

## CHAPTER III EXPERIMENTAL

### 3.1 Materials

#### 3.1.1 Maleic Anhydride Grafted on HDPE (MAH-gHDPE)

High-density polyethylene (HDPE) grafted with maleic anhydride, Fusabond<sup>®</sup> employed in this study was a grade E MB100D supplied by Creative Polymer Co. Ltd. The properties of Fusabond<sup>®</sup> EMB100D are given in Table 3.1

**Table 3.1** Physical properties of MAH-gHDPE (Fusabond<sup>®</sup> E MB100D)\*

Properties	Units	Test result	Test method
Base resin	-	HDPE	-
Density	g/cc	0.96	ASTM D 792
MAH grated content	wt%	0.9	FT-IR
Melt Flow Rate	g/10 min	2.0	ASTM D 1238,190°C/21.6kg

\*Data supplied by Dupont

#### 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 (H548S) \*\*

Properties	Units	Test result	Test method
Melt Flow Rate	g/10 min	0.80	ASTM D 1238
Density	g/cm <sup>3</sup>	0.954	ASTM D 1505
Melting point	°C	131	ASTM D 2117

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

### 3.1.3 Nylon 6 (Polyamide-6)

Nylon 6 used for the study was an injection molding grade 1013B supplied by UBE Nylon (Thailand) Ltd. The properties of Nylon 6, 1013B, are given in Table 3.3

**Table 3.3** Physical properties of Nylon6 (1013B)\*\*\*

Properties	Units	Test result
Moisture content	wt%	0.03
Relative viscosity	-	2.43
Extractable content	wt%	0.15
Amine group	X 10 <sup>-5</sup> eq/g	4.5
Carboxyl group	X 10 <sup>-5</sup> eq/g	5.8
Melting point	°C	215-225

\*\*\*Data supplied by UBE Nylon.

### 3.1.4 Zinc Acetate Dihydrate

Zinc acetate dihydrate AR. Grade was supplied by Ajax Finechem.

## 3.2 Experimental

### 3.2.1 Zinc Neutralization of Maleic Anhydride Grafted on HDPE

The required amount of MAH-gHDPE with zinc acetate dihydrate was dried and premixed in a tumble mixer for 10 min and then melt-neutralized in a twin-screw extruder (Collin D-8017 T-20) at 230°C and screw speed at 35 rpm. Levels of neutralization of MAH studied in this work were 10%, 25%, 50%, 75%, and 100% neutralization, and complete neutralization was assumed. The obtained materials were dried and kept in sealed plastic bags to prevent any contact with moisture. The neutralization reaction was confirmed by Fourier Transform Infrared Spectroscopy (FT-IR). FT-IR spectra of both unneutralized and neutralized MAH-gHDPE were obtained using a Nicolet Nexus 670 FT-IR spectrometer in the wavenumber range of

4000–400  $\text{cm}^{-1}$  with 32 scans at a resolution of 2  $\text{cm}^{-1}$ . A thin film sample for FT-IR was prepared by compression molding.

### 3.2.2 Blends and Sample Preparations

The required amount of HDPE, Nylon 6 and zinc-neutralized MAH-gHDPE were premixed in a tumble mixer for 10 min followed by drying in a vacuum oven at 60°C for 24 h. The materials were then blended in a Collin D-8017 T-20 twin-screw extruder with a screw speed of 35 rpm and temperature 230°C. The levels of compatibilizer used in these studies were 1 and 10 part per hundred of resin (phr). Test specimens were prepared using a Wabash V 50 H 50-ton compression molding machine. Blend samples were placed in a picture frame mold, and the mold was preheated at 250°C for 3 min in the press without applied force. The mold was then compressed under a force of 10 tons for a further 2 min, after which the mold was cooled to 40°C under applied force. Test specimens were cut from the molded sheets using a pneumatic punch machine.

**Table 3.4** Blend composition of Nylon 6/HDPE with zinc-neutralized MAH-gHDPE

No.	Nylon 6/HDPE blend			
	Nylon 6 (%wt)	HDPE (%wt)	Compatibilizer (phr)	Neutralization (%)
1	100	0	0	-
2	0	100	0	-
3	80	20	0	-
4	20	80	0	-
5	80	20	1	0
6	20	80	1	0
7	80	20	1	25
8	20	80	1	25
9	80	20	1	50
10	20	80	1	50

No.	Nylon 6/HDPE blend			
	Nylon 6 (%wt)	HDPE (%wt)	Compatibilizer (phr)	Neutralization (%)
11	80	20	1	75
12	20	80	1	75
13	80	20	1	100
14	20	80	1	100
15	80	20	10	0
16	20	80	10	0
17	80	20	10	25
18	20	80	10	25
19	80	20	10	50
20	20	80	10	50
21	80	20	10	75
22	20	80	10	75
23	80	20	10	100
24	20	80	10	100

**Table 3.5** Twin-screw extrusion conditions for neutralizing and blending

Conditions	Zone 1	Zone 2	Zone 3	Zone 4	Zone 5	Zone 6
Temperature (°C)	80	200	215	215	220	230
Screw speed (rpm)	35					

### 3.2.3 Scanning Electron Microscopy (SEM)

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 xylene (for HDPE minor blends) and (ii) formic acid (for Nylon 6 minor blends). The specimens were then coated with gold under vacuum and all scanning electron micrographs were taken using 15 kV with at

1500x magnification. The number average diameter ( $d_{n_i}$ ) of dispersion phase was evaluated by SEMAfore software, using equation (1):

$$d_{n_i} = \sum(n_i d_i) / n_i \quad (1)$$

where  $n_i$  is the number of droplet and  $d_{n_i}$  is the diameter of the  $i^{th}$  droplet.

#### 3.2.4. Tensile Test

An Instron Universal tester was used to measure tensile properties of the blend samples using compressed specimens. Testing was carried out following the procedure outlined in ASTM D638-91 test procedure using a crosshead speed of 50 mm/min and a gauge length 50 mm.

#### 3.2.5 Differential Scanning Calorimetry

Thermal analysis of blends was carried out on a Perkin-Elmer DSC 7. All scans were made under nitrogen atmosphere ( $N_2$ ) to minimize oxidative degradation. Temperature calibration was obtained by measuring the melting temperature of indium element. 7-10 mg samples were placed in an aluminum pan and sealed. The sealed pans were heated from 30°C to 250°C at a heating rate of 80°C/min, and held at 250°C for 5 minutes to remove any thermal history and then cooled to 30°C at 10°C/min. The samples were then heated from 30°C to 250°C at 10°C/min. Crystallinity for each polymer component of the compatibilized blends was determined from knowledge of the ratio of the melting enthalpy for 100% crystallinity of pure components. The crystallinity of each component in the blend was calculated using Equation (2):

$$\chi_{c,i} = \frac{\Delta H_i}{\Delta H_{f,i} \times W_i} \times 100\% \quad (2)$$

where  $\chi_{c,i}$  is the percent crystallinity for the  $i^{th}$  polymer component (nylon or polyethylene),  $W_i$  is the weight fractional of polymer component in the blend,  $\Delta H_i$  is

the melting enthalpy of the component present in the blends,  $\Delta H_{f,i}$  is the heat of fusion for the 100% crystallinity of the pure polymer component (190 J/g for Nylon 6, and 293 J/g for HDPE). Crystallization and melting temperatures were determined by the temperature corresponding to maximum heat flow.

### 3.2.6 Dynamic Mechanical Analysis (DMA)

Dynamic mechanical analyses (DMA) of the Nylon 6/HDPE 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 were used. All experiments were performed at 1 Hz frequency and 0.025% strain amplitude using static force tracing dynamic force.

### 3.2.7 Capillary Rheometer

A CEAST Rheologic 5000 Twin-bore Capillary Rheometer was used to evaluate melt-rheology of the zinc-neutralized MAH-gHDPE, neat Nylon 6, neat HDPE, and the as-prepared blends. The inner diameter and the length of the barrel used were 9.95 and 300 mm, respectively, with capillary diameter of 1 mm ( $L/D = 20$ ) and all measurements were operated at 240°C. The apparent viscosity of all the blends data were computed by VisualRHEO, a software based on CeastVIEW platform to be used with Rheologic and Smart RHEO instrument for process test evaluations.