

CHAPTER III EXPERIMENTAL

3.1 Materials

The materials employed in this study were used as received:

3.1.1 Polyamide6 (PA6)

PA6 employed in this study was an injection molding grade Ultramid B3, supplied by BASF (Thailand) Co.,Ltd. The properties of Ultramid B3 are given in Table 3.1

Table 3.1 Physical properties of PA6 (Ultramid B3)*

Properties	Units	Test result	Test Method
Mn	g/mol	18000	-
Density	g/cm ³	1.13	ASTM D 1505
Melting point	°C	220	ASTM D 2117

*Data supplied by BASF (Thailand) Co., Ltd.

3.1.2 Low-density Polyethylene (LDPE)

Low-density polyethylene (LDPE) used for this work was an injection molding grade LD 1450J, supplied by Thai Polyethylene Co., Ltd. The physical properties of this material are given in Table 3.2

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

Properties	Units	Test result	Test Method
Melt Flow Rate	g/10 min	50	ASTM D 1238
Density	g/cm ³	0.914	ASTM D 1505
Melting point	°C	103	ASTM D 2117

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

3.1.3 Sodium-Neutralized Poly(ethylene-co-methacrylic acid) Ionomer (Na-EMAA)

Sodium-neutralized poly(ethylene-co-methacrylic acid) ionomer used as a compatibilizer for this work was a sodium grade 8527 supplied by DuPont. Surlyn[®] 8527 or Na-EMAA 8527 is an advanced ethylene/methacrylic acid (E/MAA) copolymer, in which the MAA acid groups have been neutralized with sodium ions. Typical performance properties are shown in Table 3.3

Table 3.3 Typical performance properties of Na-EMAA (Surlyn[®] 8527)***

Properties	Units	Test result	Test Method
Melt Flow Rate	g/10 min	1.3	ASTM D 1238
Density	g/cm ³	0.94	ASTM D 1505
Melting point	°C	93	ASTM D 2117

***Data supplied by DuPont (USA).

3.2 Experimental

3.2.1 Blend Preparation

Materials, including polyamide6, low density polyethylene and sodium-neutralized ethylene methacrylic acid (Surlyn[®]) were dried in a hot air oven at 60^o C for 24 h. before use, to remove any absorbed moisture. The polymer blends were prepared in different composition in a model T-20 co-rotating twin screw extruder (Dr. Collin) using a screw speed of 40 rpm and the temperature profile used are shown in Table 3.4. The compositions of the blends are given in the table 3.5. The blends were extruded through a single stand die and cooled at ambient conditions.

Table 3.4 Temperature profile of twin screw extruder

Extruder Zone	1	2	3	4	5	6
Temperature (°C)	75	200	215	220	230	230

Table 3.5 Blend compositions

Blend systems	Concentration (wt%)		
	Ny6	LDPE	Surlyn®
LDPE/Surlyn®	-	80	20
	-	60	40
	-	50	50
	-	40	60
	-	20	80
PA6/Surlyn®	80	-	20
	60	-	40
	50	-	50
	40	-	60
	20	-	80
PA6/LDPE/Surlyn®	80	20	0, 0.5, 1.5, 5.0
	60	40	0, 0.5, 1.5, 5.0
	50	50	0, 0.5, 1.5, 5.0
	40	60	0, 0.5, 1.5, 5.0
	20	80	0, 0.5, 1.5, 5.0

3.2.2 Specimen Preparation

Samples were prepared from compression-molded sheet with a V50 H compression press (Wabash). The obtained pellets were placed in a picture frame mold, and the mold was preheated at 240°C for 3 min between the plates without any applied pressure, allowing for complete melting. After that the mold was compressed under a force of 10 tons with a residence time of 5 min and this specimen was cooled at 40°C under pressure. Test specimens were cut from the molded sheets using a pneumatic die cutter.

3.2.3 Scanning Electron Microscopic Analysis

The fracture micrographs as well as the dispersed structure of the fractured samples were studied using a scanning electron microscope, JEOL (MP 152001), operated at 15-25 kV. The samples fractured under liquid nitrogen and were also subjected to selective extraction of the LDPE and Na-EMAA ionomers phases by immersing in hot decalin to remove LDPE or in formic acid to remove the Ny6. The specimens were then coated with gold, under vacuum, to make them electrically conductive. The number average diameter (d_n) was calculated using equation (1),

$$d_n = \Sigma(n_i d_i) / (n_i) \quad (1)$$

where; n_i is the number of droplet and d_i is the diameter of the i th droplet.

3.2.4 Dynamic Mechanical Analysis

Dynamic mechanical of these blends were studied using a Solid Analyzer RSA II (Rheometric scientific). The storage modulus (G') and loss modulus (G'') were measured as a function of temperature. The film and fiber fixture were used to mount the samples and temperature step of 4 K intervals. All experiments were performed at 10 Hz frequency and 0.05 % strain amplitude using force tracing dynamic force.

3.2.5 Mechanical Properties Measurement

3.2.5.1 Tensile Properties Measurements

Tensile test was performed with an Instron Universal testing machine, Model 4206, at room temperature following the procedure describe in ASTM D 1708. A crosshead speed of 1.3 mm. min^{-1} and 100 KN load cell were used for all measurements. The value of tensile strength and elongation at break were determined from the average of five specimens for each blend ratio tested.

3.2.5.2 Izod Impact Strength Measurement

Impact tests were preformed at room temperature with 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 results will be obtained from the average of at least 5 tests for each blend ratio.

3.2.5.3 Hardness Measurements

A shore D durometer was used to measure the hardness of all blends. The tests were carried out according to ASTM D 2240 test procedure. The value of the hardness was determined form an average of ten specimens for each blend ratio.

3.2.6 The Percentage Water Absorption Measurement

The percentage water absorption measurement was conducted per ASTM D 570. All the specimens used for the study had dimensions of about $5.4 \times 1.8 \times 0.3 \text{ cm}^3$. The test samples were dried at 50°C for 24 h prior to measurement. The samples were first cooled, weighed and then placed in a container of boiling distilled water, supported on edge and entirely immersed in water. After 2 h the specimens were removed from water and cooled in distilled water maintained at room temperature. After 15 min the specimens were removed from water, wiped with dry cloth and weighed immediately. The percentage water absorption was determined as follows:

Percentage water absorption =

$$\frac{\text{weight of wet sample} - \text{conditioned weight}}{\text{conditioned weight}} \times 100$$