CHAPTER III
EXPERIMENTAL

3.1 Materials

Ultramid B3 Nylon 6 (density 1.31 g/cm^3) was supplied by BASF (Thailand) Co.,Ltd. Low-Density Polyethylene, LD 1450J, was an injection molding grade polymer (density 0.914 g/cm^3) graciously supplied by Thai Polyethylene Co.,Ltd. Sodium-neutralized poly(ethylene-co-methacrylic acid) ionomers marketed under the trademark Na-EMAA® 8527, which contain 10% acid content, and 54% neutralization, (density 0.94 g/cm^3) was supplied by DuPont (USA).

3.2 Experiment Procedures

3.2.1 Blends Preparation

Prior to blending, all the blend compositions were external mixed using a tumble mixer for 10 minutes followed by drying them under vacuum at 60°C for 24 hours. The materials were blended in a Collin D-8017 T-20 twin screw extruder using a screw speed of 40 rpm. The blends were extruded through a single strand die, the extrudates were cooled in a water bath, then dried at ambient temperature and pelletized. The compositions of the blends are given in Table 3.2

Table 3.1 Temperature profile of twin screw extruder.

Extruder Zone	1	2	3	4	5	6
Temperature (°C)	90	200	215	220	220	230

Table 3.2 Blend compositions.

Blend systems	Concentration (wt%)		
	Ny6	LDPE	Na-EMAA
LDPE/Na-EMAA	-	80	20
	-	60	40
	-	50	50
	-	40	60
	-	20	80
PA6/Na-EMAA	80	-	20
	60	-	40
	50	-	50
	40	-	60
	20	-	80
PA6/LDPE/ Na-EMAA	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 <u>Specimen Preparation</u>

Test specimens were obtained using a Wabash compression press machine. The pellets were placed in a picture frame mold and preheated at 240°C for 3 minutes between the plates without any applied pressure to allow for complete melting. After this period, a pressure of 10 tons was applied at the same temperature for 3 minutes. The sample was then slowly cooled to 40°C under pressure. Test specimens were cut from the molded sheets using a pneumatic die cutter.

3.2.3 Thermogravimetric Analysis

A DuPont TGA 2950 thermogravimetric analyzer was used to collect thermogravimetric data. 10 ± 0.5 mg samples were heated 30-600°C at three different heating rates of 10, 20, 40°C/min in a platinum pan under air. The thermal stability of the blends were evaluated by plotting the fractional conversion as a function of temperature and the values of the kinetic parameters associated to the degradation process were calculated by the Flynn and Wall method, which is based on the Arrhenius equation (Equation 3.1)

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

where;

A	=	pre-exponential factor
Ea	=	the activation energy
T	=	the temperature at a specific weight loss
R	=	the gas constant value

The fraction conversion, α, was calculated using equation 3.2

$$\alpha \quad = \quad (W_i\text{-}W)/(W_i\text{-}W_f) \qquad\qquad (3.2)$$

where;

W_i	=	the initial weight of sample
W_f	=	the final weight of sample
W	=	the weight at a particular conversion

3.2.4 Differential Scanning Calorimetric Analysis

Thermal analysis was carried out on a differential scanning calorimeter, Perkin-Elmer DSC 7. All the scans were made under nitrogen atmosphere to minimize oxidative degradation. The temperature calibration of the DSC was obtained by measuring the melting temperature of indium as a standard. 10 mg samples were encapsulated in an aluminum pan, heated from 25°C to 250°C at a heating rate of 80°C/min, held for 5 minutes at this temperature to remove their thermal history, followed by cooling to 30°C at 10°C/min. 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

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

where;

χ_c = the % wt fractional crystallinity

ΔH = the melting enthalpy of the component present in the blends

ΔH_f = the heat of fusion for the 100% crystallinity of the pure component

(190 J/g for Ny6, and 282 J/g for LDPE)

3.2.5 X-ray Diffraction Analysis

Wide angle X-ray diffraction (WAXS) investigations of the neat Ny6, LDPE, and Na-EMAA ionomers as well as their blends were carrier out at room temperature using a Rigaku Rint 2000 diffractometer equipped with a graphite monochromator and a Cu tube for generating CuKα radiation (1.5046Å). The diffraction scans were collected between 2θ values of 5 and 40° using a scan speed of 5 °C/min, CuKα radiation (λ=0.514), at 40 kV and 30mA.

3.2.6 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 for 15 minutes and by 85% formic acid for 1 hour, to remove the Ny6 phase. The specimens were then coated with gold under vacuum to make them electrically conductive. The number average diameter (d_n) was calculated using equation 3.4

$$d_n = \Sigma(n_i d_i)/\Sigma n_i \qquad (3.4)$$

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

3.2.7 Fourier Transform Infrared Spectrometry

For FTIR measurements, samples weighing 10 mg were compression molded by heating to 240°C between two sheets of Teflon, which were covered by two sheets of stainless steel. Infrared spectra were measured using a Bruker FTIR spectrometer, model Vector 3.0, with 32 scans at a resolution of 4 cm^{-1} in the 400 to 4000 cm^{-1} range.