UNIVERSITAS INDONESIA **UNIVERSITÉ DE BRETAGNE-SUD**

SIFAT – SIFAT MEKANIK DARI FLAX/POLYPROPYLENE

TESIS

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FAKULTAS TEKNIK
PROGRAM STUDI TEKNIK MESIN
DEPOK
JULI 2011

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Diajukan sebagai salah satu syarat untuk memperoleh gelar Magister Teknik

MUHAMAD ARI
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HALAMAN SAMPUL

FAKULTAS TEKNIK
PROGRAM STUDI TEKNIK MESIN
KEKHUSUSAN PERANCANGAN DAN MANUFAKTUR PRODUK
DEPOK
JULI 2011

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Telah berhasil dipertahankan dihadapan dewan penguji dan diterima sebagai bagian persyaratan yang diperlukan untuk memperoleh gelar Master 2 Université de Bretagne-Sud (Perancis) dan Magister Teknik Program Studi Teknik Mesin, Fakultas Teknik, Universitas Indonesia.

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KATA PENGANTAR

Puji syukur saya haturkan pada Allah SWT, atas rahmat-Nya lah sehingga tesis ini bisa terselesaikan.

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ABSTRAK

Nama : Muhamad Ari

Program Studi : Tekik Mesin

Judul : Sifat – sifat mekanik dari flax/polypropylene

Polypropylene diperkuat serat flax dipersiapkan untuk mengetahui pengaruh penambahan serat dan *coupling agent* terhadap sifat – sifat mekanik dari komposit. Komposit flax/polypropylene dibuat dengan kandungan yang berbeda (10,20 dan 30 % berat). Sedangkan untuk *coupling agent* dengan tipe maleic anhydride polypropylene dilakukan penambahan 4% pada masing – masing komposisi komposit. Proses pembuatan material dilakukan dengan cara ekstrusi dan injeksi. Sifat – sifat mekanik yang diteliti yaitu tarik, tekan, tekuk, puntir dan impak. Modulus dan tegangan maksimum dari flax/polypropylene meningkat dengan penambahan serat. Pengaruh yang sama juga dihasilkan melalui penambahan *coupling agent*.

Kata kunci : *flax, polypropylene,* penambahan serat, *coupling agent*

ABSTRACT

Name : Muhamad Ari
Study Program: Mechanical Engineering
Title : Mechanical Properties of Flax/Polypropylene

Flax fibre reinforced polypropylene were prepared to investigate the influence of fibre loading and coupling agent on the mechanical properties of the composite. Flax/polypropylene composite was made with different content (10, 20 and 30 wt %). Whereas, coupling agent of maleic anhydride polypropylene was added by 4% to each composite composition. The process making of materials was performed by extrusion and injection. The mechanical properties of composite such as tensile, compression, flexural, torsion and impact were investigated. Modulus and maximum stress of flax/polypropylene increased with increase of fibres loading. The same effect was also generated through the addition of coupling agent.

Keyword: flax, polypropylene, fibre loading, coupling agent.

DAFTAR ISI

DAFTAR GAMBAR

DAFTAR TABEL

CHAPTER I. INTRODUCTION

1.1 Background

Humans have known composite materials for thousands years ago; it has started with natural fibres. The ancient Egyptians have used for construction where straw reinforced mud clay to build a wall. Composite also used by Mongolian and Japan as a material for making of weapons, such as bow and swords.

In the modern world, these materials develop since fibre glass was found in the early of 1930s. The World War II and cold war became trigger for the growth of these materials because of their benefit mechanical properties with specific strength and stiffness values that were significantly higher than existing structural materials. These properties were needed to build military aircraft at that time. In addition, existing aerospace structural alloys, such as those based on aluminium, were subject to corrosion and fatigue damage, and organic-matrix composites provided an approach to overcome these issues. For 3 decades, these materials have a huge improvement; it proved that many military and commercial aircrafts have been made with these materials. Dramatic improvements in structural efficiency became possible during this period, through the introduction of high-performance carbon fibres. Improved manufacturing capabilities and design methodologies provided the background for significant increases in composite material use for military and commercial aircraft and spacecraft structures. (Donaldson, 2001)

In line with aircraft industries, automotive and marine industries also followed this phenomenon and used it as part of their body car or boat. It has been reported at 2005 the usage of composite in both sectors are 23% and 6% for automotive and marine industries, respectively (Spurr, 2010). The presence of composite material provided new solution in the manufacture of machine parts or body structure of a car which has complicated form.

However, the using glass fibre in many applications cause problem at environment, since this material is not easy to be recycled when its lifetime has run out. It's not only that, many manufactured goods are based on non-renewable materials such as fossil fuels and minerals. Unfortunately, many problems have been associated with the utilization of these products such as increasing costs, and environmental degradation due to extraction, processing, and disposal.

Besides contributing negative impact to environment, composite manufacturers which use synthetic mineral fibre (glass, rock wool, ceramic fibre) as the main raw material get more serious health problems (skin and eye irritation, upper respiratory track irritation). (Fact sheet synthetic mineral fibre, 2004).

Concern about global warming has led to renewed interest in the more sustainable use of natural fibres in composite materials. Natural fibres are emerging as low cost, lightweight and apparently environmentally superior alternatives to glass fibres in composites (S.V. Joshi, 2004) and cause less abrasive wear to processing equipment, like extruder screw. Therefore, researchers have recently focused attention on bio composites. Development of bio composites as an alternative to petroleum based materials is addressing the dependence on imported oil, reducing carbon dioxide emission, and generating more economical opportunities for the agricultural sector. Moreover, bio composites offer opportunities for environmental gains, reduced energy consumption, insulation and sound absorption properties.

Since natural fibres are short fibre in nature. They are more suitable to produce parts with complex three dimensional structures in very high quantity and in short cycle time through injection moulding. Furthermore, a high demand for natural fibre injection-moulded parts is expected in the coming years.

1.2 Objective of the study

The aim of this study is to find out the effects of fibres loading and coupling agent on mechanical properties of flax short fibre reinforced polypropylene matrix.

CHAPTER II. BIBLIOGRAPHY REVIEW

2.1. Biopolymers

Biopolymers are polymers derived from natural sources. The emergence of biopolymer is stimulated by energy crisis in 1970s where most of the synthetic polymers are made from oil. Since that, many countries invested on biopolymer, including Germany which published a call for their researcher and scientists to perform some researches in this field. (E. Grigat, 1998) realize the request and found a material fully bio degradable (BAK 1095 and BAK 2195). Ability to decompose naturally makes the biopolymer materials are environmentally friendly and gives a solution to waste-disposal problems associated with traditional petroleum-derived plastics. So that's way biopolymer also called as biodegradable polymer.

Biodegradable polymers are a growing field. A vast number of biodegradable polymers have been synthesised or are formed in nature during the growth cycles of all organisms. Some microorganisms and enzymes capable of degrading them have been identified. One of the effort to make bio degradable polymer shown by (Ming Qiu Zhang, 2005) who convert wood flour into thermoplastic through chemical treatment. Furthermore, the material produced can be fully degradable by enzyme aided.

Table 2. 1 The main classes of plant-based biopolymers

Polysaccharides (plants/algae)	**Polyphenols**
Amidon Cellulose Agar, Alginate, carrageenan Pectin, Gommes, Konjac	Lignin, Tannins, humic acid
Polysaccharides (by bacterial fermentation)	**Polyesters**
Xanthane, Dextrane, Gellane, Curdlan Pullulane, Elsinane	Polymères d'acides lactiques (PLA) Polyhydroxyalcanoates (PHA)
Proteins	**Others polymers**
Zein, Gluten, amino Poly acids	Polymers synthesized from oil (nylon) Polyisoprenes : rubber

Depending to the evolution of the synthesis process, different classifications of the different biodegradable polymers have been proposed by scientists. Table 2.1 shows an attempt at classification base on plant source (Holy Nadia Rabetafika, 2006).

(AVEROUS, 2007) also made classification of biodegradable polymer base on its constituent materials which represented at Figure 2.1.

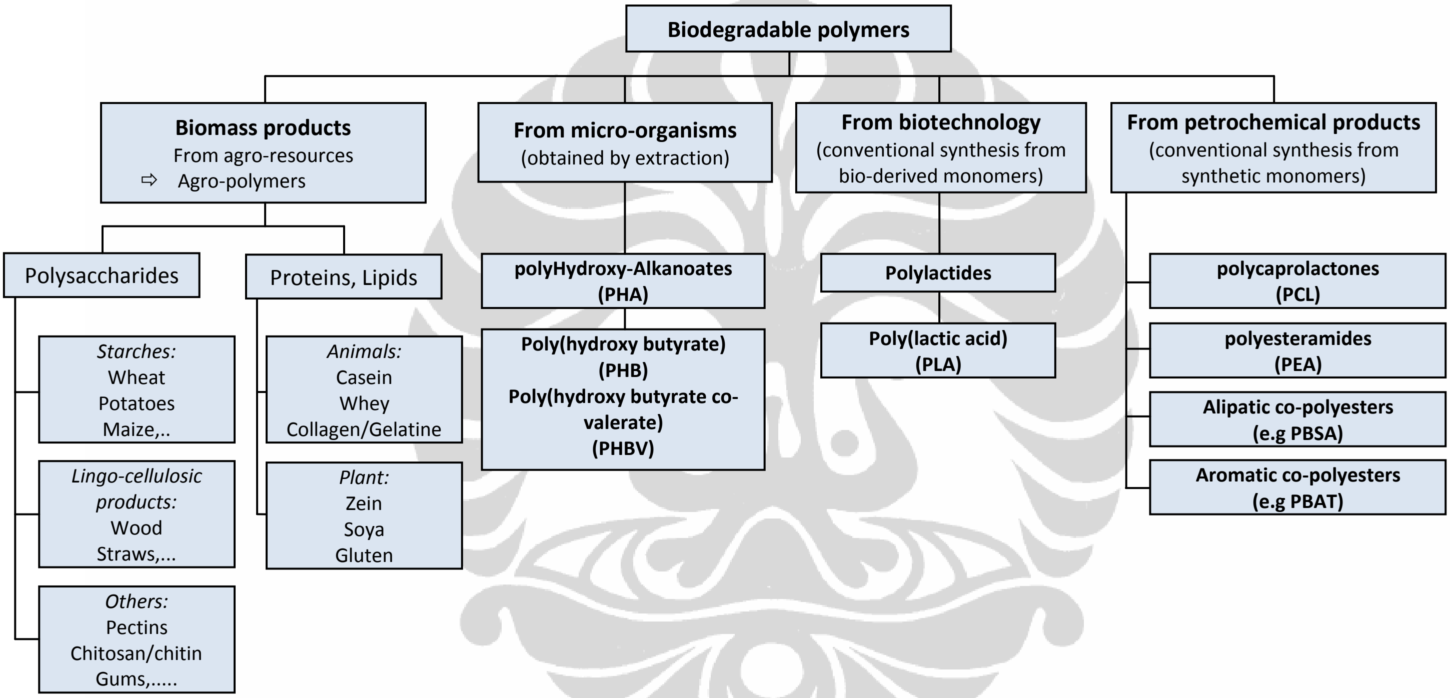


Figure 2. 1 Classification of the biodegradable polymers

We have 4 different categories. Only 3 categories (1 to 3) are obtained from renewable resources

1. Polymers from biomass such as the agro-polymers from agro-resources (e.g., starch, cellulose),
2. Polymers obtained by microbial production, e.g., the polyhydroxy-alkanoates,
3. Polymers conventionally and chemically synthesised and whose the monomers are obtained from agro-resources, e.g., the poly(lactic acid),
4. Polymers whose monomers and polymers are obtained conventionally, by chemical synthesis.

We can also classify these different biodegradable polymers into two main families: the agro-polymers (category 1) and the biodegradable polyesters (categories 2 to 4).

Recently, the usage of biopolymers to replace conventional polymers which derived from petroleum has applied in many sectors (e.g. food industry, surgery, packaging, etc). In the automotive sector, it was Henry ford who begun his experiment with making plastic parts for automobile in the early 1940s. At the time the plastics was made from soybean (Popular Research Topics, 2011).

2.2. Natural Fibre

Generally, natural fibres are coming from plants, animals or minerals. All plant fibres are composed of cellulose while animal fibres consist of proteins (hair, silk, and wool). Plant fibres include bast (or stem or soft sclerenchyma) fibres, leaf or hard fibres, seed, fruit, wood, cereal straw, and other grass fibres (Table 2.2). Asbestos is one of the mineral fibres, naturally occurring long mineral fibre.

Table 2. 2 List of vegetable and cellulose fibre classifications (James Holbery, 2006)

Blast	Leaf	Seed	Fruit	Stalk	Wood Fibres
Flax	Sisal	Cotton	Coconut	Bamboo	Hardwood
Hemps	Manila	Kapok	Coir	Wheat	Softwood
Jute	Curaua			Rice	(~10.000$^+$
Kenaf	Banana			Grass	varieties)
Ramie	Palm			Barley	
Banana				Corn	
Rattan					

So far as we know, plant fibres mainly consist of the following natural macromolecules: cellulose, hemicelluloses and lignin.

- Cellulose is a natural polymer with high strength and stiffness per weight, and it is the building material of long fibrous cells. These cells can be found in the stem, the leaves or the seeds of plants. It is also hydrophilic polymer. Therefore, all of the natural fibres are hydrophilic in nature.
- Hemicelluloses present along with cellulose in almost all plant cell walls. While cellulose is crystalline, strong, and resistant to hydrolysis, hemicelluloses have a random, amorphous structure with little strength. It is easily hydrolyzed by dilute acid.
- Lignin is a biochemical polymer which functions as a structural support material in plants. During synthesis of plant cell walls, polysaccharides such as cellulose and

hemicelluloses are laid down first, and lignin fills the spaces between the polysaccharide fibres, cementing them together. This ligninfication process cause a stiffening of cell walls, and the carbohydrate is protected from chemical and physical damage.

The chemical compositions and structural parameters of some important natural fibres are represented in table 2.3. As shown in the table that the chemical constituents of a specific natural fibre are varies and the amount of cellulose can vary depending on the species and age of the plant.

Table 2. 3 Chemical composition and structural of some natural fibres (A. K. Mohanty, 2000)

Type of fibre	Cellulose wt.-%	Lignin wt.-%	Hemi-celluloses wt.-%	Pectin wt.-%	Wax wt.-%	Micro-fibrillar/spiral angle (Deg.)	Moisture content wt.-%
Bast							
Jute	61-71.5	12-13	13.6-20.4	0.2	0.5	8.0	12.6
Flax	71	2.2	18.6-20.6	2.3	1.7	10.0	10.0
Hemp	70.2-74.4	3.7-5.7	17.9-22.4	0.9	0.8	6.2	10.8
Ramie	68.6-76.2	0.6-0.7	13.1-16.7	1.9	0.3	7.5	8.0
Kenaf	31-39	15-19	21.5	-	-	-	-
Leaf							
Sisal	67-78	8.0-11.0	10.0-14.2	10.0	2.0	20.0	11.0
PALF	70-82	5-12	-	-	-	14.0	11.8
Henequen	77.6	13.1	4-8	-	-	-	-
Seed							
Cotton	82.7	-	5.7	-	0.6	-	-
Fruit							
Coir	36-43	41-45	0.15-0.25	3-4	-	41-45	8.0

In plants, the cell wall plays a major role in ensuring the rigidity of the tissues. Plant cell wall is generally presented in the form of three elements: the middle lamella (rich in pectin substances), the primary wall and secondary wall (Fig.2.2):

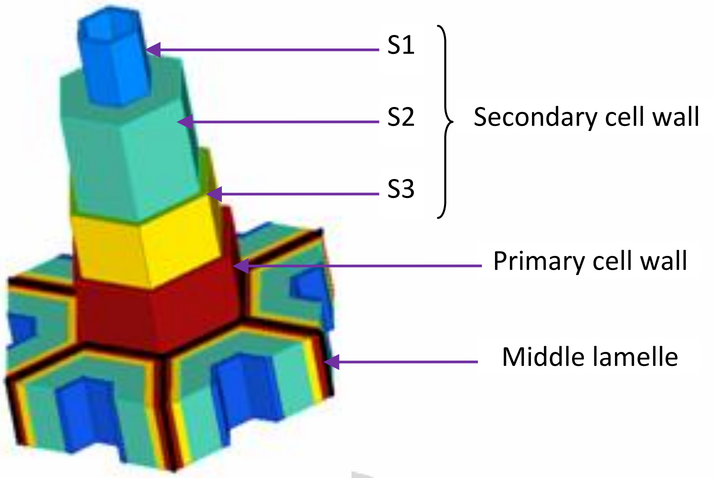


Figure 2. 2 The structure of a flax fibre cell (BALEY, 2004)

The primary wall appears as a loose network of cellulose microfibrils, encompassed in a highly hydrated amorphous matrix of pectin and hemicelluloses. The secondary wall, meanwhile, is an inextensible structure and weakly hydrophobic, consisting of cellulose and lignin. It has three layers (e.g. the walls of sclerenchyma), S1, S2 and S3, distinguished by the orientation of their cellulose fibres, and confer resistance to the wall. Poorly differentiated cells or during growth is mainly a primary wall. The latter is quite elastic and redesigned to ensure cell elongation. The differentiated cells have a wall for its primary and secondary wall, rigid. In some extreme cases such as dead cells of sclerenchyma (supporting tissue), the secondary wall can be very thick.

The natural fibres exhibit considerable variation in diameter along with the length of individual filaments. Quality as well as most of the properties depends on factors like size, maturity as well as processing methods adopted for the extraction of fibres. The modulus of fibres decreases with increase in diameter. The properties such as density, tensile strength and modulus are related to the internal structure and chemical of fibres. A comparison of properties of some natural fibres with conventional man-made fibres can be obtained from table 2.4.

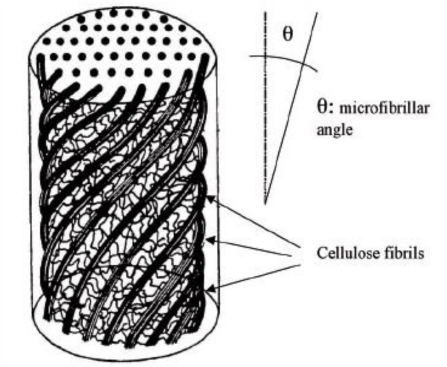


Figure 2. 3 Plant-fibre Structure (J.W.S.Hearle, 1963)

The strength and stiffness correlate with the angle between axis and fibril of the fibre,Fig.2.3 i.e. the smaller this angle, the higher the mechanical properties; the chemical constituents and complex chemical structure of natural fibres also affect the properties considerably. Coir shows least tensile strength among all the natural fibres as listed in Table 2.4 which is attributed to low cellulose content and considerably high microfibrillar angle as evidenced from Table 2.3. Again high tensile strength of flax may be attributes to its high cellulose content and comparatively low microfibrillar angle.

Table 2. 4 Comparative properties of some natural fibres with conventional manmade fibres. (A. K. Mohanty, 2000) (BALEY, 2004)

Fibre	Density g/cm^3	Diameter μm	Length mm	Ratio L/d	Tensile strength MPa	Young's modulus GPa	Elongation at break %
Cotton	1.5-1.6	-	-	-	287-800	5.5-12.6	7.0-8.0
Jute	1.3-1.45	25-200	-	-	393-773	13-26.5	1.16-1.5
Flax	1.50	5-76	4-77	1687	345-1100	27.6	2.7-3.2
Hemp	-	10-51	5-55	960	690	-	1.6
Ramie	1.50	16-126	40-250	3500	400-936	61.4-128	1.2-3.8
Sisal	1.45	7-47	0.8-8	100	468-640	9.4-22.0	3-7
PALF	-	20-80	-	-	413-1627	34.5-82.51	1.6
Coir	1.15	100-450	-	-	131-175	4-6	15-40
E-glass	2.5	-	-	-	2000-3500	70	2.5
S-glass	2.5	-	-	-	4570	86	2.8
Aramid	1.4	-	-	-	3000-3150	63-67	3.3-3.7
Carbon	1.7	-	-	-	4000	230-240	1.4-1.8

2.2.1. Flax

Flax belongs to 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 Fig.2.4 (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).

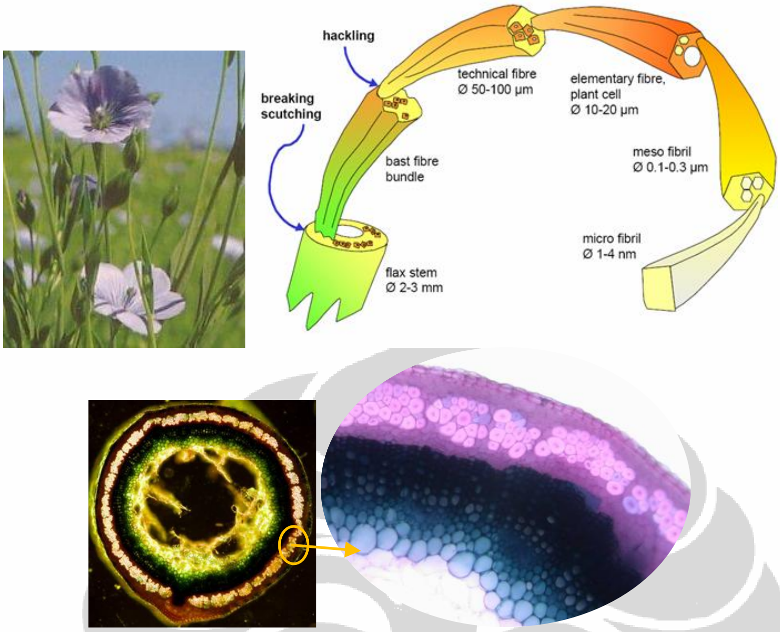


Figure 2. 4 Schematic of flax fibre

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 µm.

2.2.2. Hemp

Hemp is the common name for plants belonging to the genus Cannabis. Cannabis sativa L. subsp. sativa var. sativa is the variety grown for industrial fibre in Europe, Canada, China, and elsewhere, while C. sativa subsp. Indica with poor fibre quality is primarily grown for the production of recreational and medicinal drugs. It is an annual plant that grows in temperate climates. Hemp is considered as the oldest cultivated fibre plant in the world. The plants grow up to 4.5m (1.2 to 5 m) in height in approximately 140 to 145 days with a stem diameter of 4 to 20mm (Batra, 2007).Fig.2.5.

Figure 2. 5 Cultivating hemp plant

The fibre is the most valuable parts of the hemp plant. It is commonly called bast, which refers to the fibres that grow on the outside of the woody interior of the plant's stalk, and under the outer most part (the bark). Bast fibres give the plants strength

Figure 2. 6 Dried hemp stalk showing outer bast fibres and inner core or stick fibres

Figure 2.6 shows the bast and core or stick parts of the dried stalk. The bast fibres are covered by a thick layer of bark, which is removed by retting and represents about 15 to 25% of the total dry weight of the stalk. The average hemp bast ultimate fibre length is 25mm (5 to 55 mm) with an average fibre width of 25 μm (10 to 51 μm) and tensile strength is 690 MPa. (A. K. Mohanty, 2000).

2.2.3. Bamboo

Bamboos are giant grass which belongs to the family of the Bambusoideae. They are of notable economical and cultural significance in Asia, being used for building materials, as musical instruments, furniture, and a versatile raw product. The bamboo culm, in general, is a cylindrical shell, which is divided by transversal diaphragms at the nodes. Bamboo shells are orthotropic materials with high strength in the direction parallel to the fibres and low strength perpendicular to the fibres respectively.

Naturally, Bamboo is a composite material, consisting of long and parallel cellulose fibres embedded in a ligneous matrix. The density of the fibres in the cross-section of a bamboo shell varies along its thickness. This presents a functionally gradient material, evolved according to the state of stress distribution in its natural environment. As seen in Fig. 2.7, the fibres are concentrated in regions closer to the outer skin. This is consistent with the state of stress distribution when the culm is subjected to wind forces.

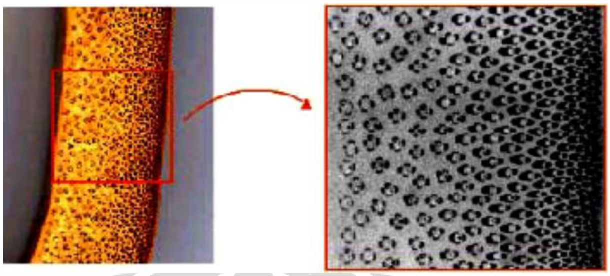


Figure 2. 7 Non-uniform fibre distribution on cross-section of bamboo **(Ghavami, 2005)**

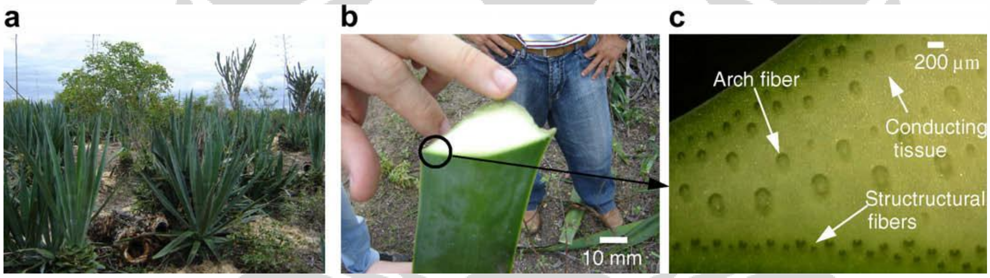
2.2.4. Jute

Jute is a tropical plant (genus Corchorus) belonging to the lily family. The stem reaches a height of 4 to 6 m with a diameter of about 3 cm (BALEY, 2004). C. capsularis (known as white jute) and C. olitorius (known as Tassa jute) are commercially grown in India and Bangladesh. The growing cycle for jute is 120 to 150 days with an average yield of 1700 kg/ha in warm and wet climates. The ultimate fibre has an average length of 2mm (2 to 5 mm) and an average width of 20 µm (10 to 25 µm).The fibre extraction technique is obtained by retting and decortications (Fig.2.8). After retting, disconnect the fibre is cleaned fibre and rinsed with water.

Figure 2. 8 Bangladesh raw jute

2.2.5. Sisal

Sisal fibre is one of the most widely used natural fibres in yarns, ropes, twines, cords, rugs, carpets, mattresses, mats, and handcrafted articles which grows with sword-shaped leaves about 1.5 to 2 m tall During the past two decades sisal fibres have also been used as reinforcement in cement and polymer based composites. Sisal fibres (Agave sisalana) are extracted from sisal plant leaves (see Fig. 2.9a) in the form of long fibre bundles. A sisal plant produces between 200 and 250 leaves before flowering, each of which contains approximately 700–1400 fibre bundles with a length of about 0.5–1.0 m. The sisal leaf consists of a sandwich structure composed of approximately 4% fibre, 1% cuticle, 8% dry matter, and 87% water (Flavio de Andrade Silva, 2008). Within the leaf, there are three basic types of fibres: structural, arch, and xylem fibres. The structural fibres give the sisal leaf its stiffness and are found in the periphery of the leaf (see Fig. 2.9b and c).



(Flavio de Andrade Silva, 2008)

Figure 2. 9 The sisal
(a) its plant,
(b) cross-section view, and
(c) optical microscopy of region selected in

2.2.6. Ramie

Ramie (Boehmeria nivea) is a family of arbuscular Urticaceae which is a flowering plant native to eastern Asia. It grows to 1 to 2.5m tall, with heart-shaped leaves 7 to15 cm long and 6 to12 cm wide (ROWELL, 2008). Fig.2.10 shows a growing ramie plant. The extraction of the fibre requires decortications and degumming very thorough. The fibres resemble other bast fibres such as jute and flax but are finer and are very strong in the dry state, but even stronger when wet. The bast fibres have a high degree of crystalline, making the fibres somewhat stiff and brittle, can reach 17 cm in length.

Figure 2. 10 Ramie plant

2.3. Behaviour of bio composite

Bio composites are the combination of *natural fibres (bio fibres)* such as wood fibres (hardwood and softwood) or non wood fibres (e.g., wheat, kenaf, hemp, jute, sisal, and flax) with polymer matrices from both of the renewable and non renewable resources. Behaviour of the composite it depend on the materials constituents. As a fibre reinforces polymer matrices, natural fibres play an important role in determining the properties of bio composite both physically and mechanically.

2.3.1. Physical behaviour

Advantages of natural fibres over traditional reinforcing materials such as glass fibres, talc and mica are low cost, low density, acceptable specific strength properties, reduced tool wear, ease of separation, enhanced energy recovery and biodegradability. The disadvantage of natural fibres is the relatively low processing temperature, because of the inability of natural fibres to resist temperatures higher than 150^{o}C for long processing durations and short-term exposures to temperatures up to 220^{o}C. Exceeding these temperatures can lead to discoloration, volatile release, poor interfacial adhesion and embrittlement of the cellulose components (James Holbery, 2006)

a. Moisture contents

The main drawback of natural fibres is their hydrophilic nature, meaning they absorb water. The fibres tend to swell when exposed to humidity, which causes a decrease in mechanical properties in the final composite. However, (Hanafi Ismail, 2002) prove that by adding coupling agent showed lower water absorption and thickness swelling than those without it.

b. Modulus - density

One of the advantages of natural fibres is having property of low specific weight, which results in a higher specific strength and stiffness than glass. This is a benefit especially in parts designed for bending stiffness. Fig. 2.11 describes performance natural material corresponding with others.

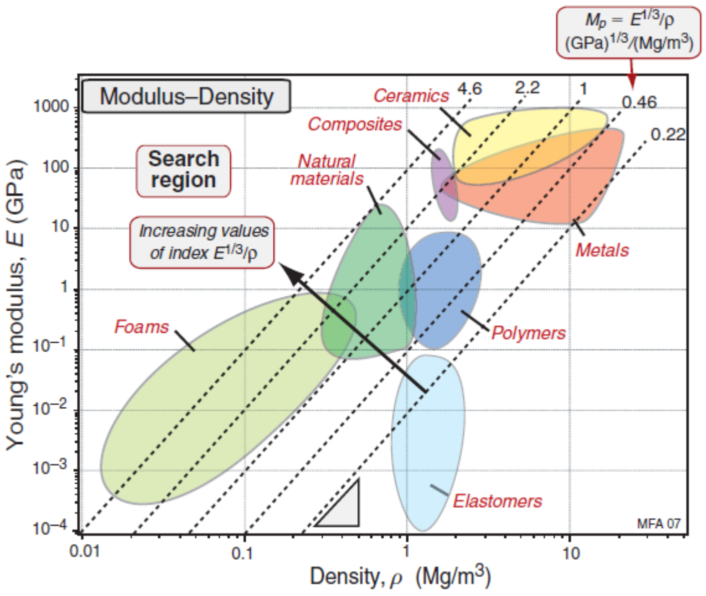


Figure 2. 11 The modulus–density chart **(Michael Ashby, 2007)**

It shows that all the materials that lie on a line of constant $M= E^{1/3}/\rho$ perform equally well as a light, stiff panel; those above the line perform better, those below less well. The figure shows a grid of lines corresponding to values of $M= E^{1/3}/\rho$ from M = 0.22 to M= 4.6. A material with M = 3 in these units gives a panel that has one-tenth the weight of one with M = 0.3.

2.3.2. Mechanical behaviour

Strength of composite materials depends on the properties of its material constituent. For bio composite, length of fibres plays main role to determine the mechanical properties, since natural fibres are naturally in short form. Furthermore, adhesion between fibres and matrix is interesting to evaluate.

a. Short fibre

Natural fibres, unlike man-made fibres, are not a continuous fibre but are in fact a composite by itself. Their schematic structure from stem to microfibril will be given in 3.1. This short fibres shape is disadvantage of the natural fibres. They have lower strength properties, particularly its impact strength. (N. SATO, 1991) has described that composite reinforced short fibres tend has microfailure at the tips of the fibres (Fig.2.12). This phenomenon can affect the mechanical properties of composite; because a crack can be easily propagate in the matrix material.

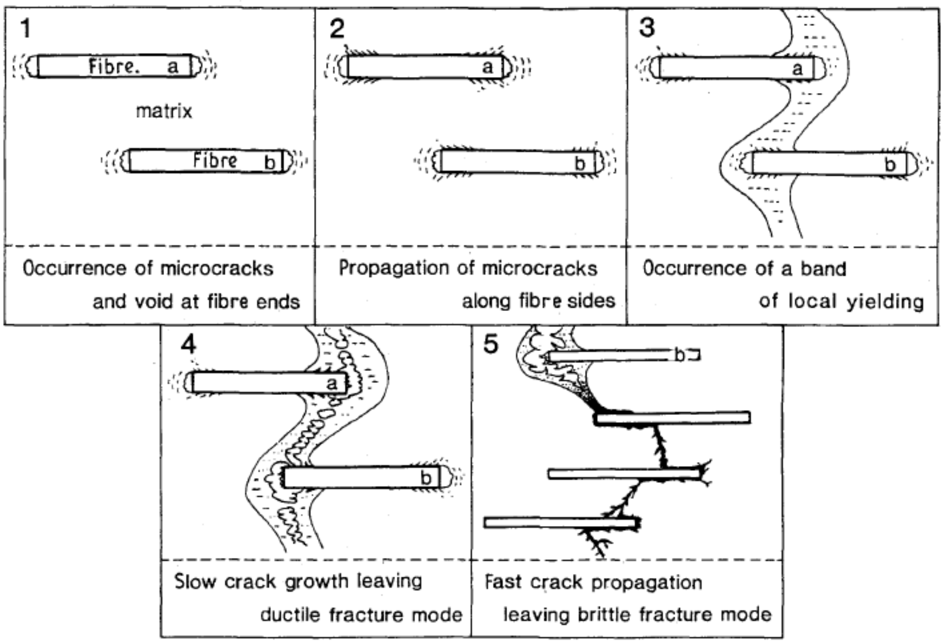


Figure 2. 12 Model of microfailure process of composites reinforced with short fibres

b. Fibre – matrix interfacial

It is well known that the interface between the reinforcing agent and the matrix plays a pivotal role in determining the mechanical properties of composite materials. Natural fibres consist mainly of cellulose, which is a natural polymer rich in hydroxyl groups. The remaining components of natural fibres (i.e. hemicelluloses, pectin and lignin) are also molecules rich in hydroxyl, carboxyl and other functional groups. Consequently, the fibres are usually strong polar materials and exhibit significant hydrophilicity. In other side, most polymer matrices tend to be apolar and mostly hydrophobic. This may contribute to a poor stress transfer between the matrix and the filler, and limited fibre dispersion in thermoplastic melt, leading to unsatisfactory final properties of the materials produced. This incompatibility between the hydrophilic natural fibres and the hydrophobic thermoplastic matrix can be overcome by fibre pre-treatment or the application of coupling agent to promote interfacial adhesion.

There are two method pre-treatment of fibre surface before mixing between fibre and polymer matrix, especially hydrophobic polymer.

I. Physical methods for surface modification

Physical treatments allow modifying the structural and surface properties of the natural fibres without the use of chemical agents, except mercerisation, which involves the use of sodium hydroxide or change the chemical composition. These methods include:

- heat treatments up to 180^{o}C at inert atmosphere.
- Corona treatment is a technique for surface activation by oxidation for example, to increase the rate of aldehyde groups on the surface of wood fibres
- Cold plasma treatments, which modify the surface energy of the fibres by formation of free radicals inducing cross-linking of surface particularly effective in the case of apolar matrices (PS, PP, PE ...),
- Alkaline treatments with sodium hydroxide, called mercerization, which were widely used in the case of textile fibres of cotton,
- Acetyl treatments.

II. Chemical methods for modification of surfaces

As its name, these methods use chemical agent to treatment fibre surface. These methods can be divided into two families:

- Treatments that modify the composition of fibre surface and create chemical bonds with the matrix, using such compounds as coupling agents (silane, titanate, zirconate), isocyanates, carboxylic acids (stearic acid),
- Functionalization treatments between the fibre surface and matrix by grafting in the presence of peroxides copolymers such as maleic anhydride grafted polypropylene

CHAPTER III. MATERIAL STUDIED AND EQUIPMENT

3.1 Flax

The flax fibre used in this study is from the Marilyn variety and comes from a 2002/2003 harvest. After steeping, the flax was scotched on the ground, combed, and cut to length. The length of fibres is 1 mm.

3.2 Polypropylene (PP)

Polypropylene (PP) is a thermoplastic polymer that has a wide range of usage. There are a number of reasons the usage of this material. First, it is easy to process and one of the cheapest polymers on the market which is of eminent importance in the ‘cost-performance’ sector. Secondly, it has a low processing temperature, which is essential because of the relatively low thermal stability of natural fibres (200–250^{0}C). Finally, it has good mechanical strength (better than polyethylene), excellent fatigue properties. In other word, it has the perfect ability to protect the hydrophilic natural fibre because of its strong hydrophobic.

Polypropylene is not bio degradable but liable to chain degradation from exposure to heat and UV radiation such as that present in sunlight. However, it is recyclable and has the number "5" as its resin identification code:

The polypropylene used in this study is 10 642 PPC supplied by Total Petrochemicals with an MFI of 44 g/10 min (230 °C - 2.16 kg). Except all above the reasons, this material is commonly used in industries, and in particular in the automotive industry.

3.3 Coupling agent

Coupling agent is a chemical substance capable of reacting with both the reinforcement and the resin matrix of a composite material. It may also bond inorganic fillers or fibres to organic resins to form or promote a stronger bond at the interface, especially natural fibre with polypropylene. The type of coupling agent was maleic-anhydride grafted PP (MAPP). It supplied by Arkema (Orevac CA 100) with an MFI of 10g/10min (190^0 C - 0.325 kg) was added to the matrix at weight fractions, 4%. Flax fibre contains functional hydroxyl groups that are able to interact chemically with the MAPP.

3.4 Production of specimen

In this experiment, composite material consisted of flax fibres length 1 mm was mixed with two matrixes (polypropylene without and polypropylene with coupling agent). Table 3.1 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 (ρ_f) and density of matrix (ρ_m).

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

Table 3. 1 List of composites composition

Materials		Matrix	Coupling agent	Fibres content	Fibres volume fraction
	unit	wt%	wt%	wt%	%
PP		100	-	-	-
PP		96	4	-	-
PP/flax		90	-	10	6.1
PP/flax		80	-	20	13.1
PP/flax		70	-	30	20.1
PP/flax		86	4	10	6.1
PP/flax		76	4	20	13.1
PP/flax		66	4	30	20.1

Specimens for this experiment were produced through three processes: extrusion, granulation and injection. These processes are illustrated in Fig.3.1.

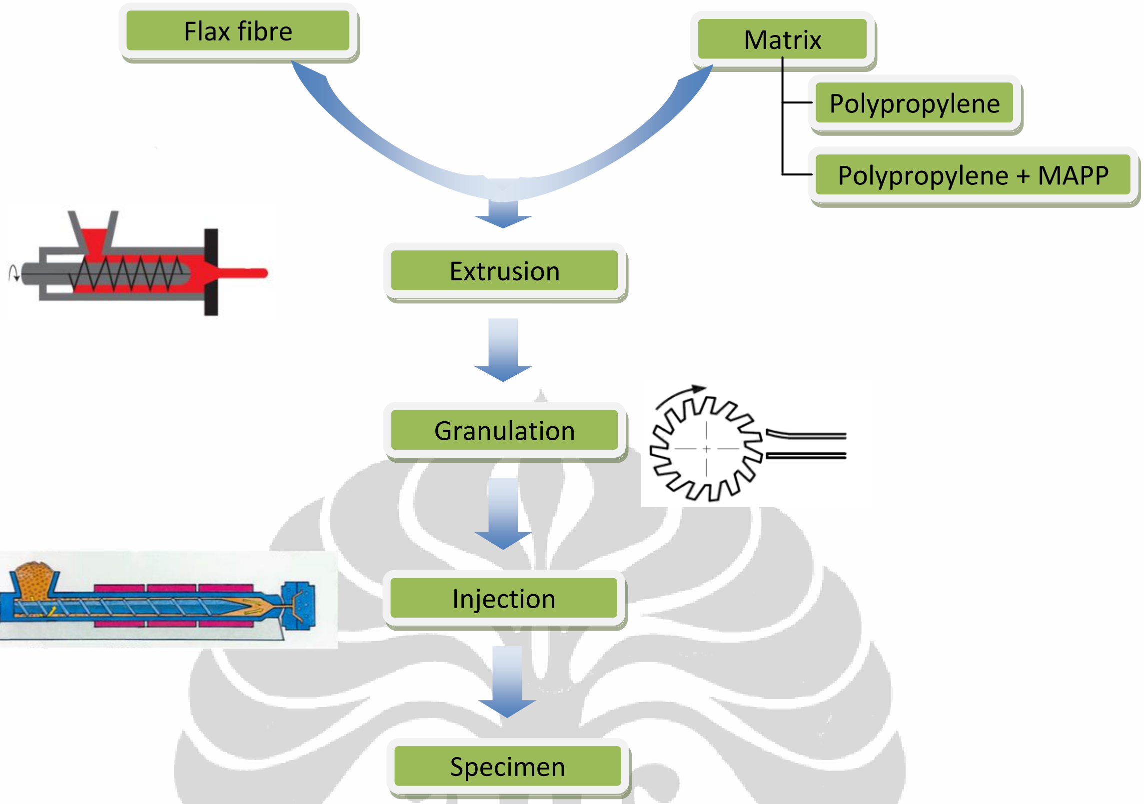


Figure 3. 1 Schematic of production of specimen

3.5 Extrusion machine

A screw extruder was used for the mixing and homogenizing of the flax fibre with the matrix material, polypropylene with and without coupling agent. The amount of coupling agent MAPP added is 4 wt. %. To expel moisture content, flax fibres were dried in an oven at 50^0C for 24 hours. The temperatures of the four zones of the extruder were 190^0 C (forming, metering, melting and compression and smooth feeding zones, Fig.3.2), this is to avoid degradation of natural fibres which mostly happen at 200^0C. In this process, the screw speed was adjusted to 30 rpm.

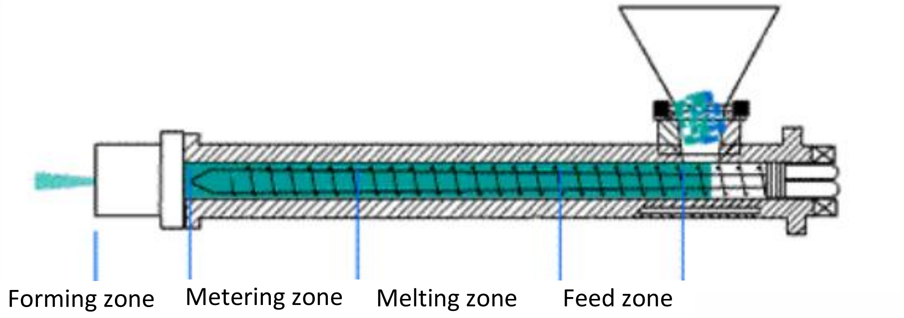


Figure 3. 2 Extrusion machine

The result of this process was the mixing between polypropylene (with or without coupling agent) with flax fibres in wire rod form at 4-5 mm diameter. The wire rod was left to cool at room temperature.

3.6 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.

3.7 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. 3 Injection machine

3.8 Testing method

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

3.8.1 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 10 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 Fig.3.4.

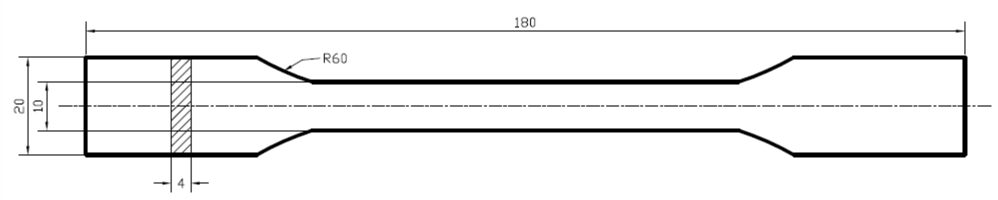


Figure 3. 4 Dimension of tensile test specimen

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

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

3.8.2 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 (Fig.3.5). 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)

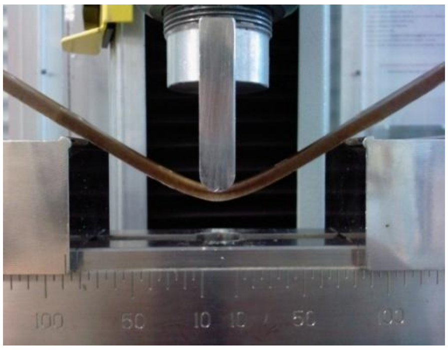


Figure 3. 5 Three-Point bending test

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.

3.8.3 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, *Φ*) 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 (Fig.3.6).

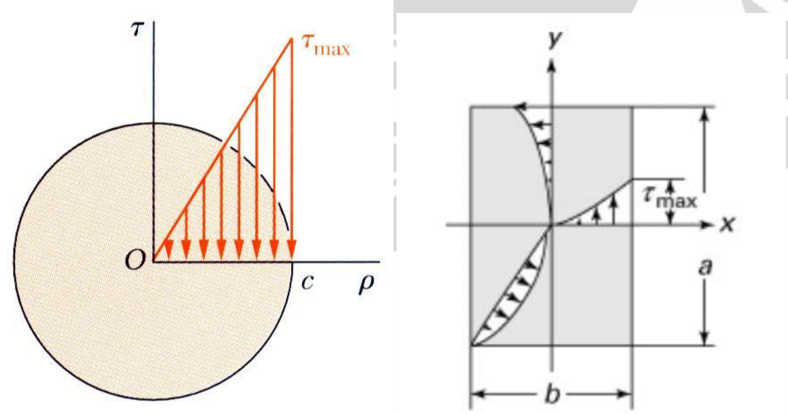


Figure 3. 6 Shear stress distribution

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 (Fig.3.7) and c_1 is a numerical factor depending upon the ratio a/b. Several values of c_1 are given in table 3.2.

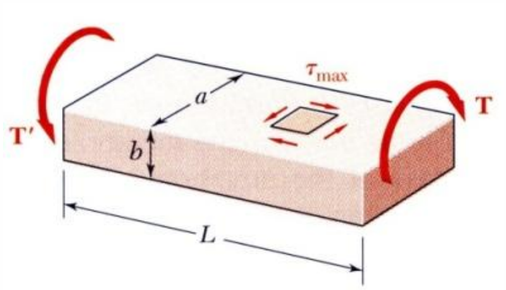
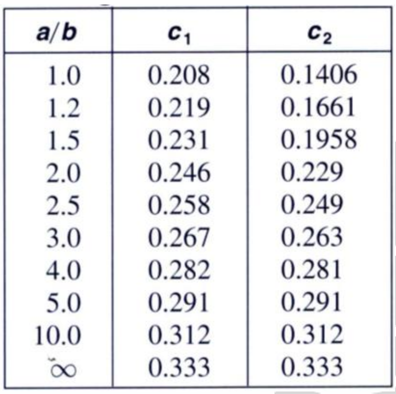


Figure 3. 7 Rectangular bars of torsion specimen

Table 3. 2 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
∞	0.333	0.333

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

$$\frac{\emptyset}{L} = \frac{T}{C_2 a b^3 G}$$

The values of the numerical factor c_2 are given in table 3.2.

3.8.4 Compression test

Shear loading method and configuration jigs compression tests as illustrated in Fig. 3.8a and 3.8b, 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.

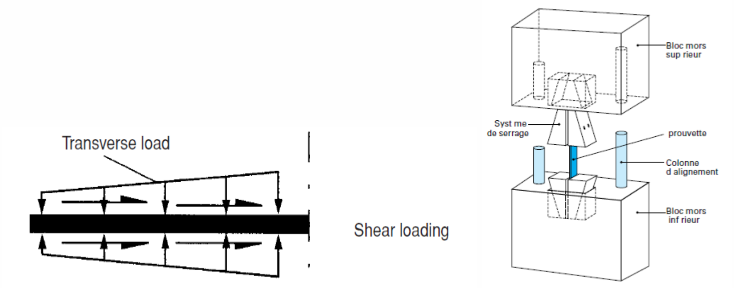
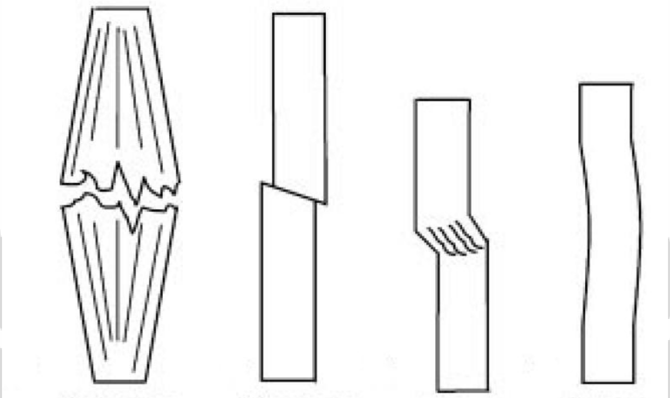


Figure 3. 8 a. Compression test method, b. Configuration of compression jigs

The typical failure mode of specimens in compressive test is represented in Fig.3.9.



a. Axial splitting b. Shear failure c. Kink zone d. Buckling

Figure 3. 9 Basic failure modes of specimens subjected to axial compressive loads. **(P J Herrera-Franco, 2008)**

3.8.5 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, Fig.3.10. 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.

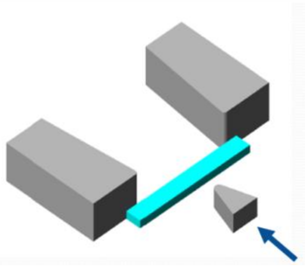


Figure 3. 10 Schematic of impact test

3.8.6 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

The tensile tests properties results for the composite material studied are shown in table 4.1. One can notice that generally stiffness and ultimate stress consistently increased when fibre volume fraction (V_f) is increased, whereas strain at stress maximum is decrease. The evolution of modulus of elasticity is represented in Fig. 4.1.

Table 4. 1 Tensile properties of flax/PP

Specimens	Tensile test		
	Elasticity modulus	Strength at break	Strain at break
unit	MPa	MPa	mm/mm
PP pure	1380.72 ± 19.23	20.68 ± 0.83	0.1838 ± 0.0574
PP 10% flax	3314.11 ± 294.51	23.22 ± 0.29	0.0910 ± 0.0207
PP 20% flax	3629.95 ± 256.62	20.43 ± 2.39	0.0730 ± 0.0060
PP 30% flax	4952.65 ± 209.47	27.20 ± 0.63	0.0443 ± 0.0067
PP+MAPP pure	1239.45 ± 28.12	18.98 ± 1.77	0.2229 ± 0.1315
PP+MAPP 10% flax	3323.49 ± 477.88	25.91 ± 1.15	0.0705 ± 0.0057
PP+MAPP 20% flax	4761.31 ± 127.64	36.83 ± 0.91	0.0310 ± 0.0045
PP+MAPP 30% flax	5666.65 ± 645.85	38.98 ± 1.77	0.0273 ± 0.0068

The other effect of fibre loading also can be seen at the stiffness and tensile strength of polypropylene reinforced by flax fibre compared to pure matrix. There is increasing about 140 – 360% in stiffness and 12 – 105% in tensile strength, whereas the strain reduces from 50 % to 90%.

The effect of coupling agent was investigated. Data in the table 4.1 shows that it can increase the stiffness and tensile strength of flax/PP composite until 15% and 45%, respectively. However, adding coupling agent reduced the ductility of the composite material of flax/PP about 22 – 40%, even all tensile properties of the pure matrix decreased.

Fig.4.2 shows representative photomicrographs of fracture surfaces of samples subjected to tensile stresses, for the specimen with and without coupling agent. The fibres surfaces of the specimen without coupling agent are completely free of any matrix material. This is an indication of fibre-matrix interfacial failure followed by extensive fibre failure and the ensuing fibre pull-out off the matrix

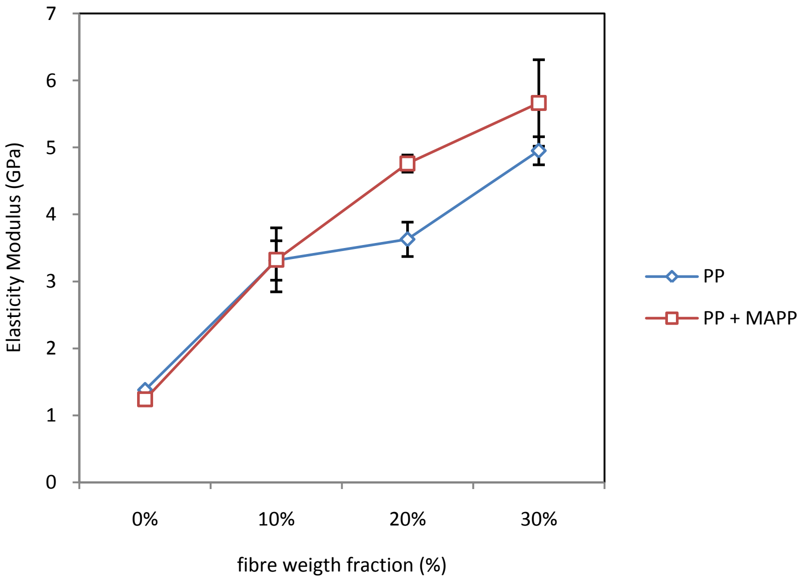
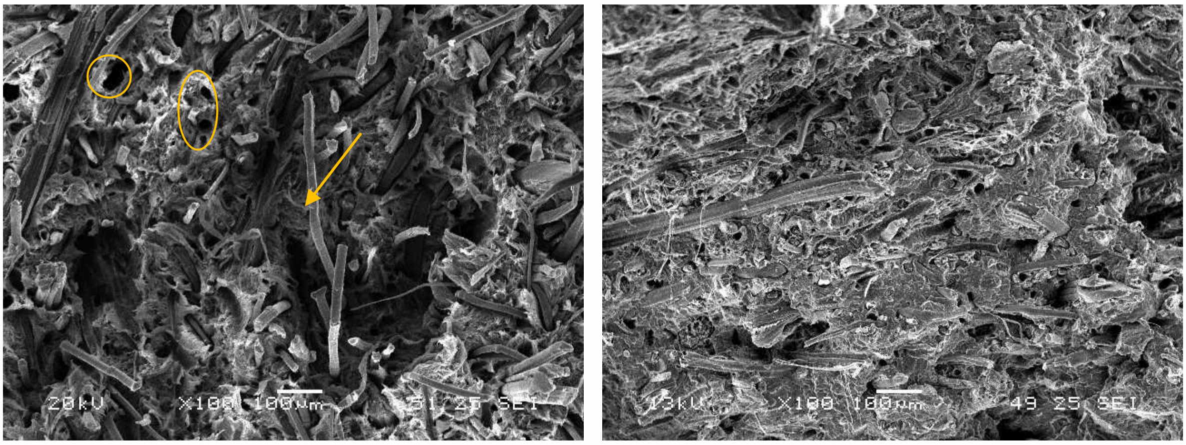


Figure 4. 1 The evolution of modulus of elasticity

In the other hand, flax/PP with coupling agent also shows interfacial failure but, in this case, there is no indication of fibre pull-out and the fibres are completely coated with the matrix. It can be said from these failure modes that the increase of the tensile strength of the composite is an effect of the fibre-matrix interface increases..



(a) (b)

Figure 4. 2 SEM micrographics of fracture surface for flax/PP in Tensile test;
a and b are without and with coupling agent, respectively.

It can also be said that a low fibre-matrix adhesion results in a failure mode dominated by fibre pull-out and matrix failure

4.2 Flexural test

The flexural tests properties results for composite material studied are shown in table 4.2. As well as, tensile properties, the results of flexural test show the same manner that fibre loading and coupling agent can increase mechanical properties, but in general reduce the strain of the material.

Table 4. 2 Flexural properties of flax/PP

Specimens		Bending test		
		Flexural modulus	Maximum stress	Strain at maximum stress
	unit	MPa	MPa	mm/mm
PP pure		1023.44 ± 9.49	27.78 ± 0.37	0.054 ± 0.002
PP 10% lin		1844.45 ± 56.11	31.97 ± 0.31	0.046 ± 0.001
PP 20% flax		2644.50 ± 90.20	34.78 ± 0.84	0.043 ± 0.002
PP 30% flax		3559.79 ± 129.36	39.45 ± 7.36	0.037 ± 0.005
PP+MAPP pure		902.02 ± 65.83	24.60 ± 0.75	0.054 ± 0.008
PP+MAPP 10% flax		2272.61 ± 44.90	33.83 ± 0.44	0.050 ± 0.002
PP+MAPP 20% flax		3214.79 ± 154.64	45.08 ± 1.26	0.044 ± 0.002
PP+MAPP 30% flax		4563.11 ± 166.41	52.22 ± 2.18	0.041 ± 0.001

Comparing to tensile properties, the stiffness of flax/PP in flexural test is lower about 56% – 81%, whereas the ultimate stress is higher about 20 -70%.

Visually, Fig. 4.3 depicts the evolution of flexural modulus. It can be seen that the adding of coupling agent reduce the stiffness of pure matrix, but increase the stiffness of polymer reinforced flax. As have been studied in the previous discussion that maleic-anhydride grafted PP (MAPP) as coupling agent can improve natural fibre – matrix adhesion.

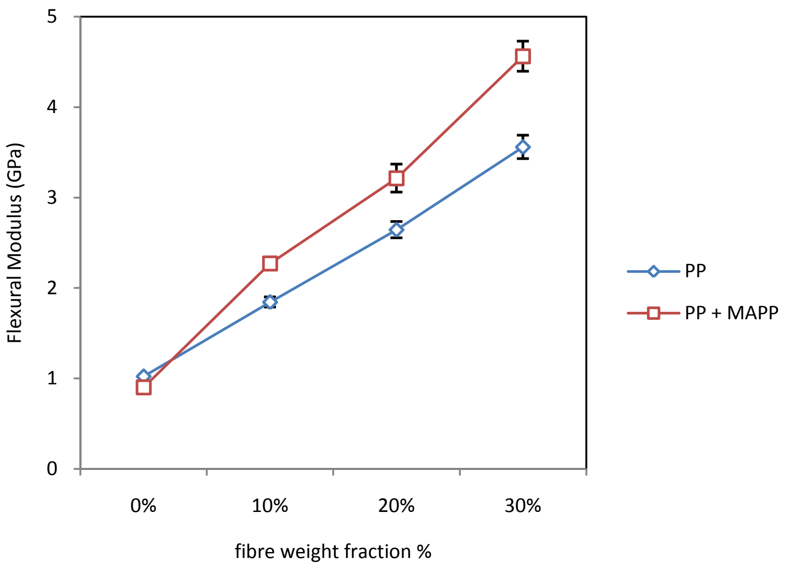


Figure 4. 3 Flexural modulus of flax/PP

4.3 Torsion

The torsion tests properties results for the composite material studied are shown in table 4.3. It's clearly that the effect of fibre loading can increase the stiffness of material composite in twisted condition. Significance increments are shown in flexural modulus and torsion strength up to 168% and 26%, respectively, while only 13% the augmentation increase in stiffness from pure PP with coupling agent matrix to 10% fibre weight fraction. However, adding fibre loading cause decrease in strain at stress maximum up to 80%. The evolution of shear modulus is represented in Fig. 4.4.

Table 4. 3 Torsion properties of flax/PP

Specimens	Torsion test		
	Shear modulus	Maximum stress	Strain at maximum stress
unit	MPa	MPa	mm/mm
PP pure	232.222 ± 14.068	33.105 ± 1.285	1.307 ± 0.214
PP 10% flax	304.578 ± 76.932	38.219 ± 5.538	1.285 ± 0.094
PP 20% flax	502.967 ± 49.055	41.786 ± 3.898	0.780 ± 0.087
PP 30% flax	622.914 ± 134.940	30.958 ± 6.017	0.414 ± 0.036
PP+MAPP pure	295.18 ± 16.367	38.570 ± 1.182	1.015 ± 0.130
PP+MAPP 10% flax	332.168 ± 11.424	44.714 ±1.302	1.052 ± 0.092
PP+MAPP 20% flax	550.132 ± 20.080	45.783 ±5.145	0.202 ± 0.020
PP+MAPP 30% flax	712.358 ± 134.940	47.204 ± 6.017	0.400 ± 0.036

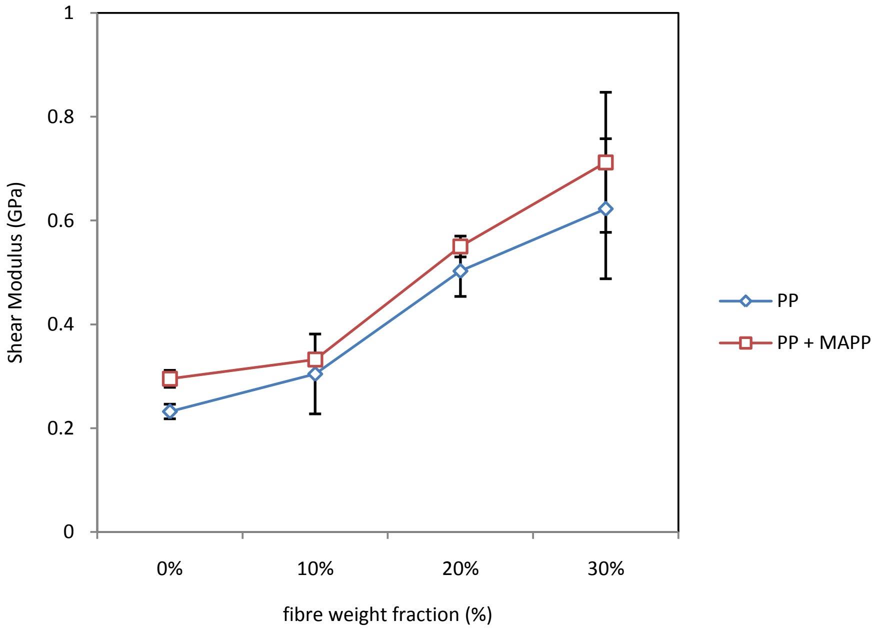
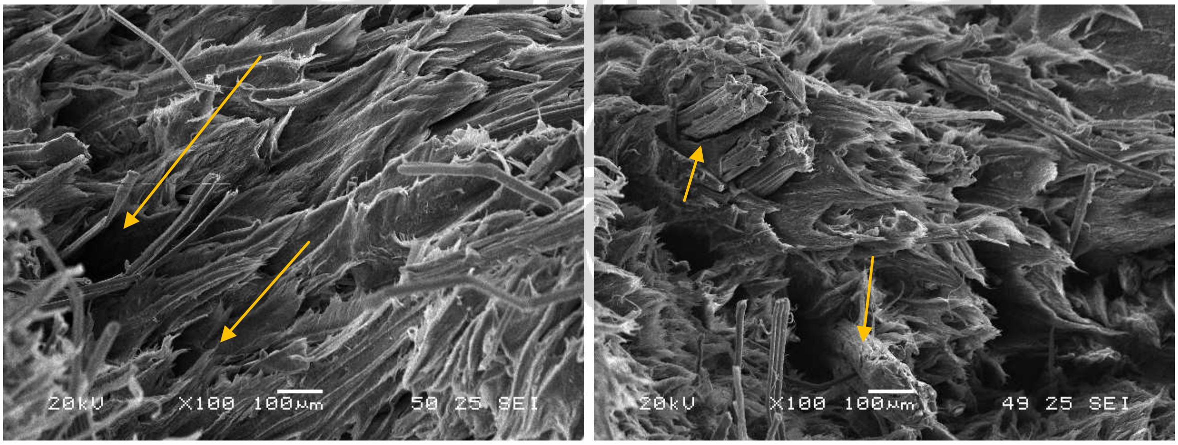


Figure 4. 4 Shear modulus of flax/PP



(a) (b)

Figure 4. 5 SEM micrographics of fracture surface for flax/PP in Torsion test;
a and b are without and with coupling agent, respectively.

Twisted fractured surface is represented in Fig.4.5. The effect of the coupling agent is shown here. Without coupling agent (Fig. 4.5a) the matrix are clearly lose their fibres, whereas with coupling agent (Fig. 4.5b), the fibres are fully coated and embedded within the PP.

4.4 Compressive

The properties in compression are presented in Table 4.4. Stiffness gradually increases with the fibre volume ratio about 3% until 124% for uncompatibilised system and 30% until 193% for compatibilitised system. Meanwhile, the maximum stress increases slightly from 26% to 31% for uncompatibilised system and from 35% to 59% for compatibilised system, whereas the strain at stress maximum decreases. The evolution of compressive modulus is represented in Fig.4.6.

Table 4. 4 Compressive properties of flax/PP

Specimens	Compression test		
	Compressive modulus	Maximum stress	Strain at maximum stress
unit	MPa	MPa	mm/mm
PP pure	1095.735 ± 121.431	25.4863 ± 0.601	0.06588 ± 0.0186
PP 10% flax	1049.987 ± 269.409	32.0131 ± 1.986	0.07143 ± 0.0137
PP 20% flax	1276.28 ± 379.441	32.1403 ± 2.0489	0.05833 ± 0.0061
PP 30% flax	2453.774 ± 687.035	32.3153 ± 2.803	0.04206 ± 0.0119
PP+MAPP pure	1000.775 ± 66.4372	26.1684 ± 0.557	0.09132 ± 0.0043
PP+MAPP 10% flax	1304.447 ± 582.751	31.84555 ± 3.117	0.06550 ± 0.0462
PP+MAPP 20% flax	1754.65 ±507.840	39.422 ± 2.0489	0.05411 ± 0.0042
PP+MAPP 30% flax	2928.874 ± 425.075	41.63925 ± 1.815	0.0370 ± 0.02571

The modulus obtained in this experiment is smaller than what we got in tensile test. Fig. 4.7 shows the correlation mechanical properties in tensile and compression for flax/PP with fibre weight fraction 30% with coupling agent. It can be seen that the stiffness of compression still low compared to tensile.

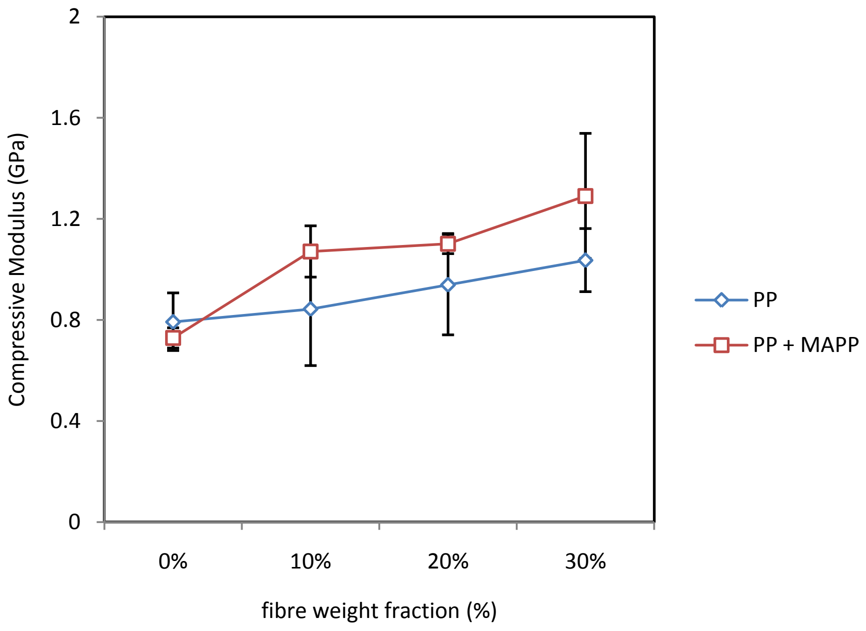


Figure 4. 6 The evolution of compressive modulus of flax/PP

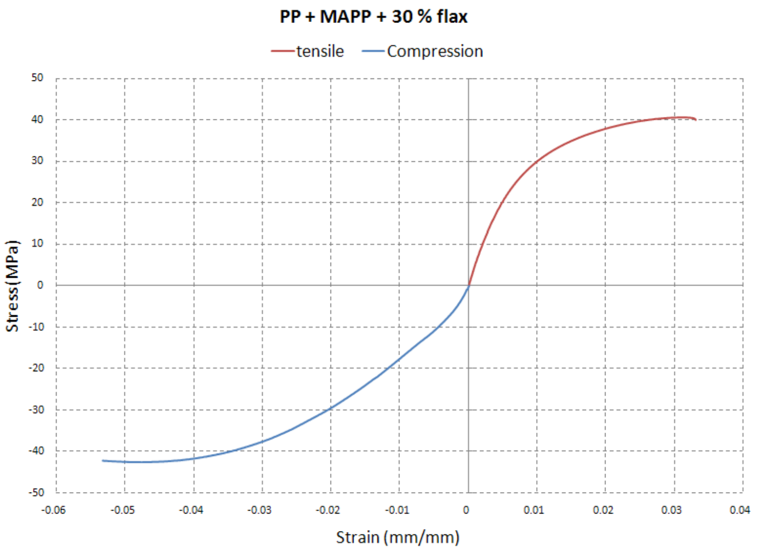


Figure 4. 7 Stress – strain curve for tensile – compression of flax/PP with fibre weight fraction 30 %

For all this, the experimental evidence shows that the compressive failure of the composite occurred by kink zone, Fig. 4.8 (according to four failure modes in compression test). This behaviour could be happen when the axial compressive cause fibres to buckle. .

Figure 4. 8 Failure mode of compressive specimen test flax/PP

4.5 Impact

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 Fig. 4.9. Impact strength is increase at higher fibre content, for the compatibilised material and reduced down for the uncompatibilised systems.

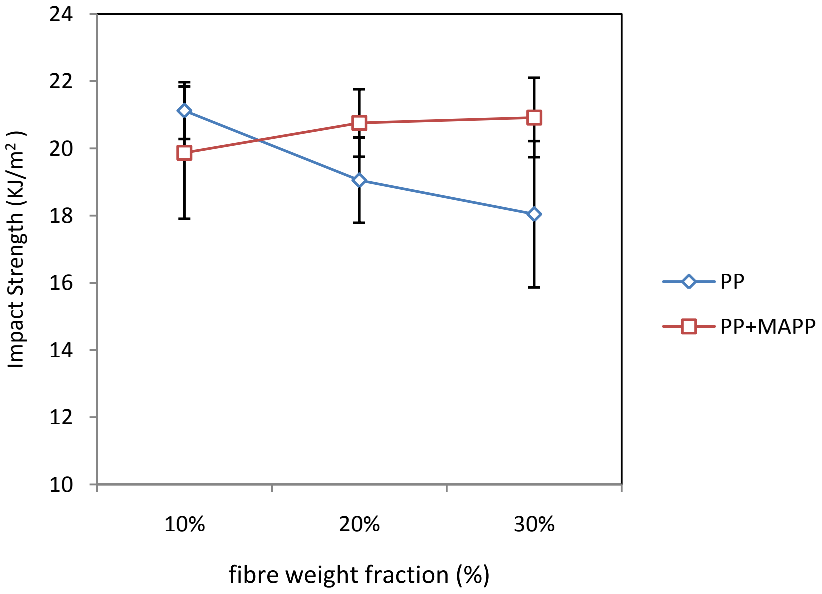


Figure 4. 9 Impact strength of unnotched charpy flax/PP

The impact failure for bio composite which consists of natural fibre and synthetic polymer with and without compabiliser was investigated. Fibres pull-out is common failure which occurs in uncompabilitised system. This means that fibre pull-out can only have a limited contribution to the energy absorption during impact. Addition of the compatibiliser will help to increase the debonding and frictional force between fibres and matrix and thus the energy dissipated during the fracture process.

Table 4. 5 Impact properties of flax/PP

Matrix	Impact Strength (KJ/m^2) Weight fibre fraction 0%	10%	20%	30%
PP	117.250 ± 22.255	21.128 ± 0.847	19.055 ± 1.269	18.043 ± 2.177
PP+MAPP	139.428 ±17.171	19.876 ± 1.971	20.758 ± 1.006	20.922 ± 1.181

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). In fact that in uncompatibilised systems has not a good adhesion between natural fibres and matrix, so a crack will propagate more quickly than in compabilitised systems. It causes lowering the values of impact strength.

CHAPTER V. CONCLUSION

The purpose of this work is to study the influence of fibre loading and coupling agent on mechanical properties of flax fibre reinforced Polypropylene. Most of mechanical properties of flax/PP increase with increase fibre loading, except impact strength for flax/PP. However, the low impact strength which shown in flax/PP due to adding of fibres content can be repair by adding coupling agent.

Furthermore, except for impact strength, adding coupling agent 4% in the compound flax fibres and polypropylene matrix could increase the others properties. In the other hand, ductility of the material generally reduces due to this addition.

Scanning electron microscopy (SEM) studies of the composites failure surfaces also indicated that there is an improved adhesion between fibre and matrix for flax/PP with coupling agent. Examination of the failure surfaces also indicated differences in the interfacial failure mode. With increasing fibre–matrix adhesion the failure mode changed from interfacial failure and considerable fibre pull-out from the matrix to matrix yielding and fibre and matrix tearing.

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