CHAPTER III
EXPERIMENTAL

3.1 Materials

3.1.1 Polyamide 6 (Nylon6)

Nylon6 used for the study was an injection molding grade 1013B supplied by UBE Nylon (Thailand) Ltd. The properties of Nylon6, 1013B, are given in Table 3.1

Table 3.1 Physical properties of Nylon6 (1013B)*

Properties	Units	Test result
Moisture content	wt%	0.03
Relative viscosity	-	2.43
Extractable content	Wt%	0.15
Amino group	$X10^{-5}$ eq/g	4.5
Carboxyl group	$X10^{-5}$ eq/g	5.8
Melting point	°C	215-225

*Data supplied by UBE Nylon.

3.1.2 High-density Polyethylene (HDPE)

High-density Polyethylene employed in the study was an injection molding grade H5480S supplied by Thai Polyethylene Co., Ltd. The properties of HDPE, H5480S, are given in table 3.2

Table 3.2 Physical properties of HDPE (H5480S)**

Properties	Units	Test result	Test Method
Melt Flow Index	g/10 min	0.80	ASTM D 1238
Density	g/cm^3	0.954	ASTM D 1505
Melting point	°C	131	ASTM D 2117

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

3.1.3 HDPE-g-MAH (Fusabond®)

Polyolefins and copolymer grafted with maleic anhydride, Fusabond® employed in this study was a grade E MB 100D was supplied by DuPont. The properties of Fusabond® E MB 100D are given ion Table 3.3

Table 3.3 Physical properties of Fusabond® E MB 100D***

Properties	Units	Test result	Test Method
Base resin	-	HDPE	-
Density	g/cc	0.96	ASTM D 792
MAH Graft Level	wt%	0.90	FTIR (DuPont)
Melt Flow Rate	g/10 min	2.0	ASTM D 1238, 190°C/2.16kg.
Melting Point	°C (°F)	136 (277)	ASTM D 3418

***data supplied by DuPont (USA)

3.2 Experimental

3.2.1 Blends Preparation

Uncompatibilized and compatibilized blends were prepared by a relatively similar procedure.

Pellets having blend ratios shown in Table 3.4 were mixed in a trumble mixer for 10 min. Followed by drying under vacuum at 60°C for 24 hrs. Materials were blended in a Collin D-8017 T-20 twin screw extruder using a screw speed of 35 rpm. The blends were extruded through a single strand die; the extrudates were cooled in a water bath, dried at ambient temperature and then pelletized. The pellets were then dried and kept in a sealed plastic bags prior to compression molding. This is to minimize moisture absorption. The operating temperature of each zone of twin-screw extruder barrel for blending are shown in Table 3.5.

Table 3.4 Blend ratio of PA 6/ HDPE/HDPE-g-MAH (Fusabond®)

compatibilizer	%compatibilizer (phr)	% ZnO base on Fusabond® (wt%)	Concentration of individual component in the blend PA6/HDPE (wt%)	
HDPE-g-MAH (Fusabond® E MB 100D)	0	0	80/20	20/80
	0.1	0	80/20	20/80
	0.1	50	80/20	20/80
	1.0	0	80/20	20/80
	1.0	50	80/20	20/80
	2.5	0	80/20	20/80
	2.5	50	80/20	20/80
	5.0	0	80/20	20/80
	5.0	50	80/20	20/80
	10.0	0	80/20	20/80
	10.0	50	80/20	20/80

Table 3.5 Operating temperature of each zone of twin-screw extruder barrel for blending

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

3.2.2 Specimen Preparation

Test specimens were prepared using a Wabash V 50 H 50 ton compression molding machine. Pellets were placed in a picture frame mold and the mold was 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.

3.2.3 Phase Morphology

Scanning electron microscope (SEM), JEOL 5200-2AE (MP152001) was used to study phase morphologies of the blends. The specimens were fractured in liquid nitrogen and etched using (i) hot decalin (for HDPE minor phase blends) and (ii) formic acid (for PA6 minor phase blends). The specimens were then coated with gold under vacuum. All SEM studied were characterized using magnification of 1500 times at 15-20 kV. 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)

$$d_n = \sum(n_i d_i)/(n_i) \qquad (1)$$

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

3.2.4 Differential Scanning Calorimetric Analysis

Thermal analysis was carried out using a differential scanning calorimeter, Perkin-Elmer DSC 7. All scans were made under nitrogen atmosphere to minimize oxidative degradation. The temperature calibration of DSC was obtained by measuring the melting temperature of indium. About 10 mg of samples were exposed to the following condition: the specimens, encapsulated in aluminum pans, were heated from 30°C to 250°C at a heating rate of 80°C/min, held for 5 minutes at this temperature to remove any thermal history, followed by cooling to 30°C at 10°C/min and the samples were reheated to 250 °C at 10 °C/min, before cooling back 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 of the blend was calculated using equation (2),

$$\chi_c = \frac{\Delta H \times 100\%}{\Delta H_f \times \text{wt.fraction}} \qquad (2)$$

where; χ_c is the % weight fractional crystallinity, ΔH is the melting enthalpy of the component present in the blends, ΔH_f is the heat of fusion for the 100% crystallinity of the pure component, (190 J/g for PA6, and 293 J/g for HDPE)

3.2.5 Mechanical and Physical Properties Testing

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

An Instron Universal testing machine was used to measure the tensile strength of the blends. The tests were conducted according to ASTM D638-91 test procedure, using a crosshead speed of 50 mm.mim^{-1}. The tensile modulus , stress at break and elongation at break were determined from the stress-strain curves. Izod impact strength was measured using a Zwick Impact tester according to ASTM D 256-92 test procedure method with a 2.7 J pendulum. Shore D hardness tester was used to measure hardness of the blends. The test was conducted according to ASTM D 2240 test procedure.

All the tests were done at room temperature (30°C) and the results were obtained from the average of ten specimens for each blend ratio

3.2.6 X-ray Diffraction Analysis

Wide angle X-ray diffraction (WAXS) investigation of the neat PA6 and HDPE as well as their blends were carrier out at room temperature using a Bruker AXS D8 Discover system with a 2-D wire detector. Samples were measured in symmetric transmission; two different source-detector angles were used and the data was combined by matching the intensities in the overlap region. Within experimental error, this procedure was identical to matching the two spectra by using the known angular correction for the two angles. No angular correction was performed for different sample absorption depending on angle for a given source-detector angle; the transmittance of all samples was fairly high (~80%) and no distortions were apparent in the overlap region. The transmittance was not used to subtract the background spectra from the sample spectra, rather the transmittance was set at a value that gave a flat profile at low angles.

3.2.7 Dynamic Mechanical Analysis

Dynamic mechanical of these blends were studied using a Solid Analyzer RSA II (Rheometric scientific). The storage modulus (E') and loss modulus (E") were measured as a function of temperature. The 3 point bend fixture was used to mount the samples and temperature step of 4 K intervals. All experiments were performed at 10 Hz frequency and 0.025 % strain amplitude using static force tracing dynamic force.