



## CHAPTER III EXPERIMENTAL

### 3.1 Materials

Ultramid B3 polyamide 6 (density 1.31 g/cm<sup>3</sup>) was obtained from BASF. Low-density polyethylene, LD 1450 J, an injection molding grade polymer (density 0.914 g/cm<sup>3</sup>) was kindly supplied by Thai Polyethylene Co., Ltd., and ionomer, Surlyn<sup>®</sup> 9650 (density 0.95 g/cm<sup>3</sup>), was supplied by DuPont

### 3.2 Experimental Procedures

#### 3.2.1 Blending Process

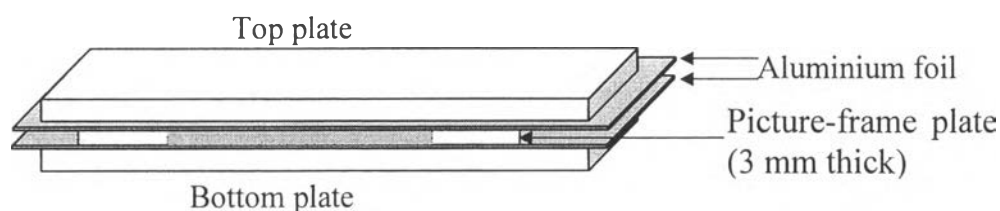
PA6, Surlyn<sup>®</sup> ionomer, and LDPE were kept in an air-circulating oven at 70 °C for 24 hours to remove moisture. Premixes of PA6/LDPE/Surlyn<sup>®</sup>, PA6/Surlyn<sup>®</sup>, and Surlyn<sup>®</sup>/LDPE were prepared at various composition ratios given in Table 3.1. These mixtures were melt-blended in a Collin D-8017 (T-20) twin-screw extruder using a temperature profile of 75/200/215/220/220/230 °C and screw rotation speed 40 rpm. Pure PA6, Surlyn<sup>®</sup>, and LDPE were also passed through the extruder in order to compare all materials having the same thermomechanical history. The extrudates, in the form of strands, were cooled and pelletized. The pellets were then dried in a hot air oven at 60 °C for 24 hours.

**Table 3.1** Blend compositions of PA6/LDPE/Surlyn<sup>®</sup> ternary blends, PA6/Surlyn<sup>®</sup> binary blends, and Surlyn<sup>®</sup>/LDPE binary blends.

Blend type	Composition (wt%)							
PA 6/Surlyn <sup>®</sup>	0/100	20/80	40/60	50/50	60/40	80/20	100/0	
PA6/LDPE/Surlyn <sup>®</sup>	20/80/0.5	40/60/0.5	50/50/0.5	60/40/0.5	80/20/0.5			
	20/80/1.5	40/60/1.5	50/50/1.5	60/40/1.5	80/20/1.5			
	20/80/5.0	40/60/5.0	50/50/5.0	60/40/5.0	80/20/5.0			
Surlyn <sup>®</sup> /LDPE	0/100	20/80	40/60	50/50	60/40	80/20	100/0	

### 3.2.2 Moulding Process

To prepare the samples for characterization, granules of the compound blends were compression moulded in a picture-frame mould (Figure 3.1). Aluminium foil was used to serve as a mould release agent. A 50 tons capacity Wabash V50 compression press was used to prepare the samples. Granules were placed in the mould and preheated to 250 °C by maintaining contact pressure on the material for 3 minutes. Then a force of 10 tons was applied to the mould, at 250 °C, for a further 3 minutes. Finally, the mould was cooled under pressure, to 40 °C before removing the pressed samples. The moulded sheets were dried in a vacuum oven for 24 hours at 60 °C before carrying out characterization tests.



**Figure 3.1** Picture-frame mould used to compress samples of blends for characterization.

### 3.2.3 Thermogravimetric Analysis

Thermogravimetric and derivative thermogravimetric tests were carried out using a Dupont 2950 thermogravimetric analyzer. The percent weight changes were monitored as a function of temperature. A sample weight of  $10 \pm 0.5$  mg was used, and a temperature range between 25-600 °C at three different heating rates (5, 10, 20 °C/min) in air. The kinetic parameters of degradation were determined by plotting fractional conversion as a function of temperature and then using the basic Arrhenius-type expression (equation 3.1) to determine kinetic parameters,

$$d\alpha/dt = f(\alpha) A \exp(-E_a/RT) \quad (3.1)$$

where:

A = pre-exponential factor (the rate of degradation at a temperature at which the sample loses a specific weight)

$E_a$  = activation energy (the energy barrier for the occurrence of degradation)

$T$  = temperature at a specific weight loss

$R$  = gas constant value.

The fraction conversion,  $\alpha$ , was calculated using equation 3.2,

$$\alpha = (w_i - w) / (w_i - w_f) \quad (3.2)$$

where:

$w_i$  = initial weight of sample

$w_f$  = final weight of sample

$w$  = weight at a particular conversion.

#### 3.2.4 Differential Scanning Calorimetric Analysis

A Perkin-Elmer differential scanning calorimetry (DSC-7) was used to analyze the samples. All measurements were performed under nitrogen flow. Temperature calibration of the DSC instrument was carried out by measuring the onset melting temperature and heat of fusion (peak area of endothermic peak) of an indium standard. The sample size was 10-13 mg, and the following procedure was used: (i) first heating run at 80 °C/min, from 30 °C to 250 °C, for deleting the thermal history of the sample; (ii) maintaining the temperature at 250 °C for 5 min; (iii) cooling to room temperature at various cooling rates (5, 10, 20, 40 °C/min); and (iv) second heating run at 10 °C/min, up to 250 °C. The onset of crystallization temperature, crystallization temperature ( $T_c$ ), and melting temperature ( $T_m$ ) were recorded. The crystallinity of the sample was also determined from a knowledge of the ratio of the melting enthalpy for 100% crystallinity of pure components.

The absolute crystallinity was calculated using equation 3.3

$$\chi_c = \frac{\Delta H \times 100\%}{\Delta H_f \times wt. fraction} \quad (3.3)$$

where:

$\chi_c$  = wt% fractional crystallinity

$\Delta H$  = melting enthalpy of the component present in the blends

$\Delta H_f$  = heat of fusion for 100% crystallinity of the pure component  
(190 J/g for PA6, and 282 J/g for LDPE).

### 3.2.5 Scanning Electron Microscopic Analysis

The samples for SEM analysis were prepared by cryogenically fracturing sheet samples at liquid nitrogen temperature followed by immersion in formic acid to remove the PA6 phase or in decalin to remove the Surlyn<sup>®</sup> phase. Cryogenically fractured surfaces of PA6/LDPE/Surlyn<sup>®</sup>, PA6/Surlyn<sup>®</sup>, and Surlyn<sup>®</sup>/LDPE were observed by scanning electron microscopy (SEM), using a JEOL (MP 152001) microscope operating at 25kV.

### 3.2.6 X-ray Diffraction Analysis

Wide angle X-ray diffraction (XRD) patterns of all blends were obtained using a Rigaku Rint 2000 diffractometer equipped with a graphite monochromator and a Cu tube for generating CuK $\alpha$  radiation (1.5046 Å). In order to delete the thermal history of samples and relate the results of XRD and DSC, samples were prepared as follows: (i) heat the sample at 20 °C/min from 25 °C to 250 °C, (ii) hold at 250 °C for 5 min and then cool from 250 °C to 25 °C at 20 °C/min. The prepared samples were put on a sample holder (glass slide) using double-sided adhesive as binder between sample and glass holder, then examined between 5° – 40° 2 $\theta$  at a scanning rate of 2° 2 $\theta$ /min in 0.02° 2 $\theta$  increments. CuK $\alpha$  radiation with  $\lambda = 0.514$  nm was used as the X-ray source, operated at 40 kV and 30 mA. The digital output of the proportional X-ray detector and the goniometric angle measurements were sent to an online microcomputer for storing the data. Percent crystallinity and amorphous contents were calculated using a deconvoluted gaussian program to separate overlapping peaks.