UNIVERSITI PUTRA MALAYSIA

CONTROLLING UREA-N RELEASE USING WOOD WASTE MATERIALS

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By

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This research attempts to perceive on the practicability of wood waste materials from oil palm frond (OPF) and rubber wood (RW) chips as the carrier for slow release fertilizer (SRF). RW residue was chosen in order to expand the usage of wood materials in the development of SRF, which previously utilize solid wood in the wood chips preparation for SRF production. Whereas the utilization of OPF can be explained by the abundant supply and high availability of this material which can be beneficial for environment and agricultural future in replacing synthetic material usage. Thus, basic anatomical characteristics and their relation to physical properties of oil palm frond and rubber wood as potential supporting material for slow release woodchips fertilizer was investigated, including microscopic structure study, quantitative fiber morphology and physical properties of both materials. This study revealed that OPF is found as more porous structure as well as having ability to absorb more water which is very favorable for nutrient deposition and release property of SRF. On the contrary, RW has thick cell wall which could be one of beneficial properties required for SRF carrier in order to hold and retain the impregnated nutrient in a longer time, thus make RW as possible material to be used in the production of wood waste chips SRF. Owing to aforementioned characteristics, it is expected that OPF and RW have their own advantageous, ability to be used as supporting material for SRF production in which they will facilitate the further process which is impregnation of nutrient fertilizer.

The treatability of OPF and RW chips with urea was carried out using pressurized and non-pressurized impregnation processes, with three different levels of urea concentrations. Findings show that types of material, impregnation process and urea concentration did influence the treatability of oil palm frond and RW chips with urea solution. OPF was found to have higher weight percent gain (WPG) and

nitrogen (N) retention compared to RW, and impregnation using pressurized method attained higher retention compared to non-pressurized method. Treatment with 15% urea concentration using pressurized process was found to be the most efficient treatment combination in the development of wood waste SRF.

Further study on the nitrogen deposition into the impregnated woodchips fertilizer as well as the effects of cumulative nitrogen release pattern were also evaluated. The morphological changes of the impregnated woodchips was shown by VP-SEM, thus provide microscopic evidence of urea penetration into the microstructure of treated OPF and RW. By energy dispersive X-ray (EDX) analysis, the characteristic signals for N was detected clearly at 0.25 keV for OPF and 1.47 keV for RW. Release patterns of the nitrogen from impregnated woodchips were found slow and steady, particularly much slower in distilled water compared to soil solution. Other than that, the cumulative N release for OPF was found higher than RW in both leaching solution at the time interval of 768 h. Based on the results, the release pattern of both woodchips were similar to the conventional SRF, hence it is suggested that urea-impregnated woodchips fertilizer could function as a SRF that release nutrients gradually. Biodegradability test showed that treated chips decompose slower than untreated chips, whereas OPF chips have higher degradation rate than RW.

In order to evaluate the effectiveness of the woodchips SRF on cultivation of crops, a field trial was conducted. Comparative effects of woodchips SRF, common NPK fertilizer, commercialized SRF and control (blank) were investigated on the growth performance and yield productivity of lady finger. Throughout the cultivation period, none of the plants were found dead. The findings also show that plant treated with urea-impregnated OPF has better vegetative growth in term of leaves and stem diameter, whereas application of urea-impregnated RW contribute to give higher fruit weight of lady fingers than OPF but did not give big influence in yield productivity of crops. Besides, significantly greater plant dry mass and N content were obtained with application of woodchips SRF. Therefore, it can be concluded that the urea-N release for slow release fertilizer can be controlled by utilization of wood waste materials from OPF and RW chips, which equally effective with the commercialized SRF in providing sufficient nutrients needed by the plant with less frequency of application than common compound fertilizer.

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

MENGAWAL PELEPASAN UREA-N MENGGUNAKAN BAHAN SISA KAYU

Oleh

NUR NABILAH ABD. KHALID

Disember 2016

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Kajian ini bertujuan untuk mengkaji kesesuaian bahan-bahan sisa dan serpihan dari kayu getah (RW) dan pelepah kelapa sawit (OPF) sebagai pembawa untuk baja berpelepasan perlahan (SRF). Sisa RW dipilih untuk memperkembangkan penggunaan bahan-bahan kayu dalam pembangunan SRF, yang sebelum ini menggunakan kayu pepejal dalam penyediaan cip kayu untuk pengeluaran SRF. Manakala penggunaan OPF pula adalah disebabkan oleh bekalan yang banyak dan ketersediaan tinggi bahan ini yang boleh memberi manfaat kepada alam sekitar dan masa depan dunia pertanian dalam menggantikan penggunaan bahan sintetik. Oleh itu, ciri-ciri asas anatomi dan hubungankait mereka dengan sifat-sifat fizikal pelepah kelapa sawit dan kayu getah sebagai bahan sokongan yang berpotensi untuk digunakan sebagai cip pembawa untuk baja berpelepasan perlahan telah dikaji, termasuklah kajian struktur mikroskopik, kuantitatif fiber morfologi dan ciri-ciri fizikal. Kajian ini mendedahkan bahawa OPF didapati sebagai struktur yang lebih poros serta mempunyai keupayaan untuk menyerap lebih banyak air yang mana ianya sangat baik untuk pemendapan dan pelepasan nutrien SRF. Sebaliknya, RW mempunyai dinding sel yang tebal yang boleh menjadi salah satu sifat berfaedah yang diperlukan cip pembawa SRF untuk memegang dan mengekalkan nutrien yang diimpregnasi dalam jangka masa yang lebih panjang, yang mana menjadikan RW sebagai bahan berpotensi dan boleh digunakan dalam pengeluaran cip sisa kayu SRF. Disebabkan ciri-ciri yang dinyatakan di atas, adalah dijangkakan bahawa OPF dan RW masing-masing mempunyai kelebihan dan manfaat tersendiri,yang berkeupayaan untuk digunakan sebagai bahan sokongan untuk pengeluaran SRF di mana mereka akan memudahkan proses selanjutnya iaitu memasukkan baja nutrient ke dalam serpihan kayu.

Kebolehrawatan cip daripada OPF dan RW dengan urea telah dijalankan dengan menggunakan proses impregnasi bertekanan dan bukan bertekanan, dengan tiga tahap kepekatan urea yang berbeza. Dapatan kajian menunjukkan jenis-jenis

bahan sisa kayu, kondisi proses impregnasi dan kepekatan urea yang digunakan mempengaruhi kebolehrawatan cip OPF dan RW dengan larutan urea. OPF didapati mempunyai lebih tinggi peratus berat serapan (WPG) dan pengekalan nitrogen (N) berbanding RW, dan impregnasi menggunakan kaedah bertekanan didapati mencapai pengekalan serapan lebih banyak berbanding dengan kaedah bukan tekanan. Rawatan dengan 15% kepekatan urea menggunakan proses bertekanan dilihat sebagai gabungan rawatan yang paling berkesan dalam pembangunan SRF sisa kayu.

Kajian lanjut pada pemendapan nitrogen ke dalam cip OPF dan RW yang diimpregnasikan telah dijalankan serta kesan corak pelepasan nitrogen terkumpul juga telah dinilai. Perubahan morfologi daripada cip yang telah diimpregnasikan ditunjukkan oleh VP-SEM, dimana ianya menyediakan bukti mikroskopik penembusan urea ke dalam mikrostruktur OPF dan RW yang telah dirawat. Selain itu, menerusi (EDX), isyarat bagi N dikesan dengan jelas pada 0.25 keV untuk OPF dan 1.47 keV untuk RW. Corak pelepasan nitrogen dari cip berimpregnasi didapati perlahan dan mantap, terutamanya lebih perlahan dalam air suling berbanding larutan tanah. Selain daripada itu, N terkumpul bagi OPF didapati lebih tinggi daripada RW dalam kedua-dua larutan larut lesap pada selang masa 768 jam. Berdasarkan keputusan, corak pembebasan kedua-dua cip adalah berkadaran dengan SRF yang konvensional, maka adalah dicadangkan bahawa cip berimpregnasi urea dapat berfungsi sebagai SRF yang melepaskan nutrien secara beransur-ansur. Ujian biodegradasi menunjukkan bahawa cip yang dirawat mengurai perlahan daripada cip yang tidak dirawat, manakala cip OPF mempunyai kadar degradasi lebih tinggi daripada RW.

Untuk menilai keberkesanan cip berimpregnasi urea terhadap penanaman tanaman, percubaan lapangan telah dijalankan. Kesan perbandingan di antara cip berimpregnasi urea SRF, baja NPK biasa, SRF komersial dan cip kawalan (kosong) telah dikaji pada prestasi pertumbuhan dan hasil produktiviti bendi. Sepanjang tempoh penanaman, tiada sebarang pokok yang ditanam ditemui mati. Dapatan kajian menunjukkan bahawa tanaman bendi yang dirawat dengan cip OPF berimpregnasi mempunyai pertumbuhan vegetatif yang lebih baik dari segi diameter daun dan batang, manakala penggunaan cip RW berimpregnasi menyumbang kepada berat buah yang lebih tinggi bendi daripada OPF, namun tidak memberikan pengaruh yang besar dalam hasil produktiviti tanaman. Selain itu, jisim kering dan kandungan N yang lebih ketara tinggi diperolehi dengan penggunaan cip berimpregnasi SRF. Oleh itu, dapat disimpulkan bahawa pelepasan urea-N untuk baja berpelepasan perlahan boleh dikawal dengan penggunaan cip bahan-bahan buangan kayu dari OPF dan RW, yang sama berkesan dengan SRF komersial dalam membekalkan nutrien mencukupi yang diperlukan oleh tumbuhan dengan kekerapan pembajaan yang kurang berbanding aplikasi baja sebatian yang sama.

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I certify that a Thesis Examination Committee has met on 2 December 2016 to conduct the final examination of Nur Nabilah bte Abd. Khalid on her thesis entitled "Controlling Urea-N Release using Wood Waste Materials" 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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LIST OF ABBREVIATIONS

ANOVA	Analysis of Variance
ASTM	American Standard Testing Method
CEC	Cation Exchange Capacity
$CO (NH_2)_2$	Urea
DDSA	Dodecenyl succinic anhydride
EDX	Energy Dispersive X-ray
EFB	Empty Fruit Bunches
FB	Fibre Bundle
GP	Ground Parenchyma
LSD	Least Significant Difference
MC	Moisture Content
MF	Mesocarp Fibre
MS	Malaysian Standard
MSW	Municipal Solid Waste
N	Nitrogen
N_2O	Nitrous Oxide
NH_3	Ammonia
NH_4	Ammonium
NO_3	Nitrate
NPK	Nitrogen,Phosphorus,Potassium
OPF	Oil Palm Frond
OPT	Oil Palm Trunk
PKC	Palm Kernel Cake

PKS	Palm Kernel Shell
POME	Palm Oil Mill Effluent
RW	Rubber wood
SAS	Statistical Analysis Software
SPSS	Statistical Package for the Social Sciences
SRF	Slow Release Fertilizer
UPM	Universiti Putra Malaysia
VB	Vascular Bundle
VP-SEM	Variable Pressure Scanning Electron Microscopy
WA	Water Absorption
WPG	Weight Percent Gain

CHAPTER 1

INTRODUCTION

1.1 General Background

Malaysian Government has identified biomass as one of the strategic and potential economic drivers for the country. Biomass is expected to contribute an additional RM30 billion to the nation's Gross National Income through sustainable utilization of biomass by higher value-added downstream activities especially in the palm oil sector (MIDA, 2015).

Besides, biomass utilization is highly emphasized in the 10th Malaysian Plan mainly for energy production as Malaysia is widely recognized as one of the country that is rich with agro-biomass resources. The Government has also launched the National Biomass Strategy 2020 (NBS2020) in 2011, focusing on oil palm biomass and subsequently intensifying to other biomass from other sources in 2013 (MIA, 2013). Through NBS2020, it is believed to offer strategies on how Malaysia could maximize the potential of the country's biomass resources over a balanced portfolio of downstream industries.

Owing to high organic matter, moisture and other minerals, this biomass bear a huge potential to be converted as an alternative and beneficial invention for numerous applications (Siti Zulaiha, 2013). Bio-plastic, bio-composite, and bio-pellet are some examples of potential commercialized products that can be made from the renewable organic matter available in Malaysia including oil palm plantation waste, forest and mill residue, rice husk and straws, sugarcane bagasse, as well as municipal solid waste. Figure 1.1 illustrates the annual biomass availability in Malaysia.

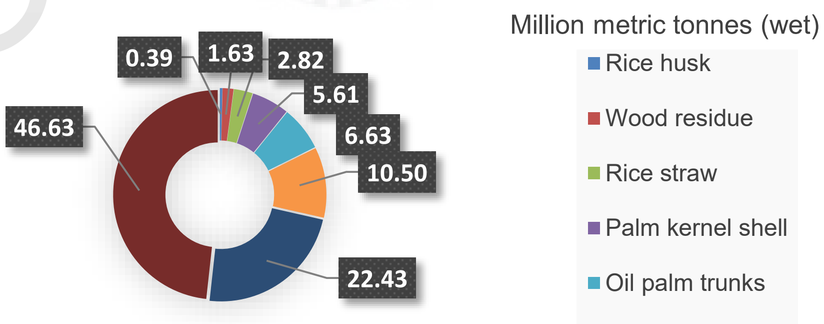

Figure 1.1: Total projected annual biomass availability in Malaysia (Tang, 2015)

Despite it wide use already predominantly in the application of co-firing ,syngas fuel, composite product and bioethanol (Roslan et al., 2011; Abdul Khalil et al., 2012; Griffin et al., 2014; Nor Afzanizam et al., 2014), there is still much more possibility to optimize the utilization of biomass resource at Malaysia.

Agricultural and wood waste is also believed to be expedient in the development of fertilizer system, particularly on organic as well as slow release fertilizer (SRF). In recent times, slow release fertilizer regime has been demanding by the agricultural consumer due to its benefits and eco-friendly characteristics. For the time being, SRF can be found in various forms including nugget, tablet, granular, stick and even sachet. With advantageous such as ability to minimize nutrient leaching losses, reduce the frequency of fertilizer application, lower fertilizer burn potential and improve the efficiency of nutrients uptake, SRF certainly become as one of the top choices for most of agricultural practitioners.

Although conventional fertilizers are mainly used because of fast acting properties, lower cost and easy to get, the usage of slow release fertilizer is much more efficient and also driven closer to best management practice (BMPs) implementation in which the nutrients are released into the soil slowly over time and become available to plants as they are needed.

Generally, the term SRF is used synonymously with delayed released, controlled release, metered release and slows acting to designate a rate of dissolution much less than is obtained by completely water soluble compounds. SRF allow the release of the active ingredient at a predetermined rate which extends its availability for plant uptake significantly longer in slower manner than conventional water-soluble fertilizer and provides adequate nutrients for crop production (AAPFCO, 1997; Haderlein et al., 2001; Ni et al., 2011).

However, the delay of initial availability or extended time of continued availability may occur by a variety of mechanisms. These involve controlled water solubility of the material by coatings, carriers, or other chemical forms, by slow hydrolysis of water-soluble low molecular weight compounds, microbial activity or by other unknown means (Trenkel, 2010).

For decades, an extensive variety of materials have been used so far to produce slow release fertilizer including conventional SRF made from poultry feathers by Choi and Nelson (1996); slow release molybdenum fertilizer by Bandyopadhyay et al. (2008); and SRF from natural attapulgite (APT) clay by Ni et al. (2010; 2011). Moreover, existing formulations have also been made on the development of granular urea-zeolite SRF by Hoeung et al. (2011); and starch-g-poly (vinyl acetate) SRF by Niu and Li (2012).

Thus, this research project attempts to observe the practicability of new kind of fertilizer that still owned the basic concept of slow release mechanisms similar to other SRF that have been studied or commercialized, yet with some added value, enhancement and green technology approach by using wood waste and agricultural biomass as carrier.

1.2 Problem Statement and Justification

Malaysia is highly dependent on external sources of chemical fertilizers which exposed to global price volatility and harmful effects to the environment. Thus, production of inexpensive and eco-friendly fertilizer to substitute the existing chemical fertilizers is required and need more attention. Development of SRF seems to be as an ideal and beneficial invention to promote green technology approach and best management practice (BMP) for agriculture world.

SRF plays an important role in improving fertilizer use efficiency by plants and also reducing the frequency of fertilization, hence alleviating environmental pollution and leading to the expansion of sustainable agriculture. At present, the development of SRFs is concentrating mainly on attaining of system in which a fertilizer granule is encapsulated, i.e., it is coated with an inert layer (Lubkowski and Grzmil, 2007; Basu and Kumar, 2008). However, the use of coating materials seems to be a crucial constraint that may result in a high production cost as well as soil contamination after their release into soil (Song et al., 2003).

A promising solution to these problems is by looking for possibility of using waste materials to produce new uncoated, biodegradable SRF. Fertilizers prepared by using clay polyester and plastic-starch as cementing agents are some examples of uncoated SRF recently developed by researcher in mixing common urea with industrial organic wastes and controlled-release inorganic materials. In another study, inorganic compound fertilizers was also mixed with N-rich and high quality organic fertilizer material (Wang et al., 2005). Nonetheless, formulation of SRF specifically from plant biomass is still lacking and need further exploration.

In this context, Malaysia have a plentiful tropical plant biomass i.e. oil palm fronds and rubber wood chips that can be used as carrier to hold the impregnated nutrient fertilizer (urea) of newly develop uncoated SRF. It has been projected that Malaysia will produce between 80 to 100 million dry tonnes of biomass annually from 2011 till 2020 (MIDA, 2015). Hence, Malaysia is capable to transform this waste into wealth. Furthermore, this will consequently help agriculturist to overcome their problems on how to get rid with the discarded biomass because they are relatively difficult to dispose and often left or burned at the site that will create more serious environmental concerns.

Moreover, a few number of prior studies have also been carried out on the formulation of SRF using wood as core matrix or carrier. For instance, Kottegoda et al. (2011) have developed urea-modified hydroxyapatite nanoparticles that were encapsulated under pressure into soft wood of *Gliricidia sepium*. While Ahmed et al.(2011) have formulated ammonium nitrate-impregnated wood chips SRF using three types of solid wood which are Japanese red pine (*Pinus densiflora*), eunsasi poplar (*Populus tomentiglandulosa*), and konara oak (*Quercus serrata*).

Upon this situation, the usage of wood materials for SRF development can be improved. Despite using solid wood which generally more expensive in unit cost, utilization of agricultural biomass seen to be relatively suitable and can be a great alternative sources in the preparation of SRF. This invention of SRF with a bi-functional will provide both carbon sequestration and soil amending properties.

After all, the formulation of SRF specifically on wood waste is still new and need to be explored. Nevertheless, it is necessary to develop and design a new kind of biodegradable, recyclable and cheap carriers that can be used as fertilizer to promote higher yield, environmental friendly and subsequently can increase profitability for agricultural business.

1.3 Objectives

The general objective of this research is to perceive on the feasibility of wood waste materials from oil palm fronds and rubber wood as carrier for slow release woodchips fertilizer. The specific objectives aim:

i) To determine the anatomical characteristics and physical properties of oil palm frond and rubber wood chips as potential carrier for slow release woodchips fertilizer.
ii) To examine the treatability of oil palm frond and rubber wood chips with urea solution using pressure and non-pressure impregnation process.
iii) To determine the deposition, release pattern of nitrogen and biodegradability of urea-treated chips.
iv) To evaluate the effectiveness of urea-impregnated woodchips slow release fertilizer on field trials of okra.

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