Faculty of Manufacturing Engineering

EFFECT OF ACID TREATED BIOCHAR ON UREA FERTILIZER FOR RETAINING AMMONIUM AND NITRATE IONS IN SOIL

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EFFECT OF ACID TREATED BIOCHAR ON UREA FERTILIZER FOR RETAINING AMMONIUM AND NITRATE IONS IN SOIL

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DECLARATION

I declare that this thesis entitled “Effect of Acid Treated Biochar on Urea Fertilizer for Retaining Ammonium and Nitrate Ions in Soil” is the result of my own research except as cited in the references. The thesis has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.

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APPROVAL

I hereby declare that I have read this thesis and in my opinion this thesis is sufficient in terms of scope and quality for the award of Master of Science in Manufacturing Engineering

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DEDICATION

To my beloved husband, children, mother and father

ABSTRACT

Urea is widely used as fertilizer because it is inexpensive, economical to produce, soluble in water and contain high amount of nitrogen (47%). Biochar impregnated urea has the potential to improve nitrogen use efficiency of plant by reducing the N losses via gaseous emission of ammonia (NH3) and nitrous oxide (N_2O) to the atmosphere and nitrate leaching into surface and ground water bodies. The aim of this study is to investigate the biochar performance in reducing the loses of ammonium and nitrate ions by increasing its negative surface charge to improve its cation exchange capacity (CEC) which may influence its ability to retain the ions. The biochar was derived from rubber wood sawdust, underwent a pyrolysis process before treated with phosphoric acid at various concentration to improve its ion retention properties. From the optimization result obtained using response surface method regarding the acid treatment process, the best setting suggested for biochar was treatment with 1.5M phosphoric acid at 90°C. Confirmation runs obtained from the best setting were 7.06 pH with negative surface charge of 7.18 mmol/gram. Ammonium and nitrate retention in soil were measured and compared between ureas impregnated with acid treated and untreated biochars. Results showed that urea with acid treated biochar was able to retain 48.7% of ammonium and 45.2% of nitrate as compared to 44.7% and 28.0% shown by urea with untreated biochar, after week 3. This study affirms that acid treated biochar increases the ability of urea to retain more ammonium and nitrate compared to the untreated biochar.

ABSTRAK

Urea digunakan secara meluas kerana ianya murah, ekonomik untuk dihasilkan, larut dalam air dan mengandungi kandungan nitrogen yang tinggi (47%). Urea yang mengandungi biochar berpotensi untuk meningkatkan kecekapan penggunaan nitrogen tumbuhan dengan mengurangkan kehilangan N melalui pelepasan gas ammonia (NH3) dan nitrous oksida (N_2O) ke atmosfera dan penembusan nitrat ke dalam tanah. Tujuan kajian ini adalah untuk mengkaji prestasi biochar dalam mengurangkan kehilangan ion ammonium dan nitrat dengan meningkatkan caj permukaan negatif untuk meningkatkan kapasiti pertukaran kation (CEC) yang mungkin mempengaruhi kemampuannya untuk mengekalkan ion. Biochar berasal dari habuk kayu getah, menjalani proses pirolisis sebelum dirawat dengan asid fosforik pada pelbagai tumpuan untuk memperbaiki sifat pengekalan ionnya. Menerapkan kaedah permukaan tindak balas untuk mengoptimumkan proses rawatan asid, tetapan terbaik yang dicadangkan untuk biochar perlu dirawat dengan asid fosforik 1.5M pada 90°C. Pengesahan yang diperolehi daripada penetapan terbaik adalah 7.06 pH dengan caj permukaan negatif sebanyak 7.18 mmol / gram. Pengekalan ammonium dan nitrat dalam tanah diukur dan dibandingkan antara urea yang mengandungi biochar terawat asid dengan sampel mengandungi biochar tidak dirawat. Keputusan menunjukkan urea mengandungi biochar yang dirawat dengan asid dapat mengekalkan 48.7% ammonium dan 45.2% daripada nitrat berbanding 44.7% dan 28.0% yang ditunjukkan oleh urea mengandungi biochar yang tidak dirawat, selepas minggu ke-3. Kajian ini membuktikan bahawa biochar yang dirawat dengan asid dapat membantu urea mengekalkan lebih banyak ammonium dan nitrate berbanding biochar yang tidak dirawat.

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TABLE OF CONTENTS

		PAGE
DECLARATION		
APPROVAL		
DEDICATION		
ABSTRACT		**i**
ABSTRAK		**ii**
ACKNOWLEDGEMENTS		**iii**
TABLE OF CONTENTS		**iv**
LIST OF TABLES		**vii**
LIST OF FIGURES		**ix**
LIST OF ABBREVIATIONS		**xi**
LIST OF SYMBOLS		**xii**
LIST OF PUBLICATIONS		**xiii**
CHAPTER		
1.	**INTRODUCTION**	**1**
1.1	Background	1
1.2	Problem statement	4
1.3	Objectives	5
1.4	Research scope	5
1.5	Thesis overview	6
2.	**LITERATURE REVIEW**	**7**
2.1	Nitrogenous fertilizer	7
	2.1.1 Urea fertilizer	9
	2.1.2 Importance of urea fertilizer to the paddy plant	10
2.2	Application of urea to the soils	11
	2.2.1 N mineralization	12
	2.2.2 Environmental effect by urea fertilizer	15
	2.2.2.1 Ammonia volatilization	15
	2.2.2.2 Nitrous oxide emission	16
	2.2.2.3 Leaching	16
2.3	Strategies to improve urea fertilizer on paddy	17
2.4	Slow release fertilizer	18
	2.4.1 Biochar based slow release fertilizer	19
2.5	Biomass derived biochar	20
	2.5.1 Slow pyrolysis of biochar	21
	2.5.2 Properties of biochar (physical and chemical)	21
2.6	Mechanism of anion and cation exchange capacity (CEC)	24
2.7	Application of biochar to the soils	25
2.8	Modified biochar treatment	27
	2.8.1 Biochar treatment with base/alkali	28
	2.8.2 Biochar treatment with acid	31
	2.8.2.1 Treatment of biochar by using phosphoric acid	32

2.9 Design of experiment (DOE) 33
2.9.1 Response surface methodology (RSM) 34
2.10 Summary 35

3. METHODOLOGY 37
3.1 Flow chart of the experiment 37
3.2 Raw materials 39
3.3 Pyrolysis of RWSD to produce biochar 40
3.4 Acid treatment of biochar 40
3.4.1 Determination of alkalinity 41
3.4.2 Determination of negative surface charge by using Boehm titration 42
3.4.3 Design and optimization process for acid treatment of biochar by using experimental design 43
3.5 Treated biochar impregnated urea 45
3.6 Characterization of acid treated biochar and biochar impregnated urea 46
3.6.1 Elemental analysis by CHNS analyzer 46
3.6.2 Determination of surface functional group via Fourier Transform Infrared (FT-IR) spectroscopy 47
3.6.3 Percentage of porosity by using mercury microporosity 48
3.6.4 Microstructure and composition analysis using Scanning Electron Microscopy (SEM) and SEM with EDX 49
3.7 Determination of ammonium and nitrate content using mineralization test by incubation 50
3.8 Leaching test 53
3.9 Summary 54

4. RESULT AND DISCUSSION 55
4.1 Characteristic of rubber wood sawdust and urea 55
4.1.1 Proximate and ultimate analysis of RWSD 56
4.1.2 Characterization using SEM and SEM-EDX 59
4.2 Acid treatment of biochar by using RSM via CCD 60
4.2.1 Effect of process parameters on response of acid treatment biochar using phosphoric acid 60
4.2.2 The determination of appropriate polynomial equation for the model 61
4.2.2.1 ANOVA analysis of pH for acid treatment biochar 62
4.2.2.2 ANOVA analysis of surface negative charge for acid treatment biochar 68
4.2.3 Optimization process 74
4.3 Characterization of acid treated biochar, acid treated biochar impregnated urea and control biochar 76
4.3.1 C, H, N and S analysis 76
4.3.2 Fourier Transform Infrared (FTiR) spectroscopy analysis 78
4.3.3 Microporosity analysis of CB, TB1 and TB2 80
4.3.4 Surface morphology of treated biochar, biochar impregnated urea and treated biochar impregnated urea (CB, TB1, TB2 and CB-1, TB1-U, TB2-U) using SEM and SEM-EDX 82
4.4 Mineralization test by incubation 87

4.4.1 Ammonium and nitrate content in soil after incubation test 88
4.5 Water column leaching test 91
4.5.1 Ammonium and nitrate leaching 91
4.6 Summary 93

5. CONCLUSION AND RECOMMENDATIONS 96
5.1 Conclusion 96
5.2 Future recommendations 97

REFERENCES 98

LIST OF TABLES

TABLE	TITLE	PAGE
1.1	World demand for fertilizer nutrients, 2015-2019 (thousand tonnes)	2
2.1	Nutrients required by plants (McKenzie, 1998)	8
2.2	Factor affecting N_2O emission and their management practice (Zaman et al., 2012)	16
2.3	Chemical modification methodologies of biochars, production conditions contaminant removals (mechanism) and their application	30
2.4	Enhanced performance of engineered biochar (Rajapaksha et al., 2016)	31
3.1	Experimental codes and levels of independent variable for response surface methodology experiment	44
3.2	Designation of samples	46
3.3	Preparation of the reagent	50
4.1	Proximate analysis of RWSD (wt% dry basis)	57
4.2	Ultimate analysis of RWSD (wt% dry basis)	57
4.3	Design of experiments and results for acid treatment in biochar	61
4.4	Fit summary for pH model	62
4.5	Fit summary for negative surface charge model	62
4.6	ANOVA analysis of acid treatment in biochar for pH before eliminating the insignificants terms	63
4.7	New ANOVA results on the pH after eliminated insignificants terms	64
4.8	ANOVA analysis on negative surface charge for acid treatment of biochar before eliminating the insignificants terms	69
4.9	New ANOVA on the negative surface charge after eliminating insignificants terms	70
4.10	The goals for factors and responses to determine the optimum parameters in RSM	75

4.11	Optimization results for acid treated biochar based on Design Expert Software	76
4.12	Set of parameters of selected biochar treated	76
4.13	Elemental composition of the biochar and treated biochar	77
4.14	Microporosity analysis using mercury porosimetry	81
4.15	Properties of control urea, urea impregnated untreated biochar and urea impregnated treated biochar	87

LIST OF FIGURES

FIGURE	TITLE	PAGE
2.1	Chemical structure of urea (Chengteh et al., 1995)	9
2.2	Mechanism of urea hydrolysis	12
2.3	Nitrogen cycle in soils (Agric gov., 2017)	13
2.4	Cation absorption onto negative surface charge of biochar (Herbert et al., 2012)	24
2.5	Mechanisms for biochar application to the soils (Mandal et al., 2016)	27
2.6	Biochar modification method	28
3.1	Flow chart of the experiment	38
3.2	Rubber Wood Sawdust (RWSD)	40
3.3	Acid treatment of biochar	41
3.4	Incubation experiment setup	51
3.5	Method determining amount of ammonium and nitrate in soil solution	51
3.6	Water column leaching test experiment set up	53
4.1	Thermogravimetric analysis (TGA) of RWSD using 700°C heating rate	58
4.2	SEM micrograph of RWSD used as raw materials	60
4.3	SEM micrograph of urea fertilizer used as raw materials	60
4.4	Normal plot residuals on pH for acid treatment biochar	65
4.5	Predicted vs actual on pH for acid treatment biochar	66
4.6	One factor plot on pH for acid treatment biochar	67
4.7	3D plot of treated biochar pH value against process temperature and acid concentration applied	67
4.8	3D plot of treated biochar pH value against time and acid concentration applied	68
4.9	Normal plot of residual on negative surface charge for acid treatment biochar	71
4.10	Predicted vs actual on negative surface charge for acid treatment biochar	71

4.11	Interaction graph on negative surface charge for acid treatment biochar for factor concentration and time	72
4.12	Interaction graph on negative surface charge for acid treatment biochar for factor temperature and time	73
4.13	3D plot of treated biochar negative surface charge value against process temperature and acid concentrations applied	73
4.14	3D plot of treated biochar on negative surface charge value against process temperature and time during treatment	74
4.15	Histogram graph of elemental composition of the biochar and treated biochar	78
4.16	FT-IR spectra of biochar and treated biochar (CB, TB1 abd TB2)	79
4.17	FT-IR spectra of urea and biochar impregnated urea sample (Urea, CB-U, TB1-U and TB2-U)	80
4.18	SEM micrograph of untreated and treated biochar (CB,TB1 and TB2)	84
4.19	SEM micrograph of untreated and treated biochar impregnated urea (CB-U, TB1-U and TB2-U)	86
4.20	Ammonium content in soil of blank, urea, untreated and treated biochar impregnated urea (CB-U, TB1-U and TB2-U) in 4 weeks	90
4.21	Nitrate content in soil of blank, urea, untreated and treated biochar impregnated urea (CB-U, TB1-U and TB2-U) in 4 weeks	90
4.22	Ammonium content in leachate for blank, urea, untreated and treated biochar impregnated urea (CB-U, TB1-U and TB2-U) after 4 weeks	92
4.23	Nitrate content leachate for blank, urea, untreated and treated biochar impregnated urea (CB-U, TB1-U and TB2-U) after 4 weeks	93

LIST OF ABBREVIATIONS

UF	-	Urea Fertilizer
N	-	Nitrogen
CEC	-	Cation Exchangeable Charge
SRF	-	Slow Release Fertilizer
SEM	-	Scanning Electron Microscope
EDX	-	Energy Dispersive X-Ray
FT-IR	-	Fourier Transform Infrared
ATR	-	Attenuated Total Reflectance
RSM	-	Response Surface Methodology
CCD	-	Central Composite Design
ANOVA	-	Analysis of Variance
RWSD	-	Rubber Wood Sawdustp

LIST OF SYMBOLS

N	-	Normality
M	-	Molarity
h	-	Hour
mmol/g	-	Mili mol per gram
α	-	Alpha
˚C	-	Degree celcius
wt%	-	Weight percentage
%	-	Percentage
µm	-	Micrometer
nm	-	Nanometer
g/mL	-	Gram per milliliter
mL/g	-	Milliliter per gram
m^2/g	-	Meter square per gram
±	-	Plus minus
ml	-	Milliliter
mg	-	Milligram
g	-	Gram

LIST OF PUBLICATIONS

1. Farhaneem, N., Dimin, M. F., Shaaban, A., N Mohamad., 2018. Optimization of Phosphoric Acid Treatment Biochar using Response Surface Method. *Journal of Advance Manufacturing Technology*, 12 (1), pp. 453-466.

2. Farhaneem, N., Se, S. M., Dimin, M. F., Shaaban, A., 2017. Microstructual Analysis of Biochar Obtained from Rubber Wood Sawdust via Slow Pyrolysis. *Solid State Phenomena, Trans Tech Publication*, Switzerland, 264, pp. 13-16.

CHAPTER 1

INTRODUCTION

This chapter focuses on the background of urea fertilizer and its function in paddy field fertilization and rice production in Malaysia. Problems related to urea fertilizer and biochar impregnated urea from the earlier researcher are also included. Lastly, goal, scope and thesis overview are explained in this chapter.

1.1 Background

The demand to optimize rice production is becoming more important in Malaysia as the nation is heavily dependent on rice as a staple food. The demand of rice increases with the increase of population in Malaysia. The optimum rice production will not only deliver sufficient supply of food for the country, but also necessary in providing food security. Therefore, it is important to improve rice production in Malaysia especially improving the used of fertilizer for the rice cultivation.

Fertilizers are the most important added nutrient for plants to consume in order to introduce higher yield. Based on the International Fertilizer Industry Association's (IFA) Fertilizer Outlook 2013-2017, the total fertilizer nutrient ($N+P_2O_5+K_2O$) consumption is forecasted at 184.67 million tonnes in 2014 and estimated to reach 186.6 million tonnes in 2015. It is expected to reach 199 million tonnes by the end of 2019 and attain successful growth of 1.6 percent per year. The global demand for fertilizer nutrients is summarized in Table 1.1. It is likely to show that the world of fertilizer consumption has been increasing over the year.

Table 1.1: World demand for fertilizer nutrients, 2015-2019 (thousand tonnes)

Year	2015	2016	2017	2018	2019
Nitrogen (N)	112539	113955	115489	116905	118222
Phosphate (P_2O_5)	42113	42865	43785	44652	45527
Potash (K_2)	31973	32802	336229	34452	352257
Total	18625	189622	192912	196009	199006

Based on the world demand fertilizer nutrient, nitrogen shows the highest consumption compared to other nutrients. The world nitrogen fertilizer demand has increased from 111,400,000 tonnes in 2013 to 113,100,000 tonnes in 2014, at a growth rate of 1.5 percent. It is expected to be around 119,400,000 tonnes in 2018 at the annual growth of 1.4 percent (FAO, 2015).

Urea fertilizers as the source of nitrogen (N) has been widely used on the soil for providing additional nutrient for plant growth and sustaining the soil fertility. The application of urea fertilizers on paddy fields has been accepted to produce high rice grain yields and improves the N availability. Based on previous report (Trenkel 1997 and Zheng et al. 2010), among the N fertilizers, urea is commonly used to be applied on paddy fields because it is the cheapest form of granular chemical N fertilizer which contains high N nutrient of about 47%. This is important as fertilizers have grown increasingly and represents about 50% of the world's N fertilizers (Fageria and Baligar, 2001). Thus, it is economical to produce, ease of handling, transport and available throughout the world (Ibrahim et al., 2014).

However, it has been estimated that about 60% of the urea N is lost to the envvvironment. N fertilizers that are lost from cultivated soils via ammonia (NH_3) volatilization, nitrate (NO_3) leaching and nitrous oxide (N_2O) emission could give high economic and environmental impact (Ramírez et al., 1997 ; Zhou et al., 2003). Thus, it is

important to search an alternative as a substrate that can regulate the release of N from urea fertilizers. Hence, many research has been made on the modification of urea fertilizer as a slow release fertilizer (SRF) to enhance the efficiency of urea. Nutrient form the urea fertilizer such as ammonium and nitrate ion which are absorbed by the plant can be retained. The used of organic and inorganic materials such as starch, biochar, neem, tar, zeolite, halloysite and etc has been introduced. From the previous study, the use of organic substance such as biochar can be used as a substrate to regulate the adsorption and desorption pattern of N in different formed that can serve as a based to develop biochar based slow release fertilizer.

Biochar is the carbon-rich product of agricultural wastes such as wood, manure and leaves heated in a closed container with or without availability of oxygen (Lehmann & Joseph, 2009; Lee et al., 2013). It is well-known as a value-added product, which can be used for several purposes such as a support material for delivering plant nutrients (González et al., 2012). Biochar is likely to be used as a soil amendment medium in agricultural application since it has the ability as a soil conditioner to improve soil fertility and nutrient-use efficiency using locally available and renewable material in a sustainable way.

Published studies suggest the modification of biochar before their combining with urea as a bio-fertilizer. These modifications include the addition of one or more nutrients either by a direct mixing process, encapsulation or palletization. Hence it was found that, modification of biochar include chemical treatment can improve their physical and chemical properties (Lin et al., 2012b). The use of phosphoric acid (H_3PO_4) treatment is reported to alter the properties of biochar surface and reduce environmental effect when it is being applied to the surface of soils (Doydora et al., 2011). Hence, the current research is to introduce the treatment on biochar with urea to increase the efficiency of N plant uptake and reduce nitrous oxide (N_2O) and ammonia (NH_3) released to the environment.

1.2 Problem statement

The major concern of the farmers is the efficiency of urea fertilizer being applied to the rice crop. The consumption efficiency or plant uptake of N from urea is very low at about 35-60% due to its water soluble property and agriculture practice (application of urea fertilizer) (Li et al., 2008). There is approximately about 60% of N of applied urea reported to have lost to the environment that can causes economic and resources loss and serious environmental pollution such as surface runoff, nitrate leaching, urea hydrolysis and ammonia volatilization (Jones et al., 2007; Liu et al., 2010).

Previously, researches has been made on modification of urea fertilizer to improve their efficiency and reduce environmental effect. Biochar application has been reported to increase the efficiency of urea fertilizer and able to lower environmental effect (Ding et al., 2010; Blackwell et al., 2009). Recent works have been done on the use of biochar as a support material to the urea fertilizer. It was found that biochar can be impregnated with urea and the compound produced has capability as a slow release mechanism. Moreover, with the supplement of biochar to urea, fertilizers tend to smoothen and retard nutrient release into the soil when it is applied (González et al., 2015). The use of these impregnated biochar with urea can also help to reduce ammonia volatilization and nitrous oxide (N_2O) emission.

Other studies have examined biochar's ion exchange and surface charge characteristics which also vary among different chars. Both of these properties are expected to influence ion adsorption during application (Mukherjee et al., 2011). Surface charge is a parameter that can be used to predict the adsorption and nutrient holding characteristics of a soil or soil component. Biochar has negative surface charge that help to increase cation exchange charge (CEC). According to Brady and Weil. (1984), CEC has been found can improve soil's ability to hold nutrient such as ammonium and nitrate.

From the previous research by Sian Meng, 2014, biochar produced at 400°C results on 2.5-3mmol/g negative surface charge. The higher negative surface charge is a potential for the retention of more nutrient by improving the CEC and able to reduce ammonia and nitrous oxide emission. Thus, for the alternative approach, these biochar properties can be improve by using chemical activation method such as KOH, NaOH, HCL, H_3PO_4 and etc. However, the role of biochar treated with phosphoric acid still unclear up to now on retaining the ammonium and nitrate.

1.3 Objectives

The objectives of this research are as follow:

1. To evaluate and characterize the phosphoric acid treated biochar.
2. To optimize the combined effect of process of treated biochar using Central Composite Design (CCD) of Response Surface Method (RSM).
3. To evaluate the performance of biochar impregnated urea with respect to ammonia and nitrous oxide loss.

1.4 Research scope

The study is focusing on acid treatment of biochar as the biochar is impregnated into urea fertilizer to produce treated biochar-urea (TBU). Rubber wood sawdust (RWSD) is selected as the biomass resources to produce biochar through pyrolysis process by using horizontal furnace. The biochar samples are treated with phosphoric acid following the designed experiment by using Response Surface Methodology (RSM) via Central Composite Design (CCD) method. Based on the result and analysis by Design Expert Software, the selected treated biochar (TB1) and treated biochar 2 (TB2) are chosen and the sample is then compared with biochar that has not been treated which is labelled as CB. All

the selected biochar are characterized by using FTiR, mercury microporosity, SEM and SEM/EDX to compare their physiochemical properties before the biochar is impregnated with urea fertilizer. For the application of the fertilizer, incubation and mineralization test is used to determine N_2O release and NH_3 loss from paddy soils including nutrient retained by using leaching test.

1.5 Thesis overview

This thesis is divided into five chapters. The first chapter is the introduction of the research including background, problem statement, research objectives, and scope of study. Chapter two presents the literature review discussing on urea fertilizer and their slow release mechanism, effect of urea fertilizer, biochar and their properties as a support material to the fertilizer, process and acid treatment in biochar and application of biochar and biochar urea to the soil. Then, it is followed by the chapter three which further explains the methodology of the research work including preparation of biochar by rubber wood sawdust (RWSD), acid treatment in biochar using experimental design, characterization and application of urea impregnated biochar to the paddy soils. Next, all the results of the research are analyzed and discussed in chapter four. Finally, chapter five summarizes and concludes the research with some recommendations for further improvement in the area.