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## A STUDY ON THE PROPE

**INFORCED BIOPLASTIC** 

## MARTIN HII CHUN YEONG

Report submitted in partial fulfilment of the requirements for the award of Bachelor

(Honour) of Engineering Technology (Energy and Environment)

# FACULTY OF ENGINEERING TECHNOLOGY UNIVERSITI MALAYSIA PAHANG

## JANUARY 2017



#### SUPERVISOR'S DECLARATION

I hereby declare that I have checked this project report and in my opinion this project is satisfactory in term of scope and quality for the award of the degree of Bachelor (Honour) of Engineering Technology (Energy and Environment).

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#### ABSTRACT

About 408,000 metric tonnes of rice husk are produced annually yet are not utilise properly as small amount of rice husk is used for fertiliser and the rest become waste. This action leaks to waste disposal problem and high methane emissions. In agriculture, biodegradable plastics are desirable alternative to the current black low density poly(ethylene) plastic. Therefore, a research on bio-polybag is carried out with the objectives to utilize agricultural biomass for the production of biodegradable polybag and to investigate the properties of obtained polybag in terms of mechanical properties, surface structure, and biodegradability. The biomass studied in this research are rice husk due to it abundance in Malaysia, and polylactic acid is used to mix with the biomasses in different compositions (10%, 30%, 50%, 70% and 90%). Three main tests including tensile strength test, Scanning Election Microscopy (SEM) and accelerated aging test are taken place to study the properties. As a result, 30% rice husk: 70% PLA is the best among the ratio, in terms of lower tensile strength, higher elasticity, and shows bigger void and gap. 30% rice husk: 70% PLA has the tensile strength of 11.3 MPa, tensile modulus at 322.12MPa, elongation at break at 3.508%, decomposite rate of 1.413% and SEM test shows many gaps yet with maximum used of biomass material. Hence, 30% rice husk: 70% PLA is the most suitable biomass to use as biodegradable polybag.

#### ABSTRAK

Dengan agaran 408,000 tan metrik sekam padi dihasilkan setiap tahun namun ia tidak digunakan sepenuhnya sebagai sebahagian sekam padi digunakan untuk baja dan selebihnya akan dibuang. Tindakan ini telah menyebabkan masalah pelupusan dan pelepasan and penglepasan metana yang tinggi. Dalam bidang pertanian, plastik yang boleh lupus adalah alternatif wajar untuk plastik semasa hitam poli berketumpatan rendah (etilena). Oleh itu, penyelidikan bio-polibeg dijalankan dengan objektif untuk menggunakan sisa pertanian untuk pengeluaran polibeg yang boleh lupus dan untuk menyiasat sifat-sifat polibeg diperolehi dari segi sifat-sifat mekanikal, struktur permukaan, dan kebolehan lupus. Bahan sisa yang dikaji dalam kajian ini ialah sekam padi kerana ia banyak terdapat di Malaysia, dan asid polylactic digunakan untuk campuran dengan bahan sisa ini dalam komposisi yang berbeza (10%, 30%, 50%, 70% dan 90%). Tiga ujian utama termasuk ujian kekuatan tegangan, Mikroskop elektron pengimbas (SEM) dan ujian penuaan dipercepatkan diambil tempat untuk mengkaji sifat-sifatnya. Akibatnya, 30% sekam padi: 70% PLA adalah yang terbaik di kalangan nisbah, dari segi kekuatan tegangan yang lebih rendah, keanjalan yang lebih tinggi, dan menunjukkan tidak sah lebih besar dan jurang. 30% sekam padi: 70% PLA mempunyai kekuatan tegangan sebanyak 11.3 MPa, modulus tegangan pada 322.12MPa, pemanjangan pada waktu rehat di 3,508%, kadar decomposite daripada 1,413% dan ujian SEM menunjukkan banyak jurang namun dengan maksimum digunakan bahan sisa. Oleh itu, 30% sekam padi: 70% PLA adalah bahan sisa yang paling sesuai untuk digunakan sebagai polibeg biodegradable.

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## LIST OF SYMBOLS

%	Percent
&	And
°C	Degree Celsius
cm	Centimeter
g	Grams
L	Litle
М	Meter
μm	Micrometer
wt %	Weight percentage
W	Watt
g L M µm wt % W	Grams Litle Meter Micrometer Weight percentage Watt

## LIST OF ABBREVIATIONS

ASTM	American Section of the International
	Association for Testing Materials
PLA	Polylactic acid
SEM	Scanning electron microscopy

## LIST OF APPENDICES

Appendix A	Graph of Tensile test
Appendix B	Product of samples mixing at difference ratio
Appendix C	Scanning electron microscope (SEM)
Appendix D	Table data

#### **CHAPTER 1**

#### **INTRODUCTION**

#### **1.1 BACKGROUND OF STUDY**

Since year 1974 production of rice in Malaysia has increase from year to year as shows in Fig. 1.1. Rice industry in Malaysia is estimated product of total 408,000 metric tonnes of rice husk are produced yearly (Kadir and Ariffin, 2013). Usually small amount of rice husk is used for fertiliser and the rest become waste. This action leaks to waste disposal problem and high methane emissions (Chungsangunsit et al., 2009).



Fig. 1.1. Rice production according to year (Kadir and Ariffin, 2013)

Bio-polybag comes with the term of biodegradable polybag, at which it has the similar function as the polybag available on market, with the capability to decompose. According to the standard ASTM D-5488-94d and European Standard EN 13432-2000,

biodegradable refers to compounds that decompose into carbon dioxide, water or methane, inorganic compounds, and new cell biomass (Paola, 2015). ASTM is known as American Section of the International Association for Testing Materials. In the available market today, various products can be found in the market labelling to be biodegradable, but not all matches fully with the significant. According to the EN 13432, each significant organic constituent of a compostable packaging material must be biodegradable. "Significant" constituent is any constituent presenting a concentration higher than 1% of dry weight of that material. This stipulation implies that a concentration of polyethylene of maximum 1% is considered to be as an acceptable contamination of the biodegradable and compostable material, while a concentration higher than 1% would make the material not compliant with the EN 13432 (Müller, 2005).

#### **1.2 PROBLEM STATEMENT**

In Malaysia, polybags used in agriculture today are still non-biodegradable and inorganic, where plastic is the main component and it is bringing a negative impact to the environment (Jalil, 2013). As just one example, for every plantation of a tree, one non-degradable material will be used and this creates a waste that does not decompose (Jalil, 2013). It is very costly and tough to recycle used polybag. Hence, most of the polybag become waste and being dispose at landfill. Furthermore, polybag take about 300 years to photo degrade and will breaks down into tiny toxic particles. This break down is not environmental friendly as the toxic particles will contaminate the soil and waterways and enter the food chain when animals accidentally ingest them (Das et al., 2016). Duel to this fact, rice husk and polylactic acid (PLA) were mixed to produce as potential bio-polybag in this research and the mechanical properties, surface structure and biodegradability of the biopolybag were studied.

#### **1.3 OBJECTIVES**

There are two main objectives in this project:

1. To utilize the agricultural biomass for the production of biodegradable polybag.

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2. To investigate and evaluate the obtained polybag for surface structure, mechanical properties and biodegradability.

#### **1.4 SCOPE OF STUDY**

Based on the objectives of this project, the scopes of study are:

- 1. Research and utilization of the most usable agriculture biomass for the production of bio-polybag.
- 2. Evaluation of obtained polybag by using three different tests including tensile test, scanning electron microscopy (SEM), and accelerated aging test.

#### **1.5 SIGNFICANCE OF STUDY**

Polybag is a plastic that uses to seed plants. Generally, synthetic polybags from petroleum sources are greatly use which are non-renewable. Furthermore, the synthetic polybag ability to disintegrate in the environment is very low. Due to the increased environmental awareness around the world, the use of renewable sources to replace the depleting petroleum sources has proliferating day by day. Agricultural biomass is extensively exploring for the production of biopolybag.

In this context, rice husk was selected as the raw material to produce biopolybag in present research. Polylactic acid (PLA) which was a biodegradable polymer mixed with rice husk to produce biopolybag. To the best of our knowledge, this approach of using rice husk and PLA as the raw materials for biopolybag preparation, has never been reported. It is expected that rice husk is a potential agricultural biomass for the production of biodegradable polybag which is anticipated to bring much greater environmental and sustainable benefit. The morphology and mechanical properties of rice husk: PLA biopolybag will be investigated in this research as well.

Over the last 35 years, plastic is the waste component that has seen the most significant growth. Damages to the environment caused by petro polymers are significant. Hence, the development in these bio-based polybags helps in greenhouse gas balances and other environmental impacts over whole life cycles. It is intended that use of biodegradable materials will contribute to sustainability and reduction in

the environmental impact associated with disposal of oil-based polymers (Song et al., 2009).

#### CHAPTER 2

#### LITERATURE REVIEW

#### 2.1 RICE HUSK

Rice husk a major by-product of the rice milling industry, is one of the most commonly available lignocellulosic materials that can be converted to different types of fuels and chemical feedstocks through a variety of thermochemical conversion processes (Kelleh et al., 1997). Proper understanding of the physical and thermochemical properties of rice husk is necessary for the design of thermochemical conversion systems (Kelleh et al., 1997). The uses of rice husk are continually growing and now it can use as biomass to fuel and co-fuel power plants. Rice husk is the outermost layer of protection encasing a rice grain. It is a yellowish colour and has a convex shape. It is slightly larger than a grain of rice thus lengths up to 7mm are possible. Typical dimension is 4mm by 6mm. It is lightweight and having a ground bulk density of 340 kg/m<sup>3</sup> to 400 kg/m<sup>3</sup>. Rice husk is a potential material, which is amenable for value addition. The usage of rice husk either in its raw form or in ash form is many. Most of the husk from the milling is either burnt or dumped as waste in open fields and a small amount is used as fuel for boilers, electricity generation, bulking agents for composting of animal manure and etc (Gallega and Rubis, 2014).

As shows in Table 2.1, the chemical composition of rice husk is similar to that of many common organic fibers and it contains of cellulose 40-50 percent, lignin 25-30 percent, ash 15-20 percent and moisture 8-15 percent (Gallega and Rubis, 2014).

#### **Table 2.1.** Rice husk's properties

Properties	Unit
Dimension	4mm by 6mm
Bulk density	$340 \text{ kg/m}^3$ to $400 \text{ kg/m}^3$
Contains of cellulose	40-50 %
Contains of lignin	25-30 %
Contains of ash	15-20 %
Moisture contains	8-15 %

#### 2.2 EXISITNG POLYBAGS

Bio-polybag, which is also defined as biodegradable polybags and other green alternatives to plastic grocery and shopping bags are becoming more popular. Biodegradable bags can be decomposed by bacteria or other living organisms; at which they are designed to break down after a period of time. They pose less of a hazard to neighbourhood ecosystems and sometimes contribute less, long-lasting, plastic material to landfills (Sara, 2016). Normally, biodegradable polybags are often made from farmed products like corn starch, which in the right conditions and it will break down into elements like carbon dioxide, water and methane. Biodegradable polybags also best suited to composting and may contribute to methane emissions if sent to landfill. To meet international standards, bags need to compost within 12 weeks and fully biodegrade within 6 months.

Technologies have been developed to speed up the decaying process of poly bags such as polythene (or polyethylene) bags will decay in the environment naturally. Bags have to survive long storage, be strong enough to hold groceries and other items and yet, still be broken down quickly once exposed to wind, water, light, or another trigger to start the decomposition process. Different technologies hasten decay at different rates (Sara, 2016). Besides that, the breaking down of biodegradable polybag will as well depend on temperature and humidity. It goes slow in cold weather, and high humidity virtually stops the process. The technology involved determines how effectively and how quickly a bag will degrade (Sara, 2016). Fig. 2.1 shows biopolybag technology.





Almost half of the biodegradable polybags used corn starch incorporated into the plastic. The corn starch component will begin to decay when exposed to a microbe rich environment. This breaks the plastic into tiny pieces. When it works well, the bag is totally deconstructed. This does not always happen as advertised. When it does not work, a bag may just become perforated but maintain its basic shape (Sara, 2016). These bags are designed to decay with prolonged exposure to the ultra violet rays in sunlight. If they're buried in a landfill or find their way into sewers, the process won't work properly and most of the plastic will remain intact (Sara, 2016).

These bags are among the most economical to manufacture and the most effective to use. They are completely biodegradable. This happens in a two-step process. First, the bag is oxidized by prolonged exposure to the oxygen in the air. After that, the oxidized fragments convert into carbon dioxide, a harmless biomass and water. It's an elegant process. Somewhat similar to using starch to encourage nature to deconstruct poly bags, Aliphatic poly bags also rely on microbes to breakdown the bag into its constituent molecules. Although these bags are effective, they're also expensive to produce (Sara, 2016).

#### 2.3 EXISITING POLYBAGS

There are many types of polybag such as Anti-Static poly bag, Drawstring poly bag, Flat poly bag, Foodservice poly bag, Garment poly bag, Gusseted poly bag, Hang Hole poly bag, Header Pak poly bag, Minigrip White Block poly bag, Reclosable poly bag, Slide Seal poly bag, UV Protective Amber poly bag, White Block poly bag, Laddawn poly bag, Elkay Plastics poly bag. Fig. 2.2 shows the existing application of polybag.

a. Drawing Polybags	b. Clear Plastic Cookie Bags	c. Mil Reclosable Polybags
Source: http://www.millersupplyinc. com/drawstring-poly-bags	Source: http://www.millersupplyinc. com/foodservice-poly-bags	Source: http://www.millersupplyinc .com/reclosable-poly-bags
d. Bottom Gusset Bags	e. Garment Bags	f. Reclosable Minigrip
		RWIN
Source:	Source:	Source:
http://www.millersupplyinc.	http://www.millersupplyinc.	http://www.millersupplyinc
-com/gusseted-poly-bags	com/garment-poly-bags	.com/minigrip-white-block-
		poly-bags

Fig. 2.2. The existing polybag (a) Drawing Polybags (b) Clear Plastic Cookie Bags
(c) Mil Reclosable Polybags (d) Bottom Gusset Bags (e) Garment Bags
(f) Reclosable Minigrip

# 2.4 POLYMER USED IN MANUFACTURING BIODEGRADABLE PLASTIC

Polymer degradation takes place mostly through scission of the main chains or side-chains of polymer molecules, induced by their thermal activation, oxidation, photolysis, radiolysis, or hydrolysis. Some polymers undergo degradation in biological environments when living cells or microorganisms are present around the polymers. Such environments include soils, seas, rivers, and lakes on the earth as well as the body of human beings and animal (Ikada & Tsuji, 2000). General polymers used in manufacturing biodegradable plastic are divided into two types which are natural polymers and synthetic polymers. Table 2.2 shows the classification of biodegradable polymers.

Natural polymers		Synthetic polymers	
Sub-classification	Example	Sub-classification	Example
Plant origin		Aliphatic polyesters	
• Polysaccharides	Cellulose, Starch, Alginate	• Glycol and dicarbonic acid polycondensates	Poly(ethylene succinate), Poly(butylene terephthalate)
Animal origin		• Polylactides	Polyglycolide, Polylactides
Polysaccharides	Chitin (Chitosan), Hyaluronate	Polylactones	Poly(e- carpolactone)
• Proteins	Collagen (Gelatin), Albumin	• Miscellaneous	Poly(butylene terephthalate)
Microbe origin		Polyol	Poly(vinyl alcohol)
Polycarbonates	Poly(ester carbonate)	Polycarbonates	Poly(ester carbonate)
• Poly(3- hydroxyalkanoate)	Poly(3- hydroxyalkanoate)	Miscellaneous	Polyanhydrides, Poly(a- cyanoacrylate)s, Polyphosphazenes, Poly(orthoesters)
Polysaccharides	Hyaluronate		

Table 2.2. Cla	assification	of biodegra	dable polymers
----------------	--------------	-------------	----------------

#### 2.4.1 Polylactic Acid (PLA)

Polylactic acid (PLA) as shows at Fig 2.12 is an aliphatic polyester made up of lactic acid which is 2-hydroxy propionic acid building blocks. It is also a biodegradable and compostable thermoplastic derived from renewable plant sources, such as starch and sugar (Lim et al., 2008). Nowadays, PLA is most common bioplastic in use today. Historically, the uses of PLA have been mainly limited to biomedical areas due to its

bioabsorbable characteristics. Over the past decade, the discovery of new polymerization routes which allow the economical production of high molecular weight PLA, along with the elevated environmental awareness of the general public, have resulted in an expanded use of PLA for consumer goods and packaging applications (Lim et al., 2008). Bioplastics are expected to contribute more benefits to environmental protection because of it will reduce carbon dioxide emission and biodegradable. Because PLA is compostable and derived from renewable sources, it has been considered as one of the solutions to alleviate solid waste disposal problems and to lessen the dependence on petroleum-based plastics for packaging materials (Lim et al., 2008).

The conversion of lactic acid to high-molecular weight PLA is achieved by a solvent-free process and a novel distillation process (Lunt, 1998). The essential novelty of the process lies in the ability to go from lactic acid to a low-molecular-weight polylactic acid, followed by controlled depolymerisation to produce the cyclic dimer, commonly referred to as lactide. This lactide is maintained in the liquid form and purified by distillation. Catalytic ring opening of the lactide intermediate results in the production of PLAs with controlled molecular weights. The process is continuous with no necessity to separate the intermediate lactide (Lunt, 1998). Figure 2.3 shows the manufacturing routes to polylactic acids.

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Fig. 2.3. Manufacturing routes to polylactic acids

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#### 2.5 EXPERIMENTAL TEST AND EVALUATION

#### 2.5.1 Tensile Test

A tensile test is a fundamental mechanical test where a carefully prepared specimen is loaded in a very controlled manner while measuring the applied load and the elongation of the specimen over some distance. Tensile tests are used to determine the modulus of elasticity, elastic limit, elongation, proportional limit, reduction in area, tensile strength, yield point, yield strength and other tensile properties. Tensile test was carried out by a Universal Testing Machine and samples were clamped on it. Crosshead speed of 5 mm/min was used for tensile test (Then et al., 2013).

#### 2.5.2 Scanning Electron Microscopy

The scanning electron microscope (SEM) uses a focused beam of high-energy electrons to generate a variety of signals at the surface of solid specimens. The signals that derive from electron-sample interactions reveal information about the sample including external morphology (texture), chemical composition, and crystalline structure and orientation of materials making up the sample (Susan, 2016). The surface morphologies of OPMF and tensile fracture surfaces of PLA bio-polybag was recorded by SEM, MV2300, CamScan Electron Optics Ltd. The scanning electron micrographs were recorded at the magnification of 200–300x (Then et al., 2013).

#### 2.5.3 Accelerated Aging Test

Accelerated Aging test is the simulation of real-time aging of the material. Samples with the mass of 2 to 5 g will covered completely with organic soil (with the present of bacteria and microorganism) and placed in incubator for 10 days at 55°C. The mass of the samples will be measured every three days to compare with initial samples' mass. The change in mass will be the study of biodegradability. The organic soil must be ensured that also wet moisture condition.

#### **CHAPTER 3**

#### **RESEARCH METHODOLOGY**

## 3.1 MATERIALS

The materials used in the projects were rice husk and polylactic acid (PLA). Rice husk was collected from PLS Marketing (M) Sdn Bhd, a rice process factory at Sekinchan. Polylactic acid was purchased from Sigma-Aldrich (M) Sdn. Bhd. The specific gravity of the polylactic acid is 1.24 g/cm<sup>3</sup> (Noorulnajwa et al., 2015). Figure 3.1 shows materials used in this study.



Fig. 3.1. Materials used in this study (a) rice husk (b) polylactic acid (PLA)

#### 3.2 MATERIALS PREPARATION

As shows in Figure 3.2, before rice husk was used to mix with PLA, prewash was carried out in order to remove impurities from the materials. Rice husk was washed with distilled water and placed on aluminium foil to dry in oven (Binder

Incubator Model RI 115 Oven) at 110 °C. Through grinding method rice husk was grind into powder form of 150 µm by using blender model Philip Daily Collection Blender 400 W, 1.5 L. The grinded size of the material was identified through sieve analysis. Lastly, grinded rice husk was weighted and stored in sealed polyethylene bag according to weigh ratio as presented at Table 3.1.



Fig. 3.2. The process of rice husk preparation

#### 3.3 EXPERIMENTAL DESIGN

The design of this samples production and samples analysis was referred to Noorulnajwa et al. (2015) with little modification. Rice husk was mixed with PLA at difference weight ratio as shows at Table 3.1.

Code	Rice husk composition (wt %)	PLA (wt %)
10 rice husk / PLA	10	90
30 rice husk / PLA	30	70
50 rice husk / PLA	50	50
70 rice husk / PLA	70	30
90 rice husk / PLA	90	10

**Table 3.1:** Formulations of PLA, rice husk-composites

Hence, in total five samples will be produce. These samples were prepared at Forest Research Institute Malaysia (FRIM) with difference loading of PLA (10-90 wt %).



Fig. 3.3. Mixing and hot press processes

Fig. 3.3 shows the mixing and hot press processes. The samples were produced by adding PLA into the mixer (Figure 3.4 (a)) at the temperature of 180 °C for 2 minutes to melt the PLA. Once the PLA was melted, with rotor speed of 60 rpm and material was mixed according to ratio based on Table 3.1 and continue the mixing for 12 minutes. The whole mixing process will take 14 minutes. After the mixing process, the samples were allowed to cool down and by using grinding machine (Fig. 3.4 (b)) to grind it into smaller pieces.



Fig. 3.4. (a) Plastic mixing machine (b) Grinding machine

After mixing process, the samples were molded at Chemical laboratory at Faculty of Chemical & Natural Resources Engineering. The samples were produced from compression- molded. The machine (Lotus Scientific LS-22025 25 Ton Hot and Cold Molding Press) as shows in Fig. 3.5 and samples were preheated for three minutes then compress mixed samples for another five minutes at 185 °C follow by cooling under pressure for one minute at 45 °C.



Fig. 3.5. Hot & cold Molding Press machine

#### 3.4 CHARACTERIZATION OF BIOPOLYBAG

Three tests were performed for characteristic analyses of the mechanical and thermal properties of the samples. The tests consist of tensile test, scanning electron microscopy (SEM) and accelerated aging test.

#### 3.4.1 Tensile Test

Tensile test was carrying out to study the tensile properties which are very important in order to understand the characterization of the samples. Through obtaining results from this testing to have a better understanding on samples. The samples were cut into dumb-bell shapes based on ASTM 638 for this test. The dimension of the dumb-bell shapes is 2.5 cm x 11.5 cm. The machine used in this test is Universal Testing Machine as shows in Fig. 3.6.



Fig. 3.6. Universal Testing Machine for tensile test

#### 3.4.2 Scanning Electron Microscopy (SEM)

JOEL JSM- 7800F was used to record the surface morphologies of the tensile fracture surfaces of the sample. JOEL JSM were operated in 6400 scanning electron microscope at 15 kV. Scanning electron micrographs recorded all the images at the magnification of 60–1.0Kx. As shows in Fig. 3.6., 70% rice husk samples after tensile test were used for scanning electron microscope (SEM) as shown in Fig 3.7.



Fig. 3.7. Scanning electron microscope (SEM) test

#### 3.4.3 Accelerated Aging Test

As shows in Fig. 3.8, each samples about 2-5 g were weighed and recorded. Then the samples were covered with soil and placed in the beaker. The soil was mixed with cafeteria waste water which obtained from Kolej Kediaman 3 cafeteria. The samples in the beaker were placed in the incubator of the model Binder Incubator (115V) RI 115 with the temperature of 55 °C. This process was involving for 10 days with initial weight recorded as day 1 and followed by samples weight taken at day 4, day 7 and day 10.



Fig. 3.8. Sample mix with soil and wastewater (a) samples from left to right (50% rice husk, 30% rice husk and 10% rice husk. (b) Samples were placed in incubator for 10 days.



Fig. 3.9. Methodology Flow Chart of Prototype.

#### **CHAPTER 4**

#### **RESULTS AND DISCUSSION**

#### 4.1 MIXING RATIO OF SAMPLES

Based on Table 4.1, samples with the ratio of 70% rice husk: 30% polylactic acid (PLA) and 90% rice husk: 10% PLA are unable to mix well together. This is because the amount of melted PLA to mix and bind with rice husk is not enough. Hence the mixing of this two ratio are unsuccessful. This observation also seen in biocomposites of 20% Polybutylene succinate: 80% oil palm mesocarp fiber (Then et al., 2013).

Code	Rice husk (wt %)	PLA (wt %)	Mixable
10 rice husk / PLA	10	90	Yes
30 rice husk / PLA	30	70	Yes
50 rice husk / PLA	50	50	Yes
70 rice husk / PLA	70	30	No
90 rice husk / PLA	90	10	No

**Table 4.1:** Formulations of PLA, rice husk-composites mixing

#### 4.2 TENSILE TEST

Fig. 4.1 shows the graph of tensile test of rice husk: PLA biocomposite. From the graph of Fig 4.1 (a), as the weight percentage of rice husk increase, tensile strength decrease. 10% and 30% rice husk shows high tensile strength. This graph similar trend with the finding from Noorulnajwa et al (2015). This may due to dispersed phase loading increase and the effective cross sectional area of continuous phase is reduced, subsequently resulting in a decrease of tensile strength (Noorulnajwa et al, 2015). 50%

rice husk: 50% PLA, has shown weak tensile strength, this is because the sample does not have a smooth surface.





By referring to Fig. 4.1 (b), tensile modulus also has the same trend like tensile strength as shows at Fig. 4.1 (a) which the tensile modulus decrease when the weight percentage of rice husk increase. However, 10% rice husk has better modulus strength than 30% rice husk and 50% rice husk. Fig. 4.1 (c) is the graph of elongation at break of the three samples of rice husk PLA biocomposites. When compare the data with Noorulnajwa at al (2015)'s findings, the readings of the elongation at break have similar results of their finding.

Based on this three result, the obvious and direct selected of the best ratio to use as polybag is sample with 10 % rice husk mixed with 90 % PLA. Hence, with economic wise, 90 % PLA of total polybag contain is a wise choice. The objective will be use as minimum PLA as possible as utilise agriculture waste is the main focus. It is however, 50 % rice husk sample had weak strength that might not able to reach the required minimum strength of polybag. Therefore, based on tensile strength with the lease use of PLA, 30 % rice husk of sample is the most suitable choice to use as bio-polybag.

#### 4.3 SCANNING ELECTRON MICROSCOPY (SEM)

It projected that the sample was build up layer over layer as it become must more obvious by referring to figure 4.2. It presented the rough surface of rice husk and some gaps in the sample which will contribute to the reduce in strength of sample. The presence of gaps was evidences of the poor interfacial adhesion resulting from the lack of compatibility between hydrophilic rice husk and hydrophobic PLA. Poor interfacial adhesion can then acted as a stress concentration point upon exertion of external forces and consequently result in premature failure due to poor stress transfer from matrix to the rice husk (Then et al., 2013). Hence, this can prove that samples can more easily to degradable compare with without biomass mixing due to it has many small holes, voids and gaps and the presence of voids and gaps are evidences of the poor interfacial adhesion resulting from the lack of compatibility between hydrophilic rice husk and hydrophobic PLA.



Fig. 4.2: The SEM of 30 % rice husk: 70 % PLA at under magnification of  $100 \times$ 

#### 4.4 ACCELERATED AGING TEST

From on Fig. 4.3, it shows that on after going through the process of accelerated aging test for 10 days, there was decreased in weight samples. This shows that it is possible for the mixing of rice husk and PLA as potential Bio-polybag can decompose or in another word, is biodegradable. There were increased weight in samples on Day 4 and Day 7, it was suspected that the samples possible had moisture as the samples might uptake oxygen consequent wettability increase (Chiellini et al., 2006). Based on the lost weight percentage on Day 10, sample with the mixed ratio of 70% rice husk and 30% PLA has the highest percentage in weight lost. Due to the difference of weight between sample of 70% rice husk and sample of 50% rice husk, it can be the possible cause of having 70% rice husk sample had higher weight lost percentage. However, it is possible that sample of 70% rice husk: 30% PLA had a higher decompose rate compare to 10% and 50% of rice husk samples. The decomposition was expected to be increasing after day 10 if the test continued at the same condition. Based on results conducted, PLA biopolybag was proved that it can be biodegradable due to the decreasing weight of samples after ten days during accelerated aging test and it can more easily to degradable and shorten the durability performance of degradable. Because of the low brittle, it was suitable for using it to produce polybag.



Fig 4.3. Graph of accelerated aging test

Having to work with budget, the team prioritized which area of cost would receive the most funds needed. In term of the whole project execution, the costs will need to cover majorly for material purchasing, transportation, utility of laboratory's equipment (if any), etc. The team has been given funding from the Faculty of Engineering Technology in the amount of RM3,600 (RM1,200 for each person in the team). Appendix D4 shows the cost analysis. This section of report presents a comprehensive budget of materials purchased by the student, including the unit costs and total costs.

#### **CHAPTER 5**

#### **CONCLUSIONS AND RECOMMENDATIONS**

#### 5.1 CONCLUSIONS

In conclusion, rice husk mix with polylactic acid (PLA) can be a potential of biodegradable polybags. Mixing ratio 30wt% rice husk: 70wt% PLA is chosen as optimum ratio for biopolybag production which are justifications as follows:

- 1. Rice husk can be utilised for production of biodegradable polybag instate of being dump as waste.
- Through tensile test and accelerated aging test, 30% rice husk: 70% PLA has the tensile strength of 11.3 MPa, tensile modulus at 322.12 MPa, elongation at break at 3.508% and decomposite rate of 1.413%

3. SEM test shows many gaps yet with maximum used of biomass material.

#### 5.2 **RECOMMENDATIONS**

In today market, the uses and development of biodegradable bag is growing larger and popular. However, develop of biodegradable bag from biomass or industrial waste is still yet to be seen or heard of. Nevertheless, with the advance technology that available in this world, biodegradable bag manufacture from biomass or industrial waste such as rice husk has the potential to replace non- degradable polybag that are currently still use in agriculture industry. There are a few recommendations that can be improved in this project which are temperature can be elevated to accelerate the aging process in biopolybag; different composition of mixed samples can be tested in future research; and pinpoint and further finding more on the unseen costs and benefits of bio-poly bag product.

#### REFERENCES

- Chiellini, E., Corti, A., D'antone, S., Baciu, R. 2006. Oxo-biodegradable carbon backbone polymers–oxidative degradation of polyethylene under accelerated test conditions. *Polymer Degradation and Stability*, **91**(11), 2739-2747.
- Chungsangunsit, T., Shabbir, H. G. and Patumsawad, S. 2009. *Emission Assessment of Rice Husk Combustion for Power Production*. International Journal of Mechanical, Aerospace, Industrial, Mechatronic and Manufacturing Engineering 3:(5)
- Das, V., Kumar, P., Gupta, A., Dr. Ameta, N. K. 2016. Dune Sand Stabilization Using Plastic (Polybags) Waste as Admixture for the Design of Flexible Pavement in Construction of Roads. IOSR Journal of Mechanical and Civil Engineering (IOSR-JMCE), 13:(6.I)
- European Standard EN 13432, 2000. *Packaging requirements for packaging recoverable through composting and biodegradation test scheme ande valuation criteria for the final acceptance of packaging.*
- Gallega, R. Rubis, J. 2014. *Risk husk and raw cotton as alternative oil absorbents* (online). http://shodhganga.inflibnet.ac.in/bitstream/10603/5599/10/10\_chapter%202.pdf (2 May 2016).
- Ikada, Y., Tsuji, H. 2000. Biodegradable polyesters for medical and ecological applications. *Macromolecular rapid communications*, **21**(3), 117-132.
- Kadir, A. A., and Ariffin, N. M. 2013. *Effects of Utilizing Rice Husk in Fired Clay Brick*. International Journal of Zero Waste Generation 1:(1)
- Kelleh, M. Ghaly, A.E. 1997. Physical and thermochemical properties of rice husk. online). https://www.researchgate.net/publication/245326953\_Physical\_and \_Thermochemical\_Properties\_of\_Rice\_Husk (2 May 2016)
- L.T, Lim. Auras, R. Rubino, M. 2008. Processing Technologies for Poly(lactic acid).
- Lunt, J. 1998. Large-scale production, properties and commercial applications of polylactic acid polymers. *Polymer degradation and stability*, **59**(1), 145-152.
- Md. Abdul. Jalil, 2013. Using Plastic Bags and Its Damaging Impact on Environment and Agriculture: An Alternative Proposal.
- Müller, R.J. 2005. Biodegradability of polymers: Regulations and methods for testing. *Biopolymers Online*.
- Nations Encyclopedia, 2016. *Malaysia's Agriculture* (online). http://www.nationsencyclopedia.com/Asia-and-Oceania/Malaysia-AGRICULTURE.html (12 May 2016)

- Noorulnajwa, D. Y. Hanafi, I. Sam, S.T. 2015. Potential use of paddy straw as filler in poly lactic acid/paddy straw powder biocomposite: thermal and thermal properties.
- Paola, R. Marco, R. Stefania, P. Emanuele, F.M. Stefania, L.C. Concetto, P. Graziella, V. 2015. Determination of polyethylene in biodegradable polymer blends and in compostable carrier bags by Py-GC/MS and TGA.
- Sara, E. 2016. *Biodegradable poly bags* (online). http://greenliving.lovetoknow.com/biodegradable-poly-bags (1 May 2016).
- Song, J., Murphy, R., Narayan, R., Davies, G. 2009. Biodegradable and compostable alternatives to conventional plastics. *Philosophical Transactions of the Royal Society of London B: Biological Sciences*, **364**(1526), 2127-2139.
- Susan, S. 2016. *Scanning Electron Microscopy* (online). http://serc.carleton.edu/research\_education/geochemsheets/techniques/SEM.html (4 May 2016).
- Then, Y.Y., Ibrahim, N.A., Zainuddin, N., Ariffin, H., Wan Yunus, W.M.Z. 2013. *Oil* palm mesocarp fiber as new lignocellulosic material for fabrication of polymer/fiber biocomposites. International Journal of Polymer Science, **2013**.

#### **APPENDICES**

## Appendix A

Graph of Tensile test

(a) 10% Rice husk, 90% PLA

**(b)** 30% Rice husk, 70% PLA

(c) 50% Rice husk, 50% PLA



# Appendix **B**

## Product of samples mixing at difference ratio



# Appendix C Scanning electron microscope (SEM)

- (a) magnification of  $200 \times$
- (b) magnification of 500×
- (c) magnification of 1.0k×



## Appendix D

## Table data

Year	Rice production (tonnes)
1974	1,884,000
1982	2,231,000
2013	2,604,000
2014	2,645,000

## D1 Rice production according to year

# D2 Tensile strength, tensile modulus and elongation at break of rice husk PLA biocomposites

Rice husk (wt%)	10	30	50
Tensile Strength (MPa)	18.06	11.3	2.14
Tensile Modulus (MPa)	547.27	322.12	288.41
Elongation at break (%)	3.3	3.508	0.742

## D3 Accelerated aging test samples weight and percentage lost

Cada		Weig	ht (g)	
Code	Day 1	Day 4	Day 7	<b>Day 10</b>
10 rice husk / PLA	2.706	2.973	2.722	2.683
30 rice husk / PLA	3.891	4.075	3.895	3.836
50 rice husk / PLA	2.537	2.770	2.578	2.511

## D4 Cost Analysis

Category	Expense / RM	
Polylactic Acid (PLA) / 200 gram	RM 2,000.00	
Transportation	RM 300.00	
Laboratory's Equipment	RM 500.00	
Others	RM 200.00	
Total	RM3,000.00	