UNIVERSITI TEKNIKAL MALAYSIA MELAKA

Faculty of Manufacturing Engineering

SLOW RELEASE BEHAVIOR OF BIODEGRADABLE STARCH POLYVINYL ALCOHOL UREA FERTILIZER

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LUM YIP HING

A thesis submitted
in fulfilment of the requirements for the degree of Doctor of Philosophy

Faculty of Manufacturing Engineering

UNIVERSTITI TEKNIKAL MALAYSIA MELAKA

2018

DECLARATION

I declare that this thesis entitled "Slow Release Behavior of Biodegradable Starch Polyvinyl Alcohol Urea Fertilizer" 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.

Signature	:	....
Name	:	....
Date	:	....

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 Doctor of Philosophy.

Signature : ....

Supervisor Name : ....

Date : ....

DEDICATION

I Humbly Dedicate to

My Beloved Father and Mother,
Lum Choong Weng and
Ng Ah Mooi

&

My Lovely Wife
Tan Fui Teng

&

All My Friends;
Who Have Bring Me

The Gaiety, Encourage, Delight and Happiness in My life!

ABSTRACT

By the year 2050, the world population will be projected to reach 9 billion. The world arable land areas are reported to decline in the past years with potentially disastrous consequences as global demand of food soars. There will be absolutely need that the use of nitrogen fertilizers as the primary vital nutrients in crop output must be increased. The most widely used is urea due to its high nitrogen content, 46% by weight which has surpassed other nitrogen fertilizer. However, approximately 70% of the high soluble urea fertilizer may be lost into the environment through surface runoff, leaching, and volatilization, thereby reduce farmer's profit and lead to serious environmental pollution. The slow release fertilizer is most often suggested to avoid losses of nitrogen from agricultural land. Nevertheless, the cost of producing slow release fertilizer has limited its development and accumulation of hydrophobic matrix is a menace to environment. The objective of this study were to examine the performance of synthesized biodegradable matrix based urea fertilizer compared to conventional urea fertilizer in relation to slow release properties, ammonium retention and ammonia loss under flooded condition. Tapioca starch was crosslinked with polyvinyl alcohol by using boric acid to form a matrix having slow release characteristic when immersed in water. The experiments were arranged in a two-level factorial design with three replications. Treatments were two levels of concentration of boric acid (-1: 1 %; +1: 4%), reaction time (-1: 1 hour; +1: 4 hours) and heating temperature (-1:60℃; +1: 90℃) in the synthesis formulation and condition of crosslinked matrix. The soil burial degradation data showed that synthesized matrix were still able to degrade in the range of 35.81% to 56.48% even in the presence of boric acid crosslinking. Compared to conventional urea fertilizer which was dissolved within seconds in water, the synthesized matrix slowly released the urea completely during the 8 hours soaking period at 30ºC with the rotation speed of 100 rpm. The release mechanism of the urea from the synthesized matrix obeyed the Korsmeyer-Peppas model with a quasi-Fickian diffusion mechanism. Soil incubation experiments showed that the matrix could retain the soil exchangeable ammonium due to partially negatively charged sites after ionization of matrix. Urea encapsulated with matrix significantly reduced ammonia loss from urea by 40% up to 55% compared with pure urea alone. Hence, there was a widely potential utility of matrix in agricultural industry as fertilizer carrier due to its simple step in manufacturing process, environmental friendly, and high nitrogen use efficiency.

ABSTRAK

Menjelang tahun 2050, kepadatan penduduk dunia dianggarkan berjumlah 9 bilion. Kawasan tanah subur dunia dilaporkan terus menurun dan ini amat merugikan apabila permintaan keatas makanan dunia meningkat. Dengan itu, penggunaan baja nitrogen sebagai nutrien utama dalam penghasilan tanaman perlu ditingkatkan. Baja nitrogen yang paling banyak digunakan adalah urea kerana kandungan nitrogennya yang lebih tinggi, iaitu 46% berbanding baja nitrogen yang lain. Walau bagaimanapun, hampir 70% daripada baja urea bersifat amat terlarut air dan akan hilang kepersekitaran melalui hanyutan permukaan, larut lesap dan volatilasi lalu merugikan ekonomi petani serta membawa pencemaran alam sekitar yang serius. Baja pelepasan perlahan sering dicadangkan untuk mengelakkan kehilangan nitrogen dari tanah pertanian. Namun disebabkan kos pengeluaran yang tinggi, pembangunan baja pelepasan perlahan menjadi terhad dan pengumpulan produk sampingan iaitu matriks hidrofobik terus menjadi ancaman kepada alam sekitar. Objektif kajian ini adalah untuk menentukan prestasi baja urea yang disaluti oleh bahan polimer matriks biodegradasi berbanding baja urea tulen dan menghubungkaitkan sifat pelepasan, kadar penukaran ammonium dan kadar pemeruapan ammonia dalam persekitaran berair. Kanji ubi kayu telah dicampurkan dengan polyvinyl alkohol dan asid borik untuk menghasilkan matriks polimer yang mempunyai ciri pelepasan perlahan pada baja urea apabila direndamkan dalam air. Kajian telah dilaksana menggunakan dua faktorial analisis dengan tiga ulangan. Dalam dua factorial analisis, pembolehubah yang dikaji adalah kepekatan asid borik (-1: 1%; 1: 4%), masa untuk bertindak balas (-1: 1 jam; 1: 4 jam) dan suhu pemanasan (-1: 60 ℃; 1 : 90 ℃). Data analisis kemusnahan biodegradasi tanah menunjukkan bahawa biodegradasi matriks polimer berlaku pada kadar 35.81% ke 56.48% dengan kehadiran rangkaian silang asid borik. Berbanding urea tulen yang melarut dalam air dalam beberapa saat, baja urea yang disaluti matriks polimer telah dibuktikan mempunyai ciri pelepasan perlahan kerana urea melarut sepenuhnya dalam masa 8 jam rendaman pada suhu 30ºC di bawah putaran 100 rpm. Pelepasan perlahan urea dari matriks polimer menuruti mekanisma model Korsmeyer-Peppas dan mekanisma penyebaran quasi-Fickian. Eksperimen inkubasi tanah menunjukkan bahawa matriks polimer dapat mengurangkan kadar penukaran ammonium kepada nitrate kerana sebahagian matriks polimer bercas negatif selepas pengionan. Selain itu, matriks polimer dapat mengurangkan kadar kehilangan ammonia sebanyak 40% hingga 55% berbanding baja urea tulen. Dengan ini, matriks polimer berpotensi luas sebagai pembawa baja urea disebabkan oleh kaedah pengeluaran yang mudah, produk mesra alam, dan meningkatkan keberkesanan penggunaan nitrogen oleh tumbuhan.

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LIST OF ABBREVIATIONS

AAPFCO	-	Association of American Plant Food Control Officials
ANOVA	-	Analysis of variance
ASTM	-	American Society for Testing and Materials
CEN	-	European Standardization Committee
DAM	-	Diacetyl monoxim
DOE	-	Design of experiment
DSC	-	Differential Scanning Calorimetry
EPI	-	Epichlorohydrin
FTIR	-	Fourier-Transform Infrared
ISO	-	International Organization for Standardization
KCl-PMA	-	Potassium Chloride-Phenylmercuric Acetate
MBF	-	Matrix based fertilizer
NUE	-	Nutrient use efficiency
PCL	-	Polycaprolactone
PMA	-	Phenylmercuric Acetate
PLA	-	Polylactic acid
PSA	-	Particle size analyzer
PVA	-	Polyvinyl alcohol
PVAc	-	Polyvinyl acetate
R^2	-	Coefficient of determination
SEM	-	Scanning electron microscopy

SP	-	Starch/PVA
SPB	-	Starch/PVA/Boric Acid
SRF	-	Slow release fertilizer
STMP	-	Sodium trimetaphosphate
STPP	-	Sodium triphosphate
XRD	-	X-ray diffraction

LIST OF SYMBOLS

wt. %	-	Weight Percentages
t	-	Time
T_g	-	Gelatinization temperature
T_m	-	Melting temperature
$\triangle H$	-	Enthalpy change
$\triangle s$	-	Entropy change

LIST OF PUBLICATIONS

Lum, Y.H., Shaaban, A., Mitan, N.M.M., Dimin, M.F., Mohamad, N., Hamid, N., and Se, S.M., 2013. Characterization of Urea Encapsulated by Biodegradable Starch-PVA-Glycerol. *Journal of Polymers and the Environment*, 21(4), pp. 1083-1087.

Lum, Y.H., Shaaban, A., Mohamad, N., Dimin, M.F., and Yatim, N.M., 2016. Boric Acid Modified Starch Polyvinyl Alcohol Matrix for Slow Release Fertilizer. *e-Polymers*, 16(2), pp. 151-158.

Lum, Y.H., Shaaban, A., Mohamad, N., and Dimin, M.F., 2016. Slow Release of Urea Encapsulated by Starch PVA Matrix. *Key Engineering Materials*, 707, pp. 28-31.

CHAPTER 1

INTRODUCTION

1.1 Research Background

According to the most recent world population data sheet (PRB, 2016), the world population is around 7.4 billion as of year 2016. The population is currently growing at the rate of around 80 million per year. By 2050, the world population will be projected to reach 9 billion. Nowadays, there are nearly 1 billion people in the world suffering from chronic hunger, and another 2 billion are malnourished. That means one in seven people do not get enough food to be healthy and lead an active life (Mykleby et al., 2016). Hence, the production of food must keep up with the demand and find the new secure sources of food.

The greatest threat to the food security may center on the Earth's dwindling arable land. Alexandratos and Bruinsma (2012) showed that the continuing decline of arable land (in use) per person (Figure 1.1) resulted that the food production was no longer able to sustain a still growing world population. Many global analysts reported the world had lost a third of its arable land due to erosion and pollution in the past years, with potentially disastrous consequences as global demand of food soars (Lal and Stewart, 2016). The degradation of arable land occurred due to overuse of fertilizer changes the soil composition and disrupts the balance of microorganism in soil (Zhang et al., 2015). The main objective for applying fertilizer to arable land is to secure the productivity of the crops. Nevertheless, the long term impacts of chemical fertilizer use are the predominant cause of soil degradation (Jie et al., 2002; Gomiero, 2016).

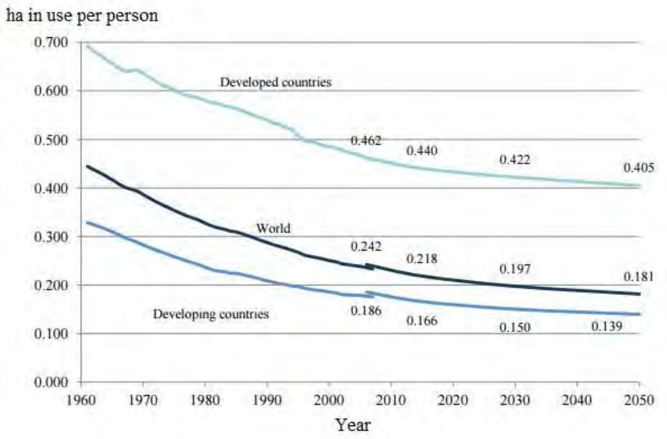

Figure 1.1: Arable Land per Capita: Historical and Projected (Alexandratos and Bruinsma, 2012)

In order to meet food production projections, the farmers, agricultural scientists and governments are working on alternative approaches to provide enough food for feeding a growing global population. Fertilizer is the most common additive nutrient to increase soil's fertility and is essential for plant life. It is largely responsible for boosting agricultural yields. The crucial crops such as corn, wheat, and rice consume large quantities of fertilizer which is more than the nature alone can produce. The application of fertilizer enhances and replenishes the fertility of the soil which has been taken up by crops. Hence, the world fertilizer consumption will increase sharply over the years (increased from 172 million tonnes in 2010 to 263 million tonnes in 2050) to support the rapid rate of population growth (Figure 1.2).

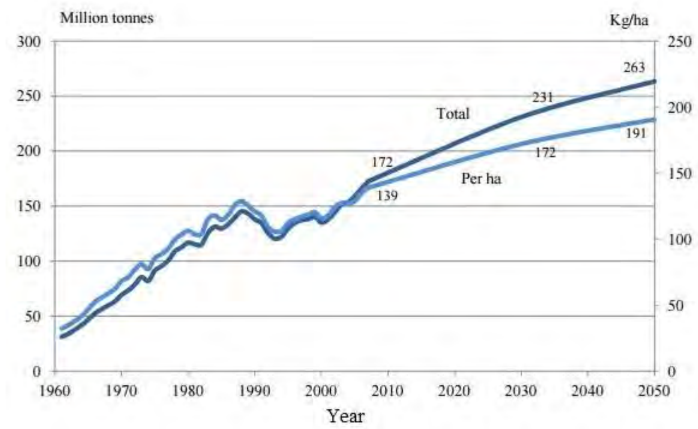

Figure 1.2: World Fertilizer Consumption: Historical and Projected (Alexandratos and Bruinsma, 2012)

Globally, the primary vital fertilizer nutrient is nitrogen and it is needed by all crops for healthy growth. Among the nitrogen fertilizer, the most widely used is urea due to its high nitrogen content, i.e. 46% by weight which has surpassed other nitrogen fertilizer. The urea fertilizer can be used for all types of crops such as rice, corn and wheat and is applied to soil in its solid or solution form. However, various environmental and economic disadvantages associated with the overuse of fertilizer have become a focus of worldwide concern. According to Ibrahim et al. (2014), approximately 70% of the applied fertilizer may be lost into the environment through surface runoff, leaching, and volatilization. Consequently, the utilization efficiency of plant uptake is generally 50% or less because only a soluble fraction of nutrients can be taken up by plants.

Hence, the proper use of fertilizer is essential to achieve crop yield and quality, environmental stewardship and profit goals. The efforts have been made by researchers to counteract the environmental pollution with the development of advanced technology on the fertilizers application. The improvement of fertilizers can be achieved through appropriate product design with the chemical and physical properties, environmental safety and stability against mechanical strength. The slow release fertilizer (SRF) arises as a new generation environmental-friendly fertilizer which delivers nutrients to plant over a defined and extended period of time (Zhong et al., 2013; Campos et al., 2014). The trend toward increasing consumption of SRF is expected to continue in the future, for example of China as shown in Figure 1.3. The conventional fertilizers can be designed to SRF by giving a protective encapsulating matrix. In essence, the encapsulating matrix comprises a plastic shell to encapsulate the fertilizer. When the plastic shell degrades slowly over time, the chemical nutrients inside the fertilizer are released into the soil over a longer period of time; therefore, the plant can utilize nutrients more fully. After the release of fertilizer, remaining matrix materials in the soil must be biodegrade to prevent accumulation of solid waste over time (Trenkel, 1997; Flemming et al., 2016).

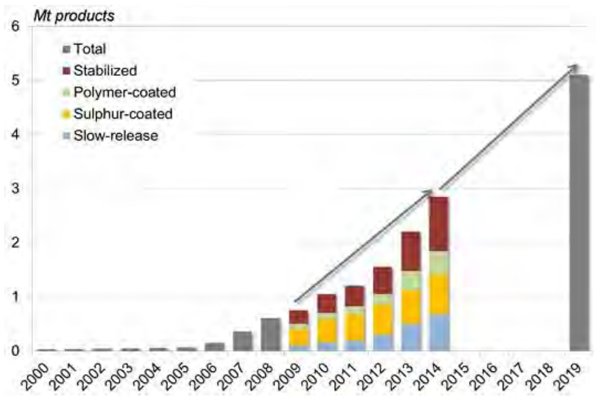

Figure 1.3: Slow Release and Stabilized Release Fertilizer Use in China (IHS, 2015)

Natural biopolymers are more favorable precursors for the preparation of fertilizer matrix in recent years as they are toxicologically harmless, bacteriologically degradable, renewable and abundant in nature (Hussain et al., 2012; Rychter et al., 2016). Among various types of natural biopolymers, starch has been shown to be a good precursor