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DEVELOPMENT OF SLOW RELEASE FERTILIZER FROM OIL PALM EMPTY FRUIT BUNCH BIOPOLYMER COMPOSITES

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By

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

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Chairman : Khalina Abdan, PhD

Institute : Tropical Forestry and Forest Products

The use of fertilizers in agricultural sector has increased drastically along with the increasing food demand and world population. The high volume usage of conventional fertilizers is still rampant and it has brought a negative effect such as underground water pollution, environmental pollution, and health issues as well. At present, the utilization of chemical fertilizer in conventional fertilizer (broadcast system) such as nitrogen, phosphorus and potassium (NPK) has need 2.5 metric ton a year for an oil palm nursery. With application of slow released fertilizer (SRF) prepared using coating method, the usage has reduced to 1.8 metric ton per year. Furthermore, in order to extend the duration of SRF degradation release time, reduce the fertilizer cycle application. The industry seeks for new method of compounding to optimise the degradation and release rate.

This study attempts to use twin screw extrusion method in compounding SRF that contains NPK fertilizer, biopolymer as well as the empty fruit bunch (EFB) fibre. EFB to improve the compatibility between biopolymer and NPK to produce good bonding of SRF composites beside it function as micro nutrient to the soil. The degradability of SRF composites is depending on the mechanism the NPK and EFB fibre in the twin screw barrel. It is anticipated that encapsulated at the outer layer of the mixture (EFB and NPK). Therefore, the encapsulate layer will be functional as slow released agent. The optimum processing temperature for all polymer used i.e. poly (lactic acid) PLA was 145-150°C, poly (butylene succinate) PBS was 120-145°C, and poly (hydroxybutyrate-co-valerate) PHBv was 150-180°C. The speed of counter rotating twin screw extruder were set at 50 rpm and the feeder screw for NPK fertilizer, polymer and oil palm fibre was running at speed of 20 rpm. In this study, the formulation of BpF composites used comprises of polymer/NPK fertilizer (40/60%) and polymer/empty fruit bunch (EFB) fibres/NPK fertilizer (30/10/60%) ratio compounding. Scanning electron microscopy SEM was used to study dispersion of the EFB and fertilizer in the polymer. The morphological results showed that EFB and NPK distribution is well dispersed besides showed good bonding with the polymers.

To ensure the effectiveness of the slow-release bioplastic fertilizer composite to be used in oil palm nursery, a biodegradation test was conducted. This test spanned over 24 weeks. Based on the results, the new invention of SRF composite namely as Biopolymer Fertilizer, (BpF) composite showed slower rate degradation rate as compared to pure NPK fertilizer. However BpF shown higher rate in degradation as compared to pure polymer mixed with NPK only. At early stage, of week 8, BpF composites experienced 40% of degradation for all formulation used. Continuous degradation until week 16 showed that BpF with the combination of PHBv/ EFB/NPK reached 100% of degradation. BpF made of PBS/EFB/NPK and PLA/EFB/NPK composite formulations have reached 80% degradation in week 24. BpF composites were further tested in leachate column in order to determine the reaction of BpF with water. From the study, all BpF composites have showed the nitrogen release between 15% and 40% at week 8 to 12. This elucidated the presence of reaction of the fertilizer in the composite systems.

Nursery field test for 4-month old of oil palm seedlings was conducted. The results showed that the development and growth of the seedlings were excellent in terms of plant growth such as diameter, height, the number of fronds, and chlorophyll content. The growth of the seedlings applied with BpF was increased every week as compared to non BpF. The diameter and height with BpF composite is increased at the rate between 20% and 30%. The chlorophyll content increased around 20% with BpF composite.

In conclusion, BpF composite has the potential as slow-release fertilizer in oil palm seedling nursery and thus reduce the relying of pure chemical fertilizer.

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

PEMBANGUNAN BAJA PERLEPASAN PERLAHAN DARI KOMPOSIT BIOPOLIMER TANDAN KOSONG SAWIT

Oleh

HARMAEN AHMAD SAFFIAN

Ogos 2016

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Penggunaan baja bagi keperluan sektor pertanian meningkat secara drastik selari dengan keperluan makanan dan pertambahan penduduk dunia. Penggunaan baja konvensional berterusan hingga kini dan mendatangkan kesan yang kurang baik dengan menyebabkan pencemaran air bawah tanah serta menjejaskan alam sekitar dan kesihatan manusia. Pada masa ini penggunaan baja kimia konvensional (sistem tabur) seperti baja sebatian nitrogen, fosforus dan potasium (NPK) telah bertambah kepada 2.5 metrik tan setahun di nursery kelapa sawit. Penggunaan baja pelepasan pelahan (SRF) yang diperbuat secara kaedah penyalutan, penggunaannya telah menurun kepada 1.8 metrik tan setahun. Tambahan lagi untuk menambah durasi masa pelepasan dan degradasi SRF dan mengurangkan kitaran penggunaan baja, pihak industri perlu mencari kaedah baru mengadun baja untuk mengoptimumkan kadar pelepasan dan degradasi tersebut.

Kajian ini mencuba untuk menggunakan kaedah penyemperitan skru berkembar dalam mengadunkan SRF yang mengandungi baja sebatian NPK, biopolimer, dan juga gentian tandan kosong sawit (EFB). EFB telah ditambah untuk meningkatkan keserasian antara baja sebatian dan polimer komposit pelepasan pelahan (SRF), selain ia berfungsi menyediakan mikro nutrisi kepada tanah juga. Kebolehleraian komposit SRF bergantung kepada mekanisme pencampuran baja NPK dan gentian sawit dalam skru berkembar. Adalah dijangkakan biopolimer akan menyeliputi pada lapisan luar bagi pencampuran (EFB dan NPK). Oleh itu lapisan yang menyeliputi akan berfungsi sebagai agen perlepasan pelahan. Suhu optimum pemprosesan bagi polimer asid polilaktik (PLA) ialah 145-150°C, polibutilena suksinat (PBS) ialah 120-145°C, dan polihidroksibutirat valerat (PHBv) ialah 150-180°C. Mesin ini digerakkan dengan kelajuan 50 rpm dan penyuap bahan baja komposit dengan kelajuan 20 rpm.

Dalam kajian ini, satu formulasi baja plastik komposit telah diadun mengikut bahagian pecahan seperti polimer/NPK (40/60%) dan polimer/gentian tandan sawit kosong /NPK (30/10/60%) bahagian. Keputusan morpologi menunjukkan

gentian tandan sawit kosong dan NPK teragih dengan baik seterusnya menunjukkan perekat yang baik dengan biopolimer.

Dalam memastikan keberkesanan baja komposit bioplastik pelepasan perlahan, satu ujian biodegradasi telah dijalankan di tapak semaian anak sawit. Berdasarkan hasil kajian tersebut, inovasi baru bagi komposit SRF yang dinamakan sebagai Baja Komposit Biopolimer (BpF) menunjukkan kadar degradasi dalam tanah yang lebih perlahan berbanding dengan baja NPK yang digabungkan dengan gentian tandan sawit. Pada peringkat awal iaitu degradasi yang berterusan sehingga minggu 16 menyaksikan PHBv yang bergabung dengan NPK dan gentian tandan sawit telah mengalami degradasi sebanyak 100%. Baja komposit bioplastik yang lain pula mencapai 80% tahap degradasi pada minggu 24. Baja komposit bioplastik, satu kajian larut resap dalam turus dijalankan. Kaedah ini amat berkesan bagi melihat tindak balas baja komposit bioplastik di dalam air menggunakan Analisa Oto (AA) dan Spektrofotometer Penyerapan Atomik (AAS) bagi menentukan kadar pembebasan nitrogen, phospoforus, dan potassium dalam baja komposit bioplastik. Minggu 8 dan 12, semua baja biopolimer komposit menunjukkan pembebasan nitrogen sebanyak 15% hingga 40%.

Baja komposit bioplastik, dijalankan kajian di tapak semaian ke atas anak sawit berusia 4 bulan. Hasil kajian mendapati perkembangan dan pertumbuhan anak sawit amat baik dari segi ukur lilit, ketinggian, pertambahan pelepah dan kandungan klorofil. Bacaan ukur lilit perepang bagi semua anak sawit yang disertakan baja komposit bioplastik bertambah setiap minggu berbanding anak sawit tanpa baja. Ketinggian dan ukur lilit perepang juga bertambah dan berkembang dengan kadar 20% hingga 30% bagi anak sawit dengan baja komposit bioplastik. Kandungan klorofil meningkat pada minggu 4 hingga 12 dengan bacaan meningkat kepada 20%.

Kesimpulannya, baja komposit biopolimer mempunyai potensi sebagai baja pelepasan perlahan ke atas anak sawit di tapak semaian dan dapat mengurangkan penggunaan baja kimia.

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I certify that a Thesis Examination Committee has met on 16 August 2016 to conduct the final examination of Harmaen Ahmad Saffian on his thesis entitled "Development Of Slow Release Fertilizer From Oil Palm Empty Fruit Bunch Biopolymer Composites" 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

ASTM	American Standard Testing Materials
BASF	Germany Chemical Company
BpF	Bioplastic Fertilizer
CDU	Urea Acetaldehyde/Cyclo Diurea
CEC	Cation Exchange Capacity
CHN	Carbon,Hidrogen,Nitrogen
CRF	Controlled Release Fertilizers
DIN	German Institute for Standardization
DSC	Differential Scanning Calorimeter
DTG	Derivative Thermogravimetric
EFB	Empty Fruit Bunch
EPA	United States Environmental Protection Agency
FAO	Food Agriculture Organization
HDT	Heat Distortion Temperature
IBDU	Isobutyledene-Diurea
NPK	Nitrogen, Phosphorus, Potassium
MFI	Melt Flow Index
MPOB	Malaysian Palm Oil Berhad
Mt	Metric ton
OC	Organic Carbon
OM	Organic Matter
OPEFB	Oil Palm Empty Fruit Bunch
PBS	Poly (Butylene succinate)
PHBv	Poly (Hydroxybutyrate-co-valerate)
PLA	Poly (Lactic acid)

POME	Palm Oil Mill Effluent
SEM	Scanning Electron Microscope
SRF	Slow Release Fertilizer
TGA	Thermogravimetric Analysis
UF	Urea formaldehyde

CHAPTER 1

INTRODUCTON

1.1 Introduction

Higher dose of fertilizers is sometimes applied during the initial stages of plant growth for the purpose of reducing the frequency of application as well as labour costs. It also means that these traditionally used fertilizers are not efficient enough and thus needs to be improved to attain sustainable agricultural ecosystems. Fertilizers with enhanced efficiency are defined as fertilizers that possess certain environmental, economic, and agronomic advantages compared to the conventional fertilizers (Shaviv, 2000; Trenkel *et al*., 2010; Lu *et al*., 2013).

Most of the recent studies on slow release fertilizers are focused on nitrogenous fertilizers (Sanchez, 2000; Hartz, 2006). This study utilizes a new processing route to produce biodegradable slow release NPK fertilizers. The use of biodegradable polymers as coating materials to achieve better control over the nutrient release mechanism while the lower production cost (Shaviv & Mikkelsen, 1993). Ni *et al*. (2010) utilized lignocellulosic residues to produce slow release fertilizers. Slow release fertilizers are expected to replace the conventional fertilizers in the future.

Some factors that influence biodegradation of polymers are the polymer properties, types of microorganisms, and the pretreatment process. Some of the polymer properties include molecular weight, crystallinity, tacticity, mobility, the presence of functional groups and substituents within its structure, as well as the incorporation of additives or plasticizers (Gu *et al*., 2000b; Artham & Doble, 2008). The breaking down of organic substances by living organisms is called biodegradation process. This term is more common in the fields of waste management, environmental remediation, and ecology because they deal with recalcitrant waste materials. The biodegradation of organic materials may occur under aerobic or anaerobic condition. In aerobic biodegradation, the products are water and carbon dioxide, but in anaerobic biodegradation, the products are methane, water, and carbon dioxide (Gu *et al*., 2000a). The breaking down of polymer into monomers may involve one organism while the utilization of these monomers to produce simpler waste compounds may involve another and these excreted waste compounds may be used by yet another different microorganism.

The use of non-biodegradable polymers such as polyethylene in many industries is a global concern because it causes serious environmental problems related to waste disposal and global warming due to incineration of these polymers. Plastic pollution must be reduced for a better future for the next generation. Therefore, biodegradable polymers have been the focus of interest to replace their counterparts. However, more research is required to improve the weaknesses of biodegradable polymers such as inferior physical properties, expensive prices, as well as lower processing stability and flexibility.

Thus, biodegradable polymers have undergone ongoing modifications in order to incorporate them in everyday use (Pilla, 1999; Bastioli, 2005; Vroman & Tighzert, 2009).

1.2 Oil Palm Empty Fruit Bunch

Presently, oil palm empty fruit bunch (OPEFB) is used as a fuel for to generate energy for oil palm mills consumption. Due to high content of postassium, attempt has been carried out to convert EFB into fertilisers by burning them into ashes. However, the process in converting efb into energy and fertilizer has raised an environmental issue due to uncontrolled burning practices (Abdul Khalil *et al*. 2010d). One of the advantage characteristic of EFB is it can be naturally degraded by microorganism thus can be used to produce environmental friendly degradation material. Moreover, in last decades, many report were published on advantages of incooperating of EFB. Incompatibility can be improved by adding compatibilizers to bind the matrix with the fibres (Tserki *et al*., 2006b).

1.3 The Economic Perspective

Oil palm is one of the important crop in Malaysia. The total oil palm planted area in the country increased by 4.3% from 4,304,914 ha in 2007 to 4,487,957 ha in 2008 (MPOB, 2008a, MPOB 2008b). The output of these plantation contributed to about 45% of total palm oil production in the world. As the plantaion size increase, the use of fertilizer in oil palm plantation is also increases. The increase of use fertilizer caused an increase in operating cost for farmers and smallholder. To be profitable, cost need to be minimized while the yield need to be continuously maintained increased in the yearly basis.

The economic perspective of slow fertilizer usage in the nursery has been tabulated in Table 3.1. This table shows the highest cost of fertilizer is convensional fertilizer of 2.5 matric ton per ha per year compared to one type of SRF called AJIB. AJIB (SRF) has been applied in the nursery almost 1.8 metric ton per year. The AJIB contionted a total cost of RM 7560 per polybag a year. It is about 15% higher then conventional fertilizer. However, the application cycle a year is lesser.

Table 1.1: Application of different fertilizer and costing in oil palm nursery (Diversatech Fertilizer, 2015)

Desciption (Nursary)	Application cycle/year	Amount of fertilizer used (kg/ poly/ year)	Cost of fertilizer (RM/ Palm /year)	Total cost fertilizer (RM/poly/ year) [12,000 polybag palm/ha]	Total of fertilizer used (kg/ha/ year)	Total of fertilizer used (mt/ha/ year)
AJIB®(SRF)	2	0.15	0.63	7,560.00	1,800	1.8
NPK Blue	9	0.21	0.57	6,840.00	2,520	2.5

1.4 Statement of Problems

The main challenge in fertilizer industry today is to produce efficient and effective product which can maximize crop yield and productivity while able to maintain minimum nutrient losses to the environment. As the nutrients are released at a slower rate throughout the season, the nutrients supply can be sustained for a prolonged time, and consequently lower the labour cost by eliminating the need for labour associated with repeated fertilizer application.

Studies showed that good slow release coatings should have both hydrophobic and hydrophilic properties. The hydrophobic part strengthen the structure of coated film to prevent nutrient from being released too fast while the hydrophilic part allows the transport of nutrients via water flow. The nutrient release from slow release fertilizers can be synchronized with the stages of plant growth, increasing its efficiency. In fact, it may prevent nutrient overdose occurrence because slow release fertilizers can be designed to closely meet the nutrient demand by plants.

Furthermore, slow release fertilizers can reduce the risk of nutrient leaching from soil, which in turn reduces the time, labour, and cost for fertilizers application. Different concentration of nutrients in soil may affect the plant ability to effectively uptake other nutrients. Therefore, a systematic way that can regulate the application of these nutrients is needed so that the plants are able to effectively and efficiently utilize the fertilizers applied, resulting in lower cost and less waste.

The hydrophobic properties of Biopolymer will extend the nutrient from released rapidly in soil. However, the combination of NPK which is hydrophilic may increase the rate of release faster if not well balanced with the amount of biopolymer. This problem will be encountered by adding EFB fibre to bond tightly NPK with biopolymer and thus slower the degradation release time.

1.5 Scope of Study

In this study, attempt has been made to develop a new method in producing Bioplastic Fertilizer (BpF) from various biodegradable polymers such as poly (lactic acid), poly (butylene succinate) and poly (hydroxybutyrate-co-valerate). Bioplastic Fertilizer (BpF) composites can control the slow release fertilizer through degradation and diffusion in soil. Bioplastic is combined with oil palm empty fruit bunch fibre and NPK fertilizer (by weight w/w percentage ratio) to produce a new type of bioplastic fertilizer (BpF) composites using the extrusion method. Bioplastic fertilizer (BpF) underwent several testing such as soil burial test, leaching test, seedling growth performance, and scanning electron microscope. The performance of BpF composite produced in this study was compared with the NPK and existing SRF available in the market.

1.6 Research Objectives

The general research objective is to develop a new type of slow release in producing bioplastic fertilizer (BpF) composites from various biodegradable polymers and EFB fibres. The specific objectives are as follows;

A. To prepare and characterise bioplastic fertilizer composites.
B. To investigate degradation properties of bioplastic fertilizer composites.
C. To examine leachate and growth performance of bioplastic fertilizer composites.

1.7 Organization of the Thesis

The thesis is divided into seven chapters. The first chapter is the introduction. The second chapter provides a comprehensive review of literature on the topic biodegradable and biodegradation of polymer. The following chapters are about the production and application of slow release fertilizer made of biodegradable polymer reinforced with natural fibre composites. Chapter 3; Characterisation and biodegradation of bioplastic filled with oil palm fibres and fertilizer for bioplastic fertilizer (BpF) composites. Chapter 4; Biodegradation of bioplastic fertilizer (BpF) composites for slow release fertilizer. Chapter 5; Leaching of slow release bioplastic fertilizer (BpF) composites using soil column method. Chapter 6; Plant growth performance using BpF fertilizer composites: oil palm seedling in nursery study. Chapter 7; Conclusions and recommendations for future research.

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BIODATA OF STUDENT

Harmaen Ahmad Saffian was born on 22nd June 1962, in Manong, Kuala Kangsar, Perak. He completed his primary education at Sekolah Kebangsaan Manong and secondary education at Sekolah Menengah Sultan Tajul Ariffin, Manong. After completing his Sijil Pelajaran Malaysia on 1979, He was joined the Universiti Pertanian Malaysia in 1981 as a Laboratory Assistant at Faculty of Forestry until October 2005. He completed his Bachelor of Science (Forestry) on 2003. In Year 2004, He registered as Master's candidate in Biocomposites Technology at INTROP, UPM. He was promoted to Science Officer at The Institute of Tropical Bio-Composite (INTROB), UPM in 2005 until 2008. In August 2008, he was promoted to Research Officer at the Institute of Tropical Forestry and Forest Products (INTROP) until present. He has experienced in Wood Science and Technology, BioComposite, and Pulp & Paper area. He attended 6 months training in Wood Composites and Wood Chemistry (JICA programme) at Forestry and Forest Product Research Institute (FFPRI), Tsukuba, Japan in 1989. He also was attended training pulp and paper in Vienna, Austria 2007. In 2010, He attached research collaboration as visiting researcher to Agriculture University, Bogor (IPB), Indonesia, Kasetsart University, Bangkok,Thailand, in 2011 and Tehran University, Iran in 2013. In Year 2014, He was awarded Putra Grant about RM 149,000 for two years duration from Research Management Center (RMC), UPM and became a project leader for Bioplastic Fertilizer Composites study.

LIST OF PUBLICATIONS

Published papers

Harmaen Ahmad Saffian, Khalina Abdan, Mohd Ali Hassan, Nor Azowa Ibrahim and Mohammad Jawaid. Characterisation and biodegradation of poly(lactic acid) blended with oil palm biomass and fertiliser for bioplastic fertiliser composites. *BioResources* 11(1), 2055-2070 (2016). Impact Factor: 1.425 (Q1).

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