CHAPTER III
EXPERIMENTAL

3.1 Materials

Poly(trimethylene terephthalate), Sorona® 3301 NC010 (density 1.32 g/cm^3) was supplied by DuPont (USA). High density polyethylene, InnoPlus® HD2208J (density 0.961 g/ cm^3) and Liner low density, InnoPlus® LL8420A (density 0.924 g/cm^3) were an injection molding grade which supplied by PTT Global Chemical Public Co.,Ltd. (Thailand). Maleic anhydride grafted high-density polyethylene (MAH–g–HDPE), Fusabond® E MB100D (density 0.960 g/cm^3) and ethylene-methacrylic acid neutralized sodium metal (Na-EMAA), Surlyn® 8940 (density 0.95 g/cm^3) were supplied by DuPont (USA).

3.2 Equipment

3.2.1 Twin screw extruder (Collin D8017 T-20)
3.2.2 Compression machine (Lab-Tech compression machine)
3.2.3 Injection molding machine (Battenfeld BA 250 CDC)
3.2.4 Universal testing machine (LLOYD AMETEX®)
3.2.5 Zwick impact tester
3.2.6 Dynamic mechanical analyzer (EPLEXOR® 100N)
3.2.7 Differential scanning calorimeter (DSC Q1000)
3.2.8 Capillary rheometer (CEAST Rhelogic 5000)
3.2.9 Melt flow indexer
3.2.10 Scanning electron microscope (JEOL JSM-S410LV)

3.3 Experiment Procedures

3.3.1 <u>Blend Preparation</u>

Prior to melt mixing, all materials have dried in oven at 60°C for 24 h. Then, the materials with different ratios (Table 3.1) were placed into a tumble mixer to premix for 10 min. Then these materials were fed through a Collin D8017 T-20 twin-screw extruder by using a screw speed of 40 rpm and temperature profile follow Table 3.2. The blends were extruded through the strands die, those extrudates were cooled in a water bath, then dried at ambient temperature and were cut from a pneumatic die cutter.

Table 3.1 Temperature profile of twin screw extruder

Extruder Zone	1	2	3	4	5	6	7	8
Temperature (°C)	120	240	245	245	250	250	250	250

Table 3.2 Blend compositions

	Concentration (%wt)			
	PPT	HDPE	LLDPE	Compatibilizer
PTT	100	-	-	-
HDPE	-	100	-	-
LLDPE	-	-	100	-
PTT/HDPE/MAH-g-HDPE	80	20	-	0, 0.1, 0.5, 1, 5 phr
	60	40	-	0, 0.1, 0.5, 1, 5phr
PTT/HDPE/Na-EMAA	80	20	-	0, 0.1, 0.5, 1, 5phr
	60	40	-	0, 0.1, 0.5, 1, 5phr
PTT/LLDPE/MAH-g-HDPE	80	-	20	0, 0.1, 0.5, 1, 5phr
	60	-	40	0, 0.1, 0.5, 1, 5phr
PTT/LLDPE/Na-EMAA	80	-	20	0, 0.1, 0.5, 1, 5phr
	60	-	40	0, 0.1, 0.5, 1, 5phr

3.3.2 Specimen Preparation

3.3.2.1 *Compression Molding*

DSC specimens were obtained by using a Lab-Tech compression machine. The pellets were placed in an aluminum frame mold and preheated at 250 °C for 10 min between the plates without any applied pressure to allow for complete melting. After this period, lading pressure of 40 kg/cm^2 to mould at the same temperature for 5 min. The sample was cooled naturally for 5 min under same pressure.

3.3.2.2 *Injection Molding*

Tensile, impact, and DMA test specimens were obtained by injection molding machine (Battenfeld BA 250 CDC) with 22 mm of diameter. The temperature profile for forming specimen was 230, 245, 250, and 250 °C respectively except for pure HDPE and pure LLDPE using 160, 165, 170, and 175 °C. The screw speed was 20 rpm, and injection pressure was set as 75 bars.

3.4 Characterization

3.4.1 Tensile Testing

A Universal testing machine was used to measure the tensile strength, Young's modulus, and elongation at break of the blends. The tests were followed according to ASTM D638 test procedure, using a crosshead speed of 50 mm/min. Results were averaged from five specimens per each batch of the blends.

3.4.2 Impact Testing

Izod impact strength was measured using a Zwick impact tester according to ASTM D256 test procedure with a 2.7 J pendulum. Results were averaged from ten specimens per each batch of the blends.

3.4.3 Differential Scanning Calorimetry (DSC)

Thermal analysis was carried out on a differential scanning calorimeter, DSC Q1000. All 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 of samples were encapsulated in an aluminum pan, heated from -85°C to 275°C at a heating rate of 10°C/min, held for 1 min at this temperature to remove their thermal history, followed by cooling to -85°C at 10°C/min, and held for 5 min again. After that, samples reheat to 275°C with heating rate of 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;

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

where; χ_c is the % weight fractional crystallinity, ΔH is the melting enthalpy of the component present in the blends, ΔHf is the heat o fusion for the 100% crystallinity of the pure component, (145 J/g for PTT, and 293 J/g for HDPE and LLDPE) (Piorkowska *et al.*, 2013).

3.4.4 Rheometry

All blends are measured for the shear viscosity by the capillary rheometer (CEAST Rheolgic 5000). The investigation is recorded at temperature 250°C with a temperature tolerance is set at ±0.5°C. The inner diameter of the barrel is 15 mm, while the inner diameter and the length of the die were 1 and 20 mm (i.e. L/D = 20), respectively. Approximately 50 ml pellets were inserted to the bore and pressed well. After preheating 300 seconds, an automatic data collection system is used to analyze the test results.

3.4.5 Melt Flow Index Testing

The weight of the polymer flow for 10 min was measured by a melt flow indexer under a 2.16-kg load at 250°C according to ASTM D1238 test procedure.

3.4.6 Scanning Electron Microscopy (SEM)

Fracture micrographs were studies using a scanning electron microscope (JEOL, JSM-S410LV), operated at 15 kV. The sample fractured under liquid nitrogen. The specimens were then coated with gold to make samples electrically conductive. The number average diameter (d_n) was calculated using

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

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