

CHAPTER III EXPERIMENTAL

3.1 Materials

3.1.1 Fusabond[®]

Polyolefins and copolymer grafted with maleic anhydride, Fusabond[®] employed in this study was a grade E MB 100D supplied by Du Pont. The properties of Fusabond[®] E MB 100D are given in Table 3.1

Table 3.1 Typical 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
Melt Point	°C (°F)	136(277)	ASTM D 3418

*Data supplied by DuPont

3.1.2 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 3.2

Table 3.2 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	X 10 ⁻⁵ eq/g	4.5
Carboxyl group	X 10 ⁻⁵ eq/g	5.8
Melting point	°C	215-225

**Data supplied by UBE Nylon.

3.1.3 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 3.3

Table 3.3 Physical properties of HDPE (H5480S) ***

Properties	Units	Test result	Test Method
Melt Flow Rate	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.

3.2 Experimental

3.2.1 Blend Preparation

All the components were dried in an oven for at least 12 hours at 60°C to remove moisture prior to use. The compositions of the blends are shown in Table 3.4 and were melt blended using a Collin twin-screw extruder (T-20) with a screw speed of 35 rpm. Each blend compositions was premixed in a tumble mixer before

introducing into the twin-screw extruder to be extruded through a single strand die, and solidified with cold water (temperature 35°C) and pelletized. The pellets obtained were dried and kept in sealed plastic bags, prior to compression molding. This is to minimize moisture regain of the blends from the atmosphere. The processing conditions used for blending are shown in Table 3.5

Table 3.4 Blend ratio of PA 6/HDPE/Fusabond[®] ternary blend.

Compatibilizer	%Compatibilizer contribution (wt %)	Concentration of individual Components in the blend PA 6/ HDPE (wt %)				
		80/20	60/40	50/50	40/60	20/80
Fusabond [®] E MB 100D	0	80/20	60/40	50/50	40/60	20/80
	0.1	80/20	60/40	50/50	40/60	20/80
	0.5	80/20	60/40	50/50	40/60	20/80
	1.0	80/20	60/40	50/50	40/60	20/80
	2.5	80/20	60/40	50/50	40/60	20/80
	5.0	80/20	60/40	50/50	40/60	20/80
	10.0	80/20	60/40	50/50	40/60	20/80
	15.0	80/20	60/40	50/50	40/60	20/80
	35.0	80/20	60/40	50/50	40/60	20/80

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

Zone 1	Zone 2	Zone 3	Zone 4	Zone 5	Zone 6
750°C	200°C	215°C	220°C	220°C	230°C

3.2.2 Specimen Preparation

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

3.2.3 Differential Scanning Calorimetry

Differential Scanning Calorimetry (DSC) measurements were carried out under nitrogen, on samples of 7-10 mg, using a Perkin-Elmer DSC7 instrument previously calibrated with indium. The analysis was carried out in the following manner:

- (i) Samples were heated from 30 to 250°C at 80°C/min and held at 250°C for 5 minutes.
- (ii) Samples were then cooled from 250 to 30°C at 10°/min
- (iii) After that, samples were immediately heated from 30 to 250°C at 10°C/min

The melting temperatures (T_m) and the crystallization temperatures (T_c) were recorded together with the weight fraction of crystallinity, which was estimated from the normalized melting enthalpy (ΔH) of the blends using Equation 3.1

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

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 for PA6 (Brandrup, J. and Immergut, E.H.(1989)., 290 J/g for HDPE Blow, H.P. *et al* (1996)).

Fusabond[®] is a copolymer, which consist of HDPE as the main component. Therefore, the melting enthalpies recorded from DSC consisted of the combined melting enthalpies of HDPE and Fusabond[®]. To calculating the percentage weight fraction crystallinity of HDPE, the amount of HDPE present in the Fusabond[®] was taken into account. For example, for the blend consisting of 1% Fusabond[®] in 80/20 PA6/HDPE, the weight fraction of HDPE was calculated as $(1+20)/(1+20+80) = 0.208 \cong 0.21$, the weight fraction of PA6 therefore being 0.79. This method of calculation assumes that the Fusabond[®] can be considered to be HDPE for the purpose of calculating the degree of crystallinity.

3.2.4 Fourier Transform Infrared Spectrometry (FTIR)

FTIR spectra of the blends are obtained from film samples. These film samples were prepared using a compression-molding machine. Fourier Transform Infrared Spectrophotometer (FTIR) is used to probe the specific interpolymer interaction between Fusabond[®] and PA6 in the blends. Measurements were made in absorbance mode using a Bruker FTIR Spectrometer, model Vector 3.0, using 32 scans at a resolution of 4 cm^{-1} .

3.2.5 Phase Morphologies

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. Magnifications of 750 and 1500 times were used in these characterizations.

3.2.6 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.

3.2.6.1 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 D638-91 test procedure. The results were obtained from the average of ten specimens per each blend ratio.

3.2.6.2 Impact Property Testing

Izod impact strength was measured using a Zwick Impact tester according to ASTM D256-92 test procedure. The results were collected from the average of ten specimens per each blend ratio.

3.2.6.3 Hardness Testing

Rockwell Hardness Tester was used to measure hardness of the blends. The test was conducted according to ASTM D785 test procedure. The results were obtained from the average of ten specimens per each blend ratio.