# CHAPTER III

# EXPERIMENTAL

## 3.1 Materials

### 3.1.1 High density polyethylene(HDPE)

In this study, high density polyethylene, under the trade name of Thai-Zex 7000F was used. It was manufactured by Bangkok Polyethylene Public Company Limited.

### 3.1.2 Linear low density polyethylene (LLDPE)

Two types of linear low density polyethylene, Dowlex D2045 and Elite 5100 were used which were produced by using Ziegler-Natta catalyst(Z-NLLDPE) and metallocene catalyst (MLLDPE), respectively. Both were manufactured by Dow Chemical Co.,Ltd. The basic properties of both resins are shown in Table 3.1

Table 3.1 : The basic properties of Dowlex D2045 and Elite 5100

| Properties | Dowlex D2045 | Elite 5100 |
|---|---|---|
| Melt flow index (g/10 min.) | 1.0 | 0.85 |
| Density ($g/cm^3$) | 0.920 | 0.920 |

## 3.2 Blending and sample preparation

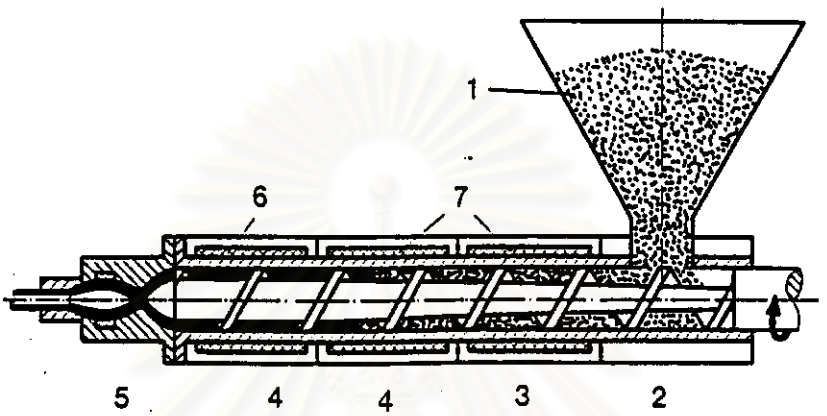
### 3.2.1 Blending

A dry blend of HDPE and LLDPE (MLLDPE or Z-NLLDPE) was performed according to the formulation shown in Table 3.2 using the high speed mixer. Mixing time for a period is 10 min., the blend was melted and extruded by single screw extruder as shown in Figure 3.1. The temperature profile of the single screw extruder was 180/200/230/230/230°C, rotor speed was kept constant at 100 rpm. The extruded was cooled in water at 30°C and subsequently cut into granules.

Table 3.2 : Formulation of HDPE/LLDPE blends

| HDPE (% by weight) | LLDPE (% by weight) |
|---|---|
| 100 | 0 |
| 95* | 5 |
| 90 | 10 |
| 85* | 15 |
| 80 | 20 |
| 75 | 25 |
| 70 | 30 |
| 60 | 40 |

* this formulation used for HDPE/MLLDPE only




zone 2 : temperature = 180°C

zone 3 : temperature = 200°C

zone 4 : temperature = 230°C

zone 5 : temperature = 230°C

Figure 3.1 : Single screw extruder

### 3.2.2 Molding and specimen preparation

Sample for stiffness testing was prepared by compression molding into plates of 2 mm. thickness, using the blend 43 g/sample. The condition of molding was set at 190°C, 5 MPa for 6 min. The molded plate was then cooled by cooling water at rate of -15°C/min. for 10 min. then the temperature was lower. After cooling to room temperature, they were punched into the rectangular shape, 1x5 cm. size.

### 3.2.3 Blown film extrusion sample

Blown film sample was processed as shown in Figure 3.2. Molten resin was extruded through a circular die into tubular extruder and blown by air until it reached the desired width (590 mm.) and thickness (25 micron or 15 micron). The polyethylene film was cut to dumbell shape for tensile strength and elongation testing. For film impact testing, it was cut into approximate 100x100 mm. from the film along its circumference. Finally, the film was sealed and tested the seal strength.

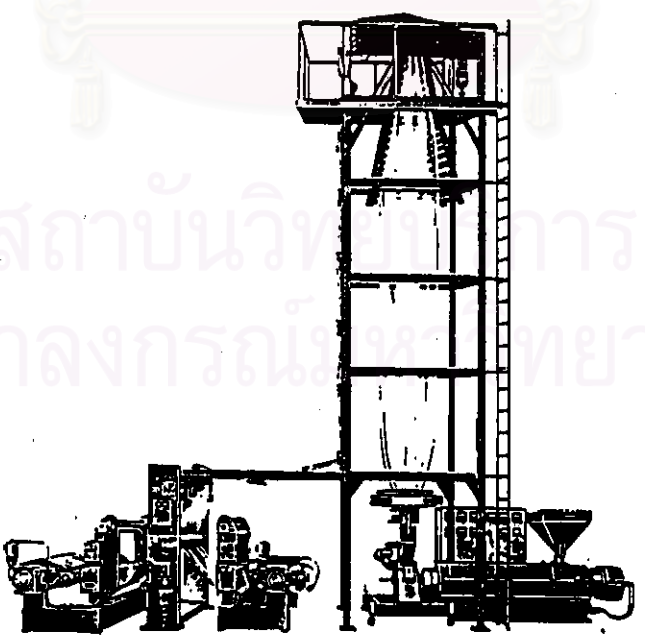
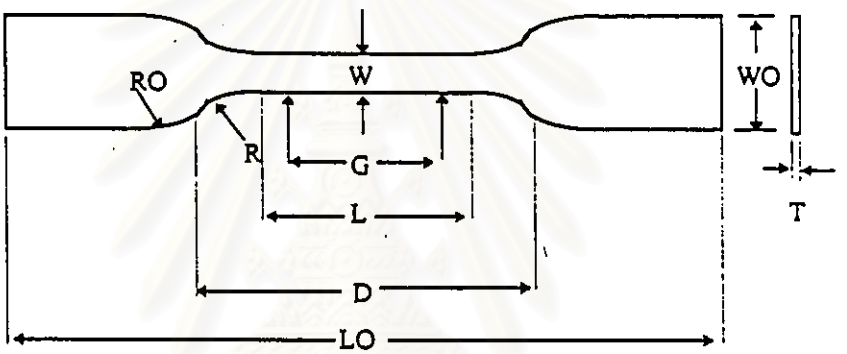


Figure 3.2 : Blown film processing

## 3.3 Mechanical testing of sample

### 3.3.1 Tensile strength and elongation testing

The tensile strength and elongation properties were measured according to ASTM D-638 using Instron Model 4501. The dumbell specimen of type IV was used, Figure 3.3 shown the dimensions of dumbell specimen of type IV. The crosshead speed was set constant at 5 mm/min., 5 specimens were tested and their average value was taken.




| Dimension (see drawing) | ASTM D-638, type IV (mm.) |
|---|---|
| W - Width of narrow section | 6 |
| L - Length of narrow section | 33 |
| WO - Width over-all, min. | 19 |
| LO - Length over-all, min. | 115 |
| G - Gage length | 25 |
| D - Distance between grips | 64 |
| R - Radius of fillet | 14 |
| RO - Outer radius | 25 |

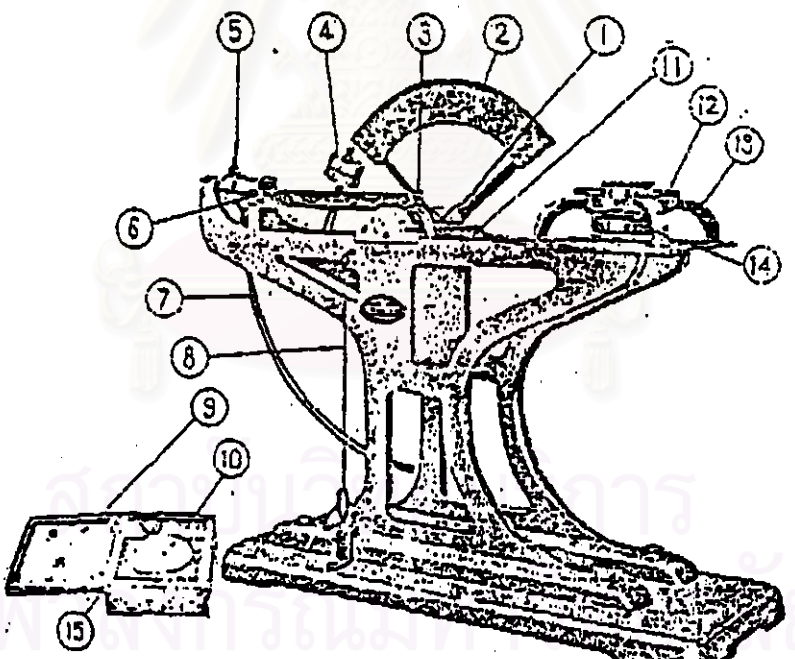
Figure 3.3 : The dimensions of dumbell specimen of type IV.

### 3.3.2 Stiffness testing

Stiffness of sheet sample was based on the conditions and performed according to ASTM D-747 (Olsen). For each blend, 5 specimens were taken and the results were average value.

### 3.3.3 Film impact testing

Film impact testing was performed according to ASTM D-256. Test produced by the impact tester as shown in Figure 3.4. The sample was placed on the tester, the pendulum was released to hit the sample by its head and measured the energy that cause the sample to fracture. Each formulation, 5 specimens were taken and the results were average value.




| | | |
|---|---|---|
| 1. Maximum Reading Pointer | 6. Pendulum | 11. Weight fixing arm |
| 2. Scale Plate | 7. Impact arm | 12. Specimen clamp handle |
| 3. Stud for moving the pointer | 8. Plumb bob | 13. Specimen clamp ring |
| 4. Balance weight | 9. Accessories box | 14. Specimen clamp frame |
| 5. Latch | 10. Dead weight | 15. Impact head |

Figure 3.4 : The impact tester

### 3.3.4 Seal strength testing

After sealing the film, the seal strength was tested as same as tensile strength testing method. Seal strength is the load(kg.) at tensile strength at break of sample. Each formulation, 5 specimens were tested and their average value was reported.

## 3.4 Clarity of film testing

The film specimen was tested for the clarity by using Densitometer Macbeth/TR 927. The light was emitted through the sample and the detector detected the transmittance of light. For each formulation, 5 points were tested and their average value was reported.

## 3.5 Sample characterization

### 3.5.1 Melt flow index (MFI) analysis

The weight in grams of extrusion of molten resin through an orifice of a specified diameter and length for 10 min. under a prescribed weight of 2.6 kg. and at a temperature of 190°C was measured, by using an Auto Melt Indexer. The test was measured according to ASTM D-1238.

### 3.5.2 Density measurement

The density measurement of resin can be performed by using the density gradient technique, the test was according to ASTM D-1505. The density of the specimen shall be determined by the density gradient tube. Toluene and carbon tetrachloride were selected for the density gradient study of the HDPE in the present study. The position along to form a calibration curve was determined the sample density.

### 3.5.3 Differential scanning calorimetry (DSC)

The melt temperature of the HDPE/LLDPE blends were tested according to ASTM D-2117 by using the Perkin-Elmer differential scanning calorimeter tester. Each formulation was weight and placed in an aluminium pan, the sample was heated from 30°C to 200°C at a heating rate of 10°C/min. A DSC scan of the specific energy against the temperature was obtained. The melt temperature was calculated based on the fact that 100% crystallinity HDPE requires an energy of 293 J/g

### 3.5.4 Dynamic mechanical thermal analysis (DMA)

The Tg of HDPE/MLLDPE (75/25), HDPE/Z-NLLDPE (75/25), HDPE, MLLDPE and Z-NLLDPE were measured by using dynamic mechanical thermal analyzer, NETZSCH DMA 242. The specimens were a rectangular sheet of 1 cm. in width, 4 cm. in length and 2 mm. in thickness. The samples were cooled to -150 $^{\circ}$C by using liquid nitrogen. Then the temperature was increased to 50$^{\circ}$C with the rate of 3$^{\circ}$C/min. The test was conducted in a single cantilever mode at a frequency of 1 Hz.