Processing and Characterisation of PE/PET Microfibrillar Composites

by

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Dissertation submitted in partial fulfillment of
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CERTIFICATION OF APPROVAL

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A project dissertation submitted to the
Mechanical Engineering Programme
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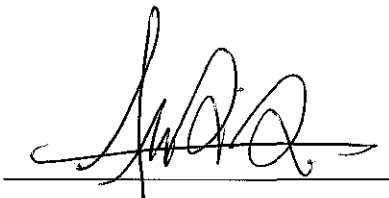
Approved by,

(Dr.Mohamad Zaki Bin Abdullah)

UNIVERSITI TEKNOLOGI PETRONAS
TRONOH, PERAK
MAY 2011

CERTIFICATION OF ORIGINALITY

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



MOHD FAIRUZ BIN MAHMUD

ABSTRACT

This project will present the feasibility study of processing and characterisation of PE/PET microfibrillar composites (MFCs). MFCs are created by processing two homopolymers with different melting temperature (T_m) of at least 40°C [1]. These new composites were reported to improve mechanical properties and had potential for wide range of applications with suitable processing under controlled condition [2]. In this study, linear density polyethylene (LDPE) was used as a matrix and recycled poly (ethylene terephthalate) (PET) was employed as reinforcement. They were blended together using twin-screw extruder at temperature above the melting temperature (T_m) for PET. The extrudate blends were drawn at temperature above the glass transition temperature (T_g) of PET. The drawn blends were then injection molded to produce samples for tensile and flexural tests. The morphology and molecular orientation of MFCs were observed using scanning electron microscope (SEM). ASTM D638 and D790 standards were used to determine the tensile and flexural properties. The morphological structure of the MFCs showed fibrils formation after drawing process. Compared to pure LDPE, tensile strength, flexural strength and modulus of the MFCs were improved by 50%, 60% and 30%, respectively.

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TABLE OF CONTENTS

LIST OF FIGURES

LIST OF TABLES

CHAPTER 1
INTRODUCTION

1.1 PROJECT BACKGROUND

Polymers have been produced and applied for various applications such as plastic bottles, toys, equipment and others. It has good and productive capabilities that can be expanded for usage in many areas and applications. It is understandable that polymers must expand their capability to ensure continuous improvements. This can be achieved by introducing the reinforcement to a matrix polymer to improve mechanical properties. A new composite called Microfibrillar Composites (MFCs) has been proven to improve mechanical properties.

Basically, in processing the MFCs, the reinforced polymer will be blend to matrix polymer to become a new blend polymer. They will mix together which the minority constituent (reinforce polymer) will dispersed into majority constituent (matrix polymer) that forms a homogenous body [2]. That means the LDPE as matrix will be reinforced with PET to become a new blend polymer. From previous researches, ratio of 90/10, 80/20, 70/30, 60/50 and 50/50 of polymer blends have been used. In this study, the author used the ratio of 70/30. The blend of the polymers would give a good partnership to the complex polymer chain microstructures [3].

The matrix was reinforced with microfibrils having a diameter in the micrometer range with aspect ratio around 100. They are prepared from polymers haves melting temperatures different of at least 40°C. The manufacturing of MFCs consists of three basic steps: (i) melt blending the two homopolymer with extrusion (mixing step); (ii) drawing the extruded with good orientation and controlled condition (fibrillation step), and (iii) thermal treatment at a temperature between the melting temperature (T_m) of the two blend polymer (isotropization step) [2]. The first step needs to be carried out above T_m of the reinforced polymer to ensure the melting of both

polymers during the extrusion. The second step is very important as it will allow the formation of reinforcing fibrils in the blends. The third step can take place during processing of the drawn blend via injection molding or compression molding to become MFCs [1].

This new composite has high potential for improve mechanical property improvement. Tensile and flexural tests were done on MFC samples, undrawn LDPE/PET and neat LDPE after the injection molding process. While the morphology and molecular orientations of the MFCs were observed using scanning electron microscope (SEM). The morphology and molecular orientation were check after the drawing step.

1.2 PROBLEM STATEMENT

LDPE may be used for certain applications because of its limited mechanical properties. The mechanical performance of LDPE can be increased by applying reinforcement to it. By applying the concept of MFCs, where polymer matrix will be blend with reinforced polymer, then cold drawing and later injection molded, until mechanical properties can be improved significantly.

1.3 OBJECTIVES AND SCOPE OF STUDY

The objectives of this study are:

- To process MFCs from LDPE and PET and perform tensile and flexural testings.
- To characterise the mechanical properties and morphological structure of the MFCs.

The scope of study for this project would involve the processing and characterisation of LDPE/PET MFCs. The characteristics of LDPE/PET MFCs were checked through SEM and mechanical properties were done to determine the tensile and flexural properties using ASTM D638 and ASTM D790 standard [4, 5], respectively.

CHAPTER 2
LITERATURE REVIEW

2.1 OVERVIEW

Previous literature relating to polymers, their blending and composites was dated as early as 1865, where the first polymer blend was created [6]. Basically, the objective of this project is to do feasibility study of processing and characterisations of MFCs. Nowadays, polymers are being used for certain applications that require higher mechanical properties and characteristics to extend their usage. It was reported that 15 wt% of PET microfibrils, would significantly improved tensile strength and modulus by 30-65% and 50-70%, respectively [7].

To achieve the improvement, sphere shapes of PET in blends must be increased up to 100 times larger which a draw ratio of 7 during cold drawing (at T_g of PET) until it become microfibrils that dispersed in PE [8]. Which such a good orientation the microfibrils would give a good bonding with PE that may improve the strength defers to undrawn PE/PET and neat PE. It also showed that MFCs of blend PE/PET were comparable with the short-glass-fibre reinforced low density PE with ratio of 70/30 [3].

MFCs were comparable rather than standard and traditional polymer composites as it have several advantages which: (i) polymer-polymer reinforcement; (ii) no requirement of mineral additives; (iii) reduced weight in comparison to equivalent glass-fibre composites; (iv) ease of processing; (v) no need for the addition of compatibilizer, and (vi) recyclability and repetition of the process [2]. Based on feature of MFCs, it relevance for author to do feasibility study about the processing and characterisation of polymer-polymer blend and make a process to improve the mechanical properties.

2.2 PE AND PET

The basic and commercial polymer, PE and PET had been reported usage for previous experiment [1-23]. It may use as PE and PET have a wide different in their properties. It had been stated that T_m for both polymer blends must have a different of at least 40°C [2]. For PE, it melting temperature are around 120-160°C while PET 245-260°C which give a different strength for both of the polymer [9]. PET, PA, ABS are commonly used to be blends with commodity thermoplastics resin such as PE, PP or PSU (special high performance polymers) [10]. Basically, PE and PET are commercially used for packaging and equipment where they are recyclability. PE and PET possess good mechanical properties that applicable for wide ranging application as shown in Table 2.1.

Table 2.1: Properties of PE and PET.

Properties of PE	Properties of PET
• Light weight • Easy processing • Good chemical resistance • Good impact strength • Excellent electrical properties • Good barrier properties • Low water absorption • Toughness and flexibility even at extremely low temperature	• Light weight • Hard, stiff, strong • Dimensionally stable • Absorbs very little water • Good gas barrier properties • Good chemical resistance except to alkalis (which hydrolyse it) • Highly transparent • Colourless

2.3 POLYMER-POLYMER BLEND

Blending two homopolymers for example PE and PET can improved the mechanical properties of polymeric materials. However, the improvements of polymer-polymer blends do not arise except the polymers are thermodynamically immiscible and incompatible [1]. This is major reason as the strength of polymer-polymer blends are being affected by their morphological structure which the reinforcement immersed into matrices that changed the bonding of the polymer. From these blending, the morphological structure possessed a variety of shape dispersed phase formed, e.g. spheres or ellipsoids, fibrils or plates [11, 12].

This variety of shape was basically depending on the processing and properties of the polymer. For example PE and PET possessed a different mechanical properties and even their T_m have a wide different. With the processing of this two homopolymer, the properties of the polymer blend may improve in term of their strength and capability of processing. This proved that blending are a new way of extend the limit of processing and usage of existing and traditional polymer for more broad application.

The increasing usage and application of polymer blends in the past decade had been done as it will improved resin/product performance through; (i) lower costs to produce a material with the full set of desired properties; (ii) extended performance by using less expensive polymers; (iii) improved specific properties; (iv) providing means of recycling industrial/municipal plastics waste; (v) rebuilding of the high molecular weight polymers from degraded polymers [6].

2.4 MFCs

Previous research had shown that mechanical properties (tensile strength and Young's modulus) of MFCs from blends of PET are better than neat PP and PE [7]. MFCs were blends of isotropic matrix with polymer reinforced and going through certain condition until there are microfibrils that proven may increased strength of the polymer composites. These microfibrils were having a micrometer range with aspect ratio of around 100 [8]. The reinforced polymer created an in-situ composition to isotropic matrix. The formation of microfibrils was very important for the improvement in their properties. These formations are done in controlled condition to ensure the deformation from sphere shape to fibrils shape.

MFCs were prepared from blend and extrude the polymer-polymer blends, cold drawing and lastly injection molding to create a new polymer composite, MFCs. With MFCs, the limit performance barrier of composites can be enhanced and applied for useful application rather than traditional and exist polymers [1-6]. Rather than have improvement in their strength, MFCs also have been identified other advantages that rarely different from other materials and also benefit to the

environment as its recyclability [1, 6]. MFCs based on blends of thermoplastic polymers and manufactured using an extrusion, drawing and matrix consolidation technique as shown in Figure 2.1.

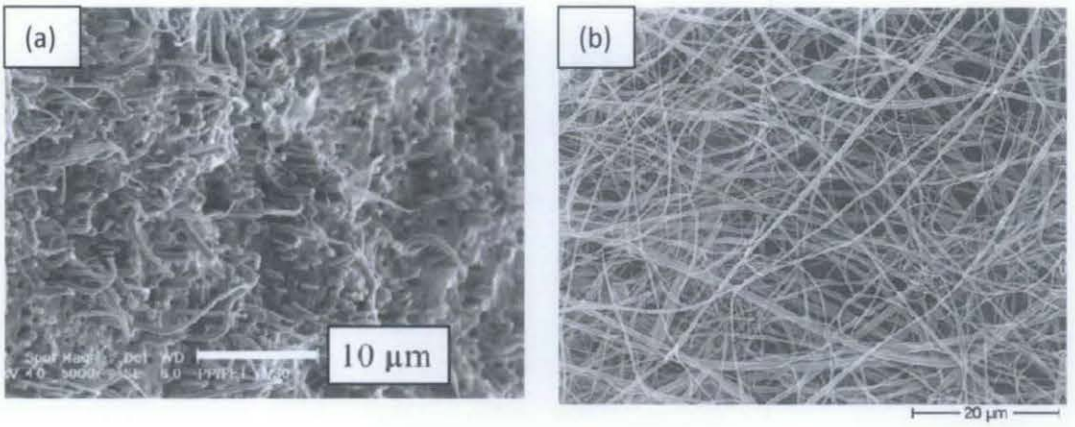

Figure 2.1: Typical structure of MFCs: (a) Composite fracture surface after compression moulding; (b) Reinforcing fibrils after extraction of the matrix polymer [6].

2.5 COLD/HOT DRAWING

Actually the melt polymer-polymer blend that had been extruded going through the process of drawing to gain the fibrils shape of MFCs. From the previous research it been stated that two process of drawing have been done which is cold and hot drawing. For cold drawing, the extruded was drawn in a dry hot air at a temperature of around 90°C (which is higher than the glass transition temperature of PET) to create microfibrillar structure of the blend with the diameter of a fibril being around 2 µm [7]. This was possibly achieved the drawing ratio 5-11 of the extruded blends.

This was basically different to hot drawing which used to drawn the extruded by a take-up device with three pinching rolls to form the microfibrils, and the roll temperature was kept at about 40°C by adjusting the volume flow rate of tap water in the cooling pipe which the speed of the rolls can be changed to obtain different hot stretching ratios [13]. One of other hot drawing technique is by cooled the extruded in a water bath at approximately 20°C and continuously drawn into through a set of roller into hot water bath at 80°C [10]. The drawing ratio of hot drawing is more than cold drawing which approximately 30-47.

2.6 MELTING TEMPERATURE

T_m for MFCs was very critical to allow fibril retention during matrix consolidation [2, 3, 8]. During the extrusion, the T_m of reinforced polymer used to ensure the blends are fully and isotropic melts and blends together. While during the injection molding the T_m of matrix polymer used to make sure the fibrils of the blends not melt together with the matrix polymer to ensure the reinforcing effect not lost. Processing of MFCs can be separated into three stages which T_m plays a crucial step in the development of MFCs (T_m different of at least 40°C) [1, 6].

2.7 GLASS TRANSITION TEMPERATURE

The T_g of reinforced polymer was important to make sure the continuous and cigar form of fibrils in the polymer blends. Theoretically, T_g of reinforcing polymer will produced molecular chain alignment that are lower than matrix structure that give consistent drawing properties between 15-90°C [3]. Somewhat, the T_g of PET were around 80-90°C which is greater than LDPE, -125°C but lower than T_m of both polymer. This was state where PET will deformed and started to change from sphere shape to fibrils or cigar shapes during cold drawing.

2.8 INJECTION MOLDING

The using of injection molding also can be referred to thermal treatment at temperature between the T_m of the polymer-polymer blend [1-11]. It depend on the T_m of the matrix polymer and not close to T_m of reinforcement, otherwise the microfibrils melted together and return from fibrils to sphere shape [1]. For example the T_m of PE is around 120-160°C which the injection molding need to be set according to the temperature and with a high pressure of injection molding, the microfibrils tended to not return to their original sphere shape. The reason injection molding are more comparable than compression molding as it had a processing with high pressure that give the MFCs to transform into isotropic matrix, reinforced with microfibrils.

2.9 EXTRUSION

The temperature profile starting from the feeding zone to die were 260, 270, 260 and 245°C and screw rotating speed was 30 rpm [1]. During the extrusion, fibril diameters and shapes also changed due to the different levels of shear stress induced by the die walls [6]. The fibril shapes may changes at the surface of the blend filament but still in sphere shapes in the cross section. The stress during the extrusion may influence on the formation of the fibrils at it difficult to be measure but through the drawing process, the formation of fibrils can be assured.

2.10 MECHANICAL PROPERTIES

MFCs are often report to have improvement in their mechanical properties defer to their constituent materials. For example, Evstatiev reported that the tensile strength of MFCs from PET/PA6 (30/70 wt%) was higher than that of the equivalent glass-fibre filled PA6 system when compression molded [6]. It is understandable that the polymer-polymer blends of LDPE/PET may have improvement defer to its neat LDPE, but by continuing drawing the LDPE/PET it would influenced the mechanical properties to increase further [14]. This improvement of mechanical properties had been integrated to change the application of the polymer and will vary according to the type of polymer-polymer blends, for example PE/PEN and PE/PET [15].

2.11 MORPHOLOGICAL CHARACTERISTICS

The morphology of the PET/PP extruded was studied with a Cambridge S440 scanning electron microscope where sample was immersed in liquid nitrogen and fractured before coated with thin platinum layers [3]. During the mix and blend, the sphere shape will shown while after the drawing the cigar and fibrils shape will shown which depend on the ratio of drawing. During the drawing process, the temperature and also stress of elongation need to be controlled to ensure the formation of fibrils from sphere shape. These fibrils will be aligning through the molecular orientation of MFCs where this was used as the parameter for the improvement in their mechanical properties.

The alignment of fibrils in MFCs was very critical as well aligned fibrils will increased the strength and cause higher crystallinity to the fibre orientation [6]. The last formation of fibrils was the determination of successful formation during drawing process. The previous researches also had been stated that MFCs will show different degrees of orientation after injection molding and compression molding. Actually after MFCs processing by injection molding will possessed higher strength than compression molding as their molecular orientation are more random rather than uniaxial orientation. The random orientation of fibrils will ensured that molecular alignment through the orientation was strong and caused higher crystallinity.

2.12 MISCIBILITY AND COMPATIBILITY

Thermodynamic miscibility was the term used to describe the propensity of a mixture to create a single homogeneous phase, through the capability to mix on a molecular level [6]. That means the miscibility showed a complete mix phase of the polymer-polymer blends which no phase separation. The miscibility was important to determine the strength of the polymer-polymer blends. Their interaction between the chemical bonding will give different properties to the polymer-polymer blend. If the mixing of the polymer was separated, it was thermodynamically immiscibility which there was weak region trough the chemical bonding of the polymer-polymer blends.

Compatibility refers to the ability to maintain two immiscible polymers in a mixed state which relates to the ability of multiphase morphology to produce synergistic advantage over single polymer materials [6]. This was very crucial to determine and maintained the mix of the polymer-polymer blends. The combination of the polymer-polymer blends must be having a good compatibility to ensure the formation of MFCs as it may affect the morphological structure of MFCs and as well as the mechanical properties. Some of the previous research is using compatibilizer to ensure the two immiscible polymers are mixed [16]. This compatibilizer tends to effect the formation of fibrils and the molecular orientation of MFCs thus lead to different effect on their strength.

2.13 PEER-REVIEWED RESEARCH

The previous research of MFCs had given a wide knowledge of improvise composites that being process from homopolymer. It really proved that there are improvement in MFCs properties and application. For a decade, there are many research have been publish relating to MFCs involved in polymer-polymer blend, properties, process and their characteristics [6]. A summary of tensile testing of MFCs, as reported in various peer-reviewed research articles in Table 2.2 are shown.

Table 2.2: Result of previous test and research [5].

Author/s	Material	Ratio (by wt)	Tensile		
			Modulus (GPa)	Strength (MPa)	Extension (%)
Evstatiev et al.	LDPE/PET	100/0	0.11	8.8	88
		70/30	0.94	18	16
		50/50	1.32	27	14
Fakirov et al.	LDPE/PET	100/0	0.10	8	87
		70/30	0.79	17	12
		50/50	1.05	26	11
Li et al.	PE/PET	95/5		22.0	
		90/10		25.2	
		85/15		31.3	
		80/20		32.8	
		75/25		35.9	
		70/30		29.2	
		65/35		24.5	
Li et al.	PE/PET	90/10	1.35	26	
		85/15	1.65	31	
		80/20	1.70	33	
		75/25	1.75	36	
Li et al.	HDPE/PET	100/0	0.99	20.5	30-340
		85/15	1.35	23-31	
		0/100	1.63	78	
Li et al.	PE/PET	100/0	0.98	20.5	
		95/5	1.07	22.3	
		90/10	1.10	23.6	
		85/15	1.16	24.4	
		80/20	1.22	25.4	

CHAPTER 3
METHODOLOGY

This chapter will discuss the methodology to process MFCs. It divided into four parts which is material, tool and equipment, experiment and gannt chart and project work. All part is important to achieve the objectives of the project. Material part discussed on the material used for the project. All parameter and properties were stated before going into processing. Tool part explained through the entire tool and equipment used in the processing of MFCs. The experiment part would have an explanation on step and processed done from raw material of LDPE and PET into MFCs. The characterisation technique and also test also explained in the experiment part. Lastly the gannt chart and project work (Figure 3.14), it explained on the timelines on doing the project. The methodology will explained all criteria and parameter needed to process the MFCs (Figure 3.13).

3.1 MATERIAL

Recycled material from PET bottles (bottle grade) with T_m range 254-256°C, T_g 82°C, tensile strength range 55-75 MPa, tensile modulus range 2.7-4.0 GPa, flexural strength range 80-120 MPa, flexural modulus range 2.3-3.0 GPa and density 1.38~1.40 g/mm3 as reinforced polymer. This material was selected to be used as it has greater mechanical properties deferred to LDPE for an easy comparison. Injection molding grade LDPE (type Titanlene LDI300YY provided by Titan Chemical) with T_m range 160-240°C, T_g -125°C, tensile strength range 8-10 MPa, tensile modulus range 0.2-0.3 GPa, flexural strength range 10-40 MPa, flexural modulus range 0.2-0.3 GPa and density 0.000920 g/mm^3 as matrix polymer [17].

3.2 TOOLS AND EQUIPMENT

The tools used in this study were low speed granulator (model SG-21P) to grinder the bottle of PET, vacuum Oven for drying LDPE and PET, Leistriz twin screw extruder (model Mi027/G6-32D) for mix and blend of LDPE and PET, palletiser machine (model C.F.SCHEER) to grinder the extruded LDPE/PET before going through injection molding, injection molding machine (model ME 20 iii) with two mold, dumbbell and bar to make samples for testing, scanning electron microscope (model LEO VP1430) to check on the morphology characteristics of MFCs and 5 kN universal testing machine (model LLOYD LR54) with ASTM D638 and D790 standard for testing the tensile and flexural properties of the samples .

3.3 EXPERIMENT

3.3.1 Preparation of Raw Materials

LDPE were raw material supplied by Titan Chemical as shown in Figure 3.2, while PET was processing from bottle by grinder using low speed granulator until become small plastic flakes shown in Figure 3.1.

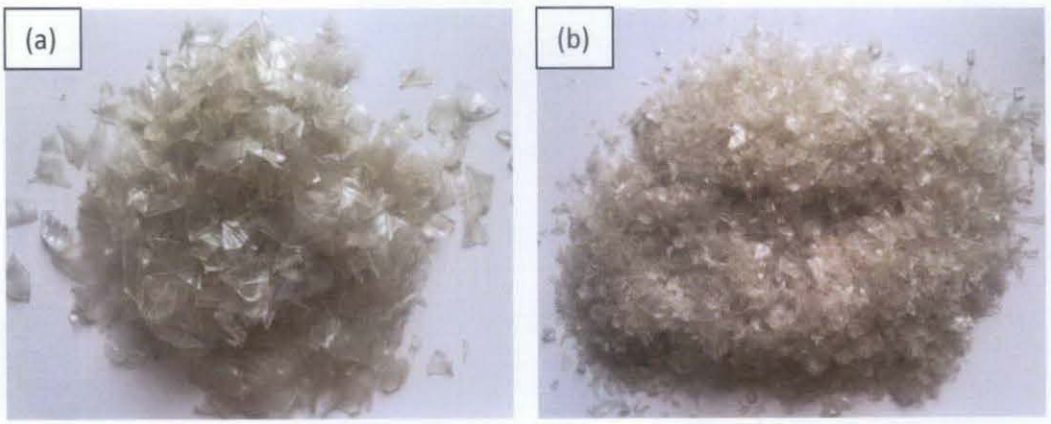

Figure 3.1: Raw materials: (a) PET before grinder; (b) PET after grinder.

Figure 3.2: Raw LDPE.

3.3.2 Composition Ratio and Drying

A melt blended LDPE/PET with 70/30 wt% (Figure 3.3) which means a total of 1000 g melt blend LDPE/PET with 300 g PET and 700 g LDPE were dried in the vacuum oven for 10-12 hour at temperature of 80°C to remove any moisture built up during storage. Drying for PET was important to prevent hydrolysis during extrusion process [5].

Figure 3.3: The mixture of LDPE/ PET with ratio 70/30 wt%.

3.3.3 Mixing and Extrusion

LDPE/PET mixed, compounded and extrusion using Leistriz twin screw extruder (Figure 3.4 (a)) with increasing temperature profiles of T_1, T_2, T_3, T_4, T_5, T_6 and T_{die} was 200, 220, 250, 260, 270 and 250°C and the speed of screw being set to 30 rpm throughout the process. These temperature profiles were using the polymer with highest T_m which is PET, 270°C. This forms an isotropic and continuous blend filament (Figure 3.4 (b)). The extruded blend filament was cold down before straightly going to drawing process of drawn LDPE/PET and some are palletised for undrawn LDPE/PET. `

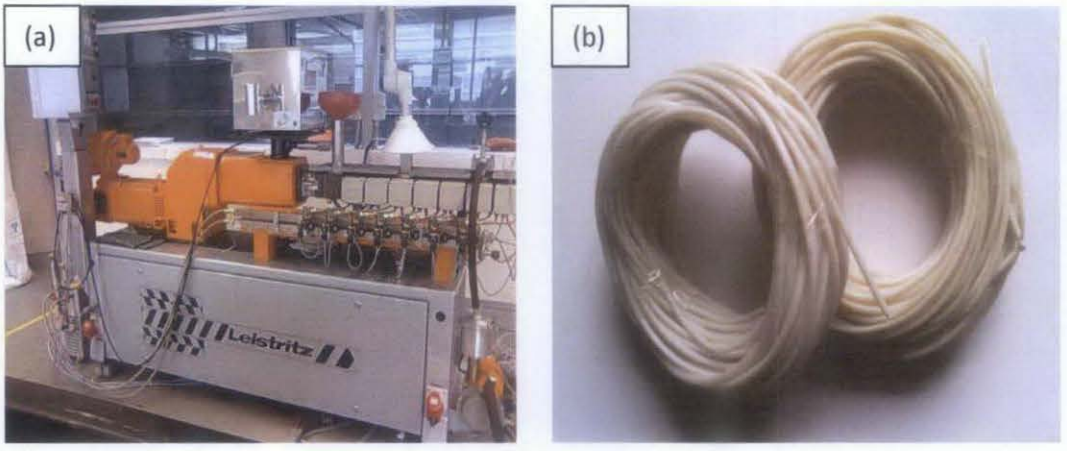

Figure 3.4: (a) Leistriz Twin Screw Extruder; (b) The extrudate LDPE/PET bled filament.

3.3.4 Drawing

The LDPE/PET blend filament was drawn to create the microfibrillar morphology essential to MFCs. Drawing was doing straightly after extrusion by cold down to T_g of PET 82°C and stretched until indicates point of necking to the blend filament (Figure 3.5 (a)), and then palletised using palletiser machine. It been done above the T_g of PET to allow the molecular chains to move freely during realignment. The blend filament was drawn to the ratio 40 that equivalent to 0.3mm diameter from 2.0mm after extrusion (Figure 3.5 (b)). During the drawing, the formation of fibrils will form in morphological structure of filament blend.

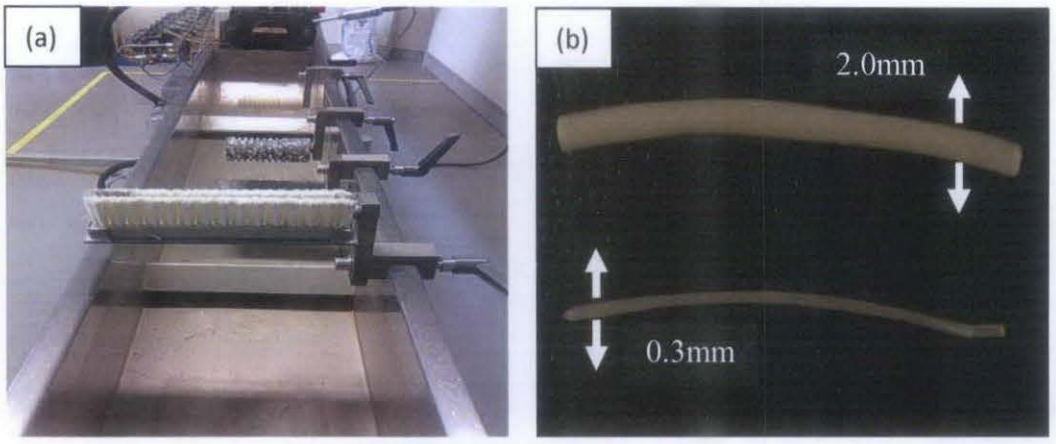

Figure 3.5: The pellet (a) The drawing process; (b) Comparison between drawn and undrawn LDPE/PET.

3.3.5 Injection Molding

The neat LDPE, undrawn LDPE/PET and drawn LDPE/ PET (MFCs) pallets (Figure 3.7) were formed into composite structures via injection molding (Figure 3.6) at T_m 185°C, pressure 800 bar and clamping force at 380 bar to make dumbbell and bar sample for tensile and flexural testing (Figure 3.8). This step was critical to the successful creation of drawn LDPE/PET into MFCs as it ensures the formation of an isotropic matrix while retaining the highly oriented reinforcing fibrils. If the processing temperature is too high the fibrils will melt and the reinforcing effect will be lost.

Figure 3.6: Injection Molding.

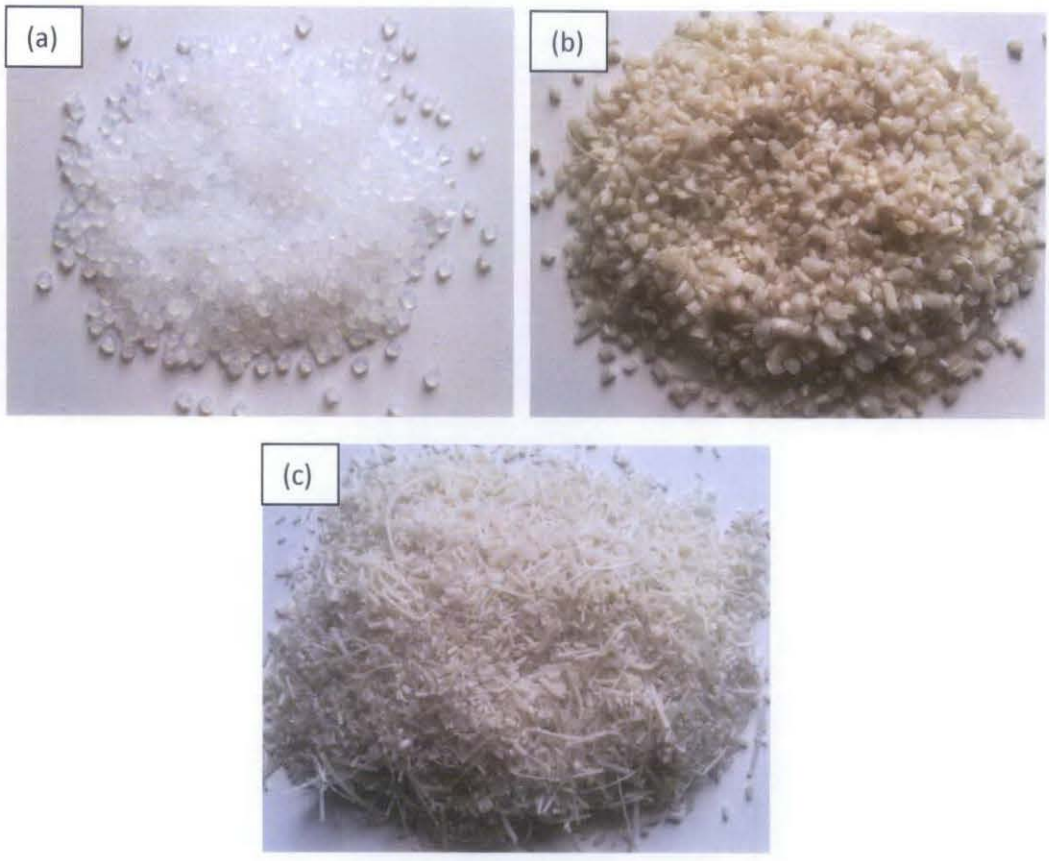

Figure 3.7: Pallets (a) LDPE; (b) Undrawn LDPE/PET; (c) Drawn LDPE/PET (MFCs).

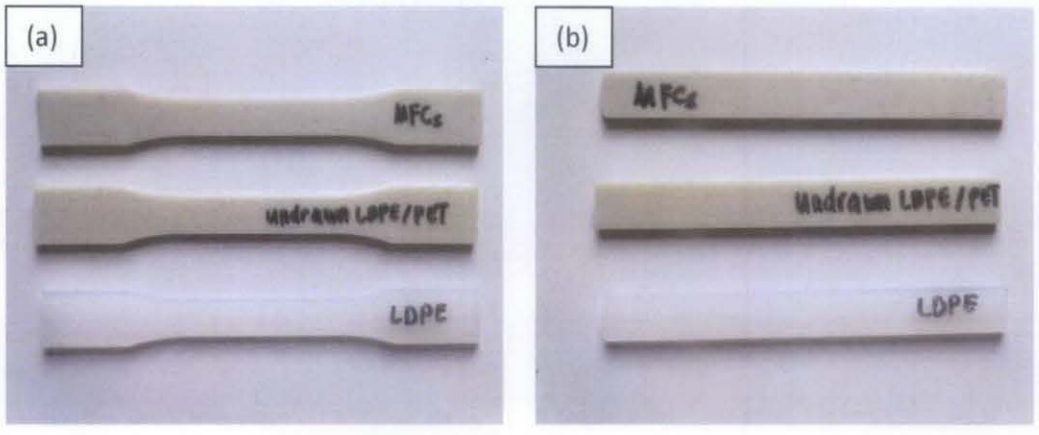

Figure 3.8: The sample (a) Dumbbell; (b) Bar sample after injection molding.

The dumbbell and bar samples were processing using the standard ASTM mold where 10 samples for each neat LDPE, undrawn LDPE/PET and drawn LDPE/PET (MFCs) were done for testing and analysis.

3.3.6 Characteristics

The morphological structure of LDPE/PET checked using the scanning electron microscope (SEM) before drawing and after drawing with 1000X magnification as shown in Figure 3.9. The samples were prepared by immersed in liquid nitrogen for at least 10 minutes and fracture which allowed performing extraction. Then, samples were coated with a fine layer of gold to aid in electron conductance for SEM analysis.

Figure 3.9: Scanning Electron Microscope.

3.3.7 Tensile and Flexural Testing

The strength of the neat LDPE, undrawn LDPE/PET and drawn LDPE/PET (MFCs) determined by using the 5 kN universal testing machine after injection molding. The tests were carried out according to ASTM D638 and D790 standard (Figure 3.10).

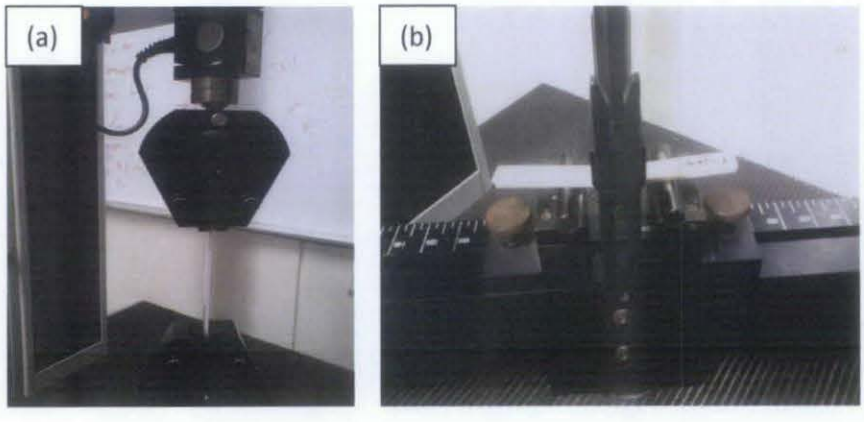

Figure 3.10: Testing (a) Tensile Strength; (b) Flexural Strength.

Test samples underwent conditioning and testing at 23 ± 2°C temperature and 50 ± 5% relative humidity. The tensile and flexural test performed on a minimum of five samples and average strength of each set of result is reported.

Tensile test were performed by followed the ASTM standard D638 (Standard Test Method for Tensile Properties of Plastics) and using thread-locking jaw grips with an abraded inner surface to allow better specimen-grip adhesion. Five samples of Type 1 used, gauge length was 50 mm, crosshead speed was 5 mm/min and preload was 5 kN as shown in Figure 3.11. The Stress/Strain graphs were used to measure on the tensile strength.

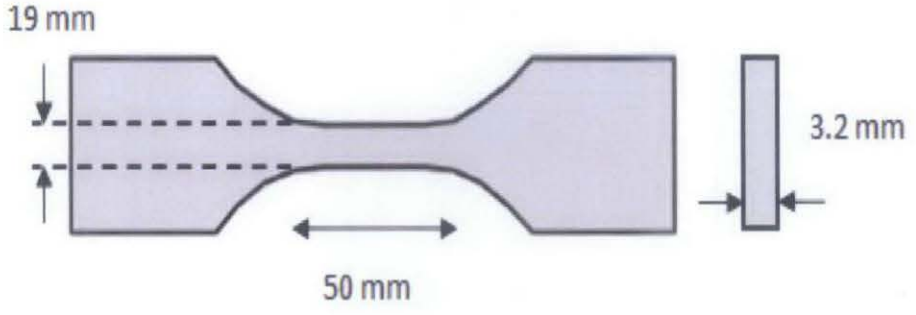

Figure 3.11: Dumbbell samples.

Flexural test were performed in accordance to ASTM standard D790 (Standard Test Methods for Flexural Properties of Unreinforced and Reinforced Plastics and Electrical Insulating Materials). A three point bending setup was used for 5 samples of procedure A with support span, 51.2 mm. The crosshead speed was 0.1 mm/min, preload was 15 kN and thickness was 3.2 mm as shown in Figure 3.12.

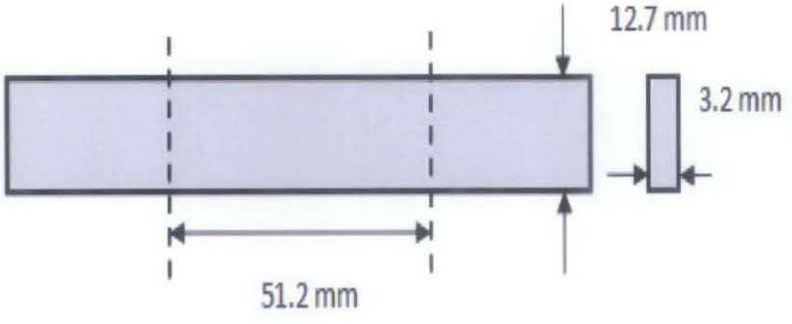

Figure 3.12: Bar samples.

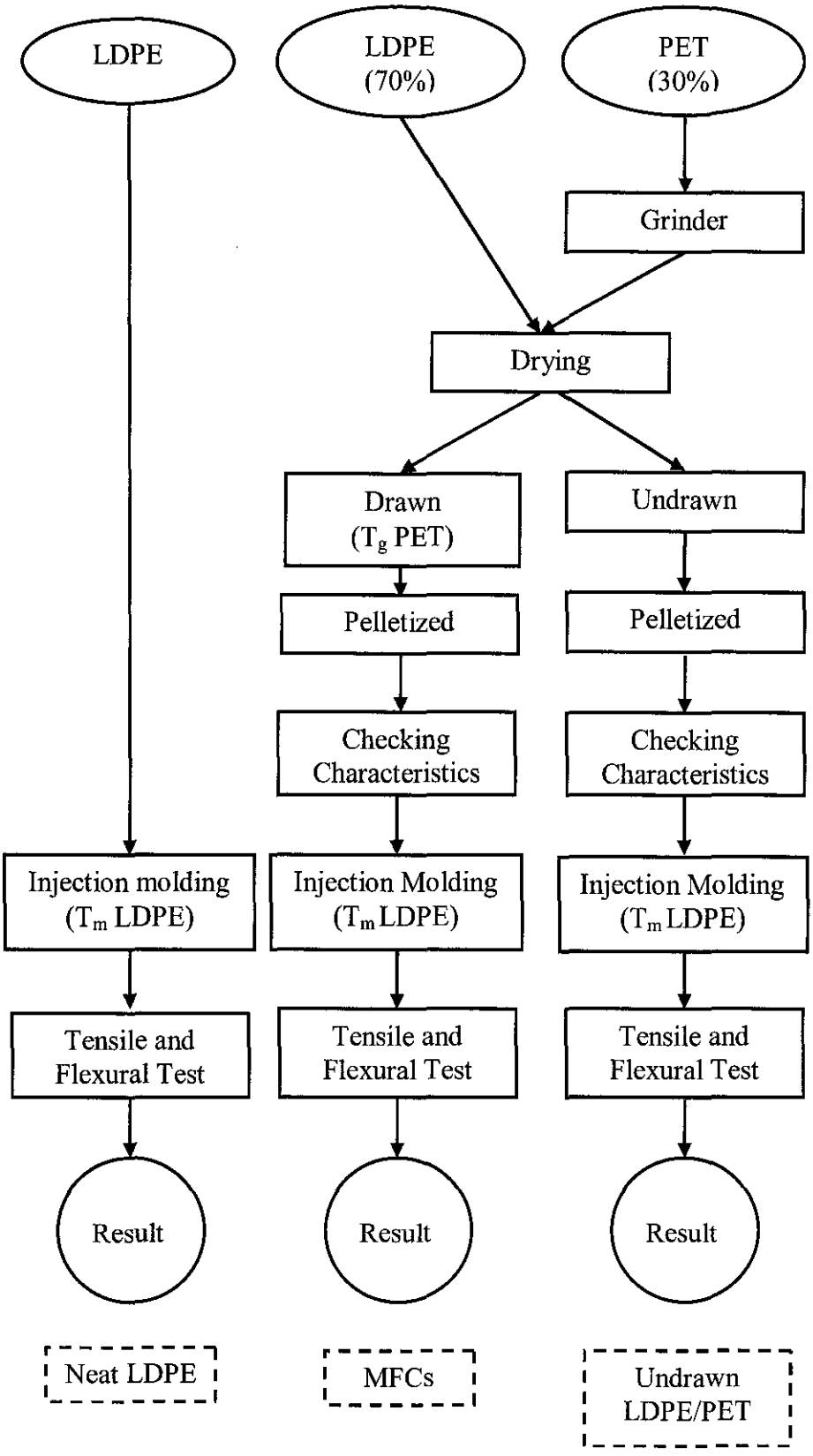

Figure 3.13: Overall Process Methodology.

3.4 GANTT CHART AND PROJECT WORK

No	Activities	Week														
		1	2	3	4	5	6	7		8	9	10	11	12	13	14	15
1	Grinder the PET																
2	Dried up LDPE and PET																
3	Mix and Blend LDPE with PET																
4	Drawing the MFCs																
5	Submission of Progress Report																
6	Check the Characteristics of MFCs																
7	Injection Molding of MFCs																
8	Tensile and Flexural Test																
9	Pre-EDX																
10	Submission of Dissertation(Soft Bound)																
11	Submission of Technical Paper																
12	Oral Presentation																
13	Submission of Dissertation(Hard Bound)																

MID SEMESTER BREAK

▲ Milestone

▯ Process

Figure 3.14: Gantt chart and Project work.

CHAPTER 4
RESULTS AND DISCUSSION

This chapter will discuss data gathering and analysis of the work done throughout the project. All manufacturing parameters were identified to ease the processing and characterisation of LDPE/PET MFCs with blend ratio of 70/30 wt%.

4.1 MORPHOLOGICAL CHARACTERISTICS

The morphological characteristic of MFCs is an important parameter to observe the formation of the fibrils. To check on the morphological characteristics, SEM analysis were done on two extruded samples which were undrawn and drawn LDPE/PET. Figure 4.1 displays the morphological structure of undrawn LDPE/PET. The molecular arrangement of undrawn LDPE/PET is characterized by an isotropic and homogenous dispersion of sphere shape of PET in the LDPE matrix. Sphere shape particles of PET from 15-49 μm in diameter existed in the blends which were aligned in the direction of extrusion. The size and shape of the dispersed phase depend on viscosity, composition, elasticity, thermal and interfacial tension of the blend [1].

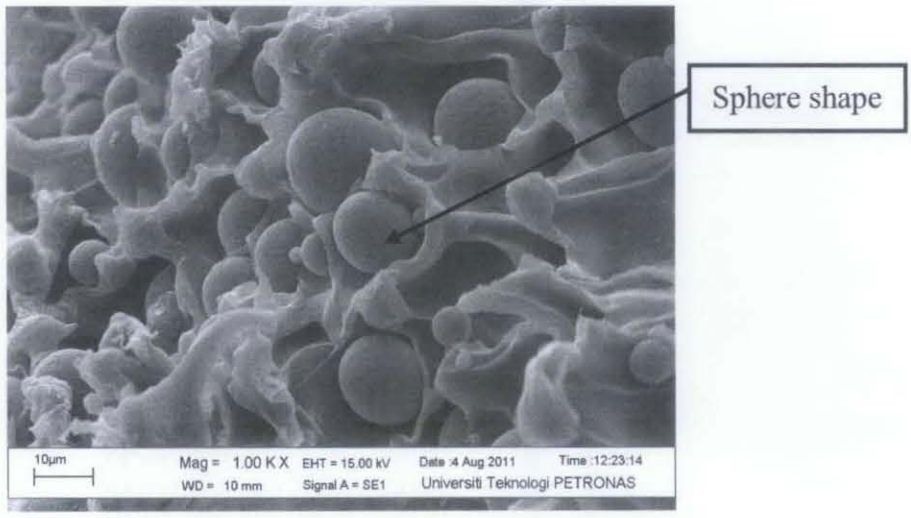

Figure 4.1: SEM image of undrawn LDPE/PET.

The morphological development of the MFCs by drawing process, the suspended and elongated of polymer blend tends to align parallel to the direction of elongation and stress flow direction as shown in the Figure 4.2. The molecular arrangement of drawn LDPE/PET shown in Figure 4.2 is the formation of the fibrils shape of PET after the drawing process. After drawing above T_g of PET, the blend components are transforming into highly oriented microfibrils with diameter of 5-7 μm that is equivalent to the drawing ratio of 7-10. The length of MFCs was more than 100 μm. The SEM analysis of the surfaces show very well oriented PET fibrils with a high aspect ratio.

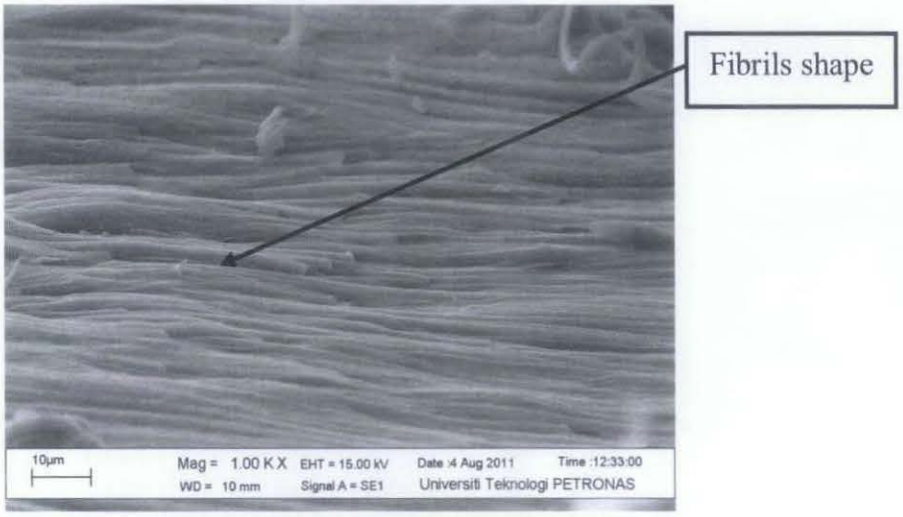

Figure 4.2: SEM image of drawn LDPE/PET (MFCs).

4.2 MECHANICAL PROPERTIES

To determine the mechanical properties of MFCs, five samples were going through the tensile and flexural tests. Another five samples of undrawn LDPE/PET and neat LDPE were tested for comparison. The tensile and flexural properties are shown in Table 4.1.

Table 4.1: Tensile and Flexural Strength.

Type	Sample	Tensile Strength (MPa)	Flexural Strength (MPa)	Flexural Modulus (GPa)
LDPE	1	8.80	9.08	0.59
	2	8.73	9.47	0.63
	3	8.70	8.92	0.59
	4	8.71	8.78	0.55
	5	8.76	10.51	0.64
	Average	8.74	9.35	0.60
Undrawn LDPE/PET	1	8.34	11.42	0.74
	2	8.24	12.24	0.70
	3	8.19	11.16	0.70
	4	8.40	11.65	0.72
	5	8.47	11.25	0.70
	Average	8.33	11.54	0.71
Drawn LDPE/PET (MFCs)	1	13.32	14.67	0.76
	2	13.00	14.95	0.79
	3	12.32	15.45	0.80
	4	13.40	15.01	0.79
	5	12.96	14.89	0.79
	Average	13.00	14.99	0.79

4.2.1 Tensile Strength

Figure 4.3 shows the tensile strength of neat LDPE, undrawn LDPE/PET and drawn LDPE/PET. The tensile strengths for neat LDPE, undrawn LDPE/PET and drawn LDPE/PET were 8.7, 8.3 and 13.0 MPa, respectively. Tensile strength of MFCs improved by 50% compared neat LDPE. The formation of fibrils has improved the strength of MFCs. This is because of the formation of fibrils which align the morphological structure of MFCs has stronger reinforcement of PET to LDPE matrices. Although PET inherits higher tensile strength compared to LDPE, the undrawn LDPE/PET has a slightly lower tensile strength. This may be due to weaker bonding between sphere shaped PET particles and LDPE. Drawing process not only makes the PET fibrils much stronger but also provides some kind of bonding between the PET fibrils and LDPE.

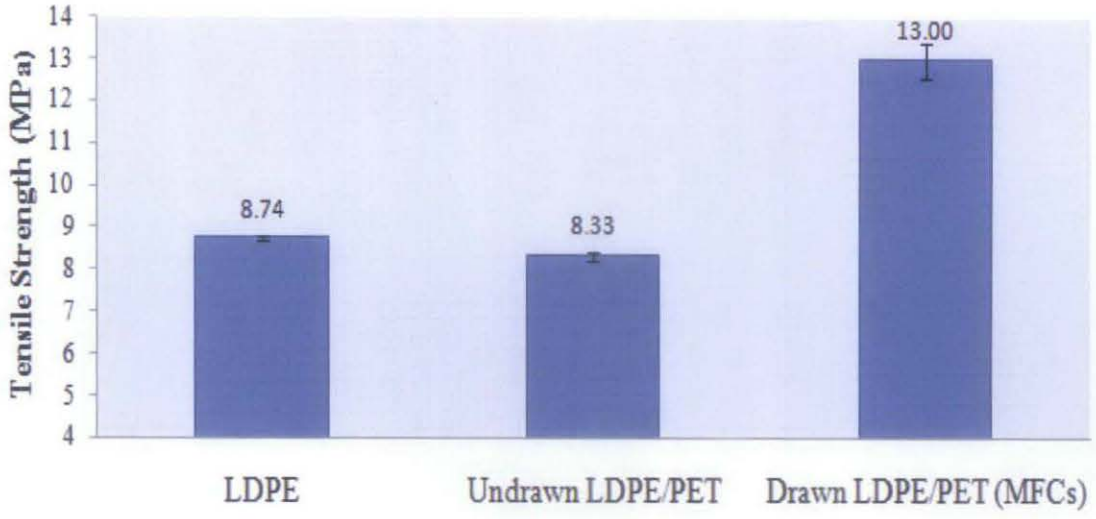

Figure 4.3: Tensile strength of LDPE, undrawn LDPE/PET and MFCs.

4.2.2 Flexural Strength and Modulus

Figure 4.4 shows the flexural strength of neat LDPE, undrawn LDPE/PET and drawn LDPE/PET. The flexural strengths for neat LDPE, undrawn LDPE/PET and drawn LDPE/PET were 9.4, 11.5 and 15.0 MPa, respectively. The flexural strength improvement of MFCs compared to neat LDPE was 60%.

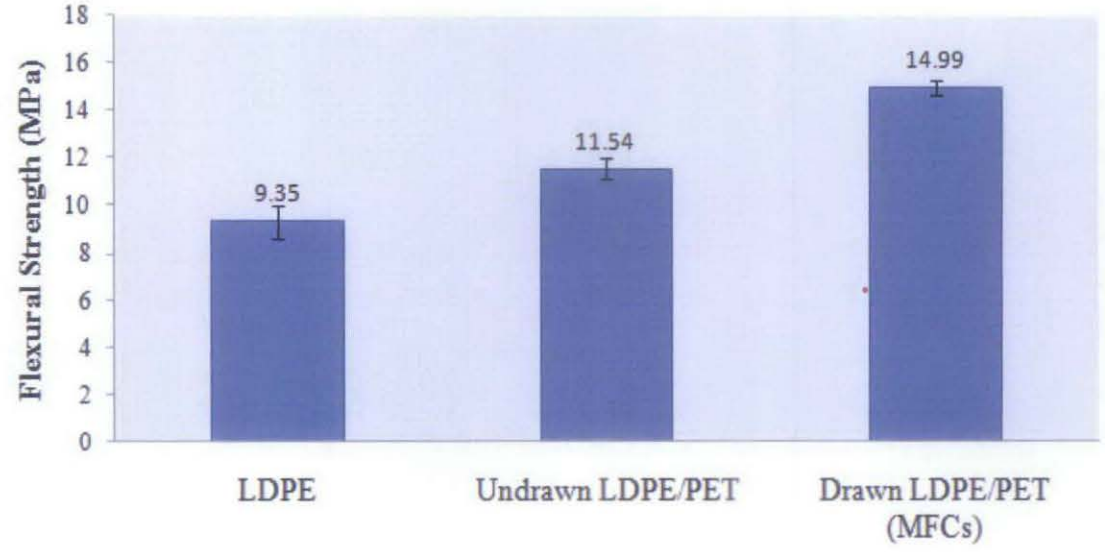

Figure 4.4: Flexural Strength of LDPE, undrawn LDPE/PET and MFCs.

Figure 4.5 shows the flexural modulus of neat LDPE, undrawn LDPE/PET and drawn LDPE/PET. The flexural modulus for neat LDPE, undrawn LDPE/PET and drawn LDPE/PET were 0.6, 0.7 and 0.8 GPa, respectively. The flexural modulus improvement of MFCs compared to neat LDPE was 30%.

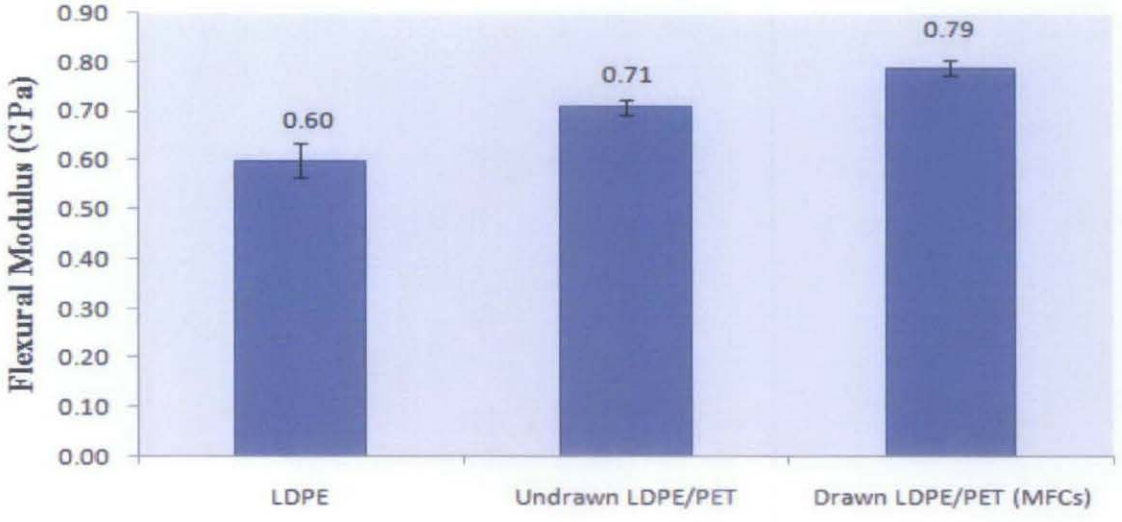

Figure 4.5: Flexural Modulus of LDPE, undrawn LDPE/PET and MFCs.

From the analysis of flexural strength and modulus, the stronger reinforcement of PET fibrils to LDPE matrices improves stiffness significantly. Unlike tensile strength, flexural properties are significantly improved with undrawn LDPE/PET because sphere shaped PET does help the stiffness of the blend. As expected, PET fibrils provide even better improvement toward flexural properties because of their stronger strength and potentially better bonding between drawn PET fibrils and LDPE.

CHAPTER 5
CONCLUSIONS AND RECOMMENDATIONS

5.1 CONCLUSIONS

The main goal of this project is to process and characterise PE/PET MFCs. The following conclusions can be drawn from this study:

- The processing of PE/PET MFCs was successfully done with 70/30 wt% ratio.
- The formation of fibrils was observed through SEM images after the drawing process.
- The sizes of fibrils after the drawing process were 5-7 μm in diameter and 100 μm in length.
- MFCs showed improvement in tensile strength of 50% compare to neat LDPE.
- For flexural strength and modulus, MFCs showed improvements of 60% and 30%, respectively.

5.2 RECOMMENDATIONS

The processing technique of MFCs can be improved by provided better analysis and equipment for the drawing process. As the drawing process was the crucial process in MFCs where it need totally controlled condition where T_g of reinforced polymer must be constant to allow the formation of fibrils from the sphere shape. With a good controlled condition, the orientation of the fibrils will be better thus enhancing the strength more than usual.

The suitable mixing condition by using the compatibilizer may be considered for MFCs. As been stated before the undrawn LDPE/PET has lower strength than neat LDPE cause of the weak interfacial bonding of LDPE and PET. By adding compatibilizer to the polymer blend to facilitate its distribution on the matrix and reinforced polymer interfaces thus improve the mechanical properties. But further study on this problem is needed to ensure compatibilizer not disturb and diminish the formation of fibrils [8].

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