

CHAPTER 1

INTRODUCTION

1.1 Background of Study

The rise of global environmental awareness, high rate of depletion of petroleum resources, and large consumption of plastics have caught the attention of many researchers to get involved with renewed curiosities in natural materials [1-7]. The utilization of high strength synthetic fibers reinforced composites such as aramid, glass and graphite are broadly employed in the applications of aerospace structures, automotive parts and building materials [3]. However, the high cost of producing these composites has led to the development of alternative materials which are of lower cost and more importantly compatible with the environment. For these purposes, the use of natural fibers in composites as substitute for synthetic fiber reinforcements has taken the central stage and is becoming the interests and attentions of many researchers over the past decades [4]. The use of natural fiber as reinforcing materials in both thermoplastic and thermosets has already found applications in furniture, packaging, building and automobile industries [5].

Generally, natural fibers can be considered as naturally occurring composites consisting mainly of cellulose fibrils embedded in lignin matrix [6]. The main components of natural fibers are cellulose, hemicelluloses, lignin, pectin and waxes [6], and these natural fibers are commonly known as lignocellulosic materials. The advantages of utilizing lignocellulosic materials as reinforcements in composites include lower specific weights which increase the specific strength and stiffness, renewable resources which are available in relative abundance, non abrasive, the ability to recycle without affecting the environment and more importantly lower costs compared to synthetic fibers [6, 7]. However, there are also some drawbacks of using natural fibers such as poor compatibility with non polar thermoplastics,

thermal instability at temperature above 220°C and difficulty in mixing using ordinary plastic mixing equipment [7].

The oil palm or *Elais guineensis* [8] has shown a great potential to be exploited due to the large quantity of biomass generated by palm oil industries. Malaysia produces about 50% of the world's palm oil with 3.1 million ha of land under palm oil cultivation producing a total of over 9 million tons of crude palm oil annually [7, 8]. However, the production of palm oil only represents 10% of the total biomass produced by the industry, of which the remaining 90% consists of mainly lignocellulosic materials [7]. One of the parts in oil palm that is most relevant to be utilized is empty fruit bunch (EFB). EFB is a major biomass waste with 15 million tons per year are being generated and burned in Malaysia which can lead to air pollution [9].

1.2 Problem Statement

The potential of natural fibers as reinforcement to substitute the synthetic fibers such as glass and graphite in composites promises many advantages. This is an alternative way which is more economical and environmentally friendly than that of synthetic fibers. Despite the fact that these fibers may not be as strong as carbon or aramid, the low cost and biodegradability are of great advantages [9].

The compatibility between polymer matrix and natural fibers plays a crucial role in determining the properties of a composite. Several studies showed that EFB of oil palm has the possibility to be an effective reinforcement in thermoplastic and thermosetting materials [7, 10-12]. However, EFB is hydrophilic, which lowers their compatibility with relatively hydrophobic polymer matrices [13]. Therefore, chemical treatments are considered in modifying the fiber surface properties to optimize the interface of fibers.

Many works have been done in order to investigate how different types of fibers can be modified in order to be compatible with certain thermoplastic such as propylene [3, 14, 15]. However, there is still lack of study to investigate the

influence of different types of polymer matrices on the mechanical properties of composites. The polymer matrix may differ in chemical composition, molecular structures and physical properties which can affect mechanical properties of the composite. In addition, different types of polymer matrices have different affinity towards the natural fiber and this affects the compatibility between them as well as the mechanical properties of the composite in general.

Therefore, the focus of this study is to investigate the influence of different types of polymer matrices specifically on thermoplastic on the natural filler filled composites. A systematic investigation is necessary to discover the suitability of different types of polymer matrices with EFB as filler in polymer composites in order to produce better mechanical properties, more economical polymer composites and at the same time benefit the environment.

1.3 Objective

The objective of this project is to investigate the effects of different types of polymer matrices at varying oil palm empty fruit bunch (EFB) content on the mechanical properties of the EFB filled composites.

1.4 Scope of Study

This study involved two (2) types of polymer as the matrix with one (1) type of natural filler. Thermoplastic such as high density polyethylene (HDPE), and low density polyethylene (LDPE) were used as the polymer matrix while oil palm empty fruit bunch (OPEFB) acted as the filler. EFB content was varied from 0, 10, 20, to 30 wt% for each type of polymer matrix. The groundwork was the preparation of samples through grinding and sieving into standard size of particulates (0.325 – 0.4 mm). In order to enhance the interface bonding between EFB and the matrices, chemical treatments using 3 wt% of vinyltrimethoxysilane (VTS) coupling agent was introduced. Injection molding process was done in order to prepare the dog-bone shaped specimen. Later, the specimens were tested for its mechanical properties such

as tensile and flexural. The tensile and flexural fractured surfaces were further analyzed through Scanning Electron Microscopy (SEM) for microstructural analysis.

CHAPTER 2

THEORY AND LITERATURE REVIEW

2.1 Theory

2.1.1 Composite

Composite materials are materials consisting of a discontinuous phase embedded in a continuous phase. The discontinuous phase may consist of at least one constituent material and is usually stiffer and stronger than the continuous phase, if composite materials of high stiffness and high strength with low density are desired. Therefore, the discontinuous phase is usually called reinforcement or reinforcing material while the continuous phase, matrix.

2.1.2 Biocomposite

Biocomposite uses natural fiber as its filler and/or reinforcing material in the composite [16]. Generally, natural fibers are classified into four (4) different types depending on their sources: leaf (sisal, henequen and pineapple fibre), bast (flax, ramie, jute and hemp), fruit (empty fruit bunch) and seed (cotton) [17].

2.1.3 Matrix Material

Composites are commonly utilized for structural applications when high performances are of great importance. Among the large variety of composites certainly polymeric matrix composites are the most used, particularly thermosetting matrix composites. The commercial availability of thermoplastic matrix composites represents a significant recent innovation in the field of composites. Specifically, the use of thermoplastic matrices avoids the presence of dangerous vapours during the composites fabricating as in the case of thermosetting matrices which also represent a strong environmental improvement. The use of thermoplastic composites also

eliminates the necessity of expensive hardware and procedures as required in the use of thermosetting composites. Most commonly used thermoplastics in composites are polyethylene, polypropylene, polystyrene and polyvinyl chloride, mainly due to their low melting point [10].

2.1.3.1 Polyethylene (PE)

PE consists of two (2) carbons and four (4) hydrogens in the basic polymer repeat unit, as shown in Figure 2-1 [18]. PE is classified into several different categories based mostly on its density and branching. The mechanical properties of PE depend significantly on variables such as the extent and type of branching, the crystal structure and the molecular weight [19]. For the purpose of this study, two types of PE will be used, which is high density polyethylene (HDPE) and low density polyethylene (LDPE).

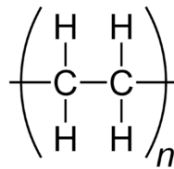


Figure 2-1 Molecular repeat unit of polyethylene

HDPE is defined by specific gravity ranging from 0.94 – 0.97 [19]. HDPE has a low degree of branching and thus stronger intermolecular forces and tensile strength. LDPE is defined by specific gravity ranging from 0.91 – 0.93 [19]. LDPE has a high degree of short and long chain branching, which means that the chains do not pack into the crystal structure as well. It has, therefore, less strong intermolecular forces as the instantaneous-dipole induced-dipole attraction is less. This results in a lower tensile strength and increased ductility. The high degree of branching with long chains gives molten LDPE unique and desirable flow properties. The low melting temperatures below 180°C [19] for both HDPE and LDPE make it possible for these thermoplastics to be used as matrices for natural fiber reinforced composites, as the low processing temperature will prevent thermal degradation of natural fibers.

2.1.4 Chemical Modifications

The problem with incorporating natural fiber in a composite is the poor compatibility of the hydrophilic natural fiber with hydrophobic polymer matrix. Therefore, in order to increase the interfacial bonding, chemical treatments are introduced. Chemicals activate the hydroxyl groups or may introduce new moieties that can effectively interlock with the matrix [20].

2.1.4.1 Silane Treatment

Silane treatment is one example of chemical modifications. The advantages of using silane treatment are commercially available, capable reacting with OH-rich surface and have a large number of functional groups [21].

Silicon (Si) is the center of the silane molecule which contains an organic functional group (R) with a second functional group (X). The functional group (R) will attach to an organic resin while the functional group (X) attaches to an inorganic material or substrate to achieve a 'coupling' effect. Silane coupling agents are predominantly used as mediators, binding organic materials to inorganic materials. Silane coupling agents are primarily used in reinforced plastics and electrical cables composed of crosslinked polyethylene. Other uses include resins, concrete, sealant primers, paint, adhesives, printing inks, and dyeing auxiliaries. The inorganic group (X) of the silane molecule will hydrolyze to produce silanol, which forms a metal hydroxide or siloxane bond with the inorganic material. The organic group (R) of the silane molecule will react with the organic material to produce a covalent bond. As a result, the organic material and the inorganic material are tightly bound together after heating.

2.2 Literature Review

Research on the influence of polymer matrices on the mechanical properties of natural filler composite was previously conducted [10]. EFB was used as natural filler to be incorporated into different types of polymer matrices, such as polystyrene

(PS), polyvinylchloride (PVC), polypropylene (PP) and HDPE [10, 11]. However, there is lack of study on natural filler being incorporated into LDPE matrix. Generally, the incorporation of natural filler resulted in reduction of tensile strength [10, 11, 14] due to poor compatibility between natural filler and matrix [11]. In order to increase interfacial bonding between natural filler and matrix, different kinds of chemical modifications were employed [12, 22, 24-26]. Silane treatment proved to be effective in modifying the surface of natural filler and resulted in higher tensile strength of the composite [12, 20, 22-24].

One study which focused on the influence of polymer matrices on the mechanical and water absorption properties of EFB filled composite was conducted by Rozman et al. [10]. Several thermoplastic were used such as polystyrene (PS), polyvinylchloride (PVC), polypropylene (PP) and high density polyethylene (HDPE). The EFB used was not treated with any surface treatment. At 30 wt%, tensile strength of HDPE-EFB was 13 MPa. Generally, the incorporation of EFB reduced the flexural and tensile strength of the composite due to poor filler-matrix interaction, size irregularity and also decreased ductile deformation.

Another study on HDPE matrix was conducted by Rozman et al [11]. HDPE matrix was incorporated with oil palm frond (OPF) and EFB filler. The materials were compounded with a single-screw extruder. Based on the study, the results showed that when filler loading was increased, tensile strength was decreased gradually. The reduction in tensile strength resulted from poor interfacial bonding due to poor compatibility between polar OPF and EFB with nonpolar PE matrix. OPF and EFB were covered with polar hydroxyl groups contributed by cellulose, hemicelluloses and lignin. It was also reported that the quality of interfacial bonding was influenced by several factors, including the composition and the nature of lignocellulosic and thermoplastic materials, the fiber aspect ratio, the types of incorporation procedures, processing conditions, and the treatment done to the polymers and fibers [11]. Filler sizes did not show any significant influence on the strength of the composites. Fibers with uniform circular cross section and a certain aspect of ratio normally improved the strength.

Apart from EFB and OPF, another part of oil palm which is the trunk wood flour was also used as filler. Oil palm trunk wood flour (OPWF) was employed as filler in polypropylene (PP) composite [14]. Zaini et al.[14] has studied on the influence of the amount and size of the oil palm wood flour (OPFW) on the mechanical properties of PP-OPFW composites. The OPFW used were in the range of 63-250 μm . The OPWF was compounded into polypropylene using a Brabender thermoplastics mixer and a twin screw compounder. Test specimens of the PP/OPWF composite at 30% filler content were prepared by injection molding process. The tensile properties, flexural modulus, impact strength, and melt flow index of the PP/OPWF composite were decreased only by a small margin when compared to those of PP/talc. In conclusion, the study proved that OPWF had a good potential to be used as alternative filler for polypropylene composites.

Generally, the tensile strength of natural filler filled composite would decrease after being incorporated with natural filler due to the poor interfacial bonding between the filler and matrix. Therefore, chemical modifications [20] should be considered to treat the filler before being incorporated into polymer matrices to increase the interfacial bonding between them. These include alkali, silane, acetylation, benzylation, acrylation, maleated coupling agents, isocyanates, and permanganates [20]. In general, chemical coupling agents are molecules possessing two (2) functions. The first function is to react with hydroxyl groups of cellulose and the second is to react with functional group of the matrix [20]. Silane coupling agents were found to be effective in modifying natural filler – polymer matrix interface in order to increase interfacial strength. Different types of silane coupling agents have been reported [20]. Three amino-propyltrimethoxysilane with concentration of 1% in a solution of acetone and water (50/50 by volume) agitated for 2 hours was reported to modify flax surface. Moreover, sisal fiber was soaked in a solution of 2% aminosilane in 95% alcohol for 5 min at a pH value of 4.5 – 5.5 followed by 30 min air drying for hydrolyzing the coupling agent [20]. Silane solution in a water and ethanol mixture with concentration of 0.033% at 1% was also used to treat henequen fibers and oil palm fibers.

In addition, M.Karina et al. [12] conducted a research on the chemical treatment of EFB fiber and found that chemical treatment of EFB increased the

characteristic strength of fiber. One of the chemical treatment used was silane treatment where it was found to be effective in modifying natural fiber-polymer matrix interface and increasing the strength. From the results they obtained, the silane treatment produced higher strength compared to alkali treatment [12]. These results were similar with that of an earlier study [22] that verified the interaction between silane modified fiber and the matrix was much stronger than that of alkaline treatment, which led to composites with higher tensile strength from silane-treated than alkaline-treated fiber.

The study on the chemical treatments of EFB fiber was also conducted by Sreekala [23] and Mohd Ishak et al. [24]. The chemical treatments that Sreekala [23] used were alkali treatment, silane treatment, acrylation, acrylonitrile grafting, permanganate treatment, acetylation and peroxide treatment. The best mechanical performances were observed for silane treated and acrylated fibers. Both tensile strength of silane treated and acrylated fibers were increased by 10%. Silane and acrylation has resulted to strong covalent bond formation and thereby improved the strength of the fiber. Mohd Ishak et al. [24] also made a study on the effects of silane coupling agent and acrylation on the mechanical properties on HDPE-EFB composites. The particles used were in the range of 270-500 μm . Generally, samples with smaller particle size filler showed higher toughness as more energy is required to break samples with smaller size particle [11].

CHAPTER 3

METHODOLOGY

3.1 Materials

3.1.1 Polymer Matrix

Two (2) types of polymers were used for the purpose of this study which included HDPE and LDPE. HDPE and LDPE used were from Titan Chemicals (M) Sdn. Bhd. The properties of each polymer matrix are listed in Table 3-1 [18].

Table 3-1 Properties of polymer matrices [18]

Types Properties	LDPE	HDPE
Density (g/cm ³)	0.925	0.959
T _m (°C)	115	137
T _g (°C)	-110	-90
Tensile Strength (MPa)	8.3 – 31.4	22.1 – 31.0
Tensile Modulus (GPa)	0.17 – 0.28	1.06 – 1.09
Melt Index (g/10min)	6.0	7.0

3.1.2 Natural Filler

EFB was used as the natural filler for this study. EFB fiber was extracted by retting process of the EFB. EFB was obtained from Sabutek (M) Sdn. Bhd. in the form of particulate.

3.2 Processing of Composite Material

3.2.1 EFB Preparation

The EFB used in this study was in particulate form with the particulate size distribution of 0.325 – 0.4 mm. The EFB was first grounded using Rocklabs Type B.T.R.M. Model 1A into particulate size and then sieved into required particulate size of using Ma Test type 425 μm . To remove the moisture content, EFB was dried in a hot air oven at 80°C for 24 hours.

3.2.2 Surface Modification of Fiber

EFB was treated with 3 wt% of silane in an ethanol/water (60/40) solution for 1 hour under agitation and the pH of the solution was adjusted to 3.5-4 with acetic acid. Later, EFB was left to dry in the oven at 80°C for 24 hours. Vinyltriethoxysilane (VTS) was used for the purpose of this study. The silane was obtained from Dow Corning Corp.

3.2.3 Composite Preparation

In order to prepare the specimen, injection molding process was involved. The matrix was mixed with EFB manually and molded using injection molding machine Tat Ming ME20 III with an injection pressure of 80 bar and temperatures of 120°C (zone 1) and 180°C (zone 2 and 3) for HDPE while temperatures of 120°C (zone 1) and 130°C (zone 2 and 3) for LDPE in order to produce dog-bone shaped specimens for mechanical testing.

3.3 Characterization Testing

3.3.1 Mechanical Testing

Tensile and flexural testing were employed in order to investigate the mechanical performance of the composites. The machine used was LLOYD Instruments LR5K Universal Testing Machine according to ISO 527-2 and ISO 178, respectively. The dog-bone shaped specimens with crosshead speed of 5 mm/min was used for tensile testing while for flexural testing, specimens with dimensions of 80x10x4 mm³ and crosshead speed of 2mm/min was employed. Five (5) specimens were tested for each type of testing. In order to determine the tensile and flexural strength, Equation 3.1 and 3.2 are needed in the calculation respectively [18].

$$\sigma_t = \frac{P}{bd} \quad (3.1)$$

where σ_t = tensile strength (MPa)

P = load (N)

b = width of specimen (mm)

d = thickness of specimen (mm)

$$\sigma_f = \frac{3PL}{2bd^2} \quad (3.2)$$

where σ_f = flexural strength (MPa)

P = load (N)

L = distance between support span (mm)

b = width of specimen (mm)

d = thickness of specimen (mm)

3.3.2 Morphology Study Using Scanning Electron Microscopy (SEM)

In order to study the fiber morphology and fractured surfaces of the specimens, Oxford Leo 1430 SEM was used. The specimens were mounted on aluminum stubs and sputter-coated with a thin layer of gold to avoid electrostatic charging during examination.

3.4 Project Activities

The Gantt chart for the project can be found in Appendix 1.

CHAPTER 4

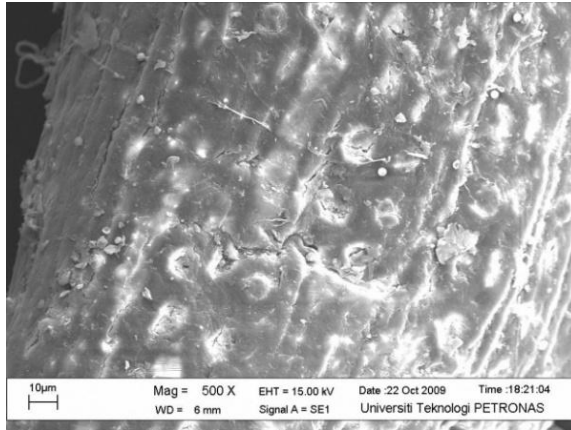
RESULTS AND DISCUSSION

This chapter is divided into two sections. The first section discusses the effects of silane treatment on the mechanical properties of the EFB filled composites. The surface morphology of untreated and treated EFB is also presented. The second section continues with the discussion of the tensile and flexural properties of HDPE-EFB and LDPE-EFB composites with EFB content varied from 0, 10, 20 to 30 wt%. The SEM micrographs of the fractured specimens are displayed.

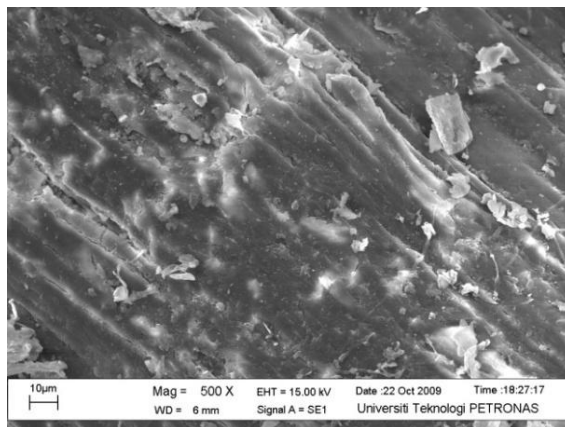
4.1 Effects of silane treatment

In order to improve the compatibility between the fiber and the matrix, chemical treatment using 3 wt% of vinyltriethoxysilane (VTS) was conducted. Fiber surface morphology was analyzed to observe the differences between untreated, dried and VTS-treated EFB.

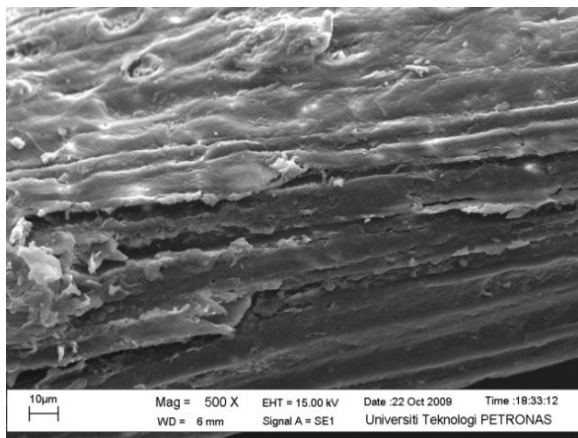
Surface morphology of EFB fiber is shown in Figure 4-1 which was observed using Scanning Electron Microscopy (SEM). Figure 4-1 (a) showed SEM micrograph of the surface of raw EFB fiber without any drying process or surface treatment. It clearly showed that the EFB fiber appeared in separated bundles with relatively smooth surface which consisted of aggregated micro-fibrils and pores. After the moisture content was removed, the surface of EFB fiber appeared slightly coarser and pores size was reduced, as shown in Figure 4-1 (b). From the SEM micrograph shown in Figure 4-1(c), it is shown that the surface of VTS treated EFB fiber appeared rough with more separation of individual fibers which indicated the removal of some cementing materials. The rough surfaces on EFB fiber after being treated are expected to support the interfacial adhesion in the composite. Surface



(a)



(b)



(c)

Figure 4-1 Scanning electron micrograph of (a) raw EFB fiber, (b) dried EFB fiber and (c) VTS treated EFB fiber

coating on VTS treated fiber revealed that the coupling agents were successfully grafted on the fiber. The coupling agents have penetrated into the micropores available on the surface of EFB and formed a mechanical interlocking coating on the surface. In addition, the micro-fibrils on the surface of the EFB fiber were slightly visible, indicating that silane coupling agent had washed out the lignin from the surface.

Treating the EFB is one way to enhance the compatibility between matrix and EFB. The mechanical properties of a composite are not only influenced by the properties of the constituent materials but the interaction between the matrix phase and reinforcing phase is also of great importance [26]. Therefore, the compatibility between polymer matrix and EFB must be enhanced to ensure good mechanical properties of the composite.

4.2 Tensile Properties of HDPE-EFB and LDPE-EFB composites

Figure 4-2 summarizes the results of the tensile strength of the HDPE-EFB and LDPE-EFB composites. The tensile strength of pure HDPE obtained was in the range of the tensile strength of HDPE (22.1 – 31.0 MPa) [18] and comparable to the tensile strength of HDPE (22 MPa) [10]. The same result was observed for pure LDPE where the tensile strength fell in the range of tensile strength of LDPE (8.3 – 31.4 MPa) [18]. However, after being incorporated with EFB, both HDPE-EFB and LDPE-EFB composites showed reduction in tensile strength by 11 and 18% at 10 wt% EFB content compared to respective pure polymer. Generally, natural filler with irregular shapes decreases the tensile strength of the resulting composites due to the inability of the filler to sustain stresses transferred from the polymer matrix [3, 10, 25]. This can be clearly seen in SEM micrograph of LDPE-EFB composites at 30 wt% EFB content as shown in Figure 4-3 where different sizes of EFB were found in LDPE-EFB composites that caused the tensile strength to decrease.

In this study, it was observed that both HDPE-EFB and LDPE-EFB composites showed a decreasing trend as the EFB content was increased from 0, 10, 20, to 30 wt%. A significant decrease (11 and 18%) can be seen as 10 wt% of EFB was incorporated into HDPE and LDPE respectively. However, the corresponding

incorporation of EFB into HDPE and LDPE matrix respectively, yielded insignificant reduction in tensile strength. Figure 4-4(a) and 4-4(b) showed the SEM micrograph of the fractured specimens of HDPE-EBF composites with 10 and 30 wt% of EFB content,

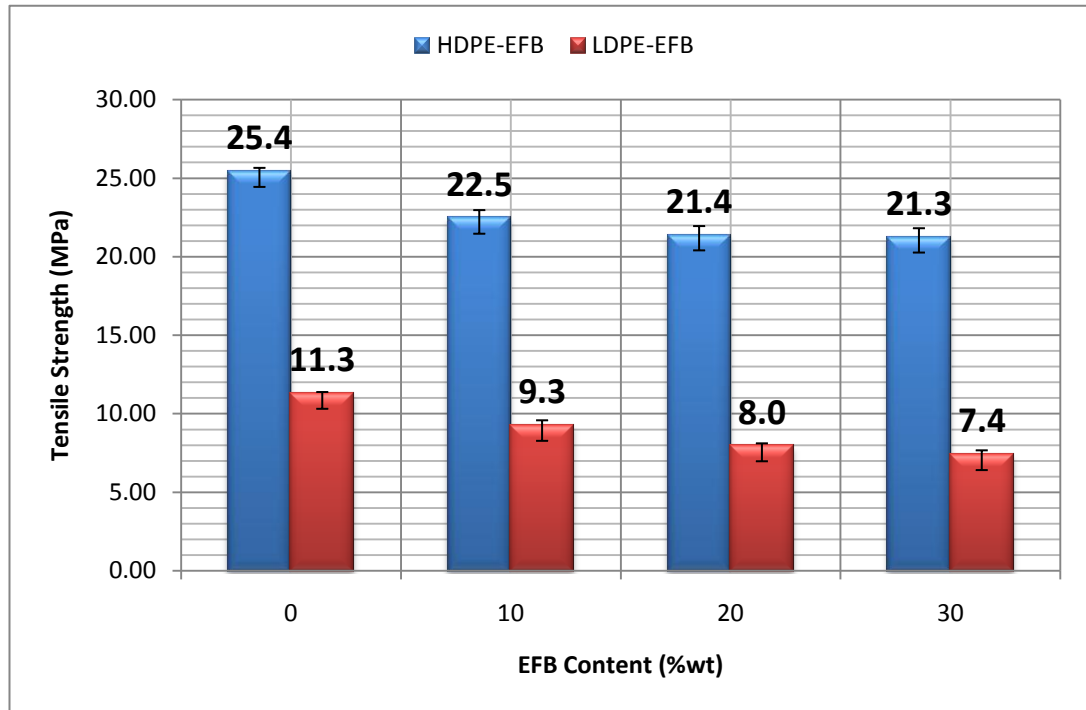


Figure 4-2 Tensile strength of HDPE-EBF and LDPE-EBF composites at varying EFB content

respectively. It can be seen that as the EFB content increases, more and larger voids appeared in the composites, which may contribute to the lower tensile strength at higher EFB content. The presence of these voids was due to the inefficient filler distribution inside the matrix during mixing process [23]. Additionally, at 30 wt% EFB content, more brittle fracture was observed, as shown in Figure 4-4 (b) when compared to a more ductile fracture at 10 wt% EFB content in Figure 4-4 (a). Apart from voids, poor wetting of EFB inside matrix can also be observed in Figure 4-4 (a) and (b). This may be due to insufficient adhesion between EFB and HDPE which contributed to the reduced tensile strength as compared to pure HDPE.

The same problems can also be seen in LDPE-EFB composites at 30 wt% EFB content as shown in Figure 4-5. The presence of voids, poor distribution of EFB and poor wetting of EFB inside LDPE matrix contributed to the fiber pull outs which indicated poor adhesion or interfacial bonding of the EFB and LDPE matrix. In addition, it can be clearly seen in Figure 4-5 that the EFB fillers remain intact which indicated that the failure does not occur through fiber breakage [10]. These factors resulted in reduced the tensile strength of the composites.

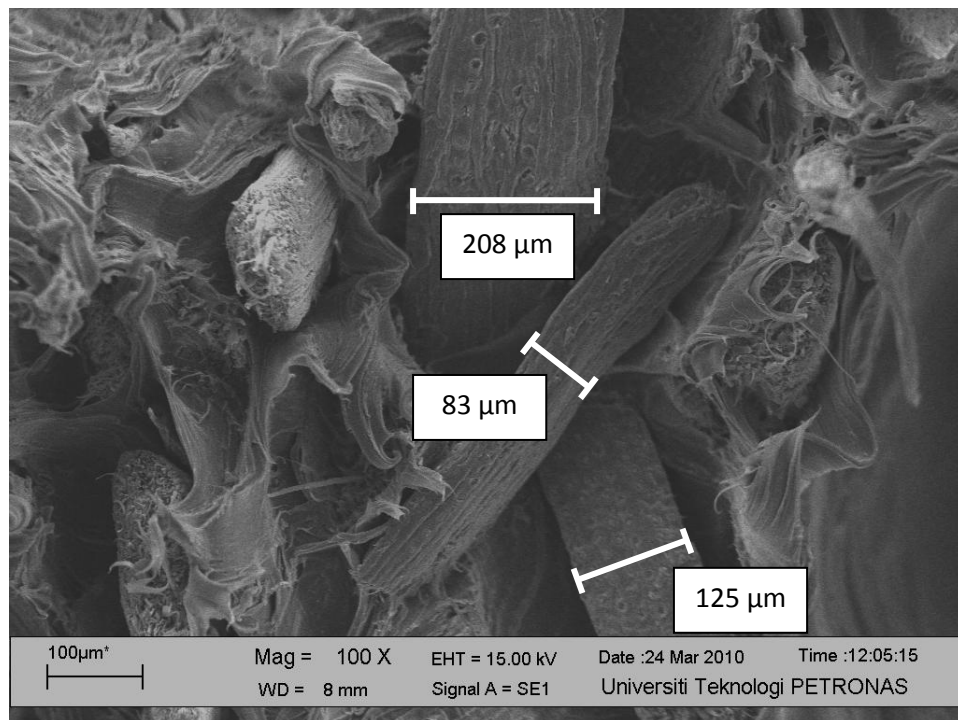
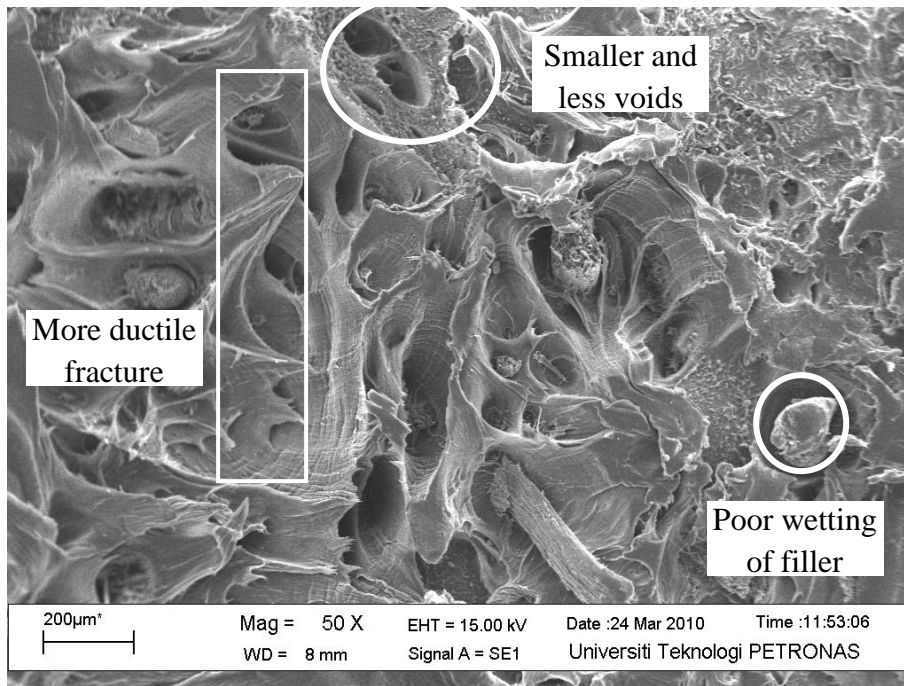
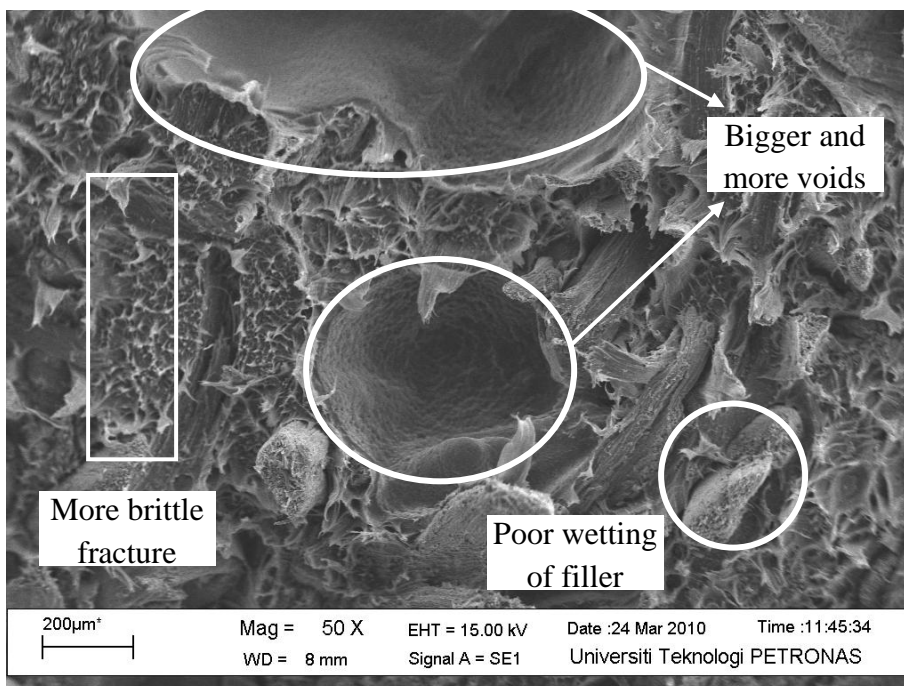


Figure 4-3 SEM micrograph of different sizes of EFB filler in LDPE-EFB composites at 30 wt% EFB content (magnification: 100 X)

Moreover, it was observed that HDPE-EFB composites result in higher tensile strength compared to LDPE-EFB composites. This was mainly due to the difference in molecular structure between HDPE and LDPE. Molecular structure characteristic such as branching plays a big role in the strength of the polymer. For instance, LDPE has a high degree of short and long chain branching. The molecules did not packed into the crystal structure which resulted in low intermolecular forces



(a)



(b)

Figure 4-4 SEM micrographs of tensile fractured surface of (a) HDPE-EFB at 10 wt% of EFB content (magnification: 50 X) and (b) HDPE-EFB at 30 wt% of EFB content (magnification: 50 X)

and subsequently lower the tensile strength [18]. As for HDPE, it has a low degree of branching, which makes the intermolecular forces stronger and higher tensile strength compared to LDPE [18].

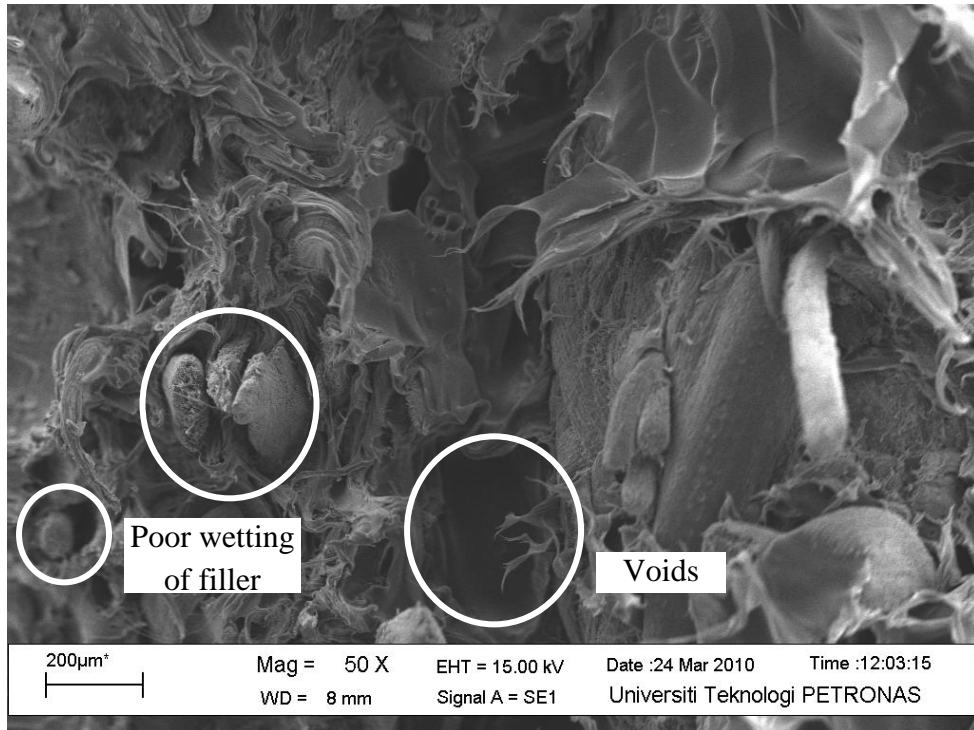


Figure 4-5 SEM micrograph of LDPE-EFB composite at 30 wt% EFB content (magnification: 50 X)

4.2 Flexural Properties of HDPE-EFB and LDPE-EFB composites

Figure 4-4 displayed the flexural strength of HDPE-EFB and LDPE-EFB composites at varying EFB content. Generally, the incorporation of EFB into matrix increased the flexural strength of the composites. The flexural strength showed an improvement as the EFB content increases from 0 to 30 wt%. The increase in flexural strength of HDPE-EFB and LDPE-EFB composites at 30 wt% EFB content was up to 22 and 24%, respectively when compared to the respective pure polymers.

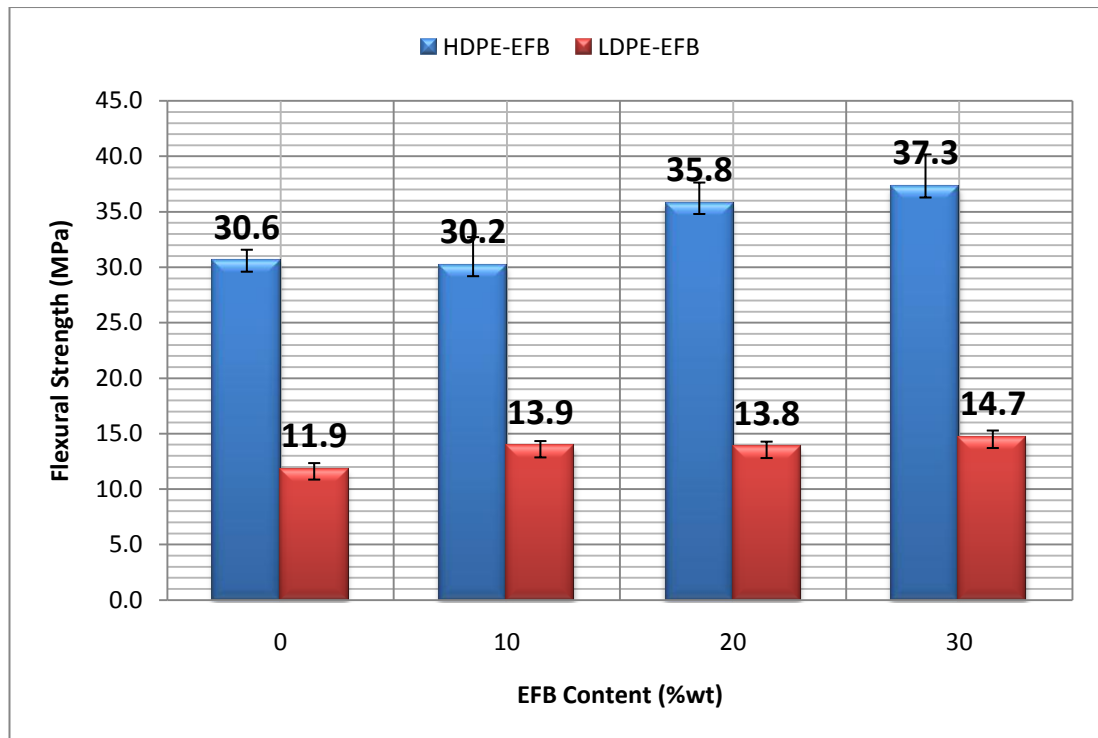
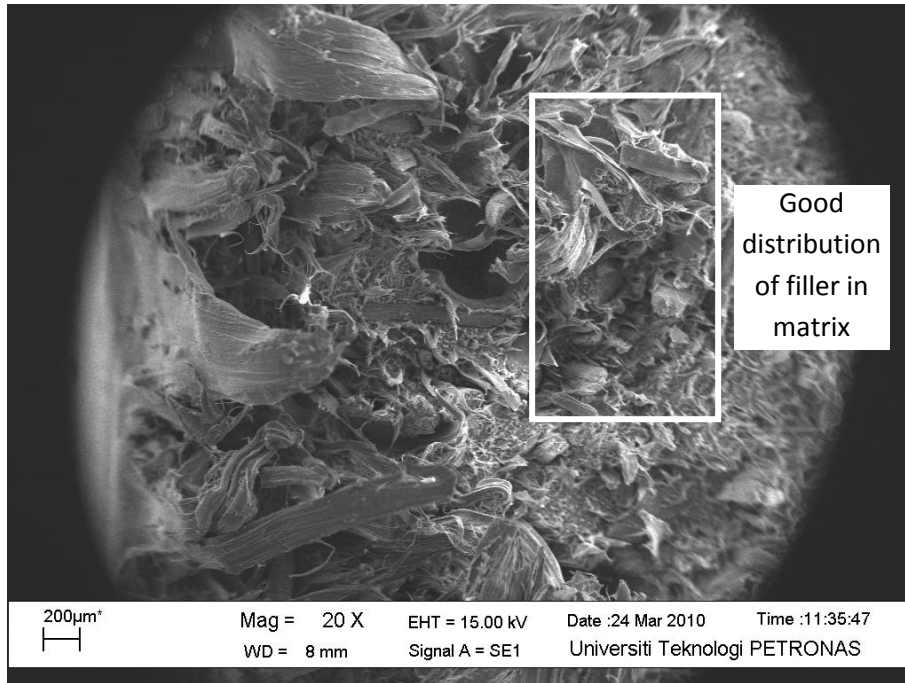
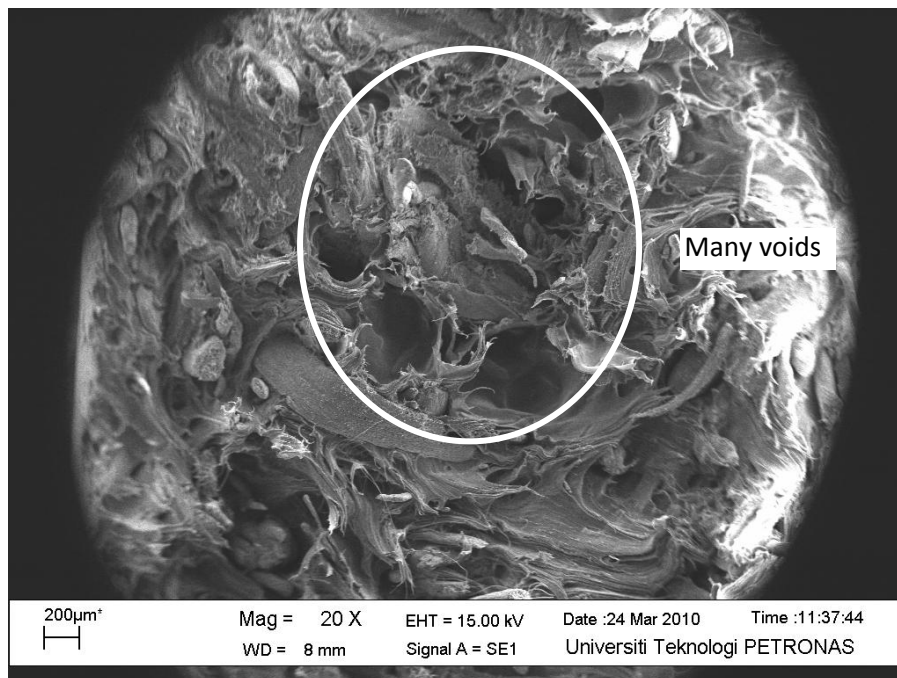


Figure 4-6 Flexural strength of HDPE-EFB and LDPE-EFB composites

However, HDPE-EFB composites showed better flexural strength than LDPE-EFB composites. This was due to the fact that the molecules in HDPE pack into crystal structure. Therefore, higher intermolecular forces between each molecule prevented them from resisting deformation longer compared to molecules in LDPE. From Figure 4-5(a) and (b), it can be seen that the distribution of EFB fibers inside HDPE matrix is better than in LDPE matrix and also, the presence of many voids can be found in LDPE-EFB composites. This showed that there was a higher fiber-matrix adhesion in HDPE-EFB composites when compared to LDPE-EFB composites. This provided a better stress transfer between EFB and HDPE which contributed to the higher flexural strength as compared to LDPE-EFB composites.



(a)



(b)

Figure 4-7 SEM micrographs of flexural fractured surfaces of (a) HDPE-EFB at 30 wt% of EFB content (magnification: 20 X) and (b) LDPE-EFB at 30 wt% of EFB content (magnification: 20 X)

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 Conclusions

The influence of the HDPE and LDPE matrix on the mechanical properties of the EFB filled composite at varying EFB content was investigated. Apart from the nature of the polymer matrix and natural fiber, the most important aspect that needs attention is the interaction between them. The compatibility between the fiber and matrix can be enhanced by chemical treatment such as silane coupling agent. The rough and more textures on the surface from the SEM micrograph revealed that the VTS coupling agent was successfully grafted on the surface of EFB. Nonetheless, the incorporation of EFB into the HDPE-EBF and LDPE-EBF composites led to reduction in tensile strength. The tensile strength for both HDPE-EBF and LDPE-EBF decreased as the EFB content was increased from 0 to 30 wt%. The poor distribution of EFB fiber inside the matrix due to poor compatibility between EFB and matrix promoted the formation of voids. However, HDPE-EBF composites results in higher tensile strength when compared to LDPE-EBF composites, which may be due to the difference in molecular structure of the matrix that influence the strength of the composites in general. In the case of flexural strength, the incorporation EFB displayed an improvement for both HDPE-EBF and LDPE-EBF composites. The flexural strength for both HDPE-EBF and LDPE-EBF increased as the EFB content was increased from 0 to 30 wt%. However, HDPE-EBF composites showed better flexural strength than LDPE-EBF composites.

5.2 Recommendations

From the study that has been done, it can be concluded that the EFB has yet to fully show its potential to reinforce in order to improve the mechanical properties of the

EFB filled composites. The incorporation of EFB into HDPE and LDPE composites had led to reduction in tensile strength. This is mainly due to the irregular shapes of EFB and due to inability of EFB to sustain stresses transferred from the polymer matrix. Therefore, perhaps a better way of sieving the EFB should be looked into in order to obtain a uniform size of the filler before being incorporated into the composite.

Moreover, from SEM micrograph displayed in Figure 4-3, bigger and more voids were observed at a higher EFB content, mainly due to poor dispersion of filler inside the matrix during mixing process. In this project, the compounding process of the filler and matrix was done inside an injection molding machine. Therefore, consistency is a problem because mixing was done manually and at higher temperatures, EFB fillers tend to degrade and unable to properly compound with the matrix. An extruder process should be considered before injection molding process to ensure better distribution of filler inside the matrix.

Based on this project, silane coupling agent was used to modify the surface of the EFB in order to increase the interfacial bonding between the filler and matrix. However, as shown from Figure 4-3, poor wetting of filler inside the matrix still can be observed. Perhaps, different kinds of surface treatments should be considered in order to find the most effective treatments to treat the natural fillers.

However, the flexural strength of both HDPE-EFB and LDPE-EFB composites showed promising results as the flexural strength improved when EFB content was increased from 0 to 30 wt%. For future work, higher EFB content should be considered in order to determine the optimum content of EFB that can be incorporated into the matrix to optimize the flexural strength of the composite.

REFERENCES

- [1] T. Peijs, H.G.H. Van Melick, S.K. Garkhail. G.T. Pott, A. Stamboulis, and C.A. Baillie, 1998, "Natural-Fiber-Mat-Reinforced Thermoplastics Based on Upgraded Flax Fibers for Improved Moisture Resistance," *Proceedings of European Conference on Composite Materials*, **2**: 119
- [2] G. Bogoeva-Gaceva, A. Dekanski, V. Panić, D. Poleti, A. Grozdanov, A. Bužarovska, M. Avella, and G. Gentile, 2006, "Natural Fibers in Polymer Composite Materials", *XLIV Meeting of the Serbian Chemical Society*, Belgrade
- [3] Nurulhuda Muhammad Ali, 2008, *The tensile properties of pineapple leaf fiber as polymer composite materials*, B.A.(Hons.) Thesis, Universiti Teknologi Malaysia, Malaysia
- [4] Singha, A.S. and Thakur, V.K., 2008, "Mechanical properties of natural fibre reinforced polymer composite", *Bull. Mater. Sci* **31** (5): 791-799
- [5] Bledzki, A.K. and Gassan, J., 1999, "Composites reinforced with cellulose fibres", *Prog Polym Sci* **24**: 74-221
- [6] Seena J., Maya J., and Sabu T., 2005, "Natural fiber-rubber composites and their applications", *Natural Fibers, Biopolymers, and Biocomposites* **13**: 435-472
- [7] Rozman, H.D., Z.A.M. Ishak, and U.S. Ishiaku, 2005, "Oil Palm Fiber - Thermoplastic Composites", *Natural Fibers, Biopolymers, and Biocomposites*, **12**: 407-434
- [8] Sabah Government, 2008, [Table illustration the world producers of palm oil], *Oil Palm Tree*, Retrieved August 8, 2009, from

<http://www.etawau.com/HTML/OilPalm/OilPalm.htm>.

- [9] S.H.A. Rahman, J.P. Choudhury, A.L. Ahmad, and A.H. Kamaruddin, 2007, "Optimization studies on acid hydrolysis of oil palm empty fruit bunch fiber for production of xylose", *Biores. Technol.* **98**: 554–559
- [10] Rozman H.D., Lim P.P., Abusamah A., Kumar R.N., Ismail H., and Mohd Ishak Z.A., 1999, "The physical properties of oil palm empty fruit bunch (EFB) composite made from various thermoplastics", *Int J Polym Matter* **44**: 95-179
- [11] Rozman, H.D., Z.A.M. Ishak, and U.S. Ishiaku, 2005, "Oil Palm Fiber - Thermoplastic Composites", *Natural Fibers, Biopolymers, and Biocomposites*, **12**: 407-434
- [12] M. Karina, H. Onggo, A.H. Dawam Abdullah and A. Syampurwadi, 2009, "Effect of Surface Treatment and Gauge Length of Empty Fruit Bunch on its Morphological and Characteristic Strength Properties", *Asian J. Mater. Sci.*, **1**: 7098
- [13] Gatenholm, P. and Felix, J., 1993, *Wood fiber/polymer composites: fundamental concepts, process and material options*, Madison, Forest Product Society, **2**: 87-88
- [14] M. J. Zaini, Z. Ismail, M. Y. A. Fuad and J. Mustafah, 1994, "Application of Oil Palm Wood Flour as Fillers in Polypropylene", *Polym. J.*, **26**: 637-642
- [15] M. Khalid, C.T. Ratnam, Chuah, T.G., Salmiaton Ali, and Choong, Thomas S.Y., 2008, "Comparative study of polypropylene composites reinforced with oil palm empty fruit bunch fiber and oil palm derived cellulose", *Materials and Design* **29**: 173–178
- [16] Mohanty, A.K., Misra, M., Drzal, L.T., Selke, S.E., Harte, B.r., and Hinrichsen, G., 2005, "Natural Fibers, Biopolymers, and Biocomposites: An Introduction", *Natural Fibers, Biopolymers, and Biocomposites*, **1**: 1 – 36
- [17] O'Donnell, A., Dweib, M.A., and Wool, R.P., 2004, "Natural fiber composites with plant oil-based resin", *Composites Science and Technology*, **64**: 1135 – 1145

- [18] Callister, W.D, 2007, "Materials science and engineering: an introduction", New York, John Wiley & Sons, Inc., 489-515, 736
- [19] Fried, J.R., 2003, "Polymer Science & Technology", New Jersey, Pearson Education, Inc., 233-235
- [20] Xue Li, Tabil, L.G., and Panigrahi, S., 2007, "Chemical treatments of natural fiber for use in natural fiber-reinforced composites: a review", *J Polym Environ*, **15**: 25-33
- [21] Abdelmouleh, M., Boufi, S., Belgacem, M.N., and Dufresne, A., 2007, "Short natural-fiber reinforced polyethylene and natural rubber composites: Effect of silane coupling agents and fibers loading", *Comp Sci and Techno*, **67**: 1627 - 1639
- [22] P.J. Herrera-Franco and A. Valadez-Gonzales, 2004, "Mechanical properties of continuous natural fibre-reinforced polymer composites", *Composites Part A: Applied Science and Manufacturing*, **35(3)**: 339–345
- [23] M. S. Sreekala, and S. Thomas, 2003, "Effect of fibre surface modification on water-sorption characteristics of oil palm fibres", *Composites Science and Technology*, **63(6)**: 861-869
- [24] Mohd Ishak, Z.A., Aminullah, A., Ismail, H. and Rozman, H.D., 1998, "The Effect of Silane Based Coupling Agents and Acrylic Acid Based Compatibilisers in Mechanical Properties of Oil Palm Empty Fruit Bunch Filled High Density Polyethylene Composites", *J. Appl. Polym. Sci.*, **68**: 2189
- [25] Rozman, H.D., Ismail, H., Jaffri, R.M., Aminullah, A., and Ishak, Z.A. Mohd., 1998, Mechanical Properties of Polyethylene-Oil Palm Empty Fruit Bunch Composites, *Polymer-Plastics Technology and Engineering*, **37(4)**: 495-507
- [26] Abrate, S., 2002, "World Wide Web Resource for Composite Materials", *Proceedings of the 17th technical conference*, West Lafayette, American Society for Composite.

APPENDICES

APPENDIX I : Project Activities

Tasks	2009												2010											
	August			September			October			November			February			March			April			May		
Progress Report	█	█	█	█	█	█																		
Seminar				█	█	█																		
Interim Final Report				█	█	█	█	█	█															
Oral Presentation 1										█	█	█												
Sample Preparation													█	█										
Silane Treatment														█	█									
Composite Preparation																								
Extrusion																█	█							
Injection Molding																			█	█				
Mechanical Testing																								
Tensile																			█	█				
Flexural																				█	█			
Analysis of Samples																								
SEM																						█	█	
Final Report													█	█	█	█	█	█	█	█	█	█	█	█
Oral Presentation 2																							█	█

APPENDIX II : TENSILE TESTING SPECIFICATIONS AND RESULTS FOR HDPE-EFB COMPOSITES

Tensile properties for HDPE-EFB composites (10 wt% EFB)							
No.	Sample	Weight(g)	Speed(mm/min)	Gauge length (mm)	Maximum Load (N)	Tensile Strength (MPa)	Tensile Modulus (MPa)
1	B1	12.42	5	50	894.08	22.35	1250
2	B2	12.47	5	50	893.38	22.33	1187.5
3	B5	12.37	5	50	929.81	23.25	1200
4	B6	12.40	5	50	874.09	21.85	1312.5
5	B7	12.36	5	50	901.15	22.53	1250
					Mean	22.46	1250
					Standard Deviation	0.50489	50,621

Tensile properties for HDPE-EFB composites (20 wt% EFB)							
No.	Sample	Weight(g)	Speed(mm/min)	Gauge length (mm)	Maximum Load (N)	Tensile Strength (MPa)	Tensile Modulus (MPa)
1	C1	12.60	5	50	887.14	22.18	937.5
2	C2	12.54	5	50	867.16	21.68	900
3	C4	12.62	5	50	830.58	20.76	1150
4	C5	12.57	5	50	851.80	21.30	700
5	C6	12.57	5	50	843.49	21.09	750
					Mean	21.40	887.5
					Standard Deviation	0.54704	177,218

Tensile properties for HDPE-EFB composites (30 wt% EFB)							
No.	Sample	Weight(g)	Speed(mm/min)	Gauge length (mm)	Maximum Load (N)	Tensile Strength (MPa)	Tensile Modulus (MPa)
1	D5	12.99	5	50	842.48	21.06	1600
2	D6	13.11	5	50	860.21	21.51	1312.5
3	D9	13.13	5	50	880.69	22.01	1166.7
4	D10	12.84	5	50	847.42	21.19	937.5
5	D11	13.15	5	50	821.12	20.53	1357.1
					Mean	21.26	1274.76
					Standard Deviation	0.54936	244,646

Tensile properties for HDPE							
No.	Sample	Weight(g)	Speed(mm/min)	Gauge length (mm)	Maximum Load (N)	Tensile Strength (MPa)	Tensile Modulus (MPa)
1	A1	12.17	50	50	1008.32	25.21	1875
2	A7	12.15	50	50	1028.64	25.72	1750
3	A11	12.20	50	50	1015.07	25.38	1583.3
4	A12	12.09	50	50	1011.75	25.29	1625
5	A15	12.12	50	50	1023.99	25.60	1500
					Mean	25.44	1666.66
					Standard Deviation	0.21261	147,319

APPENDIX II : TENSILE TESTING SPECIFICATIONS AND RESULTS FOR LDPE-EFB COMPOSITES

Tensile properties for LDPE-EFB composites (10 wt% EFB)							
No.	Sample	Weight(g)	Speed(mm/min)	Gauge length (mm)	Maximum Load (N)	Tensile Strength (MPa)	Tensile Modulus (MPa)
1	E2	12.56	5	50	375.84	9,396	106.7
2	E3	12.53	5	50	363.55	9,089	120
3	E4	12.47	5	50	380.75	9,519	125
4	E7	12.46	5	50	352.46	8,812	140
5	E8	12.54	5	50	380.69	9,517	130
					Mean	9,267	124.34
					Standard Deviation	0.30877	12,326

Tensile properties for LDPE-EFB composites (20 wt% EFB)							
No.	Sample	Weight(g)	Speed(mm/min)	Gauge length (mm)	Maximum Load (N)	Tensile Strength (MPa)	Tensile Modulus (MPa)
1	G4	13.01	5	50	324.39	8,110	180
2	G6	13.3	5	50	319.39	7,992	253.3
3	G11	13.22	5	50	323.66	8,092	240
4	G10	13.20	5	50	312.75	7,819	320
5	G13	13.33	5	50	312.93	7,823	250
					Mean	7,967	245.5
					Standard Deviation	0.14065	45,149

Tensile properties for LDPE-EFB composites (30 wt% EFB)							
No.	Sample	Weight(g)	Speed(mm/min)	Gauge length (mm)	Maximum Load (N)	Tensile Strength (MPa)	Tensile Modulus (MPa)
1	G1	14.03	5	50	286.26	7,157	366.7
2	G2	14.11	5	50	308.24	7,706	650
3	G4	13.80	5	50	291.35	7,284	280
4	G6	14.14	5	50	289.28	7,232	575
5	G7	14.11	5	50	306.44	7,661	550
					Mean	7,408	484.34
					Standard Deviation	0.25601	154,570

Tensile properties for LDPE							
No.	Sample	Weight(g)	Speed(mm/min)	Gauge length (mm)	Maximum Load (N)	Tensile Strength (MPa)	Tensile Modulus (MPa)
1	H8	12.33	50	50	454.31	11,358	400
2	H10	12.35	50	50	452.56	11,314	373.33
3	H12	12.33	50	50	451.72	11,293	254.55
4	H16	12.35	50	50	447.64	11,191	200
5	H19	12.32	50	50	455.71	11,393	333.33
					Mean	11,310	312.24
					Standard Deviation	0.06879	74,572

APPENDIX IV: FLEXURAL TESTING SPECIFICATIONS AND RESULTS FOR HDPE-EFB COMPOSITES

Flexural properties for HDPE-EFB composites (10 wt% EFB)								
No.	Sample	Weight(g)	Width, b(mm)	Depth,d (mm)	Length, l (mm)	Speed(mm/min)	Maximum Load (N)	Flexural Strength (MPa)
1	B12	12.37	10.1	4.32	77.50	2	108.38	34.50
2	B17	12.50	10.1	4.34	77.50	2	90.22	28.46
3	B18	12.49	10.1	4.30	77.46	2	89.37	28.71
4	B19	12.57	10.1	4.30	77.40	2	90.02	28.92
5	B20	12.66	10.1	4.32	76.96	2	95.47	30.39
							Mean	30.20
							Standard Deviation	2.52

Flexural properties for HDPE-EFB composites (20 wt% EFB)								
No.	Sample	Weight(g)	Width, b(mm)	Depth,d (mm)	Length, l (mm)	Speed(mm/min)	Maximum Load (N)	Flexural Strength (MPa)
1	C3	12.43	10.1	4.30	78.14	2	102.04	32.78
2	C7	12.55	10.1	4.32	77.12	2	112.68	35.89
3	C8	12.50	10.14	4.30	77.66	2	112.99	36.16
4	C11	12.86	10.2	4.32	78.32	2	119.78	37.75
5	C12	12.66	10.16	4.32	77.32	2	115.19	36,449
							Mean	35,803
							Standard Deviation	1,835

Flexural properties for HDPE-EFB composites (30 wt% EFB)								
No.	Sample	Weight(g)	Width, b(mm)	Depth,d (mm)	Length, l (mm)	Speed(mm/min)	Maximum Load (N)	Flexural Strength (MPa)
1	D8	13.30	10.1	4.30	79.12	2	114.71	36.86
2	D12	12.71	10.1	4.30	76.68	2	112.67	36.20
3	D13	13.33	10.2	4.32	77.66	2	126.03	39.72
4	D15	13.76	10.18	4.30	77.66	2	126.77	40.41
5	D16	11.94	10.1	4.28	76.48	2	102.55	33.26
							Mean	37.29
							Standard Deviation	2,884

Flexural properties for HDPE								
No.	Sample	Weight(g)	Width, b(mm)	Depth,d (mm)	Length, l (mm)	Speed(mm/min)	Maximum Load (N)	Flexural Strength (MPa)
1	A3	12.20	10.1	4.24	78.14	2	91.55	30.25
2	A5	12.12	10.08	4.26	78.12	2	92.20	30.24
3	A6	12.11	10.04	4.26	77.80	2	95.11	31.32
4	A10	12.15	10.14	4.22	77.84	2	95.79	31.83
5	A11	12.15	10.16	4.28	79.10	2	91.04	29.35
							Mean	30,599
							Standard Deviation	0.981

APPENDIX V : FLEXURAL TESTING SPECIFICATIONS AND RESULTS FOR LDPE-EFB COMPOSITES

Flexural properties for LDPE-EFB composites (10 wt% EFB)								
No.	Sample	Weight(g)	Width, b(mm)	Depth,d (mm)	Length, l (mm)	Speed(mm/min)	Maximum Load (N)	Flexural Strength (MPa)
1	E1	12.64	10.14	4.32	78.04	2	43.54	13.81
2	E5	12.57	10.10	4.32	78.60	2	44.19	14.07
3	E9	12.51	10.14	4.30	77.40	2	40.81	13.06
4	E10	12.53	10.20	4.30	77.26	2	44.69	14.22
5	E11	12.50	10.10	4.30	78.10	2	44.00	14.14
							Mean	13.86
							Standard Deviation	0.47279

Flexural properties for LDPE-EFB composites (20 wt% EFB)								
No.	Sample	Weight(g)	Width, b(mm)	Depth,d (mm)	Length, l (mm)	Speed(mm/min)	Maximum Load (N)	Flexural Strength (MPa)
1	G3	12.95	10.16	4.32	78.20	2	42.63	13.49
2	G8	12.91	10.20	4.30	78.52	2	41.88	13.32
3	G9	12.97	10.10	4.32	78.10	2	44.00	14.00
4	G11	12.96	10.16	4.32	78.20	2	45.90	14.52
5	G12	13.09	10.10	4.30	77.60	2	42.51	13.66
							Mean	13.80
							Standard Deviation	0.477

Flexural properties for LDPE-EFB composites (30 wt% EFB)								
No.	Sample	Weight(g)	Width, b(mm)	Depth,d (mm)	Length, l (mm)	Speed(mm/min)	Maximum Load (N)	Flexural Strength (MPa)
1	F8	14.06	10.20	4.26	77.00	2	45.69	14.81
2	F10	13.79	10.26	4.32	77.46	2	33.10	10.48
3	F11	13.58	10.20	4.28	77.66	2	43.84	14.08
4	F14	13.68	10.10	4.32	76.58	2	30.65	9.85
5	F15	13.68	10.20	4.26	76.68	2	46.95	15.22
							Mean	14,703
							Standard Deviation	0.5775

Flexural properties for LDPE								
No.	Sample	Weight(g)	Width, b(mm)	Depth,d (mm)	Length, l (mm)	Speed(mm/min)	Maximum Load (N)	Flexural Strength (MPa)
1	H1	12.32	10.2	4.32	78.20	2	38.60	12.17
2	H3	12.27	10.24	4.32	77.90	2	37.25	11.70
3	H4	12.32	10.24	4.32	78.10	2	35.27	11.07
4	H5	12.32	10.24	4.32	77.60	2	39.12	12.28
5	H6	12.33	10.2	4.34	78.40	2	38.62	12.06
							Mean	11.86
							Standard Deviation	0.489