CHAPTER III
EXPERIMENTAL

3.1 Materials

Ultradur® B4520 Polybutylene Terephthalate(PBT)(density 1.3 g/cm^3) was supplied by BASF Corporation Engineering Plastics Co., Ltd. High density polyethylene, EL-Lene® H6007JU(density 0.964 g/cm^3) was an injection molding grade which supplied by Siam Cement Group. Sodium-neutralized poly(ethylene-co-methacrylic acid) ionomers marketed under the trademark Surlyn® 8940(density 0.95g/cm^3) was supplied by DuPont (USA)

3.2 Equipment

3.2.1 Twin screw extruder (Collin D8017 T-20)

3.2.2 Compression Presses (WABASH®)

3.2.3 Injection mould machine (Battenfeld BA 250 CDC)

3.2.4 Differential Scanning Calorimetry (DSC Q1000)

3.2.5 Universal Testing Machine (LLOYD AMETEK®)

3.2.6 Zwick Impact tester

3.2.7 Dynamic Mechanical Analyzer (EPLEXOR 100N)

3.2.8 Capillary Rheometer (CEAST Rheologic 5000)

3.2.9 Scanning Electron Microscope (Hitachi, S-4800)

3.3 Experiment Procedures

3.3.1 Blends preparation

Prior to melt mixing, all materials have dried in vacuum oven at 60°C for 24 hr. Then, put materials with different ratios into a tumble mixer to premix 10 minutes respectively. 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 the

strands die, those extrudates were cooled in a water bath, then dried at ambient temperature andwere cut from a pneumatic die cutter.

Table 3.1 Temperature profile of twin screw extruder

Extruder Zone	1	2	3	4	5	6
Temperature(C)	200	230	240	245	245	240

Table 3.2 Blend compositions

Blend systems	Concentration (wt%)		
	PBT	HDPE	Na-EMAA
PBT/HDPE/Na-EMAA	100	0	0
	80	20	0, 1, 2.5, 5, 10
	70	30	0, 1, 2.5, 5, 10
	50	50	0, 1, 2.5, 5, 10
	30	70	0, 1, 2.5, 5, 10
	20	80	0, 1, 2.5, 5, 10
	0	100	0
PBT/Na-EMAA	80	-	20
	70	-	30
	50	-	50

3.3.2 Specimen preparation

3.3.2.1 Compression molding

DMAspecimens were obtained using a Wabash compression press machine.The pellets were placed in an iron frame mold and preheated at 250 °C for 5 minutesbetween the plates without any applied pressure to allow for complete melting. After this period, loading pressure of 10 tons to the mould at the same temperature for 5 minutes. The samplewas cooled to 60 °Cby the cooling system at thesame pressure.

DSCspecimens were obtained using a Lab-Techcompression machine.The pellets were placed in a pieceof Teflon frame and preheated at 250 °C for 3minutesbetween the plates without any applied pressure to allow for complete melting.

After this period, loading pressure of 40 kg/cm^{2}to mould at the same temperature for 3minutes. The samplewas cooled naturally to 60 °C under same pressure.

3.3.2.2 Injection molding

Impact and UTM test specimens were obtained by injection moulding (Battenfeld BA 250 CDC). The temperature profile for forming thedumbbell shape was 200, 210, 220 and 220 °C respectively except for pure PBT using 260, 285, 290 and 295°C. The screw speed is 65 rpm, diameteris 22 mm, and injection pressure was set as 80 bar. Cooling time was 30 seconds.

3.4 Characterization

3.4.1 Rheological measurement

All blends were measured for the shear viscosity by the capillary rheometer(CEAST Rheologic5000). The investigation was recorded at temperature 250 °C with a temperature tolerance was set at±0.5 °C. The inner diameter of the barrel used was 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 200 seconds, an automatic data collection system was used to analyze the test results.

3.4.2 Scanning Electron Microscope (SEM)

Twin screw extrudates of HDPE/PBT/ionomer blends were examined for the morphological micrograph by the scanning electron microscope (Hitachi, S-4800). The cryofractured or etched surfaces were put on the holder with an adhesive tape and coated with a thin layer of platinumgold to makesamples electrically conductive. The scanning electron images were investigated by using an acceleration voltage of 10 kV with a magnification in 3 kX. The number average diameter (d_n) was calculated using equation below.

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

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

3.4.3 Mechanical Testing Machine

An Universal tensile machine was used to measure the tensile strength of the blends. The tests were conducted according to ASTM D638-10 test procedure, using a crosshead speed of 50 mm/min. The tensile modulus and tensile strength at break were obtained. Izod impact strength was measured using a Zwick impact tester according to ASTM D256-10 test procedure method with a 2.7 J pendulum.

3.4.4 Dynamic Mechanical Analyzer

DMA measurements were carried out using the EPLEXOR® 100 N, at 1 HZ, and soaking time was 30 seconds. Specimen dimensions were:length = 40 mm,width = 10 mm, thickness = 3 mm. Dynamic mechanical of these blends were studied using a Solid Analyzer RSA II (Rheometric scientific). The storage modulus (E'), loss modulus (E'') and tanδ were measured as a function of temperature. All experiments were performed at 1Hz frequency and 0.025% strain amplitude using static force tracing dynamic force.

3.4.5 Differential Scanning Calorimetric Analysis

Thermal analysis was carried out on a differential scanning calorimeter, DSC Q1000. 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. 30 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 minus 80 °C at 10 °C /min,and hold for 5 minutes again. After that, samples reheat again to 250 °Cat a rate of 10°C /min.