

# **UNIVERSITI PUTRA MALAYSIA**

# CHARACTERIZATION OF OIL PALM (Elaeis guineensis Jacq.) EMPTY FRUIT BUNCH FIBRE FILLED POLYBUTYLENE SUCCINATE AND TAPIOCA STARCH BIOCOMPOSITES

# **AYU RAFIQAH BINTI SHAFI**

**IPTPH 2021 8** 



# CHARACTERIZATION OF OIL PALM (*Elaeis guineensis* Jacq.) EMPTY FRUIT BUNCH FIBRE FILLED POLYBUTYLENE SUCCINATE AND TAPIOCA STARCH BIOCOMPOSITES



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

May 2021

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Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the degree of Doctor of Philosophy

### CHARACTERIZATION OF OIL PALM (*Elaeis guineensis* Jacq.) EMPTY FRUIT BUNCH FIBRE FILLED POLYBUTYLENE SUCCINATE AND TAPIOCA STARCH BIOCOMPOSITES

By

#### **AYU RAFIQAH BINTI SHAFI**

May 2021

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In Malaysia, the production of biodegradable food packaging is not well established. Biodegradable polymers only were used in high-end products such as in medical instruments. This is due to the high cost of the raw materials caused industries are not interested to invest in the development of biodegradable food packaging. The major challenges for the development of biodegradable polymer as food packaging are the shortcomings related to brittleness and processability. In meantime, this research was come out with an alternative to replace conventional plastic and overcome the drawbacks of the biodegradable polymer by substitute a certain percentage of polymer with tapioca starch and natural fibre from oil palm.

EFB fibre was added at 10 to 50wt% fibre loading. The percentage of water absorption of the samples increased maximum by 10.15% for 50wt% fibre loading and reached the equilibrium of absorption on day 8. The same trends were observed for water vapour permeability as the reading was increased as the EFB fibre loading increased. It was incredible to note that increasing the EFB fibre up to 20 wt.% and 30 wt.%, showed an increase in tensile strength from 14.27MPa to 16.16MPa and tensile modulus from 2965.81MPa to 3177.19MPa. The flexural strength was increased from 17.15MPa to 35.16MPa. However, the thermal stability of the composites decreased by 12.65°C as the fibre loading increased.

The composites with 20-30 wt.% of fibre loading exhibited good physical and mechanical properties, respectively. Increasing trends in the performance of tensile strength and flexural properties were shown for the glycerol loadings up to 10 wt% by 45% and 31.25% respectively. The addition of glycerol in the composite from 7.5-15% significantly improves the flexibility of the composite as the elongation at break increase from 6.12% to 14.21%. Besides, thermal stability for composite with 10.wt% glycerol

loading shows the highest thermal stability. This is due to better interfacial bonding and interaction between PBS, starch and fibre compounding. In rheological testing was showed the viscosity for all fibre loading in the composite was reduced as an increase in shear rate. The composites showed a shear-thinning behaviour thus, showed a non-Newtonian behaviour.

Due to calendaring machine capacity and limitation, 20 wt.% of EFB fibre loading and 10 wt.% glycerol content were chosen as a final formulation. Two technique of thermoforming process were evaluated to thermoform the composite into a food packaging tray. The hot press technique was showed higher tensile strength compare with the vacuum forming method. In terms of strength, this development of food packaging is competitive with the current food packaging. Hence, the development of fully biodegradable food packaging is important in the effort to address the ongoing environmental problems and gradually substitute the widely used conventional packaging materials.

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

### PENCIRIAN GENTIAN KELAPA SAWIT (*Elaeis guineensis* Jacq.) DIPENUHI POLY BUTYLENE SUCCINATE DAN KANJI UBI BIOKOMPOSIT

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Di Malaysia penghasilan bungkusan plastik yang bersifat biodegradasi tidak mapan. Polimer yang mempunyai sifat biodegradasi hanya digunakan untuk produk yang mahal seperti dalam peralatan perubatan. Hal ini disebabkan kos bahan mentah yang tinggi dan menyebabkan pihak industri tidak berminat untuk melabur dalam pembangunan pembungkusan makanan yang mempunyai sifat biodegradasi. Cabaran utama untuk pembangunan biodegradsi polimer sebagai pembungkusan makanan adalah kekurangan yang berkaitan dengan kerapuhan dan kebolehprosesan. Sementara itu, penyelidikan ini dibuat sebagai alternatif untuk menggantikan plastik konvensional dan mengatasi kekurangan polimer yang dapat terurai dengan menggantikan peratusan polimer tertentu dengan kanji ubi kayu dan gentian tandan kosong kelapa sawit.

Muatan gentian tandan kosong kelapa sawit ditambahkan pada muatan daripada 10 hingga 50wt% peratus berat. Peratus penyerapan air meningkat sebanyak 10.15% apabila muatan gentian tandan kosong sawit meningkat pada 50wt% muatan peratus berat dan mencapai keseimbangan penyerapan pada hari ke-8. Kecenderungan yang sama diperhatikan untuk kebolehtelapan wap air ketika bacaanya meningkat apabila muatan serat gentian tandan kosong sawit meningkat. Peningkatan serat gentian tandan kosong sawit untuk 20% berat dan 30% berat, menunjukkan peningkatan kekuatan tegangan dari 14.27MPa menjadi 16.16MPa dan modulus tegangan dari 2965.81MPa hingga 3177.19MPa. Kekuatan lenturan meningkat dari 17.15MPa menjadi 35.16MPa. Walau bagaimanapun, kestabilan terma komposit menurun sebanyak 12.65 °C dengan peningkatan beban serat gentian.

Komposit dengan 20-30% berat serat masing-masing menunjukkan sifat fizikal dan mekanikal yang baik. Peningkatan dalam prestasi kekuatan tegangan sebanyak 10% dan sifat lenturan sebanyak 31.25% telah dicerap untuk muatan gliserol sehingga 10% berat. Penambahan gliserol dalam komposit dari 7.5-15% secara telah meningkatkan

fleksibiliti komposit kerana pemanjangan meningkat dari 6.12% menjadi 14.21%. Selain itu, kestabilan terma untuk komposit dengan 10% berat gliserol menunjukkan kestabilan terma tertinggi. Ini disebabkan oleh ikatan kimia antara bahan dan interaksi yang lebih baik antara polimer, kanji dan gentian. Dalam pengujian reologi menunjukkan kelikatan untuk semua berat gentian pada komposit berkurang apabila peningkatan pada kadar ricih. Komposit menunjukkan tingkah laku penipisan ricih, menunjukkan sifat non-Newtonian.

Disebabkan kapasiti mesin yang dihadkan, 20% berat serat EFB dan 10% kandungan gliserol dipilih sebagai formulasi akhir. Dua teknik proses pembentukan terma dinilai untuk membentuk komposit ke dalam bungkusan makanan. Teknik tekan panas menunjukkan kekuatan tegangan yang lebih tinggi dibandingkan dengan kaedah pembentuk vakum. Dari segi kekuatan, penghasilan pembungkusan makanan ini kompetitif dengan pembungkusan makanan semasa. Penghasilan pembungkusan yang bersifat biodegradasi adalah penting dalam usaha menangani masalah kerosakan alam sekitar yang berleluasa dan ia dapat direalisasikan secara beransur-ansur dengan menggantikan bahan pembungkusan konventional yang digunakan secara meluas.

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1	$\eta = \tau \ / \ \gamma$	32
2	Density $(g/cm^3) = m/v$ (Where m represents mass and v for volume)	53
3	Moisture content (%) = $M_1$ - $M_0$ x 100 $M_0$	53
4	WA (%) = $(W_2 - W_1)/W_2 \times 100$	54
5	WVP = <u>(amount of permeant (g) /time (s)) x film thickness (mm)</u> Film area (m <sup>2</sup> ) pressure difference (kPa)	54
6	y=103.45x-0.3165 R <sup>2</sup> =0.9452	65

# LIST OF ABREVIATIONS

ASTM	American Society for Testing and Materials
EFB	Empty Fruit Bunch
$CO_2$	Carbon Dioxide
DTG	Derivative Thermo-gravimetry
FTIR	Fourier Transform Infrared Spectroscopy
G	Gram
HDPE	High Density Polyethylene
IR	Infrared
KF	Kenaf Fibre
MC	Moisture Content
OPEFB	Oil Palm Empty Fruit Bunch
ОН	Hydroxyl Group
PALF	Pineapple Leaf Fibre
PBS	Polybutylene Succinate
PLA	Polylactic Acid
PE	Polyethylene
PP	Polypropylene
PU	Polyurethene
PS	Polystyrene
PCL	Polycaprolactone
РНА	Polyhaydroxyalkanoates
SEM	Scanning Electron Microscopy
Tg	Glass Transition Temperature
TGA	Thermal-gravimetric Analysis
USA	United States America
WA	Water Absorption
WVP	Water Vapour Permeability
wt.%	Weight percentage

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# LIST OF UNIT

°C	Degree celcius
°C/min	Degree celcius per minute
0	Degree
GPa	Gigapascal
gsm	gram square meter
tex	gram per 1000 meters
g/cm <sup>3</sup>	gram per cubic centimeter
J/m	Joule per meter
kJ	kilo Joule
kN	kilo Newtons
Kg/m <sup>3</sup>	Kilogram per cubic meter
MPa	Megapascal
ms <sup>-1</sup>	meter per second
μ	micro
mm	millimeter
min	minute
τ	Shear stress
γ	Shear rate
%	Percentage
cm <sup>-1</sup>	per centimeter
rpm	rotation per minute

### CHAPTER 1

### **INTRODUCTION**

#### 1.1 Background

Plastics are chemically synthesized polymer via polymerization from petroleum products containing long chains of monomers. The plastics industry has existed over the last 60–70 years. Plastics have great properties such as versatility, low weight, durability, and low costs (Chidambarampadmavathy *et al.*, 2017). The growing of environmental burden and awareness is forcing the industries to pursue more eco-friendly materials for their products. There has been a significant focus within the scientific, industrial and environmental communities on the use of eco-friendly products with terms such as renewable, recyclable, sustainable and biodegradable (Boz, Korhonen & Koelsch, 2020).

Thermoplastic polyesters are produced from non-degradable fossil-based materials. Its properties lead to the accumulation of plastic waste and petrochemical feedstock degradation (Folino *et al.*, 2020). The growing of environment concern and awareness are derived industries from seeking more eco-friendly material. The thermoplastic polymer is a type of plastic polymer with the properties of a polymer resin. This material becomes soft when heated and can be moulded to any desired shape and scale. When it is cooled, it becomes hard and rigid and remains in the developed shape and size. Recently, biodegradable polymers have gained a great deal of interest due to their wide variety of applications in environmentally friendly goods. Polymers have begun to replace current materials in every aspect of life due to its properties. The growing issue of non-biodegradable plastics has increased the need to replace into biodegradable materials and optimize the use and disposal of traditional plastics.

A biodegradable polymer such as polybutylene succinate (PBS), polycaprolactone (PCL) and polylactic acid (PLA) was attracted tremendous attention due to their economic competitiveness. Besides that, the selection of polymer depends on the requirements for specific applications and performances. Biopolymer had been studied by researchers as a potential matrix for compostable and eco-friendly composite (Balart et al., 2020). Facilitate biodegradable material in composites, PBS is often used as a matrix. PBS is a biopolymer that has been extensively studied primarily due to its excellent mechanical properties and processing capabilities. PBS was polymerized from butanediol and succinic acid, which may soon both be available from bio-based renewable resources (Han et al., 2019; Su et al., 2019). PBS exhibits good storage modulus and flexural properties compared to other biodegradable polymers and naturally degraded in nature. The utilization of PBS can minimize environmental plastic load in a landfill (Mazhandu et al., 2020). PBS now had become one of the most promising polymers for both wide applications especially as an alternative in the packaging industry and also an idea to replace conventional synthetic polymers (Nakajima *et al.*, 2017). However, it has some drawbacks such as brittleness and low impact strength (Huang et al., 2018). As a result, the production of bio-based natural fibre composites has been established and were used in many application. The use of natural fibres materials able to reduce the cost of material and consequently modify the chosen properties as well as the rates of degradation. As far as the matter is concerned, when comparing the copolymerization and modification process with the blending of biodegradable polymers, the latter was found to be a much simpler method as well as the best approach to obtain the desired properties. (Meereboer *et al.*, 2020).

Natural fibre composites are simply planted fibres, embedded in a thermoplastic polymer. The density of these natural fibres is close to that of their plastic counterparts, which are typically 40% to 50% lower than the density of glass fibres. (Pecas et al., 2018). Polymeric materials may then be reinforced or filled without having a noticeable effect on their density. Several types of natural fibres, such as kenaf fibre, oil palm fibre, sugar palm fibre, pineapple leaf fibre, banana fibre, flax, hemp, sisal, coir and jute fibre, have attracted the attention of scientists and technologists for their widespread applications (Madhu et al., 2018). These fibres are used to reinforce thermoplastic polymer matrices such as polystyrene (PS), polypropylene (PP), polyethylene (PE), polyvinyl chloride (PVC) and polyurethane (PU). Due to its lightweight and eco-friendly properties, natural fibres also was chosen as based biocomposite are centre of attraction for industries over traditional composites (Mohammed et al., 2017). Oil palm plantation produces about 55 tons of dry matter in the form of fibrous biomass annually while yielding 5.5 tons of oil. The calorific value of oil palm residues in Malaysia varies between 18 to 21 MJ/kg on a dry basis. Lignocellulosic fibres of oil palm can be extracted from the trunk, frond, fruit mesocarp and empty fruit bunches (EFBs) of oil palm trees which has good mechanical properties (Hamzah et al., 2019). Therefore, the insertion of natural fibres in a polymer will enhance the properties of composite for future applications.

## 1.2 Problem Statement

The challenges in the food packaging industry today is to produce food packaging, which is environmentally friendly to avoid negative impacts on human wellbeing. Disposal of food packaging from synthetic plastics makes an additional contribution to the carbon dioxide emissions in the atmosphere, which contributes to global warming. In view of the numerous environmental problems posed by petroleum-based plastics, the government of many developed countries enacted policies that will help mitigate the current scenario (Sanyang *et al.*, 2018). In Malaysia, biodegradable materials especially in the food packaging industry are not well established. It costs 20 - 50% more to produce bio-plastic compare with synthetic plastic. Therefore, biodegradable polymers only were used in high-end products such as in medical instruments and pharmaceutical fields (Teixeira *et al.*, 2021). Due to the high cost of the raw materials caused industries are not interested to invest in the development of biodegradable food packaging. Besides that, biodegradable plastic needs costly industrial processors and composters, especially those that require high industrial-scale temperatures to be broken down. Apart from cost, there is a need for the availability of equipment, which may be a problem for the industry.



To overcome the dependence on petroleum-based polymers, attempts were made in this work to utilize 100% renewable and biodegradable materials. From a previous study, research was conducted to evaluate the blending of biodegradable polymer with starch or natural fibres by using extrusion, melt blending, solvent casting or injection moulding in lab-scale capacity (Nazrin *et al.*, 2020, Fahrngruber *et al.*, 2020). However, such a bio-sourced is still underutilized and thus, very limited studies have been reported related to their development as green packaging materials. Hence, in this research, natural fibres and starch were employed in the current study to develop fully biodegradable food packaging materials. In terms of processing, this research was upscale the material's capacity by using industrial extrusion and calendaring machine. This provides an excellent opportunity for researchers and industry as a reference for the future development of biodegradable food packaging. The positive results from previous research have encouraged this research to be done on broader scale for countering conventional polymer based plastic packaging.

In the previous research, polylactic acid (PLA) was a chosen biodegradable polymer that has a high potential to replace current synthetic food packaging. PLA is a commercially and environmentally interesting biopolymer and possesses unique characteristics, including rigidity, good transparency, good processability and glossy appearance (Siakeng *et al.*, 2018). However, PLA processing temperature is very high, which is 160°C and required a longer processing time. Other than that, PLA also has a serious limitation on its brittleness, poor toughness, slow degradation rate and poor thermal stability, which limits its extensive application (Nofar *et al.*, 2019).

In response to these problems associated with plastic waste, this research was used PBS as the main biodegradable polymer due to rarely research on these materials for commercial food packaging application. PBS melting point is 115°C which is lower compare with PLA. These properties could save on industrial processing while it takes a shorter time to melts and blends with other material. PBS was also easy to process and handling while mixing with other materials. In terms of brittleness, PBS is more rigid but slightly brittle due to its production from petroleum-based. PBS also has good thermal stability and excellent mechanical properties (Matos et al., 2020). However, blending PBS alone caused the PBS unable to extrude and reshape into food packaging due to less flexibility and brittleness. Therefore, it was suggested to compound with other materials to overcome this drawback. Tapioca starch is a readily available source of renewable energy and can be obtained from various by-products of cultivation and harvesting industrialization of raw materials. Starch is one of the most promising natural polymers because of its inherent biodegradability and abundance. However, the starch itself was brittle and difficult to process. Therefore mixing starch with PBS has the potential to overcome this drawback. Empty fruit bunch (EFB) fibre is a versatile lignocellulosic fibre that is abundant and was extracted from an oil palm tree. EFB fibre was used as reinforcement or filler for both thermoplastic and thermosetting polymer to combine become composite material. (Pecas et al., 2018).

In this research, PBS was filled with starch, EFB fibre and glycerol to produce a composite sheet for food packaging application. Proper mixing of PBS, starch, EFB fibre and glycerol able to develop biodegradable food packaging materials which have the potential to substitute current synthetic food packaging in the market. The key requirements of composites for food packaging materials are non-toxic or harmful, standard rigidity and stiffness with acceptable barrier properties. These can be achieved by an innovative formulation of a significant amount of weight. This bio-composite has potential application in the development of biodegradable food tray.

### 1.3 Research Hypothesis

From this research, the utilization of PBS, natural fibres, starch and glycerol composite would lead to the fabrication of bio-composites for biodegradable food tray application to resolve on issues of synthetic food packaging.

### 1.4 Research Objectives

Main objective: The aim for this research is to investigate the performance of EFB fibre filled polybutylene succinate and tapioca starch bio composite for potential on development of biodegradable food packaging.

### Specific Objectives

- 1. To determine the effect of starch filled polybutylene succinate on its chemical, thermal, physical, mechanical and morphological properties.
- 2. To investigate the effect of different loading Empty Fruit Bunch (EFB) fibre filled with PBS and starch on chemical, thermal, physical, mechanical and morphological properties.
- 3. To examine the effect of glycerol content on PBS/Starch/EFB Fibre bio composite on its chemical, thermal, physical, mechanical and morphological properties.
- 4. To determine the rheological behaviour of PBS/Starch/EFB Fibre bio composite.

### 1.5 Significance of Study

- 1. The development of biodegradable materials which expected to aids in addressing environmental issues regarding substitution of plastic based products.
- 2. Development of novel composites from PBS, EFB fibre and starch to produce food packaging tray.
- 3. This study also employs method processing to produce biodegradable food packaging tray

### 1.6 Scope and Limitation

In this study, pure starch that was purchased in Vietnam has properties easy to trap moisture and solidify when mixing with PBS at a certain temperature. Therefore, pure starch was excluded for mixing with PBS and unable to characterize properties of PBS and pure starch compound. PBS, starch and natural fibre are hydrophilic in nature. Therefore, the materials need to oven-dry before processing.

Second, pores on the composite sheet surface were detected, causing an oxygen transmission rate for investigating barrier properties unable to carry out. The presence of pores on the composite causing the reading of oxygen in and out are at the same rate. Therefore, in future work, it was suggested to laminate the composite to improve the barrier properties of the composite.

Third, fibre loading was set at a maximum limit to 20wt% due to easy processing and limitation of industrial extruder machine. This industrial extruder consisted of 4 feeders of different capacities. Feeder 1 is the main feeder that can fit materials up to 50%, feeder 2 up to 30%, feeder 3 and feeder 4 only can feed 20% of the total material. However, during producing composite sheet using an extruder, only two feeders were used which are feeder 1 for PBS and feeder 3 for a dry mix of starch/EFB fibre and glycerol due to feeder 2 is unable to operate. The composite sheet was developed by using an industrial calendaring machine. The characterize of the starch and EFB fibre as filler for PBS to improve the matrix properties. Thus, the effect of starch and fibre loading on the chemical, physical, mechanical and morphological properties of the composite was carried out.

### 1.7 Thesis Outline

This thesis is structured into five chapters. The first chapter contains an overview of biodegradable polymer and natural fibres, the significance of the research, highlights of the research problems, research hypothesis and finally, the objectives, scope, and limitations of the research. Chapter two is an overview of the literature on natural fibres filled composites by focusing on EFB fibre composites and biodegradable polymer. Chapter three presents the overall research methodology for the overall structure of the research work. This chapter describes the materials, a specific approach in designing and planning the experimental design, experimental test procedures and standards. The following chapter four presents the research findings and recommendations for future works.

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### LIST OF PUBLICATIONS

### **Journal Papers**

- Ayu, R. S., Khalina, A., Harmaen, A. S., Zaman, K., Jawaid, M., & Lee, C. H. (2018). Effect of modified tapioca starch on mechanical, thermal, and morphological properties of PBS blends for food packaging. *Polymers*, 10(11), 1187.
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