



**UNIVERSITAS INDONESIA**



**UNIVERSITE DE BRETAGNE-SUD**

**MECHANICAL PROPERTIES OF PLA AND FLAX FIBRES  
FOR NATURAL FIBRE REINFORCED COMPOSITES**

**TESIS**

**MUHAMMAD SUBHAN  
0906579286**

**FAKULTAS TEKNIK  
PROGRAM STUDI TEKNIK MESIN  
DEPOK  
JANUARI 2012**



University of Indonesia



Université  
de Bretagne-Sud

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**Diajukan sebagai salah satu syarat untuk memperoleh gelar Magister Teknik**

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## KATA PENGANTAR

Puji syukur saya panjatkan kepada Tuhan Yang Maha Esa, karena atas berkat dan rahmat-Nya, saya dapat menyelesaikan tesis ini. Penulisan tesis ini dilakukan dalam rangka memenuhi salah satu syarat untuk mencapai gelar Magister Teknik Program Studi Teknik Mesin Fakultas Teknik Universitas Indonesia.

Saya menyadari bahwa, tanpa bantuan dan bimbingan dari berbagai pihak, dari masa perkuliahan sampai pada penyusunan tesis ini, sangatlah sulit bagi saya untuk menyelesaikan tesis ini. Oleh karena itu, saya mengucapkan terima kasih kepada:

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Penulis



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## ABSTRAK

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Program Studi : Teknik Mesin

Judul : Sifat-sifat Mekanik dari Komposit yang berasal dari Biopolymer  
(PLA) dan diperkuat dengan Serat Alami (Serat Flax)

Dalam dekade terakhir ini, teknologi biopolymer komposit yang diperkuat dengan serat alami semakin menjadi perhatian dengan kelebihan-kelebihannya yaitu biaya murah, performa yang baik dan ringan untuk menggantikan komposit yang berasal dari synthetic polymers atau glass fibre. Pada makalah ini akan dibahas sifat-sifat mekanik komposit yang berasal dari PLA dan serat flax dari serat alami. Komposisi material yang digunakan adalah terdiri dari PLA murni, PLA/10% volume fiber, PLA/20% volume fiber dan PLA/30% volume fiber. Perlakuan material dimulai dari mencampur PLA dan serat flax menurut komposisinya, proses ekstrusi, proses granulasi, dan proses injection molding. Pengujian yang dilakukan adalah pengujian tarik, bengkok, torsi, tekan dan impact.

Dari hasil percobaan menunjukkan nilai modulus dari masing-masing pengujian meningkat dengan bertambahnya volume fiber. Ini menunjukkan bahwa kekakuan dari komposit meningkat dengan bertambahnya volume fiber. Nilai modulus elastisitas yang tertinggi sebesar 7144.33 MPa pada komposit yang mengandung 30% serat flax dan 70% PLA. Hasil ini menunjukkan bahwa biodegradable komposit yang berasal dari serat alami dan biopolymer mempunyai potensi dikembangkan untuk menggantikan komposit yang berasal dari synthetic polymers atau glass fibre.

Kata kunci :

Biodegradable komposit, serat alami, biopolymer, sifat-sifat mekanik





## ABSTRACT

Name : Muhammad Subhan

Study Program: Mechanical Engineering

Title : Mechanical Properties of PLA and Flax Fibres for Natural Fibre  
Reinforced Composite

During the last few years, natural fibre reinforced biopolymer composites technology is focused on creating low cost, high performance, and lightweight materials to replace synthetic polymers or glass fibre composites. In this paper will discuss about the mechanical properties of PLA and flax fibre for natural fibre reinforced composites. Composition observed in this study consisted of pure PLA, 10% fibre volume, 20% fibre volume and 30% fibre volume. Preparation of specimens started from mixing PLA and flax fibre in accordance the composition, extrusion, granulation and injection molding. Testing is carried out a tensile test, flexural test, torsion test, compressive test and impact test.

From research results showed the value of the modulus of each test is increasing with the increasing fibre volume fraction. It shows the stiffness composites increased with the increasing fibre volume fraction. The highest value of modulus of elasticity is 7144.33 Mpa for composites containing 30% flax fibre and 70% PLA. The results showed that biodegradable composites derived from natural fibres and biopolymer have a great potential to be developed as a replacement composite materials derived from synthetic polymers or glass fibre.

Key words :

Biodegradable composites, natural fibre, biopolymer, mechanical properties



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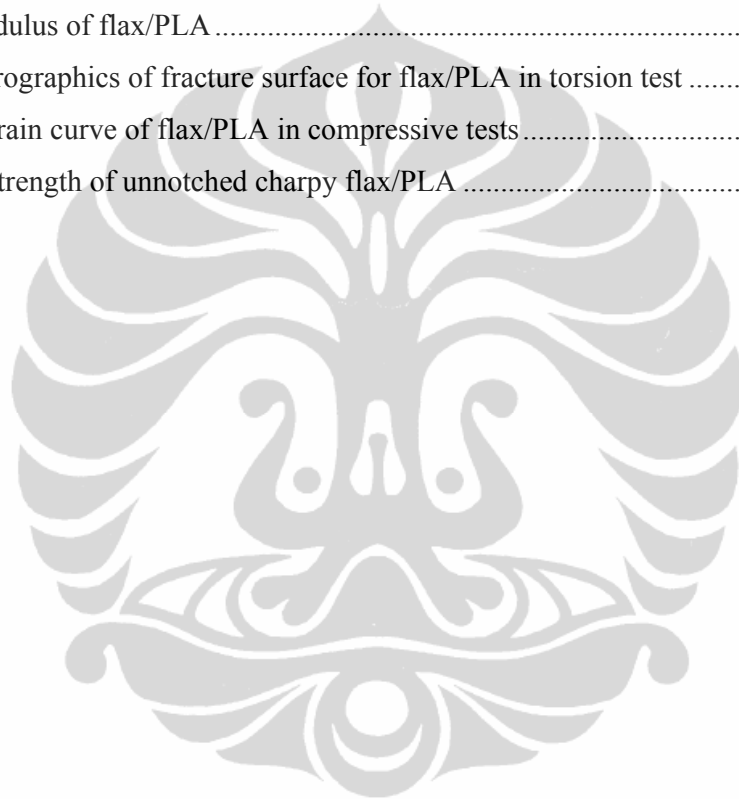
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## CHAPTER I

### INTRODUCTION

Composite materials are engineered materials made from two or more constituent materials with significantly different physical or chemical properties which remain separate and distinct on a macroscopic level within the finished structure. Composites materials are made by combining two materials where one of the materials is a reinforcement (fiber) and the other material is a matrix (resin). The combination of the fiber and matrix provide characteristics superior to either of the materials alone (Museeuw bikes) .

Composite materials have been in existence for many centuries. Some of the earliest records of their use date back to the Egyptians, who are credited with the introduction of plywood and the use of straw in mud for strengthening bricks. Similarly, the ancient Inca and Mayan civilizations used plant fibers to strengthen bricks and pottery. Swords and armor were plated to add strength in medieval times. An example is the Samurai sword, which was produced by repeated folding and reshaping to form a multilayered composite (it is estimated that several million layers could have been used). The pyramids built by the of Ancient Egyptians feature clay bricks and blocks reinforced with straw. Eskimos use moss to strengthen ice in forming igloos. Similarly, it is not uncommon to find horse hair in plaster for enhanced strength. All of these are examples of man-made composite materials. Bamboo, bone, and celery are examples of cellular composites that exist in nature. There are numerous other examples of both natural and man-made composite materials.

Originating from early agricultural societies and being almost forgotten after centuries, a true revival started of using lightweight composite structures for many technical solutions during the second half of the 20th century. Using composites to improve the structural performance of spacecraft and military aircraft became popular in the last two decades of the previous century. It is considered to be one of the first major steps in making aircraft structures primary from composites. Composites are also used in race cars, tennis rackets, golf clubs, and other sports and leisure products. The bicycle industry has seen a rapid increase of composite materials: frames, handlebars, seatposts and so on; every single component could and has been made in this exotic black material. The industry has become saturated with the terms “extremely lightweight” and “extreme stiffness” but the full potential of composite materials is



yet to be discovered. Although composite materials technology has grown rapidly, it is not fully developed. New combinations of fiber resin systems, and even new materials, are constantly being developed.

Over the years, with the discovery and developments of “cheaper” metals, composites have been more and more neglected. However, with the developments of glass fiber, carbon fibers and the discovery of natural fibers, composite technology has gradually moved back into the figure.

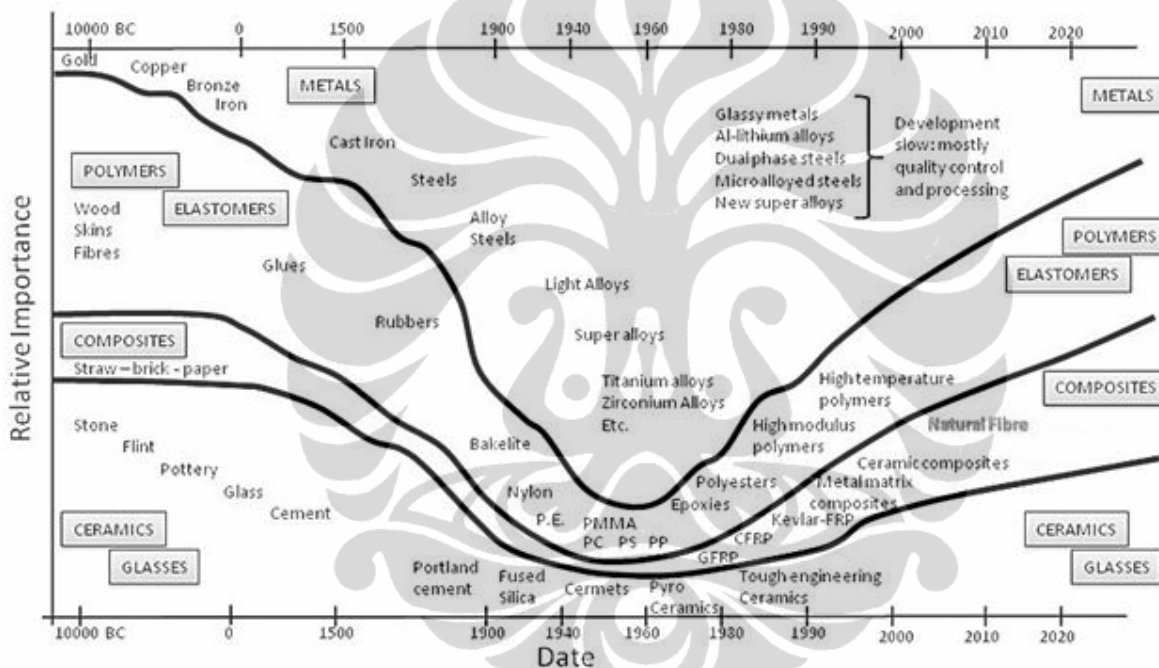


Figure 1.1 Relative importance of materials

The importance of composites has experienced steady growth since about 1960 and is projected to continue to increase through the next several decades. This has been most recently seen in the marked increase in relative importance of structural materials such as composites starting around 1960, when the race for space dominated many aspects of research and development.

The greatest value of composite materials is that they can be both lightweight and strong. The composites generally used in structural applications are best classified as high



performance. They have high strength-to-weight ratios, and require controlled manufacturing environments for optimum performance. Despite their strength and low weight, composites have not been a miracle solution for aircraft structures. Modern airliners use significant amounts of composites to achieve lighter weight. About ten percent of the structural weight of the Boeing 777, for instance, is composite material. Due to this, there have been significant developments in Composite materials. With the technological know-how available today, composite materials are constantly being adapted to the way that they are used. As a result, there are a wide variety of composites to choose from, thanks to the ever-changing technological advances that make it possible to apply composite engineering. As a result, each type of composite brings its own performance characteristics that are typically suited for specific applications.

Recently with the increased awareness of environmental degradation among the people has led the producers as well as consumers towards achieving environmental sustainability. Today the producer is aligned towards producing more environment friendly products and the consumer is more interested in products that bear a green label. One such material is fiber composites. In the past 30 – 40 years fiber composites have been competing with materials such as steel, aluminum and concrete in cars, aircraft, buildings, bridges, bicycles and everyday sports goods. It has such a wide range of application due to its possibility to combine high strength and stiffness with low weight it is non-corrosive and is considered to be less expensive in cost when compared to other material in some cases. They are also reduce the cost over the product's lifetime as they have very low maintenance cost. A lot of work is being done in order to make them more environmental friendly.

Ecological concern has resulted in a renewed interest in natural materials. An interesting environmentally friendly alternative for the use of glass fibres as reinforcement in engineering composites are ligno-cellulosic natural fibres such as flax, hemp, sisal and jute (Narayan, 2001). These fibres are renewable, nonabrasive, can be incinerated for energy recovery since they possess a good calorific value and they give less concern with health and safety during handling of fibre products. In addition, they exhibit excellent mechanical properties, low density and low price. This excellent price-performance ratio at low weight in combination with the environmental friendly character is very important for the acceptance of natural fibres in large volume engineering markets.

## CHAPTER II

### BRIEF BIBIOGRAPHY

#### 2.1 Matrix Materials

##### 2.1.1 Introduction

In matrix-based structural composites, the matrix serves two paramount purposes viz., binding the reinforcement phases in place and deforming to distribute the stresses among the constituent reinforcement materials under an applied force. Figure 2.1 helps to classify matrices :

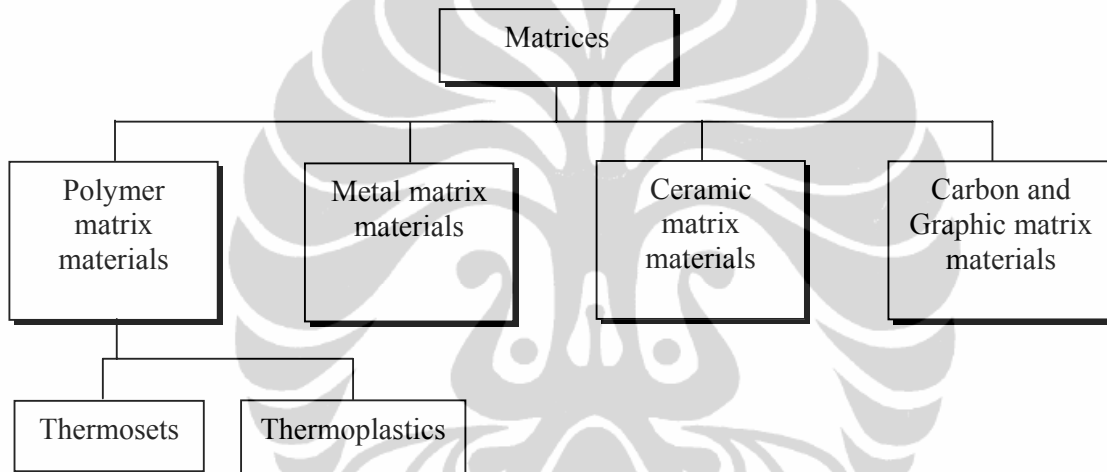


Figure 2.1 Classification of matrix-based structural composites

Polymers make ideal materials as they can be processed easily, possess lightweight, and desirable mechanical properties. It follows, therefore, that high temperature resins are extensively.

Two main kinds of polymers are thermosets and thermoplastics. Thermosets have qualities such as a well-bonded three-dimensional molecular structure after curing. They decompose instead of melting on hardening. Merely changing the basic composition of the resin is enough to alter the conditions suitably for curing and determine its other characteristics. They can be retained in a partially cured condition too over prolonged periods of time, rendering thermosets very flexible. Thus, they are most suited as matrix bases for advanced conditions fiber reinforced composites.

Thermoplastics have one- or two-dimensional molecular structure and they tend to at an elevated temperature and show exaggerated melting point. Another advantage is that the process of softening at elevated temperatures can reversed to regain its properties during cooling, facilitating applications of conventional compress techniques to mould the compounds.

Resins reinforced with thermoplastics now comprised an emerging group of composites. The theme of most experiments in this area to improve the base properties of the resins and extract the greatest functional advantages from them in new avenues, including attempts to replace metals in die-casting processes. Figure 2.2 shows kinds of thermoplastics.

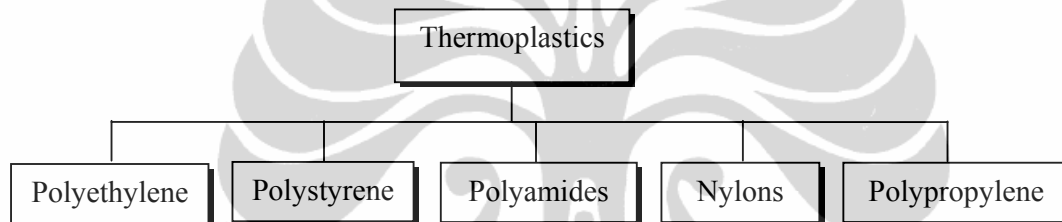


Figure 2.2 Kinds of thermoplastics

The advantage of thermoplastics systems over thermosets are that there are no chemical reactions involved, which often result in the release of gases or heat. Manufacturing is limited by the time required for heating, shaping and cooling the structures.

Thermoplastics resins are sold as moulding compounds. Fiber reinforcement is apt for these resins. Since the fibers are randomly dispersed, the reinforcement will be almost isotropic. However, when subjected to moulding processes, they can be aligned directionally.

Although it is undoubtedly true that the high strength of composites is largely due to the fibre reinforcement, the importance of matrix material cannot be underestimated as it provides support for the fibres and assists the fibres in carrying the loads. It also provides stability to the composite material. Resin matrix system acts as a binding agent in a structural component in which the fibres are embedded. When too much resin is used, the part is classified as resin rich. On the other hand if there is too little resin, the part is called resin starved. A resin rich



part is more susceptible to cracking due to lack of fibre support, whereas a resin starved part is weaker because of void areas and the fact that fibres are not held together and they are not well supported.

In a composite material, the matrix material serves the following functions:

- Holds the fibres together.
- Protects the fibres from environment.
- Distributes the loads evenly between fibres so that all fibres are subjected to the same amount of strain.
- Enhances transverse properties of a laminate.
- Improves impact and fracture resistance of a component.
- Helps to avoid propagation of crack growth through the fibres by providing alternate failure path along the interface between the fibres and the matrix.
- Carry interlaminar shear.

The matrix plays a minor role in the tensile load-carrying capacity of a composite structure. However, selection of a matrix has a major influence on the interlaminar shear as well as inplane shear properties of the composite material. The interlaminar shear strength is an important design consideration for structures under bending loads, whereas the in-plane shear strength is important under torsion loads. The matrix provides lateral support against the possibility of fibre buckling under compression loading, thus influencing to some extent the compressive strength of the composite material. The interaction between fibres and matrix is also important in designing damage tolerant structures. Finally, the processability and defects in a composite material depend strongly on the physical and thermal characteristics, such as viscosity, melting point, and curing temperature of the matrix.

The needs or desired properties of the matrix which are important for a composite structure are as follows:

- Reduced moisture absorption.
- Low shrinkage.
- Low coefficient of thermal expansion.
- Good flow characteristics so that it penetrates the fibre bundles completely and eliminates voids during the compacting/curing process.



- Reasonable strength, modulus and elongation (elongation should be greater than fibre).
- Must be elastic to transfer load to fibres.
- Strength at elevated temperature (depending on application).
- Low temperature capability (depending on application).
- Excellent chemical resistance (depending on application).
- Should be easily processable into the final composite shape.
- Dimensional stability (maintains its shape).

In selecting matrix material, following factors may be taken into consideration:

- The matrix must have a mechanical strength commensurate with that of the reinforcement i.e. both should be compatible. Thus, if a high strength fibre is used as the reinforcement, there is no point using a low strength matrix, which will not transmit stresses efficiently to the reinforcement.
- The matrix must stand up to the service conditions, viz : temperature, humidity, exposure to ultra-violet environment, exposure to chemical atmosphere, abrasion by dust particles, etc.
- The matrix must be easy to use in the selected fabrication process.
- Smoke requirements.
- Life expectancy.
- The resultant composite should be cost effective.

Thermoplastics can be repeatedly softened by heating, and hardened by cooling. They also possess improved damage tolerance, environmental resistance, fire resistance, recyclability and potential for fast processing. Primary reason for the use of thermoplastics is their cost effective processing. Increased use for thermoplastics is also due to three different reasons, viz.

- Processing can be faster than that of thermoset composites since no curing reaction is required. Thermoplastic composites require only heating, shaping and cooling.
- The properties are attractive, in particular, high delamination resistance and damage tolerance, low moisture absorption and the excellent chemical resistance of semi-crystalline polymers.
- In the light of environmental concerns, thermoplastic composites offer other advantages also. They have low toxicity since they do not contain reactive chemicals (therefore storage life is infinite).



Various options exist by which the available matrices may be modified (Ulrich Riedel and Jörg Nickel, 2005) and so the material selection must be adapted to the given requirements. The criteria to select a suitable matrix system for high-performance construction materials include the temperature used, the mechanical loading, and the manufacturing technology. An important requirement for the matrix is an adequately low viscosity to ensure good impregnation of the reinforcing fibers. Additional basic qualities such as good adhesion to the natural fibers must also be presented. Apart from further matrix qualities, the abovementioned criteria are essential for optimum fiber-reinforced composites.

## 2.1.2 Thermoplastics for natural fibre composites

### 2.1.2.1 Synthetic thermoplastics

Synthetic thermoplastics commonly used for natural fibre composites are polyethylene (PE), polypropylene (PP), polystyrene (PS) and polyamides (nylon 6 and 6,6). PP is the most widely used thermoplastic material in the natural fibre composite industry owing to its low density, excellent processibility, good mechanical properties, high temperature resistance, excellent electrical properties, good dimensional stability and good impact strength. The exciting growth of PP in the composite industry is demonstrated by the large quantity of research work and numerous publications devoted to this thermoplastic material. High impact grade polymers of polyolefins and polystyrene (blended with ethylene-propylene rubber, nitrile rubber) are also available to improve the impact strength of the corresponding polymers. Physical, mechanical and thermal properties of these polymers are given in Table 2.1 :

Table 2.1 Properties of commercially important thermoplastic polymers

Property	PP	LDPE	HDPE	PS	Nylon 6	Nylon 6.6
Density (g/cm <sup>3</sup> )	0.899 - 0.920	0.910 - 0.925	0.941 - 1	1.04 - 1.09	1.09 - 1.14	1.090 - 1.19
Water absorption after 24 hours (%)	0.01 - 0.02	< 0.015	0.01 - 0.2	0.03 - 0.10	1.3 - 1.8	1.0 - 1.6
T <sub>g</sub> (°C)	-10 to -23	-125	-133 to -100	-	48	80
T <sub>m</sub> (°C)	160 - 176	105 - 116	120 - 140	110 - 135	215 - 216	250 - 269



Heat deflection temp. $T_d$ , at 1.8 Mpa ( $^{\circ}\text{C}$ )	50 - 63	32 - 50	43 - 60	Max. 220	56 - 80	75 - 90
Coefficient of linear thermal expansion, $\alpha_T$ ( $\text{mm}/\text{mm}/^{\circ}\text{C} \times 10^5$ )	6.8 - 13.5	10	12 - 13	6 - 8	8 - 8.6	7.2 - 9
Tensile strength (MPa)	26 - 41.4	4 - 78.6	114.5 - 38	-	43 - 79	12.4 - 94
Young's Modulus (GPa)	0.95 - 1.776	0.055 - 0.38	0.413 - 1.490	4 - 5	2.9	2.5 - 3.9
Elongation (%)	15 - 700	90 - 800	12 - 1000	1 - 2.5	20 - 150	35 - >300
Izod impact strength (J/m)	21.4 - 267	> 854	26.7 - 1068	0.05 - 0.55*	42.7 - 160	16.0 - 654

\*Units ft lb/inch

### 2.1.2.2 Biopolymers

Polymers and their basic components (which are constructed predominantly from renewable resources) are termed biopolymers. Polymers of natural origin (e.g., starch and cellulose) must be modified either physically or chemically in order to make them suitable for processing as thermoplastic resins. For example, the structure of starch can be made thermoplastic by using adjuvants such as glycerol and water (Kuhne, 1998). A frequently used option to improve these properties is to add copolymers, and these may be of petrochemical origin (MaterBi is one such product) (Bastioli, 1998).

However, this effect can also be achieved by partial or complete esterification of the hydroxyl groups in the side chains with short-chained organic acids (e.g., acetic acid) or perhaps by the addition of plasticizers (e.g., the product Sconacell A). Other physical, chemical, mechanical, and thermal properties of the biopolymers are also influenced by these modifications. The esterification of hydroxyl groups at the side chain is preferred to convert cellulose into a thermoplastic material that retains its cellulose chain structure (e.g., the product Bioceta) (Kuhne, 1998).

Renewability is linked to the concept of sustainable development. The UN World commission on "Environment and Development in our Future" defines sustainability as the



development, which meets the needs of the present without compromising the ability of future generations to meet their own needs. According to (Narayan, 2001), the manufactured products e.g., packaging, must be designed and engineered from “conception to reincarnation”, the so-called “cradle-to-grave” approach. The use of annually renewable biomass, like wheat, must be understood in a complete carbon cycle. This concept is based on the development and the manufacture of products based on renewable and biodegradable resources: starch, cellulose. By collecting and composting biodegradable plastic wastes, we can generate much-needed carbon-rich compost: humic materials. These valuable soil amendments can go back to the farmland and reinitiate the carbon cycle. Besides, composting is an increasingly key point to maintain the sustainability of the agricultural system by reducing the consumption of chemical fertilizers (Blog, 2011).

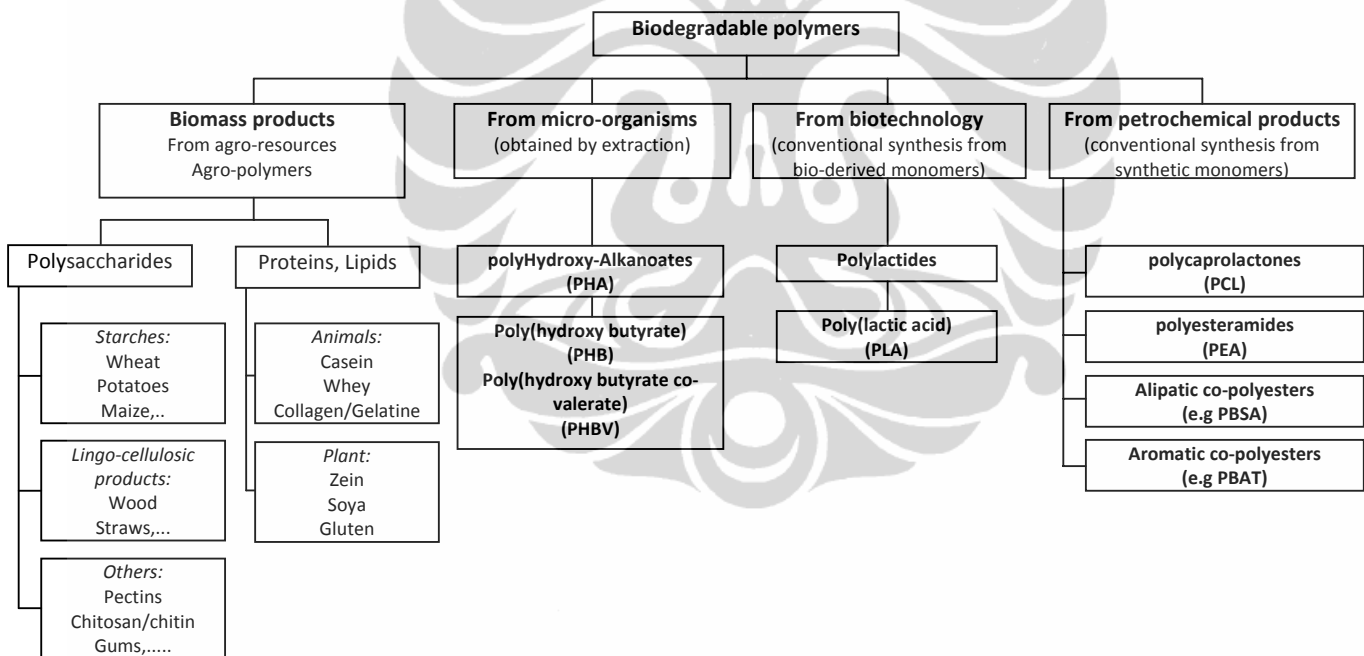


Figure 2.3 Classification of the biodegradable polymers

Figure 2.3 shows a classification of biodegradable polymers in four families (Averous and Boquillon, 2004). Except the fourth family, which is of fossil origin, most polymers (family 1–3) are obtained from renewable resources (biomass). The first family is agro-polymers (e.g. polysaccharides) obtained from biomass by fractionation. The second and third



families are polyesters, obtained, respectively by fermentation from biomass or from genetically modified plants (e.g. PHA, polyhydroxyalkanoate) and by synthesis from monomers obtained from biomass (e.g. PLA, polylactic acid). The fourth family are polyesters, totally synthesized by the petrochemical process (e.g. PCL, polycaprolactone; PEA, polyesteramide; aliphatic or aromatic copolyesters). A large number of these biodegradable polymers (biopolymers) are commercially available. They show a large range of properties and they can compete with non-biodegradable polymers in different industrial fields (e.g. packaging) (Maya Jacob John and Sabu Thomas, 2008).

Biodegradable polymers can undergo degradation processes under the action of enzymes and/or chemical dissociation with living organisms such as fungi and bacteria into harmless secretion products. The use of these polymers in natural fibre-filled composites leads to the development of economically and ecologically attractive technology. Advantages of biodegradable composites include: complete biological degradation; reduction in the volume of garbage; compostability in the natural cycle; preservation of fossil-based raw materials and protection of climate through the reduction of carbon dioxide emission. Commonly used biodegradable polymer matrices are polylactides (PLA), polyglycolic acid (PGA), poly- $\beta$ -hydroxyalkanoates (PHA), co-polymer of 3-hydroxy butyrate (HB) and 3-hydroxyl valerate (HV) (PHBV) and poly( $\omega$ -hydroxy alkanate) mainly poly( $\epsilon$ -caprolactone) (PCL) (K. Van de Velde and P. Kiekens, 2002). Physical properties of commercially important biopolymers are given in Table 2.2 (K. Van de Velde and P. Kiekens, 2001).

Table 2.2 Physical properties of PLA, PGA PCL and PHB

Type of polymer	Density (g/cm <sup>3</sup> )	Tensile strength (MPa)	Tensile modulus (GPa)	Elongation (%)	T <sub>g</sub> (°C)	T <sub>m</sub> (°C)
PLA	1.21-1.25	21-60	0.35-3.5	2.5-6	45-60	150-162
PGA	1.5-1.707	60-99.7	6-7	1.5-20	35-45	220-233
PCL	1.11-1.146	20.7-42	0.21-0.44	300-1000	-60 to -65	58-65
PHB	1.18-1.262	40	3.4-4	5-8	5-15	168-182

## 2.2 Reinforcements

Reinforcements for the composites can be fibers, fabrics particles or whiskers. Fibers are essentially characterized by one very long axis with other two axes either often circular or

near circular. Particles have no preferred orientation and so does their shape. Whiskers have a preferred shape but are small both in diameter and length as compared to fibers. Figure 2.4 shows types of reinforcements in composites.

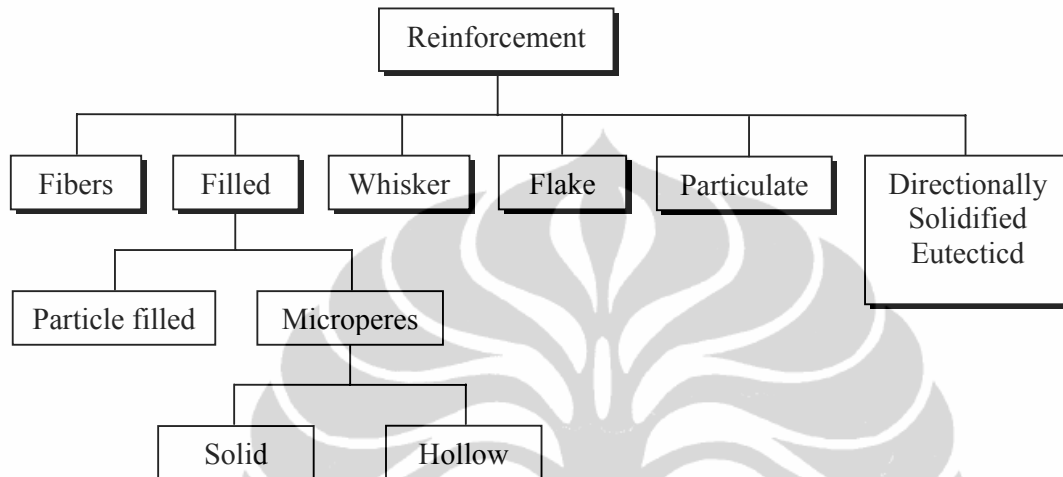


Figure 2.4 Types of reinforcements in composites

Reinforcing constituents in composites, as the word indicates, provide the strength that makes the composite what it is. But they also serve certain additional purposes of heat resistance or conduction, resistance to corrosion and provide rigidity. Reinforcement can be made to perform all or one of these functions as per the requirements.

A reinforcement that embellishes the matrix strength must be stronger and stiffer than the matrix and capable of changing failure mechanism to the advantage of the composite. This means that the ductility should be more minimum or even nil the composite must behave as brittle as possible.

### 2.2.1 Fiber Reinforcement

The history of fibres is as old as human civilization. Traces of natural fibres have been located to ancient civilizations all over the globe. For many thousand years, the usage of fiber was limited by natural fibres such as flax, cotton, silk, wool and plant fibres for different applications. Fibers can be divided into natural fibres and man-made or chemical fibres. Flax is

considered to be the oldest and the most used natural fibre since ancient times (Textileschool, 2010).

Fibers are the important class of reinforcements, as they satisfy the desired conditions and transfer strength to the matrix constituent influencing and enhancing their properties as desired.

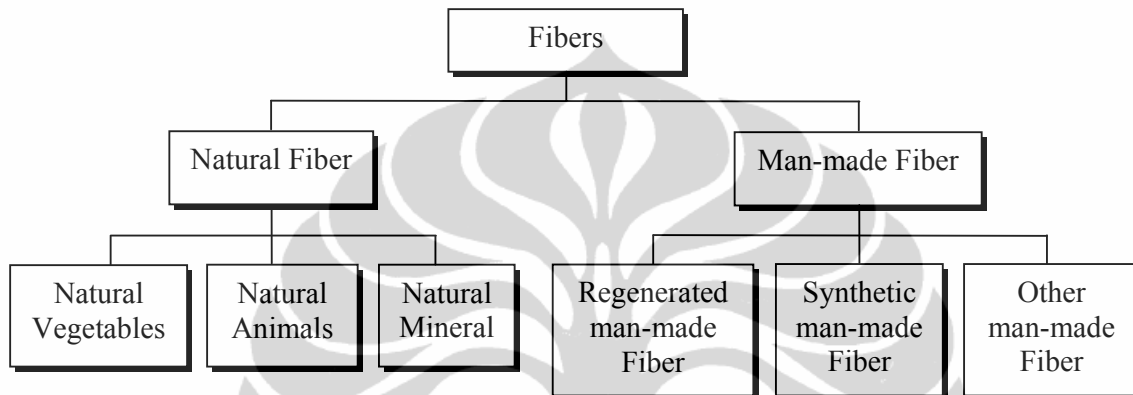


Figure 2.5 Classification of fibers

Organic and inorganic fibers are used to reinforce composite materials. Almost all organic fibers have low density, flexibility, and elasticity. Inorganic fibers have of high modulus, high thermal stability and possess great rigidity.

Fibers increase the modulus of the matrix material. The strong covalent bonds along the fiber's length gives them a very high modulus in this direction because to break or extend the fiber the bonds must also be broken or moved. The arrangement or orientation of the fibers relative to one another, the fiber concentration, and the distribution all have a significant influence on the strength and other properties of fiber-reinforced composites. Applications involving totally multidirectional applied stresses normally use discontinuous fibers, which are randomly oriented in the matrix material. Consideration of orientation and fiber length for a particular composites depends on the level and nature of the applied stress as well as fabrication cost (Elgun, 1999).

In a fiber-reinforced polymer, the fibers serve as a reinforcement and show high tensile strength and stiffness, while the matrix holds the fibers together, transmits the shear forces, and



also functions as a coating. The materials behavior of matrices is usually characterized by a functional relationship of time and temperature, a considerably lower tensile strength, and a comparatively higher elongation. Therefore, the mechanical properties of fibers determine the stiffness and tensile strength of the composite. Very thin fibers with a large surface to volume ratio generally provide good adhesion between the fibers and the matrix.

Depending on the fiber orientation, the materials behavior of composites can be either: quasi-isotropic (with all (short) fibers randomly orientated, and no privileged direction of mechanical properties); anisotropic (with all fibers orientated in one or more directions with corresponding mechanical properties); or orthotropic (fibers orientated mainly in two directions rectangular to each other and showing corresponding materials behavior).

The selection of suitable fibers is determined by the required values of stiffness and tensile strength of a composite (Michaeli W and Wegener M, 1990) (Carlsson LA and Byron Pipes R, 1989). Further criteria for the choice of suitable reinforcing fibers include:

- Elongation at failure;
- Thermal stability;
- Adhesion of the fibers and matrix;
- Dynamic behavior;
- Long-term behavior; and
- Price and processing costs.

### 2.2.2 Natural fiber

Natural fibers were utilized by mankind considerably earlier than metals, alloys, and ceramics. Indeed, it can be reasonably assumed that fibers were applied by humans long before recorded history. Moreover, even some animals use fibers, for example, when building nests (birds, mammals), webs (spiders), for protection during pupation (caterpillars, silkworms), or for retrieving insects out of narrow holes (chimpanzees). In short, some animals produce fibers for their needs, whereas others collect them. The history of the utilization of fibers is, however, much harder to trace than that of metals and ceramics because fibers often deteriorate through rot, mildew, and bacterial action. In other words, only a few specimens of early fibers have been found so far.

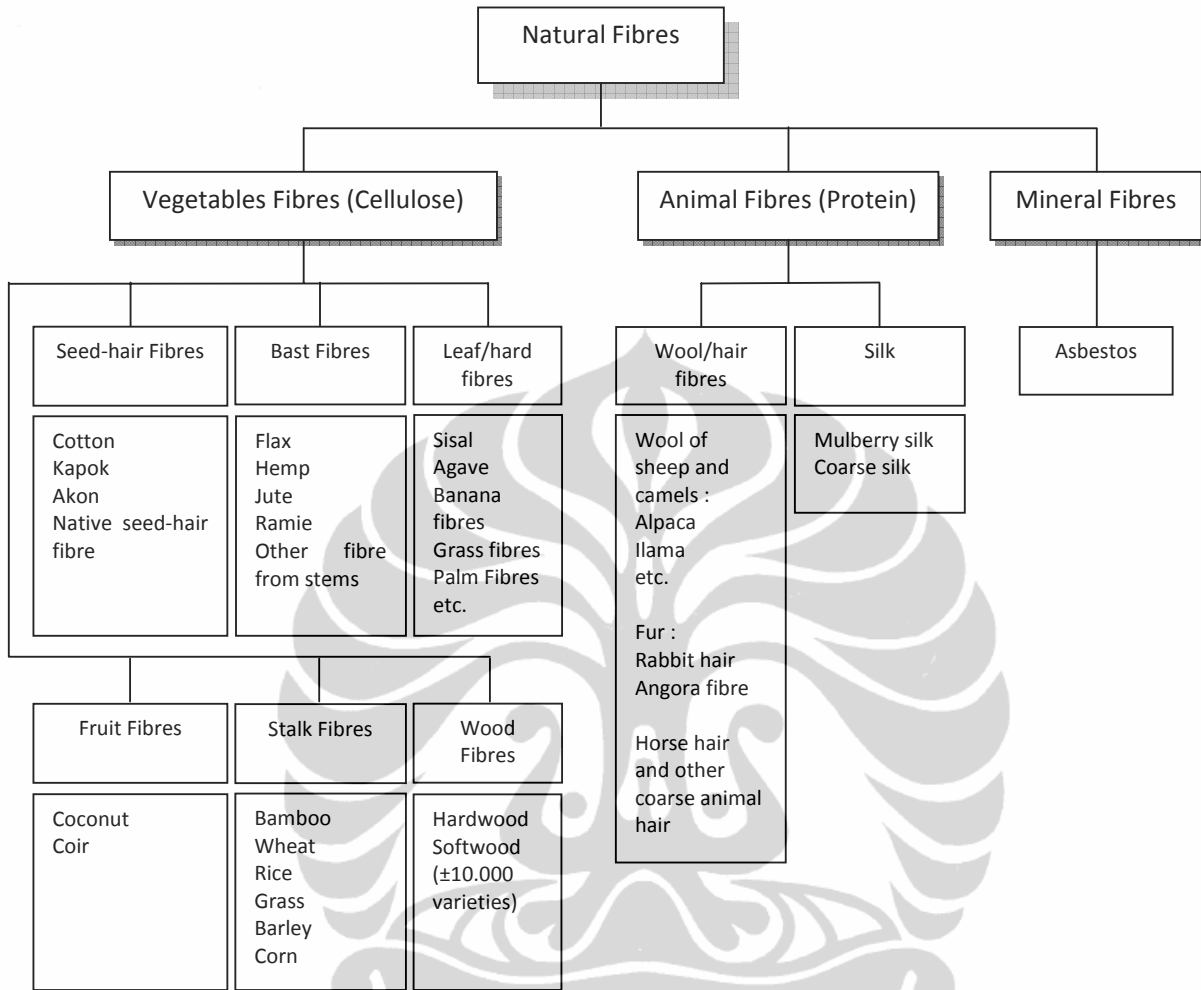


Figure 2.6 Classification of natural fibers (DIN 60001-1, 1990)

The natural fibers may be classified by their origin as cellulosic (from plants), protein (from animals), and mineral. The plant fibers may be further ordered as seed hair fibers, such as cotton; bast (stem) fibers, such as linen from the flax plant, hemp, jute, ramie; leaf (hard) fibers, such as sisal, banana; fruits ) fibers such as coconut; stalk fibers such as bamboo and wood fibers. The animal fibers are grouped under the categories of hair, such as wool, fur such as angora; and secretions or silk. The only important mineral fiber is asbestos, which because of its carcinogenic nature has been banned from consumer textiles.



The plant or vegetable fibers are mostly cellulose-based, that is, they consist of polymers derived from carbohydrates (i.e.,  $C_nH_{2n}O_n$ ) which are manufactured by the plant from water and carbon dioxide gas through photosynthesis.

Fibers retrieved from plants likewise played an important role in early civilizations because of their usefulness for clothing, storage, shelter (e.g., tents), and cordage. It is, however, not fully known when specific plants were first cultivated for fiber production.

Nevertheless, some records indicate that hemp was presumably the oldest cultivated plant for this purpose. Hemp was first grown in Southeast Asia, from where it spread to China in approximately 4500 B.C. Flax was probably cultivated in Egypt before 3400 B.C., at which time the art of spinning and weaving linen was already well developed. Woven flax and wool fabrics were found at the sites of the “Swiss lake dwellers” dating back to the seventh and sixth centuries B.C.

Advantages of biofibres are low cost, low density, strength properties, carbon dioxide sequestration and biodegradability (Mohanty, A.K., M. Misra and L.T. Drzal, 2002). However, biofibres are hydrophilic and do not bind readily to the hydrophobic polymeric matrix (Mohanty, A.K., M. Misra and G. Hinrichsen, 2000). They are also degraded at low processing temperatures required during processing.

### 2.2.3 Common natural fibres

In table 2.3 properties of the natural fibres are presented and compared to the properties of glass fibre (Bismarck A, Mishra S, Lampke T, 2005). With respect to the natural fibres one has to keep in mind that large variation in properties exist due to natural circumstances.

Table 2.3 Natural fibre properties compared to glass

Fibre	Density ( $g/cm^3$ )	Tensile Strength (Mpa)	Young's Modulus (GPa)	Elongation at break (%)	Price of row fibre (\$/kg)
Glass	2.55	2000- 3500	72	3	1.3
Flax	1.50	345-1100	50-70	2.7-3.2	0.5-1.5
Hemp	1.47	550-900	30-60	1.6	0.6-1.8
Jute	1.30	393-773	20-55	1.16-1.5	0.35
Ramie	1.50	400-938	61.4-128	1.2-3.8	1.5-2.5



Sisal	1.50	468-640	9.4-22.0	3-7	0.6-0.7
Coir	1.20	131-175	4-6	15-40	0.25-0.5
Cotton	1.55	287-800	5.5-12.6	7.0-8.0	1.5-2.2

For example from table 2.3, we see that flax has very good properties comparable to E-glass. Flax is also less dense thereby producing a light composite with good mechanical properties.

Natural fibres are complex in structure. They are generally lignocellulosic, consisting of helically wound cellulose microfibrils in an amorphous matrix of lignin and hemicellulose. Table 2.4 lists some natural fibres and their chemical constituents. Mechanical properties are dominated by the cellulose content and microfibril angle. A high cellulose content and low microfibril angle are desirable properties in a fibre to be used as reinforcement in polymer composites.

Table 2.4 Chemical composition of natural fibres

Fibrous Material	Cellulose	Hemicellulose	Lignin	Extractives	Ash
Flax	78.5	9.2	8.5	2.3	1.5
Hemp	68.1	15.1	10.6	3.6	2.6
Kenaf	60.8	20.3	11	3.2	4.7
Soft wood	40-44	25-29	25-31	5	0.2
Hard wood	43-47	25-35	16-24	2-8	0.4

The use of natural fibres as a replacement for synthetic fibres has received attention. While high performance carbon fibres remain superior to natural fibres in high-end applications, however natural fibres have comparable properties to glass fibres in high volume applications (George I and Richard P, 2000).

Depending on their origin, natural fibres can be grouped into seed, bast, leaf and fruit qualities. Bast and leaf (the hard fibres) qualities are the most commonly used in composite applications. Examples of bast fibres include hemp, jute, flax, ramie, and kenaf. Leaf fibres include, sisal and banana leaf fibres. Properties for these fibres include excellent tensile strength and modulus, high durability, low bulk density, good moldability and recyclability.





These natural fibers have an advantage over glass fibers in that they are less expensive, abundantly available from renewable resources and have a high specific strength.

Many natural fibers have a hollow space (the lumen), as well as nodes at irregular distances that divide the fiber into individual cells. The surface of natural fibers is rough and uneven and provides good adhesion to the matrix in a composite structure. As mentioned above, the specific mechanical properties of natural fibers are important when used in composites. When comparing the tenacity and elongation at failure of both natural and synthetic fibers, hemp, flax, and ramie fibers can compete with E-glass fibers that serve as a reference because of their major importance in composite technology (Flemming et al, 1995).

#### 2.2.3.1 Cotton

Cotton, see figure 2.7 is world-wide one of the most important fibres used in the textile industry. Picking is highly labour-intensive, and on large scale is often carried out by machine. In many parts of the world, however, picking is carried out by hand. The picked cotton, so called 'cotton wool' is baled. The seeds, dead leaves and other debris are removed by ginning. The clean cotton is baled again and is ready to be spun.

In comparison with other natural fibres, Cotton is rather weak. It can absorb moist up to 20% of its dry weight, without feeling wet and is also a good heat conductor. Cotton is applied for the manufacturing of clothes, carpets, blankets, mobs and medical cotton wool.



Figure 2.7 Cotton. Left: processing. Right: Field Cotton

### 2.2.3.2 Coir (Coconut fibre)

Coconut fibre is obtained from the husk of the fruit of the coconut palm, see figure 2.8. The trees can grow up to 20 m, making harvesting a difficult job. People or trained monkeys climb the tree to pick the nuts, or a pole with an attached knife is used. The fruits are dehusked with on a spike and after retting, the fibres are subtracted from the husk with beating and washing.

The fibres are strong, light and easily withstand heat and salt water. After nine months of growth, the nuts are still green and contain white fibre, which can be used for the production of yarn, rope and fishing nets. After twelve months of growth, the fibres are brown and can be used for brushes and mattresses.



Figure 2.8 Coconut palm



Figure 2.9 Raw jute in a retting tank

### 2.2.3.3 Jute

The fibres are extracted from the ribbon of the stem. When harvested the plants are cut near the ground with a sickle shaped knife. The small fibres, 5 mm, are obtained by successively retting in water, see figure 3.3, beating, stripping the fibre from the core and drying.

Due to its short fibre length, jute is the weakest stem fibre, although it withstands rotting very easily. It is used as packaging material (bags), carpet backing, ropes, yarns and wall decoration.



#### 2.2.3.4 Ramie

The Ramie plant (2 m high) can easily grow in tropical regions. Ramie is usually harvested by hand. Retting is a more complex process due to a gummy substance surrounding the fibres, which has to be removed with chemicals. This process is expensive and difficult to control, often resulting in a damaged fibre.

Ramie is an expensive and durable fibre and can be dyed very easily, and is therefore more often used in decorative fabrics than as construction material. Applications are curtains, wallpaper, sewing thread and furniture covers.

#### 2.2.3.5 Hemp

The plant itself, see figure 2.10 can be grown without artificial fertilisers and weeds don't stand a chance because hemp covers the entire ground within 4 weeks. The production is very labour intensive, especially the separation of the fibres from the bast. After mowing, the stems are bundled and dried. The seeds are removed by threshing and the stems are dew or water retted, dried and hackled to remove the fibres. Breaking softens the fibres and the top and bottom (bad) parts are removed.

A Hemp yarn is strong and has of all natural fibres the highest resistance against water, but it shouldn't be creased excessively to avoid breakage. The fibre is used for the production of rope, fishing nets, paper, sacks, fire hoses and textile.



Figure 2.10 Hemp



Figure 2.11 Sisal



#### 2.2.3.6 Sisal

The plants look like giant pineapples, see figure 2.11 and during harvest the leaves are cut as close to the ground as possible. The soft tissue is scraped from the fibres by hand or machine. The fibres are dried and brushes remove the remaining dirt, resulting in a clean fibre.

Sisal produces sturdy and strong fibres that are very well resistant against moist and heat. It is mainly used for ropes, mats, carpets and cement reinforcement.

### 2.3 Natural fiber composites

Ecological concern has resulted in a renewed interest in natural materials. An interesting environmentally friendly alternative for the use of glass fibres as reinforcement in engineering composites are ligno-cellulosic natural fibres such as flax, hemp, sisal and jute (Morton, W. E. and Hearle, J. W. S, 1993). These fibres are renewable, nonabrasive, can be incinerated for energy recovery since they possess a good calorific value and they give less concern with health and safety during handling of fibre products. In addition, they exhibit excellent mechanical properties, low density and low price. This excellent price-performance ratio at low weight in combination with the environmental friendly character is very important for the acceptance of natural fibres in large volume engineering markets such as the automotive and construction industry.

Natural fibers traditionally have been used to fill and reinforce thermosets, natural fiber reinforced thermoplastics, especially polypropylene composites, have attracted greater attention due to their added advantage of recyclability (Mohanty AK, Drazl LT, Misra M, 2002). Natural fiber composites are also claimed to offer environmental advantages such as reduced dependence on non-renewable energy/material sources, lower pollutant emissions, lower greenhouse gas emissions, enhanced energy recovery, and end of life biodegradability of components. Since, such superior environmental performance is an important driver of increased future use of natural fiber composites, a thorough comprehensive analysis of the relative environmental impacts of natural fiber composites and conventional composites, covering the entire life cycle, is warranted.



Natural fibres have good intrinsic mechanical properties, a low density compared to glass fibres as well as a lower price. Research publications nearly all show a composite stiffness for flax fiber reinforced composites close to or even higher than that of commercial glass mat reinforced thermoplastic composites (GMT) and thermoset sheet moulding compound (SMC) (Mieck K.-P. L. R., 1996), (Heijenrath R., Peijs T, 1996).

Most of natural fiber composites are short fiber composites with non-homogeneous length and orientation distributions. It is known that elastic modulus and the strength of discontinuous fiber composites are moderate compared to continuous fiber-reinforced composites.

#### 2.4 Polylactic Acid (PLA)

Poly lactide or poly lactic acid, otherwise known as PLA, is a biodegradable thermoplastic polyester that is manufactured by biotechnological processes from renewable resources (e.g. corn). Although other sources of biomass can be used, corn has the advantage of providing the required high-purity lactic acid. The use of alternative starting materials (e.g. woody biomass) is being pursued in order to reduce process costs.

Recently, with growing pressure on the world's resources as well as concerns about disposal of plastics intensifying interest and commercial activity, biodegradable polymers have received much attention (Rafael A, Bruce H and Susan S, 2004). Biopolymers offer environmental benefits such as biodegradability, greenhouse gas emissions, and renewability of the base material (Suprakas S R and Mosto B, 2005). Poly lactic acid (PLA) has received much attention of biodegradable polymers (Bhuvanesh G, Nilesh R, Jons H, 2007), (Yul L, Katherine D, Lin L, 2006). PLA has properties that are comparable to many commodity polymers (e.g. PP, PE, PVC, PS) such as high stiffness, clarity, gloss, and UV stability (Suprakas S R, Pralay M, 2002). PLA is commonly produced by two methods (Gu S, Yang M, Yu T, Rent T, Ren J, 2008). It can be commonly synthesized by ring-opening polymerization of lactide. PLA may also be produced by direct polycondensation of lactic acid. Lactic acid, the starting material for PLA synthesis, can be produced by fermentation from a number of different renewable resources. Because PLA has high strength, thermal plasticity, and biocompatibility, it has been used as package materials and other products (Tadashi Y and Masayuki Y, 2008). However,

the physical properties of PLA such as brittleness limit the PLA polymer application (Cohn D and Solomon A H, 2005). A way to improve the mechanical and thermal properties of PLA is the addition of fibers or filler materials (Masud S H, Lawrence T D, Manjusri M, 2005).

PLA, as produced by Cargill Dow LLC, uses from 20 to 50% less fossil fuel than conventional plastic resins (Figure 2.12), meaning that up to 2 to 5 times more PLA can be produced with a given amount of fossil fuel than petrochemical-derived plastics. The ability to produce the polymer on a favorable cost/performance basis is critical to realizing these human health and environmental benefits. This has been achieved through efficient use of raw materials, reduced energy utilization, and elimination of solvents.

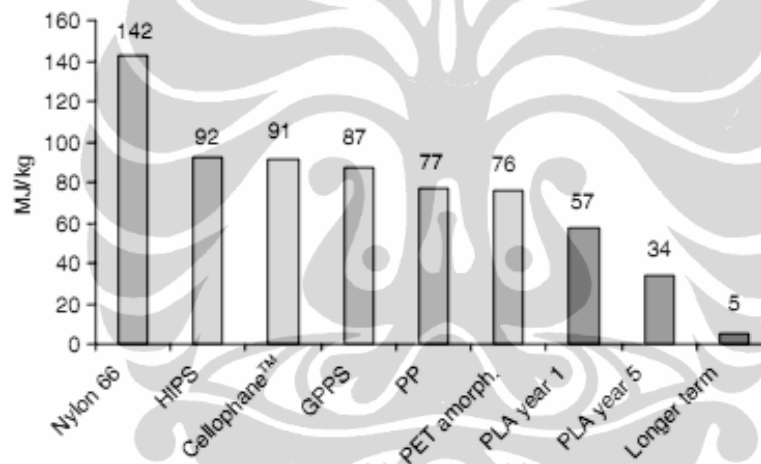


Figure 2.12 MJ/kg of energy required to produce various polymers

PLA is also a low-impact, greenhouse gas polymer because the  $\text{CO}_2$  generated during PLA biodegradation is balanced by an equal amount taken from the atmosphere during the growth of plant feedstocks. In contrast, petrochemical-based polymers contribute to volatile organic compound (VOC) emissions and  $\text{CO}_2$  generation when incinerated. LCA calculations indicate that PLA has a greenhouse gas emission rate of about 1600 kg  $\text{CO}_2$ /metric ton, while polypropylene, polystyrene, PET, and nylon have greenhouse gas values of 1850, 2740, 4140, and 7150 kg  $\text{CO}_2$  /metric ton, respectively. Figure 2.13 shows the relative comparison of greenhouse gas contribution to the environment for common plastic products. Longer term, as PLA is produced from field wastes or other biomass, PLA can become a  $\text{CO}_2$  sink and actually contribute to a net reduction in greenhouse gases.

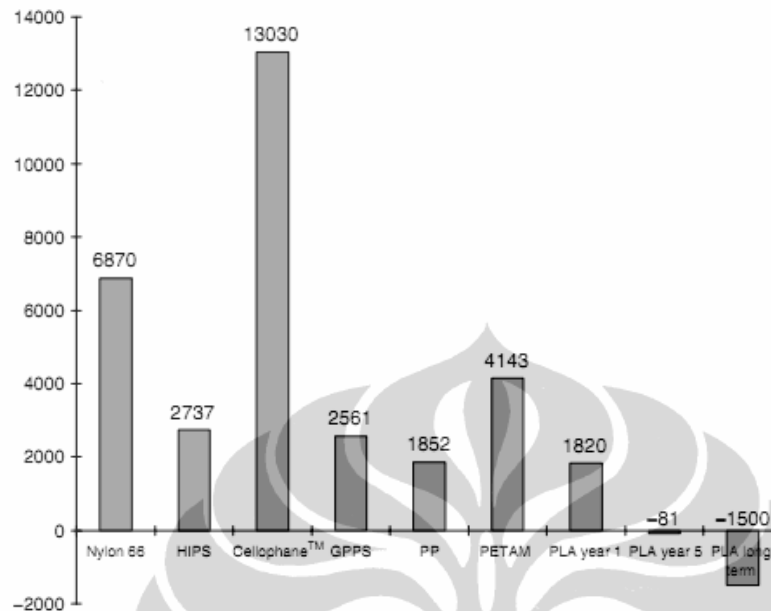


Figure 2.13 Greenhouse gas emissions (kg/ton) for production of various polymers. Long-term emissions for PLA are based on utilization of biomass for the production of lactic acid

Mechanical properties of PLA are similar to those of commodity plastics such as PP and PE; but PLA also exhibits inherent brittle behavior similar to polystyrene (PS). PLA shows high elastic modulus and tensile strength in the range of 0.35 to 2.8 GPa and 10 to 60 MPa, respectively (Baiardo, Frisoni, Scandola, 2003). However, low impact toughness and elongation at break have been limiting factors to diversification in applications of PLA (Shibata, 2005). Numbers of method have been studied to modify the stiff and rigid behavior of PLA. Among them are copolymerizations, blending with other polymers or plasticizer.

## CHAPTER III

### MATERIAL STUDIED AND EQUIPMENT

#### 3.1 Flax Fibre

Flax fibre is extracted from the bast or skin of the stem of the flax plant. Flax fibre is soft, lustrous and flexible; bundles of fibre have the appearance of blonde hair, hence the description "flaxen". It is stronger than cotton fiber but less elastic.

Flax belongs to the genus *Linum*, the family *Linaceae* and is one of the oldest fibre crops in the world. Flax fibre is one of the natural fibres which has the highest strength, 345 – 1100 MPa (A. K. Mohanty, 2000). The structure of flax fibres is composite like in itself. A schematic structure of the flax fibre, from stem to micro fibril, is given in figure 3.1 (Harriette L. Bos, 2006). The coarse bast fibre bundles are isolated from the stem by mechanical decortications (breaking, scutching and hackling), resulting in technical fibres (also called fibre bundles).

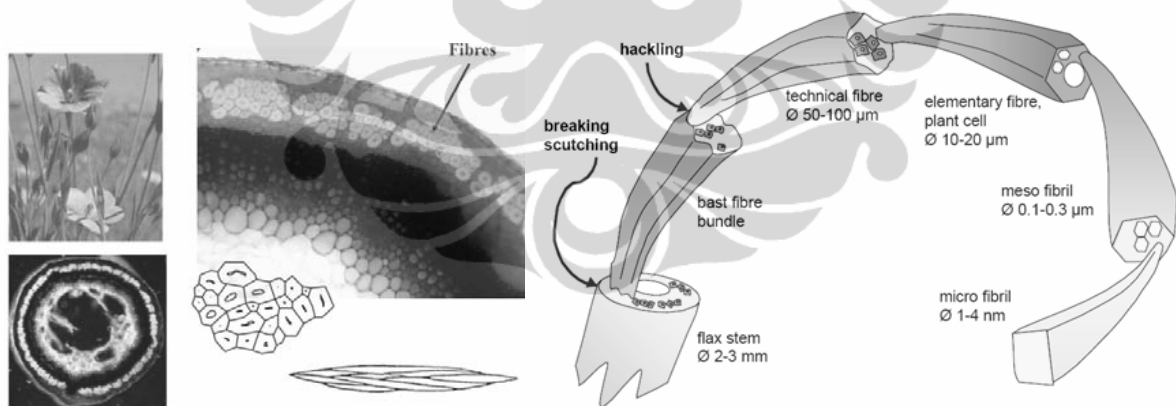


Figure 3.1 Schematic of flax fibre

The mechanical properties of these different fibre forms differ strongly. Flax fibre bundles are being obtained after the first isolation processes called breaking and scutching. These fiber bundles have an acceptable price-performance ratio and are often commercially used in natural fibre mat reinforced thermoplastic (NMT) and thermoset composites. Their





lateral strength is rather poor compared with their axial strength, mainly due to the weak pectin bonds between the so-called ‘technical fibres’. The really strong fibres are the elementary fibres, which have an average tensile strength up to 1500 MPa.

The technical fibre is approximately 1 m long and consists of about 10–40 elementary fibres in cross-section. The elementary fibres have lengths of between 2 and 5 cm, and diameters between 5 and 35  $\mu\text{m}$ . The elementary fibers are glued together by a pectin interface. They are not circular but a polyhedron with 5 to 7 sides to improve the packing in the technical fiber.

### 3.2 Polylactic Acid (PLA)

Compared to the other biodegradable polyesters, polylactic acid (PLA) is the product that at the present time has one of the highest potentials due to its availability in the market and its low price. For instance, Cargill–Dow has developed processes that use corn and other feedstock to produce different PLA grades (NatureWorks).

Table 3.1 Polylactic Acid (PLA) typical properties

<i><b>Mechanical Properties</b></i>		
Quantity	Value	Unit
Modulus of elasticity	350 - 2800	MPa
Tensile strength	10 - 60	MPa
Elongation	1.5 - 380	%
Bending strength	0.89 - 1.03	MPa
Impact strength	0.16 - 1.35	J/cm
<i><b>Physical Properties</b></i>		
Quantity	Value	Unit
Melting temperature	150 - 160	$^{\circ}\text{C}$
Glass temperature	45 - 65	$^{\circ}\text{C}$
Density	1210 - 1430	$\text{kg}/\text{m}^3$
Water absorption	0.5 - 50	%



The Polylactic Acid (PLA) used in this study is Ingeo Biopolymer 7001D, a NatureWorks LLC product, is a thermoplastic resin derived from annually renewable resources and is specifically designed for use in injection stretch blow molded (ISBM) bottle applications. Ingeo Biopolymer 7001D can be run using conventional ISBM equipment, but processes at lower temperatures than PET and can attain good resolution of mold detail on conventional tooling.

Ingeo is the trademark name for NatureWorks LLC's synthetic fiber made from corn. The process to create Ingeo makes use of the carbon stored in plants by photosynthesis. Plant starches are broken down into sugars. The carbon and other elements in these sugars are then used to make a biopolymer through a process of fermentation and separation. The resulting resin, called Ingeo biopolymer, polylactid (PLA).

Typical material and application properties and typical processing parameters of Ingeo Biopolymer 7001D are given in table 3.2 and 3.3 :

Tabel 3.2 Typical material and application properties

Physical Properties	Ingeo 7001D	ASTM Method
Specific Gravity	1.24	D792
MFR, g/10 min (210 <sup>0</sup> C, 2.16kg)	6	D1238
Glass Transition Temperature	55-60 <sup>0</sup> C	D3418
Crystalline Melt Temperature	145-160 <sup>0</sup> C	D3418
Transmission Rates		
Oxygen	675 cc-mil/m <sup>2</sup> -24hr-atm	D1434
Carbon Dioxide	2.850 cc-mil/m <sup>2</sup> -24hr-atm	Internal
Water Vapor	375 g-mil/m <sup>2</sup> -24hr	F1249
Clarity	Transparent	
General Blow Molding Guidelines		
Preform Temperature	80-100 <sup>0</sup> C (176-212 <sup>0</sup> F)	
Stretch Rod Speed	1.2-2 m/sec	
Mold Temperature	21-38 <sup>0</sup> C (70-100 <sup>0</sup> F)	

Tabel 3.3 Typical processing parameters

Parameter	Units (English)	Units (Metric)
Melt temperature	390-430 <sup>0</sup> F	200-220 <sup>0</sup> C
Feed throat	70 <sup>0</sup> F	20 <sup>0</sup> C
Feed Section	355 <sup>0</sup> F	180 <sup>0</sup> C
Compression Section	410 <sup>0</sup> F	210 <sup>0</sup> C
Matering Section	410-430 <sup>0</sup> F	210-220 <sup>0</sup> C
Nozzle	410-430 <sup>0</sup> F	210-220 <sup>0</sup> C
Mold	700-100 <sup>0</sup> F	
Back Pressure	100-200psi	

### 3.3 Production of specimen

In this experiment, composite material consisted of flax fibres length 1 mm was mixed with matrix Polylactic Acid (PLA). Table 3.4 shows material composition between fibre and matrix.

The fibre weight fraction ( $W$ ) of composite was determined by relation between weight of fibre and matrix:

$$W = \frac{m_f}{m_f + m_m}$$

where the subscripts 'f' and 'm' denote matrix and fibre, respectively. The volume fractions of fibres ( $V_f$ ) were calculated from  $W$ , density of fibre ( $\rho_f$ ) and density of matrix ( $\rho_m$ ).

$$V_f = \frac{m_f \rho_m}{m_f \rho_m + m_m \rho_f}$$

Table 3.4 List of composites composition

Materials	Matrix		Fibres content	Fibres volume fraction
	unit	wt%	wt%	%
Pure PLA		100	-	-
PLA/Flax10		90	10	8.4
PLA/Flax20		80	20	17.1
PLA/Flax30		70	30	26.2

Specimens for this experiment were produced through three processes: extrusion, granulation and injection. These processes are illustrated in Figure 3.2.

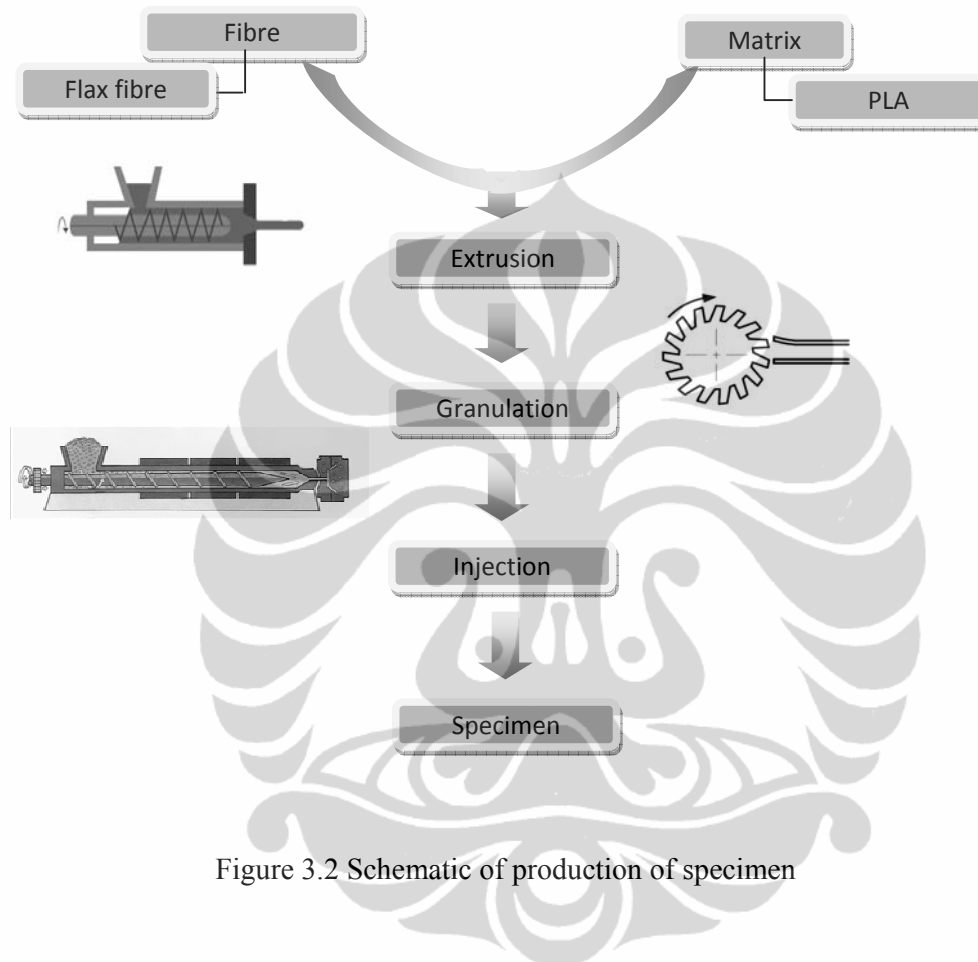


Figure 3.2 Schematic of production of specimen

#### 3.4 Extrusion machine

A screw extruder was used for the mixing and homogenizing of the flax fibre with the matrix material, polylactic acid (PLA) Ingeo Biopolymer 7001D. To expel moisture content, flax fibres were dried in an oven at 50<sup>0</sup>C for 24 hours. The temperatures of the four zones of the extruder were 190<sup>0</sup> C (forming, metering, melting and compression and smooth feeding zones, Figure 3.3), this is to avoid degradation of natural fibres which mostly happen at 200<sup>0</sup>C. In this process, the screw speed was adjusted to 30 rpm.

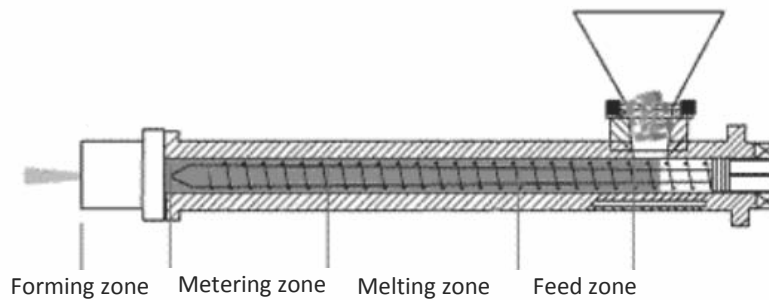


Figure 3.3 Extrusion machine

The result of this process was the mixing between polylactic acid (PLA) with flax fibres in wire rod form at 4-5 mm diameter. The wire rod was left to cool at room temperature.

### 3.5 Granulation

The objective of this process is to get the shape of the mixing flax fibres-polypropylene in grain through cutting the wire rod into small pieces with 3-5 mm length.

### 3.6 Injection machine

After a granulation step, the granulated compounds were injected on an injection moulding press Battenfeld 80 tons with a mould temperature of 30 °C and a barrel temperature of 190 °C. The injection pressure was 1000 bars and the cooling time was 15s. The shape of specimen is dog bone based on ISO standard.



Figure 3.4 Injection machine

### 3.7 Testing method

Composite specimens were characterized for their tensile, flexural, torsion and compressive properties. SEM (Scanning Electron Microscope) was used to investigate the failure surfaces and the quality of bonding between fibres and matrix.

#### 3.7.1 Tensile test

Tensile tests on composites were performed on a tensile machine type MTS Synergie RT/1000 at room temperature with sensor strength of 10kN. The speed of the ram was 1 mm/min as per ISO 527.2 standard. An extensometer with a nominal length of 25 mm was used to monitor the elongation of the specimen. Simple tensile test was performed for pure polypropylene, polypropylene with coupling agent and polylactic acid (PLA). Five specimens were tested with a dimension as describe in Figure 3.5.

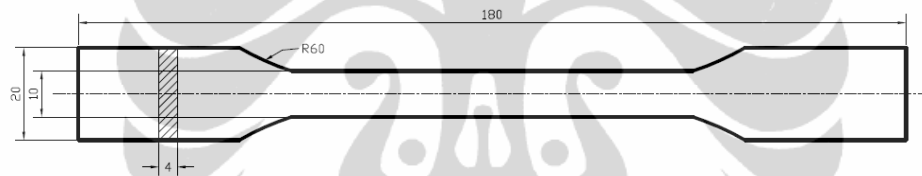


Figure 3.5 Dimension of tensile test specimen

The elastic properties are determined from the stress  $\pm$  strain curve. The initial slope is equal to the elastic stiffness or modulus in the direction of the applied load:

$$E = \frac{\Delta\sigma}{\Delta\epsilon}$$

#### 3.7.2 Bending test

Bending tests were performed to obtain flexural properties of the material. These tests were performed on a universal testing machine type MTS Synergie RT/1000 at room temperature with sensor strength of 10kN. Five specimens were tested follow the ASTM D1185 standard for 3 point bending (Figure 3.6). The speed of the ram was 2 mm/min which used for strain determination.

The flexural modulus is calculated from slope of the load – deflection curve using the equation:

$$E = \frac{L^3}{4wh^3} \frac{\Delta F}{\Delta d}$$

Where :

F : load (N)

L : distance of the two support (mm)

w : width (mm)

h : thickness (mm)

d : deflection (mm)

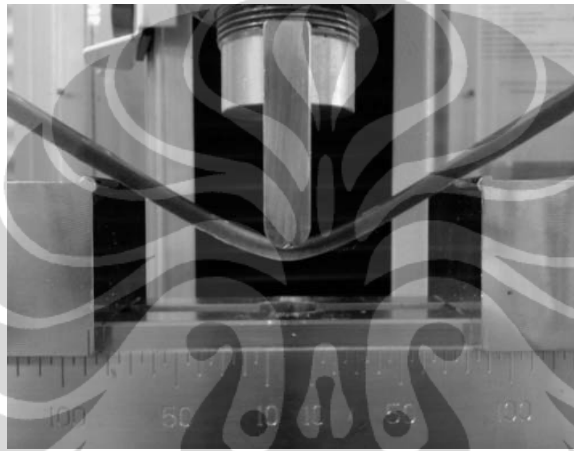


Figure 3.6 Three-Point bending test

The flexural stress in the outer fibres at the mid-span is calculated from the following equation:

$$\sigma = \frac{3FL}{2wh^2}$$

Among all of parameters for calculation the bending properties, only load (F) and deflection (d) obtained from testing.

### 3.7.3 Torsion test

Torsion tests were performed on five rectangular bar specimens with a dimension; length, width and thickness are 80, 10 and 4 in mm, respectively. The tests were conducted at angular speed of 5 degree/s.

The applied torque ( $T$ ) to the specimen and resulting deformation (angle of twist,  $\Phi$ ) are measured during the torsion test. Unlike circular bar that shear stress distribution is uniform in its cross section. The shear stress distribution for rectangular section is more complicated. The distortion of rectangular section varies along the sides of this cross section, reach a maximum value at the middle and disappear at the corners. Therefore, the shear stress varies as this distortion is maximum value at the middle of the sides and zero at the corners of the cross section (Figure 3.7).

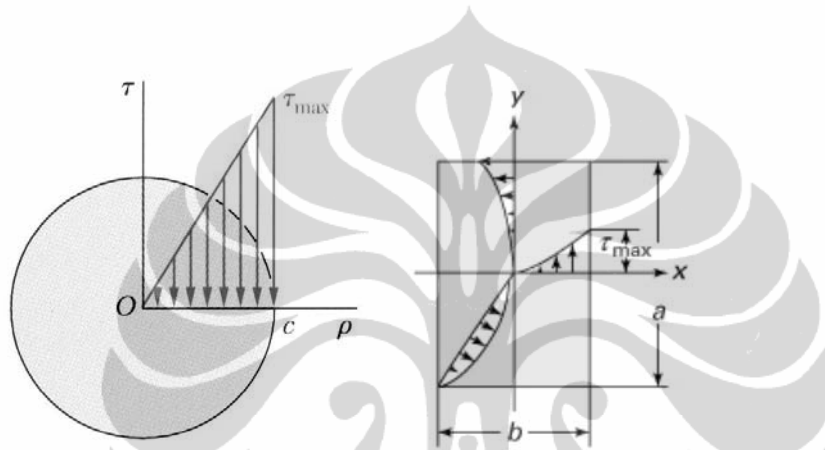


Figure 3.7 Shear stress distribution

Therefore the maximum shear stress occurs at the middle of the longer sides of the rectangular cross section is given by the equation:

$$\tau = \frac{T}{c_1 ab^2}$$

In which  $a$  is the longer and  $b$  the shorter side of the rectangular cross section (Figure 3.8) and  $c_1$  is a numerical factor depending upon the ratio  $a/b$ . Several values of  $c_1$  are given in table 3.5.

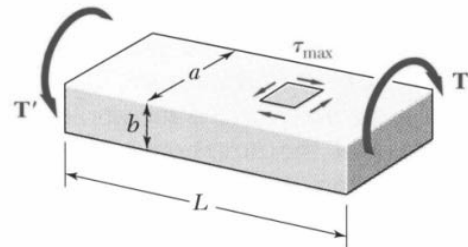


Figure 3.8 Rectangular bars of torsion specimen



Table 3.5 Coefficient geometry for rectangular bars (Timoshenko, 1948)

$a/b$	$c_1$	$c_2$
1.0	0.208	0.1406
1.2	0.219	0.1661
1.5	0.231	0.1958
2.0	0.246	0.229
2.5	0.258	0.249
3.0	0.267	0.263
4.0	0.282	0.281
5.0	0.291	0.291
10.0	0.312	0.312
$\infty$	0.333	0.333

The angle of twist per unit length in the case of rectangular cross section is given by the equation :

$$\frac{\phi}{L} = \frac{T}{C_2 ab^3 G}$$

The values of the numerical factor  $c_2$  are given in table 3.5.

### 3.7.4 Compression test

Shear loading method and configuration jigs compression tests as illustrated in Figure 3.9.a and Figure 3.9.b, respectively. Compression tests were performed at MTS Synergie RT/1000 with sensor strength 10 kN to determine behaviour of materials under crushing loads. The tests which based on ASTM D695M standard with speed of ram 1 mm/min were carried out on five samples with a width of 10 mm and a thickness of 4 mm. The specimens were compressed and deformation at various loads is recorded. Strain deformation was determined by cross head motion. The stress maximum was measured when the load reached elastic limit as well strain. The elastic properties are also determined from the stress-strain curve using the same analytical expressions used in tensile testing. The initial slope is equal to the elastic stiffness or modulus in the direction of the applied load.

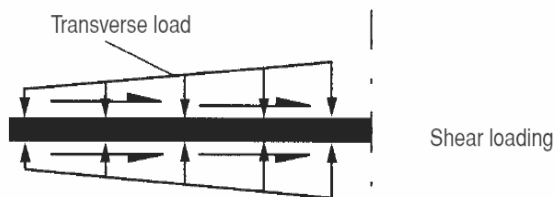
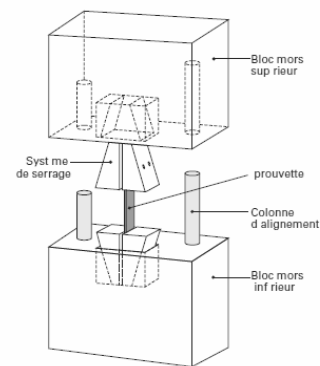


Figure 3.9.a. Compression test method,



b. Configuration of compression jigs

The typical failure mode of specimens in compressive test is represented in Figure 3.10.

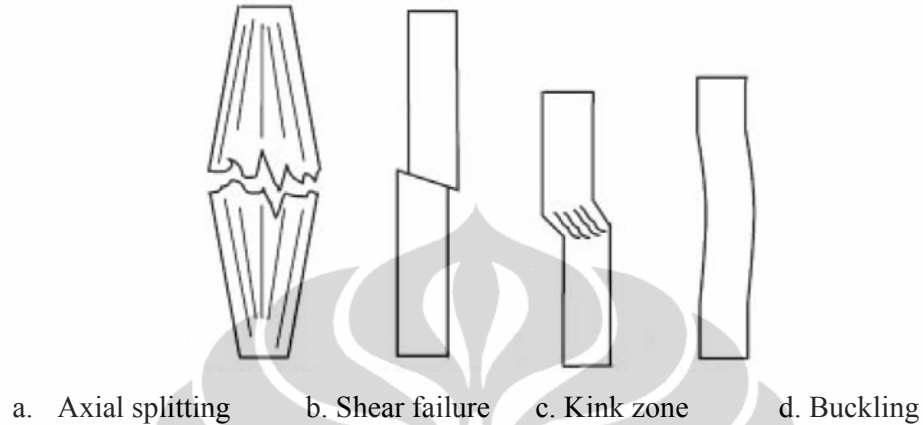


Figure 3.10 Basic failure modes of specimens subjected to axial compressive loads (**P J Herrera-Franco, 2008**)

### 3.7.5 Impact test

Impact tests were carried out to determine the resistance material due to dynamic loading. Unnotched Charpy impact testing was performed on a Tinius Olsen TI 503 testing machine. Charpy impact tests which based on ISO 179 standard were carried out in the edgewise on 10 unnotched samples with a width of 10 mm and a thickness of 4 mm. The samples were loaded over a span of 60 mm at 3.5 m/s speed of pendulum weight 24.78 N. The impact energy was calculated by dividing the total absorbed energy by the cross sectional area of the sample.

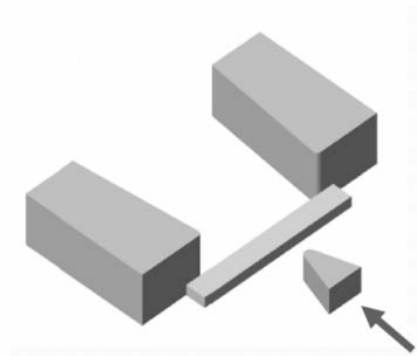


Figure 3.11 Schematic of impact test



### 3.7.6 SEM (Scanning Electron Microscope)

This microscope was used to analysis fracture surface of specimen. Fractographic study using scanning electron-microscopy (SEM) revealed the state of adhesion between the fibre and matrix. The SEM stubs are sputter coated with a thin layer of electrically conducting substances such as gold, gold/palladium alloy, platinum or graphite. The coating prevents the accumulation of static electric charge on the specimen during observation.



## CHAPTER IV RESULTS AND ANALYSIS

### 4.1 Tensile test

Figure 4.1 shows the stress vs strain curves for flax/PLA composites in tensile test. The tensile tests properties results for the composite material studied are shown in table 4.1.

Table 4.1 Tensile properties of PLA/Flax

Specimens	unit	Tensile test		
		Modulus of elasticity	Stress at break	Strain at break
		MPa	MPa	mm/mm
Pure PLA		2473.861 ± 33.661	55.065 ± 1.848	0.054 ± 0.013
PLA/Flax10		5414.932 ± 392.455	52.203 ± 0.708	0.021 ± 0.005
PLA/Flax20		6778.894 ± 278.496	56.144 ± 2.033	0.017 ± 0.005
PLA/Flax30		7144.335 ± 165.837	56.562 ± 1.992	0.013 ± 0.003

We can see that stiffness and ultimate stress gradually increased when fibre volume fraction ( $V_f$ ) is increased, whereas strain at break is decrease. The adding fibres in pure matrix increase the stiffness of the material about 120% to 190%, while the ductility reduce from 61% to 76%. In the other hand, fibres loading do not increase the stress at break for pure matrix. The evolution of modulus of elasticity with standard deviation is represented in Figure 4.2.

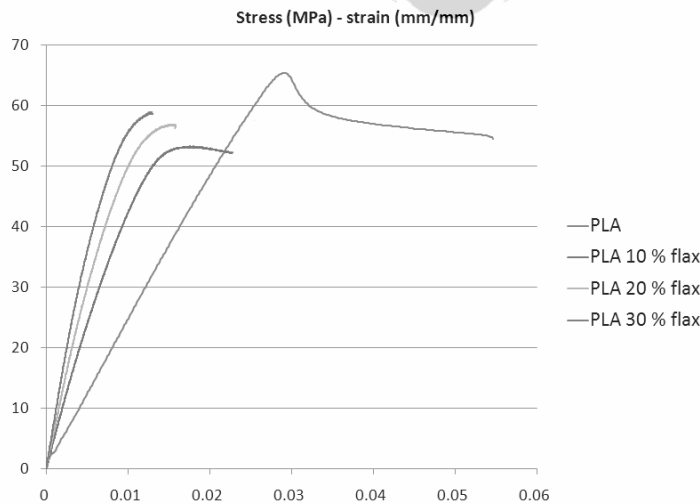


Figure 4.1 Stress vs strain curve of flax/PLA

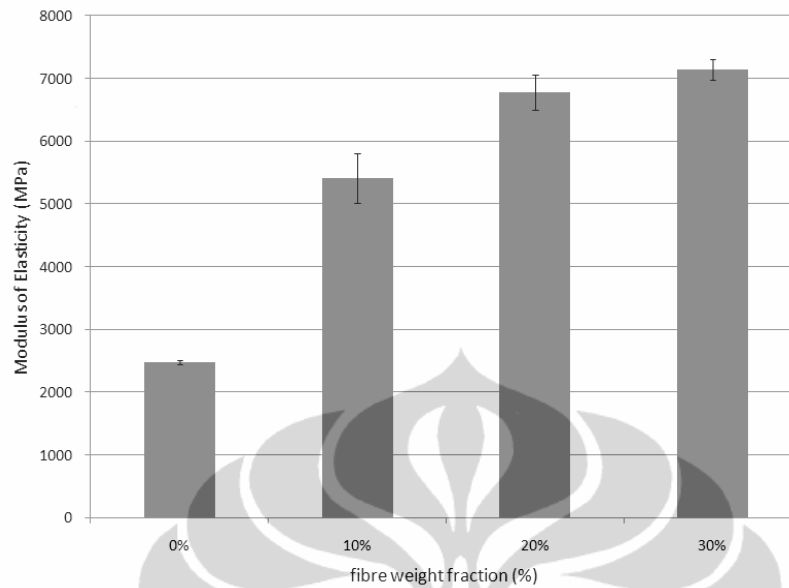


Figure 4.2 The evolution of modulus of elasticity

Figure 4.3 shows representative SEM of fracture surfaces of flax/PLA subjected to tensile stresses. In this figure, it can be noticed that fibre and matrix have a good adhesion. We can see that the fibre totally covered by PLA matrix.

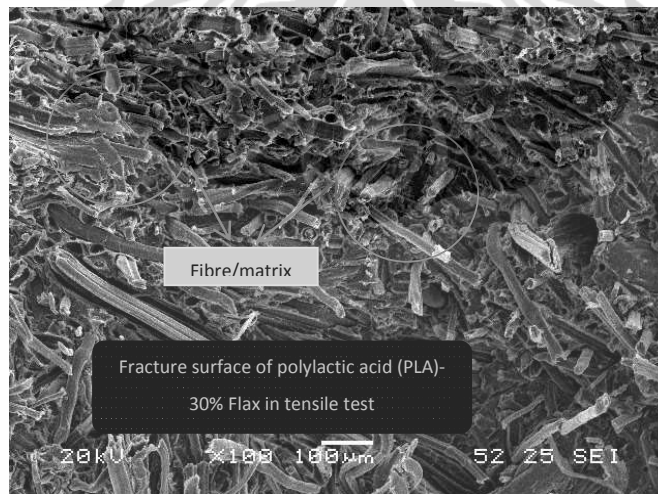


Figure 4.3 SEM micrographics of fracture surface for 70%PLA-30%Flax in tensile test

#### 4.2 Flexural test

The flexural tests properties results for composite material studied are shown in table 4.2. Stress – strain relation of flexural tests is presented in Figure 4.4. We can see that stiffness

and bending strength increase with increase the fibre loading. The effect of fibre loading can increase the stiffness of pure matrix from 2862 MPa to 5149 MPa. Its mean there is augmentation about 80%, even 158% for fibre loading 30% of flax. There are also increasing in bending strength about 2% until 10%. However, the strains are decreasing with adding fibres content.

Table 4.2 Flexural properties of PLA/Flax

Specimens	unit	Flexural test		
		Flexural modulus	Maximum stress	Strain at stress maximum
		MPa	MPa	mm/mm
Pure PLA		2862.813 ± 24.063	83.768 ± 1.776	0.043 ± 0.002
PLA/Flax10		5149.969 ± 111.206	85.498 ± 0.935	0.032 ± 0.001
PLA/Flax20		6110.623 ± 93.086	88.583 ± 3.193	0.026 ± 0.002
PLA/Flax30		7375.083 ± 312.391	92.259 ± 2.868	0.022 ± 0.001

The results of flexural tests, stiffness of flax/PLA is lower than in tensile tests about 5% – 11% except for fibre weight fraction 30%, whereas the ultimate stress is higher about 34% - 39%.

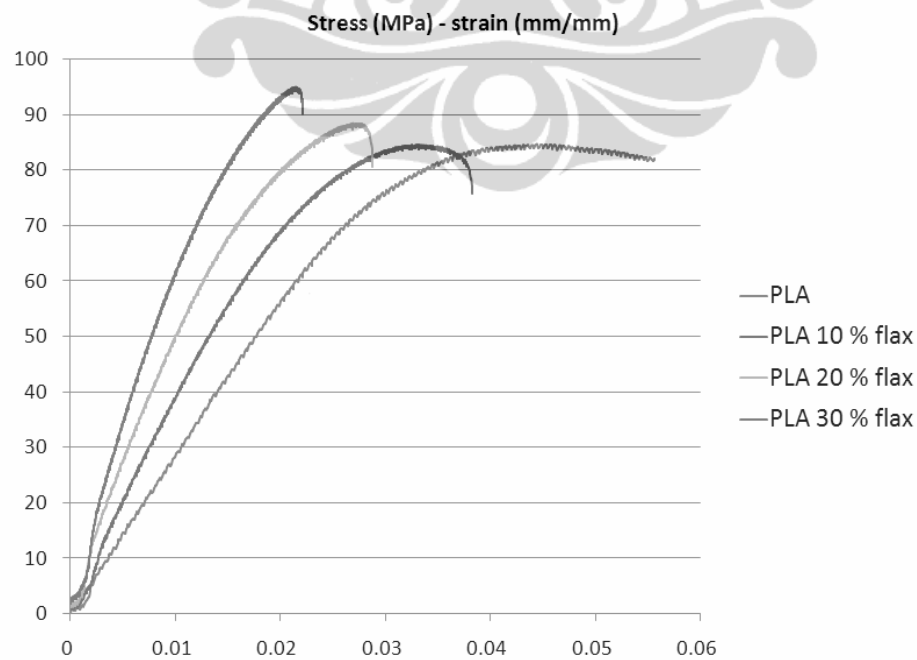


Figure 4.4 Stress - strain curve of flexural tests of flax/PLA

Visually, Figure 4.5 depicts the evolution of flexural modulus.

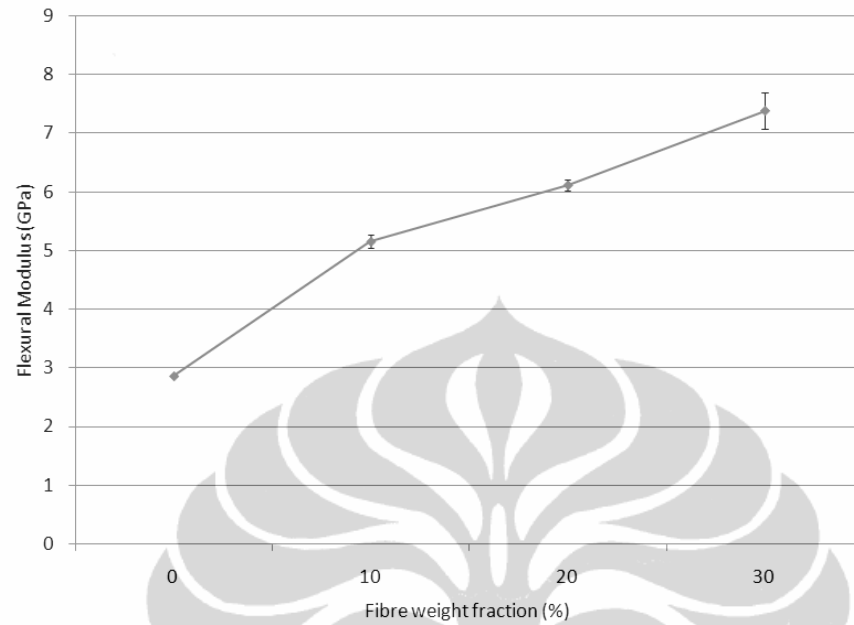


Figure 4.5 Flexural modulus of flax/PLA

#### 4.3 Torsion test

Shear stress – shear strain of flax/PLA in torsion tests are represented in Figure 4.6. The torsion tests properties results for the composite material studied are shown in table 4.3.

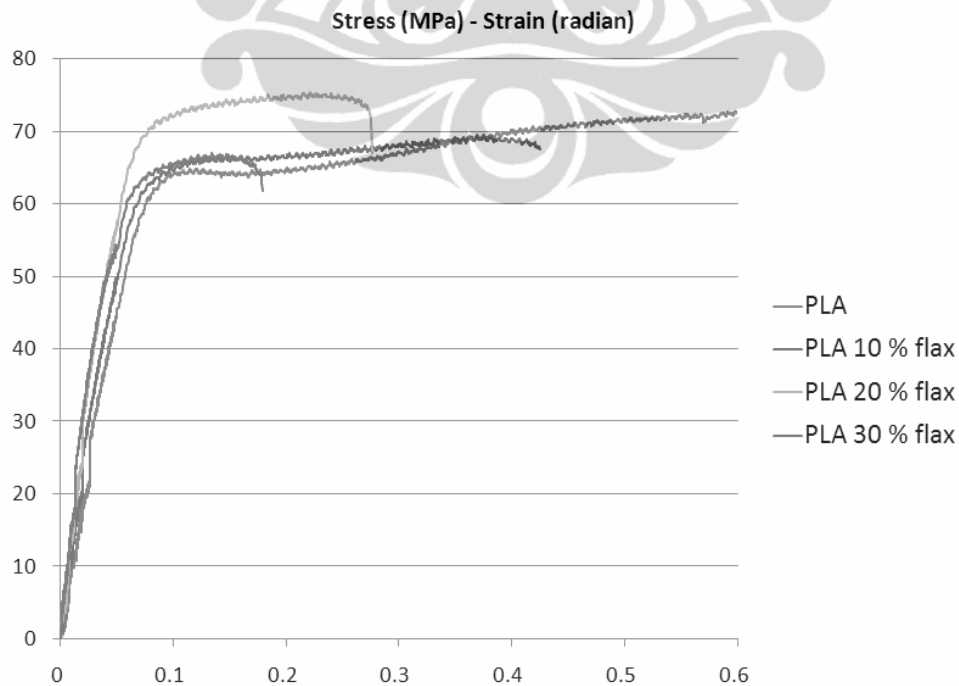


Figure 4.6 Shear stress - shear strain of flax/PLA

Table 4.3 Torsion properties of PLA/Flax

Specimens	unit	Torsion test		
		Shear modulus	Maximum stress	Strain at stress maximum
		MPa	MPa	mm/mm
Pure PLA		1008.334 ± 7.826	74.093 ± 3.551	0.627 ± 0.035
PLA/Flax10		1201.912 ± 47.672	69.289 ± 1.582	0.393 ± 0.030
PLA/Flax20		1380.407 ± 30.999	74.978 ± 3.196	0.202 ± 0.020
PLA/Flax30		1513.651 ± 42.212	71.428 ± 3.468	0.153 ± 0.024

It's clearly that the effect of fibre loading can increase the stiffness of material composite in twisted condition. Significance increments are shown in shear modulus from 19% to 50%, while augmentation in torsion strength is not so significance. Furthermore, adding fibre loading cause decrease in the ductility of the composite. The evolution of shear modulus is represented in Figure 4.7.

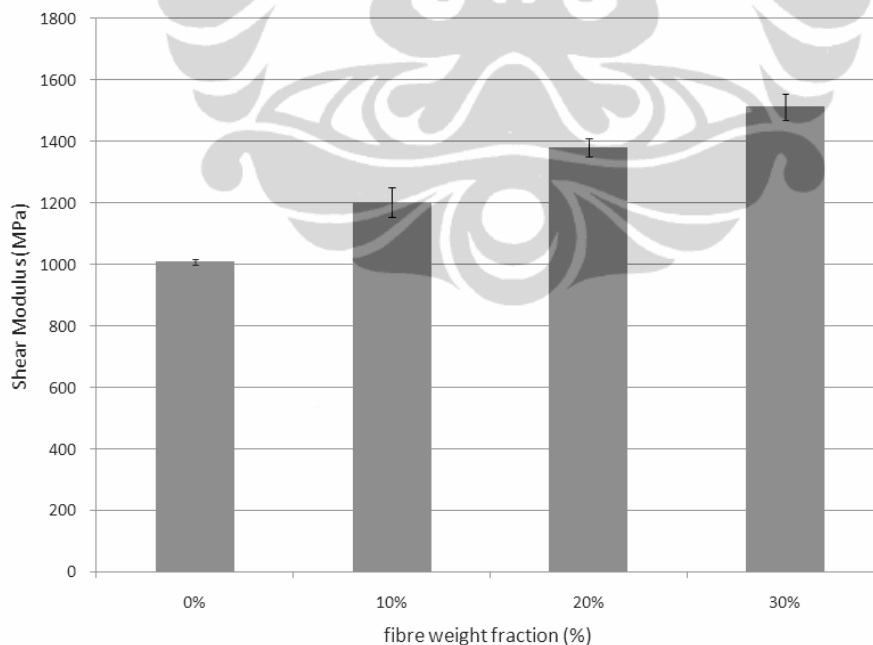


Figure 4.7 Shear modulus of flax/PLA



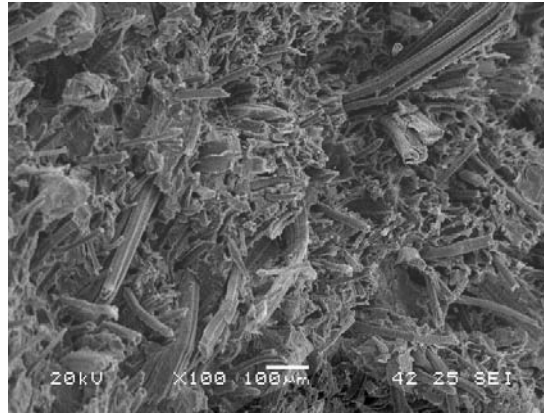


Figure 4.8 SEM micrographics of fracture surface for flax/PLA in torsion test

Twisted fractured surface of flax/PLA is represented in Figure 4.8. Based on micrographics picture, we can figure out that the PLA matrix suffers brittle fracture in torsion tests. It can be seen that there is no evidence that the matrix twisted, e.g. angle of matrix or fibres due to twist force.

#### 4.4 Compressive test

Figure 4.9 shows the compressive stress vs strain curves for flax/PLA composites. The properties in compression are presented in Table 4.4. Stiffness gradually increases with the fibre volume ratio. The maximum stress also increases, except for 20% of flax fibre content, while the strain at stress maximum decreases.

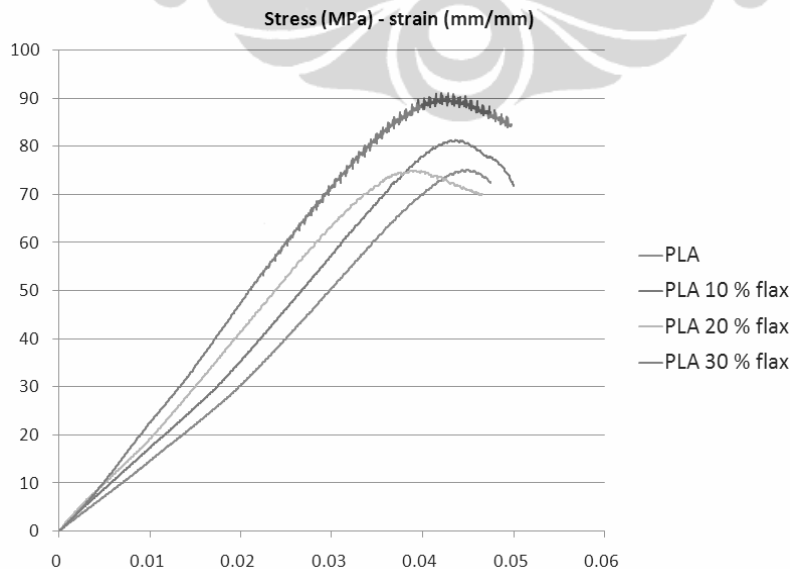


Figure 4.9 Stress - strain curve of flax/PLA in compressive tests

It's about 33% until 65 % the effect of fibre loading to the increasing of compressive modulus for pure PLA matrix. Small increment in maximum stress shown in 10% of flax fibre weight fraction comparing to pure PLA matrix, but there is a reduction for 20% of flax fibre content.

Table 4.4 Compressive properties of PLA/Flax

Specimens	unit	Compressive test		
		Compressive modulus	Maximum stress	Strain at stress maximum
		MPa	MPa	mm/mm
Pure PLA		1463.034 ± 87.588	76.668 ± 1.486	0.046 ± 0.002
PLA/Flax10		1939.814 ± 285.429	76.889 ± 10.271	0.042 ± 0.007
PLA/Flax20		1963.262 ± 207.194	72.610 ± 4.459	0.038 ± 0.002
PLA/Flax30		2407.385 ± 156.945	85.005 ± 5.791	0.042 ± 0.003

#### 4.5 Impact test

As mentioned before, the mechanical properties are significantly affected by the fibre content of the composites. This fact can be also observed in our impact investigations. The unnotched Charpy impact properties of the materials are presented in Figure 4.10. Impact strength is increase at higher fibre content. Nevertheless, the increment in impact strength is very little about 0.3% until 2.5%.

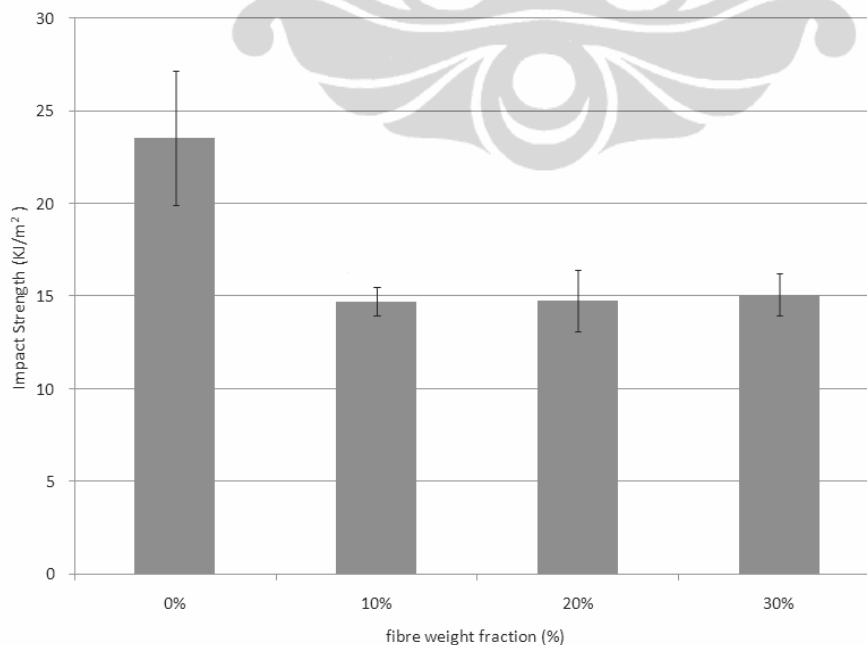


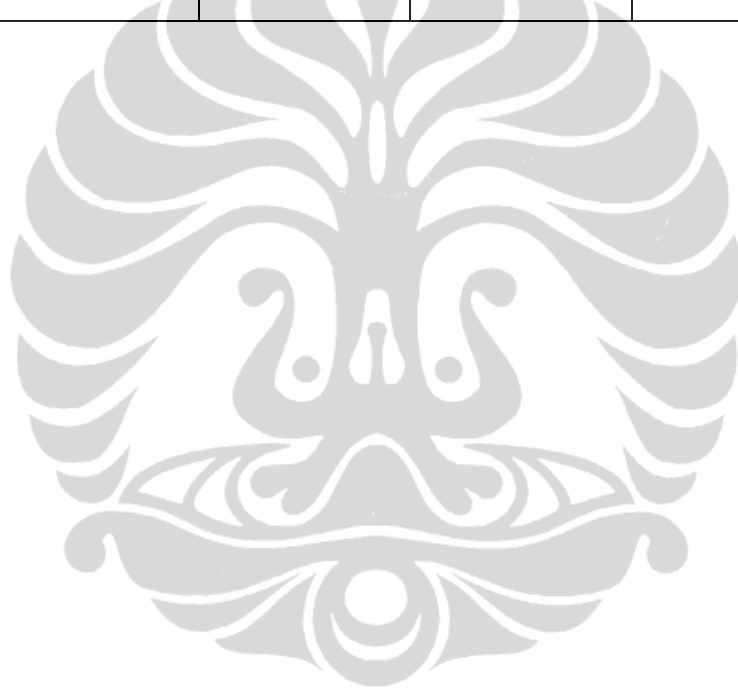
Figure 4.10 Impact strength of unnotched charpy flax/PLA



The complete results of impact test with standard deviation can be seen at Table 4.5. In fact that the impact strength decreases compared to pure matrix, it can be explained that adding short fibres in the pure matrix will increase internal void at tip of the fibres. (N. SATO, 1991). A crack will propagate more quickly when there are many defects in the material, e.g. voids.

Table 4.5 Impact properties of PLA/Flax

Matrix	Impact strength (KJ/m <sup>2</sup> )			
	Weight fibre fraction			
	0%	10%	20%	30%
PLA	23.529 ± 3.608	14.720 ± 0.789	14.769 ± 1.645	15.088 ± 1.118





## CHAPTER V

### CONCLUSION

The aim of this study is to find out the influence of fibre loading on mechanical properties of flax fibre reinforced Polylactic acid. Significant increment can be seen at all mechanical properties due to augmentation of fibre content in PLA matrix. In fact that small increment has shown at impact strength with addition of fibre content.

From research results showed the value of the modulus of each test is increasing with the increasing fibre volume fraction. It shows the stiffness composites increased with the increasing fiber volume fraction. The highest value of modulus of elasticity is 7144.33 Mpa for composites containing 30% flax fiber and 70% PLA and value of strength is 56.56 MPa .

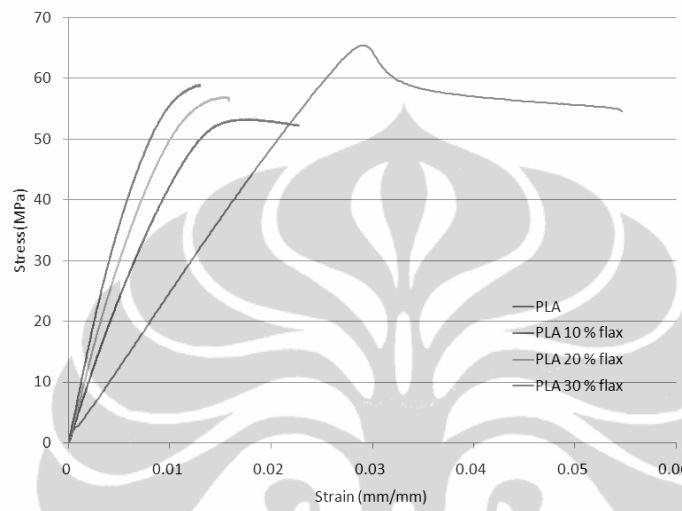
If compared with the value modulus of elasticity and strength of flax PP NMT (30% fibre volume fraction) is 5.20 GPa and 48 Mpa or the value modulus of elasticity and strength of glass PP GMT (30% fibre volume fraction) is 4.5 GPa and 70 Mpa (Bos, 2004) then this result showed that composites derived from natural fibers and biopolymer has a great potential to be developed as a replacement composite materials derived from synthetic polymers or glass fiber.

Scanning electron microscopy (SEM) studies of the composites failure surfaces indicated that flax/PLA shows brittle fracture. Even so, bio composite flax fibre and PLA matrix show that they have a good adhesion in fibre – matrix interfacial. It can be observed that all surface of the flax fibres are coated by PLA matrix.

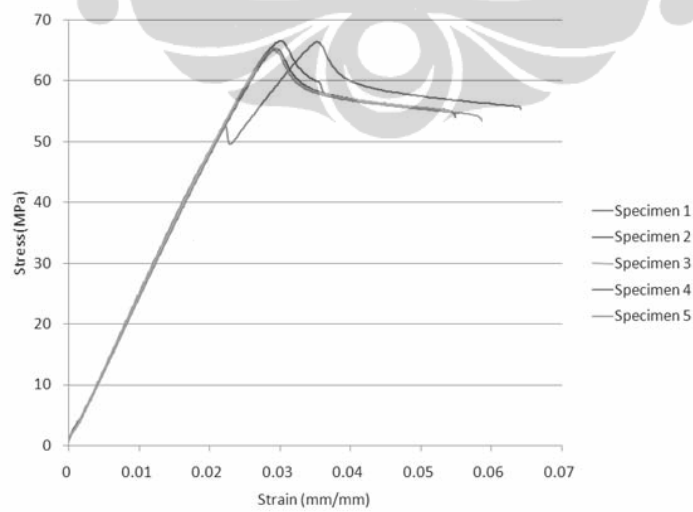
For future developments biodegradable composite was made PLA and flax fibre an interesting new candidate to substitute nonbiodegradable commodity polymers which will further expand the applications where this unique composite can be used.

### APPENDIX 1

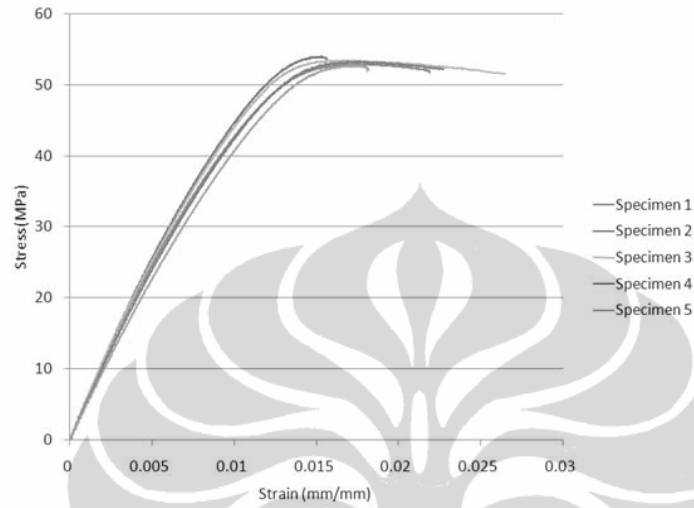
#### Stress - strain curve of tensile tests



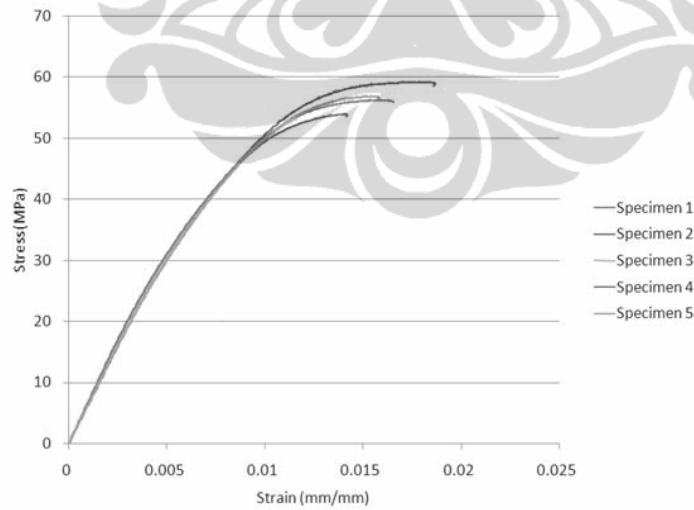
#### Stress - strain curve of tensile tests of flax/PLA



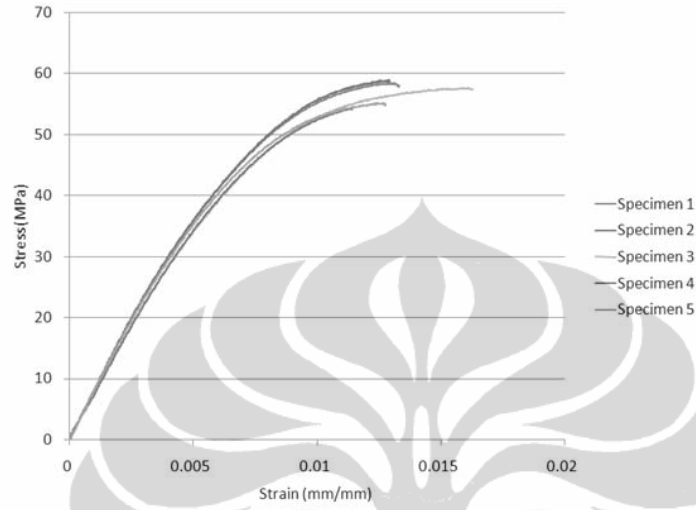
#### Stress - strain curve of tensile tests of pure PLA



Stress - strain curve of tensile tests of PLA-10% flax



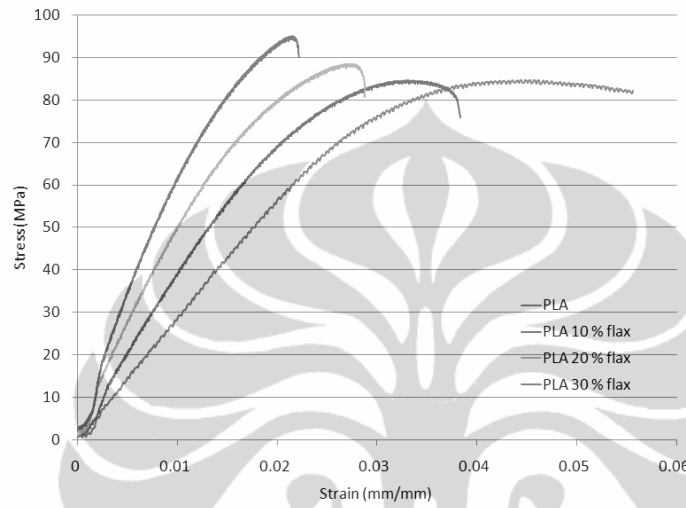
Stress - strain curve of tensile tests of PLA-20% flax



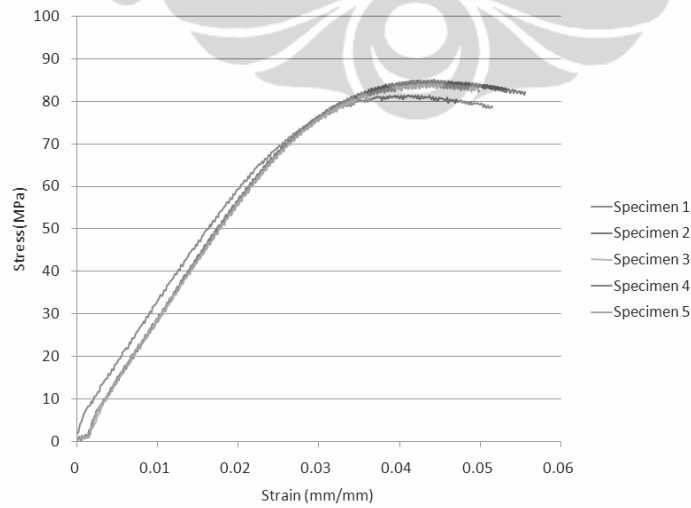
Stress - strain curve of tensile tests of PLA-30% flax

## APPENDIX 2

### Stress - strain curve of flexural tests

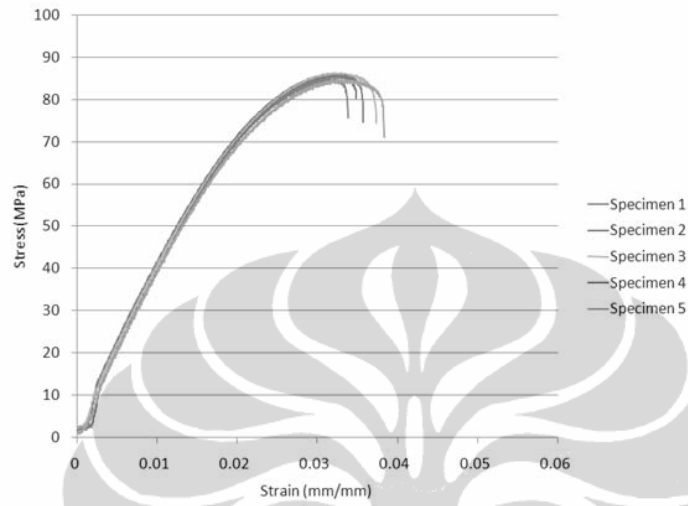


### Stress - strain curve of flexural tests of flax/PLA

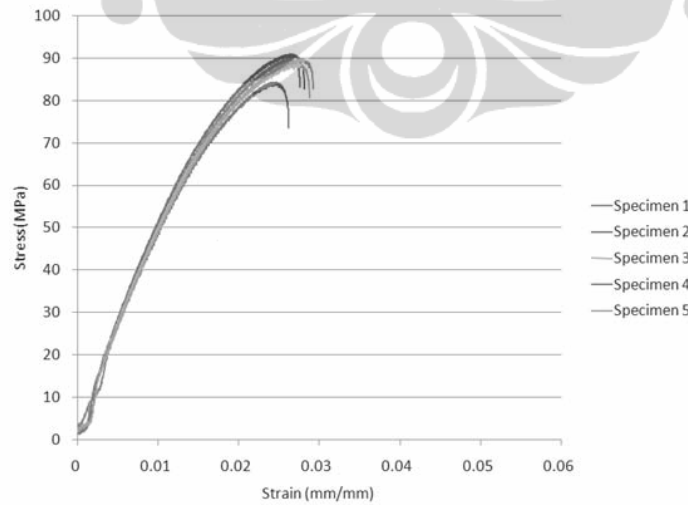


### Stress - strain curve of flexural tests of pure PLA

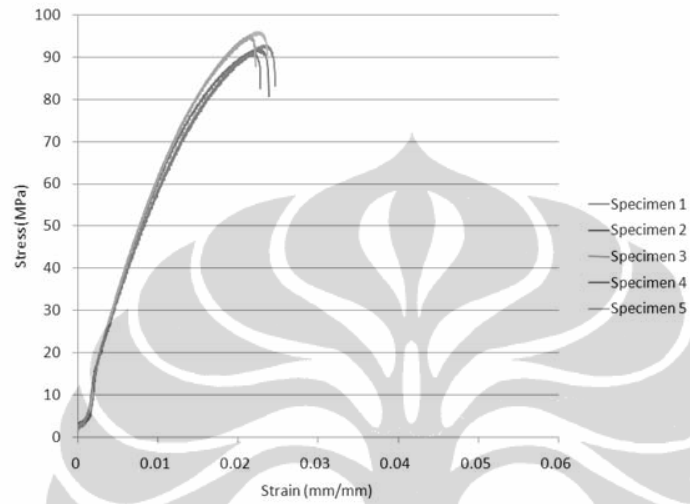




Stress - strain curve of flexural tests of PLA-10% flax



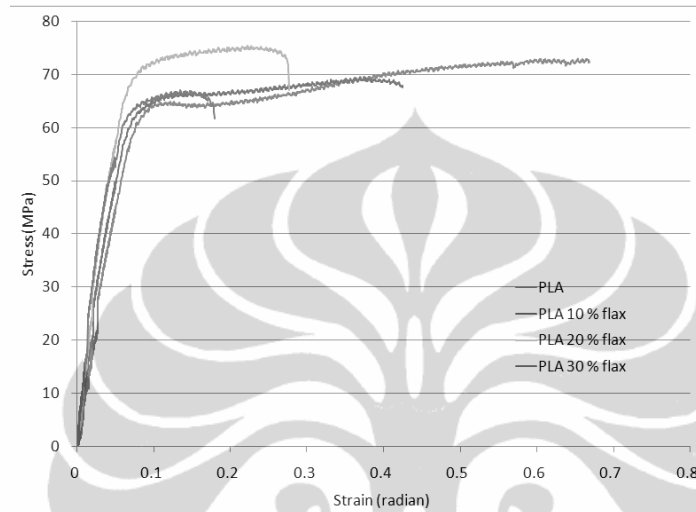
Stress - strain curve of flexural tests of PLA-20% flax



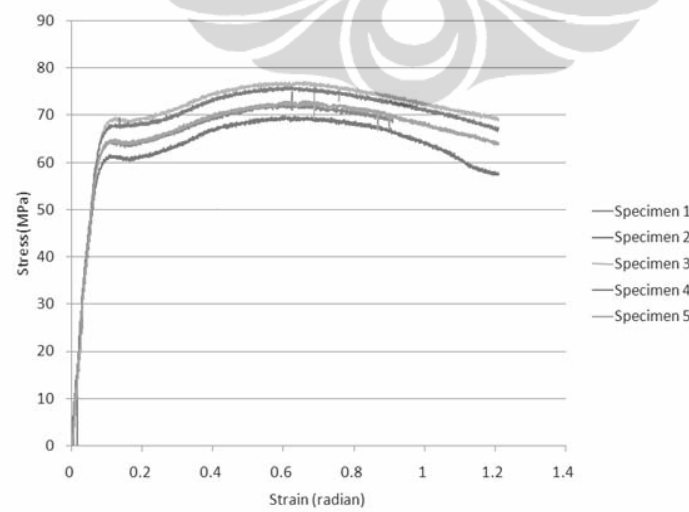
Stress - strain curve of flexural tests of PLA-30% flax

### APPENDIX 3

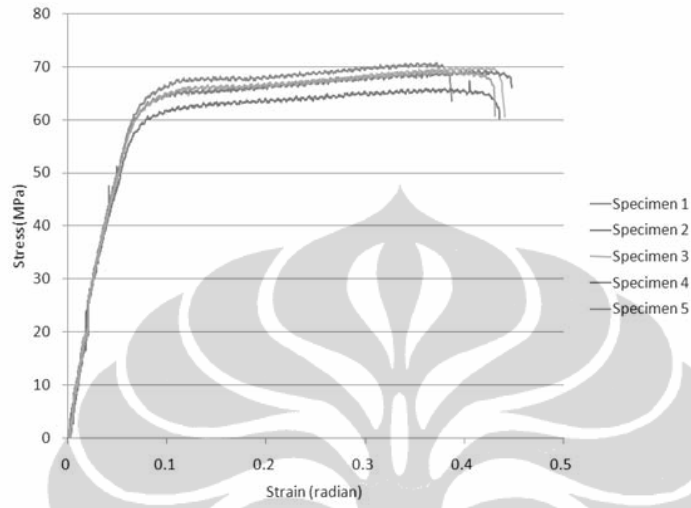
#### Stress - strain curve of torsion tests



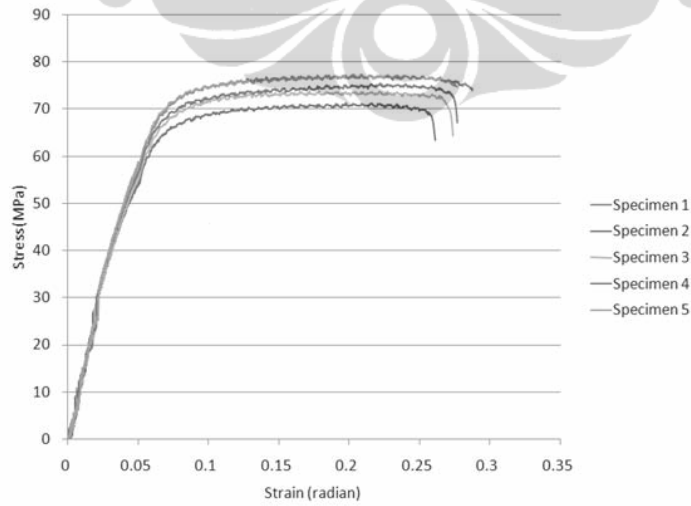
#### Stress - strain curve of torsion tests of flax/PLA



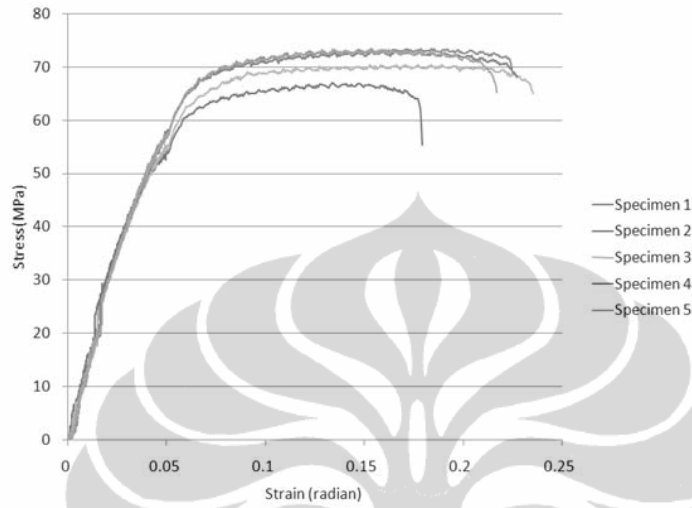
#### Stress - strain curve of torsion tests of pure PLA



Stress - strain curve of torsion tests of pure PLA-10% flax



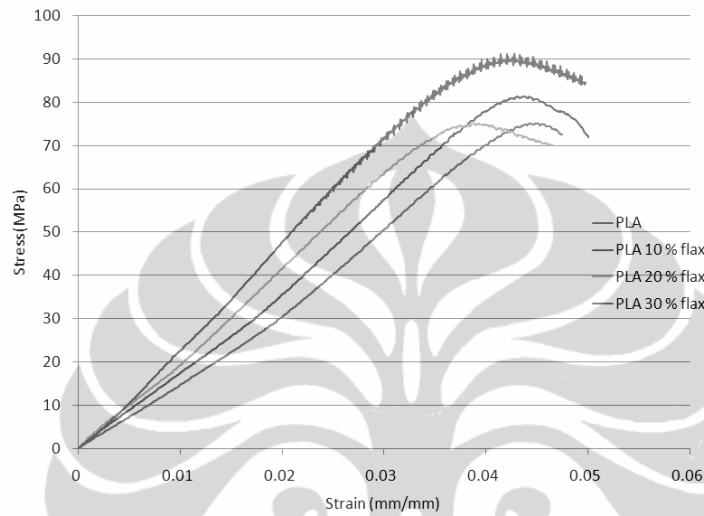
Stress - strain curve of torsion tests of pure PLA-20% flax



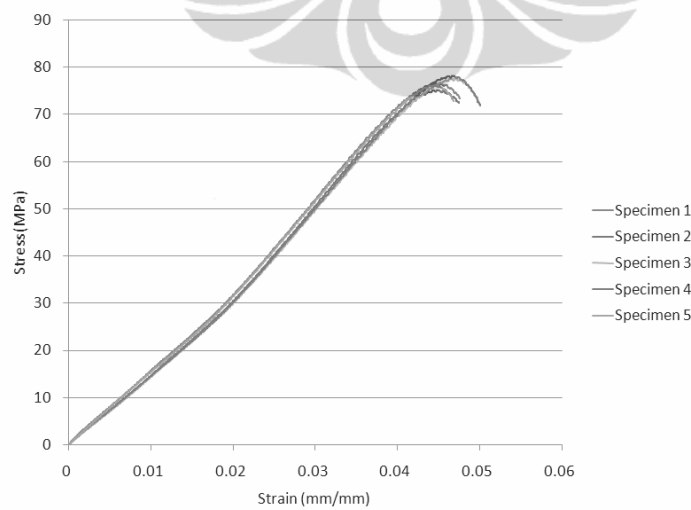
Stress - strain curve of torsion tests of pure PLA-30% flax

### APPENDIX 4

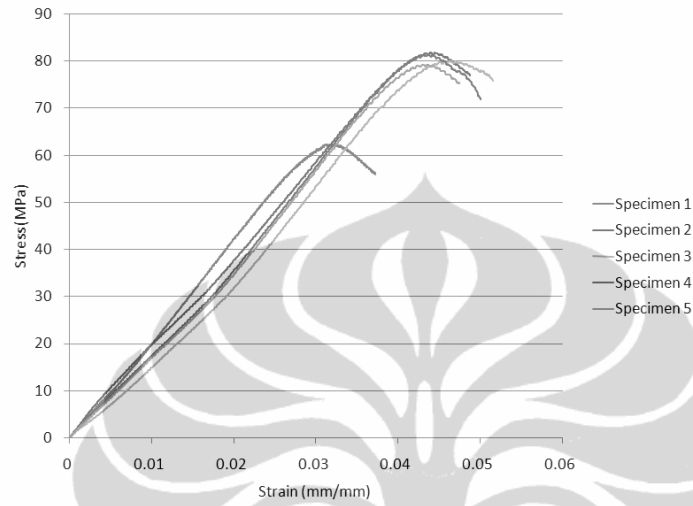
#### Stress - strain curve of compressive tests



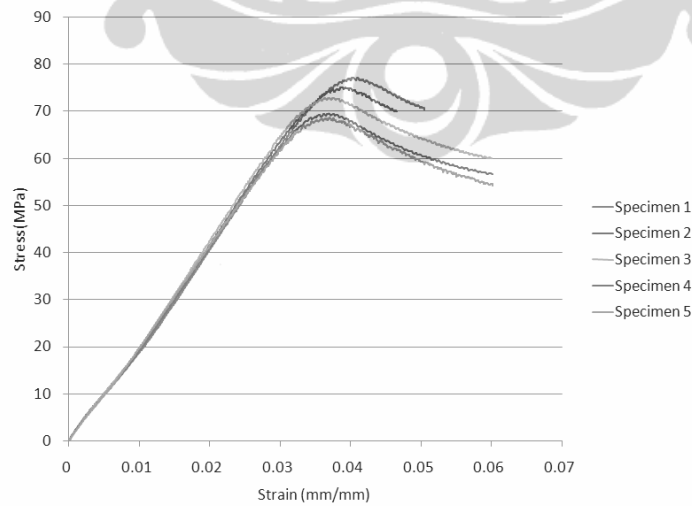
#### Stress - strain curve of compressive tests of flax/PLA



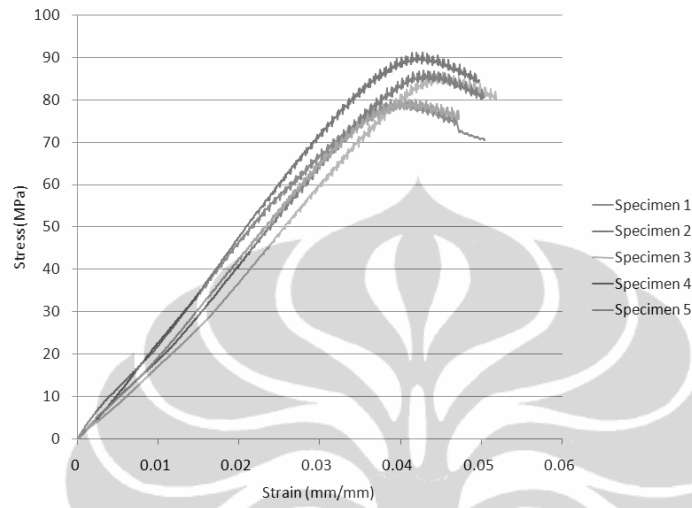
#### Stress - strain curve of compressive tests of pure PLA



Stress - strain curve of compressive tests of PLA-10% flax



Stress - strain curve of compressive tests of PLA-20% flax



Stress - strain curve of compressive tests of PLA-30% flax



**APPENDIX 5****The result of tensile tests**

The result of tensile tests of pure PLA

Specimen no.	Width mm	Thickness mm	Modulus MPa	Stress at break MPa	Strain at break mm/mm
1	10	3.900	2518.224	55.321	0.064
2	10	3.930	2449.809	53.998	0.055
3	10	3.930	2470.325	54.516	0.055
4	10	3.950	2454.498	58.115	0.036
5	10	3.960	2476.449	53.378	0.059
Average	10	3.934	2473.861	55.065	0.054
Standard deviation	0	0.023	33.661	1.848	0.013

The result of tensile tests of PLA-10% flax

Specimen no.	Width mm	Thickness mm	Modulus MPa	Stress at break MPa	Strain at break mm/mm
1	9.950	3.950	5604.669	51.707	0.022
2	10.000	3.950	5591.906	53.338	0.016
3	10.000	3.910	5630.503	51.520	0.026
4	10.000	3.910	5365.053	52.253	0.023
5	10.000	3.930	4882.526	52.196	0.018
Average	9.992	3.930	5414.932	52.203	0.021
Standard deviation	0.020	0.018	392.455	0.708	0.005



## The result of tensile tests of PLA-20% flax

Specimen no.	Width mm	Thickness mm	Modulus MPa	Stress at break MPa	Strain at break mm/mm
1	9.950	3.920	6883.350	55.911	0.017
2	10.000	3.910	6404.463	59.274	0.019
3	10.000	3.910	6818.270	55.265	0.012
4	10.000	3.910	6999.182	53.743	0.023
5	10.000	3.910	6789.207	56.526	0.016
Average	9.992	3.912	6778.894	56.144	0.017
Standard deviation	0.020	0.004	278.496	2.033	0.005

## The result of tensile tests of PLA-30% flax

Specimen no.	Width mm	Thickness mm	Modulus MPa	Stress at break MPa	Strain at break mm/mm
1	9.970	3.920	7153.806	57.735	0.011
2	9.980	3.890	7136.509	58.718	0.013
3	10.000	3.910	7023.507	57.447	0.016
4	10.000	3.910	7046.567	54.153	0.011
5	10.000	3.910	7361.285	54.757	0.013
Average	9.990	3.908	7144.335	56.562	0.013
Standard deviation	0.014	0.011	165.837	1.992	0.003

**APPENDIX 6****The result of flexural tests**

The result of flexural tests of pure PLA

Specimen no.	Width mm	Thickness mm	Flexural modulus MPa	Maximum stress MPa	Strain at stress maximum mm/mm
1	10.000	3.920	2877.374	81.322	0.040
2	10.000	3.930	2868.683	84.623	0.044
3	10.000	3.910	2842.499	83.907	0.044
4	10.000	3.910	2883.292	84.938	0.044
5	10.000	3.910	2842.216	84.050	0.044
Average	10.000	3.915	2862.813	83.768	0.043
Standard deviation	0.000	0.010	24.063	1.776	0.002

The result of flexural tests of PLA-10% flax

Specimen no.	Width mm	Thickness mm	Flexural modulus MPa	Maximum stress MPa	Strain at stress maximum mm/mm
1	10.000	3.920	5105.337	84.836	0.032
2	10.000	3.930	5199.889	86.093	0.032
3	10.000	3.910	5243.035	86.241	0.032
4	10.000	3.910	5016.788	85.750	0.033
5	10.000	3.910	5184.799	84.571	0.033
Average	10.000	3.915	5149.969	85.498	0.032
Standard deviation	0.000	0.010	111.206	0.935	0.001



The result of flexural tests of PLA-20% flax

Specimen no.	Width mm	Thickness mm	Flexural modulus MPa	Maximum stress MPa	Strain at stress maximum mm/mm
1	10.000	3.950	6086.456	89.764	0.026
2	10.000	3.930	6217.992	90.803	0.026
3	10.000	3.930	6088.212	88.434	0.028
4	10.000	3.950	6143.576	84.234	0.025
5	10.000	3.930	6016.878	89.679	0.028
Average	10.000	3.938	6110.623	88.583	0.026
Standard deviation	0.000	0.010	93.086	3.193	0.002

The result of flexural tests of PLA-30% flax

Specimen no.	Width mm	Thickness mm	Flexural modulus MPa	Maximum stress MPa	Strain at stress maximum mm/mm
1	10.000	3.950	7042.254	92.767	0.023
2	10.000	3.930	7474.045	91.863	0.023
3	10.000	3.930	7718.890	95.901	0.022
4	10.000	3.950	7255.084	90.940	0.022
5	10.000	3.930	7385.141	89.823	0.021
Average	10.000	3.915	7375.083	92.259	0.022
Standard deviation	0.000	0.010	312.391	2.868	0.001

**APPENDIX 7****The result of compressive tests**

The result of compressive tests of pure PLA

Specimen no.	Width mm	Thickness mm	Compressive modulus MPa	Maximum stress Mpa	Strain at stress maximum mm/mm
1	10	4	1536.132	76.465	0.045
2	10	4	1431.485	75.113	0.045
3	10	4	1384.919	77.556	0.047
4	10	4	1423.004	78.129	0.047
5	10	4	1539.631	76.079	0.045
Average	10	4	1463.034	76.668	0.046
Standard deviation	0	0	87.588	1.486	0.002

The result of compressive tests of PLA-10% flax

Specimen no.	Width mm	Thickness mm	Compressive modulus MPa	Maximum stress Mpa	Strain at stress maximum mm/mm
1	10	4	2087.484	76.465	0.031
2	10	4	2047.922	75.113	0.044
3	10	4	2175.183	77.556	0.046
4	10	4	1725.349	78.129	0.044
5	10	4	1663.133	76.079	0.043
Average	10	4	1939.814	76.668	0.042
Standard deviation	0	0	285.429	10.271	0.007



The result of compressive tests of PLA-20% flax

Specimen no.	Width mm	Thickness mm	Compressive modulus MPa	Maximum stress Mpa	Strain at stress maximum mm/mm
1	10	4	1864.607	77.114	0.040
2	10	4	2259.506	74.998	0.039
3	10	4	1905.279	72.826	0.037
4	10	4	1913.658	69.458	0.037
5	10	4	1873.260	68.656	0.037
Average	10	4	1963.262	72.610	0.038
Standard deviation	0	0	207.194	4.459	0.002

The result of compressive tests of PLA-30% flax

Specimen no.	Width mm	Thickness mm	Compressive modulus MPa	Maximum stress Mpa	Strain at stress maximum mm/mm
1	10	4	2492.499	80.378	0.040
2	10	4	2585.147	91.089	0.043
3	10	4	2294.175	86.435	0.045
4	10	4	2355.655	86.908	0.043
5	10	4	2309.452	80.216	0.041
Average	10	4	2407.385	85.005	0.042
Standard deviation	0	0	156.945	5.791	0.003

**APPENDIX 8****The result of torsion tests**

The result of torsion tests of pure PLA

Specimen no.	Width mm	Thickness mm	Shear modulus MPa	Maximum stress Mpa	Strain at stress maximum mm/mm
1	10	4	1000.044	74.883	0.623
2	10	4	1007.269	69.736	0.598
3	10	4	1017.768	76.997	0.659
4	10	4	1008.083	75.920	0.603
5	10	4	1008.508	72.928	0.654
Average	10	4	1008.334	74.093	0.627
Standard deviation	0	0	7.826	3.551	0.035

The result of torsion tests of pure PLA-10% flax

Specimen no.	Width mm	Thickness mm	Shear modulus MPa	Maximum stress Mpa	Strain at stress maximum mm/mm
1	10	4	1241.712	70.694	0.371
2	10	4	1138.821	67.263	0.405
3	10	4	1219.505	69.936	0.411
4	10	4	1206.747	69.257	0.414
5	10	4	1202.772	69.297	0.362
Average	10	4	1201.912	69.289	0.393
Standard deviation	0	0	47.672	1.582	0.030



The result of torsion tests of pure PLA-20% flax

Specimen no.	Width mm	Thickness mm	Shear modulus MPa	Maximum stress Mpa	Strain at stress maximum mm/mm
1	10	4	1365.471	74.97828	0.208
2	10	4	1345.294	74.97828	0.198
3	10	4	1404.054	74.97828	0.179
4	10	4	1385.537	74.97828	0.222
5	10	4	1401.679	74.97828	0.203
Average	10	4	1380.407	74.97828	0.202
Standard deviation	0	0	30.999	3.196	0.020

The result of torsion tests of pure PLA-30% flax

Specimen no.	Width mm	Thickness mm	Shear modulus MPa	Maximum stress Mpa	Strain at stress maximum mm/mm
1	10	4	1535.377	73.406	0.179
2	10	4	1526.886	66.983	0.134
3	10	4	1490.285	70.335	0.158
4	10	4	1548.866	73.087	0.160
5	10	4	1466.841	73.327	0.134
Average	10	4	1513.651	71.428	0.153
Standard deviation	0	0	42.212	3.468	0.024



**APPENDIX 9****The result of impact tests of PLA**

The result of impact tests of PLA

Specimen no.	Pure PLA	PLA-10% flax	PLA-20% flax	PLA-30% flax
1	20.8185	16.8073	12.9000	17.2335
2	31.2522	13.2476	17.4515	16.5903
3	21.3180	14.2660	17.3281	13.0072
4	19.8233	15.0491	13.8506	15.1237
5	17.8835	15.4117	17.4515	13.5489
6	29.2798	14.0259	13.5489	16.8974
7	22.0919	16.2225	14.5401	16.3222
8	27.9015	13.2607	10.3128	13.5645
9	21.3929	13.8688	15.7056	14.1736
10		15.5211	14.6012	14.4178
11		14.2346		
Average	23.529	14.720	14.769	15.088
Standard deviation	3.608	0.789	1.645	1.118



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