Analysis of Thermal and Mechanical Properties of Polypropylene (PP) with Glass Fibre (GF) Composites

by

Kew Wen Ting

14317

Dissertation submitted in partial fulfillment of

the requirements for the

Bachelor of Engineering (Hons)
(Chemical)

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CERTIFICATION OF APPROVAL

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Approved by,

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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.

KEW WEN TING

ABSTRACT

Polypropylene (PP) is known as a low cost engineering plastics with its outstanding properties. It has been widely used in various industries. Glass fibre (GF) is one of the well-known reinforcing agents. This project was conducted to study the mechanical and thermal properties of GF reinforced PP. The type of GF used was chopped strand mat. The mix formulations of GF reinforced PP that had been carried out in this project were 10 wt% GF, 15 wt% GF and 20 wt% GF. Compression molding machine was used for synthesizing process. By increasing the weight percentage of GF, the tensile strength of the composite was found to be increased. However, the percentage of elongation at break portrayed an inverse pattern. Besides, analysis of thermal properties showed that the thermal degradation temperature and degree of crystallinity increased with the weight percentage of GF. 20 wt% GF reinforced PP exhibited the highest tensile strength and thermal degradation temperature. In addition, the fracture surface morphology of each samples were obtained. It showed that with higher weight percentage of GF had fewer voids content and performed less plastically deformed. Hence, it could be concluded that the objectives of this project were achieved where the mechanical and thermal properties of GF reinforced PP were studied. There were significant improvements on PP's mechanical and thermal properties with the reinforcement of GF.

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I am truly grateful to the people that have helped and guided me throughout the final year project.

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CHAPTER 1

INTRODUCTION

1.1 BACKGROUND

Polypropylene (PP) has been one of the most widely used thermoplastic polymers, characterized by its outstanding cost-to-performance ratio. It is referred to as a low-cost engineering plastic. Its molecular formula is $(C_3H_6)_n$ and its structural formula is shown in Figure 1-1. Most of the commercial PP has an intermediate level of crystallinity between low density polyethylene (LDPE) and high density polyethylene (HDPE). It is produced by polymerization of propylene molecules, the monomer units into very long polymer molecules or chains using stereospecific Ziegler-Natta catalyst. It is well-known as a high-volume commodity plastic.

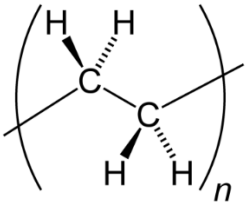

Figure 1- 1 : Structural Formula of Polypropylene

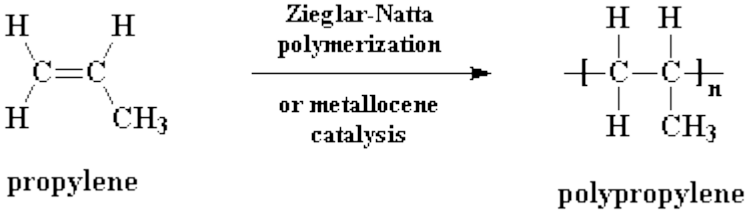

Figure 1- 2 : Structural Formula for the Formation of Polypropylene from Propylene

Polypropylene has excellent physical, mechanical and thermal properties at room temperature. The advantages offered by PP include low specific gravity, chemical resistance, stain resistance, thus it can be used for fibers and fabrics. Besides, it is also widely used for rigid packaging containers and housewares, furnitures, automotive and so on as shown in Figure 1-3. However, there are some limitations for neat polypropylene.

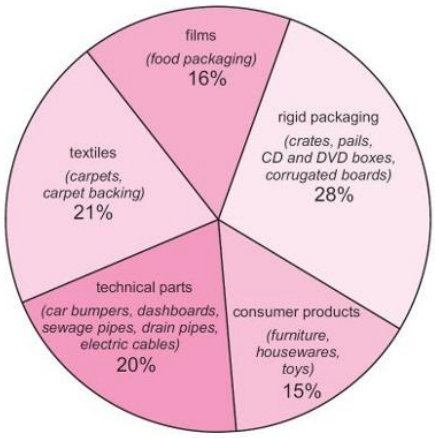

Figure 1- 3 : Usage of Polypropylene [1]

Hence, fibers are introduced to polymer in order to improve the properties to a more desirable material and at the same time it also helps to reduce the undesirable properties as well. Fiber-reinforced plastic composites, with a wide variety of matrix polymers have been developed as an alternative to thermoset fibre composites.

There are several fibers been used to introduce to polymer such as talc, mica, mineral, calcium carbonate etc. Glass fiber (GF) is also one of the common fibers that been used as reinforcing agent. It is the second man-made fibre, next to rayon, to be one of the commercial importances. Among the reinforcing agents, it is mentioned by Dwyer et. al. [2] that E-type glass fibre that made from Pyrex, are known to have higher reinforcing efficiency than talc, calcium carbonate or mica.

1.2 PROBLEM STATEMENT

Neat polypropylene exhibits certain criteria that limits its application in certain industries, thus reinforcement is required to enhance the limiting properties of polypropylene such as moderate strength, low melting temperature where it cannot be ironed when it is been used as materials of clothes and so on.

Although research on glass fibre-reinforced polypropylene has been numerously done and shown that the mechanical properties of PP could be improved with reinforcement of GF [3, 4], however, further study on this composite with chopped strand mat glass fibre is limited. Thus, this project is carried out on PP with different weight percentage of glass fibre chopped strand mat. It is expected that the mechanical and thermal properties of polypropylene will be improvised.

1.3 OBJECTIVES

The objectives of this project are:

- To synthesize and characterize glass fibre (GF) reinforced polypropylene (PP).
- To study the thermal and mechanical properties of GF reinforced PP.
- To characterize the GF reinforced PP for surface morphology.

1.4 SCOPE OF STUDY

The scope of study in this research focuses on the analysis of mechanical and thermal properties of glass fibre reinforced polypropylene.

The manipulated processing variable in this project is the weight percentage of glass fibre to be introduced to polypropylene. The mix formulation of GF reinforced PP are 10% wt GF, 15%wt GF and 20%wt GF. The composite will be synthesized by using compression moulding. Upon the completion of synthesizing, several testing will be performed in order to illustrate the effect of glass fibre addition to the properties of neat PP. The data obtained can then be further analyzed in order to determine its applications in future study.

CHAPTER 2

LITERATURE REVIEW

2.1 PROPERTIES OF COMPONENTS

The components that will be used in this project are polypropylene (PP) and glass fibre (GF). Study and research have been done on these components and the properties of each of them are discussed in this chapter.

2.1.1 POLYPROPYLENE (PP)

Polypropylene (PP) is a semi-crystalline thermoplastic, viscoelastic material that is produced by polymerization of propylene molecules. According to Karian [5], PP has excellent physical, mechanical and thermal properties when it is used in room temperature applications. It is relatively stiff and has a high melting point which is at 130 ^{0}C, its crystalline melting point ranges from 160-170 ^{o}C, low density and relatively good resistance to impact. Besides, it provides good fatigue resistance, good chemical resistance, good environmental stress cracking resistance, good detergent resistance and excellent hardness[6]. As the properties of PPs cover an extensive range, thus its applications are quite diverse.

There are two type of polypropylene, which are homopolymer PP (HPP) and copolymer PP. PP that contains only propylene monomer in the semi crystalline solid form is referred to as HPP while PP containing ethylene as a comonomer in the PP chains is referred as copolymer. Homopolymer PP are more rigid and have better resistance to high temperature than copolymers, however their impact strength at temperature below zero is limited. Applications of homopolymer PP include windshield washer tanks, housing for domestic appliances, clothing, medical fabrics and automotive interior fabrics. As for copolymerized PP, it gives softer feel to film and fibre products compared to homopolymer. It is mostly used for battery cases, bumper filler supports,

interior trim, gloves box, package trays and window mouldings, office chair, disposable container, boxes and housing appliances.

Furthermore, as PP is considered as semi-crystalline polymer, thus its crystallinity is also one of the concerns. The degree of crystallinity and crystal structure in a polymer depends on its thermal history. The formation of crystals can be suppressed by rapid cooling and give a tough clear product. In other words, slow cooling of the product leads to a brittle and hazy product. However, different degree of crysallinity has different advantages. PP with higher crystallinity has better hardness, modulus, strength, chemical resistance, barrier properties etc. while low crystallinity has better transparency and good processibility.

As for the safety of processing PP, it is claimed that it does not cause hazardous to health, but it can release volatile organic compounds into the surrounding air during high-temperature processing.

2.1.2 GLASS FIBRE

According to Mallick [7], glass fibre is one of the most common reinforcing fibre. It is the second man-made fiber, after rayon, to be of commercial importance. It has been widely used in commercial composite applications due to its competitive price, high tensile strength, good chemical resistance, excellent insulating properties and ease processing quality.

Glass fibre is an amorphous (non-crystalline) material consisting of silica (SiO_2) as the principal ingredient with other various oxide components such as Al_2O_3, CaO, B_2O_3 etc. There are various types of glass fibre which formed from different composition of oxides. However, E- glass is the most popular and most widely used in fibre-reinforced plastic industry due to its comparatively low cost, though S –glass has higher tensile strength and higher modulus than E-glass [8]. The softening point of E-glass is above 1500ºF. This temperature is beyond any reaction and processing temperature of polymer-based composite. Furthermore, in PP, E- glass is known to

exhibit higher reinforcing efficiency compared to other reinforcing agents such as talc, calcium carbonate, or mica [2].

Besides, there are varieties of glass fibre available in the market. They are chopped strand, chopped strand mat, continuous strand mat, roving and yarn. The most common used glass fibre is chopped strand mat where chopped strand are randomly oriented and distributed over a conveyor belt with an organic binder to form loosely bound open mat [6].

The comparative properties of three most common glass types are shown in Table 2-1.

Table 2- 1 : Comparative properties of three most common glass types[6]

Type of Fibre Glass	**Tensile Strength (Ksi)**	**Tensile Modulus (Msi)**	**Elongation at Failure (%)**	**Density (gm/cm^3)**	**Coefficient of Thermal Expansion (10^{-6} °C)**
E-Glass	500	11.0	4.7	2.58	4.9-6.0
S-Glass	650	12.6	5.6	2.48	2.9
Quartz	490	10.0	5.0	2.15	0.5

2.2 COMPOSITE MATERIAL

A composite material is made up of a combination of two or more distinct materials that provides a unique combination of properties; generally the properties of composite are significantly better than constituent material properties. According to Mazumdar [9], the purpose of fibres in a composite are:

- To bear the load of composite, where 70 to 90% of load is carried by fibres
- To improve the structural properties in composites such as the stiffness, strength and thermal stability
- To provide electrical conductivity or insulation based on the type of fibre

In addition, based on Hasan and Emam [10], they stated that “some of the composites offer the advantage of being tailorable so that properties, such as strength stiffness, can easily be changed by changing the amount or orientation of the

reinforcement material", The glass fibre/ polyester composite material has achieved better characteristics for dynamic forces applied in impact and hardness characteristics in their research on "Study of Impact Energy and Hardness on Reinforced Polymeric Composites".

Furthermore, it is claimed by Rahman et.al. [11] that glass fibre reinforced polymers have been extensively used in automotive and aerospace industries because of their high strength and low-weight characteristics.

2.3 GLASS FIBRE – REINFORCED POLYPROPYLENE

Based on Dwyer et.al [2], chemically coupled glass fibre- reinforced PP exhibits better tensile strength, stiffness, and heat deflection temperature compared to unmodified analogues. It is mentioned that glass fibre- reinforced PP have successfully replaced engineering thermoplastics such as polycarbonate, acrylonitrile-butadiene-styrene (ABS), polyesters etc. in various applications.

According to Jang and Lee [3] ,a research on "The Effect of Fibre Content on the Mechanical Properties of Glass Fibre Mat/ Polypropylene Composites" had been done. It showed that as glass fibre content increased the tensile and flexural modulus of the glass fibre mat/ PP composite showed a linear increment. The tensile and flexural strength modulus increased up to glass fibre content of 15-20 vol% and then decrement occurred with further glass fibre composition.

Furthermore, studies have been done on different effect of glass fibre parameter by Chu [12] .The parameters were the input chopped length of glass fibre, fibre diameter and total glass content. It was stated that if the fibre length was shorter than critical fiber length, there was not much reinforcement effect. As for fibre diameter, it was shown that the tensile and unnotched Izod impact strengths and tensile strain of glass fibre-reinforced polypropylene decreased with the increase of diameter.

In addition, based on the research that had been done by Thomason [13], the influence of fibre length and concentration on the properties of glass fibre reinforced

polypropylene was studied. The study was carried out by using injection moulding with long glass fibre reinforced polypropylene. The glass fibre content is in the range of 0-73wt %. It was shown that the composite strength and impact resistance increased and exhibited the best performance in the 40-50wt% reinforcement content range.

Furthermore, in the research of Thomason and Groenewoud [14] on another paper, the thermal properties of glass fibre reinforced polypropylene is carried out with different fibre length and concentration. It was found that the dynamic mechanical analysis (DMA) showed the higher concentration / longer fibres enhanced the high temperature modulus retention.

2.4.1 THERMAL ANALYSIS

According to Gill, Moghadam and Ranjbar [15], calorimetry is a fundamental method to determine the thermal properties of materials and is the only way to direct determine the enthalpy associated with the process of interest. Differential Scanning Calorimeter (DSC) is the most widely used apparatus.

DSC is commonly used to monitor and determine the changes of phase transitions. In this experiment, the sample cell will be placed with a reference cell, where energy will be introduced simultaneously. The temperatures of both cells are raised identically over time until an extent where the input energy is differ while maintaining the same temperature of them. The difference in the input energy will be the amount of energy absorbed or released by the molecule in the sample. The typical DSC diagram is shown in Figure 2-1. DSC usually will be used in conjunction of Thermal Gravimetric Analysis (TGA).

TGA is usually used to determine the characteristics of materials that show either mass loss or gain due to decomposition, oxidation or moisture loss with the function of increasing temperature or function of time. It works in a way that the sample is heated continuously; at certain temperature, components of the composites will decompose and result in mass change. The mass of sample is weighed throughout the process and the result of mass loss will be plotted against the temperature.

By having this two thermal analysis, thermal behavior of the composites can be determined while the melting, boiling and decomposition points of the composites will be found.

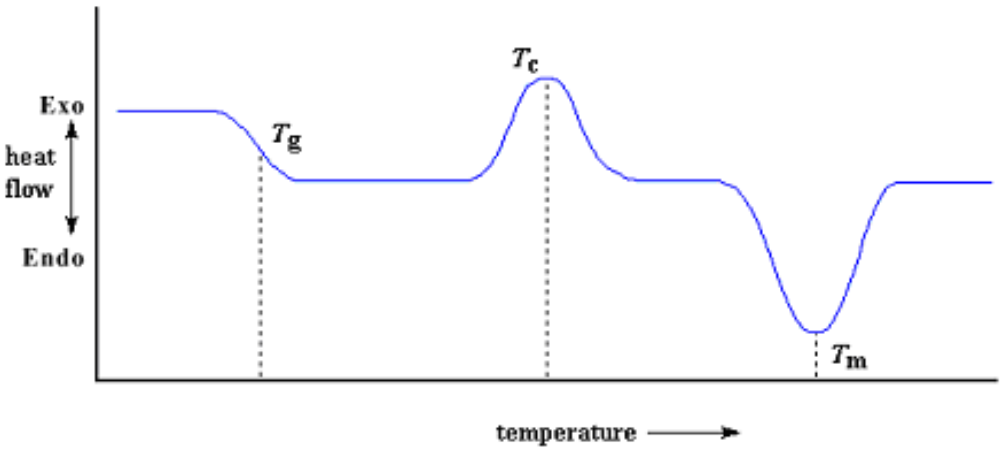

Figure 2- 1: Typical DSC Generic Diagram[16]

2.4.2 MECHANICAL ANALYSIS

Mechanical analysis is performed in order to test the physical properties of the material under variety of testing regimes. Tensile properties such as tensile strength and tensile modulus can be determined using tensile tests. These tests measure the force required to elongate a specimen to its breaking point, material properties such as the material tensile strength, elongation at break and the elastic modulus can be determined. Tensile strength is the maximum force measured divided by the original cross-sectional area. This point is also known as Ultimate Tensile Stress (UTS). The formula is shown as below.

$$Tensile\ strength\ = \frac{(load\ at\ break)}{(original\ width)\ (original\ thickness)}$$

Elongation at break is the observed strain, percent strain or draw ratio that occurs immediately prior to the sample failure. As for elastic modulus, it is defined as the ratio of tensile stress to tensile strain.

2.4.3 CHARACTERIZATION ANALYSIS

Characterization analysis is used to understand the external morphology of the composite materials, the chemical composition and the crystalline structure as well as the orientation of materials making up the sample. The equipment can be used to carry out this analysis is scanning electron microscope (SEM) and Fourier Transformation Infra-Red (FTIR). Based on Ota et al. [4], GF and PP-30wt% GF composites revealed higher amount of glass fibres, less voids and small void size. The SEM result obtained from Ota et al. [4] is shown in Figure 2-2.

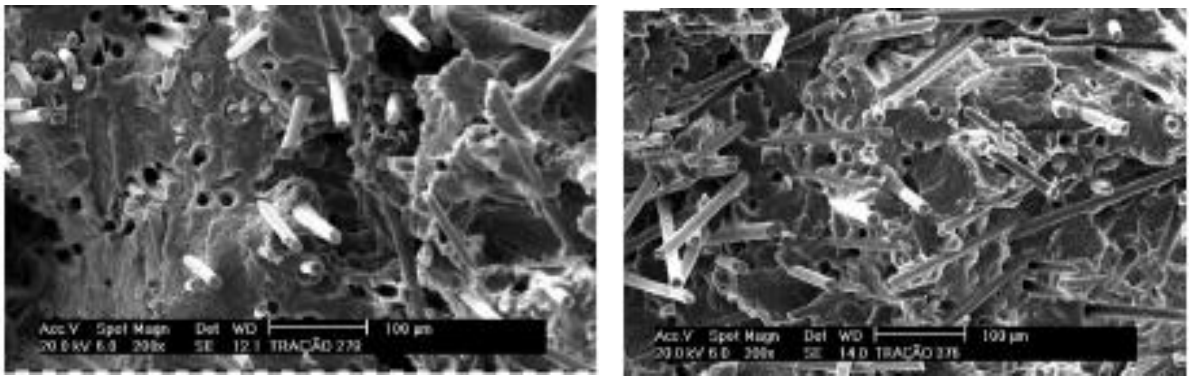

Figure 2- 2: SEM pictures - PP-20wt%GF (left) and PP-30wt% GF (right) at fractured surface [4]

CHAPTER 3

METHODOLOGY

3.1 MATERIAL

Polypropylene homopolymer was obtained from Lotte Chemical Titan (M) Sdn. Bhd. and glass fibre was obtained from local vendor at Jelapang, Malaysia. It is 450gm chopped strand mat. The properties of PP provided by the company are shown in Table 3-1. This specific type of grade is used in the production of the film extrusion and plastic bag manufacturing.

Table 3- 1 : Product Data of Polypropylene

Typical Resin Properties	Unit	Product Data	ASTM Method
Melt Flow rate, at 230ºC	g/10min	11	D 1238
Density	g/cm^3	0.9	D 1505
Tensile Strength at Yield	kg/ cm^2	360	D 638
Elongation at Yield	%	12	D 638
Flexural Modulus	kg/cm^2	17000	D790 B
Notched Izod Impact Strength at 23ºC	kgcm/cm	3.3	D 256A
Heat Deflection Temperature at $4.6kg/cm^2$	ºC	95	D 648
Rockwell Hardness	R scale	94	D785 A
Water Absorption after 24 Hours	%	0.02	D 570

3.2 EXPERIMENTAL

The experimental work of this project can be divided into three phases, which are characterization of raw material, synthesizing process and product characterization. The commercial polypropylene will first be characterized in order to ensure its composition. Then, synthesis process/ molding process can be carried out. Upon the completion of synthesizing process, the samples will then be tested with various analyses to obtain the properties. Those analyses include mechanical analysis, thermal analysis and characterize analysis. The summarized flowchart of this project is shown in Figure 3-1.

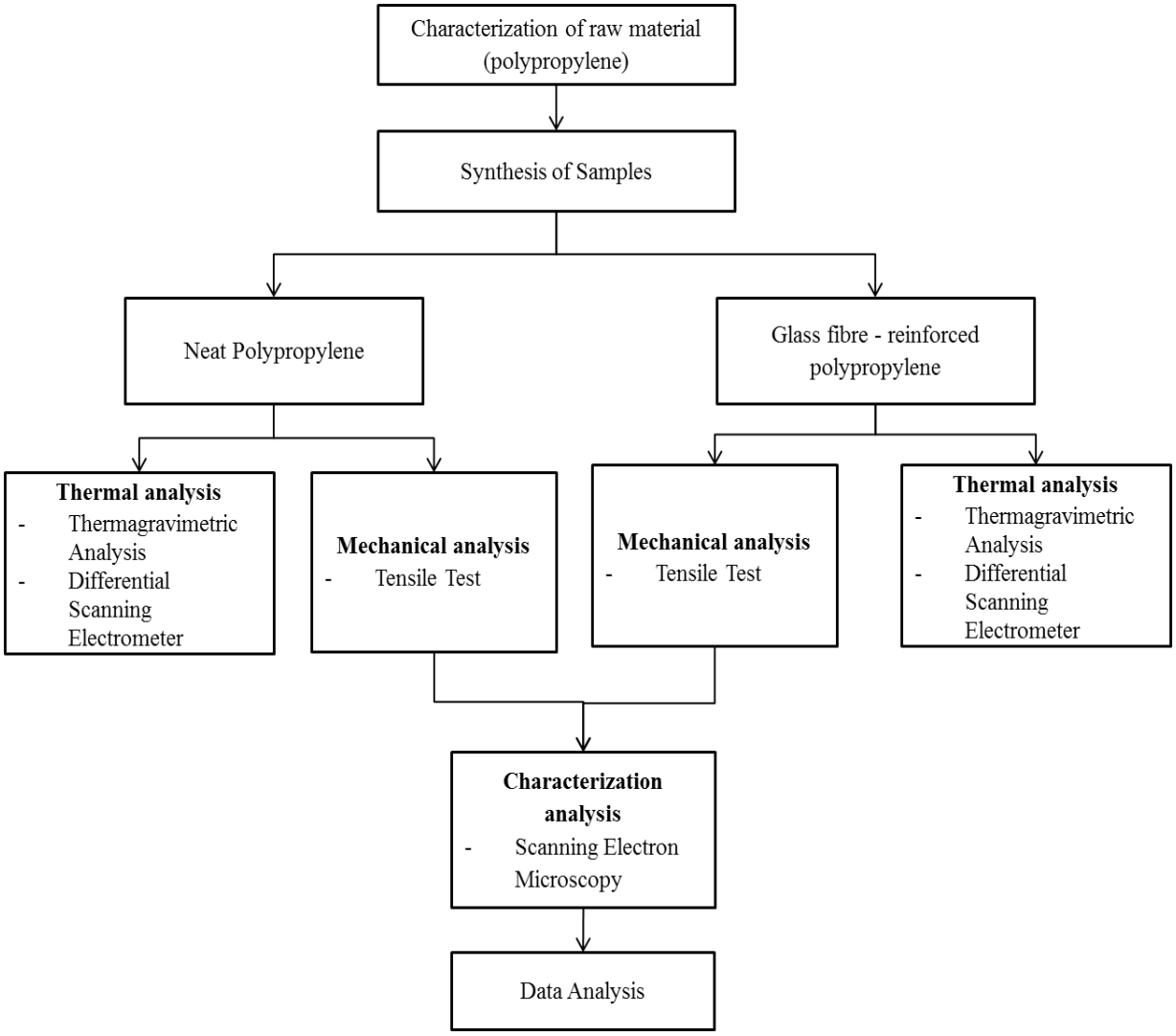

Figure 3- 1 : Overall Flowchart for the Project

3.2.1 CHARACTERIZATION OF RAW MATERIAL (POLYPROPYLENE)

Before carrying out the synthesizing process, Scanning Electron Microscope (SEM) and Fourier Transform Infrared Spectroscopy (FTIR) on lean polypropylene need to be carried out. This is to observe the surface morphology of polypropylene pellet and to ensure the composition of it. According to the material safety datasheet provided, it is stated that there is presence of additives. Hence, determination on the content of the additives is required by using Electron Diffraction Spectroscopy (EDS).

3.2.2 SYNTHESIS PROCESS

In this project, compression moulding machine is used to synthesis the samples. A standard mould is used to produce the neat polypropylene and polypropylene/glass fibre composites. The dimension of the mould is shown in Figure 3-2. It is a standard dimension to be used for ASTM D638 (Standard Test Method for Tensile Properties of Plastics).

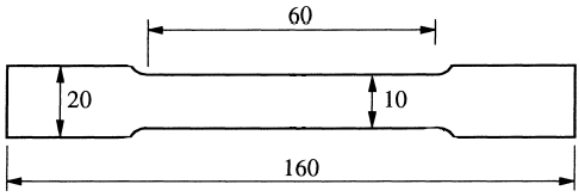

Figure 3- 2 : Dimension (mm) of Tensile Test Specimen

Generally, there are three steps for synthesis process, which are pre-heating, moulding, and cooling down. First, the mould will be preheated to the melting temperature of polypropylene, and then materials will be placed into the mould and been heated and compressed for 30 minutes. The temperature used is 190ºC and the compression pressure is 9 ton force. They are kept constant for all the samples synthesizing as well as the curing time and cooking time.

However, the weight percentage of PP and glass fibre is manipulated in order to determine the best percentage combination for the composite. The composition of the composite is shown in Table 3-2.

Table 3- 2: Weight percentage of polypropylene /glass fibre composites

Weight Percentage of Polypropylene (%)	Weight Percentage of Glass Fibre (%)
100	0
90	10
85	15
80	20

In order to obtain the desired composition, the weight of neat polypropylene that produced at 9 ton force is measured. It is then be used to calculate the weight of glass fibre that should be added to the composite based on the percentage shown in Table 3-2. The formula used to measure the composition of polypropylene and glass fibre is shown as follows.

$$Weight\ of\ glas\ fibre = \frac{\sum_{i=1}^{n=5} x_i}{n} \times desired\ percentage$$

Where x: Pure PP sample weight.

Upon completing the syntheses, the samples are then sent for mechanical analysis, thermal analysis and characterization analysis. All the data obtained from the analyses are discussed in the results section.

3.2.3 MECHANICAL ANALYSIS

In this study, tensile test is carried out to determine the tensile strength and elongation at break. All the mechanical property testing is performed at room temperature and measured in accordance with the procedures in ASTM D-638, using five ASTM specimens at a crosshead rate of 10mm/min. The results were compared with the standard reference mould which is the pure PP sample.

3.2.4 THERMAL ANALYSIS

The thermal properties of PP/ glass fibre composite will be analysed using Thermagravimetric Analysis (TGA) and Differential Scanning Electrometer (DSC). TGA is carried out to study the thermal stability of the samples while DSC is used to monitor and determine the changes of phase transitions for the composites. The pure sample's results were used as standard reference. The changes in melting point and other various thermal elements were then be studied and co-related to the chemical structure influenced by the glass fibre and analyse how different weight percentage affects the thermal stability.

Besides, the percentage of crystallinity of composites was also determined after performing Differential Scanning Calorimeter as the crystallinity of a material influences many of its characteristics, including mechanical strength, opacity, and thermal properties. The equation of determining the crystallinity percentage is shown as below.

$$Percentage\ of\ Crystallinity = \frac{Heat\ of\ Fusion\ of\ sample, \Delta H_{f,x}}{Heat\ Fusion\ of\ 100\%\ crystal, \Delta H_f}$$

3.2.5 CHARACTERIZATION ANALYSIS

In this analysis, Scanning Electron Microscopy (SEM) is used to study the characterization of the composites. It is used to observe the fracture surface morphology after tensile test has been done.

3.2.6 SUMMARY OF PROPERTIES ANALYSIS

The summary of neat polypropylene and GF reinforced PP characterization is presented in Table 3-3.

Table 3- 3 : Properties Analysis for Glass Fibre Reinforced Polypropylene

Analysis	Model	Function
Tensile Test	ASTM D 638	Tensile strength
Thermogravimetric Analysis (TGA)	Pyris 1	Thermal stability
Differential Scanning Calorimeter (DSC)	Pyris 1	Thermal stability
Scanning electron microscopy (SEM)	Phenom	Morphology

CHAPTER 4

RESULTS AND DISCUSSION

4.1 CHARACTERIZATION ANALYSIS OF RAW MATERIAL

In this section, SEM and FTIR were used to study the surface morphology and the surface functional groups of each sample. The results obtained in this test were discussed in the following sections.

4.1.1 SURFACE MORPHOLOGY OF NEAT POLYPROPYLENE

Morphology of neat polypropylene had been observed using SEM with two different magnitudes. The results obtained were displayed in Figure 4-1. The grey areas in the figure were mainly carbon and hydrogen element, while the white spots composed of silicon, carbon, oxygen and nitrogen which were determined through Electron Diffraction Spectroscopy (EDS).

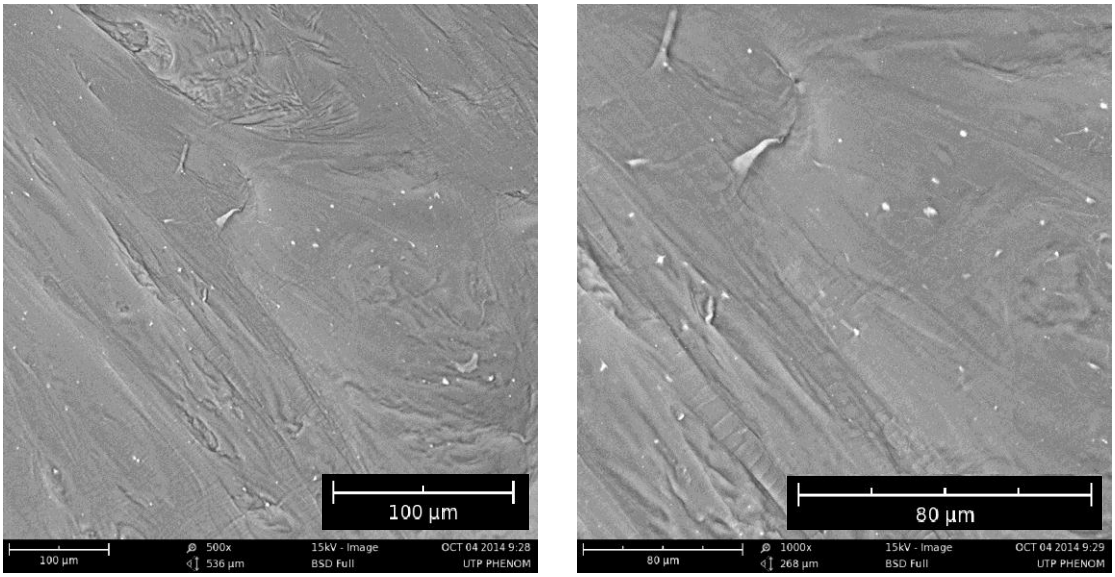

Figure 4- 1: Morphology of neat polypropylene scanned with different magnification

EDS analysis was performed on the SEM result with 80µm magnification, where four white spots were marked and undergone analysis. The reports of analysis were shown in Figure 4-3 to Figure 4-6 where they were the graphs of concentration certainty and Table 4-1 to Table 4-4 which showed the details of the graphs. Based on the results, it could be observed that the PP pellet was clean and consisted of about 98% of carbon and hydrogen. The rest of the elements were expected to be the additives of polypropylene.

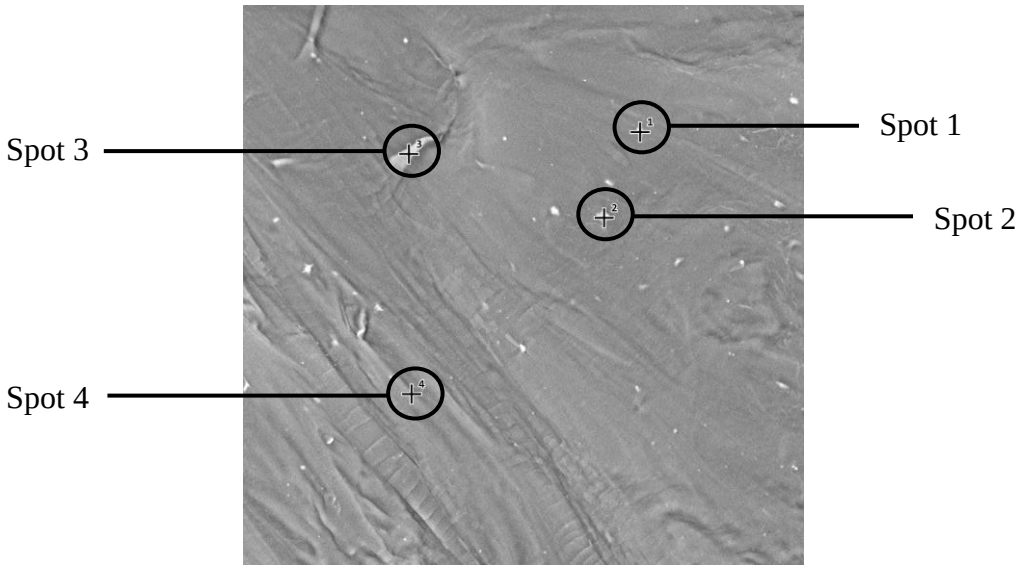

Figure 4- 2: Analysis of Electron Diffraction Spectroscopy (EDS) on Neat Polypropylene

(i) Spot 1

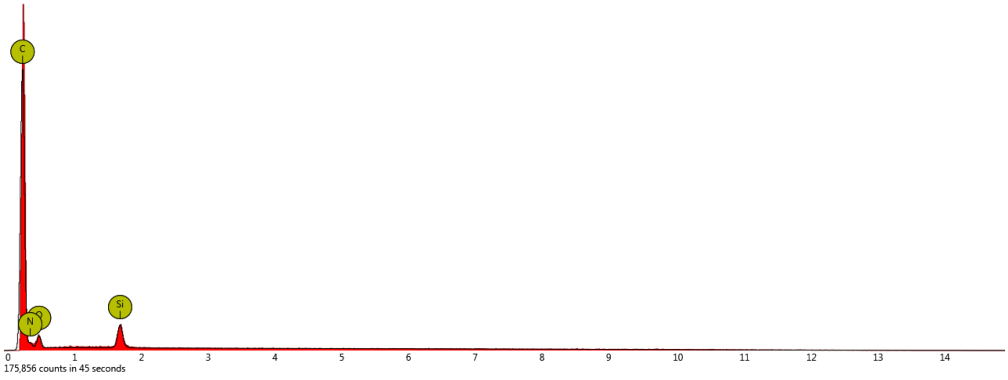

Figure 4- 3: Graph of Concentration Certainty at Spot 1

Table 4- 1: Details of the Elements at Spot 1

Element Number	Element Symbol	Element Name	Confidence	Concentration	Error
6	C	Carbon	100.0	45.6	0.4
14	Si	Silicon	100.0	5.2	1.3
8	O	Oxygen	100.0	36.8	2.1
7	N	Nitrogen	100.0	12.5	3.5

(ii) Spot 2

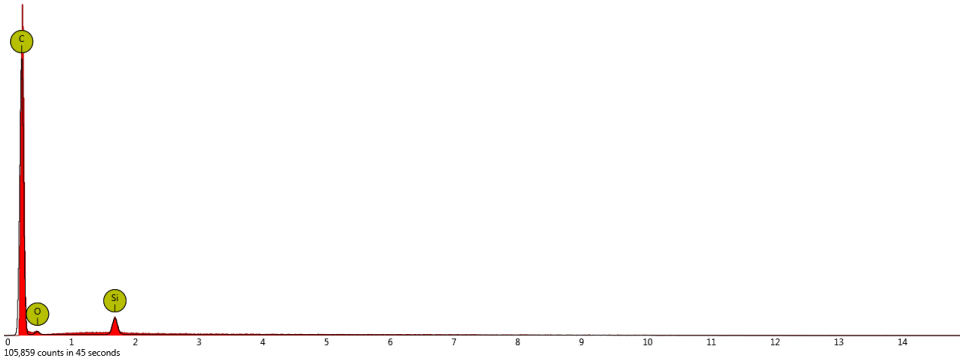

Figure 4- 4: Graph of Concentration Certainty at Spot 2

Table 4- 2: Details of the Elements at Spot 2

Element Number	Element Symbol	Element Name	Confidence	Concentration	Error
6	C	Carbon	100.0	74.6	0.4
14	Si	Silicon	100.0	6.0	1.6
8	O	Oxygen	100.0	19.3	4.8

(iii) **Spot 3**

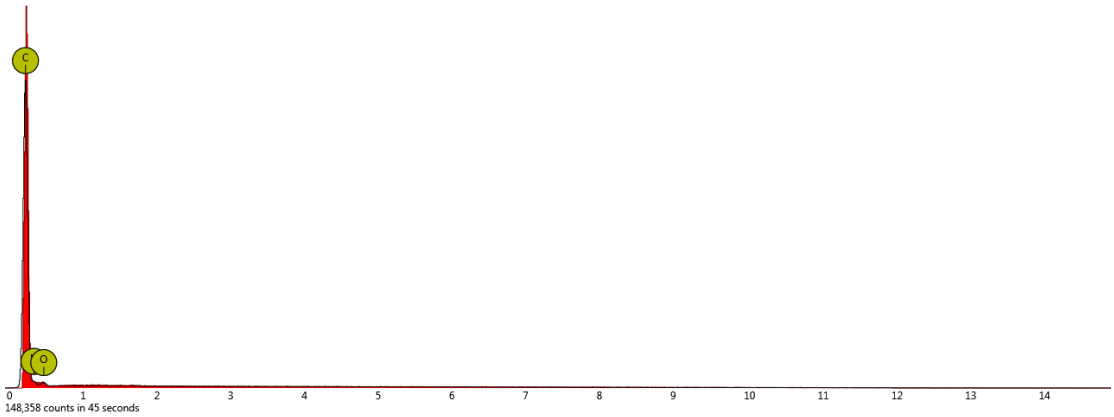

Figure 4- 5: Graph of Concentration Certainty at Spot 3

Table 4- 3: Details of the Elements at Spot 3

Element Number	Element Symbol	Element Name	Confidence	Concentration	Error
6	C	Carbon	100.0	55.5	0.4
7	N	Nitrogen	100.0	20.6	4.1
8	O	Oxygen	100.0	23.9	4.3

(iv) Spot 4

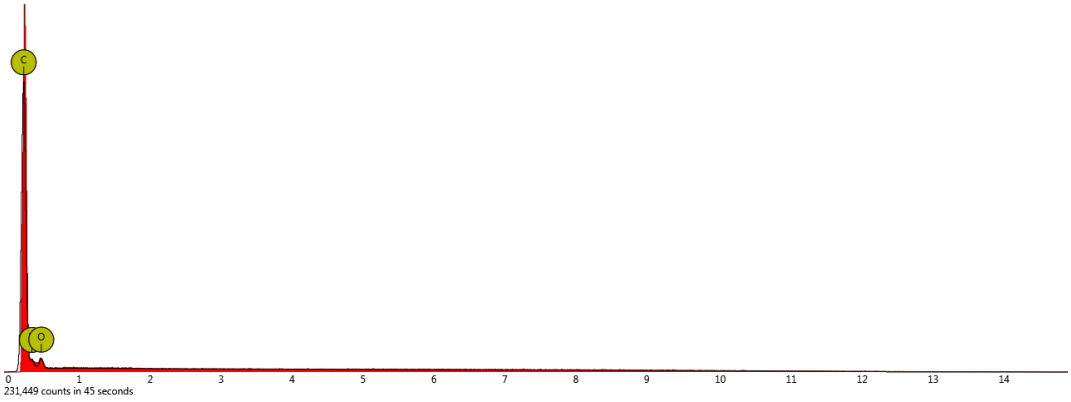

Figure 4- 6: Graph of Concentration Certainty at Spot 4

Table 4- 4: Details of the Elements at Spot 4

Element Number	Element Symbol	Element Name	Confidence	Concentration	Error
6	C	Carbon	100.0	40.9	0.4
7	N	Nitrogen	100.0	21.5	2.6
8	O	Oxygen	100.0	37.6	2.4

4.1.2 FOURIER TRANSFORMATION INFRA-RED (FTIR) OF NEAT POLYPROPYLENE

FTIR was carried out to determine the functional groups that appeared in the neat polypropylene. Figure 4-7 shows the result obtained from FTIR.

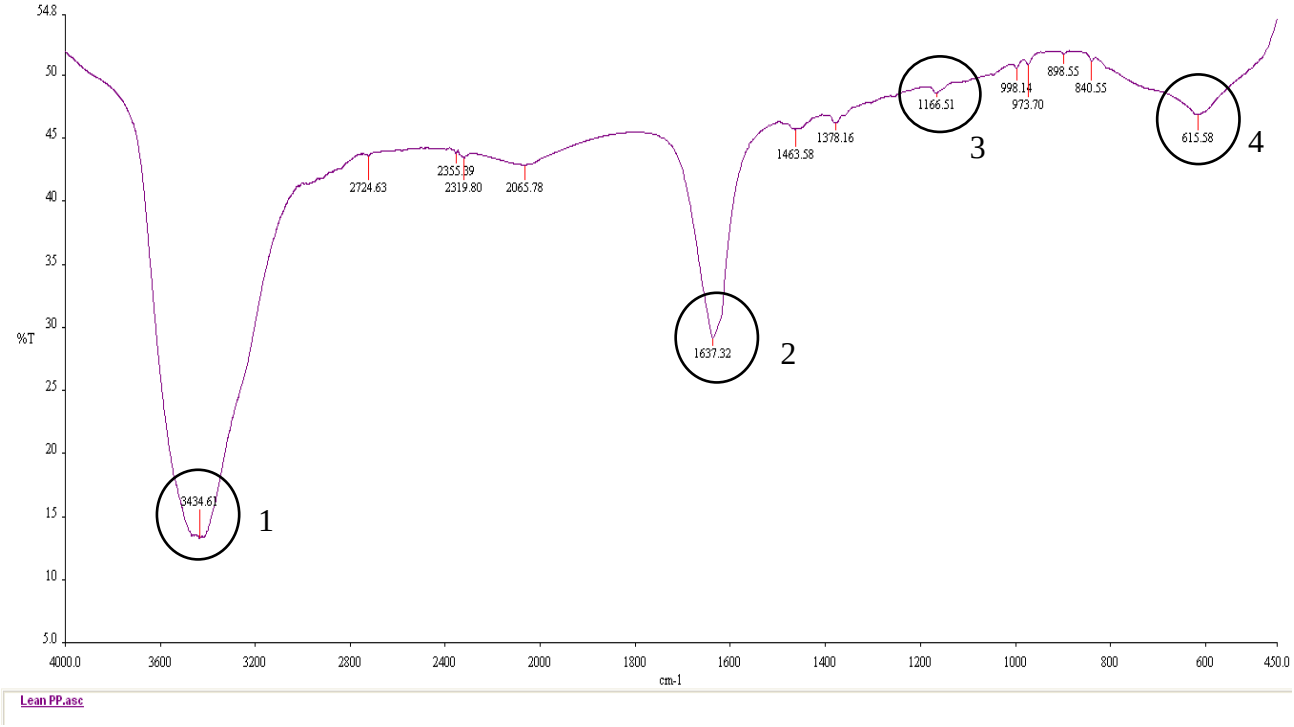

Figure 4- 7: FTIR Result of Neat Polypropylene

The peak in Figure 4-7 indicated the particular of functional group that appeared in mid-infrared region. Based on the figure, there were four significant peaks that should be analyzed. By cross referencing, the results were tabulated in Table 4-5. In the range of 3600-2900, there was alkane carbon- hydrogen bond which was the main element of polypropylene. It was made up of carbon and hydrogen chain. As for the second peak, it was in the range of 1700-1400, which showed the carbon to carbon bond. It was similar to the initial bonding in polypropylene that shown in Figure 1-1. As for the third peak, it fall in the range of 1250-1020, which consisting carbon to nitrogen bond. This could be explained by the result of EDS which showed that there was nitrogen element in the commercial polypropylene.

Table 4- 5: Analysis on FTIR Result

Peak No.	Adsorption Peak	Type of bond	Bond	Appearance
1	3600-2900	Alkanes	C-H	Strong
2	1700-1400	Acyclic	C-C	Weak
3	1250-1020	Aliphatic Amines	C-N	Medium
4	700-610	Alkynes	C-H	Broad-Strong

4.2 PROPERTIES ANALYSIS OF GLASS FIBRE REINFORCED POLYPROPYLENE

Upon the completion of synthesizing process, the prepared glass fibre reinforced polypropylenes were used for various analyses as mentioned before. The results obtained from the analysis are discussed in the following sections.

4.2.1 MECHANICAL ANALYSIS - TENSILE TEST

As mentioned in the methodology, tensile test were carried out using ASTM D 638 method to test the tensile strength of samples. The average results were tabulated in Table 4-6 and graphed in Figure 4-8 and Figure 4-9.

Based on Table 4-8, it showed that with the addition of glass fibre to polypropylene, the tensile strength increased while elongation at break decreased. According to Figure 4-8, the increase factor for 10wt% glass fibre was about 1.437, 15wt% glass fibre was 1.629 while for 20wt% glass fibre was 2.525 compared to the tensile strength of neat PP. It showed that the crosslinking between PP and glass fibre had increased the tensile strength significantly as it was expected. This was similar to the result that had been done in [3, 4, 11], which stated that mechanical properties of polypropylene was improvised with the reinforcement of glass fibre.

Based on Gedney [17], elongation at break is inversely proportional to tensile strength. This is because the greater a material's tensile strength, it requires more force to stretch the material. The results obtained showed the similar trend as mentioned where with the increase of tensile strength, the elongation at break decreased. This is mainly due to the resistance offered by the fibre during recoiling of the chain.

Table 4- 6: Results of Tensile Test using ASTM D638

Sample Reference	Tensile Strength (N/mm^2)	Elongation at Break (%)
Neat Polypropylene	18.872	1.240
PP + 10wt% Glass Fibre	27.110	1.127
PP + 15wt% Glass Fibre	30.750	1.090
PP + 20wt% Glass Fibre	47.654	1.018

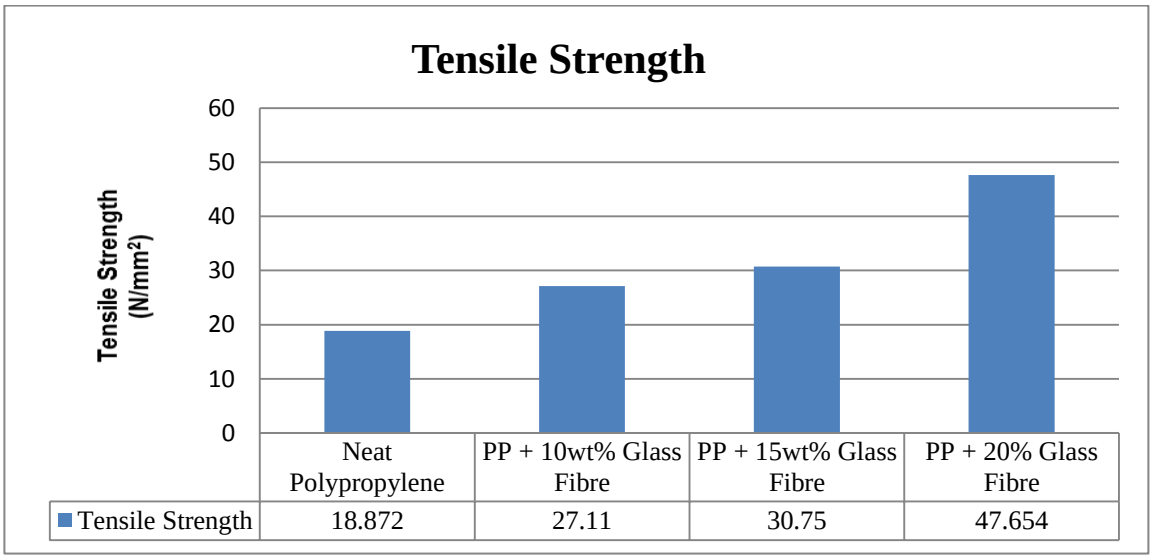

Figure 4- 8: Tensile Strength of each component

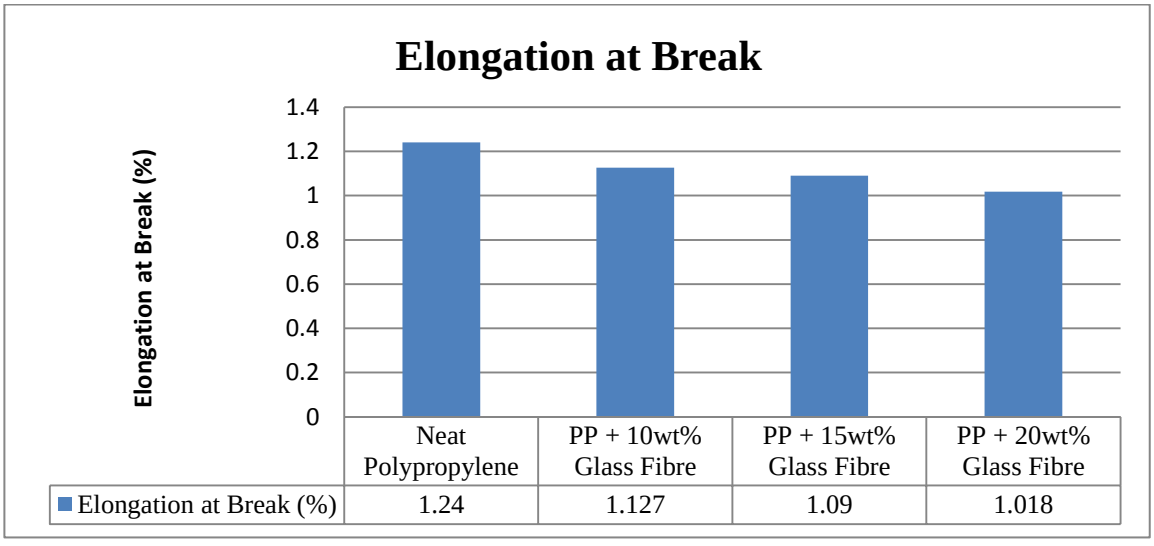

Figure 4- 9: Elongation at Break of each Component

4.2.2 THERMAL ANALYSIS

There were two analysis that had been carried out for thermal analysis, which were thermagravimetric analysis (TGA) and Differential Scanning Calorimeter (DSC). The results obtained from the testing were analyzed in the section below.

4.2.2.1 THERMAGRAVIMETRIC ANALYSIS (TGA)

TGA was performed to identify the thermal degradation of various samples. The results of neat PP and PP with varied weight percentage of glass fibre were plotted and compounded in Figure 4-10 in order to compare the degradation temperature of each sample. Based on Figure 4-10, it could be seen that the TGA curves showed a single degradation step for all the composite samples. The thermal decomposition for the entire samples was at the temperature range of 300˚ to 400˚C. The gradual weight loss happened in that temperature range indicated the matrix content of the composite which was polypropylene. There was no further weight loss been observed after 400˚C. The residue could be assigned to be the glass fibre content as it did not decompose under the condition used, while the polypropylene underwent complete degradation.

Besides, it was observed that the presence of glass fibre caused apparent alterations on the thermal stability of the composites. The thermal degradation temperature of composite was increases with the increase amount of glass fibre. It showed that the thermal degradation temperature of neat PP was at 381.11˚C, PP with 10wt% of glass fibre was 381.91˚C, PP with 15wt% of glass fibre was 386.92˚C and sample with 20wt% glass fibre had the highest degradation temperature which was at 390.56 ˚C.

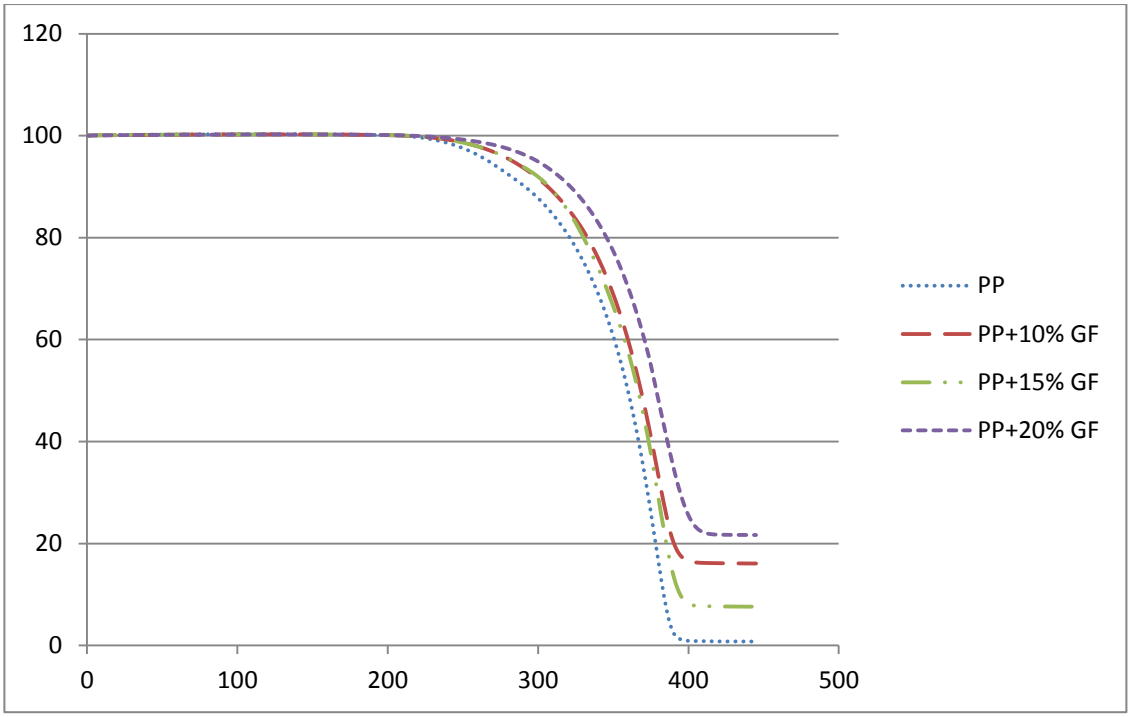

Figure 4- 10: TGA curve of Neat PP and PP with Varied Weight Percentage of Glass Fibre

4.2.2.2 Differential Scanning Calorimeter (DSC)

DSC was performed to determine the glass transition temperature, crystallization temperature and melting temperature. It was carried out after the completion of TGA. This was to ensure that the method used was within the degradation temperature range. The results obtained for DSC is shown in Figure 4-11. The peak of the curve in the figure indicated the melting temperature of the sample. It could be observed that there was no significant change in the melting temperature among them where they exhibited almost the same temperature which was 160˚C.

However, the degree of crystallinity showed slightly increment. It was because the surface of glass fibre acted as a nucleating agent for crystallization. Since the surface area for nucleation was higher with higher amount of glass fibre, hence an increased percentage of crystallinity was observed. This finding supported the earlier results of mechanical analysis as the composite was stronger with higher degree of crystanillity.

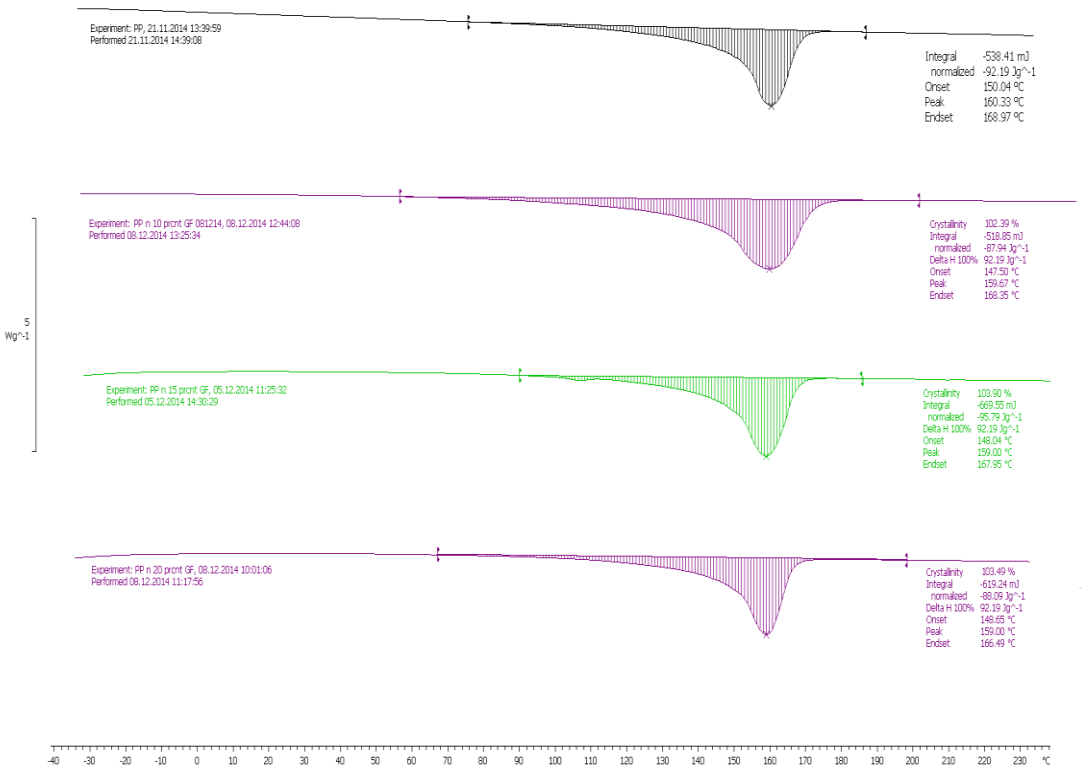

Figure 4- 11: DSC curve of Neat PP and PP with Varied Weight Percentage of PP

4.2.3 CHARACTERIZATION ANALYSIS

Upon the completion of tensile test, scanning electron microscopy was then carried out to observe the fracture surface of neat polypropylene and glass fibre reinforced polypropylene.

4.2.3.1 SURFACE MORPHOLOGY

Figure 4-12 depicts the scanning electron micrographs of the neat polypropylene and glass fibre reinforced PP. Based on Figure 4-12, it could be observed that the composite containing 10wt% of glass fibre, the amount of fibre were small and the glass fibres from the fibre pull out process were short in comparison with 15wt% and 20wt% of glass fibre. When the glass fibre content was higher, it was seen that the glass fibres protruded from the fracture surface plane were longer. Additionally, the plastic deformation of PP matric could be easily observed in Figure 4-12 (b) and (c) near the pull-out region. However, it was not observable in Figure 4-12(d) as the content of glass fibre was higher; there was less PP matrix to be plastically deformed.

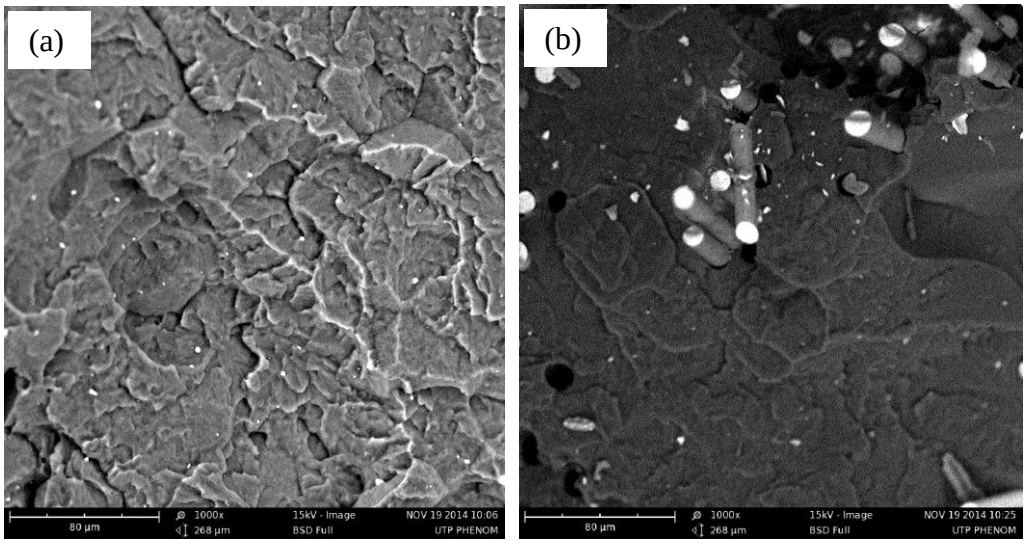

Figure 4- 12: SEM pictures: (a) Neat Polypropylene, (b) Polypropylene with 10wt% of Glass Fibre

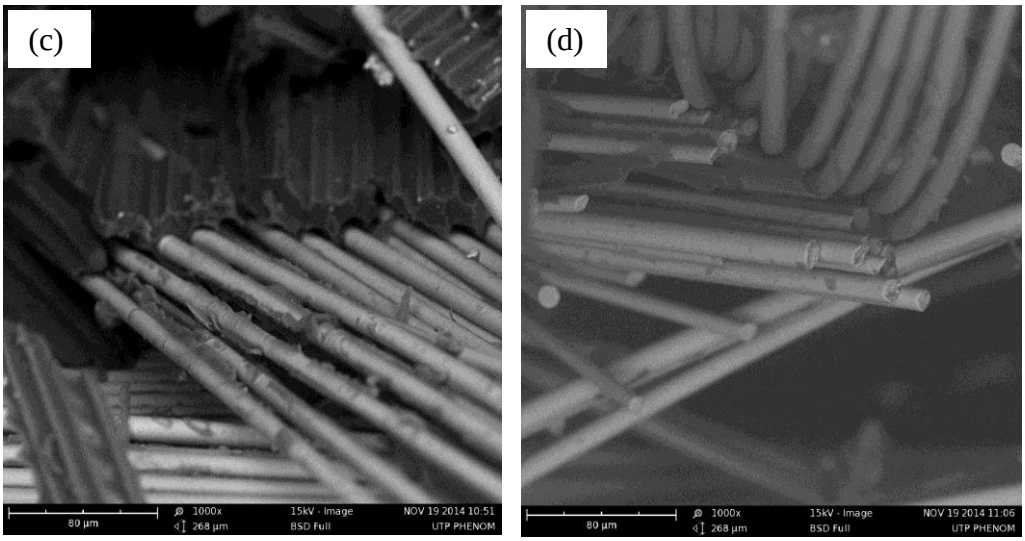

Figure 4- 12: SEM pictures: (c) Polypropylene with 15wt% of Glass Fibre and (d) Polypropylene with 20wt% of Glass Fibre

CHAPTER 5

CONCLUSION AND RECOMMENDATIONS

5.1 CONCLUSION

The glass fibre reinforced polypropylene had been successfully synthesized and properties analyses were performed. With introducing different weight percentage of glass fibre, the following results had been observed:

- The tensile strength of polypropylene increased with the weight percentage of glass fibre, but reduced in elongation at break. Polypropylene with 20wt% of glass fibre exhibited the highest tensile strength which was 33.142N/mm^2, having an increase factor of 0.584 compared to neat PP.

- The thermal degradation of polypropylene increased with the weight percentage of glass fibre. 20wt% of glass fibre- reinforced polypropylene had the highest thermal degradation temperature which was at 390.56^{o}C, having an increase factor of 1.088 compared to near polypropylene.

- The fracture surface morphology showed that with higher content of glass fibre, less PP matrix was seen to be plastically deformed.

5.2 RECOMMENDATIONS

Due to the limitation of equipment in Universiti Teknologi PETRONAS and the time allocated for the final year project, there are some constraints on this project. Hence, recommendations are given for future improvement and further development.

The weight percentage of glass fibre in this project was done until 20wt% only due to time constraint. It is recommended that further increment of glass fibre content can be done in order to obtain the best mix formulation. It is expected that there will be a peak where increment of mechanical and thermal properties starts to decrease with increasing glass fibre content.

Furthermore, compression moulding technique is used to synthesis the samples in this project. It is recommended that extruder and vertical injection moulding to be used, as the machine has better specifications which can produce better quality of composites. This can definitely made a significant change on the properties.

In addition, the experiment can be further modified by adding various chemical or organic fillers to improve the adhesion of glass fibre with polypropylene. This can improvise the bonding of fibre with polymer and increase the tensile strength as well as other thermal properties.

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APPENDICES

Appendix A – Tensile Test Report

Tensile test for each sample was performed several times in order to obtain the best results. However, the best result was not obtained in one batch. Thus, the results for tensile analysis were obtained from selected samples, the average values in the report were calculated from the chosen samples.

1. For Neat PP, the selected samples are tabulated as below:

Test No.	Tensile Strength (N/mm^2)	Elongation at Break (%)
Appendix A-1(a)		
2	17.608	1.113
4	17.262	1.106
Appendix A-1(b)		
2	19.854	1.756
3	19.979	1.020
5	19.655	1.249
Average	18.872	1.240

2. For PP with 10wt% of glass fibre, the selected samples are tabulated as below:

Test No.	Tensile Strength (N/mm^2)	Elongation at Break (%)
Appendix A-2		
1	27.855	1.806
2	27.283	0.302
4	27.404	1.186
5	25.917	1.215
Average	27.11	1.127

3. For PP with 15wt% of glass fibre, the selected samples are tabulated as below:

Test No.	Tensile Strength (N/mm^2)	Elongation at Break (%)
Appendix A-3		
1	31.560	1.888
2	20.868	0.063
3	36.668	1.254
4	33.904	1.154
Average	30.750	1.090

4. For PP with 20wt% of glass fibre, the selected samples are tabulated as below:

Test No.	Tensile Strength (N/mm^2)	Elongation at Break (%)
Appendix A-4 (a) & A-4 (b)		
1	49.434	1.613
2	45.595	1.659
4	53.991	0.039
5	41.594	0.759
Average	47.654	1.018

Appendix A- 1: Tensile Test for Neat PP

Appendix A-1 (a)

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UNIVERSITI TEKNOLOGI PETRONAS

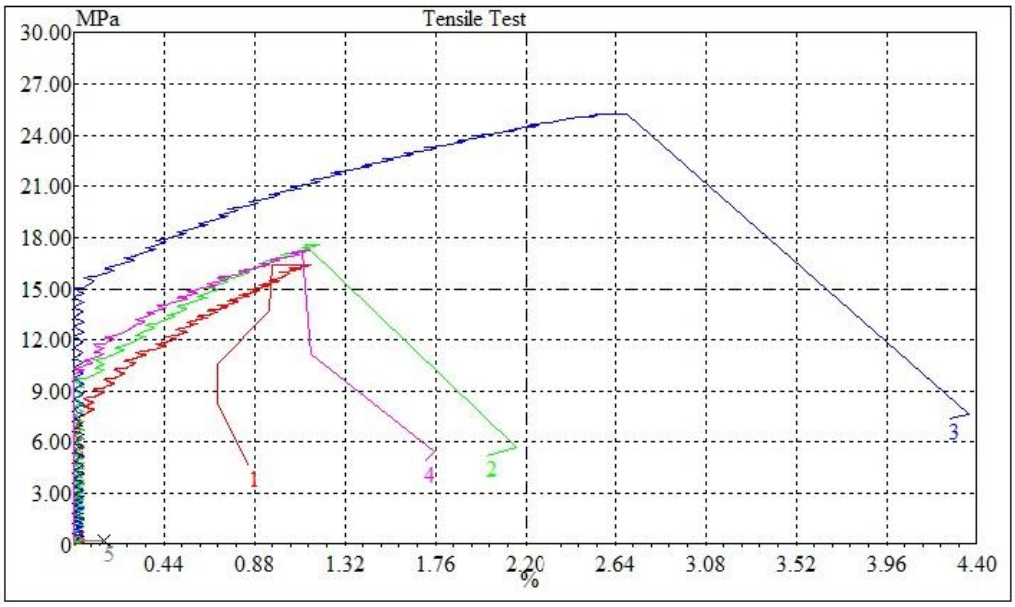
Tensile Test Report

BANDAR SERI ISKANDAR

Material : PP
Test Method : ASTM D638

Test Speed : 10.000 mm/min

Test No.	Width mm	Thickness mm	Area mm²	Elongation@Break mm	Tensile Strength N/mm²	Max. Load N	Elongation@Break %
1	12.680	4.269	54.131	0.481	16.418	888.715	0.962
2	12.670	4.281	54.240	0.556	17.608	955.049	1.113
3	12.660	4.318	54.666	1.387	25.220	1378.649	2.773
4	12.680	4.269	54.131	0.553	17.262	934.384	1.106
5	12.670	4.289	54.342	0.075	0.225	12.225	0.150
Average	12.672	4.285	54.302	0.610	15.347	833.804	1.221
$SD^{(N-1)}$	0.008	0.020	0.222	0.478	9.166	499.920	0.955



Appendix A-1 (b)

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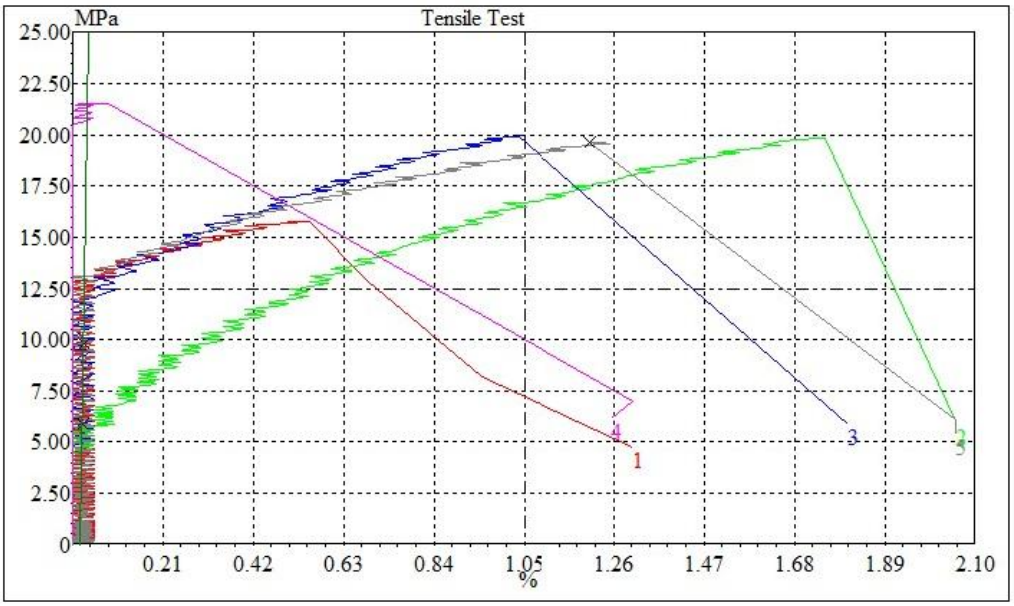
Tensile Test Report

BANDAR SERI ISKANDAR

Material : PP
Test Method : ASTM D638

Test Speed : 10.000 mm/min

Test No.	Width mm	Thickness mm	Area mm²	Elongation@Break %	Tensile Strength N/mm²	Elastic modulus kN/m²
1	12.830	5.603	71.886	0.552	15.792	44447417.694
2	12.660	4.772	60.414	1.756	19.854	1489866.403
3	12.700	4.920	62.484	1.020	19.979	97923634.041
4	12.750	4.945	63.049	0.092	21.556	181036059.554
5	12.730	4.825	61.422	1.204	19.655	82553591.494
Average	12.734	5.013	63.851	0.925	19.367	81490113.837
$SD^{(N-1)}$	0.063	0.337	4.604	0.635	2.137	66994006.834



Appendix A- 2 : Tensile Test for PP with 10wt% of Glass Fibre

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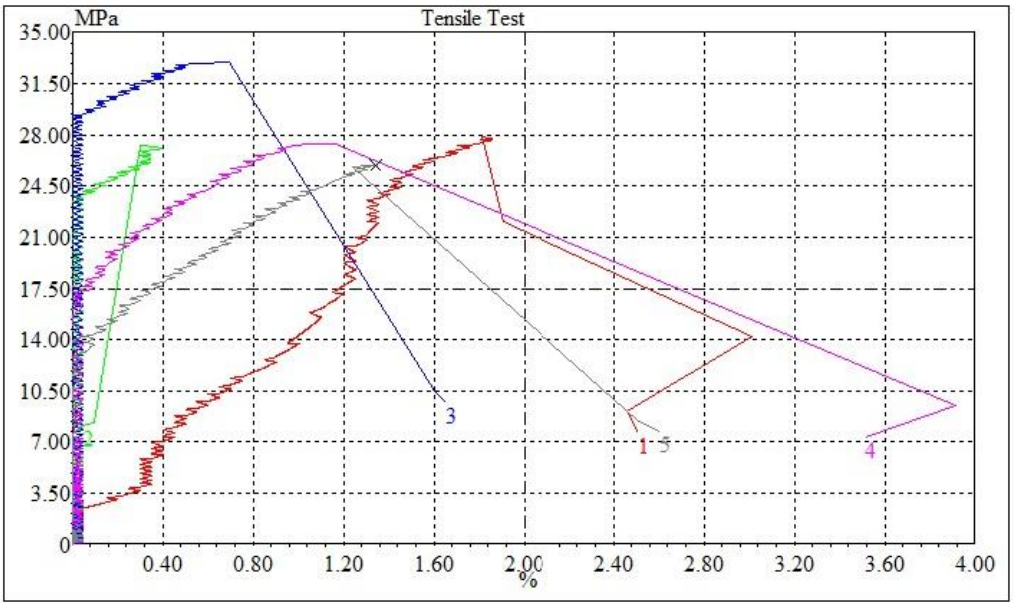
Tensile Test Report

BANDAR SERI ISKANDAR

Material : PP +10% GF
Test Method : ASTM D638

Test Speed : 10.000 mm/min

Test No.	Width mm	Thickness mm	Area mm²	Elongation@Break %	Tensile Strength N/mm²	Elastic modulus kN/m²
1	12.770	5.133	65.546	1.806	27.855	1088070.082
2	12.754	5.272	67.244	0.302	27.283	28733228.499
3	12.702	5.138	65.260	0.716	32.937	-
4	12.686	5.035	63.879	1.186	27.404	29784062.089
5	12.826	5.223	66.993	1.215	25.917	-
Average	12.748	5.160	65.784	1.045	28.279	19868453.557
$SD^{(N-1)}$	0.056	0.091	1.374	0.567	2.702	16272773.746



Appendix A- 3: Tensile Test for PP with 15wt% of Glass Fibre

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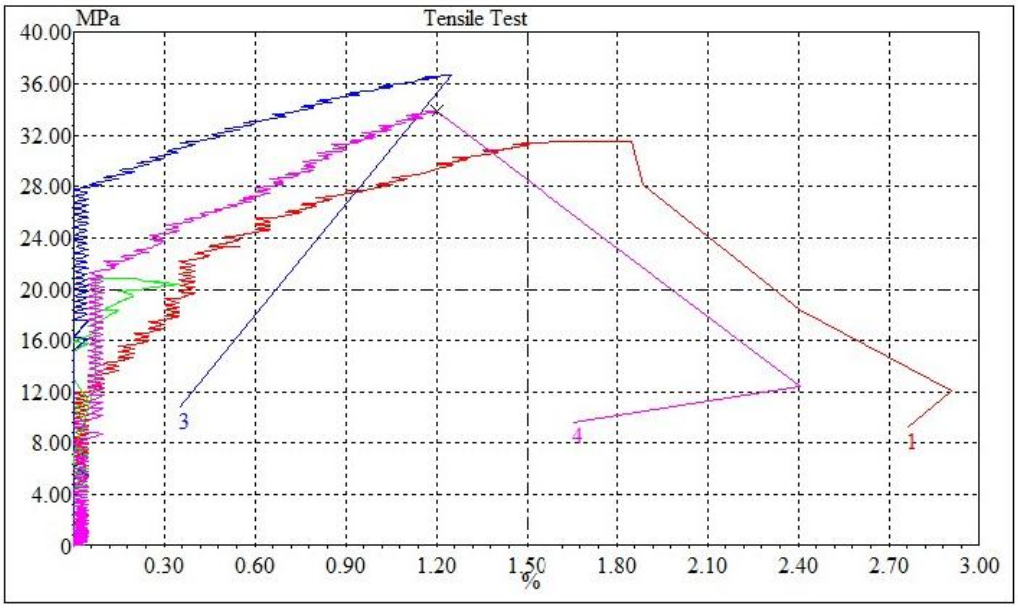
Tensile Test Report

BANDAR SERI ISKANDAR

Material : PP +15% GF
Test Method : ASTM D638

Test Speed : 10.000 mm/min

Test No.	Width mm	Thickness mm	Area mm^2	Elongation@Break mm	Tensile Strength N/mm^2	Max. Load N	Elongation@Break %
1	12.750	5.660	72.165	0.944	31.560	2277.493	1.888
2	12.780	4.731	60.462	0.031	20.868	1261.751	0.063
3	12.720	5.550	70.596	0.627	36.668	2588.588	1.254
4	12.720	4.798	61.031	0.577	33.904	2069.180	1.154
---	---	---	---	---	---	---	---
Average	12.742	5.185	66.064	0.545	30.750	2049.253	1.090
$SD^{(N-1)}$	0.029	0.488	6.177	0.379	6.911	566.725	0.758



Appendix A- 4 : Tensile Test for PP with 20wt% of Glass Fibre

Appendix A- 4(a)

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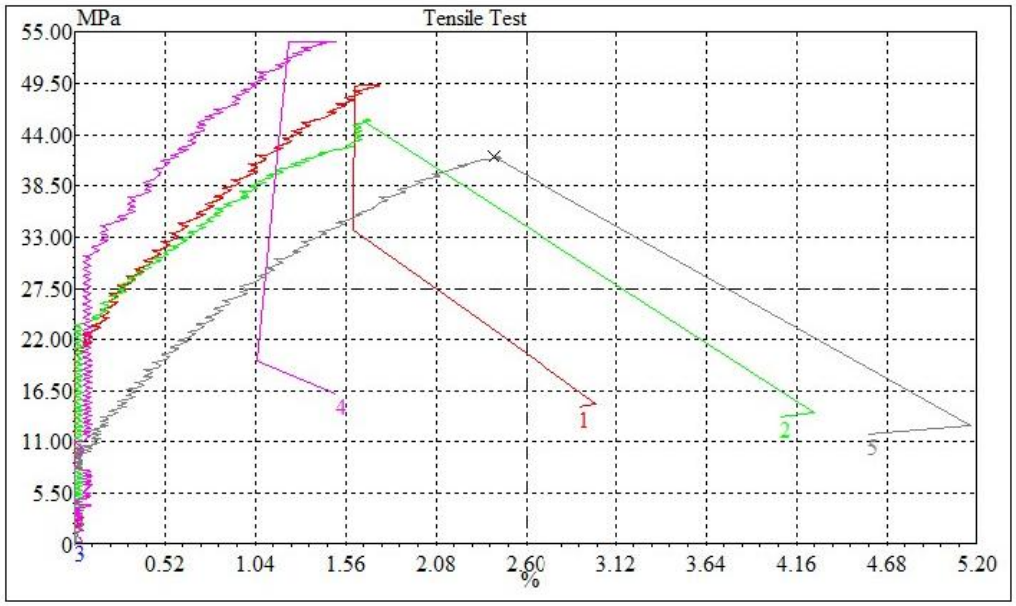
Tensile Test Report

BANDAR SERI ISKANDAR

Material : PP+20%GF
Test Method : ASTM D638

Test Speed : 10.000 mm/min

Test No.	Width mm	Thickness mm	Area mm^2	Elongation@Break mm	Tensile Strength N/mm^2	Max. Load N	Elongation@Break %
1	12.700	4.876	61.925	0.807	49.434	3061.225	1.613
2	12.740	4.953	63.101	0.830	45.595	2877.103	1.659
3	12.720	4.892	62.226	-	0.377	23.462	-
4	12.720	4.892	62.226	0.572	53.991	3359.664	1.144
5	12.720	4.892	62.226	1.205	41.594	2588.268	2.410
Average	12.720	4.901	62.341	0.854	38.198	2381.944	1.707
$SD^{(N-1)}$	0.014	0.030	0.445	0.262	21.635	1347.920	0.524



Appendix A-4 (a)

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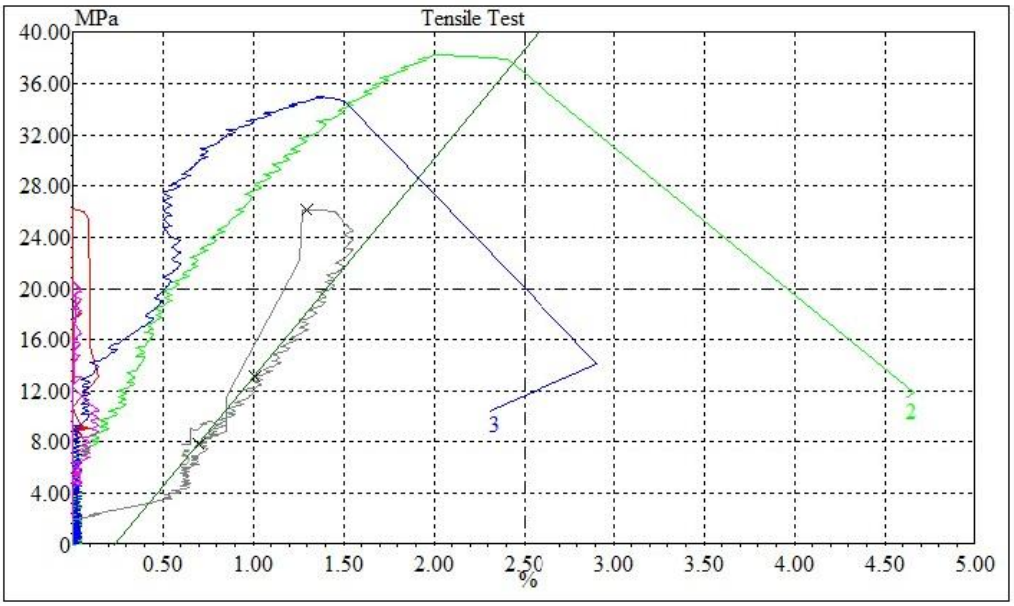
Tensile Test Report

BANDAR SERI ISKANDAR

Material : PP +20% GF
Test Method : ASTM D638

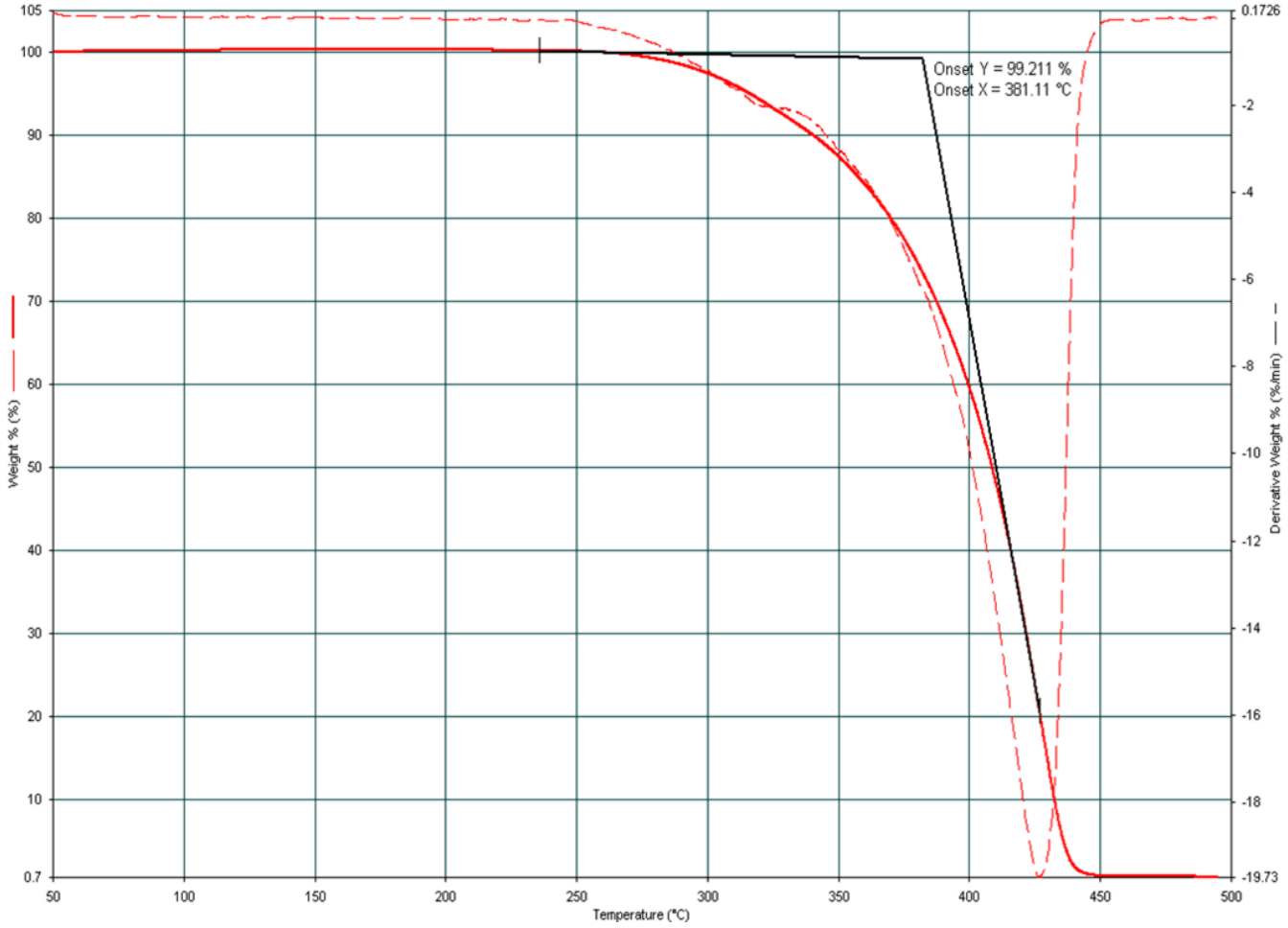
Test Speed : 10.000 mm/min

Test No.	Width mm	Thickness mm	Area mm²	Elongation@Break %	Tensile Strength N/mm²	Elastic modulus kN/m²
1	12.934	4.843	62.634	-0.030	26.240	24769613.919
2	12.922	4.805	62.085	2.809	38.290	3036212.113
3	12.776	4.830	61.703	1.605	34.960	2061443.058
4	12.806	4.831	61.868	-0.039	20.513	5786620.245
5	12.790	4.888	62.518	0.759	26.177	1703060.665
Average	12.846	4.839	62.162	1.021	29.236	7471390.000
$SD^{(N-1)}$	0.076	0.030	0.404	1.208	7.231	9801517.127

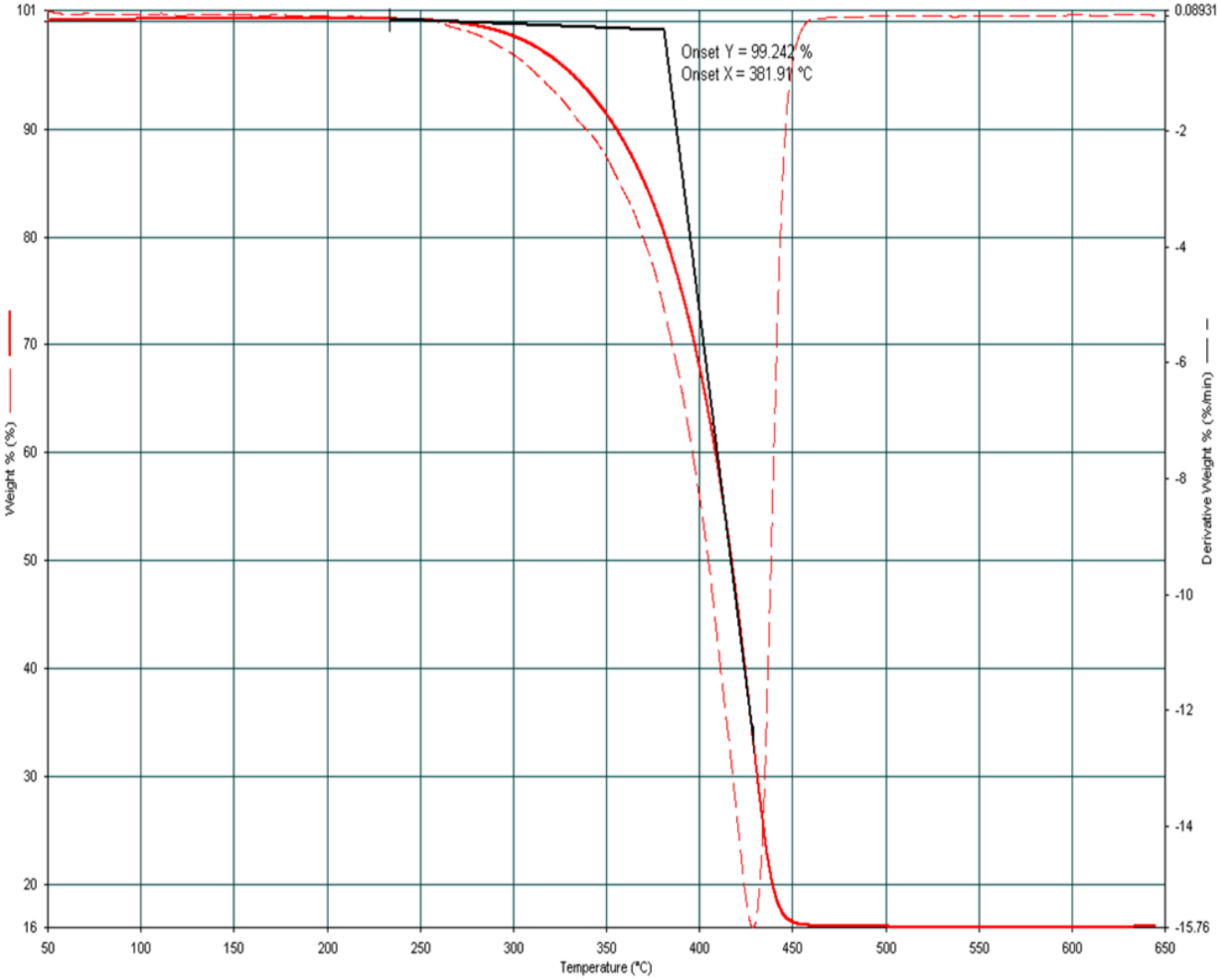


Appendix B- Thermal Gravimetric Analysis result

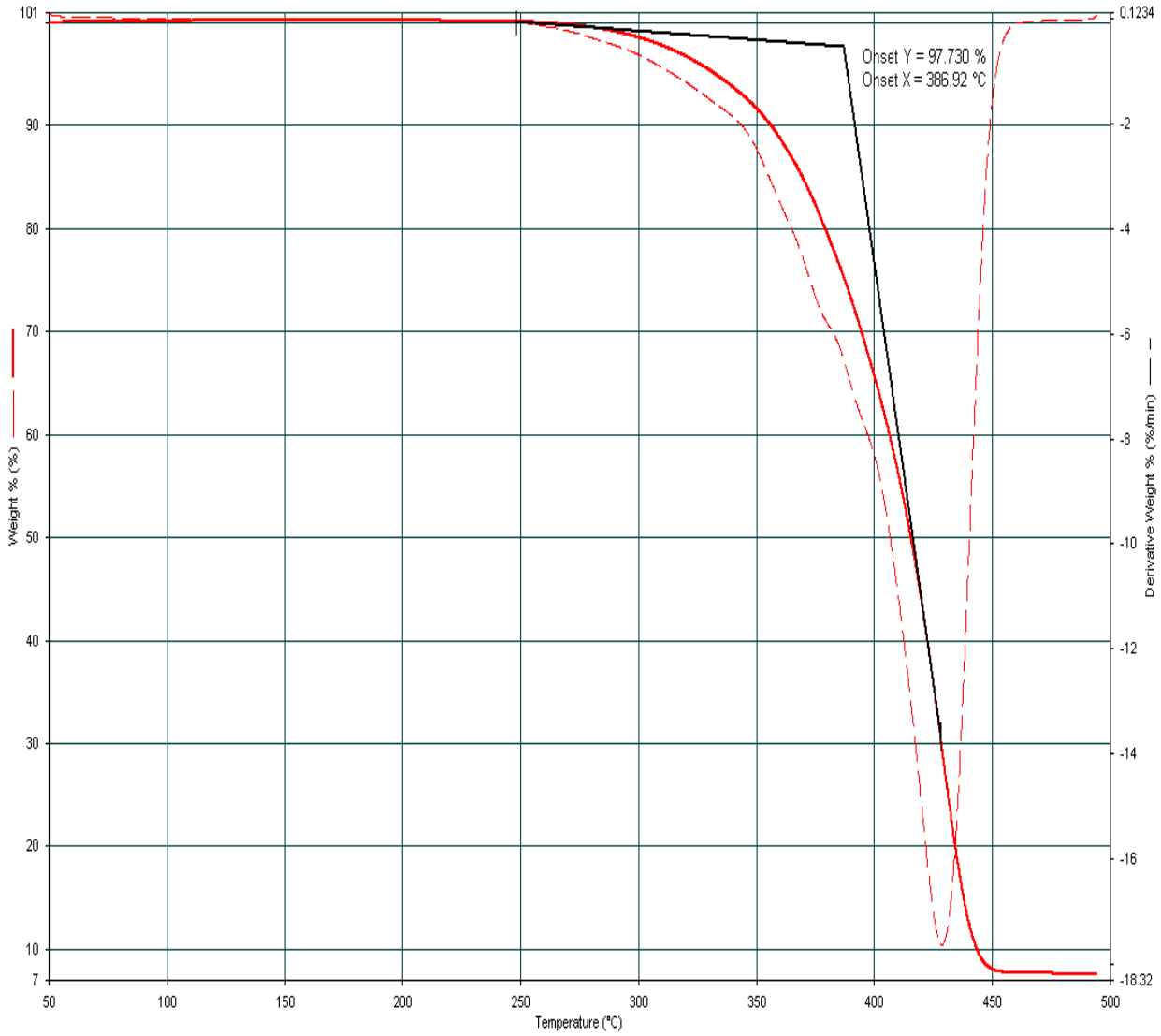
Appendix B- 1: TGA of Neat PP



Appendix B- 2: TGA of PP with 10wt% GF



Appendix B- 3: TGA of PP with 15wt % GF



Appendix B- 4: TGA of PP with 20wt % GF