CHAPTER III

EXPERIMENTAL

3.1 Materials

3.1.1 Linear low density polyethylene (LLDPE)

There are two types of linear low density polyethylene resin for using as ;

- Dowlex 2045G a product of Siam Polyethylene Company Limited, was produced by Ziegler-Natta catalyst and octane-based copolymer (hereafter, zn-LLDPE), with a melt flow index of 1.0 g/10 min and a density of 0.920 g/cm^3.

- Elite 5100G a product of Siam Polyethylene Company Limited, was produced by Metallocene catalyst and octane-based copolymer (hereafter, m-LLDPE), with a melt flow index of 0.85 g/10 min and a density of 0.920 g/cm^3.

3.1.2 High density polyethylene (HDPE)

There are two types of high density polyethylene resin for using as;

- H 5604 F a product of Thai Polyethylene Company Limited, having a melt flow index of 0.04 g/10 min and a density of 0.956 g/cm^3 (hereafter, HDPE1).

- HTA 800 is produce by ExxonMobil Chemical, having a melt flow index of 0.70 g/10 min and a density of 0.961 g/cm^3 (hereafter, HDPE2).

3.1.3 Low density polyethylene (LDPE)

Low density polyethylene resin for using as; JJ4324 a product of Thai Petrochemical Industry Public Company Limited, having a melt flow index of 5.00 g/10 min and a density of 0.922 g/cm^3 (hereafter, LDPE).

3.2 Instruments and Apparatus

There are instruments and apparatus for research as follows:

1. Blown film machine (single screw), YEI machine
2. Universal testing machine, LLOYD model LRX plus
3. Over head projector
4. Elmendorf tear tester, GOTECH model GT-7055-A
5. Dart drop tester
6. Haze measuring machine, BYK-Gardner GMBH : Haze-gard dual
7. Gloss measuring machine, BYK-Gardner GMBH : Color-guide gloss
8. Weighing machine
9. Seal temperature machine
10. Differential scanning calorimeter, Metter Toledo DSC-822

3.3 Experimentation for a blown film LLDPE/LDPE/HDPE

3.3.1 Mixing

A LLDPE (m-LLDPE or zn-LLDPE), HDPE (HDPE1 or HDPE2) and LDPE were mixed in mixing tank for 10 min, with according to the formulations (Table 3.1).

Table 3.1 : Formulation of LLDPE/HDPE/LDPE mixes (% by weight)

Polymer type / Formulation	zn-LLDPE	m-LLDPE	HDPE 1	HDPE 2	LDPE
zH000	85	0	0	0	15
mH000	0	85	0	0	15
zH105	80	0	5	0	15
zH110	75	0	10	0	15
zH115	70	0	15	0	15
zH120	65	0	20	0	15
zH130	55	0	30	0	15
zH205	80	0	0	5	15
zH210	75	0	0	10	15
zH215	70	0	0	15	15
zH215.05	80	0	0	15	5
zH215.25	60	0	0	15	25
zH220	65	0	0	20	15
zH230	55	0	0	30	15
mH105	0	80	5	0	15
mH110	0	75	10	0	15
mH115	0	70	15	0	15
mH120	0	65	20	0	15
mH130	0	55	30	0	15

Table 3.1 : Formulation of LLDPE/HDPE/LDPE mixes (% by weight) (cont.)

Polymer type / Formulation	zn-LLDPE	m-LLDPE	HDPE 1	HDPE 2	LDPE
mH205	0	80	0	5	15
mH210	0	75	0	10	15
mH215	0	70	0	15	15
mH215.05	0	80	0	15	5
mH215.25	0	60	0	15	25
mH220	0	65	0	20	15
mH230	0	55	0	30	15

3.3.2 Blown film

Blown film by formulation in Table 3.1 with Blown film machine (shown in Figure 3.1) and set up a heater temperature at 200 °C for screw zone and die zone. The molten resin was extruded by single screw (diameter 45 mm, L/D = 30), speed screw 35 rpm through a circular die (diameter 100 mm and die gab 2.0 mm) into tubular extruder and blown by air until it reached width at 280 mm and adjusting the haul off speed for thickness at 75 micron.

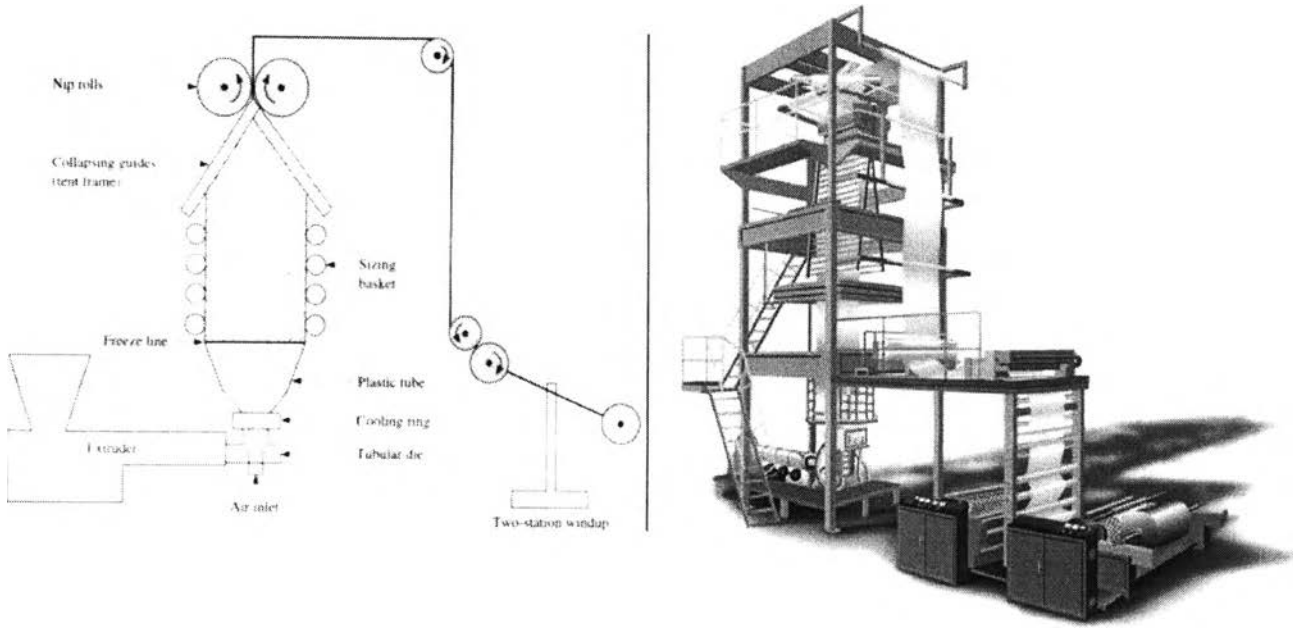

Figure 3.1 Blown film process.

3.4 Mechanical properties testing of blown film

3.4.1 Tensile strength, elongation at break and Young's modulus testing

The tensile test specimens of the blown film with dimensions of width 10 mm length 100 mm and thickness 0.075 mm were measured using Universal testing machine (LLOYD LRX plus) following the procedures described in ASTM D 882.

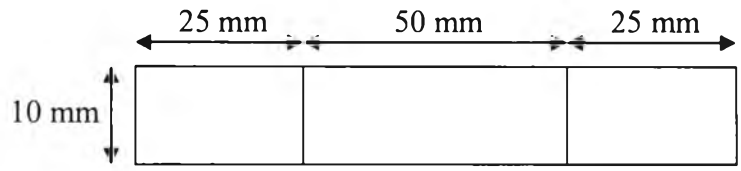

Figure 3.2 Film dimensions of tensile test.

The tensile parameters and testing condition, speed testing 500 mm/min, Gauge length 50 mm and load cell 500 N were used. Each formulation, 5 specimens were tested tensile strength (Mpa), elongation at break (%) and Young's modulus (Mpa) both

machine direction (MD) and transverse direction (TD) and their average value were reported.

3.4.2 Elmendorf tear testing

The Elmendorf tear test specimens for the blown film with dimensions shown in Figure 3.3. It was measured from Elmendorf tester (GOTECH model GT-7055-A) following the procedures described in ASTM D 1922, testing with load 3,200 g Each formulation, there are 10 specimens by machine direction (MD) and transverse direction (TD). To made a reported of average value from testing the tear strength (g).

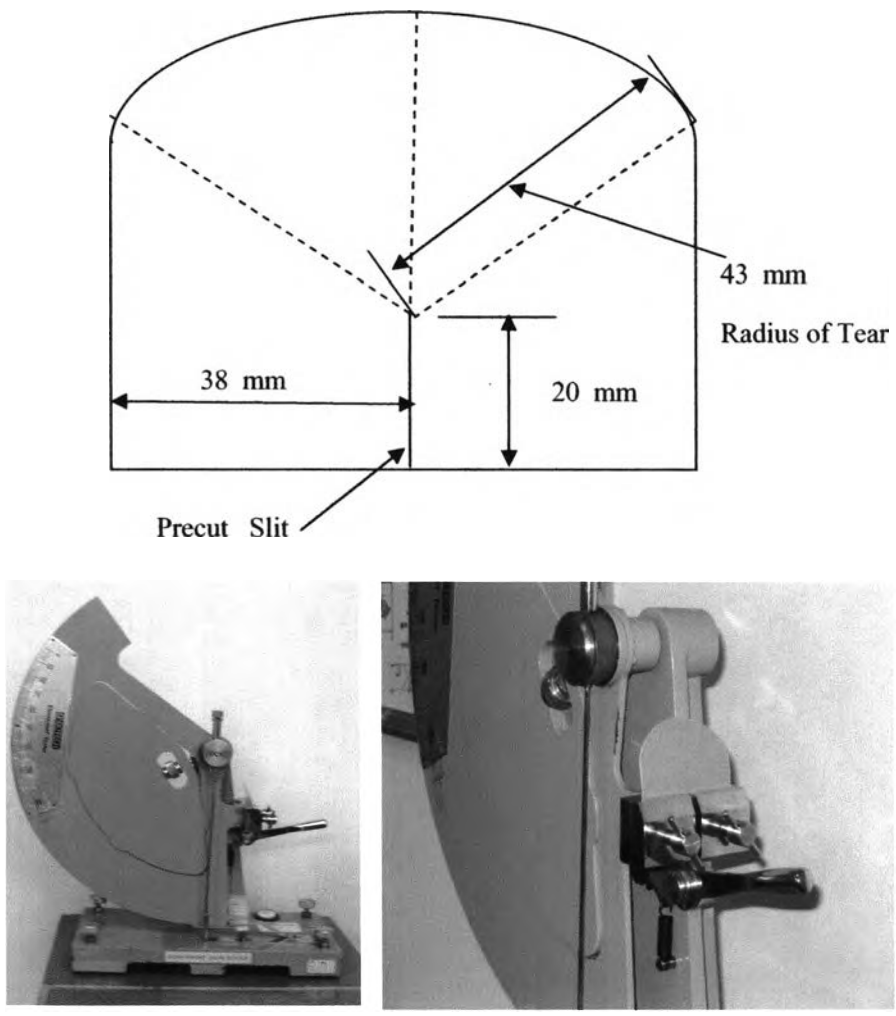

Figure 3.3 Film dimensions of Elmendorf tear test specimens and Elmendorf tear

3.4.3 Dart Impact resistance testing

The dart drop impact resistance test specimens by the blown film with dimensions width 180 mm and length 2000 mm It was measured with dart drop tester following the procedures described in ASTM D 1709 (method A). To made reported as a result of the impact resistance (g) testing.

3.4.4 Puncture resistance testing

The puncture resistance test specimens of the blown film with dimensions diameter 100 mm were measured using Universal testing Machine (LLOYD LRX plus) following the procedures described in ASTM D 3763.

The puncture resistance parameters and testing condition, speeding testing 200 mm/min, diameter of the hole in the clamp 45 mm, and load cell 500 N were used. Each formulation, 5 specimens were tested the puncture resistance (N) and their average value were reported.

3.5 Physical properties testing of blown film

3.5.1 Measuring of haze

The measurement of haze test specimens by the blown film diameter about 76.2 mm. It was measured with haze measuring machine (BYK-Gardner GMBH : HAZE-GARD DUAL) following the procedures described in ASTM D 1003. Each formulation, there are 5 specimens for tested percentage of haze. To made reported as a result of average value.

3.5.2 Measuring of gloss

The measurement of gloss test specimens by the blown film bigger than oval size length 20 mm and width 10 mm. It was measured for gloss measuring machine (BYK-Gardner GMBH : COLOR-GUIDE GLOSS) following the procedures described in ASTM D 2457. It is a measure of the ability of a surface to reflect the incident light. Figure 3.4 illustrates procedure for measuring gloss [8]. Each formulation, there are 5 specimens for tested gloss unit of gloss and made report as result of average value.

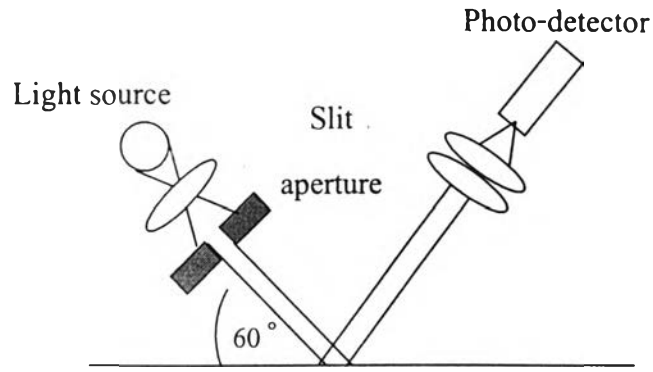

Figure 3.4 Measurement of gloss

3.5.3 Measuring of gel count

The blown film research have a result number of gels on the film. It is difficult with the unaided eyes and fatiguing to the operator. There is a method for measuring of gel counts by use an overhead projector, when light is projected to magnify the film surface ten times (10x) for better inspection and counting of gels in plastic film when show on the screen, according to ASTM D 3351.

The gel counts from 4 strips of 19 cm by 20 cm (1520 cm^2). To cutting the film at random from 7 sections of the blown film. The 4 strips blown film were placed on the overhead projector. To bring the acrylic plate size of 9.0 inch. by 9.0 inch, thickness 3.2

mm. (it had contained three standard holes, 0.4 mm, 0.8 mm and 1.5 mm in diameter) putted on these films and the image of the gel size are clearly projected on the screen. The result of this testing had a size range gel count per 1 m^2 of testing area. The blown film appearance defects were counted and classified into three different size categories, 0.4 to 0.8 mm, 0.8 to 1.5 mm and bigger than 1.5 mm.

3.5.4 Measuring of throughput

Cut and weight film from count times blown film 5 min. The weight (kg) was measured to receive throughput.

3.5.5 Initial seal temperature

The blown film is using seal temperature machine for seal and test seal strength by Universal testing Machine (LLOYD LRX plus) following the procedures described in JIS Z 1713. The Seal conditions for testing as, there is seal on transverse direction, seal bar width 5 mm., holding times 5 seconds and pressure 0.2 MPa at temperature 110 °C, 120 °C, 125 °C, 130 °C, 135 °C and 140 °C. The seal strength parameters and testing condition as, testing speed 300 mm/min, 5 specimens, width 15 mm and length 100 mm were tested. To made report an average value of seal strength (N/15mm).

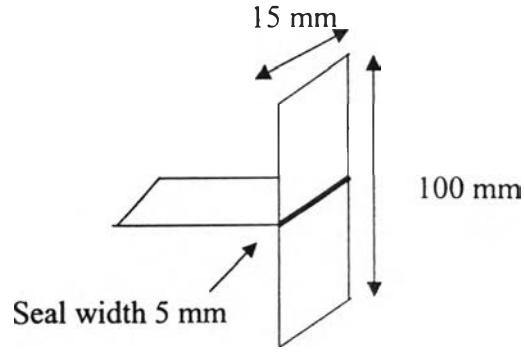

Figure 3.5 Film dimensions of seal strength test

Result of initial seal temperature is measuring by a graph of seal strength (Y) and temperature (X), and to drawing a line parallel axis X cut point with axis Y at 3 Newton. After that drawing a line parallel with axis Y at cut point of line parallel with axis X at 3 Newton and seal strength line in each formula. Line of the graph in each formulation was showed initial seal temperature at load 3 Newton.

3.5.6 Differential scanning calorimetry (DSC)

The non-isothermal melt-crystallization and subsequent melting behavior of the blown film was measured by a Mettler-Toledo DSC822 differential scanning calorimetry. The calibration was carried out with an indium standard (T_m = 156.6°C and ΔH_f = 28.5 J/g) on every other run to ensure accuracy and reliability of the data. To minimize thermal lag between the sample and the DSC furnace, each sample holder was loaded with a single disc-shaped sample cut from the filmed sample. Each sample was used only once and all the runs were carried out under nitrogen atmosphere (60 ml/min flow rate)

The measurements started with heating each sample from 25°C to 160°C at a heating rate of 80°C/min. This procedure was used to set a similar thermal history to all of the samples investigated. To ensure complete melting, each sample was melt-annealed at 160°C for 5 min. The sample was cooled at a cooling rate of 10°C/min to 25°C to observe the non-isothermal melt-crystallization behavior and the subsequent melting behavior was then investigated by reheating the sample to 160°C at a heating rate of 10°C/min. Both non-isothermal melt-crystallization exotherms and subsequent melting endotherms were recorded for further analysis.