CHAPTER III

EXPERIMENTAL

3.1 Materials

1. High-density polyethylene (HDPE) resins (grade GA3750, Thai Petrochemical Industry Public Co., Ltd., Rayong, Thailand).

   MFI loaded by 2.16 and 5 kg at 190 °C = 0.06 and 0.27 g/10 min, respectively), Density 0.951 g/cm^3.
2. Recycled polyethylene resins (Thai Bamroong Import Export Co., Ltd., Bangkok, Thailand):
   2.1 MFI of recycled HDPE (film) at loaded by 2.16 and 5 kg at 190 °C = 0.06 and 0.14 g/10 min, respectively).
   2.2 MFI of recycled HDPE (bottle) at loaded by 2.16 and 5 kg at 190 °C = 2.50 and 13.22 g/10 min, respectively).
   2.3 MFI of recycled LDPE at loaded by 2.16 and 5 kg at 190 °C = 4.30 and 18.48 g/10 min, respectively).
3. Concentrated sulfuric acid (BDH Laboratory Supplies, Poole, England).
4. 90% Phosphoric acid (BDH Laboratory Supplies, Poole, England).
5. Potassium permanganate ($KMnO_4$, Carlo Erba, Milano, Italy).

3.2 Instruments

Major instruments used are listed below.

1. JOY HDPE 45 AW high speed HDPE tubular film extrusion machine (Bangkok, Thailand)
2. WEBER ES 45 single screw extruder (Kronach, Bayern, Germany)
3. THYSSEN HEN FM 10C 5L high speed mixer (Kawata, Japan)
4. TOSHIBA IS 100G injection molding machine (Tokyo, Japan)
5. KEYNESS D 7053 automatic melt flow indexer (Morgantown, PA)
6. LLOYD LR 10K universal testing machine (Hants, England)
7. PERKIN ELMER DSC 7 differential scanning calorimeter (Connecticut, USA)
8. PERKIN ELMER DMA 7 dynamic mechanical analyzer (Connecticut, USA)
9. ROSAND RH 2000 capillary rheometer (England)
10. JEOL JSM 5800LV scanning electron microscope (Japan)
11. Siemens D5000 F8-815 x-ray diffractometer (Karlsruhe, Germany)
12. Sartorius IC 34 weighter (Goettingen, Germany)

3.3 Experimentals

3.3.1 Blend Preparation and Blown Film Extrusion.

All high-density polyethylenes (HDPE), and recycled polyethylene resins were dry mixed according to the compositions shown in Table 3.1 using a high speed mixer. All the blends were prepared by melt mixing using a 4.5-cm diameter

single screw extruder at 190-200 °C. The L/D ratio was 20:1. The extrusion rate was 12 kg/hr. Extrudate strands were water-quenched and pelletized.

All samples were tumble blended and compounded on the single screw extruder prior to film extrusion. No additional antioxidant, or slip, or antiblock agents was added. The blown film processing conditions were:

- 50-mm diameter die and 45-mm screw die
- 28:1 screw length to diameter ratio (L/D)
- air ring with internal bubble cooling
- 4 blow-up ratio (BUR), 14-inch (35.5 cm) lay flat tube
- 18 inch (45.7 cm) frost line height (FLH)
- target film thickness = 20±2 μm
- extruder temperature profile = 190/200/200 °C
- die temperature = 210 °C
- constant screw speed = 550-575 rpm
- constant take-up speed = 250 rpm

In Table 3.1, 27 blend compositions were formulated to produce recycled polyethylene films. Mechanical properties of these films were measured. Statistical analyses of these films were carried out using a personal computer program STATGRAPHICS with a one-way analysis of variance at the 95% confidence level to observe effects of significant parameter of the blend compositions. The recycled LDPE blends were chosen base on the statistical results to study the effect of the recycled resin on mechanical properties, rheological properties, tensile properties, and thermal behavior of the blended polyethylene films.

Table 3.1 Compositions of the Blended Recycled Polyethylenes.

Resins / Sample code	Virgin HDPE (%)	Recycled HDPE film (%)	Recycled HDPE Bottle (%)	Recycled LDPE (%)
F1	50	10	10	30
F2	40	20	10	30
F3	40	10	20	30
F4	30	30	10	30
F5	30	20	20	30
F6	30	10	30	30
F7	20	30	20	30
F8	20	20	30	30
F9	10	30	30	30
F10	60	10	10	20
F11	50	20	10	20
F12	50	10	20	20
F13	40	30	10	20
F14	40	20	20	20
F15	40	10	30	20
F16	30	30	20	20
F17	30	20	30	20
F18	20	30	30	20
F19	70	10	10	10
F20	60	20	10	10
F21	60	10	20	10
F22	50	30	10	10
F23	50	20	20	10
F24	50	10	30	10
F25	40	30	20	10
F26	40	20	30	10
F27	30	30	30	10

3.4 Mechanical Properties of Blended Recycled Polyethylenes Films.

3.4.1 Determination of Tensile Properties.

The tensile test was carried out using a LLOYD LR 10K universal tensile machine according to ASTM D 882 method A in standard laboratory conditions. The test film dimension is shown in Figure 3.1

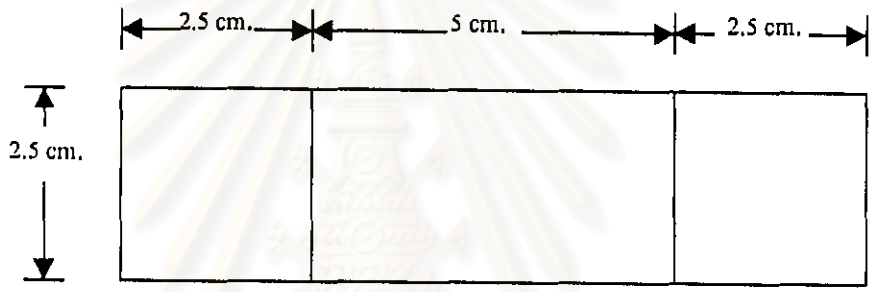

Figure 3.1 Film dimension for tensile property testing.

The tensile parameters and testing conditions were listed below:

Load cell	: 500 N
Load axis	: 50 N
Extension axis	: 500 mm.
Speed testing	: 125 mm/min
Gage length	: 5 cm. or 50 mm.
Temperature	: 23 ± 2 °C
Relative humidity	: 50 ± 5 %
Sample testing	: 5 pieces

3.4.2 Dynamic Mechanical Analysis (DMA).

DMA is a method for quantitative analysis of mechanical properties of a material expressed as function of temperature, time, and frequency under a sinusoidally oscillating load.

The compositions of each blend given in Table 3.1 were injected in a suitable form using an injection molding machine with a temperature range of 190-210 °C for the film mechanical property test.

In this study, the conditions of DMA were given below:

Scanning rate : 5 °C/min

Temperature : -140-60 °C

Frequency : 1 Hz

Specimen size : $3 \times 10 \times 1$ mm^3

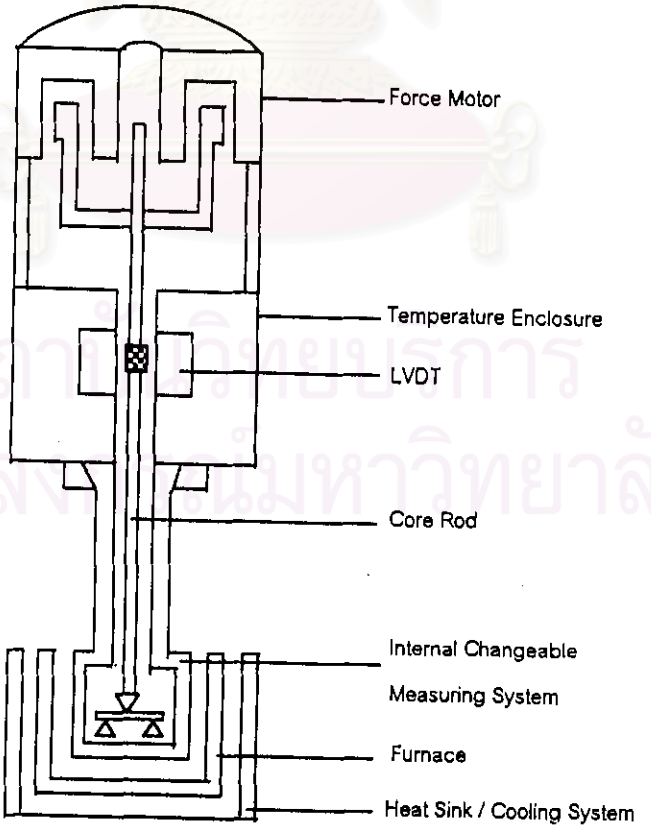

Figure 3.2 Cross section of PERKIN ELMER DMA 7.

3.5 Rheological properties of Blended Recycled Polyethylenes Resins.

3.5.1 Determination of Melt Flow Index (MFI).

MFI measurement was carried out according to ASTM method 1238, utilizing a KEYNESS 7053 melt flow indexer, at 190 °C, with a 2.16- and 5-kg dead load.

3.5.2 Determination of Rheological Property.

The melt viscosity measurement of the blended resins shown in Table 3.1 was carried out on a ROSAND RH 2000 capillary rheometer at 190 °C (Figure 3.2). This study determined the shear viscosity and shear stress as a function of melt temperature (at 190 °C) at a spectrum of shear rate (10-300,000 /s).

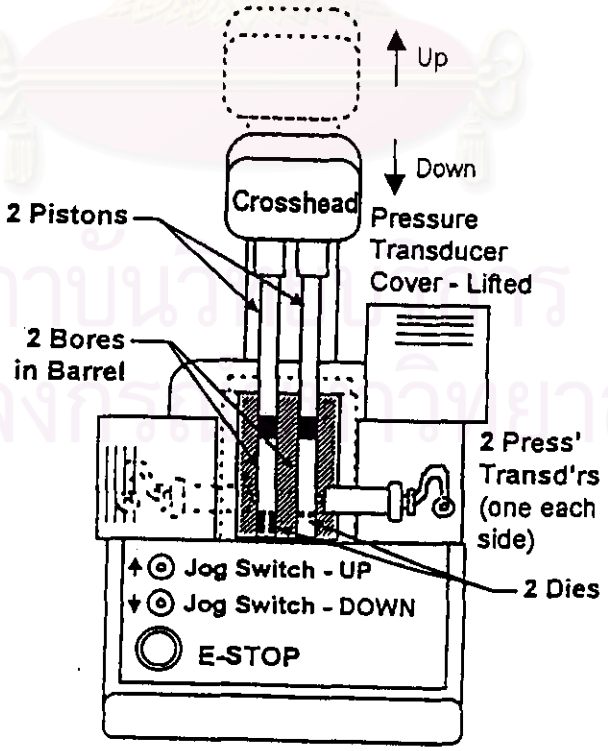

Figure 3.3 ROSAND RH 2000 capillary rheometor.

In this study, the conditions of ROSAND RH 2000 capillary rheometor were given below:

Piston temperature (°C): 190

Barrel temperature (°C): 190

Die temperature (°C) : 190

Shear rate (/s) : 50, 100, 300, 500, 700, 1000, 2000, 3000, 4000, 5000, 6000, 7000, 10000

Die diameter (mm) : 1

Preheating time (min) : 6

3.6 Thermal Behavior of Blended Recycled Polyethylenes Resins.

3.6.1 Determination of Melting Temperature (T_m) Using Differential Scanning Calorimetric (DSC) Technique.

Blended samples of 4-6 mg were initially heated in a nitrogen atmosphere from 50 to 150 °C at a heating rate of 20 °C/min, following by 20 °C/min cooling. The samples were then reheated from 50 to 150 °C at a heating rate of 20 °C/min.

3.6.2 Determination of Glass Transition Temperature (T_g) Using Dynamic Mechanical Analysis (DMA) Technique.

Blended samples of 3 × 10 × 1 mm^3 were analysed by PERKIN ELMER dynamic mechanical analyzer (DMA 7).

In this study, the conditions of determination of T_g were given below:

Scanning rate : 5 °C/min

Temperature : -140-60 °C

Frequency : 1 Hz

3.7 Morphology of Blended Recycled Polyethylenes Films.

3.7.1 Scanning Electron Microscopy (SEM) Measurement.

The film samples were etched with a 0.7-wt% solution of $KMnO_4$ in a 2:1 (v/v) mixture of concentrated sulfuric acid and 90% phosphoric acid for 1 hr at room temperature. The etched samples were washed with a 2:7 (v/v) mixture of concentrated sulfuric acid and water, then washed with demineraled water again, and finally dried in a conditioning room (temperature 23±2 °C, relative humidity 60 ± 5%). They were mounted on scanning electron microscope (SEM) stubs with double-sided adhesive tape and sputter coated with gold. The morphology of the blend film was examined by the SEM at 15-kV accelerating voltage.

3.8 The Degree of Crystallinity of Blended Recycled Polyethylenes Films.

3.8.1 X-Ray Diffraction Measurement.

X-ray diffraction patterns were obtained using the X-ray diffractometer with flat-film geometry. Nickel-filtered CuK_α radition (λ = 0.154187 nm) operated at 50 kV and 30 mA was employed. The patterns were recorded with the incident x-ray beam positioned at the angle 2θ = 10-80 degrees to the film surface.