PHYSICAL AND MECHANICAL PROPERTIES OF CORN STARCH BLENDED WITH POLYPROPYLENE FOR FUTURE AUTOMOTIVE INTERIOR PARTS

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A report submitted
In fulfilment of the requirements for the degree of
Bachelor of Mechanical Engineering (BMCG)

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2019

DECLARATION

I declare that this project report entitled “Mechanical and morphological properties of corn starch blended with polypropylene for future automotive parts” is the result of my own work except as cited in the references.

Signature : ………………………………..
Name : Rahman Bin Latuwo
Date : ………………………………..

APPROVAL

I hereby declare that I have read this project report and in my opinion this project is sufficient in terms of scope and quality for the award of the degree of Bachelor of Mechanical Engineering.

Signature : ....

Supervisor's Name : Dr. Mohd Rody Bin Mohamad Zin

Date : ....

DEDICATION

To my beloved mother and father

ABSTRACT

Biodegradable that originated from renewable natural resources are currently being paid attention and focused as alternative to synthetic polymer. Therefore, numerous effort has been exerted to develop a natural polymer plastic. Among the natural polymer, starch is one of the interests. Starch inherently biodegradable that can be degraded by others living microorganisms. Unfortunately, there are some solid confinements for applications and creating starch-based polymer because of the absence of a water obstruction property and poor mechanical properties since starch is inherently brittle caused by high intermolecular forces. A variety of corn-starch/polypropylene (PP) compounds with different corn-starch content to improve their mechanical properties were processed by compression using motorise hydraulic moulding test press. The purpose of this study was to obtain the best and appropriate concentration of the corn-starch and PP on mechanical properties. Investigation on their characteristics and mechanical properties were done using universal testing machine instron-8872, durometer test, and electronic densimeter respectively. The addition of corn-starch content to PP reduced the tensile strength and the elongation, but the Young's modulus or stiffness of material increased. The presence of high starch content adversy affected the tensile strength of corn-starch/PP blends. However, the density increases with the addition of corn-starch on PP. For the shore hardness, it was found that the PP with 10 wt.% corn-starch content had the highest hardness.

ABSTRAK

Bahan biodegradasi yang diperbuat daripada sumber semula jadi yang boleh diperbaharui kini sedang diperhatikan dan diteliti sebagai alternatif kepada polimer sintetik. Oleh itu, pelbagai usaha telah dilakukan untuk membangunkan plastik polimer semulajadi. Antara polimer semulajadi yang menjadi perhatian adalah tepung kanji. Tepung kanji secara semula jadi boleh terbiodegradasi oleh tindakbalas mikroorganisma. Malangnya,tepung kanji ini mempunyai beberapa limitasi dan batasan untuk ianya dibangunkan dan digunakan. Ini kerana sifatnya yang tidak menghalang penyerapan air dan sifat mekanikal yang lemah kerana kanji secara semulajadi adalah rapuh disebabkan oleh daya intermolekul yang tinggi. Pelbagai sebatian kanji jagung / polipropilena (PP) dengan pelbagai kandungan kanji jagung untuk meningkatkan sifat mekanikal telah disediakan dengan menggunakan mesin acuan hidraulik motor secara kaedah mampatan. Tujuan kajian ini adalah untuk mendapatkan kandungan kanji jagung dan PP yang terbaik dan tepat pada sifat mekanikalnya. Kajian terhadap ciri-ciri bahan dan sifat mekanikal telah dijalankan dengan menggunakan mesin Ujian Universal Instron-8872, ujian durometer, dan densimeter elektronik. Penambahan kandungan kanji jagung terhadap PP dapat mengurangkan kekuatan tegangan dan pemanjangan serta meningkatkan modulus atau kekauan bahan. Kewujudan kandungan kanji yang tinggi mempunyai kesan buruk kepada kekuatan tegangan terhadap campuran kanji jagung / PP. Walau bagaimanapun, ketumpatan bertambah dengan penambahan kandungan kanji jagung pada PP. Untuk kekerasan, PP dengan 10% kandungan kanji jagung mempunyai kekerasan yang tertinggi.

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I also thank my friend for giving me advice. I am profoundly thankful to my parents and siblings for the moral support and encouragement I have received from them in completing this degree. Finally, thank you to everyone who had been to the crucial parts of realization of this project.

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LIST OF ABBREVIATIONS

CS	Corn Starch
PP	Polypropylene
PVA	Polyvinyl Alcohol
SEM	Scanning Electron Microscope
TS	Tensile Strength
EB	Elongation at Break
ASTM	American Society for Testing and Materials
LDPE	Low-Density Polyethylene
LDPE-g-MA	Low-Density Polyethylene Grafted Maleic Anhydride
MFI	Melt Flow Index
MEO	Mentha Pulegium
MCS	Methylated-Cornstarch
DS	Degree of Substitution
PBAT	Poly(butylene adipate-co-terephatalate)
PC	Polycarbonate
PS	Polystyrene
PVC	Polyvinylchloride
PP-g-MA	Polypropylene Grafted Maleic Anhydride
SG	Specific Gravity
RD	Relative Density

CHAPTER 1

INTRODUCTION

1.1 Background

Mechanical properties of a material including tensile strength, elongation at break and at yields, and elasticity or stiffness are characteristics which involve an application of the load reaction and are usually achieved by tensile testing. When the tensile test have been done stress-strain graph can be obtained by using the universal testing machine. American society for testing and materials were used as a standard reference when performing the test.

Due to the non-degradability and potential environmental risk of synthetic polymers many efforts were made to produce eco-friendly alternatives to plastic inventions. Starch is one of the options as a biodegradable polymer. Unfortunately, the mechanical properties of starch are poor. Due to the poor mechanical characteristics, starch and plastic were tested to improve mechanical properties. Therefore, corn-starch and polypropylene (PP) were blend together with a various content to determine the optimum corn-starch content in PP (up to 40%) that can produce good mechanical properties as regards of tensile strength, elongation at break, young's modulus, shore hardness and density.

1.2 Problem Statement

Various endeavours were made to create polymers based on starch in order to avoid wasted overuse of petrochemical resources, reduce the natural impact and searching for more applications. Regardless, wide applications have been compelled because of the lack of water barrier property and destitute mechanical properties, such as film fragility brought by high sub-atomic strengths. However, the incorporation of other additives may improve the mechanical properties of starch film. Therefore, the focus of this research is to blend corn-starch with polypropylene (PP) in order to discover the most excellent mechanical properties for future automotive parts.

1.3 Objective

The objective of this research is:

i. To determine the optimal ratio of corn-starch blended with polypropylene (PP).
ii. To investigate the effect of corn-starch additives at various polypropylene (PP) ratios on physical and mechanical properties of the resulting blends.

1.4 Scope

In order to achieve the objective, the scopes are arranged as shown below.

i. Polypropylene are blended with different volume percentage of corn-starch content that is 10%, 20%, 30%, and 40% and 100% polypropylene ratio.
ii. Testing film specimen by Tensile Strength (TS) and Elongation at break (EB) with American Society for Testing and Materials (ASTM D-638) as reference.
iii. Testing film specimen Hardness with ASTM D-2240 as reference.
iv. Testing film specimen density by using electronic densimeter.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

Over the past century, development in science and technology, amount of synthetic polymer has been grown rapidly for various industrial uses and applications. Synthetic polymer material has been produced widely every year. Around 140 million tons are produced annually from synthetic polymers (Katarzyna Leja*, 2010). The synthetic polymers are derived from petroleum oil, and are considered to be non-degradables. The use of synthetic polymer materials, causes severe environmental issues such as plastic waste as it is not biodegradable polymers. A non-biodegradable cannot be disposal by others living organisms and its take a thousand years to degrade. One of the alternatives to dispose those plastics is by burning them for energy production. However, this simply actions can cause air pollution which is produced carbon dioxide and other gases.

Therefore, there is a lot of research and effort focus on natural polymer to protect petroleum resources as an alternative to synthetic polymer, to reduce the environmental problem and find additional applications (Lu, Xiao and Xu, 2009). Starch in one of interest in the natural polymer. Starch as an alternative to petroleum resources has been considered as it is regenerated by photosynthesis in plants from carbon dioxide and water (Lu, Xiao and Xu, 2009). Starch inherently biodegradable that can be degraded by others living microorganisms such as bacteria, fungi and algae. They will also brake down the polymer chain through non-enzymatic approaches including chemical hydrolysis (Gross and Kalra, 2002). Moreover, it is a low costs, non-toxicity, abundance of renewable resources and high production (Li *et al.*, 2015). Unfortunately, due to the lack of a water barrier property and poor mechanical properties of starch caused by high intermolecular forces, there are certain restrictions on the use of starch-based polymers. Therefore, several attempts have been made to blend starch with other additive polymer to improve and enhance the mechanical properties such as starch blended with polyvinyl alcohol (PVA) that possesses excellent mechanical properties (Azahari, Othman and Ismail, 2011).

2.2 Starch

Starch is a natural polymer which occurs extensively in plants. It is produced throughout photosynthesis and functions as the principal polysaccharide reserve material. Pure starch is a white powder, tasteless and odourless that cannot be soluble in water without blood or alcohol. Potatoes, corn, and rice are the primary bodies used in production. In all the plants. In all of those plants, starch is deposited within the form of complicated structure that known as granules, with various shapes and size relying on the botanical origin (Chandra and Rustgi, 1998). In superior plant tissues, starch is synthesised through a chain of complex enzyme reactions by means of glucose-1-photosphate (Ai and Jane, 2017). In amyloplastic storage starch is made as discrete granules with separate morphologies for a single plant life ranging from spherical, oval, ogival, or elongated to flat, in diameter of sub-microns of over 100µm, lenticular or polyhedral (Bertoft, 2017). The characteristics of the gelatinisation temperatures, viscosity and clarity of pastas, gelling ability, and retrogradation rate (relating to syneresis), dependent on the botanical source.

2.2.1 Chemistry of Starch

Starch granules consist almost totally of major polysaccharides, specifically amylose and amylopectin. Both of these polymers are composed of repeating units of α-D-glucose. The major element of maximum starches is amylopectin and it is commonly made up 70% of the polysaccharide content. There are various methods used to separate the amylose from the amylopectin that precipitates the amylose as a 1-butanol complex, a process that Schoch developed in 1942. The structure of amylose and amylopectin are shown below in Figure 1.2 and Figure 1.3, respectively.

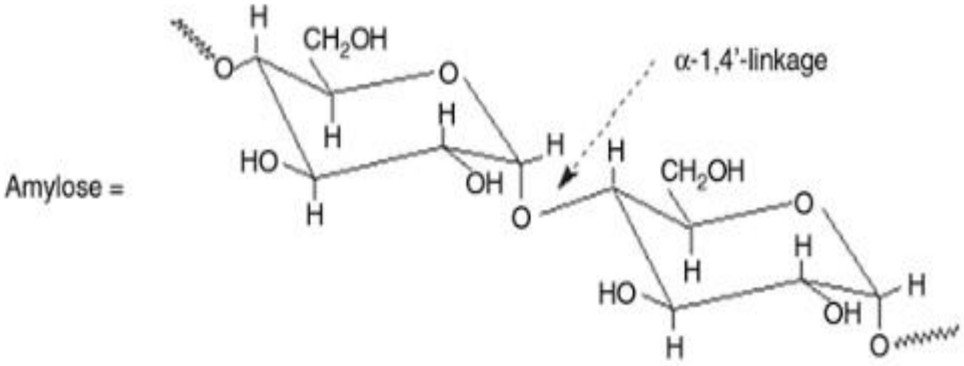


Figure 2.1 Structure of the amylose polymer.(Lu, Xiao and Xu, 2009)

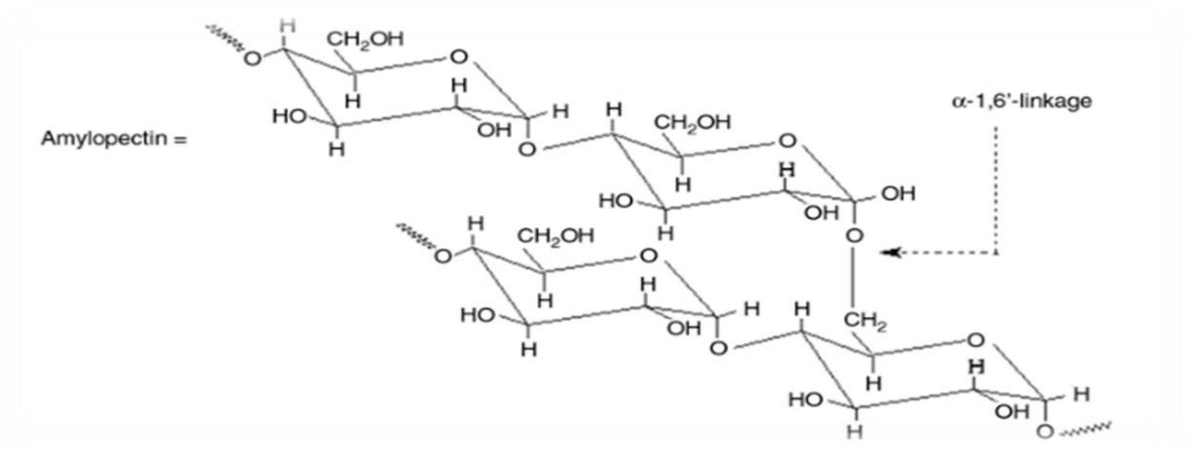


Figure 2.2 Structure of the amylopectin polymer. (Lu, Xiao, & Xu 2009)

2.2.1.1 Amylose

Amylose are a linear macromolecule containing glucose units with α-1,4 linkages. Each macromolecule has one lower end and one non-lower end. However, enzyme investigations have shown that the amylose molecule has a small degree of α-1,6-connections as with amylopectin. (Chen, 1990). The polymer strands of α-amylose in starch undertake a helical structure as shown in figure 2.3 similar that found in nucleic acids. Ordinary chain lengths for α-amylose units are about 1,000 monomer units. Amylose is very essential in plant energy storage where it is less easily digested than amylopectin, but due to its helical structure it takes up much less area compared to amylopectin. In the end, it is the recommended starch for plant storage. It accounts for approximately 30% of the starch stored in plants, but the percentage varies by species and variations (Wang *et al.*, 2017). Amylose content in starch are different with the different type of starch. For instance, corn starch is about 28% amylose and potato starch contains about 23% amylose. Common starches usually contain between 17 and 28% amylose and high amylose starches, which contain between 45 and 69% amylose (W. Banks, C. T. Greenwood, 1973).

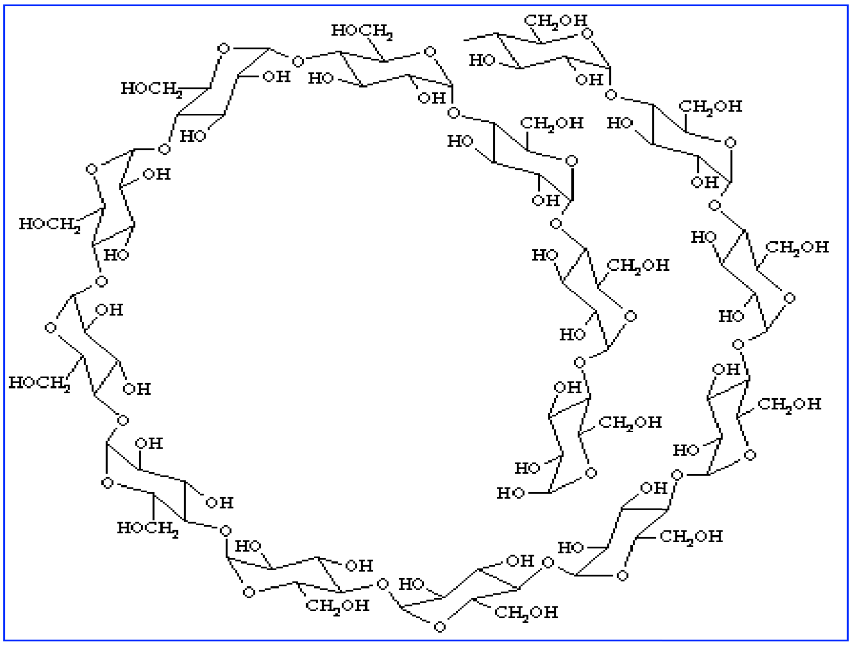


Figure 2.3 Helical Structure of the α- amylose molecule

2.2.1.2 Amylopectin

Amylopectin is a water-soluble polysaccharide that is a branched α-glucose unit component found in plants (Green, Blankenhorn and Hart, 1975). The amylopectin D-glucopyranpse is linked especially with α-1,4 bonds with 5-6 percent of α-1,6 bands at brans where the average duration of the branching chain is 20-25 units but the common duration of the chain of the high amylose corn amylopectin is higher than 30 units (C.T. Greenwood, 1966). This glucose unit is a soluble molecule that can become more rapidly degraded because there are numerous end points where enzymes can be attached. By contrast, amylose consists of just a few α-1,6 or perhaps none that produces amylose which is hydrolysed very slowly. It is however more dense and insoluble.

2.2.2 Starch Structure

The amylose and amylopectin starch molecule is composed of a glucose Monomer as a semi-crystalline polymer. Every starch molecules have two important functional groups, namely the -OH groups which have susceptible replacement reactions and high water associations which cause much of thermoplastic starch water absorption problem. Furthermore the chain vulnerable C-O-C bond. By its reaction-OH-group, changes in many properties can be made by mixing and keeping them untouched or melted and combined with the suitable molecular polymer (K. Oduola, 2015).

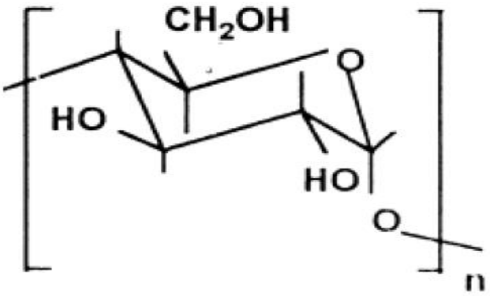


Figure 2.4 Starch Structure (K. Oduola, 2015)

2.2.3 Thermoplastic Starch

Thermoplastic starch is a plasticized starch that processes the crystalline starch structure to form an amorphous thermoplastic starch with heat and pressure. Starch plasticized is a starch mixed in with other plasticizers or additives that make processing possible. The processing of thermoplastic starch usually involves an irreversible transitional order-disorder known as gelatines. Disruption of molecular within the starch granules is called the gelatinisation of starch and this process is affected by the interactions between starch and water. Most processing of starch usually involves heating of water and several different additives, such as sugar and salt, to control gelatinisation in food industries and glycerol as a plasticizer for applications in biodegradable plastics (Halley *et al.*, 2007). Process of gelatinisation breaks up the granule structure of starch by disrupting the hydrogen bonding within adjacent glucose molecules and destroys its crystallinity (K. Oduola, 2015). Thermoplastic starch is a poor elongation and high tensile strength properties. It is due to the impact of the retro gradation on the mechanical properties that increase the tensile strength and decrease the elongation in breaks. Due to water absorption in the thermoplastic matrix, the polymer chain can be realigned by the occurrence of -OH groups (Nafchi *et al.*, 2013).

2.2.3.1 Starch Plasticization

Native starch has no thermoplastic properties without any addition of plasticizers such as water or glycerol. Product made from native starch is easily broken up into a fragment when dried in the environment, due to strong intermolecular linkages of hydrogen within the macromolecular chain of amylose and amylopectin. The high temperature and heating of the native starch melt and flow readily, allowing its utilization as an extruding, injection or blowing material similar to most traditional synthetic thermoplastic polymers. (Ma, Yu and Wang, 2007). Plasticizers attract water molecules around them and reduce the inter-molecular interactions between the starch molecules so that native starch increases their flexibility. This process of breakage by softening the form and enhancing the mobility of the macromolecular chains, resulting in a decrease in processing temperature, is called starch plasticization. (Zhang and Curtis, 2012).

Usually used plasticizes for thermoplastic starch are water, glycerol, fructose, sugar, glycols, formamide, urea, ethanolamine, and ethylene bisformamide. These chemicals are smaller and hydrophilic in their molecular size. The most common and affective plasticizers are traditionally water and glycerol. (Zhang and Curtis, 2012).

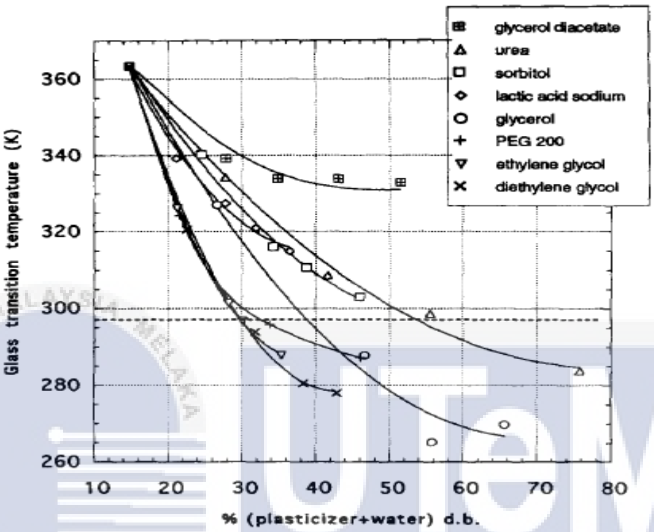


Figure 2.5 Relationship between T_g of TPS and plasticizer content and type (Lourdin *et al.*, 1997).

A minimum of 20% glycerol or any other appropriate plasticizer is required to successfully plasticize starch. The properties of thermoplastic starch can reduce the strength of the stress, Young's modulus and glass transition temperature (T g) with increasing plasticizer quantity and increase the elongation at breakage and gas permeability. Thermoplastic starch film with 25% glycerol shows the greatest tensile strength and the best elasticity modulus. (Pyshpadass, Marx and Hanna, 2008). In general, the increasing of the concentration of plasticizer will result in a lower T_g. It is because more plasticizer groups are available to bond with starch, hence increasing starch chain mobility but the intensity of this decrease is dependent on the nature of the plasticizer used (Lourdin *et al.*, 1997).

2.2.4 Water Absorption on Starch

Water absorption capacity and degradability are the most important properties for biodegradable materials. The material water absorbed allowed the microorganisms to grow and utilize the material as an energy source (Guohua *et al.*, 2006). The absorption of composite film into the polymer matrix immediately proportional to the amount of starch. In another sentence, water absorption will be increased in low-density polyethylene (LDPE) by increasing its starch content material. The hydrophilicity of starch and the ionic nature of the hydroxyl starch classes is responsible for water absorption. If the composite has lower mechanical properties the water absorption is high (Oromiehie, Lari and Rabiee, 2013).

2.2.5 Effect of Starch on Mechanical Properties

Starch have an interest to develop a biodegradable polymer composite. However, starch blended with other polymer resulting in different mechanical properties although the biodegradable starch has poor mechanical properties.

Maize starch and PVA (Polyvinyl Alcohol) combination films were made using a different ratio of starch and PVA casting solution method, followed by an investigation of the mechanical effects on the biodegradability of the film tested by the effects of gamma radiation. The result from the test show that tensile strength of the film decreased accordingly as the amount of CS increased. Elongation at break also continuously decreased as the amount of CS increased. TS and break elongation because of the amorphous nature of the starch that increases fragility and leads to a lower TS of the film. The PVA / CS dosage for γ-radiation, increases the TS value. The radiation cross-link reaction of TS of PVA / CS films was improved (Shafik, Majeed and Kamil, 2014).

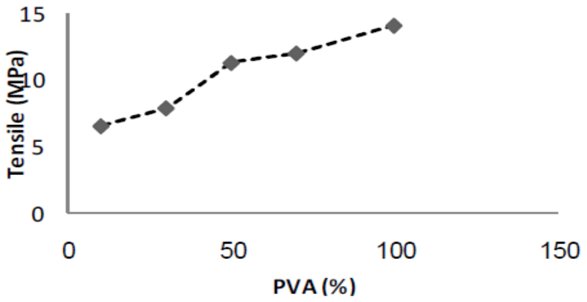


Figure 2.6 TS of PVA/CS ratio (with 20% formaldehyde) (Shafik, Majeed and Kamil, 2014)

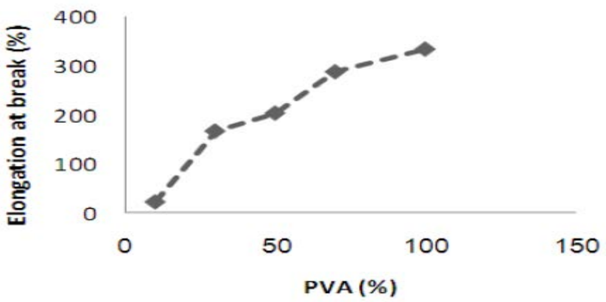


Figure 2.7 Elongation at break (%) of PV/CS ratio (with 20% formaldehyde) (Shafik, Majeed and Kamil, 2014)

Corn starch films were prepared by mixing methylated-cornstarch (MCS) with PVA and then the mechanical properties have been investigated. From the result, it was found that MCS/PVA film had higher water resistance other than the CS/PVA film. Also, the TS and elongation at break of MCS/PVA film continuously increased as the degree of substitution (DS) increased. Figure 2.8 illustrates that the TS is increased dramatically by increasing DS from 0.096 to 0.275 and TS from 0.275 to 0.864, with DS further increased from 0.275 to 0.863. (Guohua *et al.*, 2006)

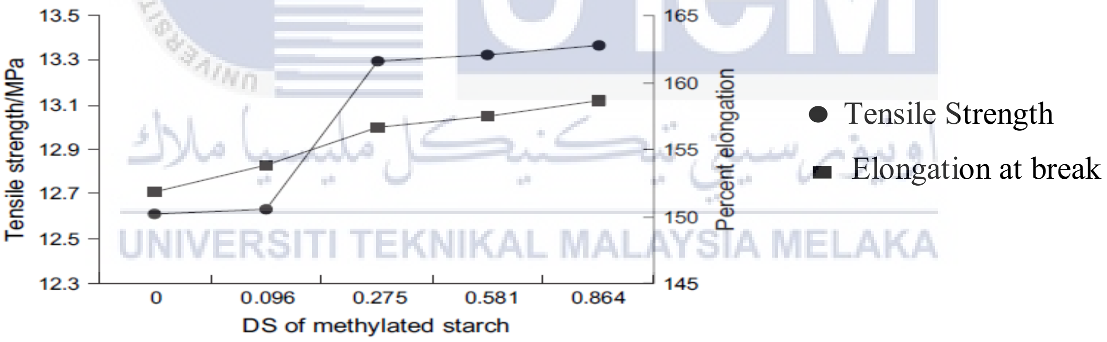


Figure 2.8 Effects of DS on the TS and elongation at break (%) of the MCS/PVA films (Guohua *et al.*, 2006)

Potato starch films was prepared by mixing with low density polyethylene (LDPE) and low density polyethylene grafted maleic anhydride (LDPE-g-MA). Starch content varied between 0 and 30% and mechanical properties was tested on the films by following ASTM Method D-638 (ASTM D638, 2015). As the amounts of starch increased the TS and elongation at break decreased. Definitely the tensile properties depend highly on the starch content on the films (Gupta, Sharma and Kumar, 2008).

Table 2.1 Average TS analysis result of LDPE and LDPE-g-MA/starch composites (Gupta, Sharma and Kumar, 2008)

Code (%)	Elongation in mm	Tensile strength in N/mm^2	Percentage elongation
0	105.0	10.0	420.2
5	101.5	9.2	406.1
10	53.3	8.8	213.2
15	40.4	8.6	161.5
20	5.7	5.7	22.9
25	5.5	6.5	22.0
30	2.7	3.6	11.0

Starch incorporated with Mentha Pulegium (MEO) film using a solution casting method was study to improve the mechanical properties. Increasing MEO content in the starch films will lead to TS decreases, which have changed substantially with the essential oil levels rising from 1 to 2 percent. However, when the contents of MEO increased from 1 to 3 percent, the elongation at the film break values improved (Ghasemlou *et al.*, 2013).

Potato starch and agar were processed into films by casting method through varying the agar ratio from 0% to 30%. TS and E of the films were then measured by using ASTM D882 (American Society for Testing and Materials. ASTM., 2002). TS and E of the starch film, agar film and starch/agar composite film are shown in Figure 2.10 respectively. The TS value of the composite film increases with the increasing amount of agar and between the value of starch and agar films. This increase can be connected to the formation, between starch and agar, of inter-molecular hydrogen connection and compact composite film structures. The concentration of agar enhanced the TS of composite film but at higher content of agar, the E values decreased (Wu *et al.*, 2009).

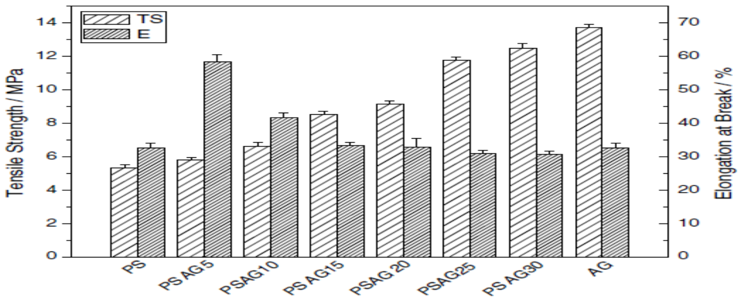


Figure 2.9 Effect of potato starch and agar content on the TS and E (Wu *et al.*, 2009)

2.3 Polypropylene

Polypropylene (PP) is a tough and rigid, crystalline thermoplastic produced from propene/propylene monomer. It is a linear hydrocarbon resin. PP is the first synthetic stereo-regular polymer for industrial uses. PP is among the cheapest plastics available, lightest and highly crystalline. PP exhibits hardness, high stiffness and tensile strength. Therefore, due to the high strength, PP is the most material used in automotive industry, industrial applications, household equipment, furniture market, and packaging application. PP is derived from non-renewable petroleum resource. PP is a non-degradable in nature, which can cause serious environmental problems if proper disposal is not taken (Gupta and Alam, 2014).

PP has the lowest density among commodity plastics such as polycarbonate (PC), polystyrene (PS), polyvinylchloride (PVC) and etc. PP has the excellent mechanical resistance and can be processed by injection moulding, extrusion moulding and compression moulding. PP belong to polyolefin family of polymers. The polymer is produced through a process of monomer connection called addition polymerization. The chemical formula of polypropylene is $(C_3H_6)_n$ (Maddah, 2016). Molecular structure of PP is shown below.

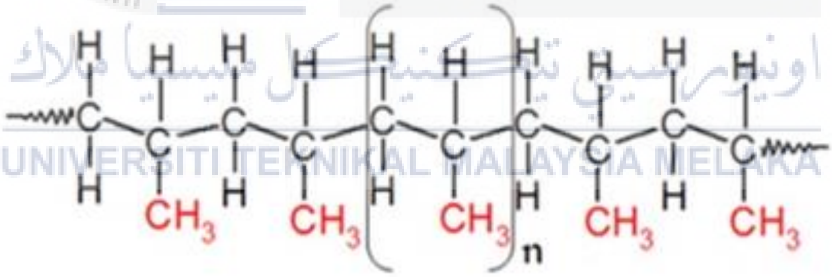
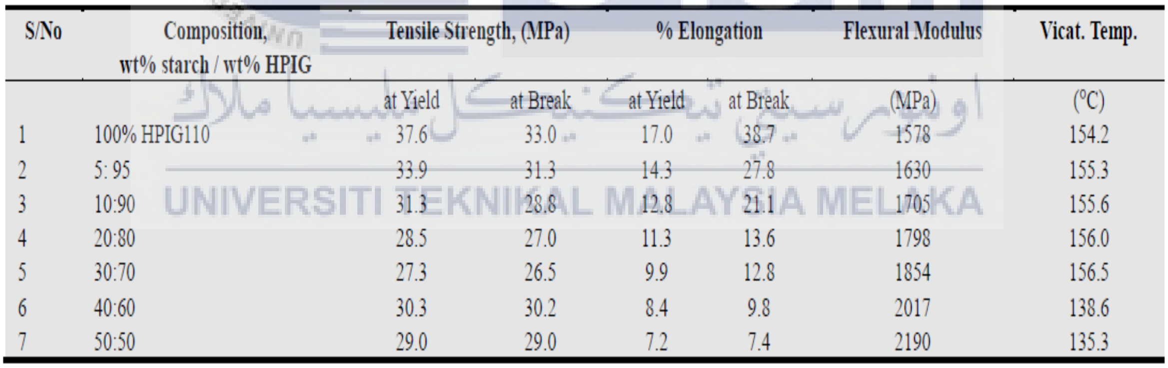


Figure 2.10 Molecular Structure of Polypropylene PP (Maddah, 2016).

2.3.1 Effect of Polypropylene on Mechanical Properties

Cassava starch/tapioca and polypropylene (PP) films were prepared by different starch content from 0% to 50% and then the films were tested. Tapioca with PP powder of grade HPIG110 (Homopolymer, Polypropylene, Injection moulding, General purpose additive) will decrease the Melt Flow Index (MFI) with increasing starch concentration which is attributable to increasing of viscosity of the blend that cause the polymer hard to flow, higher molecular weight and higher elasticity. The izod impact strength will increase if the Vicat Softening Point increase. However low softening temperature could not resist sudden load due to high starch content and cause the specimen to break when hammered. Starch and polyolefin blends usually contain low starch content and show good mechanical properties but higher starch loading in starch blended with polyolefin have poor mechanical properties since the mechanical properties of the starch are dominant. Tensile testing using ASTM D638 was using (ASTM D638, 2015). Incorporation of starch will decrease the value of Tensile Strength (TS) at yields and at break for PP because the higher the starch concentration in the blend, the lower the value of the TS (K. Oduola, 2015).

Table 2.2 Mechanical Properties of Starch and PP blends (K. Oduola, 2015)



S/No	Composition, wt% starch / wt% HPIG	Tensile Strength, (MPa)		% Elongation		Flexural Modulus	Vicat. Temp.
		at Yield	at Break	at Yield	at Break	(MPa)	(°C)
1	100% HPIG110	37.6	33.0	17.0	38.7	1578	154.2
2	5: 95	33.9	31.3	14.3	27.8	1630	155.3
3	10:90	31.3	28.8	12.8	21.1	1705	155.6
4	20:80	28.5	27.0	11.3	13.6	1798	156.0
5	30:70	27.3	26.5	9.9	12.8	1854	156.5
6	40:60	30.3	30.2	8.4	9.8	2017	138.6
7	50:50	29.0	29.0	7.2	7.4	2190	135.3

Potato starch (PS), PP (homopolymer) and maleic anhydride grafted PP (PP-g-MA) were prepared from different percentage range 0% to 30% starch by using extrusion moulding. The tensile properties for the blend have been conducted using universal testing machine model 3369 according to ASTM D638. The interfacial bonding is improved because of the plasticization of starch and the presence of PP-g-MA. Therefore, the addition of starch improved the tensile strength and impact strength flexural strength increased approximately 45%. The strain has decreased by 80% because strain is related to the ductility of a material (Gupta and Alam, 2014).

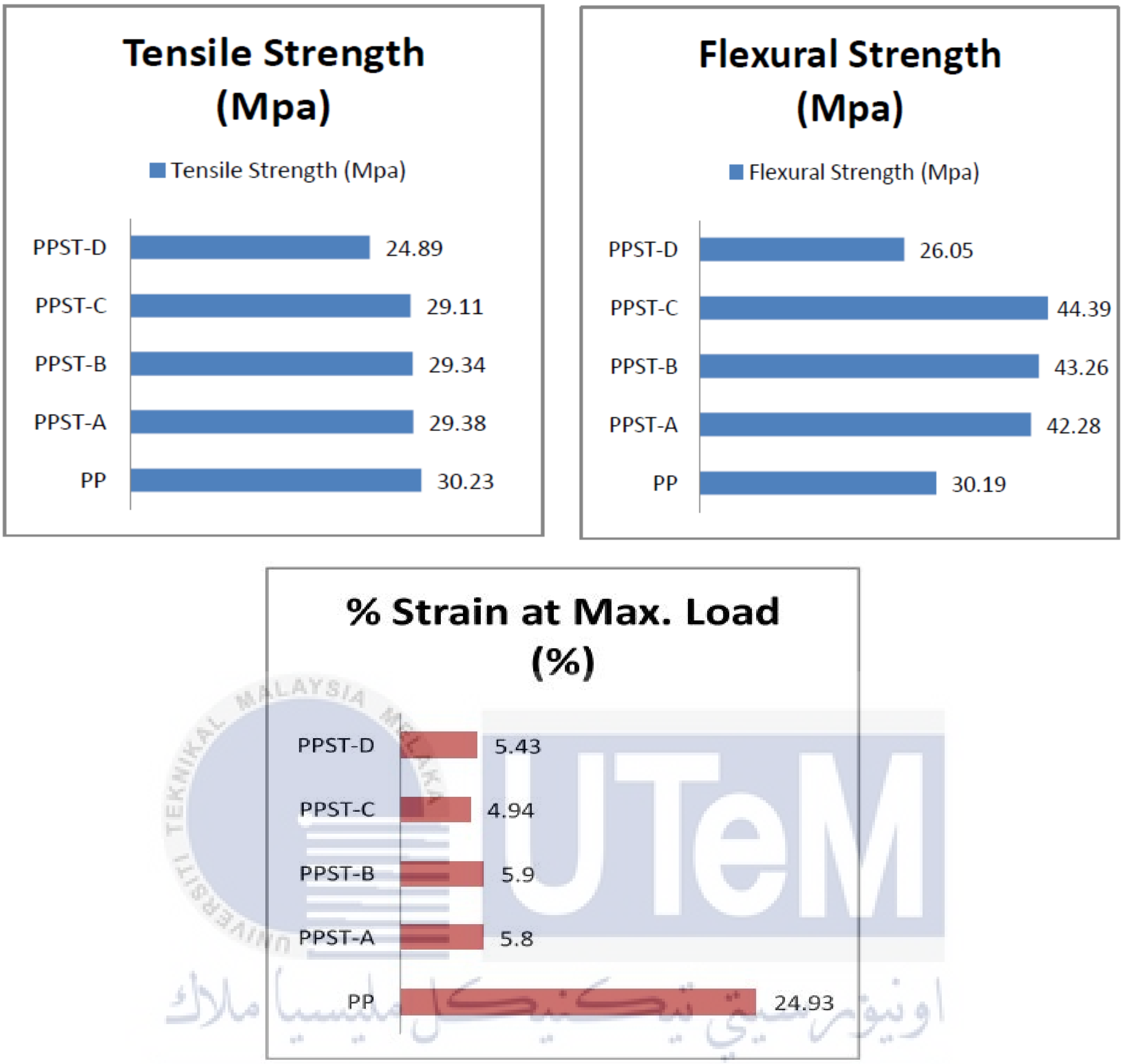


Figure 2.11 Tensile Strength (MPa), Flexural Strength (MPa), and %Strain of PS/PP/PP-g-Ma blend (Gupta and Alam, 2014)

2.4 Morphological studies

Starch granules in nature have various forms and sizes. Sphere, polygon, oval, lenticular (disk), kidney and elongated are common forms. Starch granules vary in size from submicron to about 100 µm (Ai and Jane, 2017).

Corn Starch and LDPE composite film surface were observed with a scanning electron microscope (SEM) by using Vega Tescan Model and operated at 23°C, 37% of relative humidity (RH) and 20kV of voltage. The results showed that the biodegradability of composites increased by increasing the starch content, because composite hydrophilicity due to higher water absorption and attacks on micro-organisms. It is clear that starch and the holes that have been generated increase LDPE surface and that therefore microorganisms can attack the LDPE matrix more easily, thereby creating favorable conditions for polyethylene oligomers to be consumed (Oromiehie, Lari and Rabiee, 2013).

SEM of agar/potato starch (PS) composite film with agar concentration from 0% to 30% were observed by using Microscope Philips XL-3, operating at an acceleration voltage of 20kV. The films shown in figure 2.11. The PS granules have ovate shapes and have a smooth surface. In addition to the intermediate and intramolecular hydrogen bonds and made polymer plastic, glycerol is well known for entering the inner chain of polysaccharides forming continuous plastic filming. All films of PS and agar were plasticized. The PS films fracture surface was smoother than the AG film. PS and agar content of 15% (PSAG15) film showed a relatively smooth and continuous cross section without any phase separation, which confirmed a homogeneous and dense structure with the addition of agar. Unfortunately, the fracture surface was not homogeneous like PSAG15 when more agar was added. If an appropriate starch / agar ratio has been exceeded, the compatibility of starch and agar might be reduced. There was no smooth intersection of the PSAG30 film and a multi laminar structure with fine pores and cracks. (Wu *et al.*, 2009).

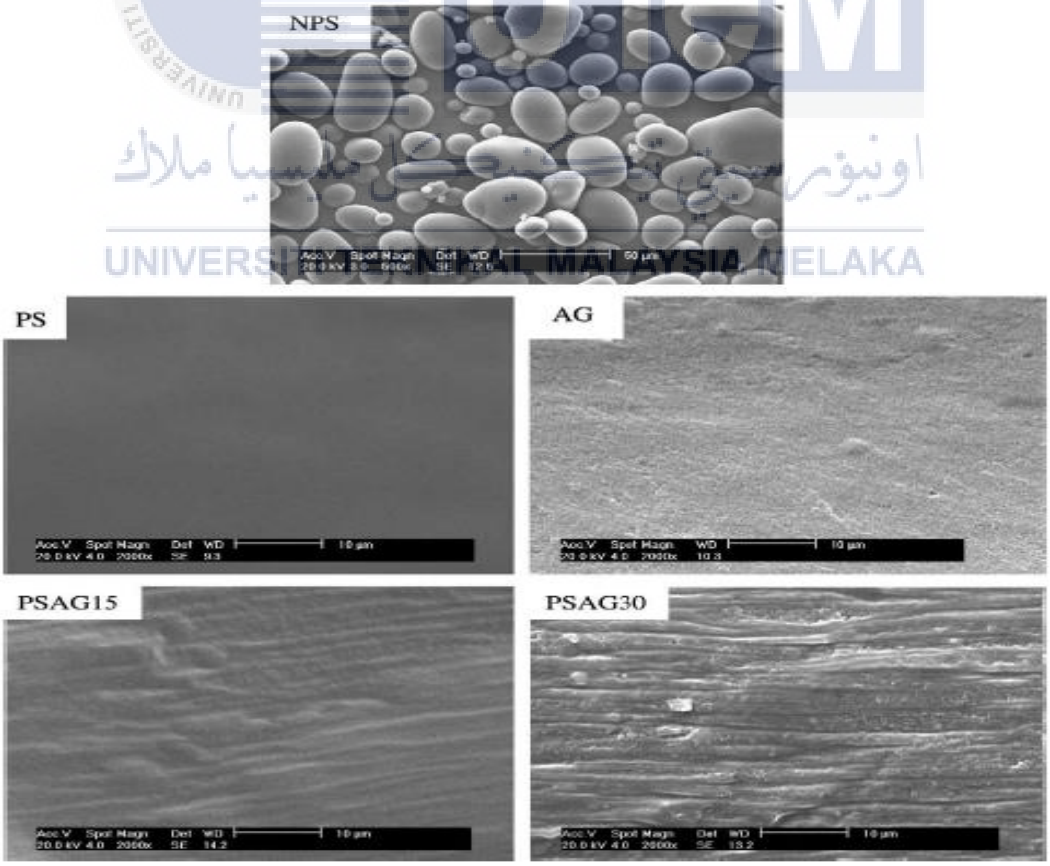


Figure 2.12 SEM images of agar/PS with concentration agar from 0%, 15% and 30% (Wu *et al.*, 2009)

CHAPTER 3

MEHODOLOGY

3.1 Introduction

This chapter describes the methodology used in this research to obtain data of the mechanical properties and morphological of corn-starch blended with polypropylene (PP). The flow chart of this research is shown in Figure 3.1. This research starts by studying the properties of starch and PP to develop biodegradable based polymers. After that, samples were prepared with different percentage of corn-starch that is 10%, 20%, 30% and 100% of PP by using hot compress moulding with a significant parameter such as temperature, pressure and time. Tensile test was done on the specimen to measure the mechanical properties that is tensile strength (TS), elongation at break (EB) and young's modulus.

Table 3.1 Details of Films Preparation

Sample no.	Corn-Starch Ratios	Polypropylene (PP) Ratio
PP	-	100%
CSPP10	10%	90%
CSPP20	20%	80%
CSPP30	30%	70%
CSPP40	40%	60%

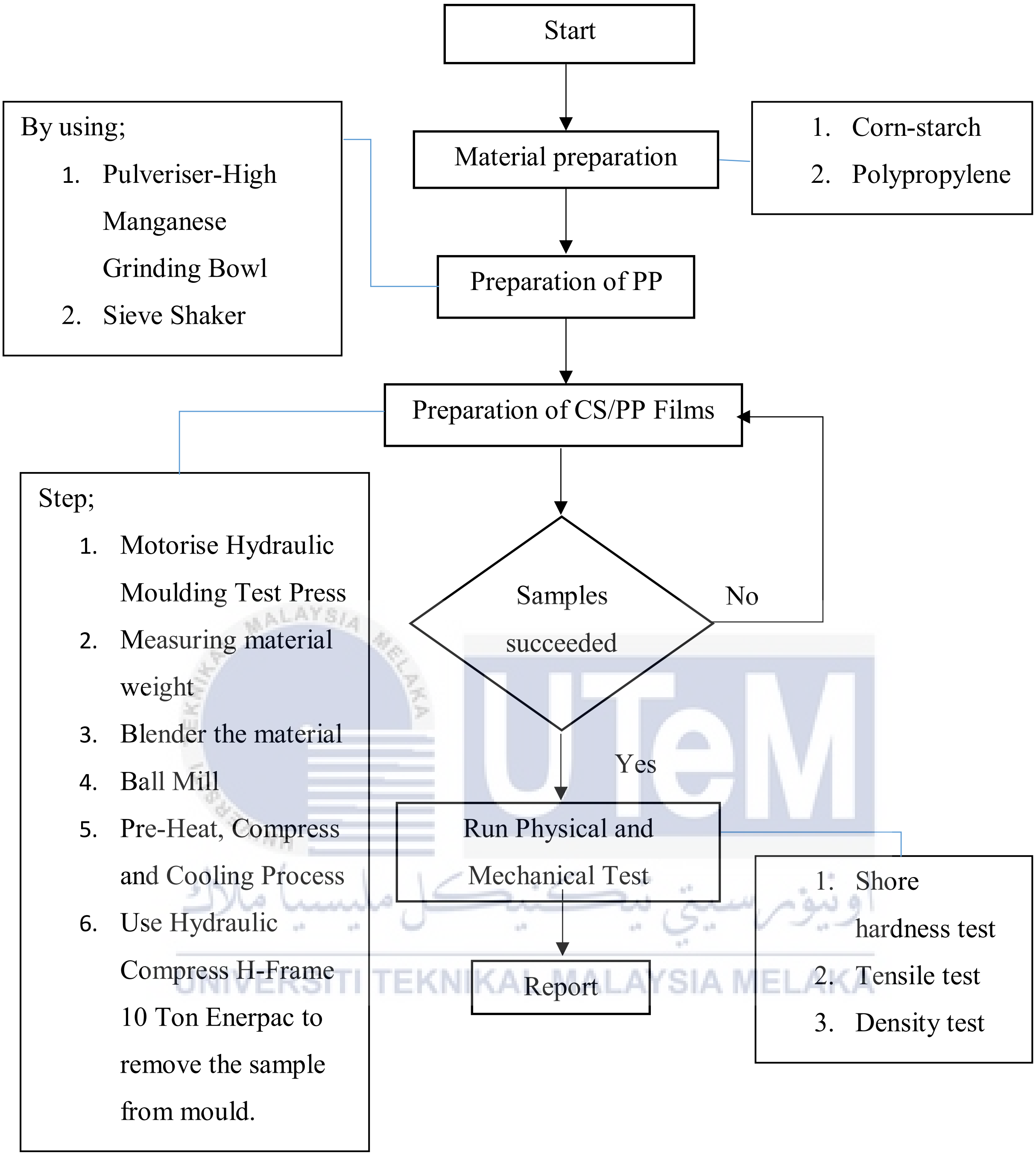


Figure 3.1 Flow chart of methodology.

3.2 Material

The raw material used to make the films is corn starch that is natural polymers which is biodegradable from renewable resources and polypropylene produced from propene monomer and cheapest plastic available.

Figure 3.2 Corn-Starch

Figure 3.3 Polypropylene

3.3 Preparation of Polypropylene

Polypropylene were processed first, from solid to powder. Pulveriser-High Manganese Grinding Bowl was used to grind the solid polypropylene to form polypropylene powder. Solid propylene was put in the bowl of pulveriser and then 'clamp' the pulveriser. The bowl can only be filled with a maximum 20 grams per use. Polypropylene will be ground about five to ten minutes.

Figure 3.4 Pulveriser-High Manganese Grinding Bowl

Figure 3.5 Bowl

Pulveriser cannot be used when it is hot. It is because it will provide polypropylene waste. The machine should be cooled first before it can be re-used to get a proper polypropylene powder. After that, polypropylene that has been grinding was blended to shrink the powder size. Then, sieve shaker was used to filter the polypropylene powder to achieve 500 micrometre (µm) polypropylene powder.

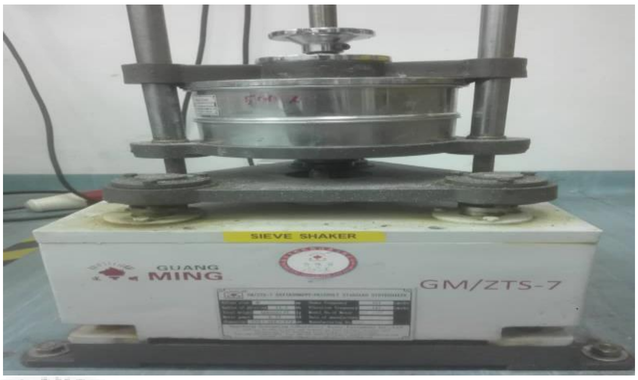


Figure 3.6 Sieve Shaker

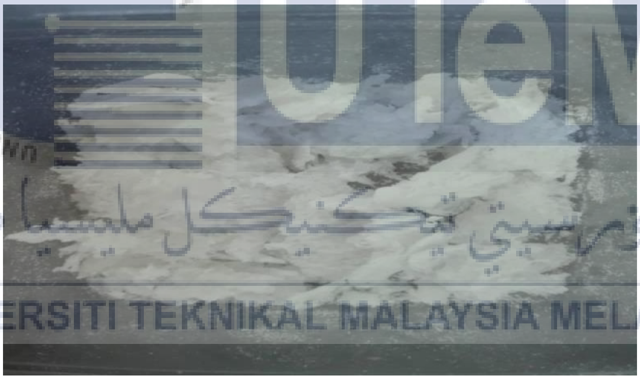


Figure 3.7 Polypropylene waste

Figure 3.8 Polypropylene Powder (500µm)

3.4 Preparation of CS/PP Films

Corn-starch/polypropylene based biodegradable polymer films were prepared by using motorise hydraulic moulding test press. The parameters of the moulding test press need to be set first that is temperature. Then, wait until the temperature reaches the desired temperature. To get the exact desired temperature, pressure and time, try and error were done to get perfect and fine films. It is because, different material composites had a different parameter. Corn-starch and polypropylene were weighted first, according to the volume of mould by using a weighing scale digital before it was blended together. Films were prepared from different weight percentage of corn-starch that is 10%, 20%, 30% and 40% with polypropylene percentage 90%, 80%, 70% and 60%, and 100% of polypropylene without mixed with corn-starch.

Figure 3.9 Motorise Hydraulic Moulding Test Press

Figure 3.10 Weighing Scale Digital

After that, weight material is placed in a transparent plastic box to isolate from 10% corn-starch content sample to 40%. The plastic box is placed with signs to prevent confusion of the corn-starch contents.

Figure 3.11 Transparent plastic box

Afterward, the polypropylene was blended five to ten minutes and then corn-starch was added and blend together for five minutes get a good mixture.

Figure 3.12 Blended Weighted Material

Then, the material was ground and blend by using a ball mill about 20 minutes to get a good homogeneity. The ball mill operates at 80 revolutions per minute (RPM).

Figure 3.13 Ball Mill

Next, the resulting mixture was poured on the mould plates of dimensions 14 x 6 cm with a thickness 0.3 cm. Before the resulting mixture is poured into the mould, the mould should be cleaned first using sandpaper and a scraper to remove the impurities founded on the mould and prevent the impurities mixed with the resulting mixture. After cleaning process, the wax will be applied to the mould to prevent the adhesion of composites with the plate.

Figure 3.14 Mould

When the temperature reaches the desired temperature, put the mould and resulting mixture into the moulding test press. Preheat the mould and resulting mixture without pressure to melt the material. Then, mould and material were compressed with pressure and then be cooled to get a crosslinking of the bond.

Figure 3.15 Pre-Heat, Compress and Cooling Process

After cooling process is done the mould were pressed by using hydraulic compress H-Frame 10 Ton Enerpac to remove the films from the mould. Mechanical tests can be performed on the films.

Figure 3.16 Hydraulic Compress H-Frame 10 Ton Enerpac

3.4 Characterization

3.4.1 Tensile Test

Tensile test is carried out to measure the capability and strength of a composite material that have been made. The TS test illustrates a composite material's capacity to resist load / forcing when the films are being pulled as well as the capacity of the material to stretch before they break and EB of the films also being measured using a universal testing machine Instron-8872 at the structural mechanics' laboratory, University Technical Malaysia Melaka (UTeM). EB also known as fracture strain. It is the ratio between changed length and initial length after breakage of the test specimen. If the strength higher, the greater it can withstand a force and the higher the elongation value, the greater the ductility.

Figure 3.17 Universal Testing Machine Instron-8872

After sample have been done, sample were cut into specimen to conduct the tensile test. The tensile test will be done by following ASTM D-638. According to ASTM D-638 Type 1 is the preferred specimen and shall be used where sufficient material having a thickness of is 7mm (0.28in) or less is available with a length of 165mm (6.5in) and width 19mm (0.75in) (ASTM D638, 2015).

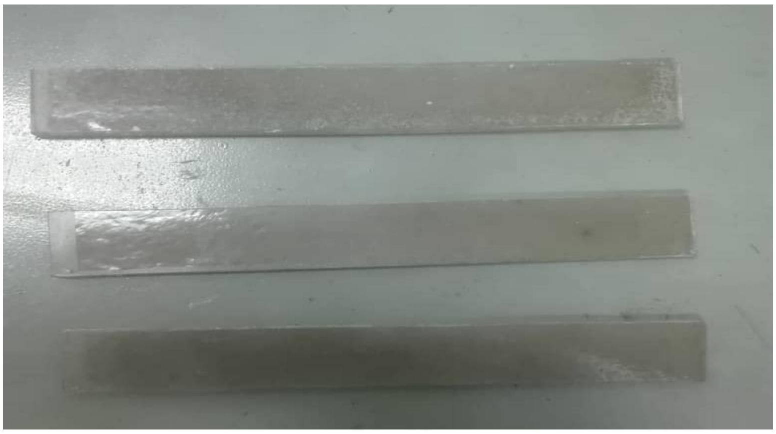


Figure 3.18 Specimen for Tensile Test

Specimens were put into the universal testing machine and adjust the grip of machines to hold the specimen tightly. According to ASTM D-638 the recommended crosshead speeds to run the tensile test is 5mm/min (0.2in/min) using the lowest speed that ruptures the specimen within 30 seconds 5 minutes (Intertek expert, 2018).

Figure 3.19 Tensile test (specimen rupture)

After the tensile test was performed, stress-strain graph is obtained by the machine. Figure 3.20 shows the stress-strain curve. Tensile test result provide stress at which observable plastic deformation or "yielding" begins, the maximum load intensity of the ultimate tensile strength that can be caused by tension, and the percent elongation or strain (the amount that will stretch the material) and the accompanying percentage reduction of the cross-sectional area caused by stretching. The rupture or fracture point can also be determined. Young’s modulus can be obtained from the stress-strain curve. Young’s modulus is a mechanical property that measures the stiffness of the material, where the formula is $E = \frac{\sigma}{\epsilon}$ where, σ is the uniaxial stress and ϵ is the strain.

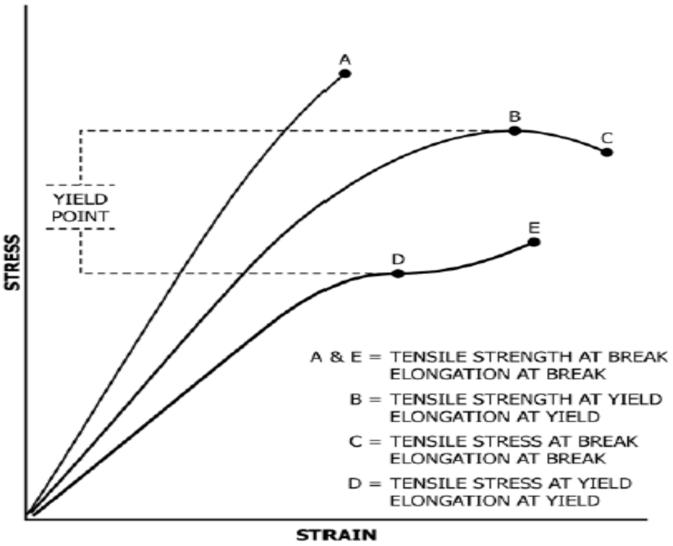


Figure 3.20 Tensile Designations (stress-strain curve) (ASTM D638, 2015)

3.4.2 Shore Hardness

Shore hardness is tested with an instrument called durometer. Shore hardness is used to determine soft material's relative hardness, usually plastic or rubber and a measure of the resistance a material has to indentation. Shore hardness is performed by using ASTM D-2240 (ASTM, 2015). The test measures a specified indenter's penetration into the material under specified force and time conditions. The hardness value is often used to determine or specify a specific elastomer hardness or as a measure of quality control on material. The specimen is placed first on a flat and hard surface. The indenter for the instrument is then pressed into the specimen ensuring that it is parallel to the surface. The hardness value is read when the firm contact with the specimen. Red needles show the hardness value of the specimen.

Figure 3.21 Durometer Test (in shore D)

3.4.3 Density

Density of a material as its mass per unit volume. The symbol used for density is ρ. Mathematically, density is defined as mass divided by volume: $\rho = \frac{m}{v}$ where m is mass (kg) and v is volume(m^3). Different materials have a different density. Therefore, density is an important concept regarding buoyancy, purity and packaging. In some cases density is expressed as the dimensionless quantities specific gravity (SG) or relative density (RD). SG less than one means that the substance floats in water. Electronic densimeter was used to measure the mass and volume of material.

Figure 3.22 Electronic Densimeter

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Introduction

Preparation of corn-starch and polypropylene sample has been done in methodology (chapter 3). After sample has been done, testing of the film specimen were performed to investigate the mechanical properties and morphological. Testing was conducted by using American Society for Testing and Material (ASTM) as a reference. Tensile test was performed by using ASTM D-638 and shore hardness using ASTM D-2240.

4.2 Results

4.2.1 Corn-starch and PP Sample

Five samples have been done by using hydraulic moulding test press in Lab FKM Fasa B. The parameters used to obtain the sample should be in accordance with the material used. It is because material that have been used and other material have different parameters. It is necessary, to find the appropriate parameters to conduct the preparation of starch and PP sample. When the temperature is too high and the time taken is too long, the material will burn and when the temperature is low, the material will not melt. Pressure should need to be considered, because when the pressure is too high, the material will be fractures and cracked. Therefore, to find the appropriate parameter to prepare the sample is by using Taguchi method. Taguchi method was used to identify the temperature, pressure and time to get the most excellent films without crack or fracture on the sample.

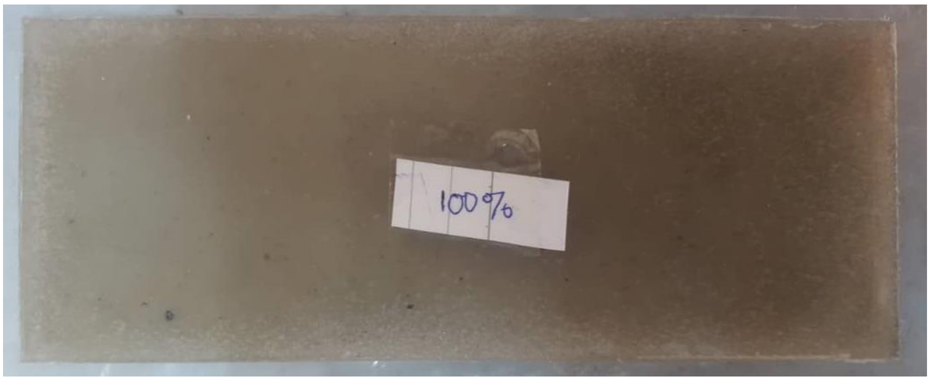


Figure 4.1 Polypropylene (PP) 100% ratio sample

Figure 4.2 Corn-starch 10% and PP 90% ratio sample

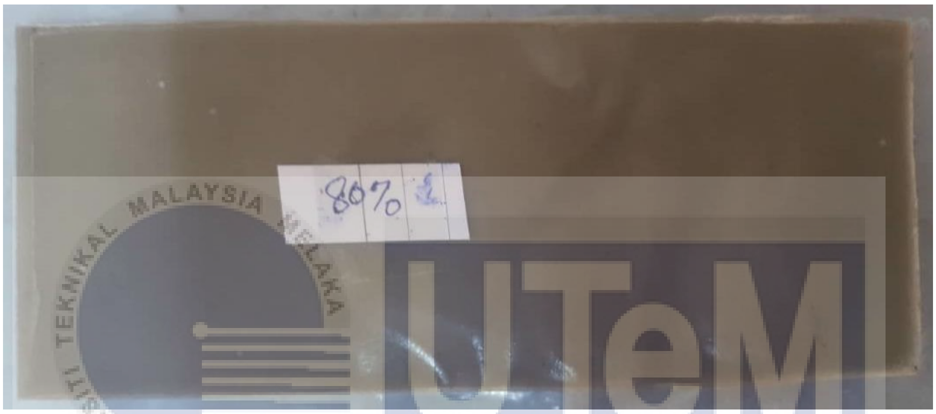


Figure 4.3 Corn-starch 20% and PP 80% ratio sample

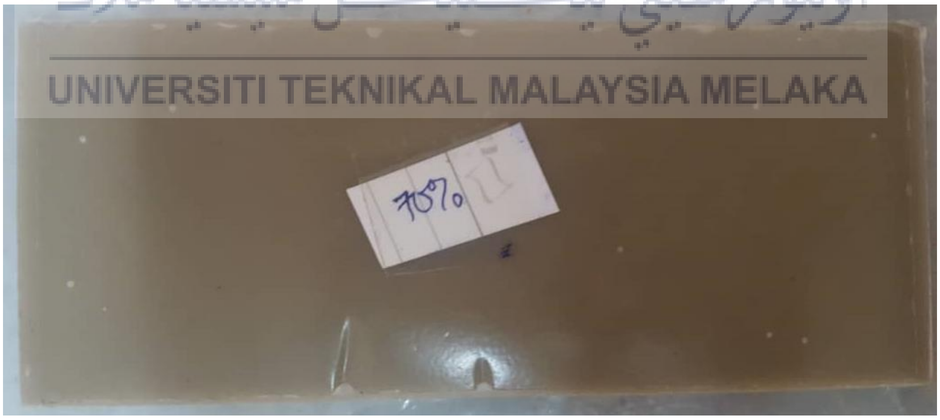


Figure 4.4 Corn-starch 30% and PP 70% ratio sample

Figure 4.5 Corn-starch 40% and PP 60% ratio sample

After the sample have been done, mechanical and physical properties were performed on the films specimen to study the properties of the films. Tensile test that is tensile strength and elongation at break were performed on the films to measure the strength of the films. Shore hardness is used to determine material's relative hardness. Scanning electron microscope also performed on the surface films after the tensile test were done to observe the interaction of the electron on the films.

4.2.2 Shore Hardness

Shore hardness show the hardness of the material composition by using durometer (in shore D). The graph shows the hardness value with the different percentage of corn-starch and PP content. The graph shows that PP with some corn-starch additive improve the hardness value, where 90% of PP and 10% of corn-starch have the highest hardness value that is 74. It is because the interface between two higher polymers, homogeneous phases, and more dissolved amounts of solid will make the cross-link between the corn-starch and PP more tightly (Eko Wahyuningtiyas *et al.*, 2018). Unfortunately, as the increasing of the corn-starch content the hardness value decrease that has lower resistance to indentation. Its mean 10% of corn-starch additives can improve the hardness value that has highest resistance to indentation and thus harder materials.

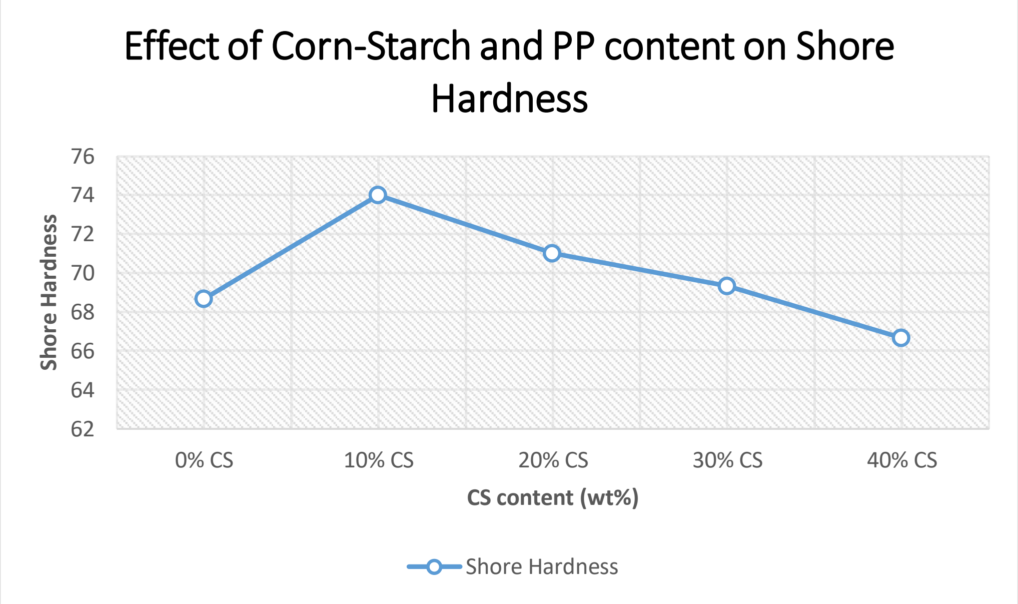


Figure 4.6 Effect of Corn-Starch and PP content on Hardness (in shore D)

4.2.3 Tensile Test

Tensile test is a measurement of the force required to pull the material until the material fracture and measure the specimen response to the stress. Universal Testing Machine Instron-8872 was used to get the results of specimen response. Figure 4.7 until 4.11 are the stress-strain graph result for each sample with three specimens.

4.2.3.1 Stress-strain curve for each sample for three specimen

Figure 4.7 show the stress-strain graph for 100% PP without addition of corn-starch content for three specimens. Specimen 1 has the highest value of the tensile stress at maximum load or tensile strength is 31.7957 MPa and the maximum load is 1907.74 N. The average load at yields (offset 0.2%) is 1095.57 N, where the tensile stress at yields (offset 0.2%) for the average specimen is 18.2595 MPa and the tensile strain at yields (offset 0.2%) is 0.02059. The composite has the highest value of stress and large strain. Before the yield, the material will have an elastic deformation which can return to the original shape when the load is removed.

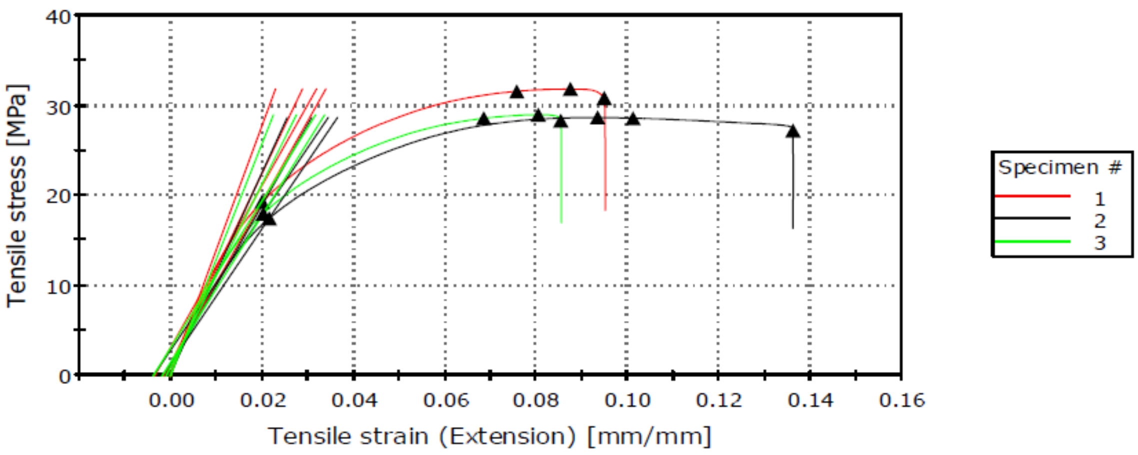


Figure 4.7 Stress-Strain Graph for 100% PP

Figure 4.8 show the stress-strain graph for corn-starch 10% content and 90% PP content for three specimens. The average of the tensile stress at maximum load or tensile strength is 21.6521 MPa and the average maximum load is 1299.12 N. The average load at yields (offset 0.2%) is 971.83 N, where the tensile stress at yields (offset 0.2%) for the average specimen is 16.1973 MPa and the tensile strain at yields (offset 0.2%) is 0.01941.

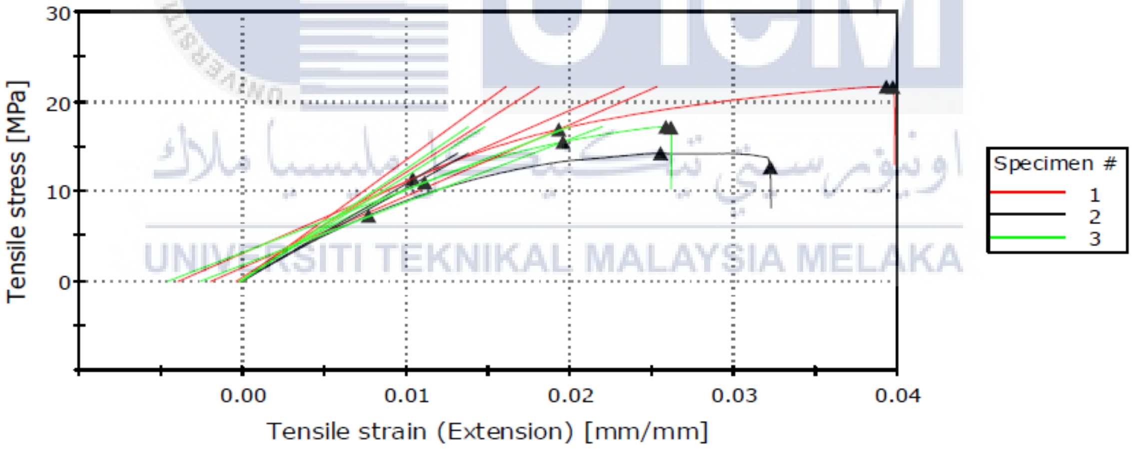


Figure 4.8 Stress-Strain Graph for Corn-starch 10% and 90% PP

Figure 4.9 show the stress-strain graph for corn-starch 20% content and 80% PP content for three specimens. The average of the tensile stress at maximum load or tensile strength is 18.5279 MPa and the average maximum load is 1111.67 N. The average load at yields (offset 0.2%) is 929.37 N, where the tensile stress at yields (offset 0.2%) for the average specimen is 15.4895 MPa and the tensile strain at yields (offset 0.2%) is 0.01629.

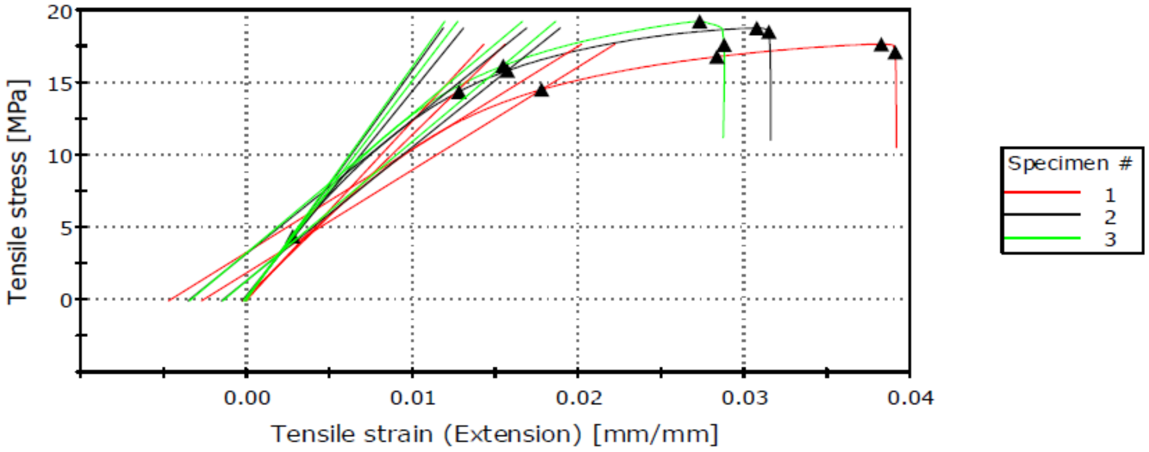


Figure 4.9 Stress-Strain Graph for Corn-starch 20% and 80% PP

Figure 4.10 show the stress-strain graph for corn-starch 30% content and 70% PP content for three specimens. The average of the tensile stress at maximum load or tensile strength is 15.9601 MPa and the average maximum load is 957.61 N. The average load at yields (offset 0.2%) is 895.97 N, where the tensile stress at yields (offset 0.2%) for the average specimen is 14.9328 MPa and the tensile strain at yields (offset 0.2%) is 0.01227.

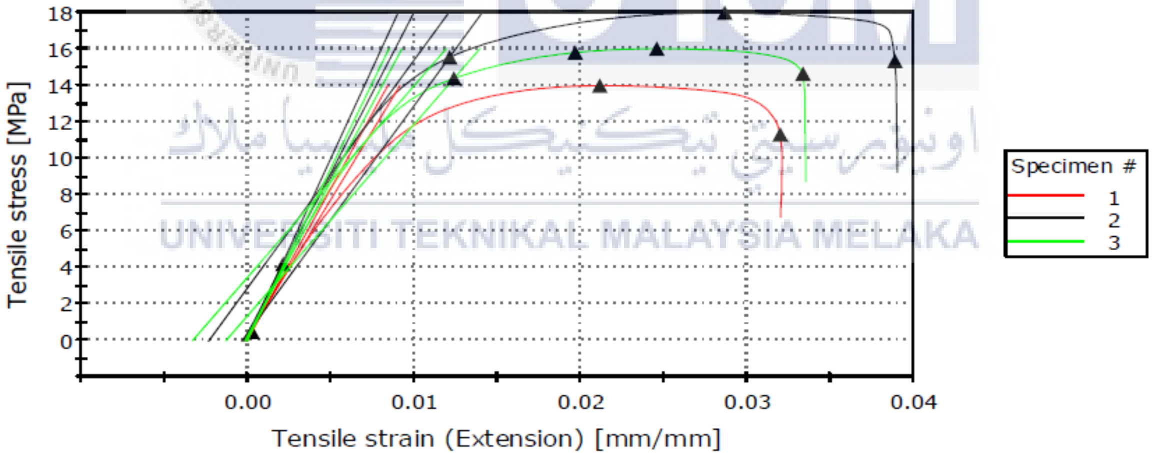


Figure 4.10 Stress-Strain Graph for Corn-starch 30% and 70% PP

Figure 4.11 show the stress-strain graph for corn-starch 40% content and 60% PP content for three specimens. The average of the tensile stress at maximum load or tensile strength is 14.9209 MPa and the average maximum load is 895.25 N. The average load at yields (offset 0.2%) is 803.39 N, where the tensile stress at yields (offset 0.2%) for the average specimen is 13.3899 MPa and the tensile strain at yields (offset 0.2%) is 0.01329.

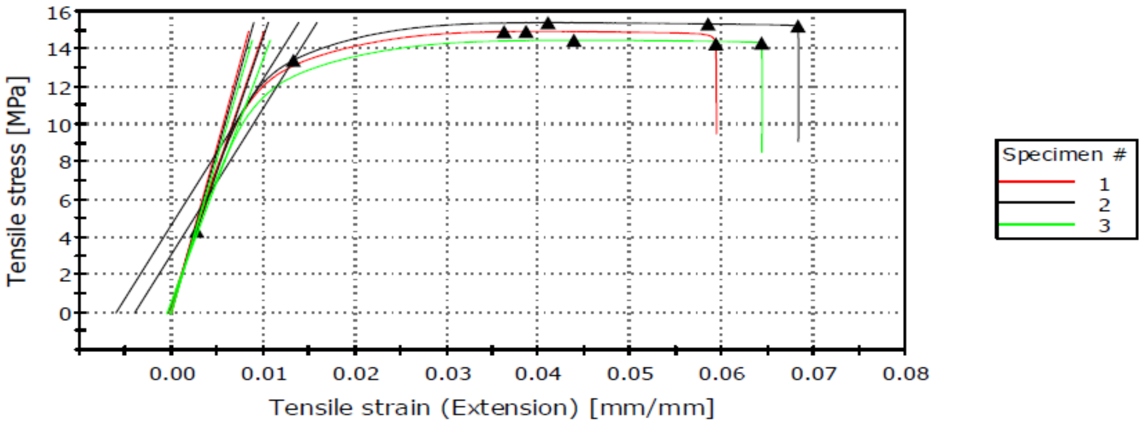


Figure 4.11 Stress-Strain Graph for Corn-starch 40% and 60% PP

4.2.3.2 Average Tensile Properties of CS/PP

Figure 4.12 and figure 4.13 shows the average effect of corn-starch and PP with a different content on tensile properties. Polypropylene without addition of corn-starch has the highest value of tensile strength that is 29.8 MPa. Tensile strength decrease with increasing in corn-starch concentration as shown in Figure 4.12. This can probably be explained by the low tensile strength of pure corn-starch that is almost 90% lower than PP (K. Oduola, 2015). The presence of corn-starch on PP reduces the tensile strength value about 27%. As the increasing content of corn-starch 10%, the tensile strength value of the composite decrease about 14%. Due to the weakness of interfacial adhesion between starch polymers, the decrease in tensile strength occurred. In addition, the sample was shown to contain bubbles. Due to the presence of moisture at the PP starch interface, the tensile strength could also be decrease (Kormin *et al.*, 2015). Therefore 100% PP content has the highest ability to withstand a greater force.

The strain or elongation of the different percentage PP show the 100% PP has the highest value of strain that is 0.087. Addition of 10% of corn-starch in PP reduces the strain value but as the increasing of corn-starch content the strain value slightly increase. When the force applied increase at yields, the deformation change from elastic deformation to plastic deformation because the elastic limit is exceeded which is the material will experience a permanent deformation when the load is removed. The bond between the atoms are broken, and new atoms are formed, making reversal to the original shape impossible, it’s called as plastic deformation. Therefore, 100% PP content undergo large strains before failure and has the greater ductility.

Young's modulus is a measure of stiffness, and ability of a material withstand changes in length when under lengthwise tension or compression. As the concentration of corn-starch increases the value of Young's modulus, also increase but the tensile strength decrease.

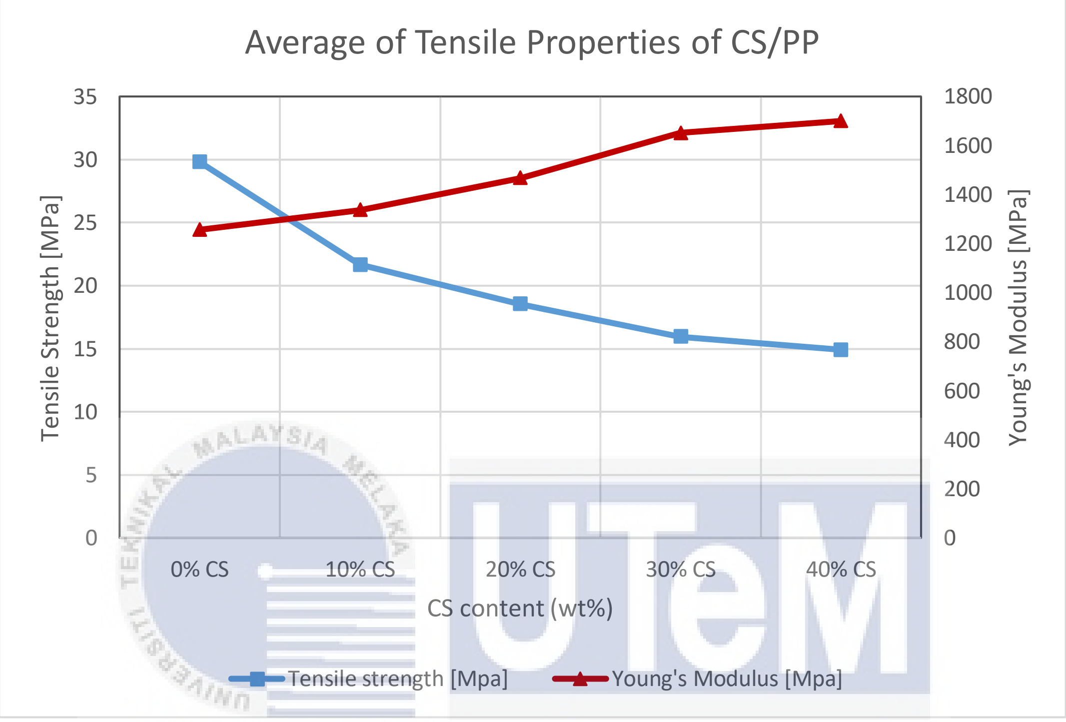


Figure 4.12 Effect of corn-starch and polypropylene content on tensile properties (Tensile Strength and Young's Modulus)

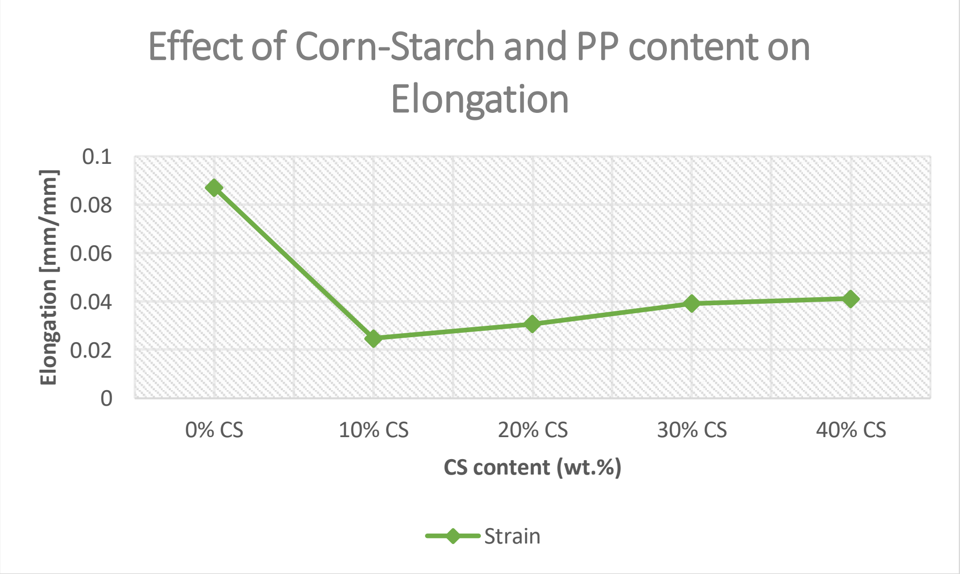


Figure 4.13 Effect of Corn-starch and PP content on Elongation

Table 4.1 show the tensile properties of corn-starch blended with polypropylene compositions at yields and at break such as load, tensile strength, elongation, and young's modulus. Before the yield occurs, the composite will undergo elastic deformation and after the yield it will undergo plastic deformation which cannot return to original shape. The material will fracture at break. At higher corn-starch concentration in CS/PP composite, mechanical properties are poor since the mechanical properties of the starch dominant. The higher the starch concentration in the mixture, the lower the tensile strength value (K. Oduola, 2015). At 100% PP content the load in Newton (N) at yields and at break is decreasing as the corn-starch content is increase. As the load is decreasing the tensile strength also decreasing. Measurement of tensile strength showed that the higher the starch content in the composite, the lower the mechanical properties. By contrast, as the starch concentration increase, the young's modulus increases (Kormin *et al.*, 2017).

Table 4.1 Tensile Properties of CS/PP composite

S/No.	Composition, wt% CS/ wt% PP	Load [N]		Tensile Strength [Mpa]		Elongation [mm/mm]		Young's Modulus [Mpa]
		at Yield	at Break	at Yield	at Break	at Yield	at Break	
1	100% PP	1095.57	1847.4	18.26	30.79	0.02059	0.09477	1256.49
2	10% : 90%	971.83	1296.8	16.2	21.61	0.01941	0.03966	1336.94
3	20% : 80%	929.37	1108.9	15.49	18.48	0.01629	0.03144	1467.07
4	30% : 70%	895.97	918.4	14.93	15.31	0.01227	0.03883	1651.3
5	40% : 60%	803.39	855.8	13.39	14.26	0.01329	0.05925	1699.87

4.2.4 Density

Table 4.2 shows the density of CS/PP composites. In general, slight increase in the density was evidenced by increasing the corn-starch content in the composites. The 10% of corn-starch and 90% of PP content show 2% higher density than the 100% of PP content. Moreover, as the increasing of corn-starch content the density also increase. Increasing in density of composites following the incorporation of starch was also reported in previous work (Kormin *et al.*, 2017). Kormin et al. conducted a study on the effect of LDPE incorporated with different starch sources, where increasing of starch content was shown to increase the density of the composites. The increase in density of corn-starch composites in this study because starch is a polar substance, and PP is a non-polar. This contributes to a poor connection between the filler and the matrix. Because of a poor plastic moistening on the filler, fillers and plastic result in a simple breakup of the cell walls (Kormin *et al.*, 2017).

Table 4.2 Density of CS/PP composite

S/No.	Composition wt% CS/ wt% PP	Mass (g)	Volume (cm³)	Density (g/cm³)
1	100% PP	4.29	4.329	0.9901
2	10% : 90%	3.22	3.179	1.013
3	20% : 80%	2.83	2.698	1.049
4	30% : 70%	1.25	1.159	1.079
5	40% : 60%	1.48	1.364	1.085

CHAPTER 5

CONCLUSION AND RECOMMENDATIONS

5.1 Conclusion

The obtained result can be summarised as follows: the addition of corn-starch to the polypropylene matrix increases the shore hardness value of the sample, where 10% of the corn-starch content has the highest value of hardness that is 74 in shore D. As the increasing of the corn-starch content in PP the hardness value was decreases. The best tensile strength of a corn-starch/PP content was at 21.65 MPa, its elongation at break was 0.03966 mm/mm and its corn-starch/PP content was 10/90. Presence of corn-starch in PP reduces the tensile strength but increases the Young's modulus, the stiffness of the material. Measurement of tensile strength showed that the higher the starch content in the composite, the lower the mechanical properties. At higher corn-starch concentration in CS/PP composite, mechanical properties are poor since the mechanical properties of the starch dominant. Due to the weakness of interfacial adhesion between starch polymers, the decrease in tensile strength occurred. In addition, bubbles were found in the samples. The presence of moisture on the PP starch interface, there was also a possibility to reduce tensile strength. Slight increase in the density by increasing the corn-starch content in the composite because of poor bonding between the corn-starch and PP. This study shows that 10% corn-starch in PP is the best composition on the tensile strength. Although corn-starch and PP blends decrease the tensile strength value about 27% from pure PP, this composite can be used for other applications.

5.2 Recommendations

After thorough analysis of data and results, the following recommendations are hereby made. This research study suggests that add a material to the corn-starch and polypropylene blends to study the mechanical and physical properties. The material that have been suggested is fibre that possess excellent mechanical properties which can improve the mechanical properties of the blends. Natural fibre is recommended such as kenaf fibre which is biodegradable.

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APPENDICES

Gantt chart for FYP 1 and FYP 2

Weeks Tasks	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
FYP 1															
Topic Selection	■														
Topic Confirmation with Supervisor		■													
Problem Statement			■	■											
Literature Review			■	■	■	■	■	■	■	■	■	■	■	■	■
Gantt chart and Flow Chart					■										
Report Preparation						■	■	■	■	■	■	■	■	■	■
Presentation Slide with Supervisor							■								
Submission Progress Report								■							
Sample Preparation									■	■	■	■	■	■	■
Submission Report FYP 1														■	
Seminar FYP 1															■

FYP 2															
Sample Preparation (Corn-starch/Agar/Glycerol)	■	■													
Meet Supervisor about the sample (CS/Agar/Glycerol)			■												
New Topic (Corn-starch and PP)			■												
Preparation of PP			■	■	■										
Preparation of CS/PP sample					■	■	■	■	■						
Submission Progress Report 2								■							
Hardness Test									■						
Tensile Test										■	■				
Density Test												■			
Result and Discussion								■	■	■	■	■	■		
Conclusion and Recommendation												■	■		
Submission Report FYP 2														■	
Seminar FYP 2															■