CHAPTER 1

INTRODUCTION

1.1 Project Background

Biocomposite are composite materials made from natural fibre as the reinforcement and petroleum-derived non-biodegradable polymers like Polypropylene (PP), Polyethylene (PE) and epoxies or biopolymers like PLA and PHAs. Nowadays, carbon fibre and glass fibre are widely being used in the vehicles manufacturing industries and construction industries. Those composites have shown undeniable quality however it also creates lots of unrecyclable trash around the world which pose threat to the environment. So, biocomposite are being given more attention lately and more studies have been done on it. Plant fibre is being utilized widely for the biocomposite production. The advantages of plant fibre are low density, low cost, acceptable specific strength, good thermal insulation properties, reduced tool wear, and most importantly, it is renewable resources and recyclable. Recycling could extend the material's useful life and thus minimizing the raw material consumption. This helps in conserving the carbon storage on earth as well. In addition, these lignocellulosic fibres are neutral with respect of the emission of CO_2 which is put the lignocellulosic material in context with the Kyoto Protocol [1].

Coconut plants are a widely spread plant all over the tropical and subtropical regions where Malaysia resides in. Locally, coconuts are widely used in the food industries where only the interior of the fruit is being extracted while the other part of the fruit including the coconut husk is disregarded. Large consumption of the coconut in the food industries produced huge amount of coconut waste where the coir of the coconut are the biggest parts of the waste. With the enormous source of this fruit, coconut fibre is a high potential replacing fibre for those non-recyclable fibre in the composite production. The coconut fibre also known as Coir is the coarse fibre extracted from fibrous outer shell of coconut. There are two types of coir which are the brown coir and the white coir. Brown coir which is harvested from fully ripened coconut will be utilized in this project. Brown Coir is thick, strong and high abrasion resistance. Besides, it is also among the

very few natural fibre that is resistant to both the salt and fresh water. Matured brown coir fibre contains more lignin and less cellulose than fibres such as flax and cotton which make it stronger however less flexibility. In this project, Coconut natural fibre (brown coir) is used as the reinforcement.

Polyethylene (PE) is a type of thermoplastic polymer which composed of ethylene monomer. PE is produced through polymerization of ethylene, for example radial polymerization, cationic addition polymerization. Polyethylene is classified into different categories based on its density and branching. The most commonly used PE is the High Density Polyethylene (HDPE) and Low Density Polyethylene (LDPE). These both PE are the semi crystalline type of polymer. The mechanical properties of PE is depends on the variables such as extent & type of branching, the crystal structure, and molecular weight. Polyethylene specifically HDPE is used as the matrix of the biocomposite in this research

1.2 Problem Statement

As known, Coconut-HDPE biocomposite is a recyclable material and it is an effective way of reducing the composite waste and minimizing the raw materials consumption. However, recycling will cause mechanical properties degradation of both the matrix and the reinforcement which reduce the quality of the composite. In the process of recycling, crashing and reforming of the composite will shorten the fibre length which affect the mechanical properties of the recycled composite. Besides, during the recycling process, contaminants from surrounding also might affect the recycled composite quality. The main concern is that to which extent that the recycling effects affect the mechanical properties of the biocomposite. Is the material is still usable after recycle?

1.3 **Objectives**

The purpose of this research is to analyse the recyclability of Coconut-HDPE biocomposite. The main objectives of the research are:

- 1. To determine the mechanical properties behaviour (Tensile and Flexural) variability of different fibre weight percentage Coconut-HDPE biocomposite using Universal Testing Machine for different recycling cycle.
- 2. To determine the crystallization and melting behaviour variability of different fibre weight percentage Coconut-HDPE biocomposite using Differential Scanning Calorimetric (DSC) machine for different recycling cycle.
- 3. To determine the carbon content change of the Coconut-HDPE biocomposite after recycled with CHNS test.

1.4 Scope of studies

Reinforcement	: Coconut coir fibre
Matrix	: High Density Polyethylene (HDPE)
Fabrication method	: Injection Molding
Recycling method	: Particle recycling /reprocessing
Conducted test	: 1. Tensile test
	2. Flexural test
	3. Thermal property test
	4. Chemical test
Observation	: 1. Optical Microscope analysis
	2. Scanning Electron Microscope analysis
Studied properties	: 1. Tensile strength and strain, Young Modulus
	2. Flexural strength, Flexural Modulus
	3. Crystallization and Melting behavior
	4. Carbon content

There are huge varieties of natural fibre either cellulose-rich fibre or lignin-rich fibre are utilised widely in the industries especially in the composite industries. In this research, lignin-rich fibre which is the coconut coir fibre is chosen as the reinforcement of the biocomposite (Appendix F4). The coconut short fibre is utilised for the fabrication

purpose. A specific range of fibre diameters are specified and utilised throughout the research. This is to maintain the consistency of the research. On the other hand, High Density Polyethylene (HDPE) is chosen as the matrix of the composite.

Five different fibre weight percentages (10.00%, 13.75%, 17.50%, 21.25%, 25.00%) are utilised for the research analysis.

For the composite fabrication purpose, there are two main methods which are the hot compression molding and the injection molding. As the type of fibre utilised here is short fibre type, so injection molding method is applicable in this research. This method can produce higher quality of composite in a shorter time and the process is simpler. Major problem like having voids within the composite can be avoided with this method.

For recycling process, there are several types of recycling methods used in the industry which are mechanical type, chemical recycling and the energy recycling. The recycling method that is utilised in this project is the mechanical type which is the grinding/ reprocessing techniques.

After every cycle of recycling, the mechanical properties of the biocomposite are analysed and compared. The analysed mechanical properties are:

- 1. Tensile properties
- 2. Flexural properties

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Besides, the crystallization and melting behaviour also will be analysed for every cycle of recycling. The melting temperature, crystallisation temperature and degree of crystallinity are analysed.

In addition, the effect of recycling on chemical elements of the biocomposite will be analysed by using the CHNS Elements Analyser. The specific components to be analysed are Carbon, Hydrogen, Nitrogen, and Sulpher level.

CHAPTER 2

LITERATURE REVIEW

2.1 Materials

Coconut fibre is the lignin-rich fibre. It contains a thin continuous surface layer of an aliphatic compound which is called the waxy layer. This long-chain aliphatic molecules and compound have been used as adhesion promoters in wood fibre-reinforced non-polar thermoplastic composites. So, coconut fibre can be used as the reinforcement for the polyethylene without any surface treatment or modification [2]. Apart from that, coir fibre is relatively waterproof and is the only natural fibre resistant to damage by salt water [3]. By using this fibre, the main disadvantage of the natural fibre which is the high water absorption behaviour can be reduced. Those are the two main properties that drive this research choose coconut fibre as the reinforcement. The general properties of the coconut fibre are given in the table 1 [4]. For the processing of the coconut fibre, the fibre is washed with the water and dried in a hot air oven at 80°C for 4-6 hours [4]. For the powder form of fibre, the particle size can be controlled by controlling the crushing time duration [7]. [Weilin, 7] who do the research on the wool powder use this method; SEM figures are given in appendix F4.

Polyethylene (PE) is the most widely used plastic in the industries and raw materials for the daily used product. The general properties are given in the Table 1 below [4]. The most commonly used PE is the High Density Polyethylene (HDPE) and Low Density Polyethylene (LDPE). The characteristic of the HDPE and LDPE are listed in the table 2 [5]. The melting temperature for HDPE is around $120^{\circ}C - 130^{\circ}C$ and for LDPE is around $105^{\circ}C - 115^{\circ}C$. More detail analysis on the LDPE also been done where the tensile test and flexural test has been done as shown in table 3 and table 4 respectively (refer to the pure LDPE). [6]

Table 1: Tensile properties of coconut fibre and the polyethylene matrix. [4]

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Material	Strength (MPa)	Modulus (GPa)	Failure strain (%)
Coconut fibre	140-225	3–5	25–40
Polyethylene	9.2	0.14	200

Table 2: Characteristic of the polyethylene [5]

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Olefin (grade name)	Density (g/cm3)	MI	HDT (°C)	Tensile strength	Code
		(g/10min)		(kg/cm2)	
High density polyethylene	0.954	0.8	123	350	HDPE
(3300)					
Low density polyethylene	0.919	3.0	90	120	LDPE
(FB300)					
Linear low density	0.918	2.1	98	350	LLDPE
polyethylene (FT810)					

MI: melt index; HDT: heat distortion temperature.

Table 3: Tensile test for LDPE/starch compounds [6].

Sample	Tensile strength (MPa)	Elongation (%)	Young modulus (MPa)
Pure LDPE	12.9 ± 0.2	131.9 ± 4.8	139.3±6.8
LDPE + 5 wt.% native starch	12.7 ± 0.2	58.0 ± 1.5	113.1 ± 7.0
LDPE + 10 wt.% native starch	11.9 ± 0.1	50.1 ± 1.5	122.6 ± 5.1
LDPE + 20 wt.% native starch	11.3 ± 0.1	30.9 ± 1.6	151.7 ± 11.2
LDPE + 5 wt.% RD125	12.5 ± 0.2	55.6 ± 3.7	118.9 ± 6.1
LDPE + 10 wt.% RD125	12.1 ± 0.1	50.3 ± 1.5	131.9 ± 5.6
LDPE + 20 wt.% RD125	11.1 ± 0.1	35.0 ± 1.8	151.2 ± 9.7
LDPE + 5 wt.% adipate starch	12.9 ± 0.1	52.6 ± 1.4	118.5 ± 6.5
LDPE + 10 wt.% adipate starch	12.2 ± 0.1	43.5 ± 1.0	129.3 ± 9.7
LDPE + 20 wt.% adipate starch	11.3 ± 0.1	33.7 ± 1.1	150.8 ± 3.6
LDPE + 5 wt.% cassava starch	12.7 ± 0.1	55.6 ± 1.9	113.3 ± 5.3
LDPE + 10 wt.% cassava starch	12.0 ± 0.2	49.2 ± 1.4	119.0 ± 7.8
LDPE + 20 wt.% cassava starch	11.4 ± 0.2	36.9 ± 1.3	149.9 ± 6.2

2.2 Fabrication

Injection molding is the most common method of producing the thermoplastic composite. It can produce more homogeneous thermoplastic product in shorter time if compared to the hot compression process where it also reduce the chance of void happening in the composite. LDPE and HDPE both require different production parameter suggested by previous other people's research. For LDPE, according to [E.M.Nakamura, 6] who had research on the LDPE/starch, they produce the injected specimen using the 25 mm diameter Arburg injector, L/D=20. The pressure is set to 1500 bar while holding pressure and time were relatively 800 bars and 20 sec. Cooling time is fixed at 5s and the heating zone varied from 110° C to 160°C. The product is following the ASTM D-638 standard [6]. For HDPE, according to [J.G.J. Beijer, 8], the research is using the HDPE 7058Z (MFI=4.4 dg/min, ρ =953 kg/m³) supplied by DSM. The research

is following the ISO 527 standard which produces the 3.8 thick tensile bars. The production is done at relatively high nozzle temperature which is 250°C and long cycle time of 55 sec to minimize the effect of anisotropy and internal stress [8]. On the other hand, [I. Rex, 9] research which use the HDPE (HD-6605) powder for injection moulding through Arburg 55-tonne injection moulding machine. The processing parameters are processing temperature and injection flow rates were varied between 190, 225, and 270 °C; and 70, 90, and 110cm³/s, respectively. Over the temperature and flow rate range disclosed, the wall shear rate varied from 4000 to 7500 s⁻¹ in the sprue and runners of the mould [9].

2.3 <u>Recycling</u>

Recyclable is the main reason that natural fibre being utilized in the composite production. In European country, flax fibre and hemp fibre are the widely used natural fibre in the composite area. The recyclability of Flax/PLLA biocomposite has been done by [A.L.Duigou, 10]. This research is utilizing the injection molding machine for the fabrication of the new and recycled composite. The PLLA were extruded with the flax fibre before the injection molding at 20rpm and with the following temperature profile: 175/180/185 and 185°C in the nozzle. Then, it was been injected. The recycling will keep all the parameters constant with the temperature profile kept as: 165/170/175/180 and 180°C at the nozzle with the injection pressure 190 bars and injection time 0.95s. The research is carried out with six injection cycles and testing was done after each cycle. The testing involved are tensile test to determine mechanical properties, Differential Scanning calorimeter(DSC) to determine the crystallization and melting behaviours, and SEM to analyse the microstructure. The research showed that the biocompoisite exhibit interesting recycling properties and it is possible to be recycled to produce good quality product. By implement the method of include both virgin and recycled matter into the production, the quality will be increased [10].

There are three main methods of recycling which are the chemical, particles, or energy recycling [11]. Example of chemical recycling is the pyrolysis process where the material is heated in an oxygen-free environment to produce one or more recoverable substances [11]. Particle recycling is based on the idea that the thermoset composite can be mechanically milled into particles which can subsequently be used as filler in new plastic or composite application as the replacement for calcium carbonate or talc. This method is adopted for the thermoplastic type material. Finally, energy recycling is the method of incinerating the material to recover energy from its organic portion. Particle recycling which involve grinding/reprocessing is the most viable technique as it is cost-effective and from performance spec, it homogenizes the fibre length and distribution [11]. However, there are some drawbacks for this technique, the grinding and reprocessing steps will degrade the performance of thermoplastic composite due to the fibre length attrition, polymer degradation and fibre/matrix interface degradation [12, 13]. The recycling steps taken by [David, 11] with the recycling of *poly (butylenes terephthalate)* (PBT) are firstly the moulded composite plate size is reduced so that it can be fed into the granulator. The plates were cut into strips with blade, which subsequently fed into a variable-speed Nelmor granulator (Model G810M1) with the setting of 260 rpm, screen opening of 9.5 mm and 0.15 mm clearance.

2.4 Testing

For the composite mechanical testing, ASTM and ISO are the standards that usually been applied. For tensile testing, ASTM D-638 or equivalently ISO527-1 is the standard being applied. The specimen's shape and dimension is as shown as fig1 [14]. [A. Bernasconi, 14] utilize ISO 527-1 as the reference for their test of the mechanical properties of glass fibre. It uses the crosshead speed of 5mm/min on an MTS Alliance RF150 machine. Strains were measured by using the MTS 634.25 extensometer of 50 mm base length. [E.M. Nakamura, 6] researches on the LDPE/starch composite utilize the ASTM D-638 standard. The test is done by using universal test equipment EMIC, model DL2000, 5 kN load cell. The tensile test velocity was 50 mm/min, for specimen 3.3 mm thickness [6]. The result of the test is shown in Table 3.



Fig.1: Shape and dimensions of the type 1A specimen, according to ISO 527-2 standard [14].

Flexural test is another important mechanical test that has to be done to examine the behaviour of the composite material. For flexural test, ASTM D-790 standard is being utilized. The test velocity is determined by calculation according to the width and thickness of the specimen and also the strain property of the material. Example research of the flexural test result of the LDPE/starch is shown in Table 4 [6]. [S. Harish, 3] had done the flexural test on the coir composite by adopting ASTM D-790 standard as well. Three point-tensile tests were carried out on the specimen at room temperature. The specimen is placed onto two supports having a 50-mm span length between the supports. The speed of the jaws was set to 2 mm/min [3].

Sample	Tensile strength (MPa)	Young modulus (MPa)
Pure LDPE	6.3 ± 0.2	164.3 ± 14.6
LDPE + 5 wt.% native starch	8.2 ± 0.4	131.5 ± 3.8
LDPE + 10 wt.% native starch	8.9 ± 0.2	147.2 ± 6.6
LDPE + 20 wt.% native starch	10.1 ± 0.1	172.8 ± 15.3
LDPE + 5 wt.% RD125	8.3 ± 0.3	138.5 ± 3.5
LDPE + 10 wt.% RD125	8.5 ± 0.3	145.9 ± 6.0
LDPE + 20 wt.% RD125	9.1 ± 0.2	174.3 ± 5.3
LDPE + 5 wt.% adipate starch	8.8 ± 0.1	148.2 ± 5.8
LDPE + 10 wt.% adipate starch	9.4 ± 0.3	163.5 ± 5.3
LDPE + 20 wt.% adipate starch	9.9 ± 0.2	182.3 ± 7.7
LDPE + 5 wt.% cassava starch	9.7 ± 0.2	168.7 ± 6.2
LDPE + 10 wt.% cassava starch	9.9 ± 0.1	173.8 ± 3.4
LDPE + 20 wt.% cassava starch	10.1 ± 0.2	192.4 ± 6.0

Table 4: Flexural test for LDPE/starch compounds [6].

Crystallization and melting behaviour is an important for the thermoplastic type composite. Differential scanning Calorimeter (DSC) is utilised for this purpose. The melting and crystallisation temperature can be obtained from this testing and the degree of crystallinity also been analysed. [A.L.Duigou^{a,} 10] has done the flexural test on the flax/poly (L-Lactide) composite.

2.5 Observation

For the observation, both optical and Scanning Electron Microscope (SEM) is required to analyse the composite microstructure condition. Defects mechanics can be analysed through these observations. 2000 magnification power with the machine Hitachi S-4300 has been utilized to obtain the image of fracture surface of specimen [5]. Three samples from three different location were analysed (top neck, middle, and bottom neck of the specimen).Example of observation as shown in Fig 2.



Fig 2: SEM photograph of the calcite filled HDPE for various draw ratios including neck part at 5 mm/min with an initial grip distance of 10 mm.

2.6 Chemical Element Analysis

In this analysis, the CHNS Element Analyser will be utilised. The purpose of this analysis is to determine the element content of carbon, hydrogen, nitrogen and sulphur in the material.

The method is used extensively across a wide range of applications, including pharmaceuticals, chemicals, oil-related products, catalysts and food. The analysers are often constructed in modular form such that they can be set up in a number of different configurations to determine, for example, CHN, CHNS, CNS or N depending on the application.

The simplest form of analysis involves the analysis of all four CHNS simultaneously. This requires high temperature combustion in an oxygen-rich environment and is based on the classical Pregl-Dumas method. This combustion can be carried out under both static conditions i.e. introduction of a set volume of oxygen or dynamic conditions i.e. a constant flow of oxygen for a set period of time. Often, catalysts are also added to the combustion tube in order to aid conversion.

CHAPTER 3

METHODOLOGY

3.1 General Project Activities



Fig 3: Methodology Chart of the whole research project

3.2 Specific Project Activities

3.2.1 <u>Research and Review Literatures</u>

In order to build up the background information on the field the project, research and study had been carried out prior to the commencement of this project. Different findings and methodologies were gathered from the research works of other researchers and to be assimilated into this project. In order to have proper information gathering, firstly the categories of information that this research project required were drawn out: (1) ASTM/ISO Standards, (2) Materials background, (3) Recycling, (4) Mechanical and Thermal testing. Then, the journal papers were searched based on these four categories and extracted the related information. With the information gathered from the research project. Most importantly, this research was conducted closely based on ASTM/ISO Standards.

3.2.2 Fibre preparation

First of all, the dried coconut coir fibres were processed. The raw coconut coir fibres got from the university store were long and entangled. Besides, it also has different variety of diameter. In order to have more even length of fibres, by using Low Speed Granulator SG 16-21 machine (figure in appendix F6), the fibres were grinded to shorter fibre with the maximum length of around 2.0 cm.

Then by using the sieve machine (figure in appendix F5), the grinded fibres were categorised into different diameter range. The distribution of diameter of the fibres were analysed by the sieve analysis. A specified range of diameter value fibre was selected to fabricate the composite for the research. This is to have more consistent result. During this sieving process, the fibre that was too long will be separated as well. In addition, the sieving also separated out the contaminants such as small leaves.

3.2.3 Biocomposite Fabrication

The biocomposite specimens were fabricated with injection moulding method. The machine model used was ME 20 (III) Injection Moulding machine. The research involved the use of coconut fibre as the reinforcement and High Density Polyethylene (HDPE) as the matrix. The injection moulding process produced the specimens which were adapted to the ASTM D-638 dimension and shape. The temperature being utilised for the fabrication is 120 °C as reference to the other's research paper [6]. Additional specimens were fabricated to prepare backup specimens for the broken failure specimens during the tensile test and the flexural test.

3.2.4 Mechanical Testing

In the testing stage, two main mechanical testing done in this research were the tensile test and the flexural test.

For tensile test, it was conducted under the parameter condition set by the standard ASTM D-638 under the room temperature and humidity with Universal Testing Machine LLOYD. The specimen used was in dumbbell shape with the dimension of 200 mm X 10 mm X 4 mm. The loading speed set was 2 mm/min. A Laserscan Non-contacting Extensometer was used with a nominal length of 50 mm to determine Young's modulus. A constant 10N preload was applied for 5 seconds at the initial of the testing, the purpose was to tighten the gripper and prevent slipping from happening during the testing. For each weight fibre percentage group, 3 sample tests were conducted.

For the flexural test, it was conducted under the parameter condition set by ASTM D-790 standard where three point bending tests are conducted with Universal Testing Machine LLOYD. The tests were conducted under room temperature and humidity. The specimen was placed onto two supports having a 64 mm span length between the supports (calculated from given equation). The speed of the jaws was set to 17 mm/min as calculated from the formula given by the standard ASTM D-790. (The rate of straining used is 0.1 mm/mm instead of 0.01 mm/mm as recommended by the standard as the specimen does not break within the 5% strain limit with the rate of straining 0.01 mm/mm.). For each weight fibre percentage group, 3 samples tests were conducted. The speed was calculated with the formula below:

$$R = \frac{ZL^2}{6d}$$
[1]

with

R = rate of crosshead motion, mm/min,

L = support span, mm

d = depth of beam, mm

Z = rate of straining

3.2.5 Thermal testing

For each sample, 10 mg specimen will be utilised for the analysis. The specimen was heated from 30°C to 200°C with the heating rate of 10°C/min. Then, the specimen was maintained at 200°C for 2 min to remove the thermal history. After that, the specimen then being cooled down to 30°C with the collong rate of 10°C/min as well. The non-isothermal crystallisation and melting temperatures, Tc and Tm were determined from the crystallisation peak extreme in experiments. The degree of crystalline (Xc) was calculated using following equation below, Δ H100% crystalline is the enthalpy value where the material have 100% crystalline:

$$\chi_{\rm c} = \frac{\Delta H_{\rm m}}{\Delta H_{100\%}}$$

$$\Delta H_m = \text{Melting Enthalpy} \qquad [2]$$

3.2.6 Recycling

In the recycling stage, the tested biocomposite materials were recycled by using the mechanical grinding/ recycling method. The granulator machine used was the Low Speed Granulator SG 16-21 machine with a total of 2 fixed blades and 9 rotating blades. Then the grinded products were then reprocessed by using

the injection moulding process to produce recycled composite. After each recycling, the mechanical test, thermal test and microscopic analysis were done on the specimens.

3.2.7 Microstructure Analysis

Two types of observation were done which are by using Optical Microscope and the SEM. For Optical Microscope, the magnification range utilised were 5X, 10X, 50X and 100X. Pictures were captured for each of the different magnification power.

For the SEM analysis, the tensile fracture surfaces were observed. Samples were sputter-coated with a thin layer of gold in an Edwards Sputter Coater before being scanned with SEM machine. Three main locations were focused for the observations: the fibre surface, matrix surface and also the interfacial between the fibre and matrix. After each cycle of recycling, optical microscope and SEM observation will be done on the specimen.

3.2.8 Chemical Elements Testing

Additional test on the chemical properties were done where the biocomposite went through the CHNS Element analysis to determine the change of level of the basic elements Carbon, Hydrogen, Nitrogen, and Sulphur in the material after several recycling process. Basically, the working theory behind this method is simple. In the combustion process (furnace at ca. 1000°C), carbon was converted to carbon dioxide; hydrogen to water; nitrogen to nitrogen gas/ oxides of nitrogen and sulphur to sulphur dioxide. If other elements such as chlorine were present, they were converted to combustion products, such as hydrogen chloride. A variety of absorbents were used to remove these additional combustion products as well as some of the principal elements, sulphur for example, if no determination of these additional elements is required. Then, the amount level of each element can be obtained.

CHAPTER 4 RESULT AND DISCUSSION

4.1 Fibre preparation with granulator

Fig 4 shows the coconut fibre after being grinded with the granulator Low Speed Granulator SG 16-21 machine. The entangled fibres were successfully been shortened into averagely even length.



Fig 4: As-received coconut fibre (left) and Fibre after grinded with granulator (right).

4.2 Sieve analysis

After grinded, although the fibre lengths were mostly even, but there were some contaminants noticed in the fibres such as small leaves. In addition, there were also obvious variations in the diameter of the fibre.

So, sieve analysis was conducted to analyse and determine the diameter range of fibre to be used for the composite sample production. This helped in increasing the consistency of the result. At the same time, this process has filter out the contaminants. The range of apertures used were from the biggest size of 2.000 mm to the smallest 0.063 mm (2.000mm, 1.180mm, 0.600mm, 0.425mm, 0.300mm, 0.212mm, 0.150mm, 0.063mm). Total mass of the before separate fibre is 97.30g.

Table 5 shows the weight of fibre of different diameter range. From the Table 5, log graph is being plotted as shown in Fig 5. From the graph, it can be observed that the stiffest curve is between 0.212 mm to 1.180mm. The stiffer of the graph means more

proportion amount of the fibre has the diameter within that range. Meanwhile, the contaminants and those fibres that has larger diameter were trapped in the aperture of 2.000mm and 1.180mm size. So, with the advice of supervisor, it was decided that fibres with the diameter range of within 0.063mm to 1.180mm were utilised for the research project.

Sieve	Mass of fiber retained	Percentage of each sieve Bn	Cumulative	Percentage Finer
(mm)	(g)	(%)	(%)	(%)
2,000	3,46	3,55	3,58	96,42
1,180	11,10	11,40	14,98	85,02
0,600	28,46	29,25	44,23	55,77
0,425	23,89	24,55	68,78	31,22
0,300	15,60	16,03	84,81	15,19
0,212	7,32	7,52	92,33	7,67
0,150	3,16	3,24	95,57	4,43
0,063	3,47	3,56	99,13	0,87
Pan	0,88	0,90	100,03	
Total	97,30	100,00		

Table 5: Weight and percentage of fibre of different diameter range.



Fig 5: Fibre diameter distribution chart.

4.3 Tensile Test

For each of the testing, two important graphs are plotted which are the Load (N) vs Strain (%) graph and Stress (MPa) vs Strain (%) graph. Fig 6 and Fig 7 show the example graphs for composite with 21.25 wt% of coconut fibre. From the graph, it is noticeable that the biocomposites have ductile behaviour where yielding happens before the fracture.



Fig 6: Graph of Load (N) vs. longitudinal strain (%)



Fig 7: Graph of Stress (MPa) vs. longitudinal strain (%)

4.3.1 Tensile properties of injected (without recycled) Coconut-HDPE biocomposite

Table 6 presents the average tensile test result for different fibre weight percentage composite without recycled. For each of the weight percentage, 3 samples tests were conducted.

Fibre Fraction:	0.00%	10.00%	13.75%	17.50%	21.25%	25.00%
Number of cycle	0	0	0	0	0	0
Specimen No :	AVG	AVG	AVG	AVG	AVG	AVG
Laboratory :	Block 17					
Machine :	LLOYD	LLOYD	LLOYD	LLOYD	LLOYD	LLOYD
Speed (mm/min) :	2	2	2	2	2	2
Max Load (N) :	814.29	786.30	798.61	762.23	759.78	749.30
Disp. At Max Load(mm) :	13.20	11.47	11.70	9.79	9.91	9.74
Strain At Max Load(%) :	18.85	16.33	16.71	13.99	14.16	13.92
Max Disp (mm):	46.48	23.73	27.38	14.64	12.98	13.07
Max Long. strain (%):	66.41	33.85	39.11	20.91	18.55	18.67
Max Stress, σ (MPa) :	20.36	19.66	19.96	19.06	19.00	18.73
Work to Maximum (J)/ toughness:	8.02	5.50	6.41	6.14	5.13	4.77
Stiffness (N/m):	146335.22	161683.27	167480.02	245943.11	191201.90	185433.93
Young Modulus, E (MPa)	256.08	282.95	293.09	334.59	334.60	324.51

Table 6: Overall Tensile test result without recycled for different fibre weight percentage.

From the Table 6, graphs are plotted to analyse the influence of the fibre weight percentage on the tensile properties. Result shows that with the increment of the coconut fibre weight percentage, the composite still able to maintain the tensile strength at around 20 MPa which is the tensile strength of the pure HDPE (refer Fig 8). On the other hand, with respect to Fig 9, results show a positive increase of Young's Modulus with the increment of coconut fibre weight percentage. So, this means that higher coconut weight percentage composite can sustain the almost same amount of tensile stress with lower strain which is desirable condition.



Fig 8: Graph of Tensile Strength (MPa) vs. Coconut fibre weight percentage (%) (without recycled biocomposite.)



Fig 9: Graph of Young Modulus (MPa) vs. Coconut fibre weight percentage (%) (without recycled biocomposite.)

A comparison of between the mechanical properties of the Coconut-HDPE biocomposite of present study (17.50wt%) with properties of other previous others work on natural fibre composite with most comparable fibre weight percentage is presented in Table 7. The natural fibre chosen here are the commonly and easily found in Malaysia. From the comparison with the Coconut fibre /Epoxy CY205, the present work biocomposite has slightly higher tensile strength. Oil palm empty fruit bunch/ Polyurethane (PU) biocomposite and Rice husk/ Polyethylene glycol biocomposite both have much lower tensile strength than the Coconut fibre/HDPE biocomposite.

Table 7: Tensile properties of Coconut-PE biocomposite compared to other natural fibre composite.

Material	Process	Fibre Content (Wt%)	Tensile strength (MPa)	Tensile Modulus (MPa)	Reference
Coconut fibre/HDPE (present work)	Injection molding	17.50	19.06	334.59	-
Coconut fibre /Epoxy CY205	Compression	-	17.86	-	[3]
Oil palm empty fruit bunch/ Polyurethane (PU)	Compression	30.00	9.00	1100.00	[16]
Rice husk/ Polyethylene glycol	Compression	30.00	5.00	0.10	[17]

4.3.2 <u>Tensile properties of First cycle recycled Coconut-HDPE biocomposite</u>

Table 8 present the average tensile test result for different fibre weight percentage composite underwent recycling process. It presents that the tensile strength is maintaining at around 18 MPa to 19 MPa for different fibre weight percentage as shown in Fig 10. This shows that increment of fibre weight percentage up until 25 % do not give significant impact on the biocomposite even after being recycled. The Young Modulus of the recycled biocomposites are relatively high as shown in Fig 11 where biocomposite of 21.25 fiber weight percentage shows the highest young modulus value of 579.14 MPa

Table 8: Tensile test result of 1st cycle recycled biocomposite for different fibre weight percentage.

Fibre Fraction:	0.00%	10.00%	13.75%	17.50%	21.25%	25.00%
Number of cycle	1	1	1	1	1	1
Specimen No :	AVG	AVG	AVG	AVG	AVG	AVG
Laboratory :	Block 17					
Machine :	LLOYD	LLOYD	LLOYD	LLOYD	LLOYD	LLOYD
Speed (mm/min) :	2	2	2	2	2	2
Max Load (N):	756.23	763.28	760.18	753.46	736.86	743.70
Disp. At Max Load(mm) :	13.11	11.24	6.99	9.66	6.77	9.45
Strain At Max Load(%) :	18.73	16.05	9.98	13.80	9.67	13.50
Max Disp (mm):	31.49	18.25	12.07	13.75	9.97	15.93
Max Long. strain (%):	44.98	26.08	17.24	19.64	14.24	22.76
Max Stress, σ (MPa) :	18.91	19.08	18.99	18.84	18.42	18.59
Work to Maximum (J)/ toughness:	7.14	6.06	4.58	5.35	3.53	5.24
Stiffness (N/m):	178391.10	200604.33	269536.19	273632.96	237682.75	278758.26
Young Modulus, E (MPa)	312.18	351.06	471.68	478.85	579.14	487.83



Fig 10: Graph of Tensile Strength (MPa) vs. Coconut fibre weight percentage (%) (1st recycled biocomposite.)



Fig 11: Graph of Young Modulus (MPa) vs. Coconut fibre weight percentage (%) (1st recycled biocomposite.)

Fig 12 shows that after one cycle recycling process, the coconut-HDPE biocomposite is still able to maintain its tensile strength. From Fig 13, it can be noticed that there are large increment of Young's modulus of the biocomposite after being recycled. So, this means that the recycled biocomposite is able to sustain the same amount of load with smaller deflection compared to the without recycled biocomposite.

This is also showing that the recycling process has made the biocomposite to become more brittle.



Fig.12: Comparison of tensile strength between the without recycled biocomposite and also the 1st cycle recycled biocomposite.



Fig.13: Comparison of Young's modulus between the without recycled biocomposite and also the 1st cycle recycled biocomposite.

4.4 Flexural test

4.4.1 <u>Flexural properties of injected (without recycled) Coconut-HDPE</u> <u>biocomposites</u>

Universal Testing Machine LLOYD is used for the flexural testing where 3 points bending tests are being conducted. For each of the testing, the Load (N) vs Deflection from preload (mm) graph are plotted. Fig 14 show the example graph for composite with 21.25 wt% of coconut fibre. All the specimens show no breakage phenomenon even after testing have been done till 20% (13.5mm) deflection strain percentage.



Fig 14: Graph of Bending Load (N) vs. Deflection from preload (mm)

Table 9 shows the change of average Flexural properties with the increment of coconut fibre weight percentage. The flexural stress is increasing with the increment of coconut weight percentage as shown in Fig 15. The maximum flexural stress 39.05 MPa is shown by the 21.25 wt% coconut fibre biocomposite which is much higher than the flexural stress of the pure HDPE. At 25.00wt%, we notice a slight drop of flexural strength. It

might be has reach the flexural properties limit of the coconut fibre. However, test with higher weight percentage composite needed to be done in order to proof this hypothesis.

For flexural modulus, by referring to Fig 16, it shows significant increment with the increment of coconut fibre weight percentage. The highest flexural modulus 1777.43 MPa is shown by 21.25 wt% coconut fibre biocomposite which much higher than the flexural modulus of pure HDPE 1071.00 MPa. In other words, 21.25 wt% coconut fibre biocomposite is able to sustain higher stress with lower deflection compared to the pure HDPE composite. In flexural modulus, it also noticeable that at 25.00wt% coconut fibre, it shows a slight decrement, further testing need to be done to explain this trend. From the result, it shows that the coconut fibre has large contribution in improving the flexural properties of the biocomposite.

Fibre Weight Percentage:	0.00%	10.00%	13.75%	17.50%	21.25%	25.00%
Number of cycle	0	0	0	0	0	0
Specimen No :	AVG	AVG	AVG	AVG	AVG	AVG
Laboratory :	Block 17	Block 17	Block 17	Block 17	Block 17	Block 17
Machine :	LLOYD	LLOYD	LLOYD	LLOYD	LLOYD	LLOYD
Speed (mm/min) :	17.00	17.00	17.00	17.00	17.00	17.00
Max Load (N):	54.06	59.68	58.96	62.84	65.09	64.44
After preload Deflection at Max Load, (mm)	12.78	10.52	10.49	10.58	10.45	10.72
Max Flexural Stress, σ (MPa) :	32.44	35.81	35.48	37.70	39.05	38.66
Modulus of Elasticity in Bending (Mpa)	1071.00	1478.79	1487.12	1570.34	1777.43	1681.27

Table 9: Overall Flexural test result without recycled for different fibre weight percentage.



Fig 15: Graph of Flexural strength, MPa vs Fibre weight fraction, % (without recycled)



Fig 16: Graph of Flexural modulus, MPa vs Fibre weight fraction, % (without recycled)

4.4.2 Flexural properties of First recycled Coconut-HDPE biocomposite

Table 10 shows the collection of flexural properties of the recycled Coconut-HDPE biocomposite for the first cycle. Similar to the without recycled biocomposites, it shows flexural strength shows increment with the increment of the coconut fibre weight percentage up until 21.25 %, and there is decrement at 25.00% (Fig 17). Flexural modulus of the biocomposites as shown in Fig. 18 demonstrates continuous increment with the increment of the coconut fibre weight percentage. The steep increment of flexural strength and modulus from pure HDPE to 10wt% biocomposite shows that coconut fibre has great positive impact on the improvement of the flexural properties of the biocomposite.

Table 10: Flexural test result of 1st cycle recycled biocomposite for different fibre weight percentage.

Fibre Fraction:	0.00%	10.00%	13.75%	17.50%	21.25%	25.00%
Number of cycle	1	1	1	1	1	1
Specimen No :	AVG	AVG	AVG	AVG	AVG	AVG
Laboratory :	Block 17					
Machine :	LLOYD	LLOYD	LLOYD	LLOYD	LLOYD	LLOYD
Speed (mm/min) :	17.00	17.00	17.00	17.00	17.00	17.00
Max Load (N) :	48.38	61.14	62.59	62.87	71.36	65.20
After preload Deflection at Max Load, (mm)	12.75	12.74	11.38	10.15	9.89	8.78
Max Flexural Stress, σ (MPa) :	29.03	36.69	37.55	37.72	42.82	39.12
Modulus of Elasticity in Bending (Mpa)	1020.93	1477.02	1609.42	1709.77	1752.78	1853.64







Fig. 18: Graph of Flexural modulus, MPa vs Fibre weight fraction, %

(1st recycled biocomposite.)

Instead of reducing the mechanical properties, Fig. 19 and Fig. 20 shows that recycling process actually promote improvement of the flexural strength of the biocomposites. This might be caused by the more evenly distributed coconut fibre within the biocomposite after being recycled.



Fig.19: Comparison of flexural strength between the without recycled biocomposite and also the 1st cycle recycled biocomposite.



Fig.20: Comparison of Flexural modulus between the without recycled biocomposite and also the 1st cycle recycled biocomposite.

4.5 Thermal Test

Fig. 21 shows that the recycled composite has relative sharper and stronger melting peaks than the without recycled composite. This indicates the increase of fusion enthalpy and degree of crystallinity of the composite after being recycled. Thus, the recycled composite have the presence of more homogeneous crystalline structures in composites as higher degree of crystallinity structure material require higher heat energy addition to 1 mole of a substance to change its state from a solid to a liquid form. This structure change due to recycling has explained the increment of mechanical properties of the composites after being recycled. For the crystallisation peaks, both curves show almost same width and peak temperature, this show that both have almost same crystallisation rate. In addition, both without recycled and recycled composite have the same initial crystallization temperatures (T_{onset}).



Fig.21: DSC heating and cooling curves of the without recycled and after recycled Coconut-HDPE composites of 21.25 % weight percentage.

4.6 Tensile Fracture analysis

Generally, it is noticeable that with the increase of the fibre weight percentage, the breakage mechanism becomes more brittle. Refer to Fig 22, for the 10.00% and 13.75%, it is obvious there are sign of ductile fracture by having the long HDPE tail on the breakage point.



Fig 22: Composite sample of different fibre weight % after testing (From up to bottom: 10.00%, 13.75%, 17.50%, 21.50%, and 25.00%)

SEM examination also been done on the tensile fracture surface to analyse the qualitative information about the fibre, matrix and interfacial condition. From Fig 23, for biocomposite that haven't go through recycling, we can observe that bundles of fibres are noticeable and the distribution of fibres is not homogeneous. We can observe there are fibres are gathered at the bottom right and top left in the figure and at the middle only have HDPE without fibre bundle. On the other hand, Fig 24 shows that the distribution of fibre among the biocomposite is more homogeneous. Fibres can be seen distributed more even within the HDPE. From Fig 25, it shows that cracks propagations happen on the coconut fibre bundles. This shows that the coconut fibres contribute to the tensile properties for the resistance to break. Fig 26 shows the interface between the fibre and the

HDPE. We can see that there are still small voids between the fibre and HDPE showing that the wetting is still not efficient in the coconut fibre and interfacial bonding is not that good. This might due to the short duration of pressing during injection moulding that do not allow enough time for the HDPE to penetrate the fibre.



Fig 23: SEM micrograph of the fracture surface of 21.25wt% **without recycled** Coconut-HDPE biocomposite (magnification level- 30X)



Fig 24: SEM micrograph of the fracture surface of 21.25wt% **after recycled** Coconut-HDPE biocomposite (magnification level- 30X)



Fig 25: SEM micrograph of the fracture surface of 13.75wt% Coconut-HDPE biocomposite (magnification level- 100X)



Fig 26: SEM micrograph of the fracture surface of 21.25wt% Coconut-HDPE biocomposite (magnification level- 200X)

4.7 Chemical Element Analysis

Table 11 shows the Carbon, Hydrogen, Nitrogen and Sulphur content of two chosen fibre weight percentage Coconut-HDPE biocomposite (13.75 wt% and 21.25%). The result shows that dominant element in this material is Carbon with little hydrogen while there are no sign of Nitrogen and Sulphur element. The CHNS test result shows that there is increment in Carbon percentage of the biocomposite after been recycled as shown in Fig 27. The 13.75 fibre wt% biocomposite shows increment of 5.97 carbon % while the 21.75 fibre wt% biocomposite shows increment of 7.66 carbon %. Generally, it is noticeable that with the increment of fibre weight percentage, the increment of carbon % is more significant. This increment of carbon percentage after the recycling process is one of reason and factor that explains the increment of mechanical properties of the biocomposite after being recycled.

Fibre Fraction:	13.75%		21.25%	
Number of cycle	0	1	0	1
Specimen weight (mg)	1.992	1.623	1.691	1.772
Carbon (%)	77.67	83.63	76.86	84.52
Hydrogen (%)	11.95	12.80	11.60	13.28
Nitrogen (%)	0.08	0.00	0.03	0.00
Sulphur (%)	0.00	0.00	0.00	0.00

Table 11: Carbon, Hydrogen, Nitrogen and Sulphur content of the Coconut-HDPE biocomposite.



Fig 27: Graph of Carbon Content (%) of without recycled and after recycled biocomposite.

CHAPTER 5

CONCLUSION & RECOMMENDATION

5.1 Conclusion

The main purpose of this research is to study the recyclability of Coconut-HDPE biocomposite with different fibre weight percentage. Mechanical, thermal and chemical properties of the Coconut-HDPE biocomposite have been investigated.

Firstly, it has shown that with the increment of coconut fibre weight percentage, the Coconut-HDPE biocomposite is able to maintain the tensile strength and increase in Young's modulus. While for the Flexural properties, both flexural strength and flexural modulus show major positive increment. The Coconut fibre play more important role in reinforce the flexural strength of the biocomposite compared to tensile strength.

For the recyclability, the recycling process has shown influence in different parameters such as:

- Mechanical Properties. After recycled, it still able to maintain the tensile strength properties while the Young's modulus of the biocomposite shows positive increment. For flexural property, the biocomposite shows major increment in both flexural strength and flexural modulus. The SEM analysis has shown that the recycled biocomposite has more evenly distributed fibre in th biocomposite which is in more homogeneous distribution.
- Thermal Properties. The calorimetric study shows that the degree of crystallinity
 of the biocomposite increase after being recycled. There microstructure of the
 biocomposite is more homogeneous. This phenomenon explains the increment of
 mechanical properties of the biocomposite after being recycled.
- Chemical Properties. The CHNS study shows that the Carbon percentage of the biocomposite increases after being recycled. This increase of Carbon percentage is one of the factors for the increment of mechanical properties of the

biocomposite after being recycled. It also shows that higher Coconut fibre content in the biocomposite will cause more significant increment of Carbon percentage after recycling.

Finally, it is concluded that Coconut-HDPE composite is recyclable with favourable mechanical properties. Moreover, usually in industry, recycled material is always mixed with virgin material.

5.2 Future Work Recommendation

The effect of recycling on the rheological behaviour of HDPE of the biocomposite analysis is recommended for future study. This is to analyse the degradation degree of the HDPE after being recycled. The reinforcement geometry analysis also been recommended for future study to analyse the change of reinforcement aspect ratio due to recycling.

In addition, the water absorption analysis can be done on the biocomposite as well to prove the water resistance of the Cocount fibre in the biocomposite.

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APPENDICES

F 1: (a) SEM micrograph of the fracture surface of BC-20% after one injection. (b) SEM micrograph of the fracture surface of BC-20% after six injection. Source: A.L.Duigou^a, I.Pillin, A.Bourmaud^a, P.Davies^a, C.Baley^b [10]



F2: Evolution of molecular weight as a function of injection cycles. Source: D.A.Steenkamer, J.L.Sullivan [11]



F3: Wool fibre and the wool powder. (1) Wool fibre ground for 5 min (single fibre) (sample 1#), (2) wool fibre was ground for 0.5 h (sample 2#), (3) wool powder ground for 3 h [7] (1) (2)





F4: Cross section of the coconut



<u>F</u>5: Mechanical Sieve shaker and apertures



F6: Low Speed Granulator SG 16-21 machine



F7: ME 20 (III) Injection Molding machine



F8: 5 KN Universal Testing Machine LLOYD with Laserscan Non-contacting Extensormeter.



F9: Final Year Project 1 planning:

	JAN	FEBRUARY					Μ	ARC	Н			MAY			
Details/ Weeks	1	2	3	4	5	6		7	8	9	10	11	12	13	14
Topic selection/confirmation	Δ														
Preliminary Research Study coconut fibre / LDPE,HDPE/ natural fibre composite															
Submission of Preliminary Report				Δ			~								
Further research studies on Natural fibre composite properties/ fabrication/ testing							Brea								
Laboratory equipments familiarization and experiments							ester								
Material Preparation: Fibre grinding and cleaning							Seme								
Submission of Progress Report							id-	Δ							
FYP 1 Seminar							Σ	Δ							
Fibre diameter determination (Sieve analysis)															
Composite specimens production															
Observation (Optical Microscope)															
Tensile test (Without recycled)															
Submission of Interim Report														Δ	
Oral Presentation															Δ

A = Key milestone

F10: Final Year Project 2 planning:

	JULY	AUGUST					SEP	ΓΕΜΕ	BER		OCTOBER				NOVEMBER
Details/ Weeks	1	2	3	4	5	6		7	8	9	10	11	12	13	14
Planning for FYP 2 (With SV)															
Flexural Test (Without recycled)															
SEM test (Without recycled)															
DSC Test (Without recycled)															
CHNS Test (Without recycled)															
1st Sub-Analysis Conclusion							eak								
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Submission of Progress Report 2									Δ						
Seminar									Δ						
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Final Report Preparation															
Submission of Dissertation Final Draft															Δ
Final Presentation															Δ

A = Key milestone