Effects of Particle Size on Mechanical Properties of Oil Palm Fruit Bunch Composites

by

Mohd Roziee bin Yahaya

Dissertation submitted in partial fulfilment of
the requirements for the
Bachelor of Engineering (Hons)
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Universiti Teknologi PETRONAS
Bandar Seri Iskandar
31750 Tronoh
Perak Darul Ridzuan

CERTIFICATION OF APPROVAL

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A project dissertation submitted to the
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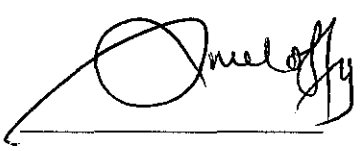
Approved by,

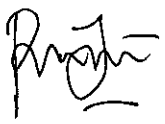


(Dr. Puteri Sri Melor Megat Yusoff)
Main Supervisor

Universiti Teknologi PETRONAS
Tronoh, Perak
JANUARY 2008

CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.



MOHD ROZIEE BIN YAHAYA

ABSTRACT

Oil palm empty fruit bunch (EFB) reinforced composite is an emerging area in polymer technology. EFB is a low cost filler with low density, high specific properties as well as being nonabrasive. The objective of the work reported is to study the effect of particle size on mechanical properties (flexural and tensile properties) of EFB-polyethylene composites.

Three different particle sizes of EFB have been prepared using 0-150 μm, 150-300 μm, and 300-425 μm. Five formulations of varying content of EFB have been prepared for each filler sizes (0%, 10%, 20%, 30% and 40%). Flexural and tensile test has been carried out to determine the mechanical properties of the composites.

For all three filler sizes, tensile strength decrease gradually with increase in EFB fiber content. However, both tensile and flexural modulus showed no significant improvement because the modulus had to be calculated manually and subjected to human error but there were part of the result showed considerable improvement.

Samples have also been subjected to microstructure observation using scanning electron microscopy (SEM). From the morphology, it showed that the fiber and matrix interface is very low thus the stress concentration is high. Besides, irregular orientation of the fiber distribution have resulted small improvement of mechanical properties. Particle size is important to achieve good combination of mechanical properties.

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LIST OF ABBREVIATIONS

ASTM	American Society for Testing and Materials
EFB	Oil palm empty fruit bunch
ESCR	Environmental stress crack resistance
HDPE	High-density polyethylene
MOR	Modulus of rupture
MWDs	Molecular weight distributions
OPF	Oil palm fronds
OPT	Oil palm trunk
PP	Polypropylene
PS	Polystyrene
PVC	Polyvinylchloride
SEM	Scanning electron microscopy
UTM	Universal Tensile Machine

CHAPTER 1
INTRODUCTION

1.1 Background of Study

Interest in the use of natural fillers has grown during the last decade and most of the natural fibers used in thermoplastics composites are derived from wood. Research on the production of composites from natural fibres is receiving widespread attention because of the growing environmental awareness throughout the world. The use of natural fillers in USA plastic industry in 2000 was estimated of 0.4 billion pounds. In fact, industry experts believe that the demand for natural fillers for plastics composite applications will grow at least six-fold in the next 5 to 7 years [1]. The rationale behind this interest is that the use of natural fibers offers several benefits, including lower cost, less abrasiveness to equipment, high specific properties such as higher specific modulus (modulus/specific gravity), renewable nature and biodegradability. Besides, the advantage of the lignocellulosic materials over synthetic fibres like aramide, carbon or glass fibre is also including their acceptable specific strength properties, low density and enhanced energy recovery [2].

The usage of biofibres as reinforcing fibres in both thermoplastic and thermoset composites provide positive response to such environmental issues through their ultimate biodegradability and as annually renewable raw material. In fiber-filled composites, the fibers serve as reinforcement by giving strength and stiffness to the structure while the plastic matrix serves as the adhesive to hold the fibers in place so that suitable structural components can be made. Furthermore they have good thermal stability and provide excellent insulation against heat and noise which increased the value of these biofibres. Besides, ease in processing also gives an advantage to biofibres over synthetics fibres. Even though natural fibers offer several advantages, there are also disadvantages such as incompatibility of fiber with the thermoplastics matrix, the tendency to form aggregates, and poor resistance to moisture that will greatly reduce the potential of these fibers to be used as reinforcement [1]. Plastics alone are not suitable for load–bearing applications due to their lack of sufficient strength, stiffness, and dimensional stability.

Generally, natural fibres are grouped into four different types depending on their sources: leaf, bast, fruit and seed [2]. One of the most common natural fillers that have been utilised by researchers is oil palm empty fruit bunch (EFB) as shown in Figure 1.1. The oil palm or *Elais guineensis* was first introduced into Malaysia in 1870. At present, there are about 3.1 million hector of land under oil palm cultivation in Malaysia producing a total of over 9 million tonne of crude palm oil annually [3].

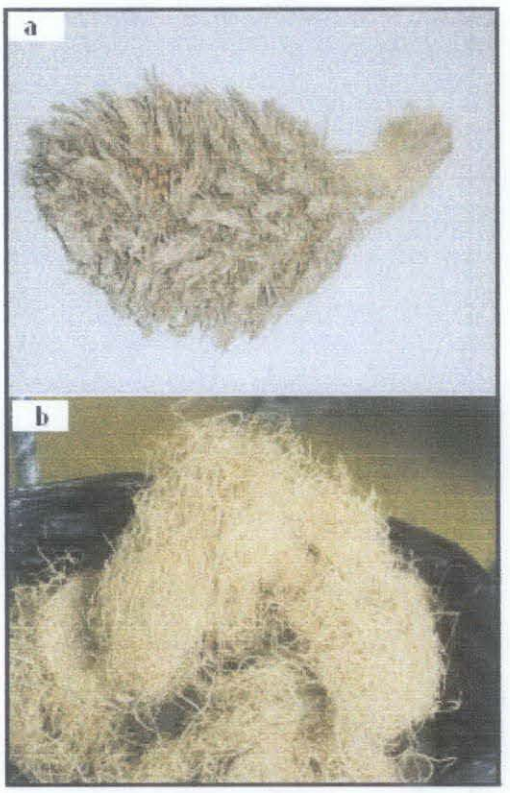

Figure 1.1: (a) Picture of EFB; (b) EFB in fibrous form

EFB is one of the natural fillers obtained from oil palm. Most of the research works on EFB so far used polymers such as polyethylene, polypropylene, polyester, polystyrene and phenol-formaldehyde resin as the matrix material. As lignocellulosic materials, EFB surfaces are covered by polar hydroxyl groups contributed by cellulose, hemicelluloses and lignin [2]. The EFB has the highest fiber yield and is the only material commercially utilized for fiber extraction but there are good potentials for the exploitation of the other two materials namely oil palm fronds and trunks[3].

Oil palm empty fruit bunch (EFB), one of the by-products of oil palm milling process, can be found in abundance in palm oil mills in Malaysia. Compared to other by-products such as oil palm trunk (OPT) and oil palm fronds (OPF), the EFB has the economical advantage of being collected at the oil palm mills. Currently, Sabutek Sdn. Bhd, Ipoh, Perak, Malaysia has commercially produced the pulverized EFB fibres and fillers. Table 1.1 shows the details of the chemical constituent of oil palm EFB fiber. Meanwhile Table 1.2 shows physical and mechanical properties of oil palm EFB fiber [4].

Table 1.1: Chemical constituents of EFB fiber [4]

Chemical constituents	**Composition**
Cellulose	49.6%
Lignin	21.2%
Hemicellulose	18
Pentosan	17.8
Ash	2%

Table 1.2: Physical and Mechanical Properties of EFB fiber [4]

Properties	**Values**
Tensile strength (MPa)	0.1–0.4
Young's modulus (GPa)	1–9
Elongation at break (%)	8–18
Size diameter (μm)	50–500

1.2 Problem Statement

One of the most important aspect in the materials development of engineering thermoplastics is to achieve a good combination of properties and processability at a moderate cost. As far as mechanical properties are concerned, the main target is to achieve a balance between stiffness, strength and toughness. So far, there have not been many studies reported on particle size impact to EFB-HDPE composites. Most of the researches concentrated to inorganic and other natural fiber such as fiber glass and woods. Commonly, property effects occur mostly when grain or particle size reflects aspect of the crystallographic character, thus affects the mechanical properties. [5]. Besides, the homogeneity of the final products is also depending on the size of the particle. Therefore, it is important to study the effect of the particle size in EFB-HDPE composites.

1.3 Objective of the Study

The objective of this study is to investigate the effect of particle size on mechanical properties (tensile and flexural strength) of oil palm empty fruit bunch – polyethylene composites using extrusion process.

1.4 Scope of the Study

In this study, three different particle sizes, which range between 0-150 μm, 150-300 μm and 300-425 μm are employed. These sizes can be achieved using Endeacotts machine which will extract the fiber to the desired sizes. For each particle size, five formulations will be prepared with varying amount of EFB content from 0%, 10%, 20%, 30%, and 40%.

Preparation of the EFB-HDPE samples will be done using extrusion machine. Then, the extruded samples will undergo moulding process through injection molding machine to create 'dog bone' shape. After that, the samples will be tested in flexural and tensile mode to obtain the relevant mechanical properties. Samples will also be subjected to microstructure and morphology observations using scanning electron micrograph (SEM).

CHAPTER 2
LITERATURE REVIEW

2.1 Recent Works

A recent work by Arif [6] investigated on the effects of research reinforcement shape and fiber treatment on the mechanical properties of oil palm empty fruit bunch –polyethylene composites. Oil palm empty fruit bunch (EFB) was used in this work because oil palm trees are abundant in Malaysia and they produce high amount of biomass waste. The biomass waste generated by oil palm industry can be reduced significantly using this natural fiber. The use of thermoplastic polymers with particulate fillers and short fiber reinforcements has grown quickly because of their processability and recycle ability.

In this research, high-density polyethylene (HDPE) composites were fabricated using EFB as the reinforcing material. The effect of reinforcement shape on the tensile and flexural properties have been studied using 5 mm average length of short fiber and 325-400 μm size distribution of particulate fiber. Five samples have been prepared according to the EFB content including the untreated one for both types of fibers. Both tensile and flexural test were carried out using LLOYD Instruments LR5K Universal Tensile Machine according to ISO 527-2 and ISO 178 respectively. Similar steps of experiment will be carried out by the author to prepare the sample employing different particle sizes.

In the work, it was found out that for both reinforcement shape, the tensile strength values decreased as the EFB content increased and the EFB short-fiber HDPE composites displayed higher tensile strength compared to that of the EFB particulate filler-HDPE composites. The reason was that the particulate filler have poor capability to support load transferred from the matrix compared to the short fiber. However, different trends were observed for flexural properties. Flexural strength of both types of fiber composites increased when the EFB content increased. It could be explained that the orientation of the fiber, as the product of injection moulding, played an important role in increasing the values of the flexural strength. Therefore, this study concluded that EFB short fiber-HDPE composites showed higher mechanical properties compared to EFB particulate-HDPE composites.

Considerable improvements were observed in tensile modulus and flexural properties for both types of composites.

Work done by Rozman et al.[2] attempted to produce EFB composites with mesh size of 35 to 60 with various commonly known thermoplastics matrix such as polystyrene (PS), polyvinylchloride (PVC), polypropylene (PP) and HDPE. All types of composites displayed a decreasing trend in modulus of rupture (MOR) as the filler loading was increased as shown in Figure 2.1. As mentioned earlier, the incorporation of fillers may disrupt the continuity of polymer matrix, which may result in the creation of more stress concentration points. Tensile strength results, Figure 2.2 showed that the strength of EFB composites with different thermoplastic matrix depended on the type of matrix used. EFB composites with PVC matrix displayed the highest tensile strength followed by PP, HDPE and PS. The impact strength for various composites showed a decreasing trend as the filler loading was increasing as demonstrated in Figure 2.3.The impact strength of EFB-HDPE composite was significantly higher than the others. This may be attributed to the ability of HDPE matrix to undergo plastic deformation in the form of crazing and shear yielding during the crack propagation process.

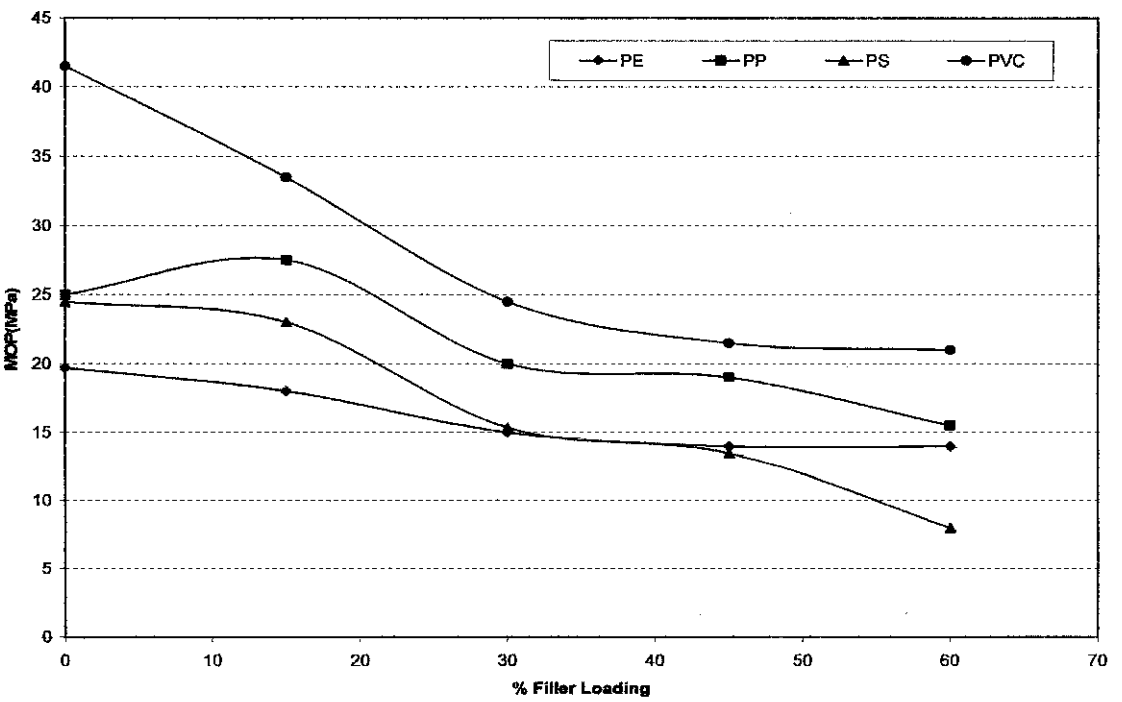

Figure 2.1: MOR of various EFB-thermoplastic composites

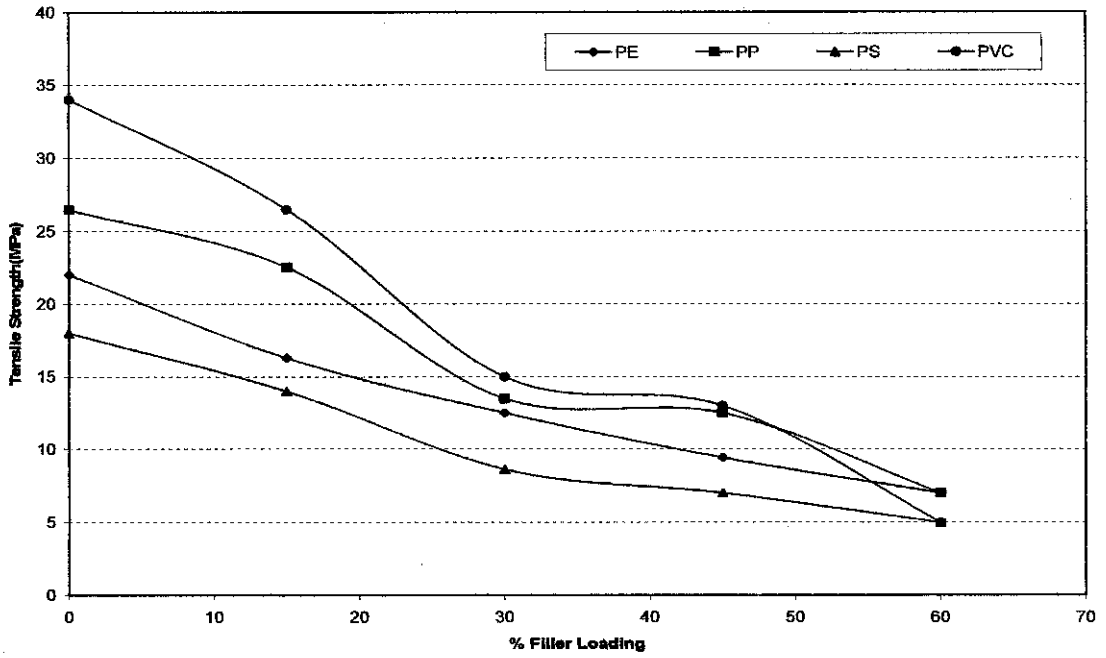

Figure 2.2: Tensile strength of EFB composites made from various thermoplastics

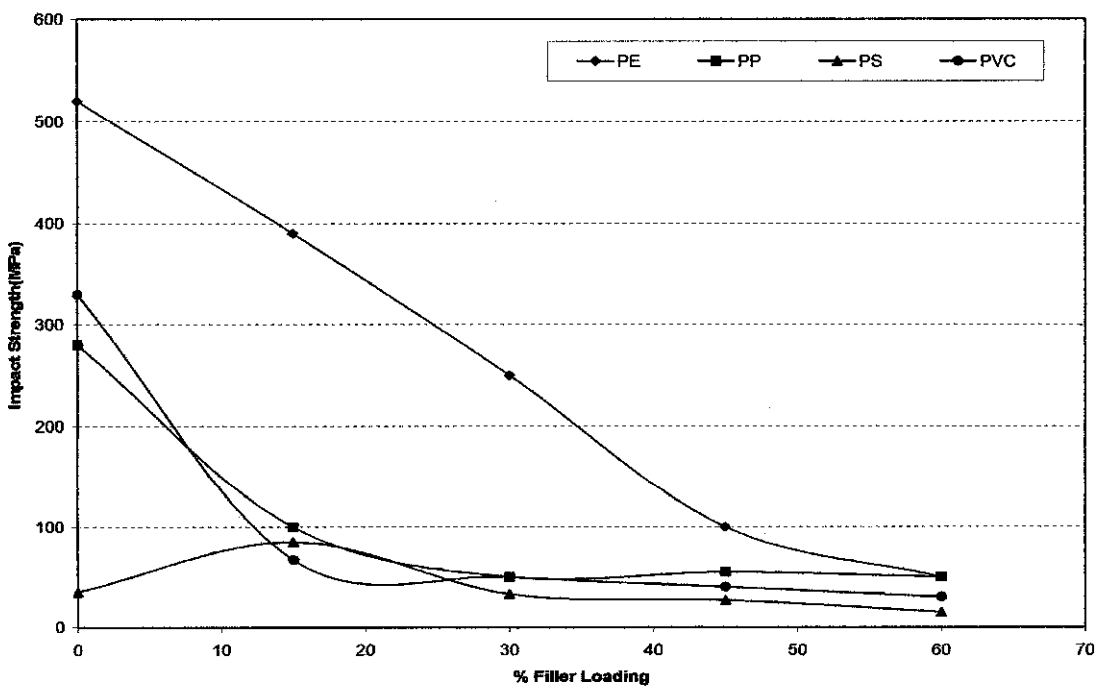

Figure 2.3: Impact strength of EFB composites made from various thermoplastics

Other research related to this work which has also been done by Rozman, H.D. et al. [2] in comparing different particle sizes of EFB-HDPE composites has been discovered. EFB-HDPE composites were first ground to different mesh sizes before use. The materials were compounded with a single-screw extruder. The addition of EFB content for all three sizes does not seem to produce any significant effect on the modulus of rupture (MOR) of the composites as summarised in Table 2.1. However, it can be seen that samples with smaller sizes filler displayed greater MOR than those with the largest ones. This implied that the samples were capable of withstanding higher stress before failure than the ones with larger particle size. EFB composites with filler size of 80 meshes showed the least reduction in MOR as compared to those with larger size. As shown by studies on other lignocellulosic fillers, this could be attributed to the greater interaction and/or dispersion of the finer particles.

Table 2.1: Mechanical properties of EFB-HDPE Composites

Sample % of filler	Flexural			Tensile		
	MOE (MPa)	MOR (MPa)	Toughness	Modulus (MPa)	Strength (MPa)	Impact Strength
Mesh 80-100						
30%	370.3	15.5	26.0	322.4	13.0	70.9
40%	383.2	15.3	25.5	312.5	11.9	52.4
50%	431.3	14.9	21.5	341.9	9.2	48.1
60%	439.0	14.0	19.0	347.8	7.3	57.3
Mesh 60-80						
30%	318.5	11.2	28.9	226.3	9.7	59.7
40%	409.2	10.9	24.6	283.7	9.7	63.2
50%	470.2	9.9	20.8	338.3	7.5	55.7
60%	451.0	7.7	12.9	358.2	7.3	48.7
Mesh 35-60						
30%	299.8	9.89	13.68	328.5	13.2	73.6
40%	360.1	9.9	14.1	364.6	11.7	65.5
50%	412.7	9.8	18.2	438.2	11.1	65.2
60%	453.0	7.0	14.6	375.4	9.7	52.4

In EFB-HDPE composites, poor interfacial interaction is expected owing to poor compatibility, which forms weak interfacial regions. As lignocellulosic materials, EFB surfaces are covered by polar hydroxyl groups contributed by cellulose, hemicelluloses and lignin. This wetting is further decreased as more filler is added. As more filler is incorporated in the composites, more incompatible interfacial regions between polar lignocellulosic and nonpolar polyethylene (PE) are created. The weak interfacial regions result in reduction in the efficiency of stress transfer from the matrix to the reinforcement component.

Rozman et. al also found that the stiffness or modulus of elasticity (MOE) of the EFB-HDPE composites increased as the filler loading was increased. As shown by various studies, incorporation of fillers is able to impart greater stiffness in composites. Generally, samples with smaller particle size filler showed higher modulus. The toughness of the samples decreased as the filler loading was increased. The results showed that samples with smaller particle size displayed higher toughness than those with bigger particle size. As toughness is a measure of energy is needed for failure, the results generally demonstrate that more energy is needed to break samples with smaller size particles.

Tensile strength of the EFB-HDPE composites decreased gradually with filler loading. Filler size did not show any significant influence on the strength of the composites. This is true because incorporation of filler into a thermoplastic matrix does not necessarily increase the tensile strength of a composite. Fibers with uniform circular cross section and a certain aspect ratio normally improve the strength. However, the capability of irregularly shaped fillers with low aspect ratio, as in EFB, to support stresses transferred from the polymer matrix is significantly reduced. EFB fibers have the tendency to exist in fiber bundles. This would mean that the fibers embedded in the matrix would have greater diameters as compared to other wood-based fillers or inorganic filler such as glass fiber. This in turn would reduce the aspect ratio of the fibers.

Significant improvement in the tensile modulus was observed with increasing filler loading. EFB composite with smaller filler size displayed better modulus than those with largest filler sizes. Smaller or finer particles with larger specific area may impart greater interaction with the polymer matrix and can result in uniform filler dispersion in the composite. This indicates the ability of EFB fillers to impart greater stiffness to the HDPE

composites. These results are in agreement with the trends observed in other lignocellulosic-filled thermoplastics.

The impact strength of EFB composites showed a decreasing trend as the filler loading was increased. This clearly indicated that the presence of EFB fibers has reduced the energy absorbing capabilities of the composites. The poor adhesion or bonding between the fibers and polymer matrix creates weak interfacial regions, which will result in debonding and frictional pullout of fiber bundles and inhibit the ductile deformation and mobility of the matrix. This will in turn lower the ability of the composite system to absorb energy during fracture propagation. A similar trend has been reported in the case of wood-flour filled HDPE.

2.2 Theory

2.2.1 High-density polyethylene (HDPE)

HDPE as shown in Figure 2.4 is a polyethylene thermoplastic made from petroleum. HDPE is defined by a density of greater or equal to 0.941 g/cm3 and is determined by a compression molded sheet that has been cooled at the rate of 27°F or 15°C per minutes. HDPE has a low degree of branching and thus stronger intermolecular forces and tensile strength. HDPE is a partially crystalline, partially amorphous thermoplastic material. The degree of crystallinity depends on the molecular weight, the amount of comonomer present, and the heat treatment given [7]. HDPE is used in products and packaging such as milk jugs, detergent bottles, margarine tubs, water pipes, blow moulded bottles, drums, carboys, automotive gas tanks and garbage containers.

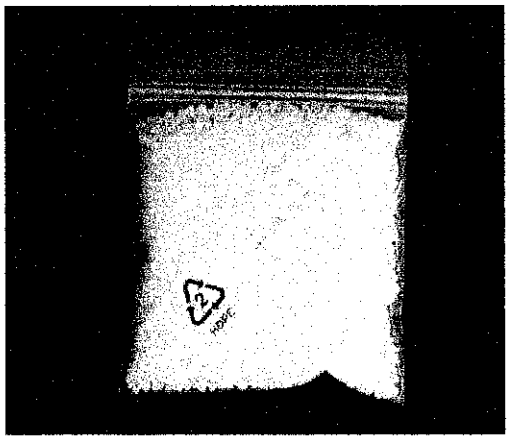

Figure 2.4: Sample of High Density Polyethylene (HDPE)

There are three major commercial processes used for polymerization of HDPE: solution, slurry, and gas-phase processes. The catalysts used in the manufacture of HDPE are usually either a transition metal oxide type or a Ziegler/Natta type. It is important to note that the performance of HDPE resins having identical melt indexes, densities, and molecular weight distributions (MWDs) can vary if the resins are produced by different processes. These differences normally are seen only in critical applications having very narrow processing windows. For most applications, HDPE resins selected from more than one supplier will perform adequately, even if the resins are made by different processes [8].

HDPE has little branching, giving it stronger intermolecular forces and tensile strength than lower density polyethylene. It is also harder and more opaque and can withstand high temperatures of 120 °C for short periods or 110 °C continuously. The lack of branching is ensured by an appropriate choice of catalyst (*e.g.* Ziegler-Natta catalysts and reaction conditions [7]. Figure 2.5 shows the molecular structure of HDPE [9].

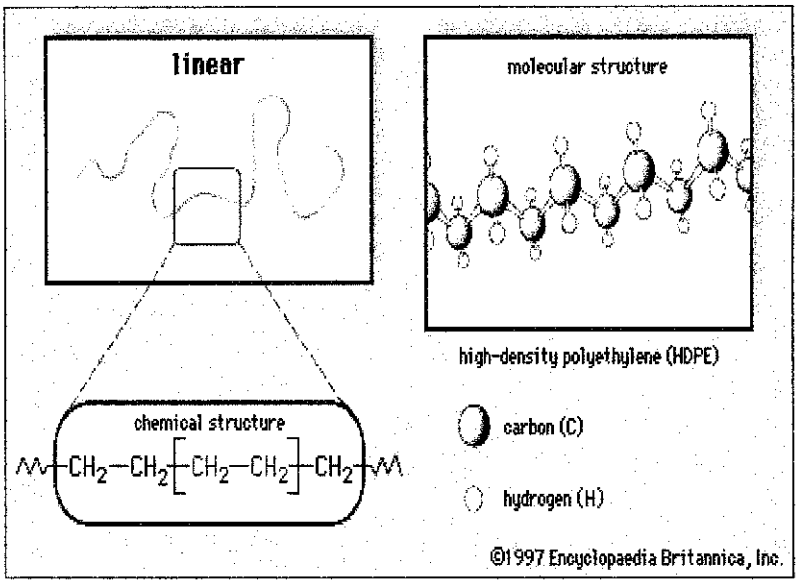

Figure 2.5: Molecular structure of HDPE

In HDPE, the properties of tensile yield strength, stiffness, creep resistance, impermeability, abrasion resistance, mold shrinkage, and hardness increase with increasing density. In contrast, the impact strength, flexibility, and environmental stress crack resistance (ESCR) increase with decreasing density [8].

As the average molecular weight of HDPE increases, the polymer's molten flow decreases. The standard test for measuring the molten flow of HDPE is called the melt index. Melt index is inversely proportional to the average molecular weight. The properties of ESCR, impact strength, tensile strength, elongation, melt strength, and die swell improve with decreasing melt index (and with increasing average molecular weight). The properties of processability melt drawdown, and optics decrease with decreasing melt index. As the average molecular weight of HDPE increases, there is more shrinkage and warpage present in the molded parts[8].

These brief comments on melt index and density indicate that it is not possible to maximize all of the properties of HDPE in a single resin. Therefore, compromises are necessary in designing any HDPE resin. For that reason, most manufacturers of HDPE offer many different HDPE resin grades in their product mix. Selection of an HDPE resin for a given application involves careful evaluation of the application

requirements, in order to select the HDPE resin that most closely satisfies the most important requirements [8].

Table 2.2: Mechanical Properties of HDPE [8]

Mechanical Properties	Test Method	English Units	Value	Metric Units	Value
Density	D792	lb/ft3	58.7-60.3	g/cm3	0.941-0.967
Tensile Strength	D638	psi	2700-4400	MPa	18.6-30.3
Tensile Modulus	D638	psi	1.06-1.09	GPa	1.06-1.09
Flexural Modulus	D790	psi	100,000-240,000	Mpa	689-1654
Elongation To Break	D638	%	100-1000	%	100-1000
Notched Izod At Room Temp	D256	ft-lb/in.	0.5-3.0	J/m	27-160

2.2.2 Endecotts Laboratory Test Sieve

Endecotts Laboratory Test Sieve as shown in Figure 2.6 designed to ensure that the design performance provides optimum sieving action to the sieves to give rapid accurate results. There are certain essential characteristics to look for in a good test sieve shaker. It should generate an effective sieving action for tests to reach an ultimate end point where the end point should be reached in the shortest possible time. Besides, the results achieved should be reproducible [10].

Figure 2.6: Endecotts Laboratory Test Sieve

The EFL has been specifically designed to operate with heavy or light samples without loss of performance. It is equipped with a dynamic power source which ensures the right vibration is imparted to the sieves and sample for fast, accurate and reproducible tests. The sample in the test sieves will vibrate vertically and rotate over the full surface of the sieve as shown in Figure 2.7 where it can be presented to the maximum number of apertures in the minimum time and also helps keep the apertures clear and free from pegging [10].

Figure 2.7: Motion of Test Sieves

The Endecotts shakers are equipped with a unique clamping device enabling the clamp plate to be fitted in seconds. The device also ensures the clamp plate secures the sieves with consistent pressure providing positive clamping and reproducible results. The timer can be pre-set for any duration up to 60 minutes. Non-corrodible and non metallic springs are fitted thus making the Endecotts maintenance free.

2.2.3 Extrusion process

Extrusion is a process where a solid plastic (also called resin), usually in the form of beads or pellets, is continuously fed to a heated chamber and carried along by a feed screw within. Figure 2.8 shows a schematic diagram of a basic extruder machine (typical extruder machine can be seen in Appendix B). The feed screw is driven via drive/motor and tight speed and torque control is critical to product quality. As it is conveyed it is compressed, melted, and forced out of the chamber at a steady rate through a die. The immediate cooling of the melt results in resolidification of the plastic into a continually drawn piece whose cross section matches the die pattern. This die has been engineered and machined to ensure that the melt flows in a precise desired shape [11].

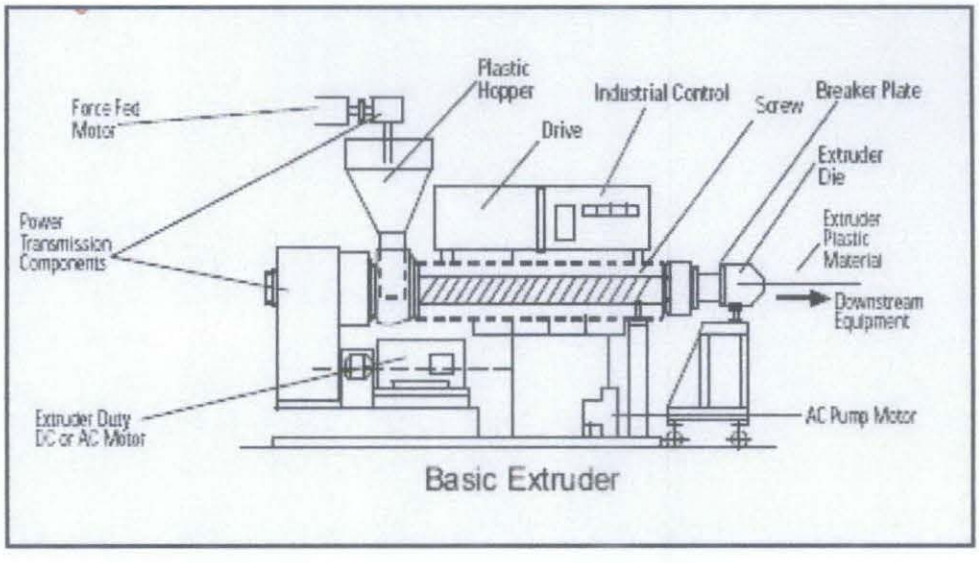

Figure 2.8: Component of Basic Extruder Machine

Examples of extruders products are blown film, pipe, coated paper, plastic filaments for brush bristles, carpet fibers and vinyl siding. There is almost always downstream processing equipment that is fed by the extruder. Depending on the end product, the extrusion may be blown into film, wound, spun, folded, and rolled, plus a number of other possibilities.

Plastics are very common materials for extrusion. Rubber and foodstuffs are also quite often processed via extrusion. Occasionally, metals such as aluminum are extruded plus trends and new technologies are allowing an ever-widening variety of materials and composites to be extruded at continually increasing throughput rates.

2.2.4 Tensile Strength

Tensile strength is a maximum load applied in breaking a tensile test piece divided by the original cross-sectional area of the test piece. It is originally quoted as Pa (Pascal) or psi (pound per square inch) where 1 MPa is equal to 145 psi [12]. Figure 2.9 shows stress-strain diagram indicating the tensile strength and the breaking point [13].

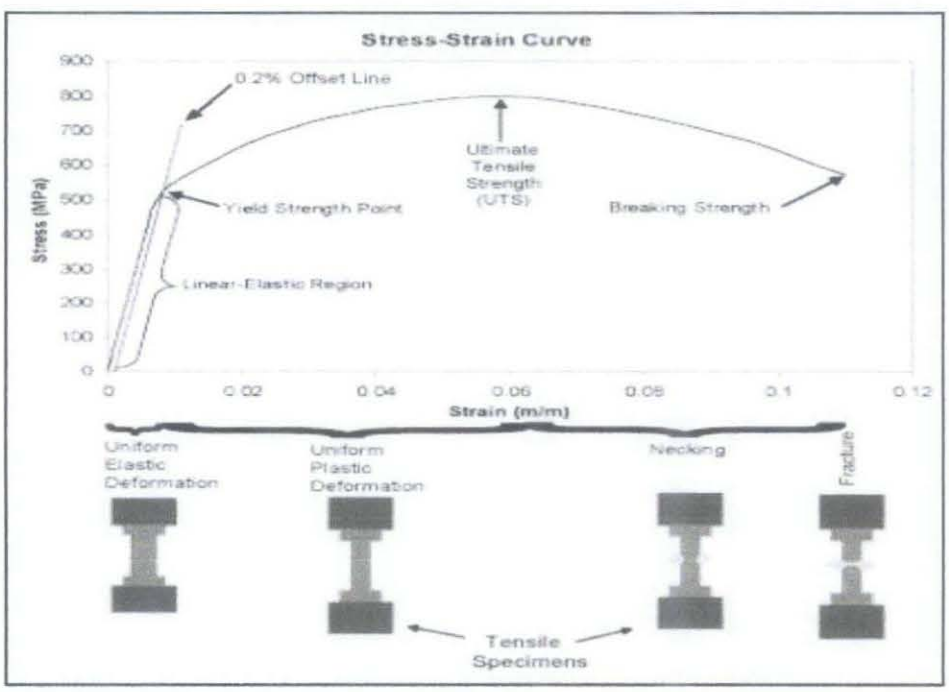

Figure 2.9: Stress-strain Diagram

Fibers need good tensile strength. Tensile strength is important for a material that is going to be stretched or under tension. As stated above, the tensile strength of a material is the maximum amount of tensile stress that it can be subjected to without failure. The definition of failure can vary according to material type and design methodology. This is an important concept in engineering, especially in the fields of material science, mechanical engineering and structural engineering [14].

There are three typical definitions of strengths which are yield strength, ultimate strength, breaking strength [14]. Yield strength is the stress at which material strain changes from elastic deformation to plastic deformation, causing it to deform permanently meanwhile ultimate strength is the maximum stress a material can withstand. The third one which is breaking strength is the stress coordinates on the stress-strain curve at the point of rupture.

2.2.5 Flexural Strength

Flexural strength of a material is defined as its ability to resist deformation under bending load. For materials that deform significantly but do not break, the load at yield, typically measured at 5% deformation/strain of the outer surface, is reported as the flexural strength or flexural yield strength. The test beam is under compressive stress at the concave surface and tensile stress at the convex surface. Figure 2.10 shows the test geometry for ASTM D790 [15].

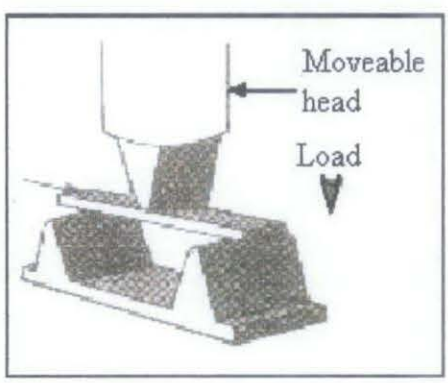

Figure 2.10: Support Span Arrangement for Flexural Testing (ASTM D790).

The analogous test to measure flexural strength in the ISO system is ISO 178. The values reported in the ASTM D790 and ISO 178 tests seldom differ significantly. These tests also give the procedure to measure a material's flexural modulus (the ratio of stress to strain in flexural deformation).

CHAPTER 3
METHODOLOGY

3.1 Experimental Procedures

3.1.1 Materials

The most important part to start this research is obtaining raw material involved. Filler types used are in the form of pulverized and 5mm average length of short fiber. HDPE used has a melt flow index of 7g/min and a density of 0.961 g/cm3 supplied by Titan Petchem (M) Sdn. Bhd. Figure 3.1 shows the sample of EFB fiber obtained from Sabutek (M) Sdn. Bhd.

Figure 3.1: Sample of the EFB fiber

3.1.2 Filler Preparations

The EFB fibers was ground several times using grinder model OM to obtain fibers of sizes ranging from 400 to 700 μm and was further refined using grinder model FDS-IHP to get fibers of sizes less than 300 μm. The fibers were then passed through sieves of 600, 425, 300 and 150 μm using Endecotts Laboratory Test Sieve. Those sizes are 0-150 μm, 150-300 μm, and 300-425 μm. Each mesh sizes will be having 5 samples. To remove moisture content, EFB then need to put in the oven at 80oC for 24 hours.

3.1.3 Sample Fabrications

The mixing of EFB and HDPE was carried out using the twin screw extruder Leistritz Type Mio27/GI-32D as shown in Figure 3.2. The compound was extruded through a single 2.5 mm rod die and palletized. The extrusion products was transferred to injection molding machine, Tat Ming ME20 III as shown in Figure 3.3 to create 'dog bone' shape. The EFB content is varied from 0, 10, 20 30 to 40 %wt. at each filler size.

Figure 3.2: Leistritz Type Mio27/GI-32D Twin Screw Extruder

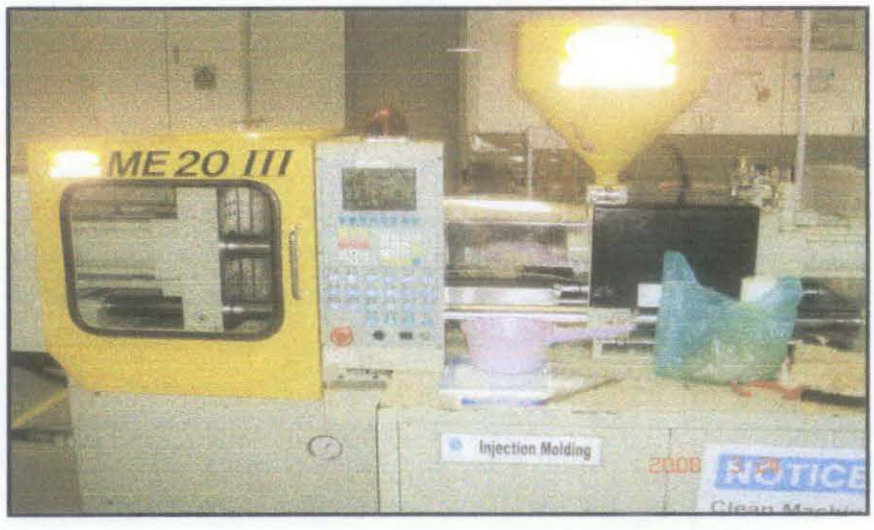

Figure 3.3: Tat Ming ME20 III Injection Molding

3.2 Testing Techniques

3.2.1 Flexural Test

Flexural test determines the strength of material when a force is applied perpendicular to the longitudinal axis sample. Flexural test ware carried out using the Universal Testing Machine (UTM) 5kN Llyod type machine as shown in Figure 3.4 according to the ASTM D790 standard, three point bending system.

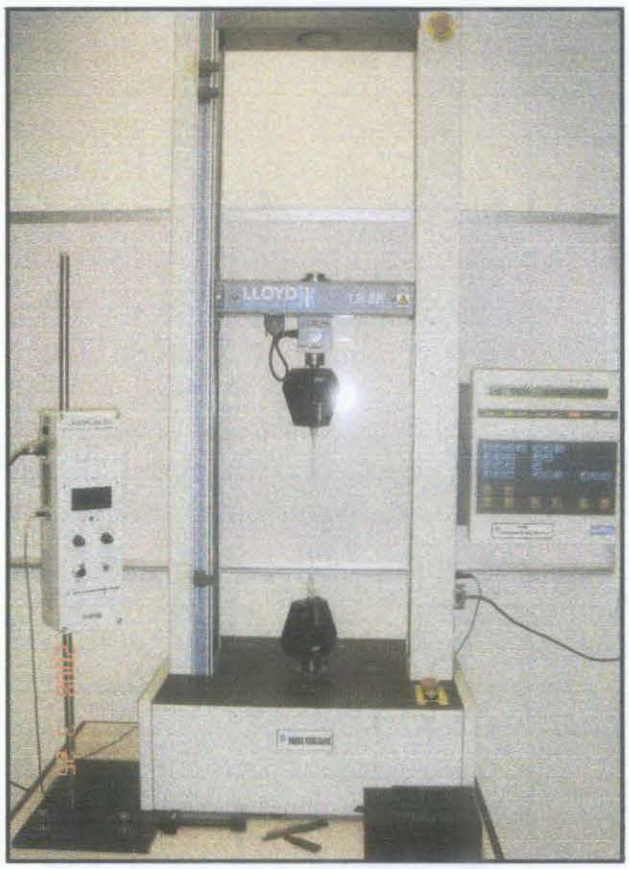

Figure 3.4: Lyold machine

Flexural properties were determined using a universal tensile testing machine MTS (20/MH) model according to ASTM D-790-03. The load displacement curves were obtained and the flexural strength and modulus of composites fabricated from treated and untreated DPF were calculated. The crosshead speed used was 2 mm/min and the dimensions of the specimen were 70 mm x 10 mm x 4 m mm. A span-to-depth The support span for the flexural tests was 55 mm and the diameter of the loading tip was 10 mm. The temperature and relative humidity for all experiments were at room 23 $\pm$ 2°C and 65 $\pm$ 5%, respectively.

The load deflection curve was recorded until failure onset. The flexural strength was calculated using:

$$\sigma_f = \frac{3PL}{2bd^2}$$

where P is the force (N), L is the distance between the support span (mm), b is the specimen width (mm), and d is the specimen thickness (mm). The flexural modulus (E*f*) was calculated using:

$$E_f = \frac{L^3 m}{4bd^3}$$

where m is the slope of the initial straight-line portion of the load-deflection curve.

3.2.2 Tensile Test

In this research the tensile test was carried out according to ASTM D638 on a Lyold machine as shown in Figure 3.3. Dumb-bell specimens 1mm thick was cut from the molded sheets. Two sets of vice jaws are used to clamp the test specimens at the top and at the bottom; hydraulic power is then applied to force the specimen apart. The crosshead speed used was 2 mm/min and the dimensions of the specimen were 70 mm x 10 mm x 4 m mm. A dial usually calibrated in pounds, tonnes or Newton, records the load applied. As the load increases, the dial registers the amount until fracture occurs.

3.2.3 Scanning Electron Microscopy (SEM)

The SEM will be used to obtain some information regarding filler dispersion and bonding quality between filler and matrix, and to correlate between fracture surface and energy absorbed. The good specimens which will be choose from every mesh sizes after the test. The fracture ends of the specimens were mounted on an aluminium stubs and sputter coating with a thin layer of gold to avoid electrostatic charging during examination. Philip XL 40 was used to examine the coated surface.

The author had managed to obtain SEM picture of pure EFB fibers as shown in Figure 3.4 from related research which had been done bye Khairiah et al. [17].

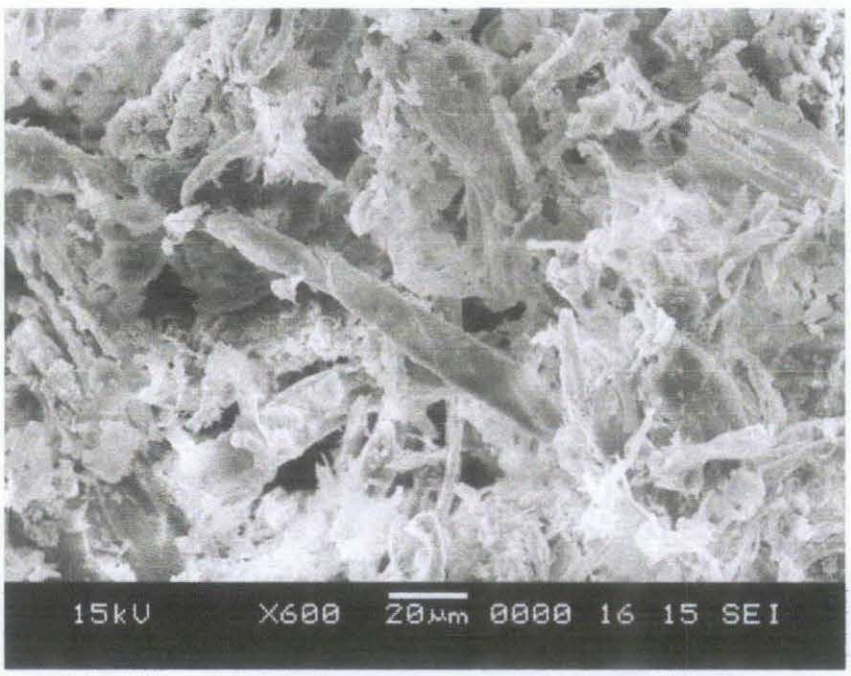

Figure 3.4: Morphology of EFB fillers (Magnification of 600 x)

CHAPTER 4
RESULT AND DISCUSSION

4.1 Mechanical properties of HDPE filled with EFB

Three different sizes of EFB composites were produced according to its mesh sizes. The materials were compounded with a single-screw extruder. Both flexural and tensile test had were carried out using different type Universal Testing Machine (UTM) machine which are Amsler HA 100 kN machine and 5 kN Llyod type machine respectively. This is because the software of 5 kn Llyod type machine encountered major setback while the author doing the flexural testing.Table 4.1 displayed the results of tensile and flexural properties for 100% pure HPDE followed by three different filler sizes at varying filler contents. Flexural properties for 100% HPDE has been taked from Mr. Arif study [6] because the Amsler machine does not give desired values.

Table 4.1: Mechanical properties of EFB-HDPE composites

Sample % of filler	Flexural		Tensile	
	Modulus (MPa)	Strength (MPa)	Modulus (MPa)	Strength (MPa)
100% HDPE	998.36	28.21	555.73	24.94
150-300μm				
10%	1364.79	35.63	946.75	23.81
20%	1559.77	35.42	775.54	21.40
30%	2079.69	36.59	912.86	20.89
40%	1451.45	29.56	973.30	17.73
300-425 μm				
10%	1819.23	36.64	920.41	23.25
20%	1676.75	36.99	977.54	22.31
30%	1787.23	37.54	800.47	21.21
40%	1455.78	30.10	1147.22	17.98
425-600μm				
10%	1490.44	34.46	920.58	23.09
20%	1338.80	32.47	997.36	21.43
30%	1559.76	35.41	965.25	21.12
40%	1503.44	30.49	1248.70	17.75

The addition of both fillers of all three sizes does not seem to produce any significant effect on the flexural strength of the composites as shown in Table 4.1.Flexural strength has been calculated manually form the graphs obtained since the Aimsler HA 100 kN machine did not give the value directly. By using the equation;

$$\sigma_f = \frac{3PL}{2bd^2}$$

where P is maximum load acting to the sample in Newton(N), L is the distance between the support span which is constant (55 mm), b is the specimen width which is also constant (10 mm), and d is the specimen thickness which is also constant (4mm).

As shown in figure 4.1, it does not show any significant effect as the filler sizes increase except for 40% filler content. For 40% filler content, the sample with larger size displayed greater flexural strength than the smaller one but the increment is very small. This shows that flexural strength increase with the decrease in EFB filler content. This would also explained that the orientation of the fiber, as the product of injection molding, play an important role in increasing the value of flexural strength [6]. The increase in flexural strength is from only up to 1.83%. This unrealistic trend occur because of the Aimsler HA 100 kN machine is not suitable to display the effect of mechanical properties for polymer and composites. It seems that the huge margin (100 kN) may affected the values where maximum load needed for polymers and composites are below 1 kN.

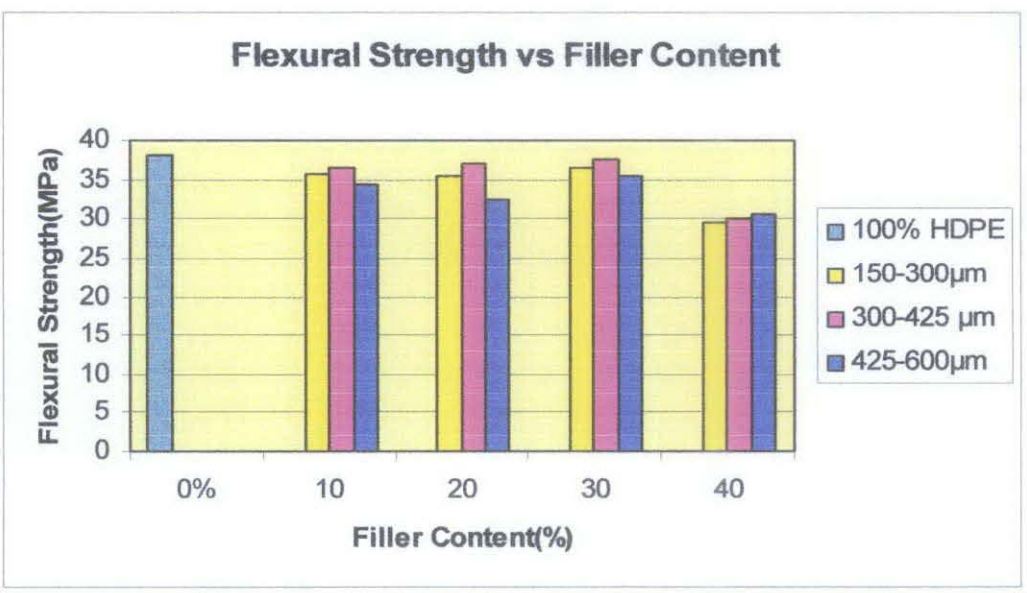

Figure 4.1: Flexural strength of EFB-HDPE composites at varying of EFB filler content.

Same situation happened for flexural modulus. Flexural modulus (E_f) is calculated manually by using the equation;

$$E_f = \frac{L^3 m}{4bd^3}$$

where m is the slope of the initial straight-line portion of the load-deflection curve. It could also be seen that flexural modulus does not seems to have any realistic trend as shown in Figure 4.2.The exact value for m cannot be obtained because the range of the graph are not precise and human error tends to occur when the straight line are not properly drawn. However, by referring filler sizes for 150-300 µm, it shows an increasing trend as the EFB content increased. This implies that, by increasing the filler content, it will increase the ability to impart greater stiffness in the composites, as shown by various studies [2]. An increasing trend also displayed by 40% EFB filler content as the filler sizes increased. The small increment in flexural modulus is up to 3.27%.

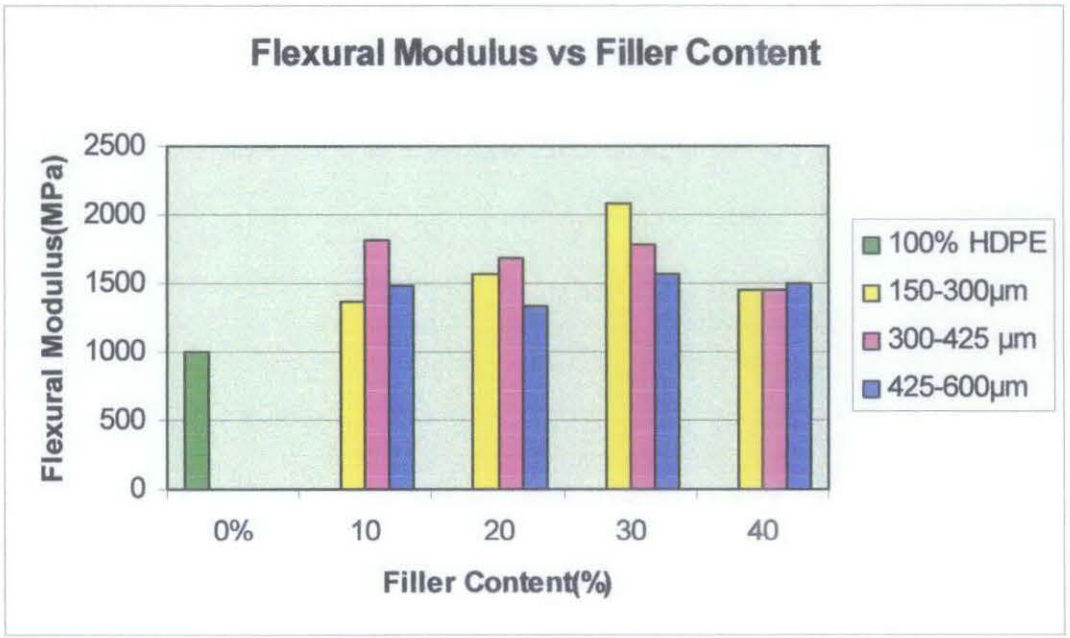

Figure 4.2: Flexural modulus of EFB-HDPE composites at varying of EFB filler content.

Figure 4.3 shows that the effect of EFB content on the tensile properties of EFB-HDPE composites. It is clear that the tensile strength decreased gradually as the EFB filler content increased. The decreased is up to 1.5 and 16%. The decrease is in tensile strength

happened because of poor adhesion between EFB and matrix, which created micro crack formation at the interface [6]. Higher EFB contents results in more void formation during processing, which lead to micro crack formation under loading and therefore reduces the tensile strength. However, all three filler sizes did not show any significant effect on the strength of the EFB-HDPE composites. As shown by previous studies done by Rozman et al, this is true because incorporation of filler into thermoplastic matrix composites does not necessarily increase the tensile strength of the composites [2].

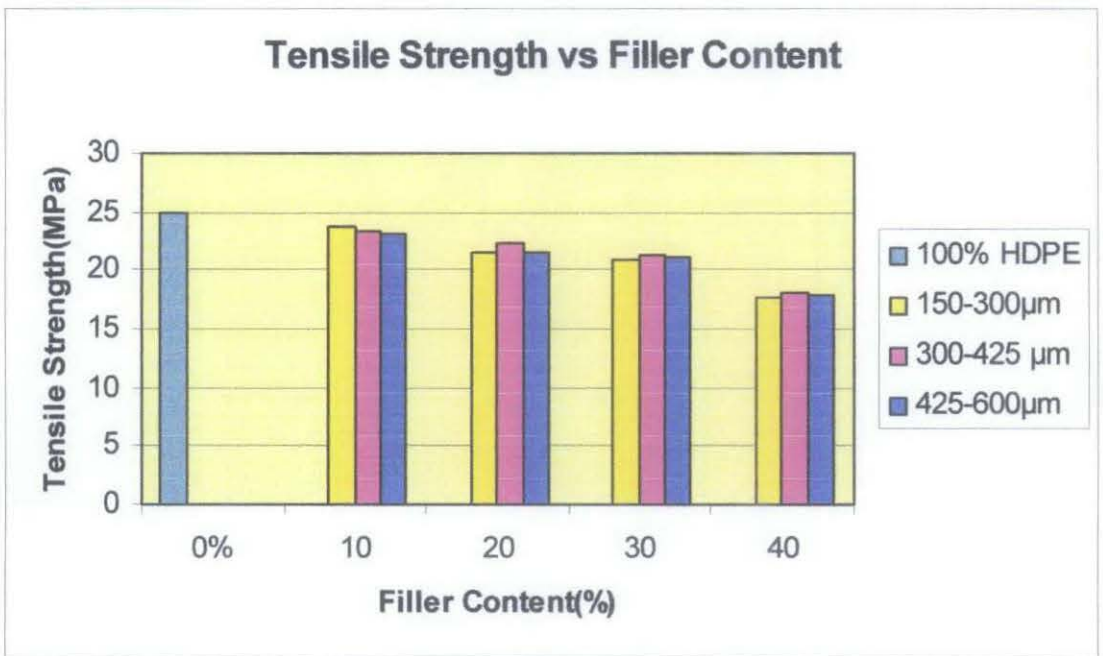

Figure 4.3: Tensile strength of EFB-HDPE composites at varying of EFB filler content.

However, significant improvement in the tensile modulus was observed with the increasing of fiber content. As shown in Figure 4.4, it shows that for 150-300µm, the tensile modulus started increased at 20% fiber content up to 40% filler content. The increment is up to 17.7%. Different trend was obtained for tensile modulus. This would be resulted from human error while conducting the test. The samples were not properly clamped and therefore, the extension does not start at 0 as the initial point. These have affected the tensile modulus where the value of slope had changed. Therefore, the author have calculated the tensile modulus manually by referring the greatest slope from the stress vs. strain curve.

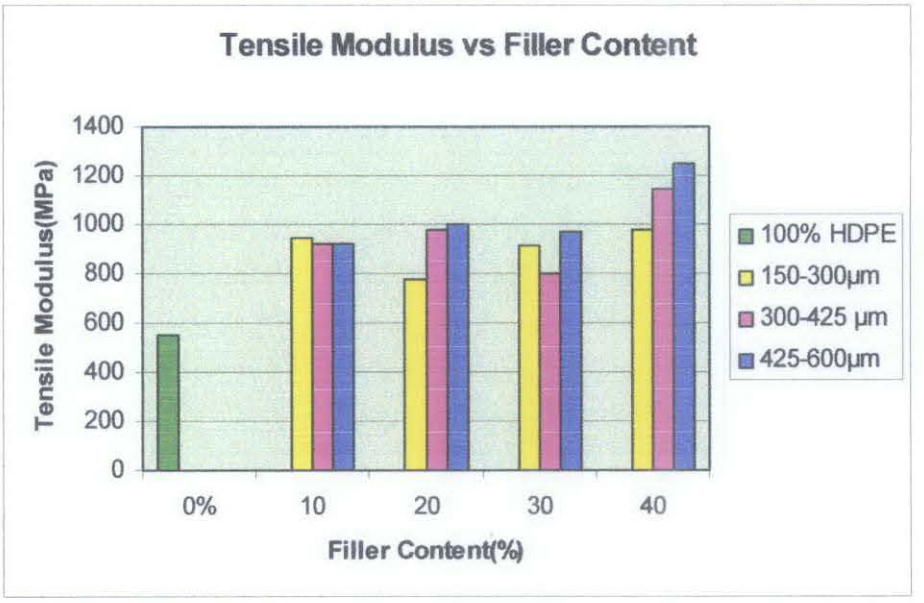

Figure 4.4: Tensile modulus of EFB-HDPE composites at varying of EFB filler content

4.2 SEM Micrograph

The morphology of the composites had been done using Philip XL 40 machine where the best of fractured sample were chosen and being coated before it can be transferred to the machine. As shown in Figure 4.5, there is a huge hole appear in the surface area where there fiber content should be located. After the fracture occurred, the fiber had been taken out and as a result, a huge hole exists. This is because of there is an interfacial region between the pure HDPE and EFB fiber which created stress concentration at that area. Therefore, the sample would easily break because the stress cannot past from one point to another. The morphology also showed that there were irregular orientations of the fiber distribution. When this happened, it will create more stress concentration point, which will lower the tensile and flexural properties.

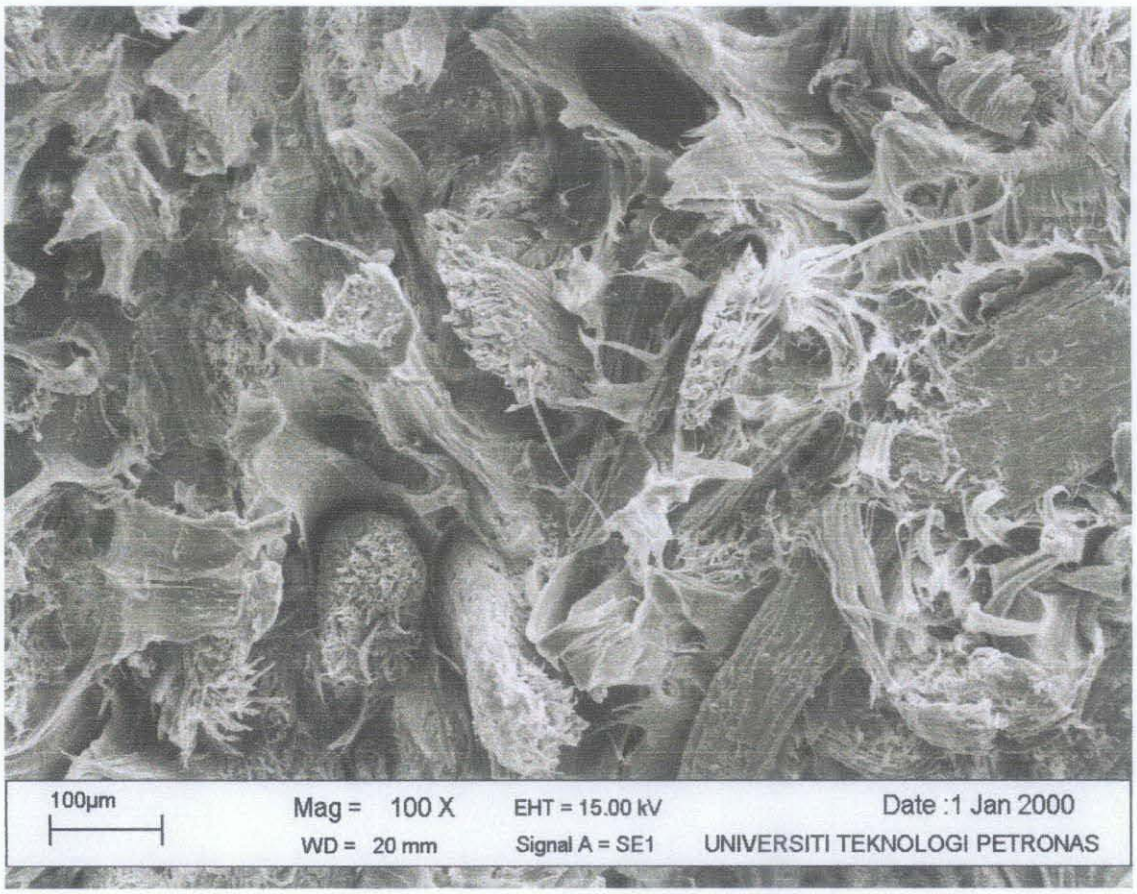

Figure 4.5: Morphology of 20% EFB content for 150-300 µm filler size

Figure 4.6 showed the morphology for the bigger size where, the hole can be seen clearly. This also shows that the filler and matrix interface is very low. This would create. Stress concentration point where, the sample would tend to break easily. Referring to the result obtained in Table 4.1, it shows that when filler size is higher, tensile modulus would decrease. This would satisfy the morphology of both samples in Figure 4.5 and Figure 4.6 when the filler size is larger, it increase the stress concentration area. Figure 4.7 also showed same conditions with both figure discussed before but with larger filler size.

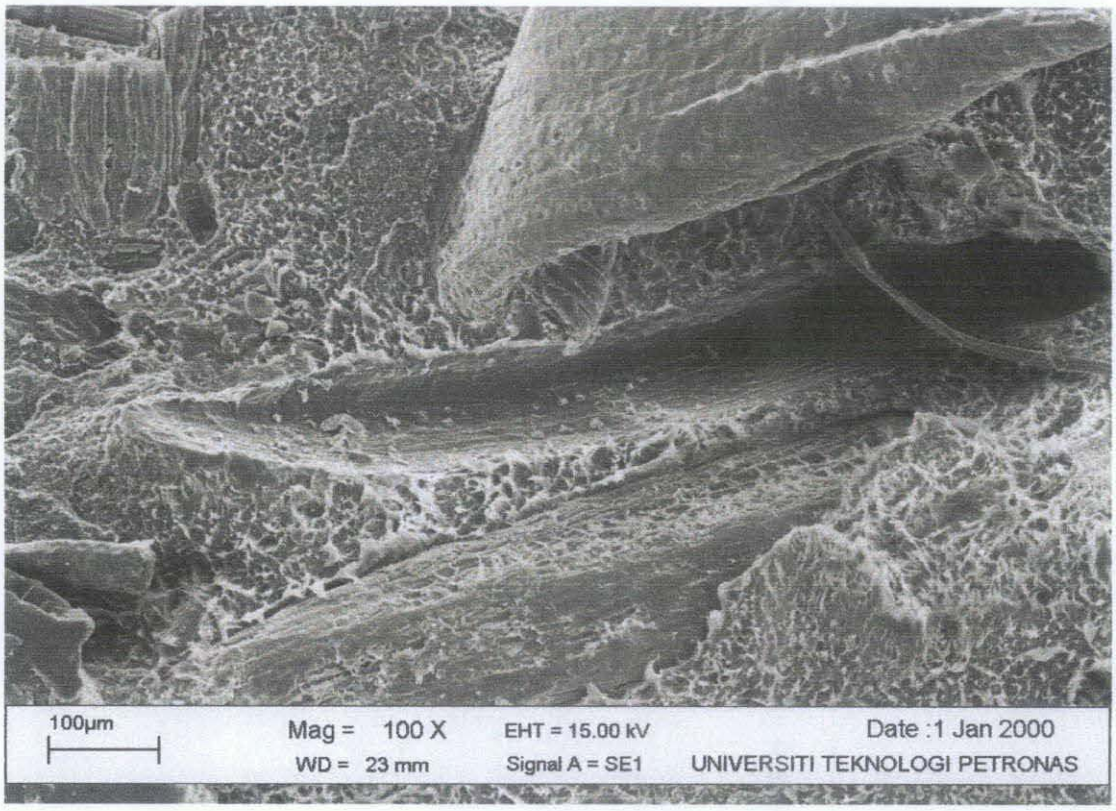

Figure 4.6: Morphology of 20% EFB content for 300-425 µm filler size

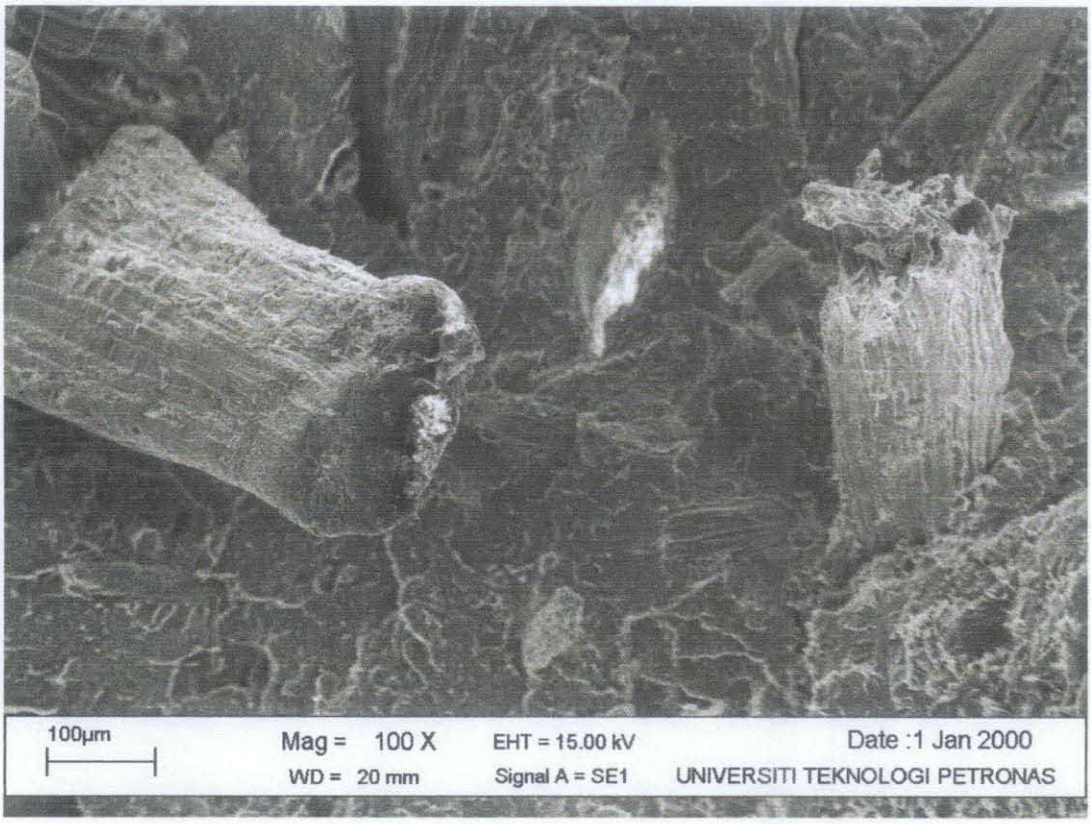

Figure 4.7: Morphology of 20% EFB content for 425-600 µm filler size

CHAPTER 5
CONCLUSION

This study has focused on the effect of particle size on mechanical properties (tensile and flexural strength) of oil palm EFB-HDPE composites by using extrusion process. Three filler sizes have been defined with five formulation of varying content of filler loading from 0 to 40 %wt. for each filler sizes. Extrusion process has been chosen as the processing technique thus becomes a fix parameter in order to achieve the objective of the study.

As shown by mechanical properties results, the percentages of fiber content do not affect much on the flexural strength. However, Flexural increased as the filler size of increased. However, the result would be more accurate only if UTM Llyod 5 kN machine is used. Therefore, the flexural strength and flexural modulus will obtain directly from the software connected to the machine.

The trend observed from the, particle size of EFB-HDPE did not show any significant influence on the tensile strength. For EFB with uniform circular cross section area normally improved the strength. However, the tendency to exist as fiber bundles and capability of irregularly shaped fillers would reduce the aspect ratio of the fibers this in turn would also reduce the tensile strength. For tensile modulus, no significant improvement obtained because the modulus were calculated manually and subjected to human and software error. Therefore, the testing should be done carefully without any distraction.

After referring to many works done by others, the author would also recommend that the fiber should undergo chemical treatment, so that there will be huge improvement on the mechanical properties. Tensile properties are depending on stress where stress is force applied divided by area. Therefore, to increase the stress, area must be small and therefore, using nano material instead of micro material is one way to obtained good mechanical properties.

Overall, oil palm EFB has huge potential which posses a variety of appealing properties to be used in matrix composites. With their low cost and consistency in availability, these natural fibers can serve as new or alternative filler to wood. It demonstrates a promising future in lignocellulosic-thermoplastic composites.

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APPENDICES

APPENDIX A

Figure A1: Oil Palm Fruit Bunch and Oil Palm Frond

Figure A2; Mature Oil Palm Plantation.

APPENDIX B

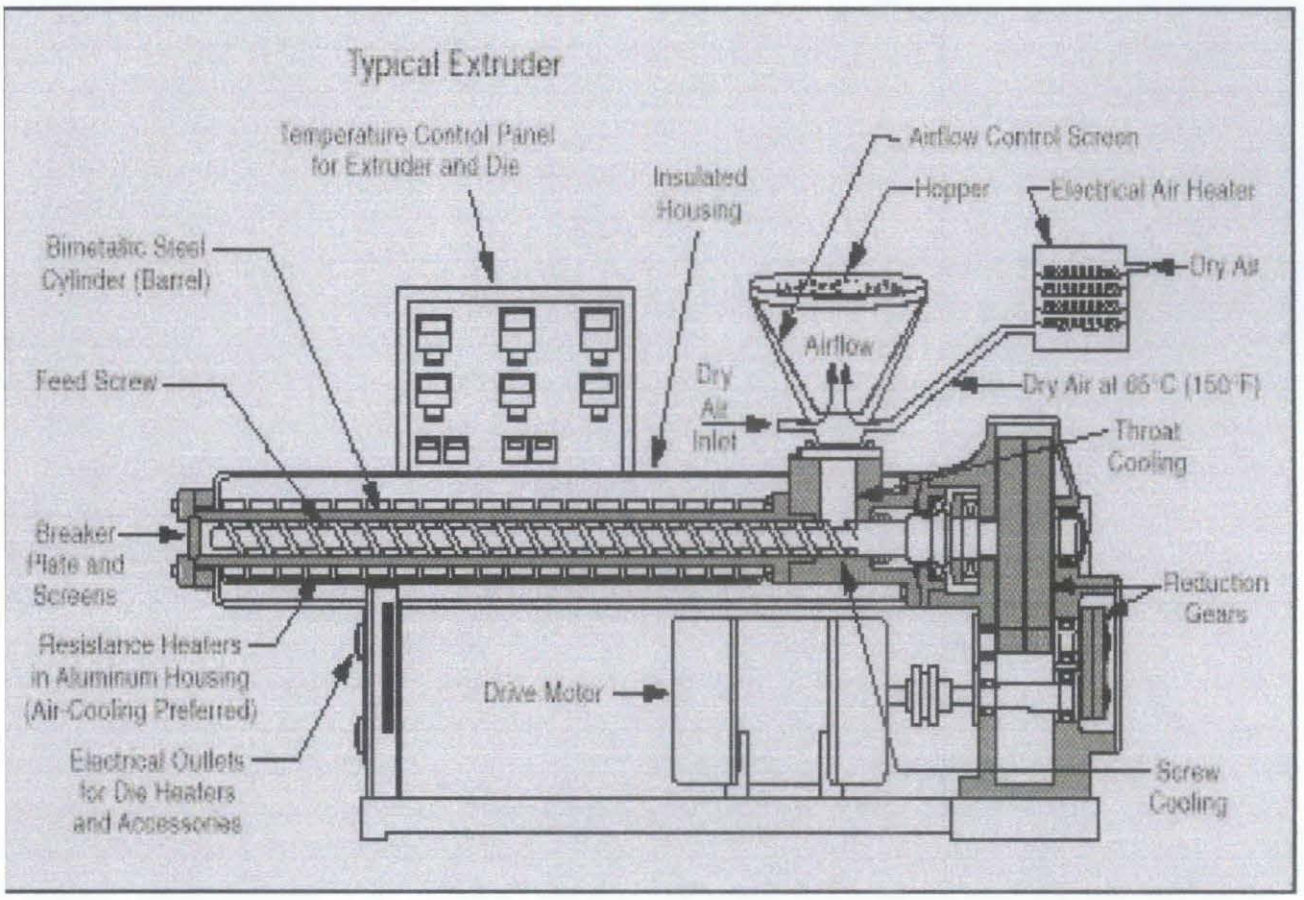
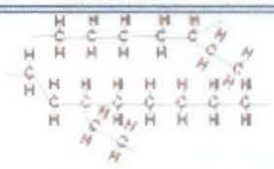


Figure B1: Typical Extruder

APPENDIX C

Table C1: Properties Comparison between LDPE and HDPE

Property	Low Density Polyethylene (LDPE)	High Density Polyethylene (HDPE)
Melting Point	~115°C	~135°C
Crystallinity	low crystallinity (50-60% crystalline) Main chain contains many side chains of 2-4 carbon atoms leading to irregular packing and low crystallinity (amorphous)	highly crystalline (>90% crystalline) contains less than 1 side chain per 200 carbon atoms in the main chain leading to long linear chains that result in regular packing and high crystallinity
Flexibility	more flexible than HDPE due to lower crystallinity	more rigid than LDPE due to higher crystallinity
Strength	not as strong as HDPE due to irregular packing of polymer chains	strong as a result of regular packing of polymer chains
Heat Resistance	Retains toughness & pliability over a wide temperature range, but density drops off dramatically above room temperature.	useful above 100°C
Transparency	good transparency since it is more amorphous (has non-crystalline regions) than HDPE	less transparent than LDPE because it is more crystalline
Density	0.91-0.94 g/cm^3 lower density than HDPE	0.95-0.97 g/cm^3 higher density than LDPE
Chemical Properties	Chemically inert Insolvent at room temperature in most solvents.	chemically inert

	Good resistance to acids and alkalis. Exposure to light and oxygen results in loss of strength and loss of tear resistance.	
Schematic diagram		
Uses	sandwich bags, cling wrap, car covers, squeeze bottles, liners for tanks and ponds, moisture barriers in construction	freezer bags, water pipes, wire and cable insulation, extrusion coating

APPENDIX D

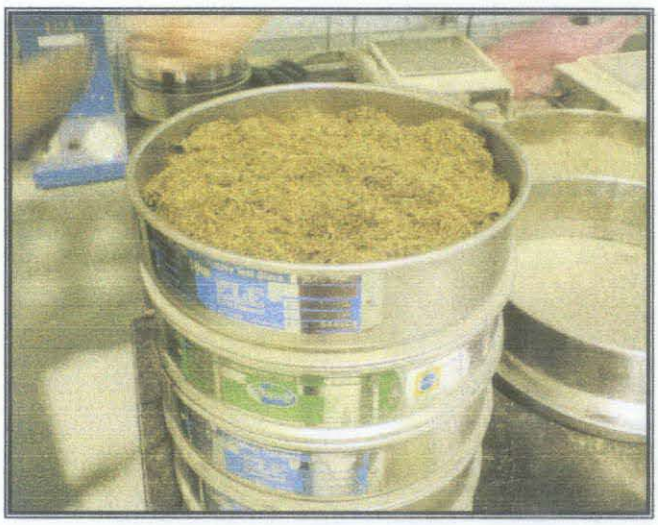


Figure D1: Sample of EFB fiber ready to be sieved

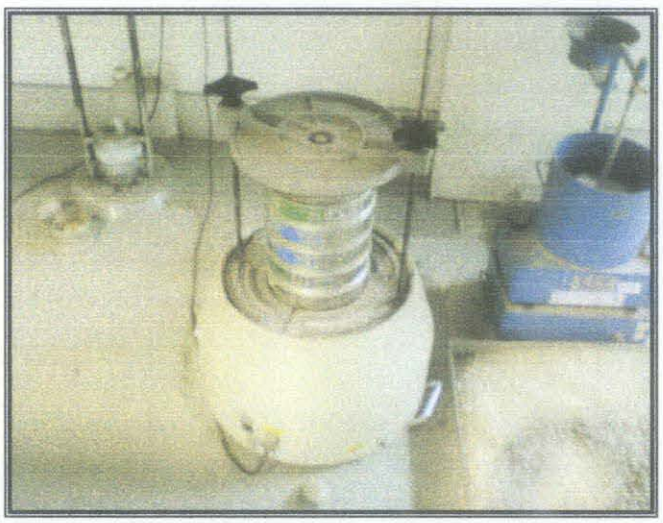


Figure D2: Sieve shaker is running

APPENDIX E

Figure E1: EFB-HDPE composites after compounding in extrusion machine

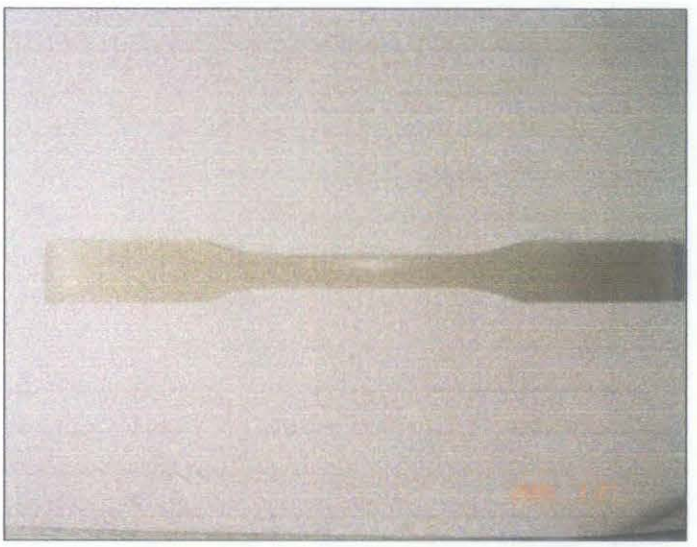


Figure E2: Sample of 100% HDPE using injection molding

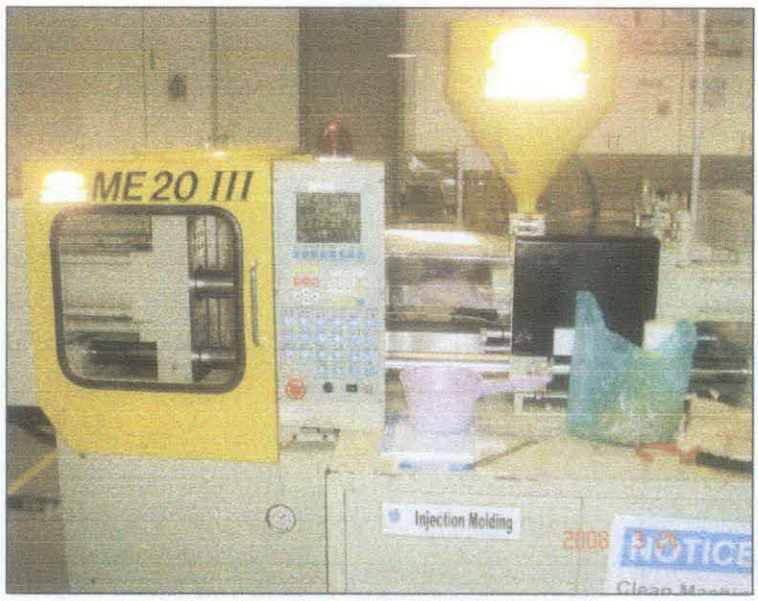


Figure E3: Injection Molding machine

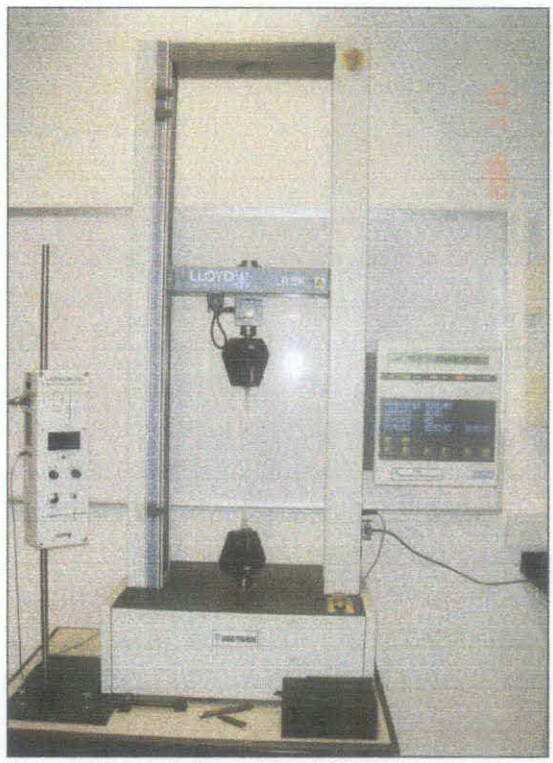


Figure E4: Tensile test for 100% HDPE using Lloyd machine

Appendix F: Sample Calculation for Flexural properties

1. Flexural strength

$$\sigma_f = \frac{3PL}{2bd^2}$$

Where; L = 55 x 10^{-3} m, b = 10 x 10^{-3} m and d = 4 x 10^{-3} m

Therefore;

$$\sigma_f = 515625\ P$$

For 10% EFB filler size 150-300μm, average P is 0.0691 N

∴ σ_f = 515625 (0.0691) = 35629.69 Pa = **35.63 MPa**

2. Flexural Modulus

$$E_f = \frac{L^3 m}{4bd^3}$$

Where; L = 55 x 10^{-3} m, b = 10 x 10^{-3} m and d = 4 x 10^{-3} m

Therefore;

E_f = 64990.234 **m,**

where **m** is the highest slope compute from graph obtained as shown in Figure F1 below.

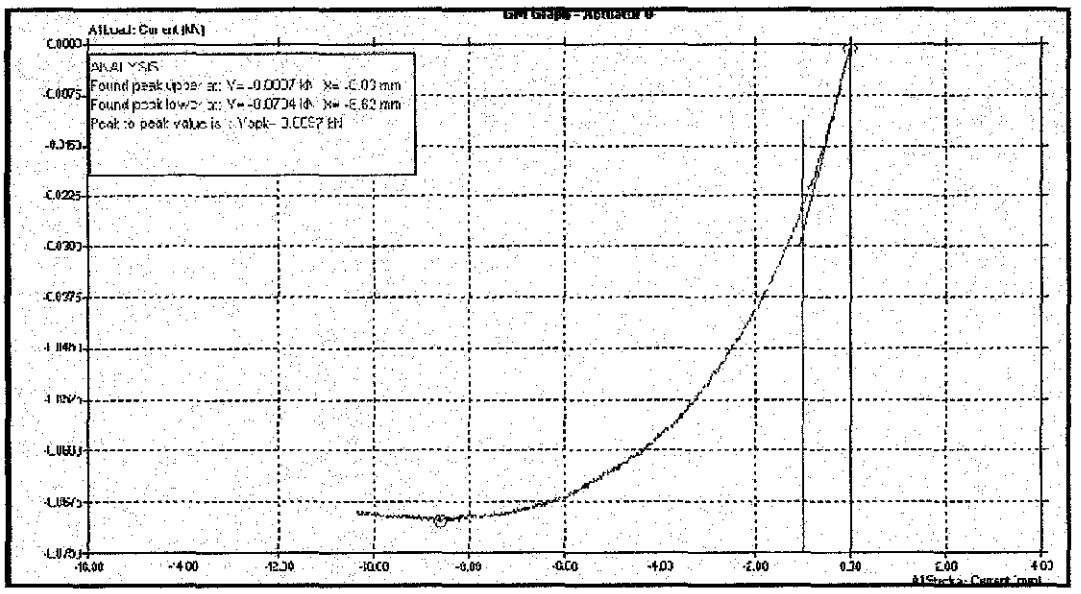


Figure F1: Graph obtained from flexural testing

For 10% EFB filler size 150-300μm, average m is 0.021 m.

$\mathbf{E_f}$ = 64990.234 (0.021) = **1364.79 MPa**

Appendix G: Gantt Chart for FYP II

No.	Detail/ Week	1	2	3	4	5	6	7	8	9	10	11	12	13	14	SW	EW
1	Filler preparation																
	- EFB sieve																
	- HDPE																
2	Sample fabrication																
	- Extrusion process																
	- Injection Moulding																
3	Submission of Progress Report I				15/2												
4	Tensile test																
5	Flexural test																
5	Submission of Progress Report II									21/3							
7	Submission of Dissertation Report													02/5			
8	Oral Presentation & Dissertation															09/5	

SW: Study Week

EW: Exam Week