CHAPTER III
EXPERIMENTAL

3.1. Materials

The commercial grade of high density polyethylene (HDPE) was obtained from different sources as shown in Table 3.1

Table 3.1 The physical properties of high density polyethylene.

Grade	**Type of processing**	**MFI (g/10 min.)* (Supplier)**	**MFI (g/10 min.)* (Experiment)**
H6205JU	Injection molding	5.50	6.3
H5818J	Injection molding	18.00	12.316
H5603B	Blow molding	0.03	0.027
1600J	Injection molding	14.00	11
2208J	Injection molding	4.00	4.31
5000S	Extrusion	0.80	0.8154
HDPE_X	Blown film	0.04	0.2 (190°C/10 kg)

*At 190 °C /2.16 kg

In this research work, raw materials were supported by two suppliers which were Thai Polyethylene Co.,Ltd. and Bangkok Polyethylene Co.Ltd.

3.1.1. Characterization

The molecular parameters by mean of molecular weight (Mw) and molecular weight distribution (MWD) of the samples were determined by gel permeation chromatography using a Water Sci GPC-150 C instrument at 140°C with 1,2-dichlorobenzene (ODCB) as a solvent. The three columns (10^4, 10^5, and 10^6 Å) were calibrated by using 10 polystyrene standard samples. The results are listed in Table 3.2. The raw data is shown in Appendix A.

Table 3.2 The molecular parameters of HDPE.

Grade	Mw	MWD	Code
H6205JU	64,997	23.1	LW1
H5603B	176,604	26.1	HW1
HDPE_X	64,482	33.1	LW2
5000S	70,560	30.5	HW2
2208J	64,920	17.7	LD3
1600J	48,361	12.7	LD2
H5818J	51,073	12.5	'LD1

3.2. Equipment

3.2.1. Melt Flow Index Tester

Melt Flow Index (MFI) was measured by using Zwick 405 Extrusion Plastometer with piston load weight of 2.16 kg at 190°C. The geometry of die were 1.180 mm for diameter and 8.00 mm for length. The test conditions were done according to ASTM 1238-82. Appendix B provides the measured data.

3.2.2. The Instron Capillary Rheometer

The pellet samples were measured for their viscosity by using an Instron Capillary Rheometer model 3213 with a 25 kN load cell at constant shear rate which covered the range of shear rate that occurred in the Brabender Single Screw Extruder. The experiment was run at 170 °C. Bagley correction was not applied to the measurement because the used die had tapered entrance with angle of 45 degree. Robinowitsch correction was applied

3.2.3. Universal Testing Machine

The tensile strength of the sample was measured according to ASTM D638-91. The determinations of tensile properties were done on the standard dumbbell-shaped of specimens under the crosshead speed of 450 mm/min for blend system 1 and at the crosshead speed of 100 mm/min for blend system 2 and 3. The different crosshead speed were used, due to the toughness of material; i.e. these data had to be received under the breaking situation of all samples. Appendix C contains the measured data.

3.2.4. Lab Tech Compression Molding

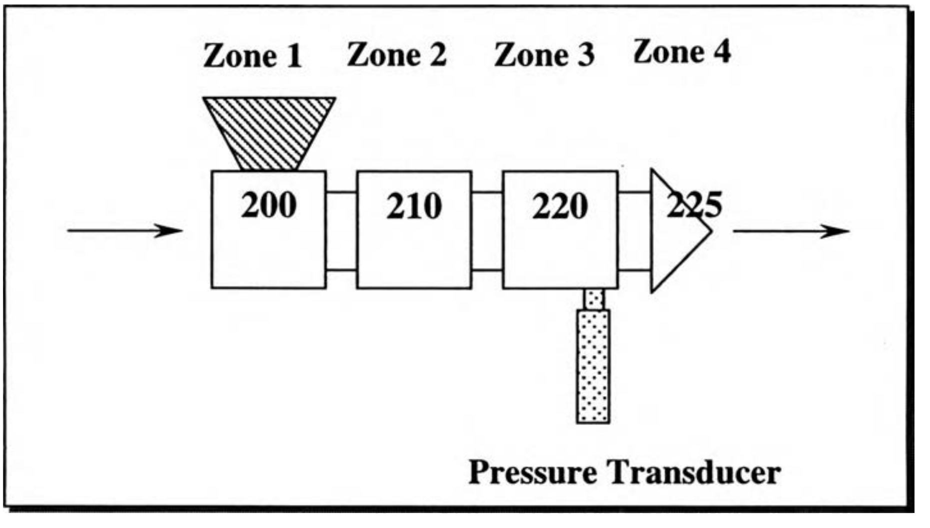
Lab Tech compression molding was used to mold specimens in sheet form for mechanical testing. The pellets were pre-heated at 170°C for 5 min, compressed at 10 tons for 8 min, and then cooled down to 40°C for 8 min.

3.2.5. Zoome Stereo Microscope

The skin texture photographs were obtained from a Zoom stereo microscope, OLYMPUS B071, with a magnification range of 4-80 times. The extrudate were examined at 20x magnification.

3.2.6. The Brabender Single Screw Extruder

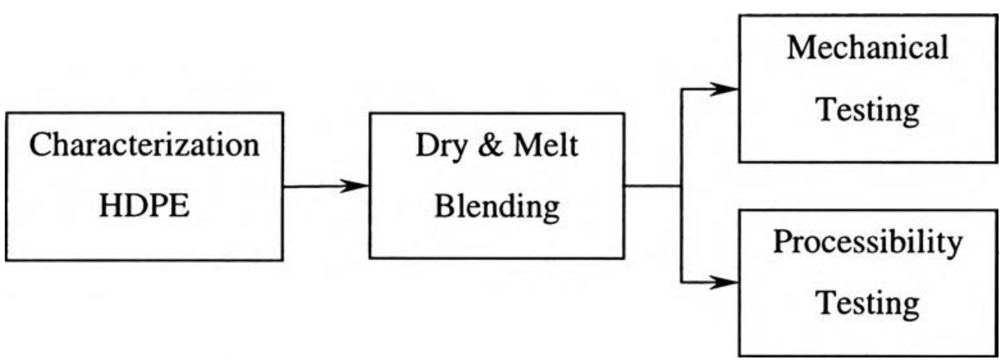
The Brabender single screw extruder was 19/25D of L/D ratio. Pressure transducer was attached at zone 4 to measure pressure build-up during screw running. Screw speed varied from 10 to 30 rpm. Temperature profiles as shown below were set up in the degree Celsius based on the recommendation from instrumental guidebook.



By doing the open discharge, round dies having different diameter of 1 and 4 mm with the same length of 23 mm were used to determine the operating point. The open discharge gave maximum output rate without pressure build-up during screw running. Meanwhile, pressure could raise up to the maximum point by closing die. This practice was called closed discharge, which gave zero output rate. If there was an orifice with a tiny hole in the middle at the end of the screw, pressure drop would be varied and the output rate would be altered. However, in this work, only pressure build-up at the end of the screw was detected by a pressure transducer during extrusion process. Appendix D gives raw data of pressure measurement.

3.3. Methodology

In the experimental procedures, characterization of various types of HDPE was first conducted. Second, the conditions for blending were set up then after that dry blending and melt blending were done to prepare homopolymer blends. Finally, all samples were kept for further testing on the mechanical properties and processibility. All steps were shown in the following diagram.



3.3.1. Condition for Blending

Condition of sample was set up as a function both of Mw and MWD in the composition ratio of 100/0, 75/25, 50/50, and 0/100 by weight. There were 3 blend systems as following.

1. Blends of HDPE with different Mw but same MWD.

LW1 / HW1	LW2 / HW2
Mw = 64,997 / 176,604	Mw = 64,482 / 70,560
MWD = 23/ 26	MWD = 33/ 30
Big Difference in Mw	Small Difference in Mw

Note: Since it was difficult to obtain exactly the same MWD of two HDPEs. Thus, HDPEs having closed MWD (±3) were chosen instead. It was also noticed that broad MWD was chosen rather than narrow MWD to avoid difference in MWD caused by ±3 error, i.e. to diminish the effect from the ±3 error.

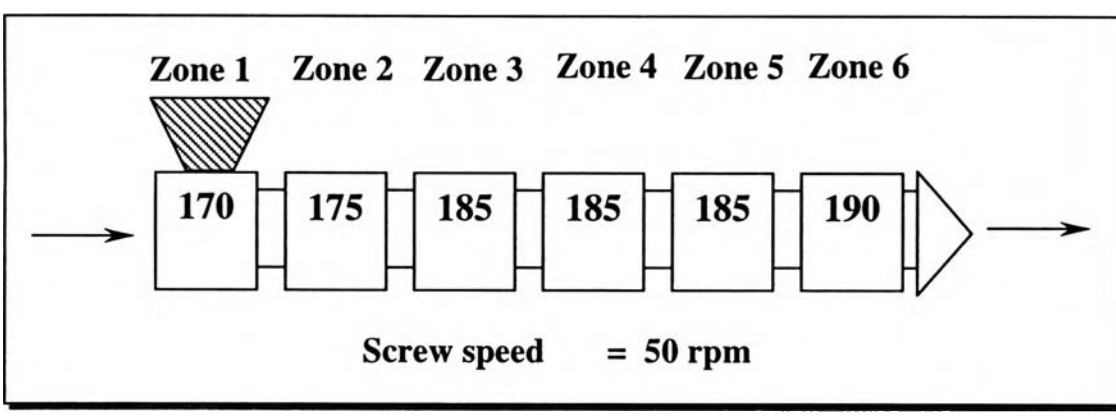
2. Blends of HDPE with difference MWD but the same Mw.

LD3 / LW2	LD3 / LW1
Mw = 64,920 / 64,482	Mw = 64,920 / 64,997
MWD = 17/ 33	MWD = 17/ 23
Big Difference in MWD	Small Difference in MWD

3. Blends of HDPE having similar Mw and MWD and used as a reference sample.

LD2 / LD1
Mw = 48,361 / 51,073
MWD = 12/ 12
Reference Sample

Homopolymer blending were firstly prepared by dry blending in Bosco Pot and then melt blending by COLLIN co-rotating twin screw Kneader ZK-25 (25 mm diameter X 30 D long). The temperature profiles (^{o}C) of these processing were shown in the diagram below based on the recommendation from suppliers.



The extrudate was cooled in the water bath around 25^{o}C and then fed to a Planetrol 075D2 pelletizer to cut into pellet form. The samples of were collected for further testing on the processibility and mechanical testing.

3.3.2. The Operating Point

According to extrusion process, the throughput of the extruder and its die can be determined by collecting the sample within 1 min and converting mass flow rate to volumetric flow rate by applying density. Therefore, the operating point was determined as the output rate at a certain pressure developed. Theoretically, the operating point is determined by the intersection of screw and die characteristics (which is shown by the plot of throughput versus pressure drop). These concepts are developed as follows.

For Newtonian fluid, the output flow rate is determined by the combination of drag flow and pressure flow (Strong, 1996).

$$Q = 1/2\pi^2 D^2 NH \sin\phi\cos\phi - \frac{\pi DH^3 \sin^2\phi\Delta P}{12\eta L} \tag{3.1}$$

Where, Q = flow rate (m^3/min) D = screw diameter (m)

N = screw speed (rpm) H = channel depth (m)

ϕ = helix angle (degree) e = flight width (m)

w = channel width (m) L = screw length (m)

η = fluid viscosity (Pa·s)

1. In the screw, the flow rate is due to drag flow and pressure flow. If there is no pressure build-up, for example, no breaker plate or die, the output will be maximum, Q_{max}. The drag flow ideal equation which occurs in the screw when there is no die (open discharge) is shown below.

Open discharge : $$Q = Q_{max} = 1/2\pi^2 D^2 NH \sin\phi\cos\phi \tag{3.2}$$

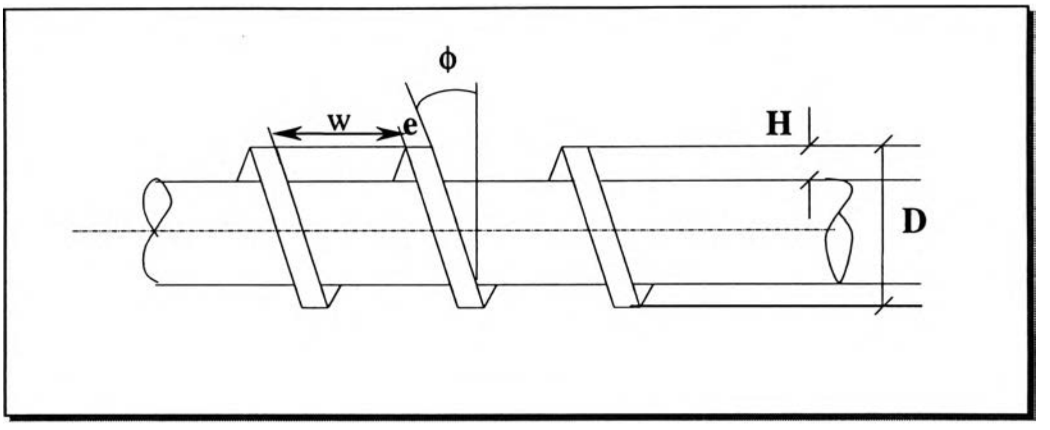


Figure 3.1 The screw geometry.

2. For close discharge, there is maximum resistance and Q = 0, and we can equate the drag and pressure flow expressions

Close discharge : $\Delta P = \Delta P_{max}$, $Q = 0$;

i.e. $$1/2\pi^2 D^2 NH \sin\phi\cos\phi = \frac{\pi DH^3 \sin^2\phi\Delta P}{12\eta L} \qquad (3.2)$$

$$\Delta P_{max} = \frac{6\pi DLN\eta}{H^2 \tan\phi} \qquad (3.3)$$

These points represent the extremes for screw characteristic (Figure 3.2).

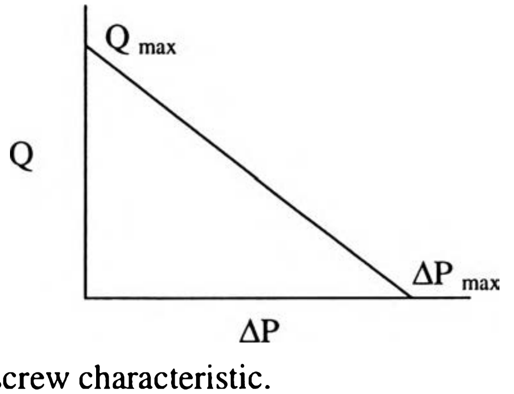


Figure 3.2 The screw characteristic.

3. For die, the pressure is needed to force the melt through the die. Therefore, output rate in die is dependent on the drag flow. Die characteristic shows the proportional dependence of flow rate on pressure drop. The output increases with increasing pressure. This can be written as the diagram given in Figure 3.3. The intersection point is the operating point, where the optimum throughput and pressure drop are.

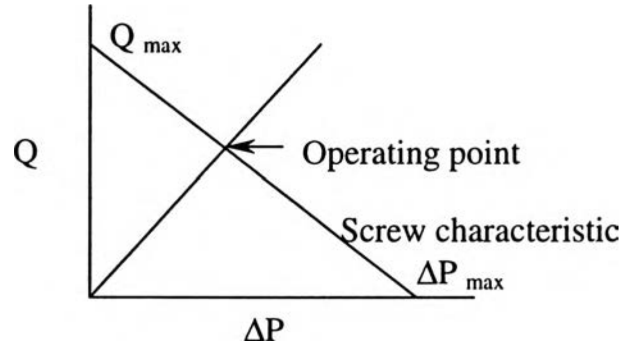


Figure 3.3 Intersection of screw and die characteristics.

The output of individual dies depends obviously on their shapes. In general

$$Q = KP/\eta \tag{3.5}$$

Where, K is the shape-dependent and η is the viscosity.

For round capillary die :

$$K = \frac{\pi R^4}{8L} \tag{3.6}$$

Where, R = radius (m)

L = length (m)

The positions of lines in Fig. 3.3 are changed by changes in operating conditions. If the extruder speed (N) is increased, the screw characteristic moves up. If the geometry of die is changed, e.g. the radius of cylinder (R) is increased, the slope of the die characteristic is increased.