A Study On Mechanical And Thermal Properties Of High Density Polyethylene (HDPE) Glass Fiber Composites

by

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14327

Dissertation submitted in partial fulfillment of
the requirements for the
Bachelor of Engineering (Hons)
(Chemical)

SEPTEMBER 2014

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CERTIFICATION OF APPROVAL

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(CHEMICAL)

Approved by,

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(Prof. Dr. Khairun Azizi bt Mohd. Azizli)

UNIVERSITI TEKNOLOGI PETRONAS
TRONOH, PERAK
September 2014

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.

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(NUR FATIN FILZATI BINTI MAHMUD)

ACKNOWLEDGEMENTS

In the name of Allah, the Most Gracious and the Most Merciful. Praise Him the Almighty that in His will and given strength, the final year project is successfully completed within the allocated eight months period. Upon completing the project undertake, I owed a great thanks to a great many people for their help and support, as well as their contribution in time, effort, advice, supervision, discussion and help during the entire period.

First and foremost, deepest appreciation to Universiti Teknologi PETRONAS especially for Chemical Engineering Department for giving me the opportunity to experience the challenging Final Year Project (FYP) course. Special thanks to my beloved and helpful supervisor, Prof Dr. Khairun Azizi bt Mohd.Azizli. The supervision and support that she gave truly helped the progression and smoothness of my project. The co-operation is much indeed appreciated. Thank you, Madam.

My grateful appreciation also goes to all the technicians and post graduate students from Chemical Engineering and Mechanical Engineering Department who always give me advice and support throughout the experiments in the laboratory. Not to forget, a deep sense of gratitude to my friends and willingness to help me in several tasks that I cannot perform alone and for all the support. To my late mother and family members, you all are my spirit that have kept me going my best.

Last but not least, endless thanks to those who helped me to finish up my project directly or indirectly for their guidance, sharing and support throughout the entire two semesters. Their help are much appreciated.

ABSTRACT

The effects of glass fiber addition on mechanical and thermal properties of high density polyethylene (HDPE) glass fiber composites were studied. HDPE pellets were obtained from PT Lotte Chemical Titan along with chopped strand glass fiber mat, and the synthesis of neat samples and composites were carried out following the mix formulation of 10% wt., 15% wt. and 20% wt. of glass fibers. The properties of the synthesized samples were studied by characterization analysis, mechanical testing and thermal decompostition analysis. It was found that the 20% wt. HDPE glass fiber composites obtained the highest value for tensile strength and modulus, which were 38.83 N/mm^2 and 5000000 kN/m^{2,}, respectively. However, the percentage of elongation portrays an inverse pattern, with the 20% HDPE glass fiber composites only showing an elongation of 13.7%, compared to neat HDPE with elongation of 46.3%. Furthermore, the decomposition temperature of 20% wt. HDPE glass fiber composites marked the highest, which was 435°C. Based on SEM analysis, the differences in microstructure for different ranges of glass fibers used were observed, along with the disintegration of glass fibers that occured at samples' failure points.

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CHAPTER 1

INTRODUCTION

1.1 Background of Study

The discovery of composite materials has brought numerous contributions towards the development of industries; namely the aircraft industry which seeks for lighter and more durable aircraft components, structural industry through high-stress tolerant construction materials for road bridges, buildings and plumbing components, and also benefits for the massive oil and gas industry throughout the world. Following World War II, the natural gas industry grew at a very aggressive rate. The 40-year period between 1948 and 1999 saw about 570,000 miles of transmission pipeline capability being built. According to Mohipour et. al (2001), as of 1998 there were more than 532,000 miles of natural gas pipelines, with almost half located in North America.

In this era, technologists and engineers are expanding their researches based on the improvement of available resources in order to meet the recent estimate which requires the assembly of 43,000 miles of pipeline additions to satisfy an expanding market by the year 2015. The challenge to the pipeline industry is to meet the increased worldwide demand while reducing the cost. The US Energy Information Administration (n.d.) predicts that the price of natural gas on the average will be in the \$3 to \$4 range through 2020. In order to meet the delivery demands and overcome the flat price, the pipeline industry will have to come up with innovative methods to transport natural gas from wellhead to household and commercial users.

Experts in the industry have all agreed that future transmission pipelines will have to operate at higher pressure hence opens up to alternatives other than the conventional steel pipe, which have lead to the application of Composite Reinforced Line Pipe (CRLP). The use of steel pipe with a composite overwrap together make a new type of pipe that has exceptional strength characteristics with positive advantages in weight and corrosion control.(Mohipour et al., 2001) The next engineering step in the transmission sector is to go to total composite pipe for natural gas transmission.

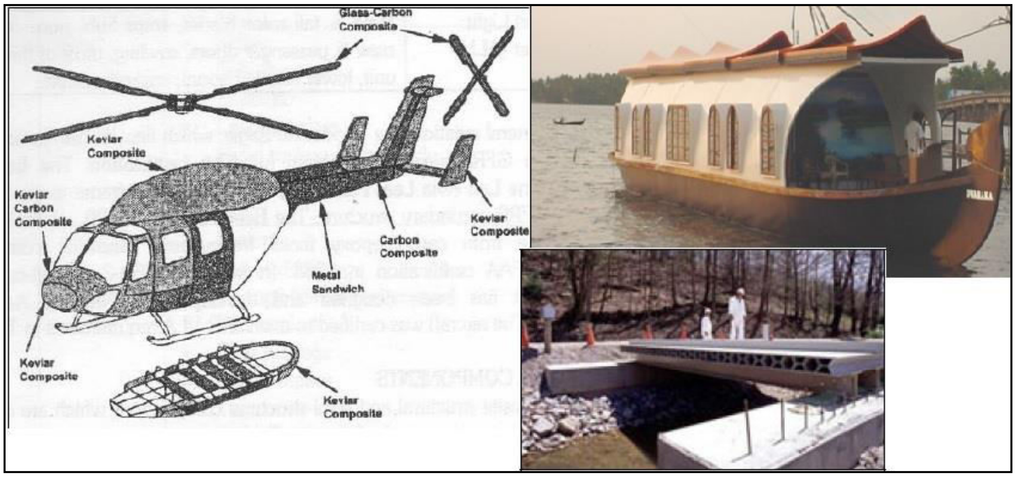


Figure 1.1: Composite materials in various industries (Mohipour et al., 2001)

Composites are one of the most widely used materials because of their adaptability to different situations and the relative ease of combination with other materials to serve specific purposes and exhibit desirable properties, as shown in Figure 1.1. This combination produces a material that is further improved compared to their respective building blocks (Taranu et al., 2012).

In this paper, the material being highlighted is high density polyethylene (HDPE), a kind of polyethylene thermoplastic sourced from petroleum. It is widely used in the production of plastic bottles and non-corrosive piping system. In this study, fiber glass composite has been chosen as the component to be added to HDPE hence producing the HDPE fiber glass composite. The mechanical and thermal properties of this material will be studied and discussed in further chapters.

1.2 Problem Statement

Polyethylene thermoplastics are already widely recognized in various industries such as automotive, household and food packaging. Although HDPE already possesses long linear chains with high crystallinity and more rigidity which contributes towards its robustness, the polymer can further be added with reinforcements to obtain the desired properties. According to Arostegui et al. (2001), it is known that the robustness of polymers can be greatly improved thorugh the mixing with a rubber or elastomer. However, the effects that have been studied included the reduction in thermoplastic stiffness, thermal stability, which further limiting its application in the industry. As a result, some researches in recent years have studied the impact of rigid particles addition towards the toughening of thermoplastics, as reported by Huang et al. (2013) and others, in Table 1.1.

Table 1.1: Previous Studies Conducted for Reinforced Polyethylene Thermoplastics

Previous Studies	Researchers (year)
High Density Polyethylene Composites Reinforced with Hybrid Inorganic Fillers: Morphology, mechanical and thermal expansion performance.	Huang et al. (2013)
The use of glass fiber reinforced polymer composites as reinforcement for tubular concrete poles.	Taranu et al. (2009)
Mechanical and thermal properties of High Density Polyethylene toughened with glass beads.	Yuan et al. (2002)

Alternatively, with the property of glass fiber itself being a reliable reinforcing material with a dominant factor in the resistivity against corrosive environment, high temperature durability and stability, its effect towards the improvement of HDPE is yet to be studied, specifically in terms of mechanical and thermal properties of HDPE fiber glass composite, for its further use in the oil and gas piping industry.

1.3 Objectives of Study

This research is to be carried out based on the stated objectives:

i) To synthesize HDPE glass fiber composites based on the required mixing formulations.

ii) To study the effect of glass fiber addition to the mechanical and thermal properties of HDPE glass fiber composites.

iii) To observe the morphology and characterize the HDPE glass fiber composites.

1.4 Scope of Study

The scope of this study includes the comparison of HDPE glass fiber composite with regard to its pure component properties. In the sample preparation process, neat HDPE samples are produced along with HDPE glass fiber composites, with mix formulations of 10% wt., 15% wt. and 20% wt. of glass fiber. In order to illustrate the effect of glass fiber addition towards the properties of the composites, mechanical tests are conducted to study the tensile and elongation properties, whereas thermal tests were done to detemine the heating and decomposition effect, as well as SEM analysis for more understanding regarding the morphology of the composite. By completing the tests and analysis of the results, a definitive understanding of the properties were obtained through further correlation.

CHAPTER 2

LITERATURE REVIEW

2.1 Glass fiber

Glass fiber, also known as glass reinforced plastic (GRP) , is a material made from extremely fine fibers of glass, as shown in Figure 2.1. Raw materials such as silicates, soda, clay, limestone, boric acid, fluorspar or various metallic oxides are blended to form a glass batch which is melted in a furnace and refined during lateral flow to the fore hearth. They are manufactured in a wide range of fine diameters. Some of them are so fine that they can be seen only through a microscope. Hence various manufacturers produce types of glass fibers for different end uses; home furnishing fabrics, apparels and garments, and purpose tires and reinforced plastics.

Figure 2.1 Glass fiber

As reported by Hartman, D. et al. (2006), currently there exists various glass compositions which were developed with the aim to provide combinations of fiber properties directed at specific end use applications - namely A glass, C glass, D glass, E glass, ECR glass, AR glass, R glass, and S-2 glass as shown in Table 2.1:

Table 2.1: Type of glass based on composition (Hartman, D. et al., 2006)

Type of glass	Description
A GLASS	Soda lime silicate glasses used where the strength, durability, and good electrical resistivity of E GLASS are not required.
C GLASS	Calcium borosilicate glasses used for their chemical stability in corrosive acid environments.
D GLASS	Borosilicate glasses with a low dielectric constant for electrical appliances.
E GLASS	Alumina-calcium-borosilicate glasses with maximum alkali content of 2 wt. % used as general purpose fibers where strength and high electrical resistivity are required.
ECR GLASS	Calcium aluminosilicate glasses with a maximum alkali content of 2 wt. % used as general purpose fibers where strength and high electrical resistivity are required.
AR GLASS	Alkali resistant glasses composed of alkali zirconium silicates used in cement substrates and concrete.
R GLASS	Calcium aluminosilicate glasses used for reinforcement where added strength and acid corrosion resistance are required.
S-2 GLASS	Magnesium aluminosilicate glasses used for textile substrates or reinforcement in composite structure applications which require high strength, modulus and stability under extreme temperature and corrosive environments.

However, the most common glass fibers are made of E glass and S glass since the former is the least expensive of all glass types and has a wide application whereby the latter is reported to attain higher tensile strength and modulus (Taranu, N. et al., 2012) The main properties of these two glasses are summarized in a table (refer **Appendix 1**).

2.1.1 Mechanical properties

According to AlMadeed et al. (2012), glass fibers are applied as reinforcements (or fillers) to plastics due to their low cost and fairly good mechanical properties compared to synthetic fibers. However, the mechanical properties of glass fiber depend heavily on the sizing system applied to the glass fiber surface. They are useful because of their high ratio of surface area to weight. However, the increased surface area makes them much more susceptible to chemical attack. Because glass has an amorphous structure, its properties are the same along the fiber and across the fiber. The freshest, thinnest fibers are the strongest since thinner fibers are more ductile compared to others (Textile Learner, n.d.). In general, glass fiber possess these useful bulk properties which are hardness, transparency, resistance to chemical attack, stability and inertness, as well as strength, flexibility and stiffness. Tensile strength of glass fibers is usually reported as the pristine single-filament or the multifilament strand measured in air at room temperatures (Hartman , D. et al., 2006), as shown in Table 2.2:

Table 2.2: Physical/Mechanical properties of glass fiber

(Hartman, D. et al, 2006)

Characteristics/Fiber	E-glass	S-glass
Density (kg/m^3)	2500	2500
Tensile strength (MPa)	3450	4580
Young Modulus (GPa)	72.4	85.5
Ultimate Tensile Strain (%)	2.4	3.3
Thermal exp. Coefficient (10^{-6}/°C)	5	2.9
Poission's coefficient	0.22	0.22

2.1.2 Thermal properties

The viscosity of glass decreases as the temperature increases. As shown in **Appendix 1**, several reference viscosity points are defined by the glass industry, and the softening point is the temperature at which as glass fiber of uniform diameter elongates at a specific rate under its own weight when when measured. Hence the softening point is defined as the temperature at which glass will deform at its own weight by ASTM C 338 (American Society for Testing and Materials), and as reported by Hartman, D. et al. (2006), it occurs at a viscosity of approximately $10^{6.6}$ Pa.s ($10^{7.6}$P).

At the annealing point of glass, internal stresses are substantially relieved in the matter of minutes whereby at the strain point of glass, internal stresses are substantially relieved in a matter of hours. Thermal conductivity characteristics in glasses differ considerably from those found on crystalline materials. Also, the conductivity of glasses drops steadily with temperature and reaches very low values, near absolute zero. For crystals, the conductivity continues to rise with decreasing temperature until very low temperatures are reached. (Kittel, C., 1949). It is essential to determine the thermal properties since applications such as aerospace equipments acquire high ability to withstand extreme temperatures.

2.2 High density polyethylene (HDPE)

According to Anatole, A. (2007), High density polyethylene (HDPE) is a thermoplastic material composed of carbon and hydrogen atoms joined together forming high molecular weight products. The polymer chain may be 500,000 to 1,000,000 carbon units long. Short and long side chain molecules exist with the polymer’s long main chain molecules, as depicted in Figure 2.2. The longer the main chain, the greater the number of atoms, and consequently, the greater the molecular weight. The molecular weight, the molecular weight distribution and the amount of branching determine many of the mechanical and chemical properties of the end product.

Figure 1-1a: Methane Figure 1-1b: Ethylene

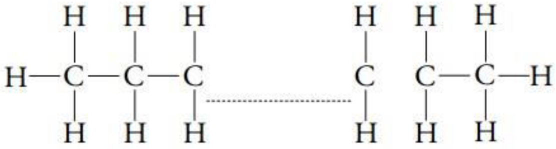


Figure 1-1c: Polyethylene Molecular Chain

Figure 2.2: Structure of polyethylene (Anatole, A., 2007)

HDPE resin has a greater proportion of crystalline regions than LDPE. The size and distribution of crystalline regions are determinants of the tensile strength and environmental stress crack resistance of the end product. HDPE, with fewer branches than MDPE or LDPE, has a greater proportion of crystals, which results in greater density and greater strength (Anatole, A., 2007).

Hypothetically, Figure 2.3 shows that a completely crystalline polyethylene would be too brittle to be functional and a completely amorphous polyethylene would be waxlike, much like paraffin. Upon heating, the ordered crystalline structure regresses to the disordered amorphous state; with cooling, the partially crystalline structure is recovered (Plastic Pipe Institute, 1993). According to Bensason et al. (1996), HDPE is stronger, abrasion resistant, stiffer and less translucent compared to low density polyethylene.

Today, plastic highway drainage pipes are commonly manufactured from HDPE and PVC, due to their distinguished attributes such as toughness, flexibility, chemical resistance and non-conductors of electricity. According to Plastic Pipe Institute (1993), thermoplastic highway drainage pipes have been used for highway drainage since the early 1970s. Since then, growing out of applications for agricultural drainage, more HDPE drainage pipes have been installed than all other plastic pipes combined. They are being used for storm sewers, perforated underdrains, storm drains, slope drains, cross drains, and culverts.

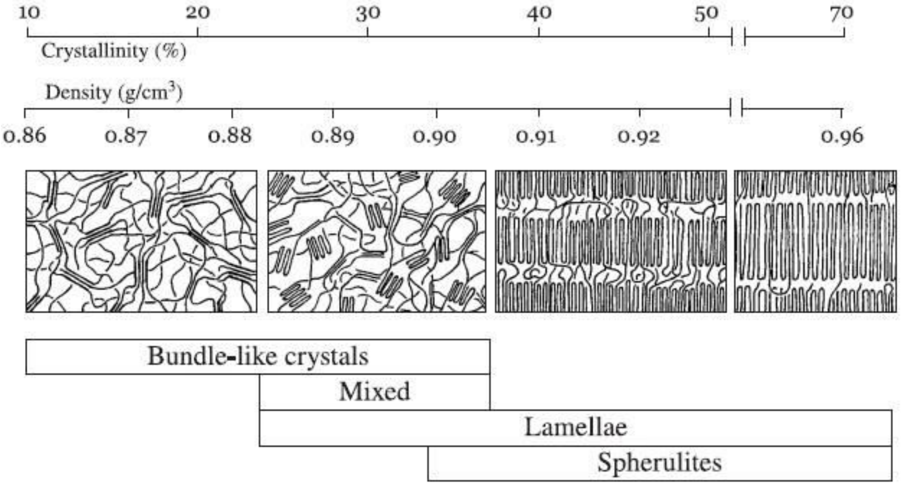


Figure 2.3: Crystal structure classification of polymers (Bensason et al., 1996)

2.2.1 Mechanical Properties

Ductile failure is a type of failure that generally occurs over a short amount of time at high stress levels. At the macroscopic level, ductile tensile failure results in observation of visible deformation (necking) in the polymer sample. For polyethylene, tensile ductile behaviour is influenced by the semicrystalline nature of the material. In the figure below, the stress-strain curve for tensile ductile deformation is accompanied by illustrations of what occurs before within the material at the micro scale (Alvarado-Contreras A., 2007).

As observed in Figure 2.4, at the beginning, before the yield point, no visible deformation of material is observed and the load is mainly carried by rigid crystalline lamellae. As strain increases, stress increases as well as yield occurs. During the time period between the point of yield and the onset of strain hardening, the load on the test sample remains at a relatively constant level. The deformation in this region is due to a combination of amorphous phase rearranging itself and crystal lamellae slipping past each other, but each individual crystal itself is still intact (Cheng, J., 2008).

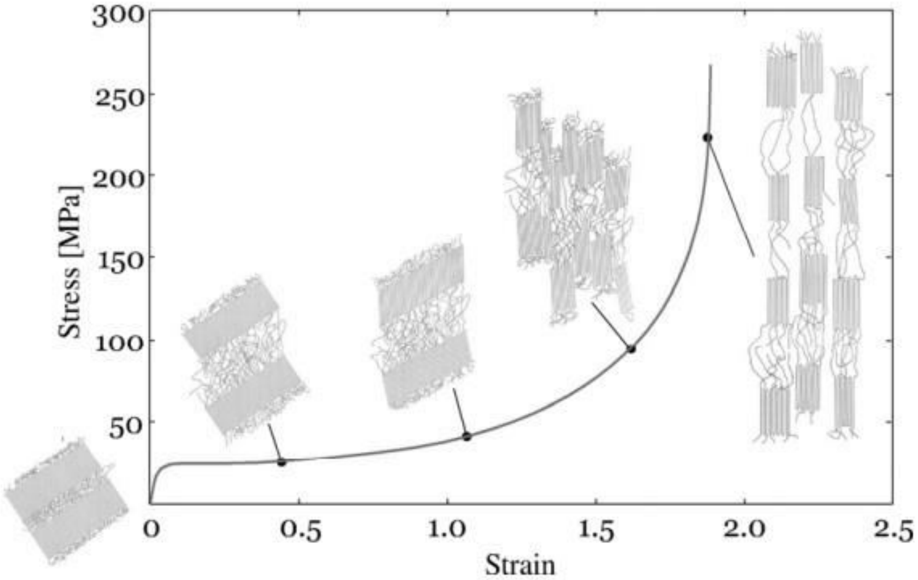


Figure 2.4: Stress-Strain behaviour of polyethylene (Cheng, J., 2008)

On the other hand, a polymer that undergoes brittle failure has a clean break with little material deformation (Mori, S. & Barth, G., 1999). To the naked eye, the fracture surface appears smooth, but under scanning electron microscopy (SEM), the surface actually consists of short random pullouts. Brittle-type failure occurs when a low stress is applied over a long period of time, causing the inter-lamellar links under stress start to untangle from each other until the number of remaining linkages become very low.

As shown in Figure 2.5, when the few inter-lamellar links are stretched to the limit, they are unable to pull apart lamellae, consequently a brittle fracture of the polymer occurs. According to Mathot, F. (1993), strain hardening is a phenomenon observed in fully drawn (and cold-drawing) of polymers. The cause of increasing stress during hardening is reported to be due to molecular alignment of polymer chains that result in increasing strength of the material. The type of chain alignment can be seen as a form of strain-induced crystallization and increasing crystallinity is known to increase tensile strength of polyethylene.

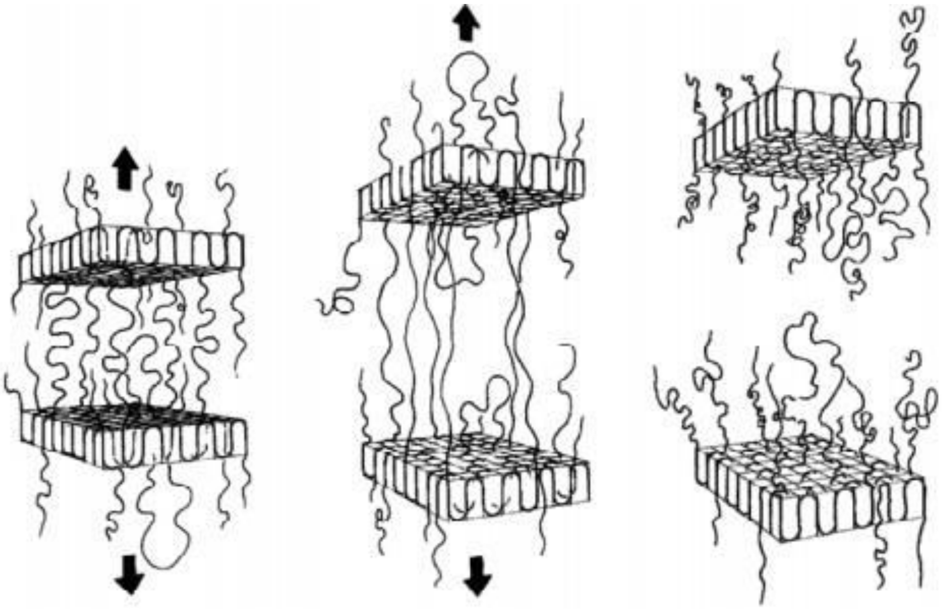


Figure 2.5: Stages of brittle fracture (Cheng, J., 2008)

2.2.2 Thermal Properties

According to Cheng, J. (2008), polyethylene is classified as a viscoelastic material, with both solid-like and liquid-like properties. With changes in temperature, mechanical behaviour of polyethylene changes due to movement of chains. As illustrated in the figure below, changes in storage modulus (E’) in polyethylene can be detected with changes in temperature. At temperature lower than the glass transition temperature, where only local movement of the polymer backbone and the bending of side chains are possible, polyethylene behaves like a rigid solid with high E’ values (Huang, B. et al., 1994).

At temperature above glass transition, larger scale movements in the amorphous phase result in polyethylene taking on more liquid-like behaviour and the E’ value decreases. At the melting temperature, crystalline lamellae inside the polyethylene matrix start to melt, large scale slippage occurs and the E’ value dramatically decreases. At temperature higher than the melting temperature, polyethylene loses its structural integrity and becomes a viscous melt (Huang, B. et al., 1994). This trend is further illustrated in Figure 2.6.

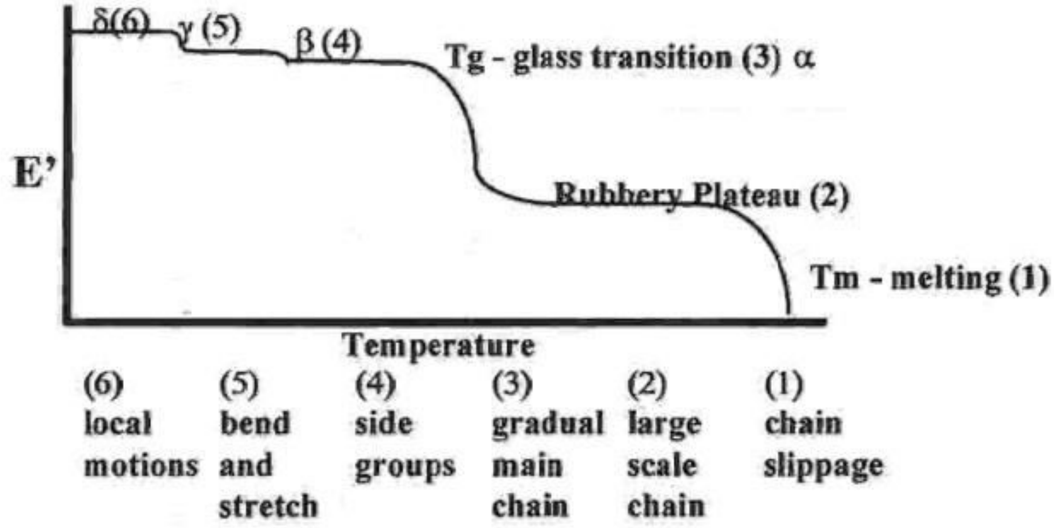


Figure 2.6: Thermal transition stage of polyethylene (Cheng, J., 2008)

2.3 HDPE reinforced composite

The concept of using fibers to improve the performance of materials is not new. The modern developments of fiber reinforcement started in the early 1960s. A multitude of fibers and the introduction of fiber materials are continuously done in the market as new applications such as polyester fiber, asbestos fiber, glass fiber, polypropylene fiber, carbon fiber and cellulose fiber (Serfass et al., 1996). The principal functions of fiber as reinforcement material is to provide additional tensile strength in the resulting composite. This may increase the amount of strain energy that can be absorbed during the fatigue and fracture process of the mix.

According to AlMadeed, A. et al. (2012), previous research has been conducted to investigate the effect of chain branching on the dispersion and adhesion in HDPE glass fiber composite. The interaction between the polyethylene matrix and the glass fibers was investigated in terms of differences in mechanical behaviour, morphological characteristics, rheological and thermal properties.

2.3.1 Mechanical Properties

Tensile Strength

Based on the results, a graph represented in Figure 2.7 is shown with the tensile strength and modulus with respect to different kinds of polyethylene and their glass fiber reinforced composites. These results are consistent with the density values of the sample. Usually, higher density translates into higher tensile strength and lower elongation due to higher crystallinity (AlMadeed, A. et al., 2012). There is a significant improvement in mechanical properties, both modulus and strength, by addition of glass fibers.

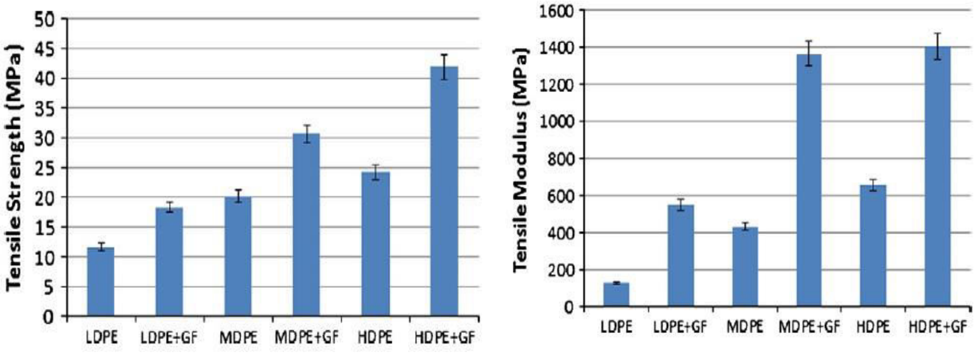
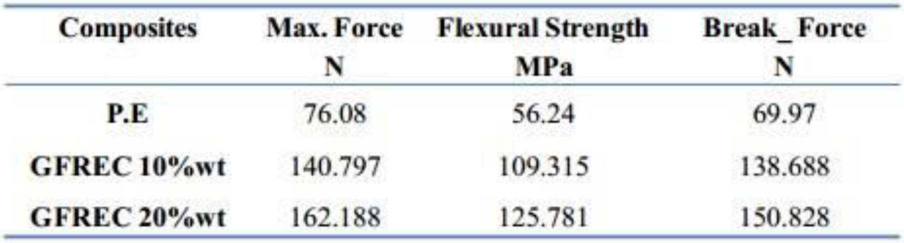


Figure 2.7: Strength and modulus for polymer glass fiber composite (AlMadeed et al., 2012)

Flexural Strength

Apart from that, a study conducted by Singh S. et al. (2013) regarding the mechanical properties of glass fiber reinforced epoxy composites have proven that by altering the fiber contents in each specimen by 10 and 20% wt., the flexural strength also increases due to the increased stiffness of composites. Respective data is shown in Table 2.3. Flexural strength is also known as modulus of rupture, bends strength, which is a mechanical parameter of materials. It represents the highest stress bearing capacity of the material at its moment of rupture. Glass fibers are lighter in weight and are stiffer which resists bending loads.

Table 2.3: Comparisons of flexural test for pure epoxy (P.E), GFREC 10% wt. And GFREC 20% wt. (AlMadeed et al., 2012)



Composites	Max. Force N	Flexural Strength MPa	Break_ Force N
P.E	76.08	56.24	69.97
GFREC 10%wt	140.797	109.315	138.688
GFREC 20%wt	162.188	125.781	150.828

2.3.2 Thermal Properties

Thermal gravimetric Analysis (TGA)

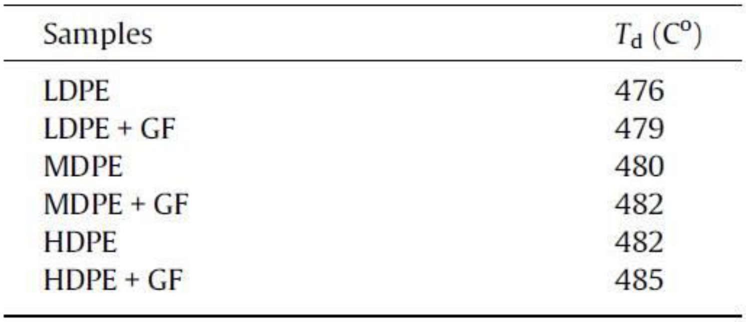
As explained by Mettler Toledo User Manual, thermal analysis comprises of a group of techniques that are used to determine the deviation in physical and chemical properties of the test sample is recorded accordingly with the temperature or time as an increasing function. If the increasing function is temperature, then heating rate will be constant. If it is in function of time, then the temperature will be constant or the mass loss will be constant. Among them is by conducting TGA.

Apart form that, this analysis enables a lot of valuable information to be recorded, such as the second order phase transitions which includes vaporisation, sublimation, adsorption and absorption can be gathered through this analysis. At the same instance, information on chemical properties also can be gathered through this system such as decomposition, chemisorption, oxidation and reduction and several others.

From Table 2.4 as shown below, it can be seen that final degradation temperatures of neat HDPE is higher than LDPE polymers. On the other hand, thermal stability of the polymers increased with addition of glass fiber in the composites

(AlMadeed, A. et al., 2012). This phenomenon was also observed by Shuying Y. et al. (2007), who found that thermal stability of HDPE is enhanced due to the presence of carbon nano fiber in the HDPE matrix.

Table 2.4: Degradation temperatures for polymer glass fiber composite



Samples	T_d (C°)
LDPE	476
LDPE + GF	479
MDPE	480
MDPE + GF	482
HDPE	482
HDPE + GF	485

These researches have provided the possibilities of further enhancing the readily existing HDPE material properties with glass fiber as reinforcement for the application in various industries, such as aerospace elements, automotive parts, marine structures, structural members and anti-vibration material.

Differential Scanning Calorimetry (DSC)

DSC is a technique that measures the heat flow of samples as a function of temperature or time. This method allows physical transitions and chemical reactions to be quantitatively measured (Mettler Toledo). The glass transition is a reversible transition that occurs when amorphous material is heated or cooled in a particular temperature change. On cooling, the material becomes brittle (less flexible) like a glass, and on heating becomes soft.

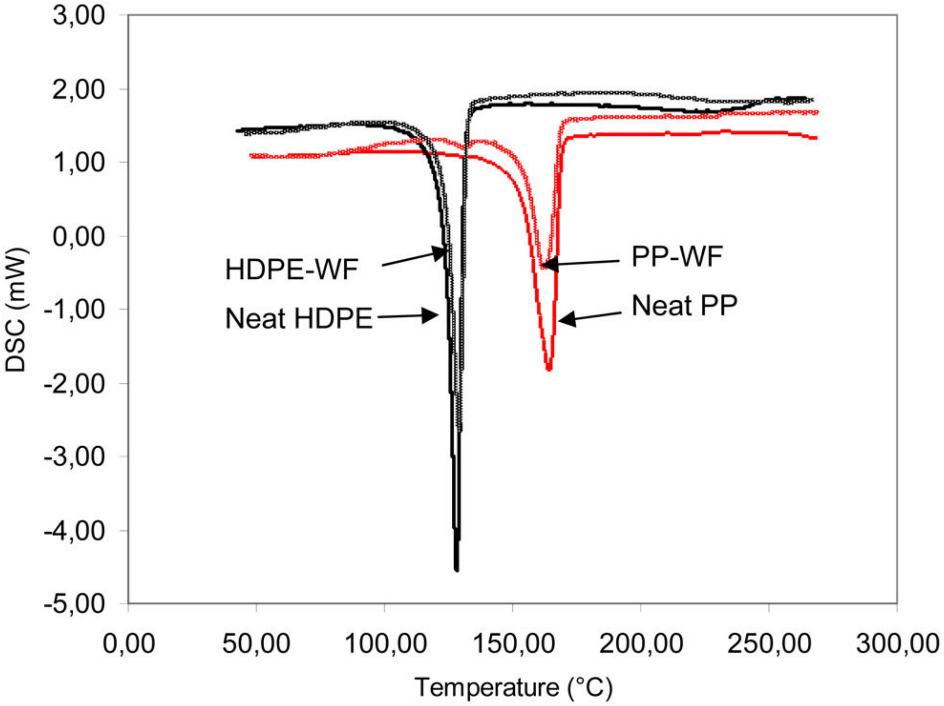


Figure 2.8: DSC curve of thermoplastic wheat straw reinforced composite (Karakus et al., 2008)

As shown in Figure 2.8, the typical heating curve for reinforced HDPE can be observed. The glass transition exhibits enthalpy relaxation, which is shown by the overlapping endothermic peak. This is considered as a kinetic phenomenon, whereby the measured value of the glass transition depends on the cooling rate, the thermomechanical history of the sample and the evaluation conditions. The lower the cooling rate, the lower the resulting glass transition that is measured in the following heating run.

CHAPTER 3

METHODOLOGY

3.1 Research methodology

As this project is mainly an empirical research, the results obtained from this research can be used to compare with other literature results. The results can further enhance the research and development on the properties of HDPE glass fiber composites. The step-by-step process of this research is illustrated in Figure 3.1 for further understanding, which are also captured in **Appendix 2, Appendix 3, Appendix 4 and Appendix 5**.

3.1 Materials

The neat HDPE pellets are purchased from PT Lotte Chemical Titan in Ipoh with Melt Flow Index at 2.16 kg/190°C of 18, whereby glass fiber is purchased in chopped strand mat form with density of 4.5g/m². The product data for HDPE is shown in Table 3.1, as given by the supplier company.

Table 3.1: Product Data for HDPE (PT Lotte Chemical Titan)

Properties	Value	Unit
Melt Flow Rate (190°C/2.16180. kg)	18	g/10 min
Nominal Density	950	kg/m^3
Vicat Softening Point	124	°C
Melting Point	130	°C

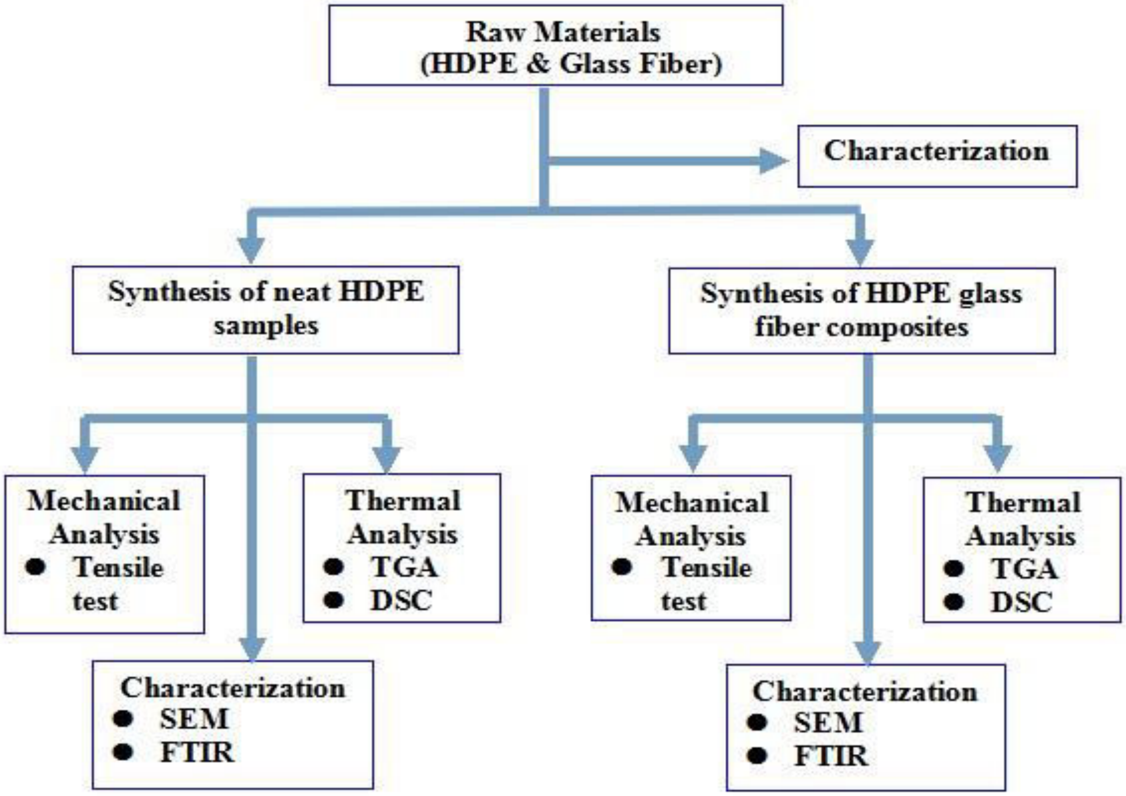


Figure 3.1: Research methodology

3.2 Methods

3.2.1 Characterization of materials

Before the experiment started, the raw materials obtained are analyzed first in order to determine their original arrangement and properties. The characterization data are obtained through the Scanning Electron Microscopy (SEM) analysis to observe the initial morphological properties of HDPE and glass fiber separately.

The pure HDPE pellets are also analyzed with the Fourier Transform Infra-Red (FTIR) spectroscopy in order to determine the branching systems of the respective material which later can be correlated to its reaction to the mechanical and thermal tests.

3.2.2 Synthesis of samples

The neat and composite samples production of HDPE and glass fiber is carried out using the standard compression molding machine. The processing temperature is 170°C, which is the maximum melting temperature for HDPE to ensure thorough heating process. The cooling time is set within the range of 90 to 120 minutes after the heating platen is turned off.

During synthesis of the sample, mix formulation of 10% wt., 15% wt. and 20% wt. of glass fiber are applied for the respective composite samples, which were subsequently layered into the mold area, as specified in Figure 3.2.

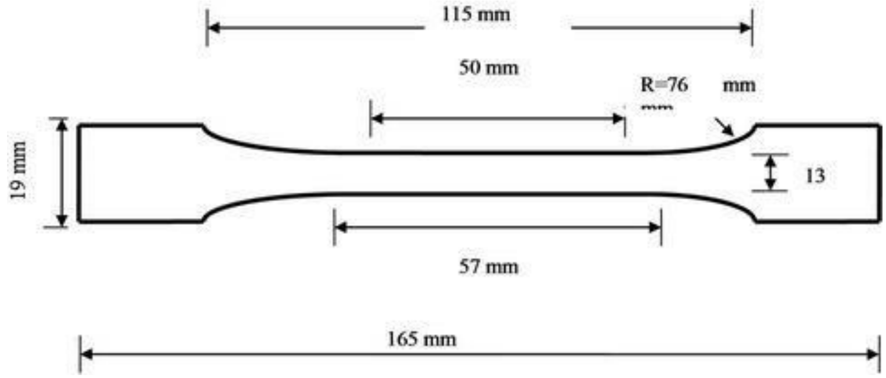


Figure 3.2: Sample specifications for ASTM D638

3.2.3 Tensile testing

Tensile tests were performed to measure the tensile properties like tensile strength, tensile modulus and elongation at break of the neat HDPE and HDPE glass fiber composite samples. The tensile tests were measured by using a universal testing machine at a crosshead speed of 10 mm/min. The tensile samples were prepared and tested according to the ASTM: D638. In each case, five samples were tested and an average value was reported, as included in **Appendix 6**.

3.2.4 Morphology Analysis

The neat and composite samples done with tensile testing were subsequently sent to the SEM analysis to examine the cross-section of the failure of the samples and hence help to study the bonding between the fiber and the matrix.

3.2.5 Differential Scanning Calorimetry (DSC)

A DSC analysis of the various HDPE samples and their glass fiber reinforced composites was completed using a Perkin Elmer, Pyris Thermal Analyzer. All measurements were taken under nitrogen atmosphere and at a constant heating and cooling rate of 10°C/min. The percentage of crystallinity (%Xc) of the sampleswas calculated according to equation (1):

$$Xc(\%) = \frac{\Delta H_f}{\Delta H_f^{\circ} w} x100 \qquad \text{....... Equation (1)}$$

3.2.6 Thermo Gravimetric Analysis (TGA)

The thermal decomposition of neat polymer matrix and polymer composites were evaluated by thermo gravimetric analysis (TGA). Thermo gravimetric analysis (TGA) measurements were performed in nitrogen atmosphere using Perkin Elmer Thermal instrument at a heating rate of 10°C/min until the maximum temperature of 650°C, to yield the onset temperature of decomposition, mass loss and maximum decomposition peak.

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Raw Materials Characterization

4.1.1 SEM analysis

The scanning electron microscopy (SEM) was used to characterize the morphology of the neat pellets of the high density polyethylene (HDPE), which was conducted at the beginning of the study. Typical SEM micrographs are obtained as per shown in Figure 4.1, with scales of (a) 100µm and (b) 80µm.

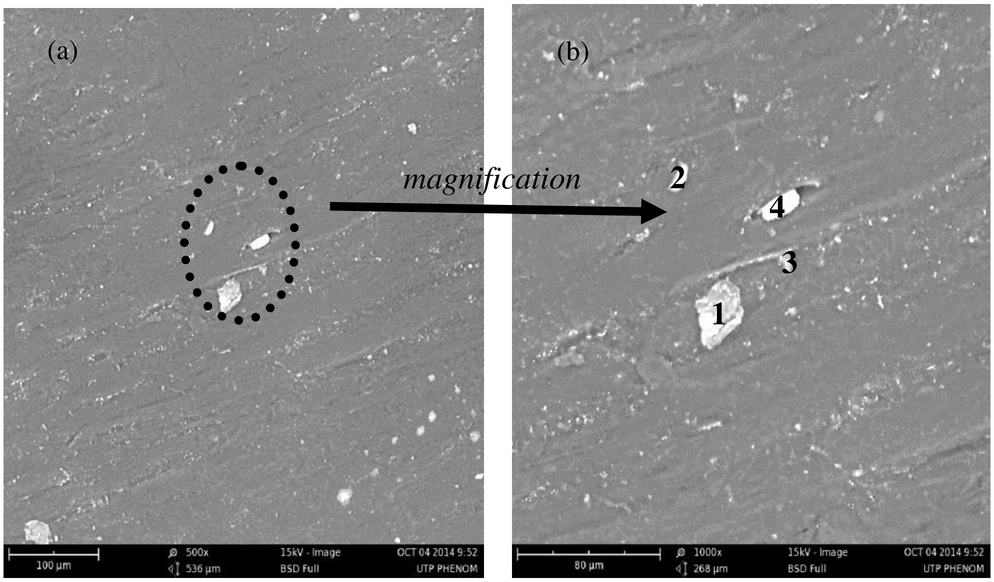
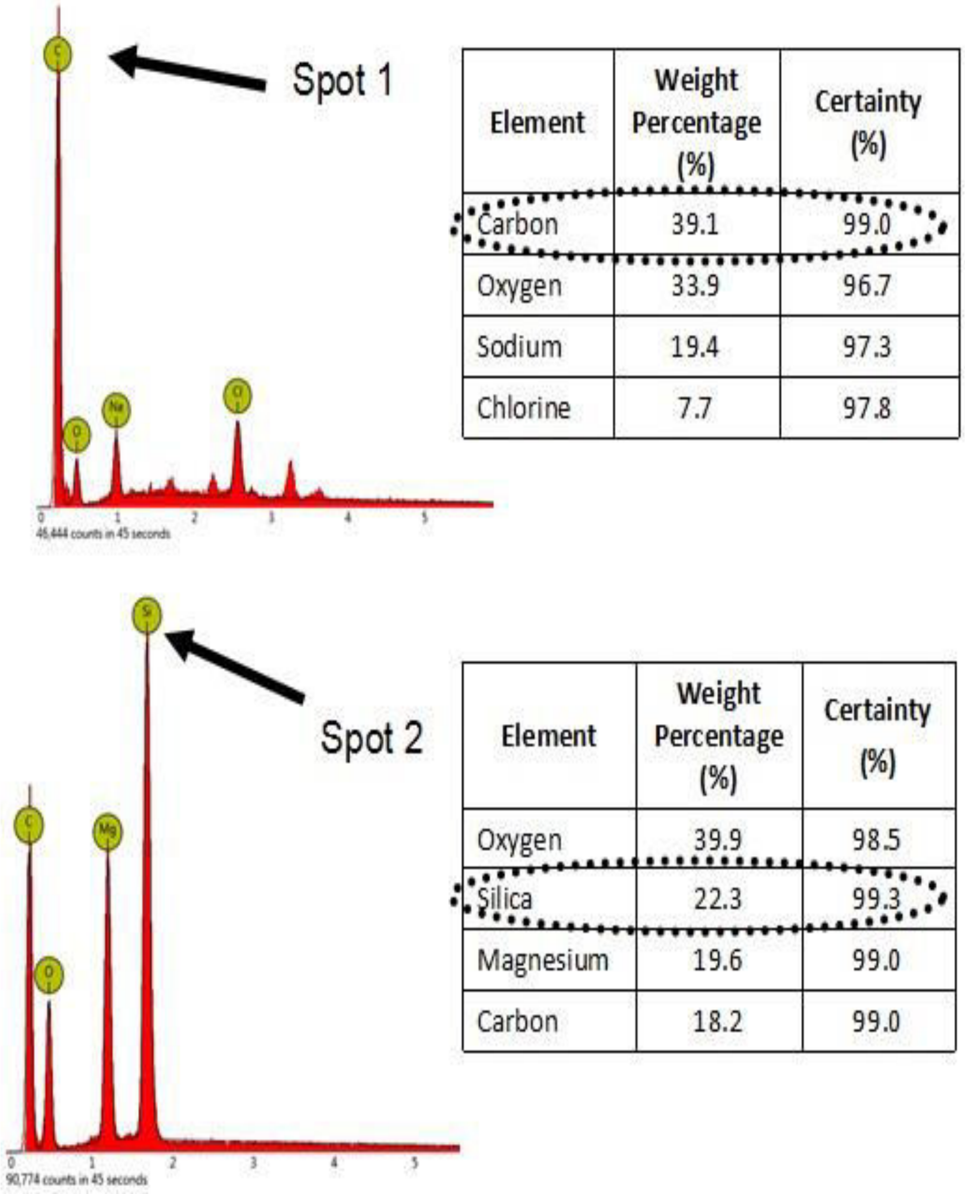


Figure 4.1: Scanning electron microscopy (SEM) micrographs of neat HDPE pellets. (a) scale 100µm ; (b) scale 80µm.

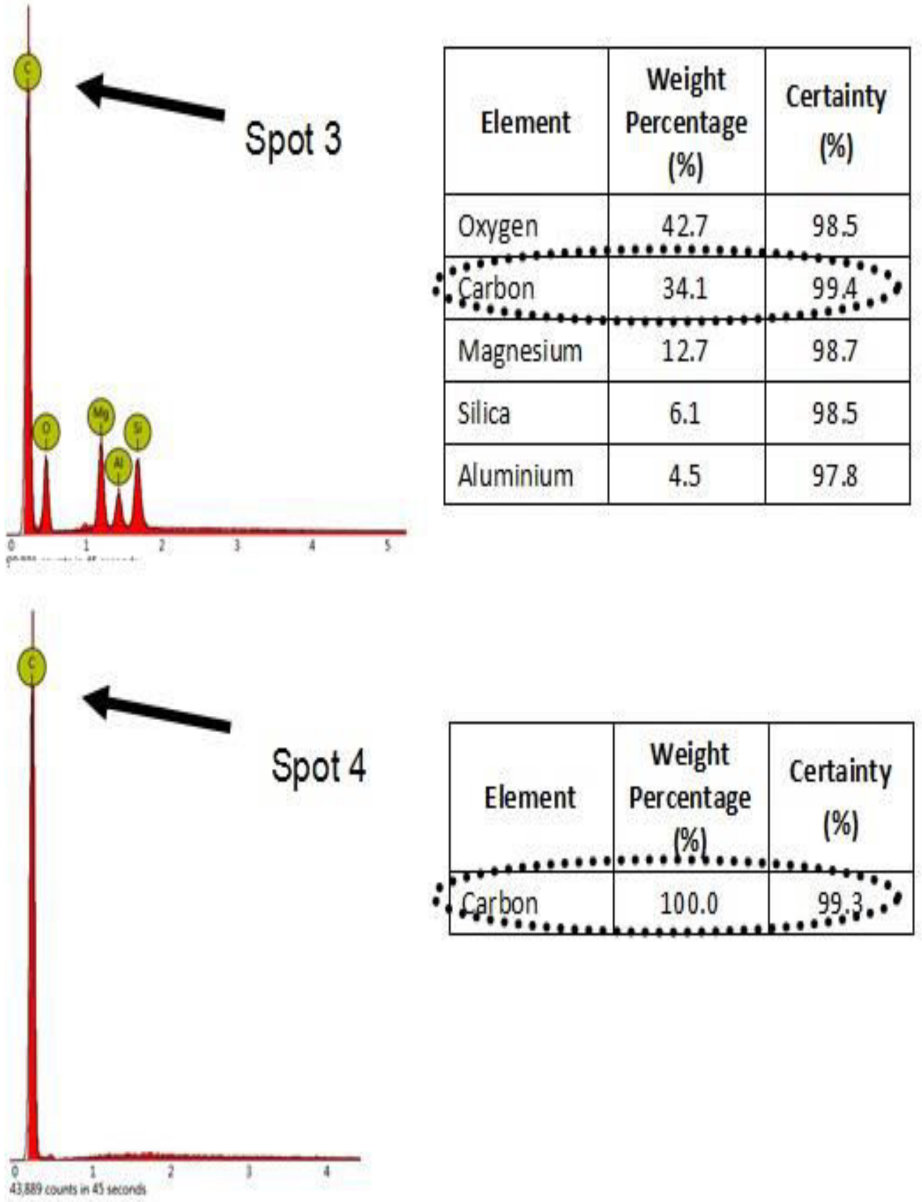
SEM study revealed the dense and compact surface morphologies of neat HDPE, which will later be compared with the image of HDPE samples containing various percentages of glass fiber as reinforcement.



Element	Weight Percentage (%)	Certainty (%)
Carbon	39.1	99.0
Oxygen	33.9	96.7
Sodium	19.4	97.3
Chlorine	7.7	97.8

Element	Weight Percentage (%)	Certainty (%)
Oxygen	39.9	98.5
Silica	22.3	99.3
Magnesium	19.6	99.0
Carbon	18.2	99.0

Figure 4.2 (a) and (b) : Spectrum analysis for tiny spots 1 and 2



Element	Weight Percentage (%)	Certainty (%)
Oxygen	42.7	98.5
Carbon	34.1	99.4
Magnesium	12.7	98.7
Silica	6.1	98.5
Aluminium	4.5	97.8

Element	Weight Percentage (%)	Certainty (%)
Carbon	100.0	99.3

Figure 4.2 (c) and (d) : Spectrum analysis for tiny spots 3 and 4

The characterization has provided useful information on the elemental composition of small contaminant particles that existed in tiny specks, as highlighted in Figure 4.1 (b), which were identified as **carbons** and **silica** through further quantification technique (Figure 4.2). The presence of contaminants may be due to the exposure to various materials in the manufacturing process at the factory.

4.1.2 Chemical analysis

The data from supplier was acquired in order to gain the chemical composition of HDPE pellets as basis understanding. HDPE is a type of polyethylene created through the polymerization of ethylene hence possessed a density of 0.941 g/cm³, with low degree of branching produced from chromium/silica catalysts. Figure depicts the process involved in the formation of dense HDPE molecules.

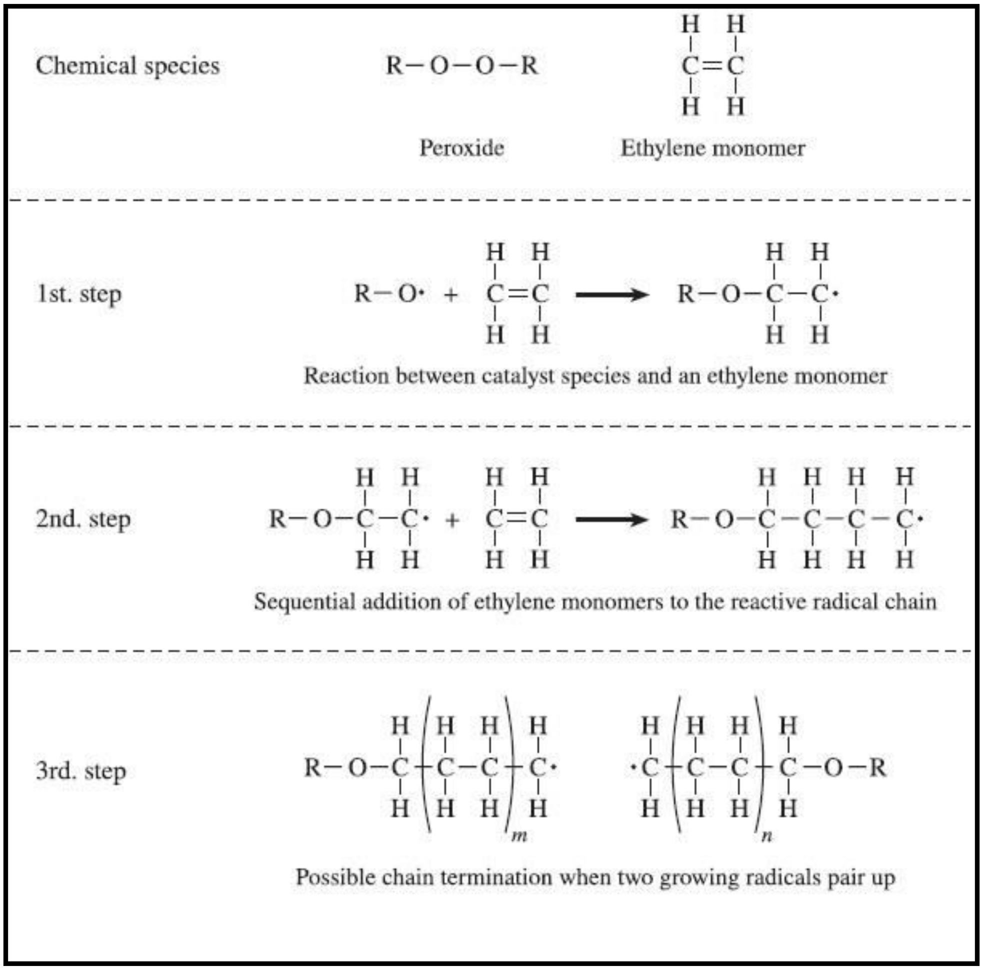


Figure 4.3: Possible polymerization scheme for polymer (Alvarado-Contreras, A., 2007)

4.1.3 Fourier-Transform Infra Red (FTIR) Analysis

Based on the test conducted, the analysis of data from FTIR spectroscopy revealed the absorptions of infra-red light by chemical bonds in all molecules, including polymers. Figure 4.4 shows the absorbance graph for HDPE pellet, which will later be compared with the graph of HDPE glass fiber composite.

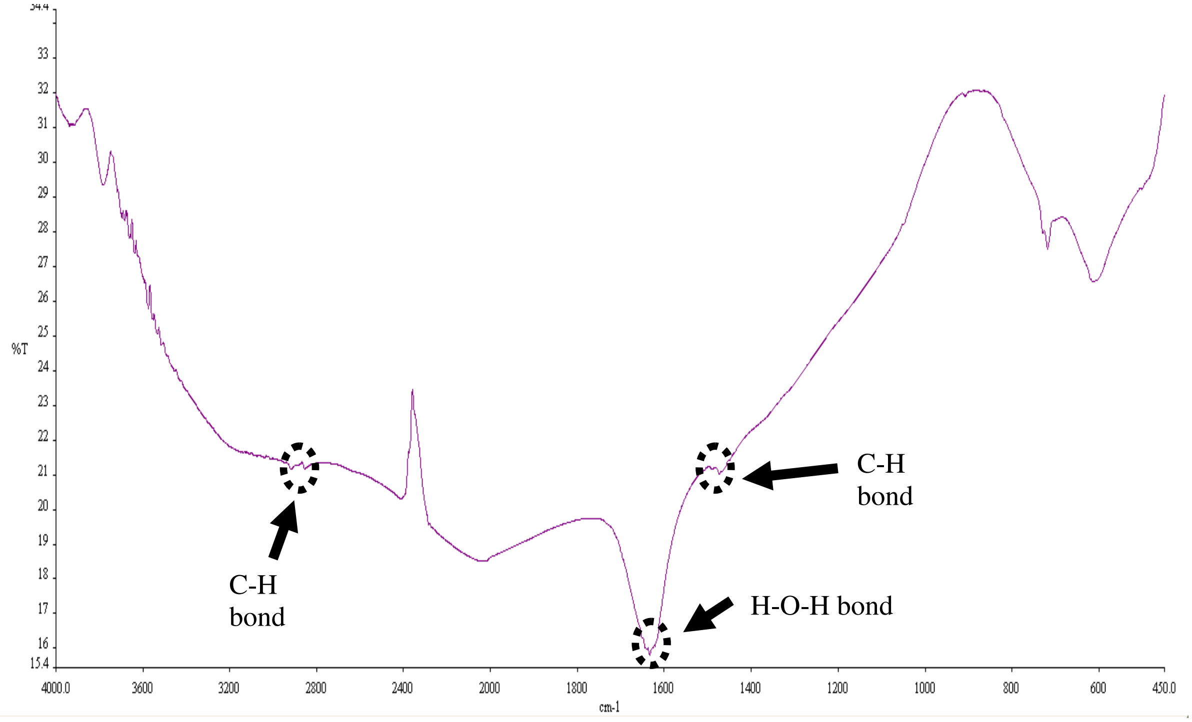


Figure 4.4: FTIR Analysis Curve for neat HDPE pellets

4.2 Characterization of Samples

4.2.1 Mechanical Analysis

Tensile properties

The failure of polymer composites can be viewed as any change of properties which makes the material or component functionally, structurally or aesthetically unacceptable. Figure 4.5 and 4.6 show the tensile strength and modulus, respectively of the neat HDPE sample and the HDPE composites with 10% wt.,15% wt. and 20% wt. of glass fiber.

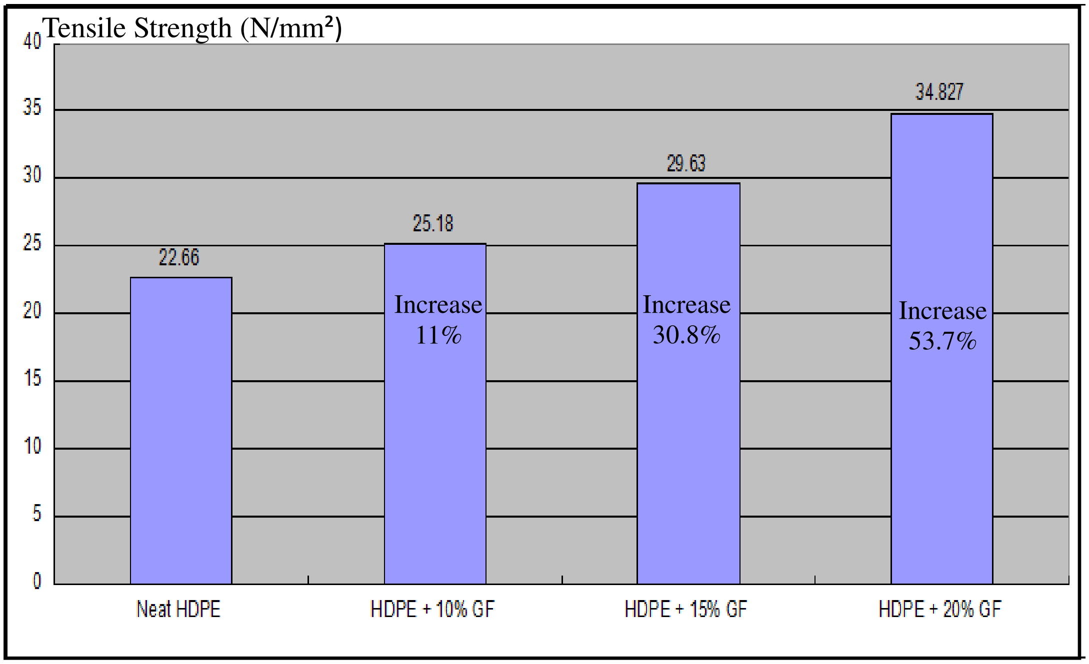


Figure 4.5: Effect of glass fiber compositions on the tensile strength of HDPE glass fiber composites.

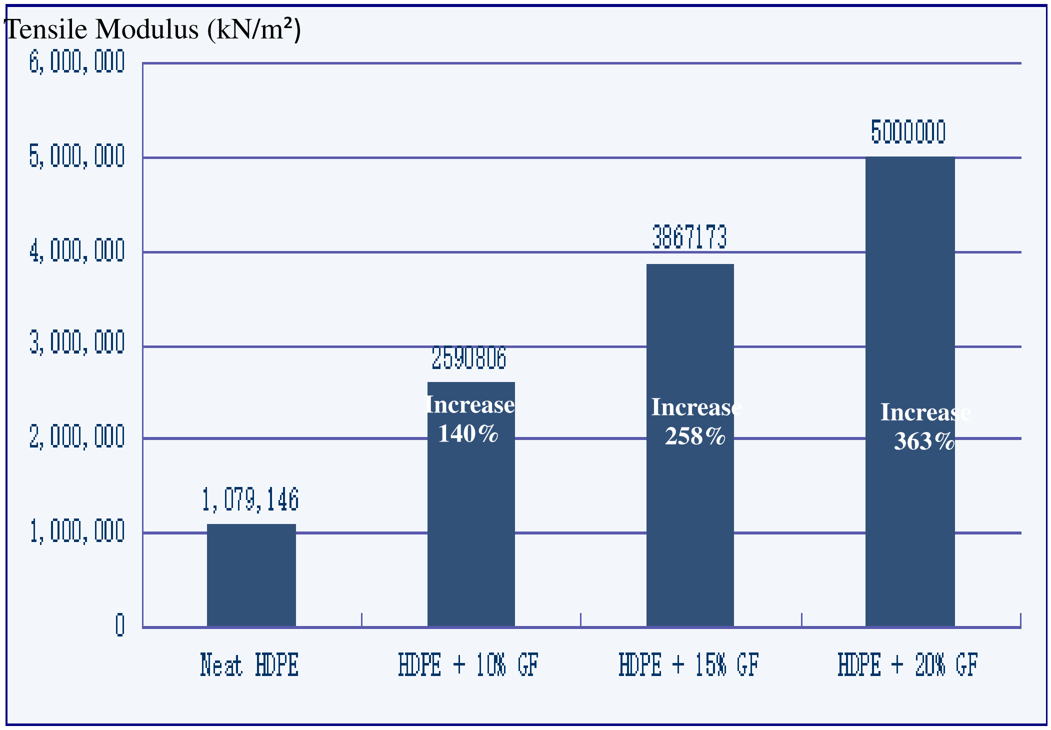


Figure 4.6: Effect of glass fiber compositions on the tensile modulus of HDPE glass fiber composites.

Based on the data, it revealed that both the tensile strength and modulus of the HDPE glass fiber composites are significantly improved with increasing glass fiber content. For the sample mixture containing 20% glass fiber, the relative tensile strength and modulus are 53.7% and 363% higher than that of pure HDPE sample. Similar results have been achieved by Somnuk et al. (2004) who studied the adhesion between the glass fiber and HDPE. They have found that tensile and flexural strength were improved with glass fiber addition.

On the other hand, the results for percentage of elongation for various percentages of glass fiber addition in HDPE are illustrated in Figure 4.7, showing the decrease in elongation as more percentage of fiber glass is added to the composites. Initially, neat HDPE was able to elongate 46.3% from its original length. However, with maximum addition of 20% glass fiber, the resulting composite was only able to show 13.7% of elongation, which is the least among others. The comparison of trend is further plotted in Figure 4.8, to demonstrate the resulting effect of tensile strength towards the composites' percentage of elongation based on the overall data from Table 4.1.

As shown in Figure 4.8, elongation at break follows an opposite trend to that of tensile strength and modulus. For an increasing % wt. of glass fiber in the composite, the value of elongation at break decreases. This indicates HDPE, which contains a high percentage of crystallinity due to the long linear chains, gradually loses its ductility property with more addition of glass fiber which is rigid and hard in nature. Hence it is proven that the impact of glass fiber addition to HDPE matrix is more significant for the tensile strength and modulus value, while decreasing the elongation at break value due to high crystallinity.

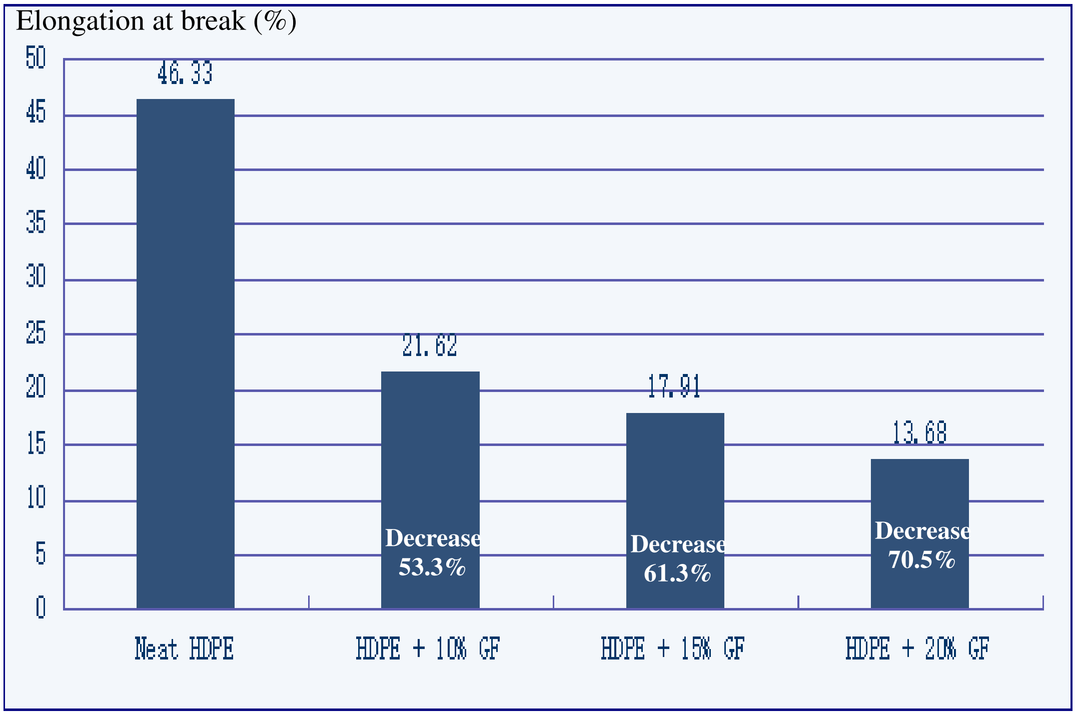


Figure 4.7: Elongation at break for pure HDPE and HDPE glass fiber composites.

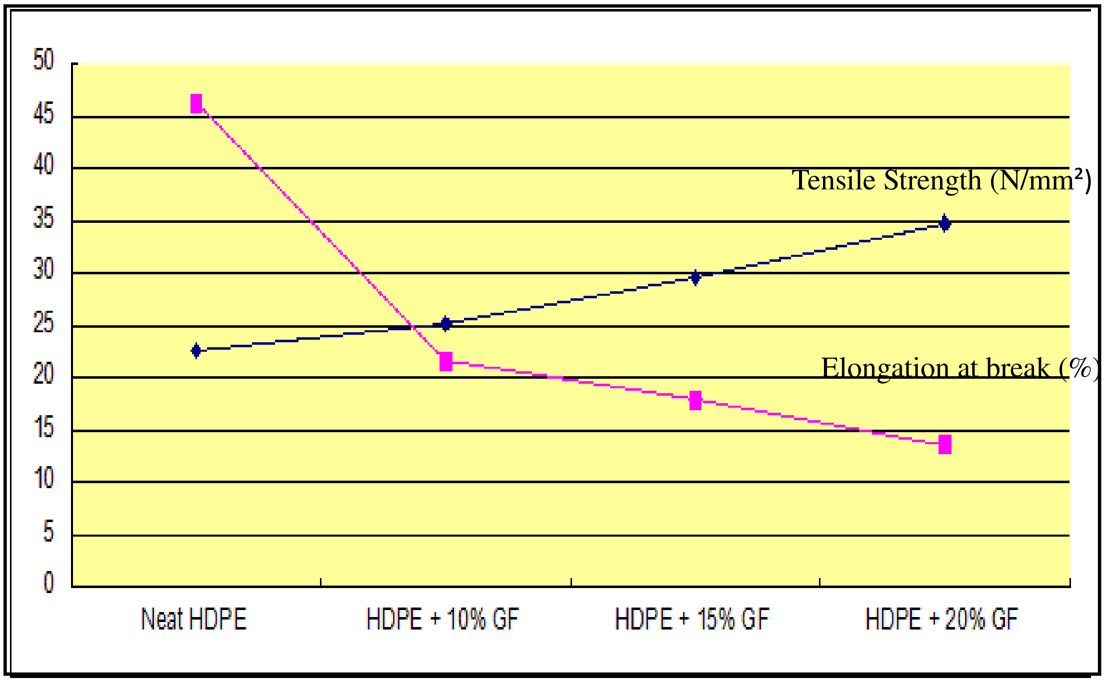


Figure 4.8: Comparison between the effects of glass fiber addition towards tensile strength and percentage of elongation.

Table 4.1: Effect of glass fiber addition on mechanical properties

Type of sample	Tensile strength (N/mm²)	Tensile modulus (kN/m²)	Elongation at break (%)
Neat HDPE	22.66	1079146	46.33
HDPE+10% glass fiber	25.18	2590806	21.62
HDPE+15% glass fiber	29.63	3867173	17.91
HDPE+20% glass fiber	34.83	5000000	13.68

Table 4.1 summarizes the overall comparison for all three parameters for mechanical analysis, which indicates that as glass fiber percentage increases, the tensile strength and modulus is improved, whereby the elongation at break decreases. This proves that, the presence of glass fiber within the dense and crystalline HDPE matrix gradually decreases its ductile properties, hencemaking it brittle. Figure 4.9 (a) and (b) shows the brittle fracture that occured in HDPE glass fiber composite sample.

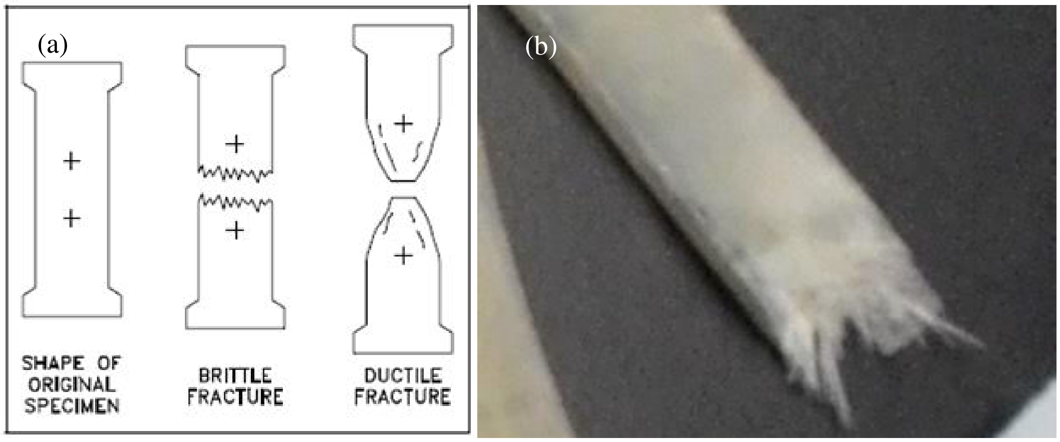


Figure 4.9: (a) Comparison between brittle and ductile fracture (b) Sample fracture of HDPE glass fiber composite.

4.2.2 Thermal Analysis

Thermal Stability

Thermal stability of pure polymers and various composites were measured by TGA (thermogravimetric analysis). Table 4.2 shows the decomposition temperatures of different compositions of HDPE glass fiber composites.

Table 4.2 Degradation temperatures of pure HDPE and HDPE glass fiber reinforced composites

Samples	T_d (°C)
Neat HDPE	413.59
HDPE + 10% glass fiber	415.66
HDPE + 15% glass fiber	422.00
HDPE + 20% glass fiber	435.00

By comparing the degradation temperatures of neat HDPE with HDPE glass fiber composites, it can be compared that the composites can endure a higher temperature before being fully decomposed. Although the amount of temperature increased is not significant, the effect of glass fiber addition can still be distinguished.

It can be seen from the graph in Figure 4.10 that the final degradation temperatures of HDPE with 20% glass fiber addition is the highest, which is 435°C and also the curve takes a higher temperature tu decline, unlike the neat HDPE. Hence, it is shown that thermal stability of the HDPE composites increased with more addition of glass fiber., due to the original property of glass fiber which is heat resistant.

This phenomenon was also observed by Shuying Yang et al. (2007) who found that thermal stability of HDPE is enhanced due to the presence of carbon nanofibre in the HDPE matrix. Giuliana Gorrasi (2011) reported that, incorporation of carbon nano tubes into the polymer matrix greatly improved the thermal stability of polyethylene composites.

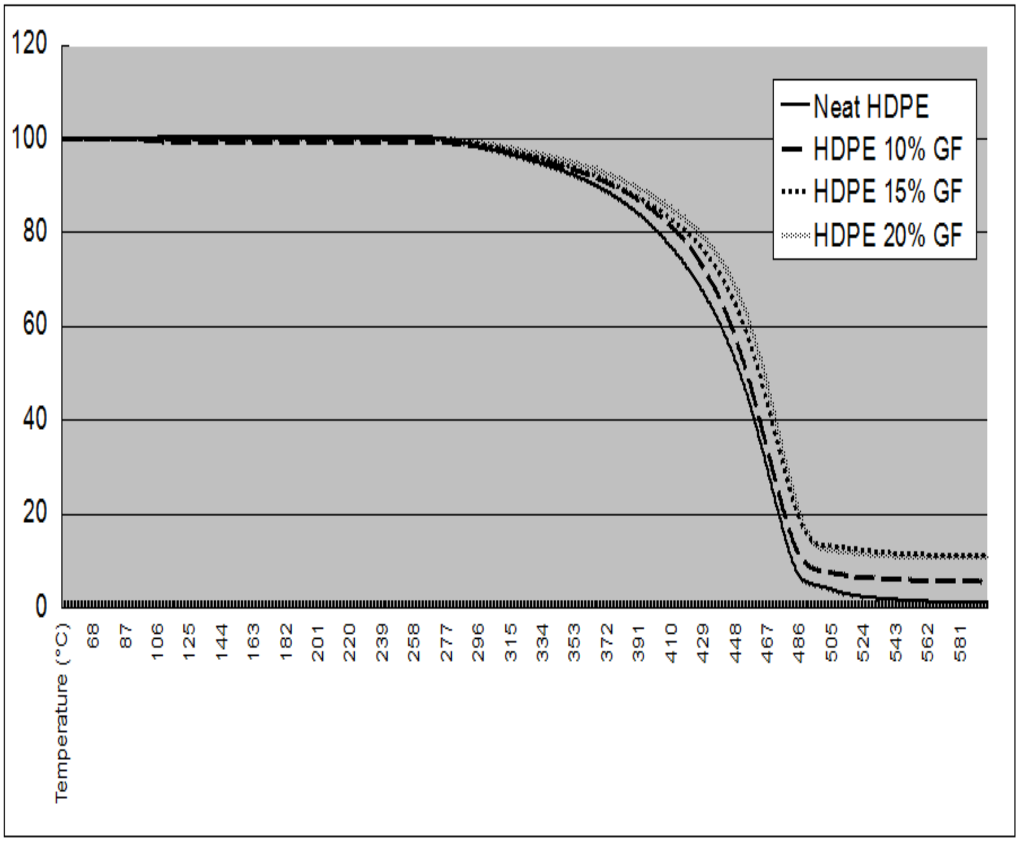


Figure 4.10: Decomposition curve for HDPE glass fiber composites

Crystallinity

Based on the data from Differential Scanning Calorimetry, crystallinity percentages for each composites samples were obtained. Theoretically, with increasing addition of glass fiber the percentage of crystallinity would also increase since glass fiber acts as nucleating agents to enhance the nucleation of HDPE particles.

The percentages of crystallinity for different HDPE glass fiber composites are shown in Figure 4.11. The trends do not follow theoretical assumption, since the crystallinity for HDPE with 20% wt. glass fiber was seen to be the least, which is 90.6% compared to others. This deviation may be due to the method of composite sample preparation, or the disturbance of specimen obtained for the test.

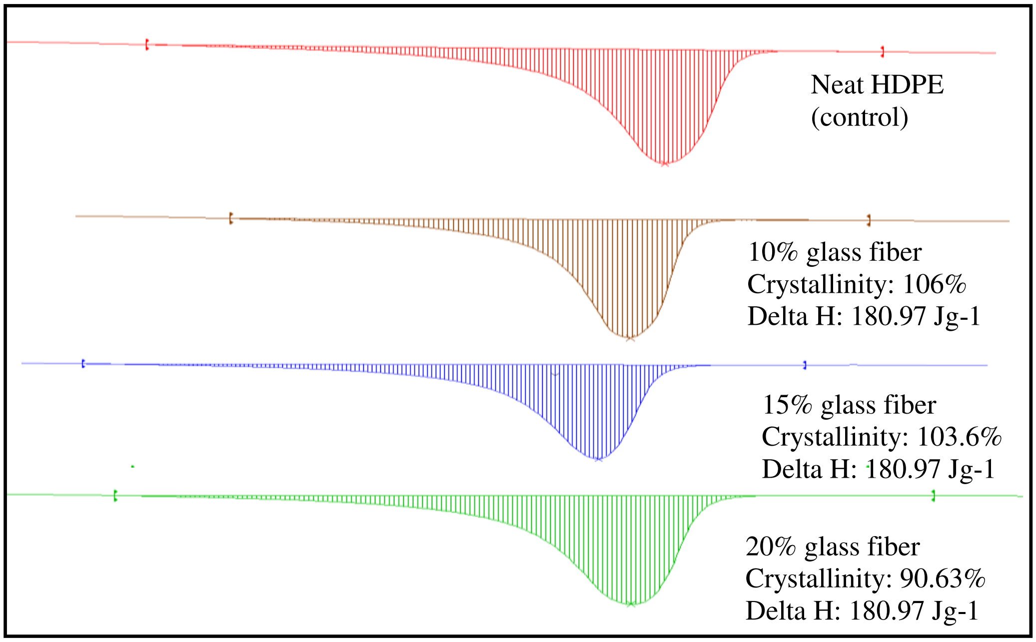


Figure 4.11: DSC graph showing percentages of crystallinity

4.2.3 FTIR Analysis

The FTIR spectroscopy graph for HDPE with 10% wt. fiber glass is obtained and compared with the graph of neat HDPE pellets, as shown in Figure 4.12

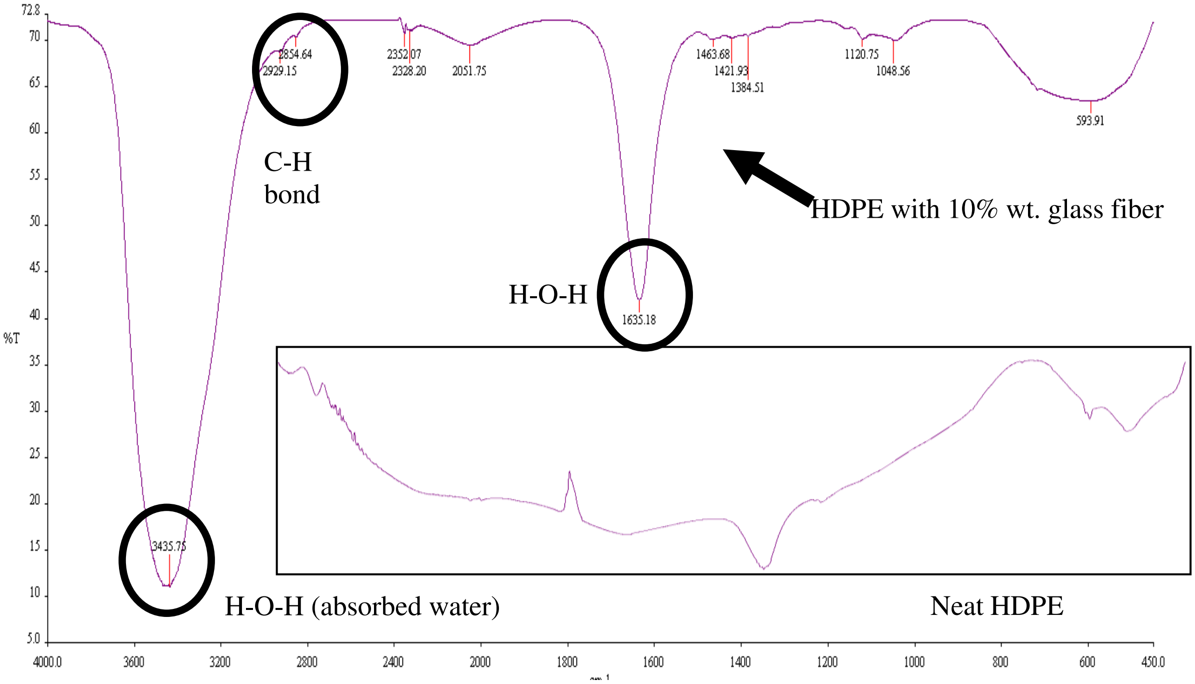


Figure 4.12: FTIR Analysis Curve for HDPE with 10% wt. glass fiber

4.2.4 SEM analysis of tensile fractured samples

SEM analysis was conducted after all samples have undergone tensile test, to observe the microstructure especially at the failure points. Figure 4.13 (a) to (d) show the micrographs obtained of the fractures surfaces, comparing the initial magnification (larger picture) of 1000x with the closer focus onto the fracture points using 3000x magnification.

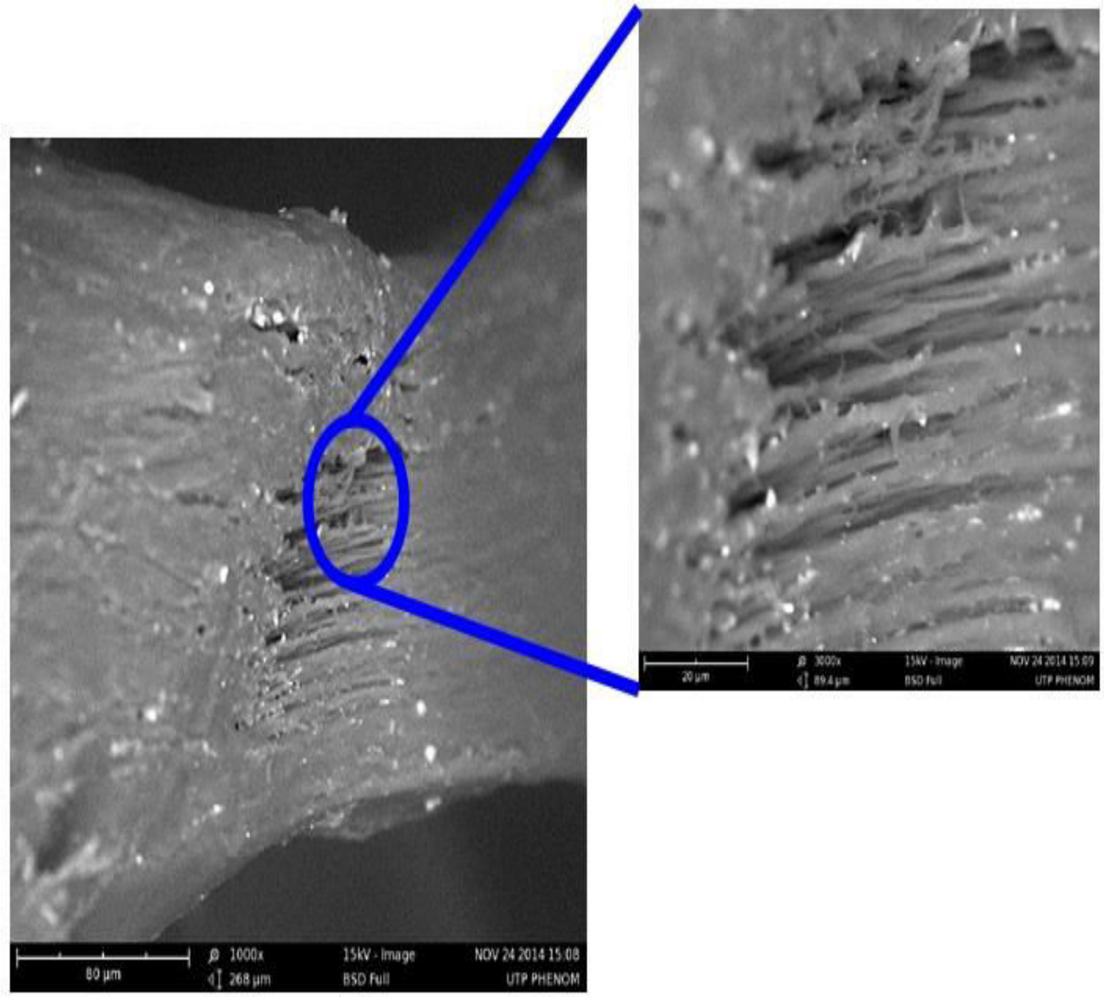


Figure 4.13 (a): SEM micrograph of neat HDPE sample

For neat HDPE, the **uniformity of microstructure** throughout the sample surface is observed in Figure 4.13 (a), whereas at the point of fracture, the **stretching of HDPE matrix** occurs due to the force exerted on it, which indicates the ductile property of the neat HDPE sample.

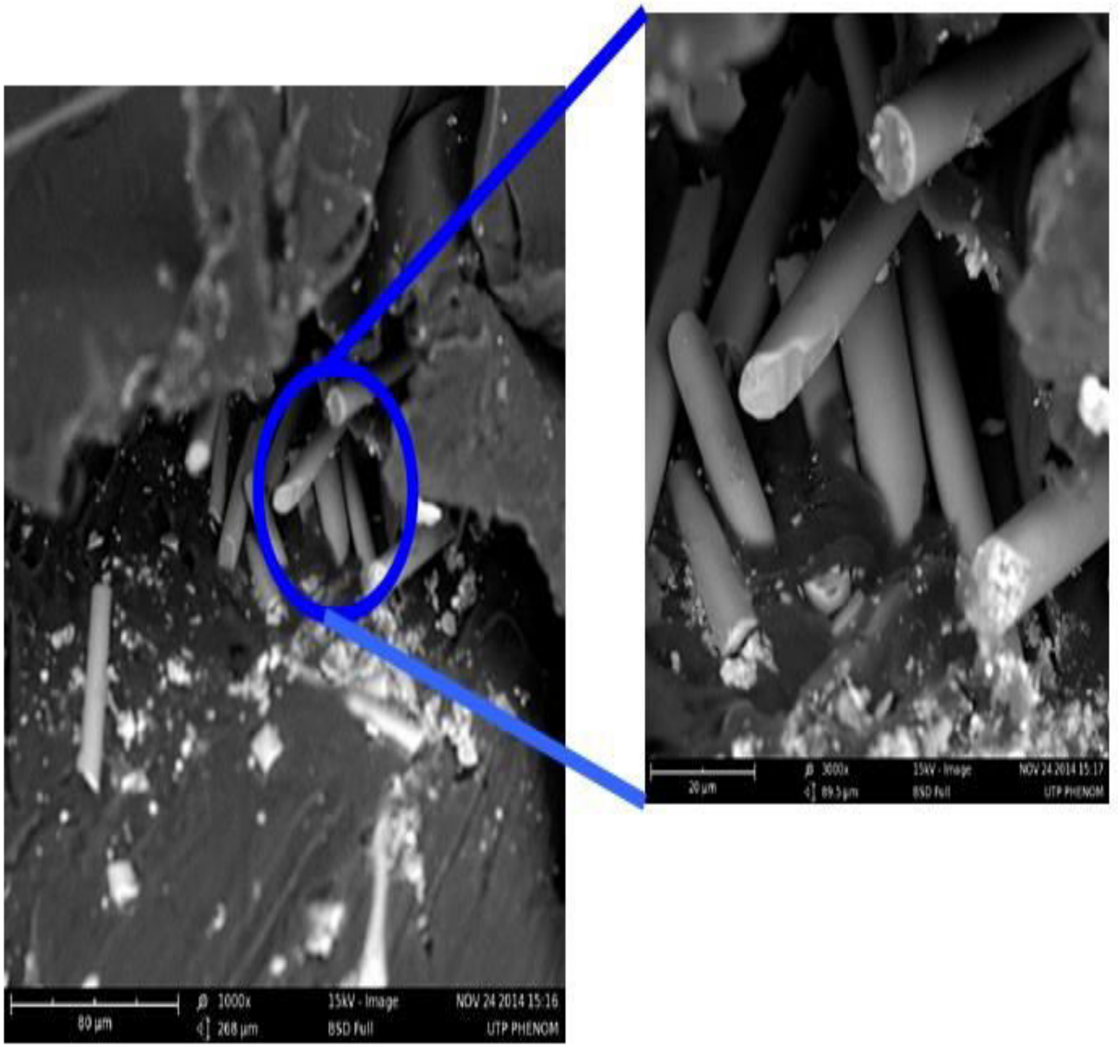


Figure 4.13 (b): SEM micrograph of HDPE 10% glass fiber sample

Non-uniform dispersion of glass fiber is observed in Figure 4.13 (b) which consists of 10% wt. from the whole sample. **Fiber pull-outs** are seen, whereby the small content of glass fiber has not gained strong adhesion to the HDPE matrix and to other glass fibers in the sample causing them to be ruptured separately.

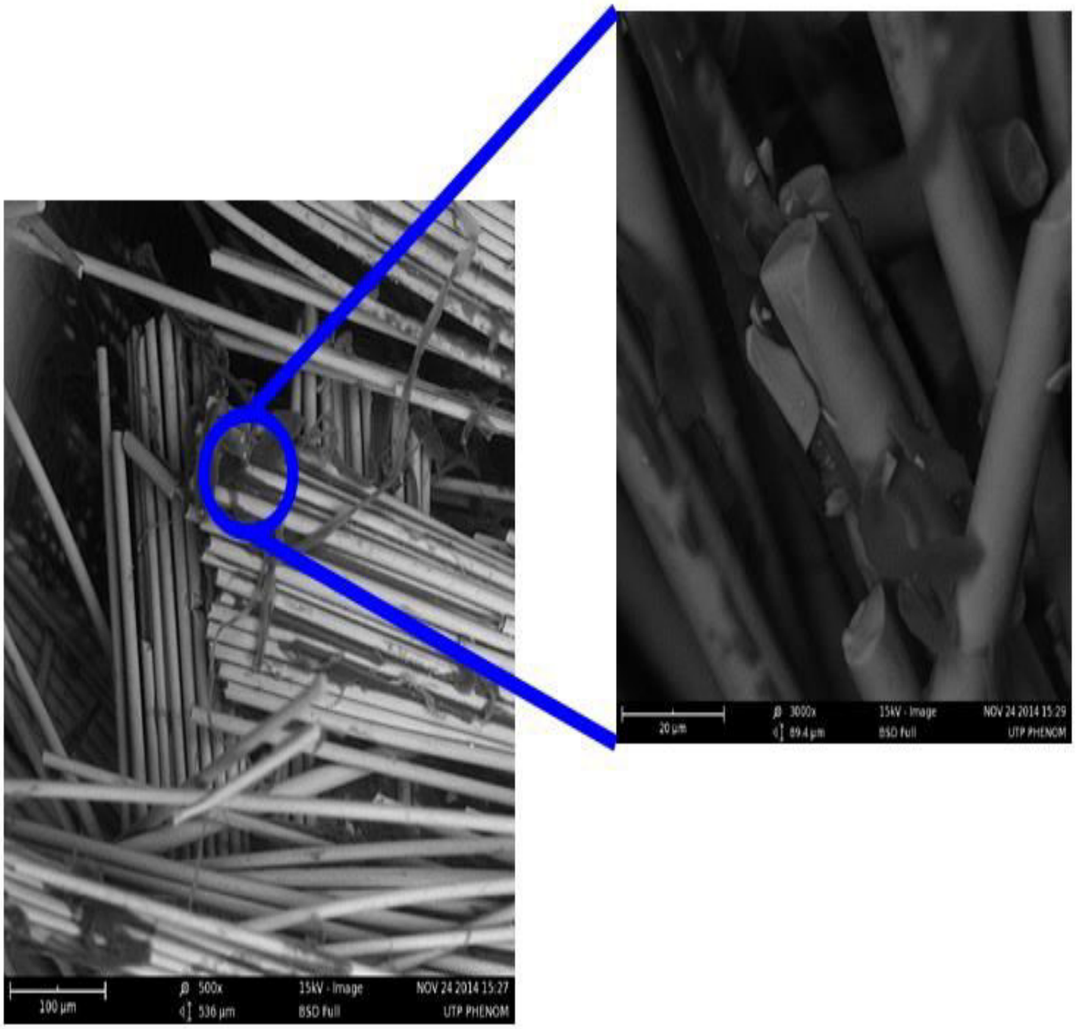


Figure 4.13 (c): SEM micrograph of HDPE 15% glass fiber sample

Less fiber pull-outs are observed for HDPE with 15% glass fiber, based on Figure 4.13 (c) and the glass fibers are **clustered** with one another, increasing the **fiber-fiber and fiber-matrix adhesion**, which results in higher tensile strength and modulus, as recorded.

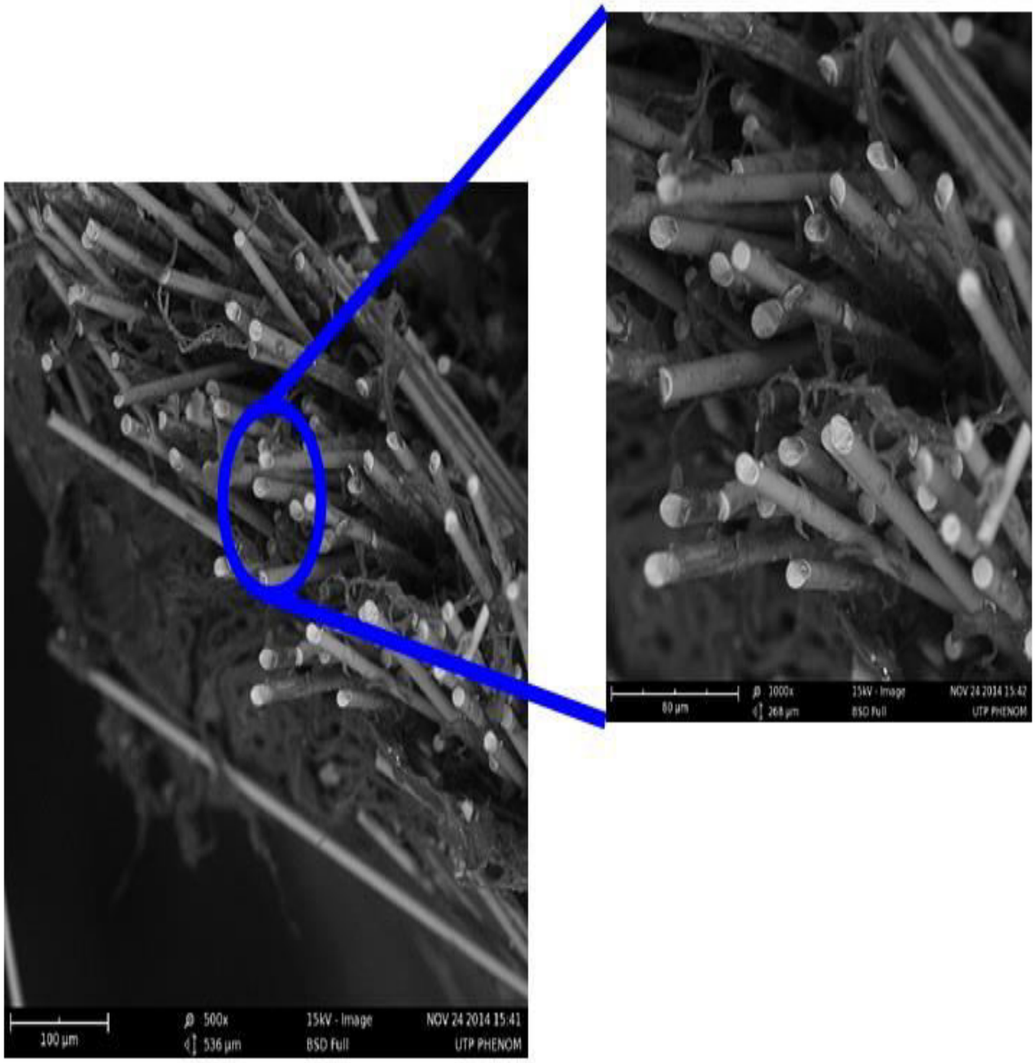


Figure 4.13 (d): SEM micrograph of HDPE 20% glass fiber sample

Based on the surface morphology shown in Figure 4.13 (d), the **compact and regular alignment** of glass fiber inside the HDPE with 20% glass fiber composite can be observed, providing strength and stability to the system.

Generally, based on Figure 4.13 (a), (b), (c) and (d), **fibre pull-out** is a phenomenon commonly seen when fibre-matrix adhesion is not very good between the two materials. This is indicated by the circular voids in the fracture plane. The even patterns of **fiber glass distribution** can be observed thorughout the composites with 15% wt. and 20% wt. fiber glass content, compared to uneven distribution of glass fiber within the 10% HDPE glass fiber composite. Uneven distribution of reinforcement will prevent the HDPE matrix from having more surface contact with glass fiber, hence preventing the fiber glass properties to truly strengthen the composite, as proven by the tensile test results.

These micrographs also reveal that the **fracture surfaces** of tensile test are relatively smooth for lower glass-bead contents, whereas they are much more deformed for higher glass fiber contents. These results were also recorded by Yuan et al. (2002), who justified that glass fiber cannot be easily deformed due to their high stiffness property. In other words, no energy can be absorbed by glass fiber during the tensile test, thus mainly absorbed by the deformation of the HDPE matrix for the fracture.

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

As a conclusion, the required composites of HDPE reinforced glass fiber were successfully synthesized and characterized according to the conditions and mix formulations specified.

- HDPE composite with 20% glass fiber content has the highest tensile strength and modulus values, which are **34.8 N/mm^2** and **5000000 kN/m^2** respectively.
- The percentage of elongation showed an inverse pattern whereby lean HDPE value is **46.3%**, compared to HDPE composite with 20% glass fiber with the value of **13.68%**.
- Decomposition temperature for lean HDPE is the lowest, **413.6°C**, compared to the composites, whereby HDPE with 20% glass fiber content is the most stable with decomposition temperature of **435°C**.
- SEM analysis showed the lean HDPE and HDPE glass fiber composites' microstructure at the fracture surface of tensile test. Fibre pull-out and the distribution of glass fiber were compared based on the data.

Hence it is observed that the addition of glass fiber into HDPE matrix has greatly increased the original properties of tensile strength, modulus, and thermal stability. Meanwhile, the ductile properties were decreased resulting in lower elongation percentage, which indicates that this combination is suitable for static applications, rather than dynamic due to the minimal elongation features.

5.2 Recommendations

For recommendation, the author would like to propose:

- **More variety of fiber glass percentage** should be used in analysis with HDPE to gain a more explicit knowledge regarding the behaviour and property of HDPE fiber glass composite.
- Also, the **chemical resistance** property of this composite should be taken into account in order to suit the desired application for a long-tem basis.
- Further research should be conducted regarding the presence of certain **additives or binders** that would further improve the adhesion between fiber glass and HDPE matrix.

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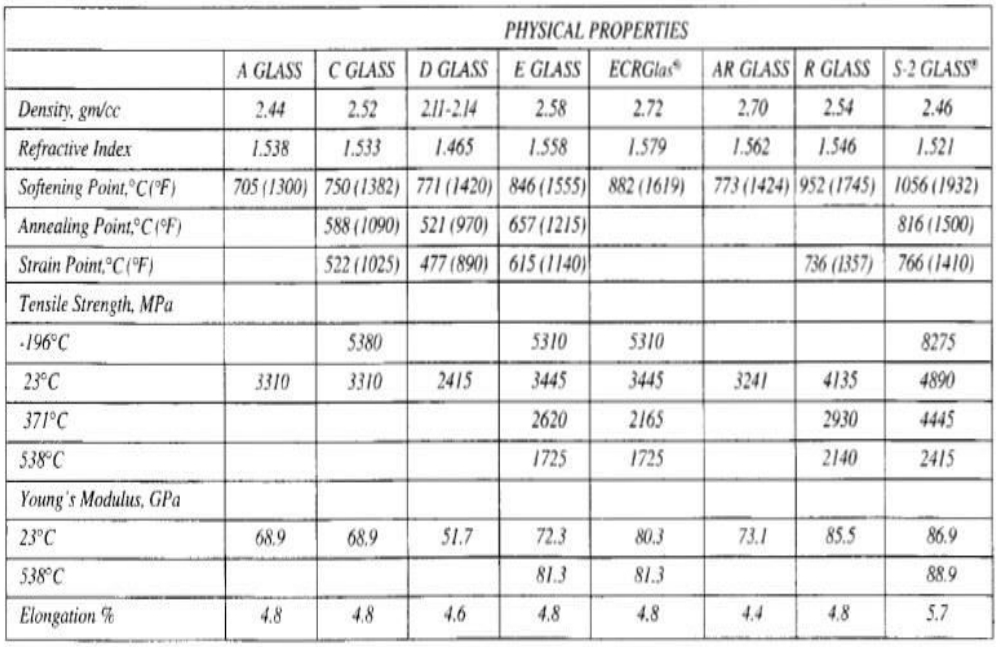
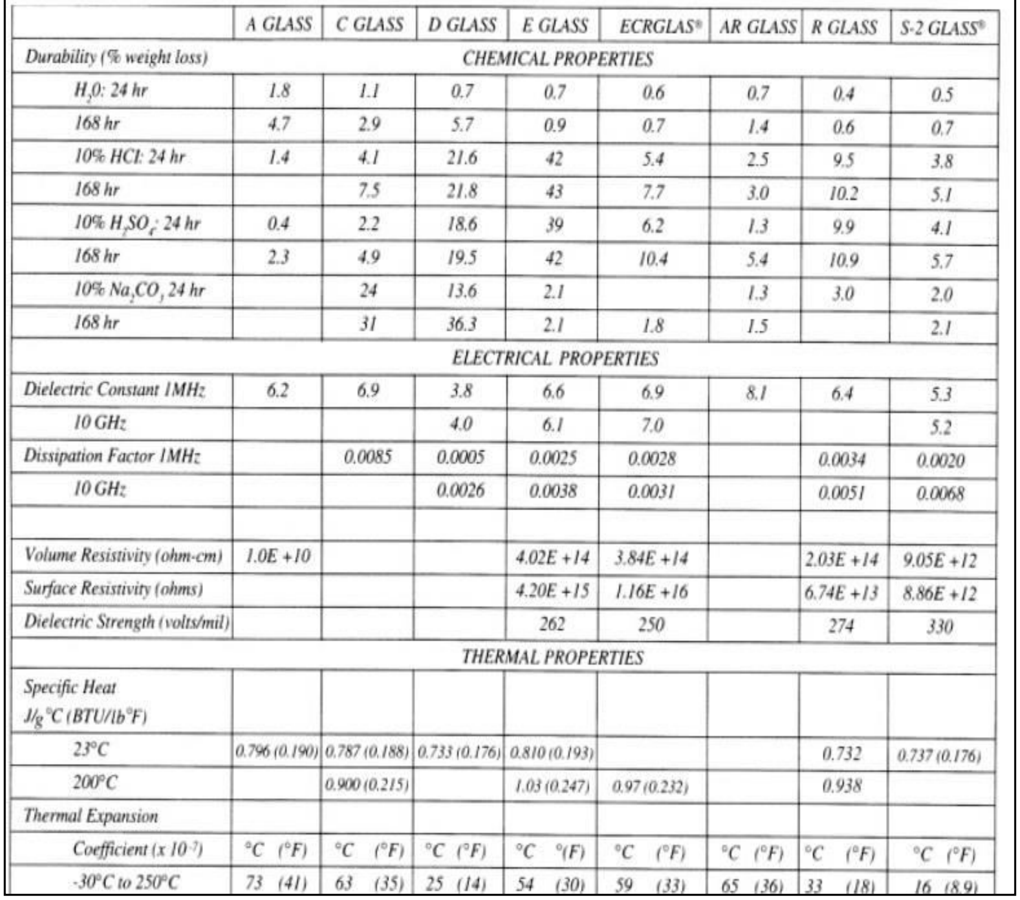
APPENDICES

Appendix 1 - Glass fiber properties



PHYSICAL PROPERTIES								
	A GLASS	C GLASS	D GLASS	E GLASS	ECRGlas®	AR GLASS	R GLASS	S-2 GLASS®
Density, gm/cc	2.44	2.52	2.11-2.14	2.58	2.72	2.70	2.54	2.46
Refractive Index	1.538	1.533	1.465	1.558	1.579	1.562	1.546	1.521
Softening Point,°C(°F)	705 (1300)	750 (1382)	771 (1420)	846 (1555)	882 (1619)	773 (1424)	952 (1745)	1056 (1932)
Annealing Point,°C(°F)		588 (1090)	521 (970)	657 (1215)				816 (1500)
Strain Point,°C(°F)		522 (1025)	477 (890)	615 (1140)			736 (1357)	766 (1410)
Tensile Strength, MPa								
-196°C		5380		5310	5310			8275
23°C	3310	3310	2415	3445	3445	3241	4135	4890
371°C				2620	2165		2930	4445
538°C				1725	1725		2140	2415
Young's Modulus, GPa								
23°C	68.9	68.9	51.7	72.3	80.3	73.1	85.5	86.9
538°C				81.3	81.3			88.9
Elongation %	4.8	4.8	4.6	4.8	4.8	4.4	4.8	5.7

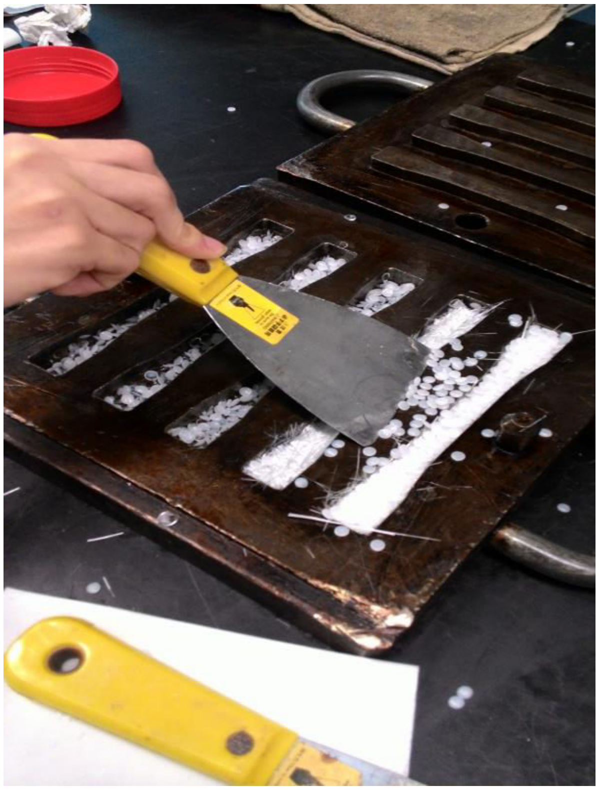


	A GLASS	C GLASS	D GLASS	E GLASS	ECRGLAS®	AR GLASS	R GLASS	S-2 GLASS®
Durability (% weight loss)				CHEMICAL PROPERTIES				
H_2O: 24 hr	1.8	1.1	0.7	0.7	0.6	0.7	0.4	0.5
168 hr	4.7	2.9	5.7	0.9	0.7	1.4	0.6	0.7
10% HCl: 24 hr	1.4	4.1	21.6	42	5.4	2.5	9.5	3.8
168 hr		7.5	21.8	43	7.7	3.0	10.2	5.1
10% H_2SO_4: 24 hr	0.4	2.2	18.6	39	6.2	1.3	9.9	4.1
168 hr	2.3	4.9	19.5	42	10.4	5.4	10.9	5.7
10% Na_2CO_3 24 hr		24	13.6	2.1		1.3	3.0	2.0
168 hr		31	36.3	2.1	1.8	1.5		2.1
				ELECTRICAL PROPERTIES				
Dielectric Constant 1MHz	6.2	6.9	3.8	6.6	6.9	8.1	6.4	5.3
10 GHz			4.0	6.1	7.0			5.2
Dissipation Factor 1MHz		0.0085	0.0005	0.0025	0.0028		0.0034	0.0020
10 GHz			0.0026	0.0038	0.0031		0.0051	0.0068
Volume Resistivity (ohm-cm)	1.0E +10			4.02E +14	3.84E +14		2.03E +14	9.05E +12
Surface Resistivity (ohms)				4.20E +15	1.16E +16		6.74E +13	8.86E +12
Dielectric Strength (volts/mil)				262	250		274	330
				THERMAL PROPERTIES				
Specific Heat J/g°C (BTU/lb°F)								
23°C	0.796 (0.190)	0.787 (0.188)	0.733 (0.176)	0.810 (0.193)			0.732	0.737 (0.176)
200°C		0.900 (0.215)		1.03 (0.247)	0.97 (0.232)		0.938	
Thermal Expansion								
Coefficient ($x 10^{-7}$)	°C (°F)	°C (°F)	°C (°F)	°C °(F)	°C (°F)	°C (°F)	°C (°F)	°C (°F)
-30°C to 250°C	73 (41)	63 (35)	25 (14)	54 (30)	59 (33)	65 (36)	33 (18)	16 (8.9)

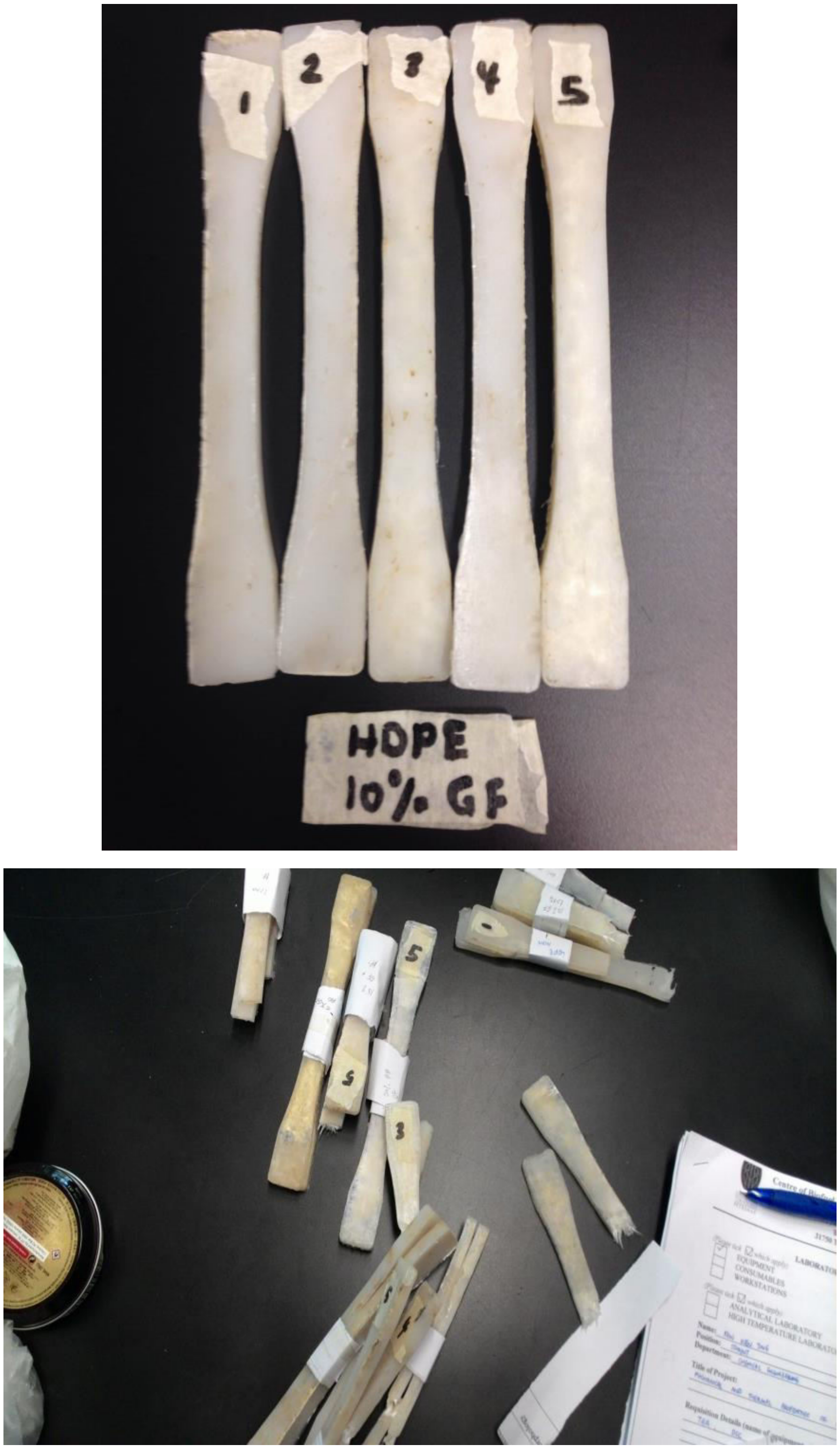
Appendix 2 - Purchasing of materials

Appendix 3 - Samples preparation

Appendix 4: Layering into mold compartments, and heating process

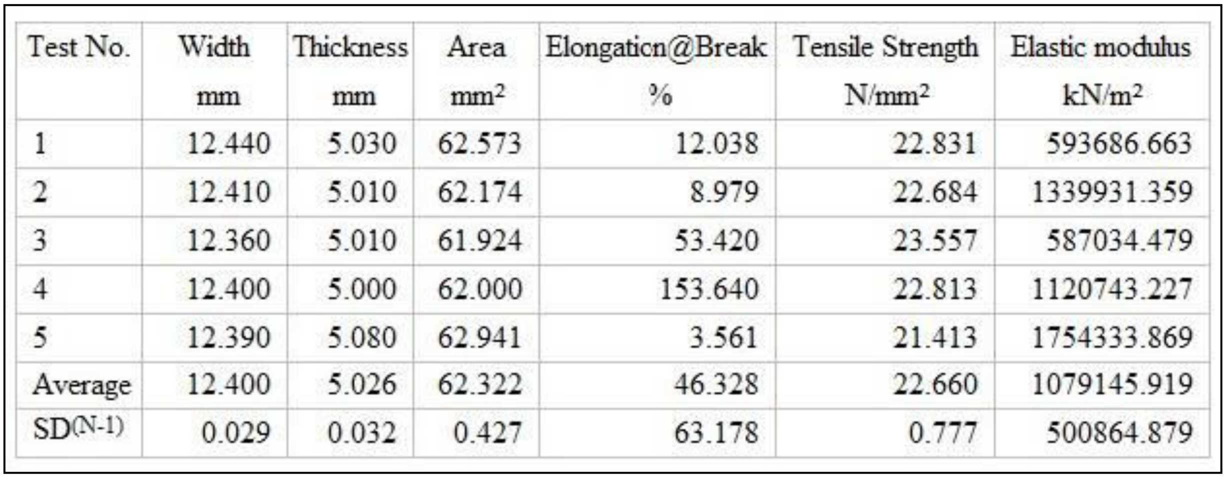


Appendix 5: Prepared samples, before and after tensile test



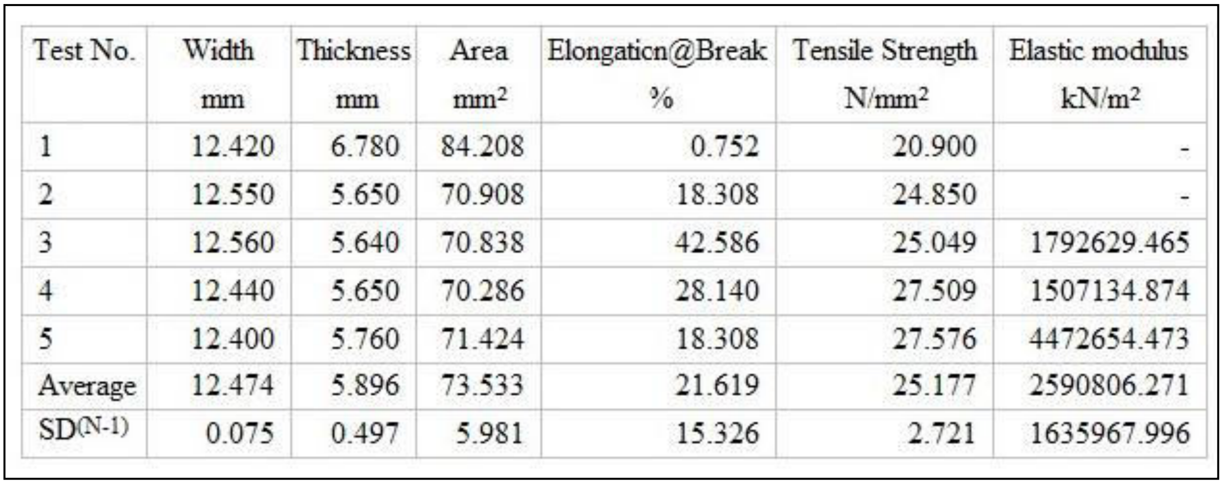
Appendix 6: Tensile Test Reports

Sample: Neat HDPE



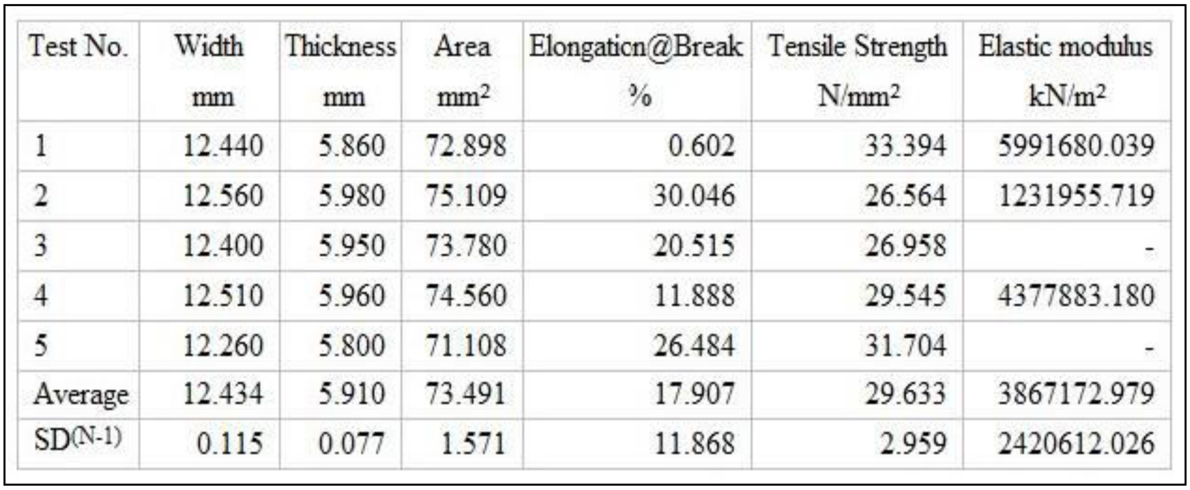
Test No.	Width mm	Thickness mm	Area mm^2	Elongaticn@Break %	Tensile Strength N/mm^2	Elastic modulus kN/m^2
1	12.440	5.030	62.573	12.038	22.831	593686.663
2	12.410	5.010	62.174	8.979	22.684	1339931.359
3	12.360	5.010	61.924	53.420	23.557	587034.479
4	12.400	5.000	62.000	153.640	22.813	1120743.227
5	12.390	5.080	62.941	3.561	21.413	1754333.869
Average	12.400	5.026	62.322	46.328	22.660	1079145.919
$SD^{(N-1)}$	0.029	0.032	0.427	63.178	0.777	500864.879

Sample: HDPE with 10% glass fiber



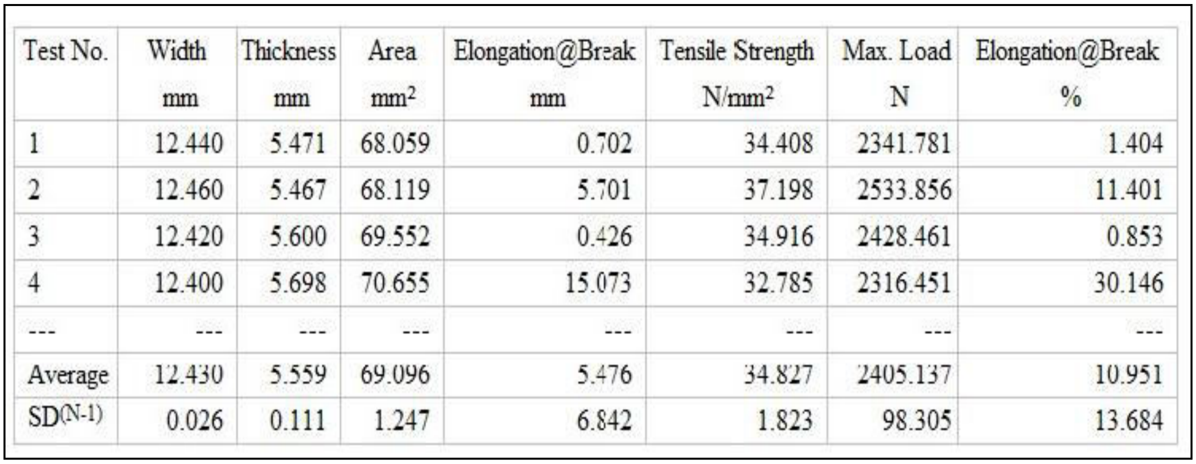
Test No.	Width mm	Thickness mm	Area mm^2	Elongaticn@Break %	Tensile Strength N/mm^2	Elastic modulus kN/m^2
1	12.420	6.780	84.208	0.752	20.900	-
2	12.550	5.650	70.908	18.308	24.850	-
3	12.560	5.640	70.838	42.586	25.049	1792629.465
4	12.440	5.650	70.286	28.140	27.509	1507134.874
5	12.400	5.760	71.424	18.308	27.576	4472654.473
Average	12.474	5.896	73.533	21.619	25.177	2590806.271
$SD^{(N-1)}$	0.075	0.497	5.981	15.326	2.721	1635967.996

Sample: HDPE with 15% glass fiber



Test No.	Width mm	Thickness mm	Area mm^2	Elongation@Break %	Tensile Strength N/mm^2	Elastic modulus kN/m^2
1	12.440	5.860	72.898	0.602	33.394	5991680.039
2	12.560	5.980	75.109	30.046	26.564	1231955.719
3	12.400	5.950	73.780	20.515	26.958	-
4	12.510	5.960	74.560	11.888	29.545	4377883.180
5	12.260	5.800	71.108	26.484	31.704	-
Average	12.434	5.910	73.491	17.907	29.633	3867172.979
$SD^{(N-1)}$	0.115	0.077	1.571	11.868	2.959	2420612.026

Sample: HDPE with 20% glass fiber



Test No.	Width mm	Thickness mm	Area mm^2	Elongation@Break mm	Tensile Strength N/mm^2	Max. Load N	Elongation@Break %
1	12.440	5.471	68.059	0.702	34.408	2341.781	1.404
2	12.460	5.467	68.119	5.701	37.198	2533.856	11.401
3	12.420	5.600	69.552	0.426	34.916	2428.461	0.853
4	12.400	5.698	70.655	15.073	32.785	2316.451	30.146
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Average	12.430	5.559	69.096	5.476	34.827	2405.137	10.951
$SD^{(N-1)}$	0.026	0.111	1.247	6.842	1.823	98.305	13.684