

CHAPTER III

EXPERIMENTAL

3.1 Materials

Polycarbonate (PC), Wonderlite[®] PC110 from Chi Mei Corporation and poly(methyl methacrylate) (PMMA), Maxiglas[®] MG845 from Shanghai Jing Qi Polymer Science Co., Ltd. were kindly supported by PTT Phenol.

Ethylene methacrylic acid (EMAA) copolymer in which methacrylic acid part is neutralized by sodium ions; Surlyn[®] 8940 from DuPont Co., Ltd. was purchased from Innovation group (Thailand) Co., Ltd.

Ethylene methylacrylate copolymer (EMA); Elvaloy[®] 1820 AC from DuPont Co., Ltd., was purchased from Creative polymer Co., Ltd.

Samarium(III) acetylacetonate hydrate from Sigma-Aldrich Co., Ltd. was purchased from S.M. Chemical Supplies Co., Ltd.

Tin(II) chloride dihydrate (stannous chloride) from QREC Chemical Co., Ltd. was purchased from Virotevidthayaphan.

3.2 Equipments

3.2.1 Twin Screw Extruder

Twin screw extruder (Lab Tech) was used in the first to melt blend PC/PMMA alloys with various ratios of PMMA and compatibilizer content. L/D of screw = 40/1, diameter screw = 20 mm.

3.2.2 Injection Molding Machine

The specimens for mechanical testing such as tensile strength (ASTM D638), notched izod impact (ASTM D256), and flexural strength (ASTM D790) were shaped by injection molding machine (Asia-Plastic-90 Injection molding) which belong to Pontex (Thailand) Co., Ltd.

3.2.3 Melt Flow Index (MFI)

Melt Flow Rate (MFR) or Melt Flow Index (MFI) of neat PC, neat PMMA, and PC/PMMA alloys were measured by Dynisco polymer test. The condition of measurement for neat PC, neat PMMA and PC/PMMA alloys were set at 250 °C, 2.16 kg load.

3.2.4 Density Kit

The density of PC/PMMA pellets was measured by Density Kit. The condition of measurement was followed ASTM D792 standard.

5.2.5 Differential Scanning Calorimeter (DSC)

Thermal analysis of PC/PMMA alloys were characterized by using Mettler Toledo DSC 822[®] which was used to observe the change in phase behavior such as glass transition temperature (T_g), melting temperature (T_m) and so on. The condition comprised three steps (heat-cool-heat). First, the temperature was set from 30°C to 300°C at rate 10°C/min to heat the sample for removing thermal history. Second, the temperature was cooled down to 30°C at the same rate. Finally, the samples were subsequently heated to 300°C once more with the same rate for corresponding melting behavior investigations. All of three steps were run under nitrogen atmosphere.

3.2.6 Thermalgravimetric Analysis (TGA)

TGA (Perkin-Elmer Pyris Diamond TG/DTA instrument) was used to observe onset degradation temperature (T_d) and % weight loss of alloys. The samples were analyzed at the temperature in range of 30-900°C with rate 10 °C/min and nitrogen flow of 50 ml/min.

3.2.7 Dynamic Mechanical Analysis (DMA)

DMA can measure stiffness and damping which reported as modulus and tan delta ($\tan \delta$), respectively. In this research, DMA (Gabo Eplexor[®] 100N) was used to determine phase behavior of the blends like T_g . The temperature scan started from 30 to 200°C at frequency of 1 Hz and heating rate 2°C/min under tensile mode.

3.2.8 Instron Universal Testing Machine

The tensile and flexural tests were carried out by using Instron 5569. The tensile specimens were prepared according to the ASTM D638 standard with the crosshead speed of 50 mm/min and load cells 10 kN. For flexural test, the specimens were followed ASTM D790 in method I/procedure B. with support span and crosshead motion speed were 25x80x3.2 mm³, 80 mm, and 13 mm/min, respectively.

3.2.9 Impact Tester

The impact strength was measured by the Zwick impact testing machine with the pendulum load = 21.6 J. The specimens shape and method were followed the ASTM D256 (notched izod type)

3.2.10 Scanning Electron Microscope (SEM)

SEM (Hitachi S-4800) was used to observe the morphology of PC/PMMA alloy. The samples were obtained from the fractured surface of PC/PMMA specimens after impact test immersing in dimethylacetamide to etch PC out for 3 min and coated by platinum under vacuum.

3.3 Methodology

2.3.1 Preparation of PC/PMMA Alloys

PC and PMMA were dried at 60 °C for 12 hours prior to mixing in twin screw extruder.

EMAA, EMA and EMG were used as compatibilizers which were added into PC/PMMA alloy. The content of compatibilizer was varied 0.5, 1, 1.5 phr for EMAA ,1, 5 phr for EMA and 1, 3 phr for EMG

Samarium(III) acetylacetonate hydrate and tin(II) chloride dihydrate were used as transesterification catalysts for PC/PMMA alloy by varying at 0.025, 0.05 and 0.075 phr.

PC, PMMA and compatibilizer or catalyst were mixed in twin screw extruder and the specimens were prepared by the injection molding.

2.3.2 Characterizations and Testing of PC/PMMA Alloys

2.3.2.1 *Physical Properties Testing*

The physical properties (the melt flow rate (MFR) and density) were measured by following ASTM D1238 and ASTM D792 respectively.

2.3.2.2 *Morphology*

The impact-fracture surfaces were etched by dimethylacetamide and coated by platinum under vacuum before observation by SEM.

2.3.2.3 *Miscibility and Thermal Properties Observation*

DSC was used to determine the miscibility and thermal properties. The samples were scanned with the rate of 10 °C/min under nitrogen atmosphere by heat-cool-heat method from 30°C to 300°C. The miscibility of alloys can be explained by glass transition temperature (T_g).

2.3.2.4 *Thermal Stability Analysis*

TGA was used to investigate the degradation temperature (T_d) and % weight loss. The samples were analyzed at temperature between 30-900 °C with the heating rate of 10 °C/min under the nitrogen flow at 50 ml/min

2.3.2.5 *Mechanical Properties Testing*

The tensile strength (ASTM D638) and flexural strength (ASTM D790) were tested by using Instron 5569. The impact test was performed at room temperature using Zwick testing equipment according to ASTM D256. At least five specimens were tested for each report value.

2.3.2.6 *The Pencil Hardness Test*

The pencil hardness test was used to investigate the scratch resistance of the materials according to ASTM D3363. For each test, at least three specimens were used to evaluate the mean value.