

INVESTIGATION OF MECHANICAL BEHAVIOUR ON CORN STARCH



BACHELOR OF MANUFACTURING ENGINEERING TECHNOLOGY (PROCESS AND TECHNOLOGY) WITH HONOURS



Faculty of Mechanical and Manufacturing Engineering Technology



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Bachelor of Manufacturing Engineering Technology (Process and Technology) with Honours

INVESTIGATION OF MECHANICAL BEHAVIOUR ON CORN STARCH

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DECLARATION

I declare that this thesis entitled "Investigation Of Mechanical Behaviour On Corn Starch" is the result of my own research except as cited in the references. The thesis has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.



APPROVAL

I hereby declare that I have checked this thesis and in my opinion, this thesis is adequate in terms of scope and quality for the award of the Bachelor of Manufactuing Engineering Technology (Process and Technology) with Honours.

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Date : 18 JANUARY 2022	

DEDICATION

This thesis I dedicated to my parents, Mr Ahamad Bin Eni and Mrs. Salhah Binti Haji Tani for the never ending support, love and prayers.



ABSTRACT

Renewable resources are typically plant-based resources such as starch, agar, and cellulose. Starch, which is produced as an energy store by many green plants, has been considered an appealing and ideal candidate to replace petroleum-based materials. The main aim of this research is to investigate the mechanical behaviour of biodegradable matrix derived from corn starch. The objectives of this research is to fabricate the composite matrix using biodegradable derived from corn starch and to understand the fundementals mechanical behaviour and morphological of thermoplastic corn starch. During material preparation, both thermoplastic starch and glycerol must be weight using different type of ratio before mixing it alltogether. Then, the mixture of thermoplastic cornstarch and glycerol were mix together using either hand or high speed blender for about 2 to 3 minutes. The mixture then put into the mould with the dimension of 140 x 60 mm. After prepping the mixture inside the mould, the mould will be put into the hot press machine for pre – heating process for about 15 minutes then hot press at pressure of 25 kg/cm2 with 165°C of temperature for 15 minutes followed by cooling process for another 15 minutes. The material testing involved in this research is tensile test following the ASTM D-638 and flexural test by following the ASTM D790. As for morphological characteristics, scanning electron microscope (SEM) will be involved. The highest tensile strength for tensile testing is concertration of 70 wt.% thermoplastic corn starch while whereas the highest flexural strength is achieved at concertration of 70 wt.% thermoplastic corn starch. In conclusion, the addition of glycerol substantially increased the tensile strenght and flexural strenght of the composites matrix.

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ABSTRAK

Sumber boleh diperbaharui biasanya sumber berasaskan tumbuhan seperti kanji, agar-agar dan selulosa. Kanji, yang dihasilkan sebagai simpanan tenaga oleh banyak tumbuhan hijau, telah dianggap sebagai calon yang menarik dan ideal untuk menggantikan bahan berasaskan petroleum. Matlamat utama penyelidikan ini adalah untuk menyiasat tingkah laku mekanikal matriks terbiodegradasi yang diperoleh daripada kanji jagung. Objektif penyelidikan ini adalah untuk merekacipta matriks komposit menggunakan terbiodegradasi yang diperolehi daripada kanji jagung dan untuk memahami kelakuan mekanikal asas dan morfologi kanji jagung termoplastik. Semasa penyediaan bahan, kedua-dua kanji termoplastik dan gliserol mestilah berat menggunakan nisbah jenis yang berbeza sebelum mencampurkannya kesemuanya. Kemudian, campuran tepung jagung termoplastik dan gliserol digaul bersama sama ada menggunakan tangan atau pengisar kelajuan tinggi selama kira-kira 2 hingga 3 minit. Campuran kemudian dimasukkan ke dalam acuan berdimensi 140 x 60 mm. Selepas menyediakan adunan di dalam acuan, acuan akan dimasukkan ke dalam mesin penekan panas untuk proses pra-pemanasan selama kira-kira 15 minit kemudian tekan panas pada tekanan 25 kg/cm2 dengan suhu 165°C selama 15 minit diikuti dengan proses penyejukan untuk yang lain. 15 minit. Ujian bahan yang terlibat dalam penyelidikan ini ialah ujian tegangan mengikuti ASTM D-638 dan ujian lenturan dengan mengikuti ASTM D790. Bagi ciri morfologi, mikroskop elektron pengimbasan (SEM) akan terlibat. Kekuatan tegangan tertinggi untuk ujian tegangan ialah gabungan 70 wt.% kanji jagung termoplastik manakala kekuatan lentur tertinggi dicapai pada gabungan 70 wt.% kanji jagung termoplastik. Kesimpulannya, penambahan gliserol telah meningkatkan dengan ketara kekuatan tegangan dan kekuatan lenturan matriks komposit. 1.1 10 0.

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#### **CHAPTER 1**

#### **INTRODUCTION**

#### 1.1 Background

Composite materials are created by combining two or more materials with distinct properties that do not dissolve or mix together. The different materials in the composite work together to give the composite unique properties (Canyon Hydro et al., 2013). The composites industry is still developing today, with much of the recent development centred on renewable energy. The growth of composite applications is due to a number of factors, the most important of which is that composite-fabricated products are stronger and lighter (Manohar, 2019).

Plastic products made from petroleum-based polymers have had a negative impact on the environment due to the accumulation of non-biodegradable waste (Jumaidin et al., 2017). In the last 20 years, people has been focusing on developing starch-based thermoplastics to reduce the use of non-biodegradable synthetics such as thermoplastics (Averous & Boquillon, 2004). Starch-based thermoplastics are environmentally friendly, cheap to produce and has widely available raw materials and highly biodegradable.

Renewable resources are typically plant-based resources such as starch, agar, and cellulose. Starch, which is produced as an energy store by many green plants, has been considered an appealing and ideal candidate to replace petroleum-based materials. Starch has been considered as the most promising resource for biopolymer development due to its advantages, including the potential of starch in the development of rigid materials, namely thermoplastic starch (TPS) (Jumaidin et al., 2017). Among other known advantages of starch includes its inherent biodegradability, widespread availability by which the raw materials are obtainable even at remote places and also for its low cost production. (Teixeira et al., 2007). Starch can be obtain from variety type of crop like tapioca, corn and potatoes.

Glycerol is used as a plasticiser to produce starch-based biodegradable films where Plasticisers create greater flexibility in the polymer structure by reducing the intermolecular forces and the glass transition temperature of the material, which increases the mobility of the polymer chains in the starch films (Bassi et al., 2020). Glycerol can improve the solubility, lightness, and water absorption of starch films made by casting, as well as create more compact structures because glycerol able to decreased the water vapour permeability of starch films by up to 30%, and more glycerol increased it. Glycerol reduced tension at break and Young's modulus in starch films, but increased elongation. Glycerol can help starch films enhance their mechanical properties (Farahnaky et al., 2013).

## 1.2 Problem Statement

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Petroleum based plastics have been widely used throughout the world. With increased applications, the disposal of waste plastics has become a major problem as it contains toxic chemicals that will harm the earth, air, and water. The accumulation of petroleum-based plastic wastes has caused serious environmental problems because they are neither biodegradable nor renewable (Alkbir et al., 2016). Typical petroleum-based plastic takes a long time to degrade due to the molecular bonds that make the plastic so durable and equally resistant to natural processes of degradation (Aini, 2010).

In order to overcome these problems, several studies are concentrated on the development of new biodegradable matrixs which can be derived from any natural resources.

Several types of bio-based polymers have been developed from natural resources, and starch is one of the most promising materials due to its renewability and widespread availability at a low cost. Polysaccharides such as starches, for example, have many advantages over synthetic polymers in the plastics industry, including low cost, non-toxicity, biodegradability, and availability (Šimkovic, 2013). Starch is a polymeric carbohydrate made up mainly of two biopolymers: amylose (straight chain) and amylopectin (branched chain). However, since starch-based films are brittle and hydrophilic, their processing and application are limited. Starch can be combined with a variety of synthetic and natural polymers to overcome these disadvantages (Manohar, 2019).

#### **1.3 Research Objective**

The main aim of this research is to investigate the mechanical and thermal behaviour of biodegradable matrix derived from corn starch. Specifically, the objectives are as follows:

- a) To fabricate the composite matrix using biodegradable derived from corn Ustarch_RSITI TEKNIKAL MALAYSIA MELAKA
- b) To understand the fundementals mechanical behaviour of corn starch

#### 1.4 Scope of Research

The scope of this research are as follows:

- Preparation of matrix sample test derived from corn starch mix with glycerol as plasticizer.
- Conducting tensile test onto the sample to determine the tensile strength of the material.
- Conducting flexural testing onto the sample to determine the stiffness of the material.
- Conducting morphological properties by performing Scanning Electron
   Microscope (SEM) to reveal the detailed surface characteristics of a specimen and provide information relating to its three-dimensional structure

#### **CHAPTER 2**

#### LITERATURE REVIEW

#### 2.1 Introduction

Plastic production and consumption have been increasing globally for more than 50 years. In 2019, the global production of plastics reached 368 million metric tonnes, with Europe producing 57.9 million metric tonnes of that total. Plastics are high molecular mass, synthetic organic polymers derived from hydrocarbons extracted from crude oil and natural gas, and they are used for a wide range of applications. More than one third of the entire mass of plastics produced globally is used to make packaging, which typically is not recycled, but instead ends up as waste (Rhodes, 2018).

With such big number of plastic production recorded, it will give negative impacts to the global environment. Plastic pollution can afflict oceans, land, and waterways. It is estimated that about 1.1 to 8.8 million metric tons of plastic waste enters the world ocean from coastal communities every year (Godswill & Gospel, 2019). Ingestion, suffocation, and entanglement of hundreds of marine species are the most apparent and unpleasant effects of marine plastics. Marine wildlife such as seabirds, whales, fishes and turtles, mistake plastic waste for prey, and most die of starvation as their stomachs are filled with plastic debris.

#### 2.2 Composite

#### 2.2.1 Introduction to composite

One of the earliest uses of composite material was by the ancient Mesopotamians around 3400 B.C. (Šerifi et al., 2018), when they glued wood strips at different angles to create plywood. The concept of "composite" building construction has existed since ancient times where civilizations throughout the world have used basic elements of their surrounding environment in the fabrication of dwellings including mud/straw and wood/clay. This clearly shows that composite has existed among us and played an important role throughout human history, from housing early civilizations to enabling future innovations.

Most composites nowadays provide a lot of benefits compared to traditional material where composite offers industry a world of new opportunities. Composite basically are combination of components or in other word consist of two or more different materials that become stronger when combined. Furthermore, the material used for the combination involves in different properties which result in unique composite properties (Ngo, 2020). The term composite covers a wide range of material combinations, including agglomeration, surface coating or reinforced materials.

Generally, a composite material is composed of reinforcement (fibers, particles, flakes, and/or fillers) embedded in a matrix (polymers, metals, or ceramics). The matrix holds the reinforcement to form the desired shape while the reinforcement improves the overall mechanical properties of the matrix. When designed properly, the new combined material exhibits better strength than would each individual material (Nagavally, 2017).

Compared to regular materials, composite materials have numerous characteristics that improve or overcome the weakness of a single material which fully give advantages to each material and provide the material new properties. The physical properties of the composite are not isotropic in nature but typically they are anisotropic depend on the direction of force application.

#### 2.2.2 Classifications of composite

Composite material is a material composed of two or more distinct phases. The two phases can be classify as matrix phase and dispersed phase (Nielsen, 2005). Matrix is the primary phase, having a continuous characteristic which usually ductile and less hard phase. The secondary phase (or phases) is embedded in the matrix in a discontinuous form called dispersed phase. Dispersed phase is usually stronger than the matrix, therefore it is sometimes called reinforcing phase.

In addition, composites can classify by the type of material used for the matrix. According to Ibrahim, (2015) composite materials are classified into three types: polymer– matrix composites, metal–matrix composites, and ceramic–matrix composites, and they are widely used in a variety of engineering applications, as illustrated in Figure 2.1.





al., 2015)

Depending on the reinforcement type, composite materials can be classified into particu- late composites, fibre-reinforced composites and struc- tural composites shown in Figure 2.2





#### 2.3 Matrix

#### 2.3.1 Introdution to matrix

The matrix is basically a homogeneous and monolithic material in which is embedded. Matrix is completely continuous and provide medium for binding and holding reinforcements together into a solid. It offers protection to the reinforcements from environmental damage, serves to transfer load, and provides finish, texture, color, durability, and functionality. The role of the matrix is to keep the reinforcement particles in place and to support them. In general, reinforcements affect mechanical and physical characteristics or on any other tailored characteristics improved from the matrix material (Sharma et al., 2020).

#### 2.3.2 Type of matrix

There are three main types of composites developed and widely used in numerous kinds of applications known as polymer matrix composites (PMC), ceramic matrix composites (CMC), and metal matrix composites (MMC) as illustrated in Figure 2.3.



2.3.2.1 Polymer matrix composites (PMCs)

A polymer matrix composite (PMC) is a composite material made up of a variety of short or continuous fibres held together by an organic polymer matrix. Currently, PMCs or known as Polymer matrix are commonly used composite and very popular due to their low cost and simple fabrication methods (Goda et al., 2013). The matrix in PMCs is typically reinforced with ceramic fibres due to their high strength in comparison to the matrix material. The matrix, reinforcement, process parameters, microstructure, composition, and interphase are all factors that influence the characteristics of PMCs. Polymer is a large molecule composed of repeating structural units connected by covalent chemical bonds. PMC's consist of a polymer matrix combined with a fibrous reinforcing dispersed phase. They are cheaper with easier fabrication methods (Sharma et al., 2020) Many commercial polymers are 'filled' by blending with solid particles, most commonly glass or minerals such as calcium carbonate (chalk), talc or clay, or carbon black (especially in rubbers) as it offer benefits in terms of cost reduction, improved processing, density control, optical effects, thermal conductivity, control of thermal expansion, electrical properties, magnetic properties, flame retardancy and improved mechanical properties, such as hardness and tear resistance (Ashby et al., 2012).

PMCs can classified into two type known as thermoplastics and thermosets based upon different types of matrix material as shown in Figure 2.4. Thermoplastics and thermosetting plastics are two different classes of polymers characterised by their behaviour in the presence of heat. The difference between the two is that thermoplastics can be remelted, whereas thermoset plastics are permanently solid once hardened (Osborne Industries Ltd., 2017).



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Figure 2.4 Classification of Polymer Matrix Composites (Singh et al., 2019).

#### 2.3.2.2 Ceramic matrix composites (CMCs)

Ceramic Matrix Composites are a mixture of ceramic particulates, fibers and whiskers with a matrix of another ceramic and may be defined as solid materials that normally show highly strong bonding generally ionic, but in a few cases, it may be covalent (Sharma et al., 2020). Ceramic matrix composites (CMCs) are a subgroup of composite materials as well as a subgroup of technical ceramics consist of ceramic fibers embedded in a ceramic matrix, thus forming a ceramic fiber reinforced ceramic (CFRC) material (Chaturvedi et al., 2021). CMC materials were designed to overcome the major disadvantages such as low fracture toughness, brittleness, and limited thermal shock resistance, faced by the traditional technical ceramics.

#### 2.3.2.3 Metal matrix composites (MMCs)

A metal matrix composite (MMC) is composite material with at least two constituent parts, one being a metal necessarily, the other material may be a different metal or another material, such as a ceramic or organic compound. Metal matrix composites (MMCs) usually consist of a low-density metal, such as aluminum or magnesium, reinforced with particulate or fibers of a ceramic material, such as silicon carbide or graphite. MMCs offers higher specific strength and stiffness, a higher operation temperature, and better wear resistance than unreinforced metals, as well as the ability to tune these qualities to a specific application (Lynch & Kershaw, 2018). The metal matrix is reinforced with the other material to improve strength and wear. In structural applications, the matrix is usually composed of a lighter metal such as magnesium, titanium, or aluminum (Sharma et al., 2020).

#### 2.4 Starch

#### 2.4.1 Introduction to starch

Biodegradable polymers play a very important role in plastic engineering by replacing non-biodegradable and nonrenewable petrol-based polymers. Starch is a renewable, biodegradable, low-cost natural polymer with high availability. Natural polymers can be blended with synthetic polymers to improve their properties significantly (Pokhrel, 2015). Due to its high inherent biodegradability, abundance, and annual renewability, starch is the most promising natural polymer that can be easily modified (Lomelí Ramírez et al., 2010). Therefore, it is not surprising that starch and its derivatives have received increased attention as biodegradable alternatives to conventional petroleum-based plastics.

Among these, starch is a potentially useful material for biodegradable plastics because of its natural abundance and most abundant biopolymers. Starch is the major carbohydrate in plant tubes and seed endosperm, where it is found as granules. Each granule contains several million amylopectin molecules accompanied by a much larger number of smaller amylose molecules. The largest source of starch is corn and the other commonly used sources are wheat, potato, and rice.

Generally, corn starch consists of 20-30% amylose and 70-80% amylopectin (Gáspár et al., 2005). Starch is a polysaccharide produced by the green plant as the energy storage. Starch possess an interesting characteristic where it can be transformed into plastic in the presence of plasticizer and heat (Ridhwan Jumaidin et al., 2018). Figure 2.5 displayed industrial starch image.



**Figure 2.5 Industrial starch** 

#### 2.4.2 Structure of Starch

Starch is a mixture of two glucose polymers. These polymers are initially enclosed within a semicrystalline granule formed inside starch-synthesizing plant organelles (Jackson, 2003) .The linear and helical amylose and the branched amylopectin are the two types of molecules present in starch (Sanyang et al., 2017). Starch exists in the form of granules, each of which consist of several million amylopectin molecules together with an even larger number of amylose molecules. From (Pérez & Bertoft, 2010) research, most starch granules are produced individually in separate amyloplasts but there are cases where more than one granule is produced simultaneously in a single amyloplast, consequently they are more difficult to separate.

In general, normal starches contain around 20–30 % of amylose and 70–80 % of amylopectin depending on source of plant as reported by Svihus, (2005). Amylose is a polymer of low molecular weight which consists of linear chains bound by  $\alpha$ -1,4 glycosides links and behaves essentially as a non-branched molecule. In Figure 2.6 shows that amylose is a linear polymer composed of D-glucose residues linked by a-1,4 bonds. Based on Pérez-

Pacheco, (2016) amylose molecules consist of approximately 200–20,000 glucose units joined by  $\alpha$ -1,4 glycoside bonds in unbranched chains or coiled helix displayed in Figure 2.7.



Figure 2.6 Structure of Amylose (Joye, 2018).

Figure 2.7 Segment of an amylose molecule (Pérez-Pacheco et al., 2016).

The molecular structure of the major, extensively branched starch component is considerably more complex than that of amylose because amylopectin is considered as as a much larger macromolecule than amylose (Pérez & Bertoft, 2010). Joye, (2018) state that the building blocks of amylopectin are also a-D-glucose units, but in this case, a significant portion of the linkages are a-1,6 bonds (5–6%), giving the molecule a branched structure shown in Figure 2.8. From Figure 2.9 the glycosidic bonds join the glucose molecules in the main amylopectin chain. Branches of the main chain are often found, which are due to the  $\alpha$ -1,6 glycosidic bonds with other glucose molecules (Pérez-Pacheco et al., 2016).



Figure 2.8 Structure of Amylose (Joye, 2018).



Figure 2.9 Segment of an amylopectin molecule (Pérez-Pacheco et al., 2016).

#### 2.4.3 Characteristic of starches

Pure starch is white in color. The starch powder does not possess any specific taste or odor. Furthermore, pure starch cannot be dissolved in cold water or alcohol. It is nontoxic, biologically absorbable, and semi-permeable to carbon dioxide (Marichelvam et al., 2019).

High-amylose starch produces strong films and is suitable for making biodegradable plastics, and smallgranule starch is an appropriate polyethylene film filler,

Various physical, chemical, and enzymatic modifications will change and improve functional properties of starch and facilitate its utilization for different purposes (Jane, 1995)

#### 2.4.4 Type of starch

Starch can be obtained from a great variety of botanical species, which determines the compound's grain size, size distribution, shape, amylose/amylopectin ratio, molecular weight, and phosphate monoester content (Alkbir et al., 2016). Starch is stored most abundantly in tubers (e.g., potato), roots (e.g., the sweet potato), the grains of cereals (e.g. wheat, corn, barley) and fruits (Mira, 2006). According to (Pfister & Zeeman, 2016) starches from different botanical sources vary in terms of their functional properties such as gelatinization onset temperature, final viscosity of paste, formation of two phase pastes or paste stickiness and thus in their end-uses. Table 2.2 shows the amylose and amylopectin concentration of various starch sources (Marichelvam et al., 2019).

Cassava is highly tolerant to harsh climatic conditions and has great productivity on marginal lands. The supply of cassava starch, the major component of root, is thus sustainable and cheap (Zhu, 2015). Cassava starch also have several other numerous uses such as an additive in cement to improve the setting time, and it is used to improve the viscosity of drilling muds in oil wells. It is also used to seal the walls of bore holes and prevent fluid loss. In the glue and adhesive industries, starch is also a main raw material where cassava starch is being utilized as a glue in paper manufacture to achieve brightness and strength.

Sago is a type of starch that is commonly extracted from a palm called Metroxylon sago, tropical Southeast Asian plant that is native to Malaysia, Indonesia, Papua New Guinea, and Thailand. Growing in a suitable environment with organized farming practices, sago palm could have a yield potential of up to 25 tons of starch per hectare per year. Sago starch yield per unit area could be about 3 to 4 times higher than that of rice, corn, or wheat, and about 17 times higher than that of cassava (Karim et al., 2008).

Sugar palm also known as Arenga pinnata is the most important sugar palm of the humid tropics. Besides yielding sugar, it provides a great number of products and benefits to its users and is one of the most diverse multipurpose tree species in culture. Among the numerous green material sources in Malaysia, the sugar palm tree is a versatile plant that can produce biofibers, biomatrices, and bio composites for a wide range of applications (Sahari et al., 2012) . Table 2.1 shows amylose and amylopectin concentration of various starch sources.

Table 2.1 Amylose and amylopectin concentration of various starch sources.

E		
Source	Amylose (%)	Amylopectin (%)
Arrowroot	20.5	79.5
Banana - U	17- 9- 6	83
Cassava	KAL MA18.61 SIA M	ELAKA81.4
Corn	28	72
Potato	17.8	82.2
Rice	35	65
Tapioca	16.7	83.3
Wheat	20	80

(Marichelvam et al., 2019)

#### 2.4.5 Corn starch

Corn is the largest source of commercial starch in the world and is the basis of a wide range of chemical-, enzyme-, and physical-modified functional products, as well as hydrolyzed maltodextrins and syrups. Corn (maize) is one of the most abundant sources of plant residues, which as biomass provides multiple advantages such as high concentration of starch, excellent consistency, cost-effectiveness, availability, and biodegradability (Mendes et al. 2016).

Corn plant is the predominant sources of native starches commercially available where almost more than 85% of starch production in the world is extracted from corn tree (Ibrahim et al., 2019). According to (BeMiller, 2019), two common commercial corn starches, called high-amylose corn starches and amylomaize starches, have apparent amylose contents of 50%-60% and 70%-80%, respectively.

Table 2.2 reveals the chemical composition of corn starch. The comparative analysis of composition indicated that corn starch is characterized by a relatively high concentration of polysaccharides, (amylopectin and amylose), while the amounts of extractives, such as crude fats, protein, and ash, were quite low.

Content	Amount	Units
Amylose	24.64	g/100g
Amylopectin	75.36	g/100g
Crude Fat	7.13	g/100g
Crude Protien	7.70	g/100g
Ash	0.62	g/100g
Phosphor	0.09	%
Moisture content	10.45	%
Density PLAYSIA	1.4029	g/cm ³

Table 2.2 Chemical composition of corn starch (Ibrahim et al., 2019).

#### 2.4.6 Advantage & Disadvantage of Starch

Starch has several advantages including its renewability, strong oxygen barrier in the dry state, abundance, low cost, and biodegradability. In plastic manufacturing, starch has strong oxygen barrier as result of the structure that is compact and low solubility (Hatmi et al., 2020). Other than that, starch is a renewable natural product with a host of advantages: low density, cost-effectiveness, abundant supply, and environmental amity (Tang et al., 2006).

Despite their numerous benefits, such as biodegradability, availability, recyclability, and low cost, starch-based materials are also known to have several drawbacks, including high water sensitivity (hydrophilic character) and lower mechanical performance when compared to traditional industrial polymers (Averous & Boquillon, 2004). Therefore, plasticizers need to enhance the mechanical performance of these biomasses due to its role

to reduce strong attraction of hydrogen bonds within amylose and amylopectin molecules in the starch network (M. I. J. Ibrahim et al., 2019b).

#### 2.5 Plasticizer

#### 2.5.1 Introduction of plasticizer

The use of plasticizers for plastic products manufacture is not a new practice considered the application is to modify polymer characteristics began in the 1800s (Vieira et al., 2011). Based on Sejidov et al., (2005), plasticizers are an important class of low molecular weight non-volatile compounds that are widely used in polymer industries as additives. Plasticizers can be define as additives that able to increase plasticity of a substance while decreasing its viscosity and this additives usually added into a substance purposely to modify the propeties of the substance (Ester et al., 2014). According to Rahman & Brazel, (2004) the plasticizer's primary function is to improve the mechanical characteristics of polymers by increasing flexibility, lowering tensile strength, and decreasing the second order transition temperature. Plasticizers are typically inert organic compound with low molecular weights, high boiling temperatures, and low vapour pressures (Wojciechowska, 2012). It is a solid with low volatility that is used to toughen and flexibilize a plastic basis or soften it at practical temperatures in some rare cases (Misra, 1993). A research by Di Bella et al., (2010) conclude that plasticizer contents can vary from 3.0 to 80% by plastic weight which produces a considerable effect on material properties which makes the plasticizers migrate to plastic article surface carrying other components of the composition (e.g., stabilizers) with them.

#### 2.5.2 Classification of plasticizer

Generally, plasticizer can be divided into internal and external plasticizers. There are numerous classes depending on the variety of a plasticizer's chemical structure and molecular weight, as well as the type of plasticization of the initial polymer as shown in Figure 2.10. Rahman & Brazel, (2004) state that external plasticizer molecules are not attached to polymer chains by primary bonds and can therefore be lost by evaporation, migration or extraction. Internal plasticizers, on the other hand, are an inherent part of the polymer chain and can be reacted with the native polymer or co-polymerized into the polymer arrangement (Tyagi & Bhattacharya, 2019). External plasticizer later divided into two categories called primary and secondary plasticizer shown in Figure 2.10.

Primary plasticizers are compounds containing polar groups and are characterized by high solvation capabilities with PVC. They have the ability to efficiently increase polymer flexibility. A primary plasticizer is used as the sole plasticizer and is generally miscible with the polymer in all proportions (Langer et al., 2020). Secondary plasticizers are used in smaller quantities in a blend with a primary plasticizer in order to improve a specific property such as low temperature flexibility, reduced flammability, improved processing, or reduced costs (Godwin, 2017). Extenders are a subset of secondary plasticizers, which although relatively incompatible with the original polymer, extends the material or fills out its bulk at a low cost without causing a significant loss of flexibility as would occur if using a solid filler as an extender (Langer et al., 2020).



Figure 2.10 Classification of Plasticizer (Langer et al., 2020)

#### 2.5.3 Glycerol

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Glycerol or glycerin is a simple polyol compound with physical properties of colorless, odorless, viscous liquid that is sweet-tasting and non-toxic. According to (Quispe et al., 2013) glycerol is an oily liquid; it is viscous, odorless, colorless, and has a syrupy-sweet taste. Aside from that glycerol also known as propane-1,2,3-triol according to IUPAC. (Rahmat et al., 2010) record that other commercial name for glycerol is 1,2,3-propanetriol, 1,2,3-trihydroxypropane, glyceritol or glycyl alcohol.

Due to three hydroxyl group, glycerol dissolve easily in water and triglyceride formed when combined with fatty acid where it is the main constituent of fats and oils which is found naturally in plants and animals (Mbamalu, 2013). Figure 2.11 and Figure 2.12 exhibits the chemical structure of glycerol.



Figure 2.11 Chemical Structure of glycerol (Mbamalu, 2013)



Previous studies recorded that glycerol and sorbitol does giving the plasticization effects on various starches from different source to prepare biodegradable or edible film. The most common plasticizer utilized for starch-based films are polyols such as glycerol and sorbitol, amongst many others (Sanyang et al., 2015).

#### **CHAPTER 3**

#### METHODOLOGY

#### 3.1 Introduction

This chapter will covers about the proposed methodology that will be implemented throughout the research development. This chapter is consist of project planning and process flow of the research to ensure the research will finished on time to complete this project with the correct guideline.

Chapter three will discuss about the scope of the projects in order to achived the objective of this research which include some other processes such as raw material selection, material preparation and fabrication and lastly material tensting.

The purposes of material testing is to investigate the mechanical and thermal behaviour on biodegradable matrix derived from corn starch. The selected material testing fot this research is Tensile Testing, Flexural Testing and Scanning electron microscope (SEM).

#### 3.2 Flow Chart



**Figure 3.1 Process Flow Chart** 

#### 3.3 Raw Material

Starch is one of the most promising natural polymers because of its inherent biodegradability, overwhelming abundance and annual renewability. For this research, biodegradable matrix derived from corn starch has been chosen as the main raw material. As for plasticizer, glycerol with 99.5% glycerol concerntration were used. Both of the material was obtained from Polyscientific Enterprice Sdn. Bhd, a local supplier based in Malacca, Malaysia.



UNIVERS Figure 3.2 Industrial Cornstarch MELAKA



Figure 3.3 Glycerol (Plasticizer)

#### **3.4** Material Preparation and Fabrication

During material preparation based on Figure 3.7, both thermoplastic starch and glycerol must be weight using different type of ratio before mixing it alltogether. Then, the mixture of thermoplastic cornstarch and glycerol were mix together using either hand or high speed blender for about 2 to 3 minutes. The mixture then weight again using digital scale before putting the mixture into the mould with the dimension of 140 x 60 mm shown in Figure 3.6. After prepping the mixture inside the mould, the mould will be put into the hot press machien shown in Figure 3.4 for pre – heating process for about 15 minutes. After that, the mould later hot press at pressure of 25 kg/cm2 with 165°C of temperature for 15 minutes followed by cooling process for another 15 minutes. Lastly, the mould then removed by H frame 10 tonne machine in order to the get the final sample. Before testing, the finalize sample will be cut into specific testing size by using table saw cutting machine.



Figure 3.4 Hot press machine



Figure 3.5 Compression Moulding Machine (Hot Press) Tool D



Figure 3.6 Dimension of Compression Moulding Machine Tool Die



Figure 3.7 Step by step Material Preparation



Figure 3.8 Final Sample

3.4.1 Cutting Process

After cooling process, the material will be cut into specific sample sizes based on the test that will be conducted. A cutting machine will be used to cut the materials into desired sample size.



Figure 3.9 Cutting machine



Figure 3.10 Example of final product sample

### 3.5 Material testing

#### 3.5.1 Tensile Testing

The tensile test specimen was prepared and conducted according to ASTM D638 (American Society for Testing and Materials, 2016). Four specimens as shown in Figure 3.11 will be tested using dimensions of 140 mm (L) × 13 mm (W) × 3mm (T) and later mounted in the grips of the Universal Testing Machine (Shimadzu AGX-V) Figure 3.12. Tensile test conducted at the temperature of  $23 \pm 1$  °C and relative humidity of  $50 \pm 5\%$  with 5 kN load cell with speed testing of 5mm/min.



Figure 3.11 Example of specimen tested



Figure 3.12 Tensile test using Universal Testing Machine (Shimadzu AGX-V)

## 3.5.2 Flexural Testing

A flexural test was conducted according to ASTM D790 (ASTM International, 2002) performed by the three-point bending method using Universal Testing Machine (Shimadzu AGX-V) Figure 3.13. The test sample or specimen were prepared with dimensions of 140 mm (L)  $\times$  13 mm (W)  $\times$  3mm (T) at crosshead speed of testing was 2 mm/min and load test 5 kN.



Figure 3.13 Flexural Test using Universal Testing Machine (Shimadzu AGX-V)

#### 3.5.3 Scanning Electron Microscopy (SEM)



Figure 3.14 Scanning Electron Microscope (Zeiss Evo 18)

A previous specimen from tensile test will be used for Scanning Electron Microscope by using Scanning Electron Microscope (Zeiss Evo 18). The specimen will be cut into tiny pieces shown in Figure 3.15 and coated with platinum according to (Heu et al., 2019). After coating, this test conducted to examine the morphology of tensile fractured surfaces with 10kv of acceleration voltage.



Figure 3.15 Tiny Specimen used for SEM

#### **CHAPTER 4**

#### **RESULT AND DISCUSSION**

#### 4.1 Introduction

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This chapter discussed about the results and findings based on observation from day one and testing that had been conducted to the final sample produced. There are three results will be discussed in this chapter which is involving mechanical testing and thermal testing. For mechanical testing, tensile test and flexural test has been conducted while for physical and morphological test will involving Scanning Electron Microscope (SEM)

Three sample from each result has been carried out with different type of ratios and labelled as A, B, C based on Table 4.1. Label A represent concentration of 60% thermoplastic cornstarch and 40% glycerol. Label B represent concentration of 65% thermoplastic cornstarch and 35% glycerol. Lastly label C represent concentration of 70% thermoplastic cornstarch and 30% glycerol.

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Table 4.1 Label of each ratio

LABEL	А	В	С
RATIO	60:40	65:35	70:30

#### 4.2 Mechanical Testing

#### 4.2.1 Tensile Test



### Figure 4.1 Tensile strength of thermoplastic cornstarch

Tensile test performed in order to determine the reaction of the materials to resist as forces are applied in stress consists of tensile properties. Tensile properties frequently are included in material specifications to ensure quality and used to predict the behavior of a material under forms of loading other than uniaxial tension (Davis, 2004). This test was conducted to precisely determine the tensile performance stress in various concentrations of starch and plastisizer.

Figure 4.1 shows the result of tensile strength for each ratio of thermoplastic corn starch and glycerol where ratio A was in the range of 0.64 MPa while ratio B in the range of 0.91 MPa and lastly ratio C in the range of 1.24 MPa. (Hazrol et al., 2021) stated that by reducing the plasticizer concentration from 40% to 30%, it will increase the tensile strength of the tested specimen. The expected interpretation of the high tensile stress at low plasticizer content is related to hydrogen bonds formed between starch and plasticizer molecules; these

bonds are strongly dominated at low plasticizer content and weaken as plasticizer content increases (Ibrahim, 2019). Therefore, ratio C with concentration of 70% thermoplastic cornstarch and 30% glycerol sustain high load before failure compared to the other two ratio.



Figure 4.2 Tensile Modulus of thermoplastic cornstarch

Young's modulus or modulus of elasticity is a material parameter that describes stiffness and is hence one of the most important qualities of solid materials (Elasticity, 2005). UNVERSITE TEXNIKAL MALAYSIA MELAKA (Ibrahim et al., 2019) also stated that in terms of elastic modulus (Young's modulus), where it determines the stiffness of materials, the higher the elastic modulus value, the greater the stiffness.

Figure 4.2 above shows the result of elastic modulus of each ratio of thermoplastic cornstarch where the result recorded the same trend with tensile strength result. Ratio A in the range of 2.2 MPa. Ratio B was in the range 18.89MPa while the highest elastic modulus is in the range 58.94 MPa. From the result, rato C has the highest elastic modulus while ratio A has the lowest elastic modulus.

The amount of plasticizer concentration may effect stiffness of the specimen where the specimen stiffness increases as the plasticizer concentration decreases from 40% to 30% based on the result. Plasticizers' role in modifying starch network structure, as stated by (Hazrol et al., 2021), can be explained by the fact that when they are incorporated into starch chains, they facilitate the formation of hydrogen bonds between molecules while weakening the solid intramolecular attraction within the starch matrix. Therefore, ratio C has the most stiffness compare to the other two ratio.



#### 4.2.2 Flexural Testing

**Figure 4.3 Flexural Strength of thermoplasic cornstarch** 

A flexural test is most commonly used to determine flexural strength, which is defined as the maximum stress at the outermost fibre on either the compression or tension side of the specimen. (Alfredo, 2008) state that flexural strength is a material's ability to withstand bending forces applied perpendicular to its longitudinal axis, where the stresses induced by the flexural load are a combination of compressive and tensile stresses.

Three sample of specimen has been prepared to conduct this test with different concentration of thermoplastic starch and glycerol. The test has been performed three time with each different type of ratio starting from 60wt% to 70wt%. Figure 4.3 above shows result of each specimen where based on the result, ratio A with 60wt% of starch recorded 0.83 MPa while ratio B and ratio C result with concentration of 65wt% and 70wt% recorded 3.86 MPa and 3.71 MPa.

The highest value of flexural strength is 3.86 MPa with concentration of 65wt% of thermoplastic corn starch. Therefore, ratio B has the higher value of flexural strength. The lowest flexural strength is at value 0.83 MPa (ratio C) with concentration of 60wt% of thermoplastic corn starch.

4.3 Morphological properties

#### 4.3.1 Scanning Electron Microscope (SEM)





Figure 4.4 SEM micrograph of tensile fracture surface

A scanning electron microscope uses a finely focused beam of electrons to reveal the detailed surface characteristics of a specimen and provide information relating to its three-dimensional structure (Holgate, 2017). Based on research by (Horovitz et al., 2011), scanning electron microscopy (SEM) test proved to be valuable method for surface characteristic of starch.

For this research, scanning electron microscopy (SEM) conducted to evaluate tensile fracture behaviour. Figure 4.4 above shows the result of (SEM) tensile fracture surface of thermoplastic cornstarch and glycerol with three type of ratio under magnification of 500x where thermoplastic starch content in (A) is 60wt%, thermoplastic starch content in (B) is 65wt% and thermoplastic starch content in (C) is 70wt%.

As a result, the surface of the thermoplastic cornstarch content 60wt% micrograph has a few cracks and a large fracture groove. Regarding the thermoplastic cornstarch content of 65wt %, the micrograph from the surface of the tensile tested sample indicated cracks and a few voids. Finally, the micrograph from the surface of the tensile tested sample for thermoplastic cornstarch content 70% wt contains smaller cracks and a few voids.

From the result observation, the specimen with higher contain of starch tend to be ductile, hard and easily to break and for the lower contain of starch tend to be brittle, sticky and the specimen is difficult to be removed from the mould. Thus, different concentration of thermoplastic corn strach may affect the ductile and brittle characteristic of the specimen.

#### **CHAPTER 5**

#### CONCLUSION AND RECOMMENDATION

#### 5.1 Conclusion

Nowadays, bioplastic sources are receiving a lot of attention because they are made mostly of natural materials. Moreover, bioplastic was indeed biodegradable and derived from renewable raw materials. The thermoplastic starch (TPS) is currently the most widely used bioplastic, either alone or in mixture with natural or synthetic polymers. TPS has some limitations, most of which are related to its high hydrophilicity, low processability, and tendency to increase brittleness (Paiva et al., 2018).

For this research, different variety and concentrations of thermoplastic cornstarch and glycerol (plasticizer) were used to analyze the mechanical behaviour of thermoplastic cornstarch. Different type of concertration of thermoplastic cornstarch and glycerol may affect the characteristic and mechanical behaviour of the specimen.

#### 5.2 **Recommendation**

There are a few recommendations provided after completing the research;

i. Study the mechanical properties introducing varies type of plasticizer such as sorbitol, fructose, urea and other related plasticizer.

ii.Enhancing the properties of thermoplastic cornstarch and glycerol by adding fibre such as kenaf, pineapple and other related fibre.

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