CHAPTER III
EXPERIMENTAL

3.1 PA 6/LDPE/Ionomer Ternary Blends

3.1.1 Materials

Ethylene-methacrylic acid copolymer neutralized with zinc was graciously supplied by DuPont. These particular materials contain an additional comonomer to facilitate toughening with PA and is sold under the trademark Surlyn® 9020. Low-density polyethylene, LD1450J, was an injection molding grade polymer, kindly supplied by Thai Polyethylene Co., Ltd. PA 6 employed in this study was also an injection molding grade, 1013B, supplied by UBE Nylon (Thailand).

3.1.2 Blend Preparation

Table 3.1 Blend ratios of PA 6/LDPE/Surlyn® 9020 ionomer

Compatibilizer	Compatibilizer contribution (wt %)	Concentration of individual components in the blend PA 6/LDPE (wt %)			
No compatibilizer	-	80/20	60/40	40/60	20/80
Surlyn® 9020 grade	0.1	80/20	60/40	40/60	20/80
	0.5	80/20	60/40	40/60	20/80
	1.0	80/20	60/40	40/60	20/80
	2.5	80/20	60/40	40/60	20/80
	5.0	80/20	60/40	40/60	20/80
	10.0	80/20	60/40	40/60	20/80
	15.0	80/20	60/40	40/60	20/80
	35.0	80/20	60/40	40/60	20/80

Prior to use, the blend materials were dried in an oven at 60 °C for 5 hours to remove moisture. Polymer blends having blend ratios shown in Table 3.1 were prepared by melt mixing in a Collin twin screw extruder (T-20) using a screw speed of 35 rpm.

The operating conditions of the twin-screw extruder, which were chosen to minimize the possibility of degradation based on data from thermogravimetric tests, are shown in Table 3.2. The melt was extruded through a single strand die, solidified under water (temperature 35 °C) and pelletized. The pellets were then dried in a hot air oven at 60 °C for 2 days and kept in sealed plastic bags to minimize absorbed moisture, prior to compression molding.

Table 3.2 Operating temperature of each zone of twin-screw extruder barrel for PA 6/LDPE/ionomer ternary blends

Zone 1	Zone 2	Zone 3	Zone 4	Zone 5	Zone 6
75 °C	200 °C	215 °C	220 °C	220 °C	230 °C

3.1.3 Specimen Preparation

Test specimens were prepared using a Wabash V 50 H 50 ton compression press machine. Pellets were placed in a picture frame mold and the mold preheated at 240 °C for 3 minutes in the press without application of pressure. The mold was then compressed under a force of 10 tons for a further 3 minutes after which the mold was cooled to 40 °C under pressure. Test specimens were cut from the molded sheets using a pneumatic die cutter.

3.1.4 Differential Scanning Calorimetry

Samples weighing 10-13 mg were used for DSC tests, which were conducted under constant nitrogen flow using a Perkin-Elmer DSC-7 instrument previously calibrated with indium. Samples were scanned at 10 °C/min from 50 °C to 250 °C. The melting temperatures (T_m) were recorded together with the weight fraction of crystallinity, which was estimated from the normalized melting enthalpies of the blends using Equation 3.1.

$$\chi_c = \frac{\Delta H \times 100\%}{\Delta H_f \times Wt.\ fraction} \qquad \text{(Equation 3.1)}$$

where χ_c is % wt fraction crystallinity

ΔH is melting enthalpy of the components present in the blend

ΔH_f is heat of fusion for 100 % crystallinity of the pure component, 190 J g^{-1} and 282 J g^{-1} for PA 6 and LDPE, respectively (Brandrup, J. and Immergut, E.H. (1989).

Surlyn® ionomer is a copolymer which consist of LDPE as the main component. Therefore, the melting enthalpies recorded from DSC consisted of the combined melting enthalpies of LDPE and Surlyn® ionomer. For calculating the percentage weight fraction crystallinity of LDPE, the amount of LDPE present in the Surlyn® ionomer was taken into account. For example, for the blend consisting of 5 % Surlyn® in 80/20 PA 6/LDPE, the weight fraction of LDPE was calculated as (5+20)/(5+20+80) = 0.238 ≈ 0.24, the weight fraction of PA 6 therefore being 0.76. This method of calculation assumes that the Surlyn® can be considered to be LDPE for the purpose of calculating the degree of crystallinity.

3.1.5 Dynamic Mechanical Analysis

A Solid Analyzer RSA II (Rheometric Scientific) was used to measure the storage and loss moduli as a function of temperature. A film and fiber fixture was used to mount the samples and temperature steps of 3 K intervals were used. All experiments were performed at 10 Hz frequency and 0.1 % strain amplitude using static force tracing dynamic force.

3.1.6 Mechanical Property Measurements

Tensile strength and elongation at break of the blends were carried out on an Instron universal testing machine, Model 4206, at room temperature following the procedure described in ASTM D 1708. A crosshead speed of 1.3 mm min^{-1} and 100 kN load cell was used for all measurements. The value of tensile strength and

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The mold was then compressed under a force of 10 tons for a further 3 minutes after which the mold was cooled to 40 °C under pressure. Test specimens were cut from the molded sheets using a pneumatic die cutter.

3.2.4 Fourier Transform Infrared Spectrometry

FTIR spectra of the blends were obtained from thin polymer films which were prepared by compression molding 20-30 mg between two Teflon® sheets. Pressure was released five times during molding in order to degas the film. Measurements were made in absorbance mode using a Bruker FTIR Spectrometer, model Vector 3.0, with 32 scans at a resolution of 4 cm^{-1}.

3.2.5 Morphological Studies

Blend morphologies were studied using a JEOL 5200-2AE (MP152001) scanning electron microscope. The specimens were fractured in liquid nitrogen and etched using (i) decalin (for removing the ionomer dispersed phase) and (ii) formic acid (for removing the PA 6 dispersed phase). The specimens were then coated with gold. Magnifications of X5000 were used.

3.2.6 Dynamic Mechanical Analysis

A Netzch DMA 242 was used to measure the storage and loss moduli of the blends as a function of temperature using dual cantilever deformation mode. Specimen dimensions were approximately 10 mm x 56 mm x 2.5 mm. All tests were performed at a frequency of 50 Hz and 3 K temperature step/min.

3.2.7 Rheological Measurements

PA 6/ionomer blend pellets were compressed into discs 25 mm in diameter and about 1 mm in thickness. An ARES rheometrics Dynamic Analyzer RDA-II with parallel plate geometry was used to measure the dynamic viscoelastic properties of the blend specimens at a temperature of 250 °C. The distance between the two plates (gap) was 0.5 mm. The following procedure was used (i) for allowing rearrangement of copolymer molecules, disc sample was pre-sheared at frequency

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3.2.10 Mechanical Property Measurements

3.2.10.1 Tensile property measurements

Tensile property measurements were carried out on an Instron universal testing machine, Model 4206, at room temperature following the procedure described in ASTM D 638. A crosshead speed of 50 mm min^{-1} and 100 kN load cell were used for all measurements. The value of tensile property was determined from an average of five specimens for each blend ratio.

3.2.10.2 Izod impact strength measurements

Izod impact strength measurements were carried out on a Zwick impact tester following the procedure described in ASTM D 256. Specimens with a notch radius of 0.025 cm were tested using a 2.7 joule pendulum. The value of izod impact strength was determined from an average of ten specimens for each blend ratio.

3.2.10.3 Hardness Measurements

A Shore D durometer was used to measure the hardness of the PA 6/ionomer blends. The tests were carried out according to ASTM D 2240 test procedure. The value of hardness was determined from an average of ten specimens for each blend ratio.

3.2.11 Gloss

The surface gloss of the blends was measured using a BYK Gardner Gloss-Haze reflectometer at 60 ° and 85 ° reflectance angles at room temperature according to ASTM D 523. The value of gloss was determined from an average of ten specimens for each blend ratio.