CHAPTER III
EXPERIMENTAL

3.1 Materials

The materials employed in this study were used as received:

3.1.1 Ionomer

Surlyn® ionomer used as a compatibilizer for the study was a zinc grade 9020 supplied by Du Pont. The properties of Surlyn® 9020, ionomer are given in Table 3.1

Table 3.1 Mechanical and thermal properties of Surlyn® (9020) ionomer*.

Analysis item	Units	Test result
Melt index 2160 g	(g/10 min)	1.0
Vicat softening point	(°C)	57
Melting point	(°C)	81
Tensile strength (break 73 F)	(MPa)	26.2
Elongation at break (73 F)	(%)	510
Flexural modulus (73 F)	(MPa)	100
Hardness	(Shore D)	55

* Data supplied by Du Pont.

3.1.2 Low-density Polyethylene

Low density polyethylene (LDPE) used for the experiment was an injection molding grade LD1450J from Thai Polyethylene Co., Ltd., supplied by MC Industrial Chemical Ltd. The properties of LDPE, LD 1450J, are given in Table 3.2

Table 3.2 Physical and rheological properties of LDPE (LD 1450J)**.

Analysis item	Units	Test result	Test method
Melt index (G)	(g/10 min)	48	ASTM D 1238
Density (G)	(g/cm^3)	0.916	ASTM D 1505

** Data supplied by Thai Polyethylene Co., Ltd.

3.1.3 Polycarprolactam (Nylon 6)

Nylon 6 employed in the study was also an injection molding grade 1013B, supplied by Ube Nylon (Thailand). The properties of nylon 6, 1013B, are given in Table 3.3

Table 3.3 Chemical and rheological properties of nylon 6 (1013B)***.

Analysis item	Units	Test result
Color	(YI)	-7.8
Moisture content	(wt%)	0.03
Relative viscosity	-	2.43
Extractable content	(wt%)	0.15
Amino group	($\times 10^{-5}$ eq/g)	4.5
Carboxyl group	($\times 10^{-5}$ eq/g)	5.8

*** Data supplied by Ube nylon.

3.2 Experimental Procedures

3.2.1 Blends Preparation

Both uncompatibilized and compatibilized blends were prepared by the same method to study effect of composition of the blend and the effect of ionomer compatibilizer on mechanical property and physical property and morphology of these blends.

3.2.1.1 Nylon 6/LDPE blend (without compatibilizer)

Nylon 6 and LDPE were dried in a hot-air oven at 60 °C for 5 hours prior to use. The polymer blends were prepared in a Collin twin screw extruder (T-20). Nylon 6/LDPE (with the following ratios: 0.0:1.0, 0.2:0.8, 0.4:0.6, 0.6:0.4, 0.8:0.2, 1.0:0.0) were premixed in a tumble mixer before introducing into the extruder. The melt was extruded through a single strand die, solidified with cold water (temperature 35 °C) and pelletized. The pellets obtained were dried and kept in the sealed plastic bags prior to compression molding. This was to minimize moisture regain of the blends from the atmosphere. The processing conditions used for blending are shown in Table 3.4

3.2.1.2 Nylon 6/LDPE blend (with Surlyn® ionomer as a compatibilizer)

The same composition of the blends with 0.1 %, 0.5 %, 1.0 %, 2.5 %, 3.5 %, 5.0 %, 10.0 %, 15.0 %, 25.0 % and 35.0 % of ionomer, were prepared using the same processing conditions adopted by the uncompatibilized blends. The processing conditions used for blending are also shown in Table 3.4

Table 3.4 Processing conditions of Collin twin screw extruder (T-20) used for blending (with/without compatibilizer).

Extrusion Parameter	Units	Operating Value
Extruder Temperature	(°C)	-
Kannel I	(°C)	75
Kannel II	(°C)	200
Kannel III	(°C)	215
Kannel IV	(°C)	220
Kannel V	(°C)	220
Kannel VI	(°C)	230
Screw Speed	(rpm)	-
Kannel IX	(rpm)	35

3.2.2 Compression Molding

Samples for mechanical and physical property tests were prepared from compression molded sheet using a Wabash V 50 H compression press machine. The pellets were placed in a picture frame mold and the mold was preheated at 240 °C for 3 minutes. The mold was then compressed under a force of 10 tons for 3 minutes. The compression molded sheet was then cooled to 40 °C at cooling rate of 20 °C/min. The test specimens for each test were cut from the molded sheets.

3.2.3 FT-IR Analysis

FT-IR spectra of the specimens were obtained from film samples prepared using Wabash V 50 H compression press machine, employing the

same processing temperature adopted for compression molding of polymer blends.

3.2.4 Testing

Various tests were used to characterize and determine properties of the prepared blends. These tests are outlined below:

3.2.4.1 Thermal Analysis

3.2.4.1.1 Thermal Gravimetric Analysis (TGA)

Thermal gravimetric analysis (TGA) was used to determined the degradation temperature of nylon 6, LDPE and Surlyn® ionomer to established the optimum blending conditions before blending was carried out in the Collin twin screw extruder (T-20).

3.2.4.1.2 Differential Scanning Calorimeter (DSC)

Netzsch DSC 200 was used to measure the melting point and the fractional crystallinity of nylon 6, LDPE and Surlyn® ionomer to establish the optimum blending conditions before blending was carried out in the Collin twin screw extruder (T-20).

3.2.4.2 Spectrophotometer Analysis

FT-IR spectra of the blends were obtained from film samples. These films samples were prepared using a compression molding machine. Fourier Transform Infrared Spectrophotometer (FT-IR) was used to probe the specific interpolymer interaction between Surlyn® ionomer and the blends.

3.2.4.3 Mechanical and Physical Properties Testing Lab

Tensile properties, impact property and hardness of the blends were determined from the compressed specimens following the test conditions suggested by ASTM.

3.2.4.3.1 Tensile Properties Testing

An Instron Universal testing machine was used to measure the tensile strength and tensile modulus of the blends. The test was conducted according to ASTM D 638-91 test procedure. The results were obtained from the average of ten specimens.

3.2.4.3.2 Impact Property Testing

Izod impact strength was measured using a Zwick Impact tester according to ASTM D 256-92 test procedure method. The results were collected from the average of ten specimens.

3.2.4.3.3 Hardness Testing

A durometer was used to measure hardness of the blends. The test was conducted according to type D, ASTM D 22 40-91 test procedure. The results were obtained from the average of ten specimens.

3.2.4.4 Microstructure Characterization

Scanning electron microscope (SEM), JEOL 5200-2AE (MP152001) was used in microstructure characterization. Fracture surface of the specimens were etched using decalin and formic acid to remove the dispersed phase. The specimens were coated with gold to facilitate the decipating of charges. In studying morphology, the secondary electrons are detected to generate signal and create the secondary image (SEI) with a magnification 2000 and 3500 times were used.