selected temperature which are 30°C and 50°C, and urea concentration of 4% and 8%. The material characterisations were carried out using FT-IR, XRD and SEM for hydroxyl group analysis, phase analyses and microstructural change respectively.

5.0 Report Outline

In this research, to create the slow release urea fertilizer, the effect of temperature and concentration of urea solution in urea impregnation process into biochar will be investigated. Basically, for chapter 1, this thesis is going to discuss on the introduction which consist of general view on research selected following by background, objective, problem statement and others. In chapter 2, this thesis will be referring to journal to produce a literature review. Moving on to chapter 3, it will talk about theoretical approaches, experimental design and methods and standard procedure description. In chapter 4, this section presents or describe the data in the preceding section and contains the analyses or interpretations of the result obtained.

Lastly, for chapter 5, which is conclusion and recommendation, it will be based on all the analysis that has been conducted, comparison, and overall improvement. After that will have to come out with recommendation for future work.

CHAPTER 2

LITERATURE REVIEW

2.1 Biochar

2.1.1 Physical and Chemical Properties

The physical and chemical properties of biochar such as presence of functional groups on its surface and high porosity of structure provide various advantages as a buffer to infertile soil. The presence of surface functional groups on its surface help to improve the cation exchange capacity and anion exchange capacity for nutrient retention in the soil. The pore structure of biochar provides a habitat for microbial activities and helps to increase the water holding capacity.

2.1.1.1 Structural Composition

At temperature between 350^0C and 400^0C, through thermal degradation of cellulose, the mass loss in the form of volatiles by leaving behind a rigid amorphous C matrix. Baldock and Smernik, (2002); Demirbas, (2004) investigated that as the pyrolysis temperature increase, so thus the proportion of aromatic carbon in the biochar, due to relatives increase in the loss of volatile matter (mostly water, followed by hydrocarbon, hydrogen, carbon oxide and carbon dioxide) and the conversion of alkyl and O-alkyl C to aryl C. Besides, around temperature 400^0C, at the amorphous C phase, the polyromatic graphene sheets begin to grow and eventually join together. While at above 600^0C, carbonization will be a dominant process. Carbonization is the removal of most remaining non C-atoms and relatively increase of C content up to