

CHAPTER III EXPERIMENTAL

3.1. Materials

Three different types of low-density polyethylene resins labeled A, B, C. Blown films produced from the three resins A, B, and C under typical commercial processing conditions.

Both the resin and film samples were provided by Thantawan Industry Public Co. Ltd.

3.2. Equipment and Methods

3.2.1. Resin Characterization

- The density was measured by using a water-isopropanol density gradient column according to ASTM D1505. The accuracy was $\pm 0.001 \text{ g/cm}^3$.
- Melt Flow Index (MI) I_2 was measured according to ASTM D1238 (condition E) at 190°C and 2.16 kg weight load. High Load Melt Index (HLMI) I_{10} was measured according to ASTM D1238 (condition F) at 190°C and 10kg weight load.
- The molecular weights were determined by High Temperature Gel Permeation Chromatography (HTGPC) at approximately 140°C using o-dichlorobenzene

as a solvent and a flow rate of 1 cm³/min GPC runs were carried out on Waters 150C ALC/GPC instrument.

- Fourier Transform Infrared Spectroscopy (FTIR). The measurements were performed using Bruker RFS 100 FT-IR spectrophotometer with a resolution of 4 cm⁻¹. The determination of methyl content in the resins was conducted according to ASTM D2238- Method B. The reference used was high-density polyethylene grade with a density of 0.956 g/cm³ and a MI of 0.03 g/10min. Samples were prepared for FTIR spectroscopy by hot pressing approximately 100 mg of the polymer between polyester sheets in a mold at 150°C. Samples were pressed for 3 min at 20 ton pressure, immediately removed from the press, and air cooled to ambient temperature. Film thickness was measured by Peacock Digital Linear Gauge, model PDN 12N. The sample film thickness was in the range of 0.03mm to 0.15mm, while the reference film thickness was in the range of 0.07mm to 0.22 mm. The spectrum from 1343 to 1400cm⁻¹ was recorded and the absorbance at the center of the methyl band (A₁₃₇₈) was measured.
- Differential Scanning Calorimetry (DSC). The crystallinity, melting point and crystallization point were determined from the thermogram obtained by Perkin-Elmer Differential Scanning Calorimeter. Sample sizes were typically 5-6 mg. Samples were held at a starting temperature of 50°C for 1 min then heated up to 150°C with the rate of 10°C/min. Samples were then held at 150°C for 2 min followed by cooling down to 25°C at 10°C/min. The second scan was recorded in order to eliminate the thermal history of the resin.

- Rheological measurements. The steady shearing properties were determined using an Instron 4303 Capillary Rheometer. Capillary number 614 with 0.0301 inches in diameter and 1.0002 inches in length was used. The test was carried out at 150°C in the range of shear rate from 10 to 1000 s⁻¹. Crosshead velocity (mm/min) was calculated by $v = 15d^3\dot{\gamma}/2D^2$, where d and D are the diameter and length of the capillary respectively.
- Tensile testing was carried out on an Instron Universal Testing Machine according to ASTM D 638. Crosshead speed used was 400 mm/min Six specimens were tested for each sample and the results were averaged to obtain a mean value.
- Extraction The resins were extracted by n-hexane as the solvent. Low molecular weight of LDPE is soluble in hot hexane. 10 grams of resin pellet was immersed in 100 ml n-hexane and extracted for 8 hours at 80°C. The solvent was then decanted and fresh 100 ml n-hexane was added. Extraction was then continued for another 8 hours. After extraction, the solvent was evaporated by slow heating and the extractable materials were obtained.

3.2.2. Blown Film Characterization

- Gloss measurement was carried out on a Haze-Gloss Tester BYK-Gardner according to ASTM D- 2457. Gloss is the amount of light reflected from the surface of a specimen in the mirror direction, i.e. if the incident light angle is 60° to the normal then light reflected from the surface at 60° to the normal is used to determine the gloss of the specimen. The amount of gloss depends on

several factors such as the surface finish, refractive index, and the angles of incidence. In this work, the incident angles were taken at 20° and 60° which are typical for semi-gloss material.

- Tensile properties testing. Tensile strength and elongation at break of blown films were determined in both the MD and TD directions. The tests were conducted on a Lloyd Universal Testing machine according to ASTM D 882. The test specimens were prepared by cutting strips of uniform width and thickness from the film samples. The specimens used were 170 mm long, 20 mm wide and 0.045 to 0.055 mm in thickness. Twenty specimens, ten in MD and ten in TD, were tested for each sample to obtain a mean value. Test conditions are summarized in Table 3.1.

Table 3.1 Tensile testing conditions.

Parameter	Value
Load cell (N)	500
Initial grip separation (mm)	50
Crosshead speed (mm/min)	500
Initial strain rate (mm/mm.min)	10

- Shrinkage testing. Shrinkage of film in MD was tested on a fiber/film shrinkage tester Yasuda No.435 at 135°C. The initial grip separation is 50 mm (original length). Five specimens were tested for each sample and the results were averaged to obtain a mean value. Shrinkage was calculated in percentage.

- Tear resistance testing. The force required to propagate tearing across the film specimens was measured using an Elmendorf 2000 Tear Tester with a “B” pendulum (1600mN full scale load). The test was conducted according to ASTM D1922. Test specimens were cut as shown in Fig. 3.1 to form a constant-radius testing length to compensate for oblique tearing. Ten specimens were tested for each sample in both directions and the results were averaged to obtain a mean value.

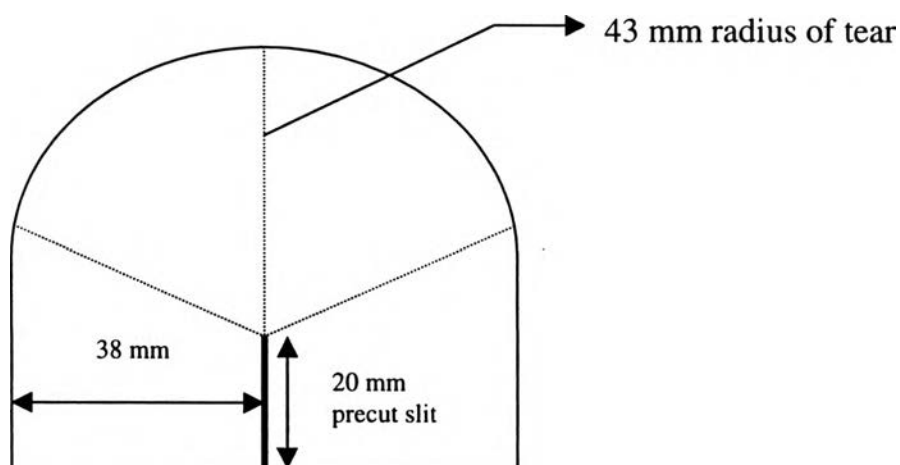


Figure 3.1 Constant- radius test specimen for tear resistance test.

- X-Ray Diffractometry. WAXD used to study the crystallinity of the blown films and the extractable fractions. For the blown film, the films were folded 6 times at different directions to compensate for the effect of orientation. WAXD spectra were obtained from D/Max-2000 series of Rigaku/X-ray Diffractometer system. X-ray of Cu K-alpha at 40kV/30 MA was used as the source. K-beta filter was used for eliminating interference peak and the

divergent slit and scattering slit were set at 1 deg. together with a 0.3 mm receiving slit. The experiment was performed in the range of 5-40 degree with the scan speed of 5 deg./min and 0.02 deg. scan step. Crystalline fractions were calculated from the area under the peak.

- Polarized Optical Microscope was used to study the molecular orientation. Pictures were taken on a Leica Model DMRX, Xe 75 lamp polarizer. The polarizer was set such that the plane of polarization was at 45° to the vertical axis and a prominent axis of the specimen (i.e. MD in this case) was set parallel to the vertical direction.