CHAPTER IV

EXPERIMENTS

4.1 Materials

4.1.1 Polymer blend

Poly-carbonate (PC) used as one of components of the blends is Makrolon® 2800. It was supplied from Bayer Co.,Ltd. The counterpart of the components of the blends, Acrylonitrile-Butadiene-Styrene graft copolymer (ABS), was Lustran® 440 from Lanxess (Thailand) Co.,Ltd.

4.1.2 Reinforcement fiber

The aramid fiber used in this study was Kevlar-29 plain weave type. The Kevlar properties are shown below.

Kevlar fiber properties	Value
Diameter (μm)	12
Glass transition temperature (°C)	348
Degradation temperature (°C)	536
Char yield at 700 (°C)	44
Tensile Modulus (GPa)	67
Elongation (%)	5.5

4.2 Gel Permeation Chromatography (GPC)

The average molecular weight of the PC, ABS and their blends was determined by gel permeation chromatography. The analysis was performed at 40 °C with a Waters 600 GPC apparatus using three Waters Styragel® HT columns (Styragel® HT 0.5, Styragel® HT 1, and Styragel® HT 4). The detector was Waters 2414 refractive index measurement (RID). The PC/ABS blends were dissolved in tetrahydrofuran (THF) with a concentration of about 0.1 % (w/v) and then 10 ml of the sample was injected into the column. Polystyrene standards of different molecular weights were used to construct a calibration curve of the column.

4.3 Preparation of Polymer Blend

Melt mixing of PC and ABS was carried out by a twin screw extruder (Rheocord 300p of Haake Inc.). The extruder system is composed of feeding unit, extruder, pelletizing unit. Diameter (D) of screw is 16 mm and its length (L) is 400 mm, which L/D is 25. Temperatures of five zones were set 250, 250, 240, 230 and 220°C from die side, respectively. And screw speed was fixed around 90 rpm for all mixing experiments.

PC and ABS were dried at 100 °C for at least 6 hours in an air oven. PC was blended with ABS at different weight ratios, which were 25/75, 40/60, 60/40, 75/25, in plastic bag and then supplied in the extruder. PC/ABS blended were cooled in water bath and then cut by a pelletizer. The samples were coded as 25/75, 40/60, 60/40, 75/25 respectively

4.4 Preparation of PC/ABS Blended Test Specimens

The particles were dried at 100°C for at least 6 hours in an air oven and then compression-molded by using a compression molder at 250°C under the pressure of 250 kg/cm^2. The test specimens consist of

1) Sheet for flexural testing 25 x 60 mm, thickness 3 mm
3) Sheet for puncher impact testing 100 x 100 mm, thickness 3 mm
2) Sheet for rheological properties measurements radius 20 mm, thickness 1.3 mm

Tensile test specimens according to ASTM D638 were processed by an injection machine at 180 °C. The test specimens were dumbbell (dog bone) shape with a uniform thickness.

4.5 Film Blowing

PC/ABS film was produced using the same twin screws extruder. In the process, the extruder was fitted with film blowing die. The film die had a diameter of 35 mm and a die gap of 1 mm. The molten polymer was extruded through the die and then was drawn up by nip rolls without air blowing. The molten polymer was cooled using an air cooling ring. Temperatures of five zones were set at 255, 250, 240, 230 and 220°C from die side, respectively. Screw speed was fixed at 90 rpm. The film's thickness was controlled by nip rolls take-up velocity.

4.6 Composite Processing

Kevlar-reinforced PC/ABS composites were manufactured by film-stacking method. The woven Kevlar fiber and PC/ABS film were cut and dried in an air oven and then the woven fibers and PC/ABS films were stacked alternately with a designed number of layers. The composites were made by hot pressing with compression molder with using the most appropriate condition.

4.7 Flexural Testing

A universal testing machine (model 5567) with a 1 kN static load cell was used to determine flexural properties of PC/ABS matrices and composites by using three-point bending mode. The distance between the support span was 48 mm. The

cross head speed of 5 mm/min was used. Flexural modulus and Flexural strength of each specimen were averaged from 4 to 6 samples.

4.8 High Speed Impact Properties

Radmana ITR-2000 driven dart impact tester was used to determine puncher impact property. The samples were clamped horizontally between two plates with an inner diameter of 7.5 cm. The impact tip was hemispherical with a diameter of 1.76 cm. The impact velocity was fixed at 4.0 m/sec. Load-displacement curves were recorded and total energy was calculated. The total impact energy was defined as the sum of the energy absorbed until the maximum load (initiation energy) and the energy absorbed after the maximum load (propagation energy). The dimension of the test specimens is 10 cm × 10 cm with the thickness of 3 mm. The impact energies were averaged from 3-5 test pieces.

4.9 Rheological Properties Measurements

The melt viscosities of blends taken at a ratio of PC/ABS as 100/0, 75/25, 60/40, 40/60, 25/75, and 0/100 at constant shear rate (1 sec^{-1}) were determined using a parallel plate Haake Rheo Stress 600, Thermo Electron Cooperation. The diameter of the plate is 20 mm and the gap between the plates was fixed at 1 mm.

4.10 Morphological Observation

The relationship between composition and intrinsic morphological characteristics of the blending systems and also the interfacial interaction between Kevlar fiber and polymer matrix was studied employing scanning electron microscope (JEOL-JSM 5800LV) at an acceleration voltage of 15 kV. The delamination surfaces of the composite specimens were gold sputtered (3 nm thickness) and dried in vacuum at room temperature. The obtained micrographs were used to investigate qualitatively the interfacial interaction between the fiber and the PC/ABS matrix.

In order to investigate morphology of PC/ABS blends, the flexural tested specimens were etched in an aqueous NaOH solution (30% w/v) at 150°C for 20 min to remove the PC fraction from their surfaces before obtaining the SEM micrographs.

4.11 Dynamic Mechanical Analysis

DMA was used to characterize the viscoelastic properties of the studied material. The dimension of tested specimens is 55mm x 10mm x 2mm. The DMA tests were carried out at 1 a frequency of 1Hz in the three-point bending mode using NETZSCH DMA242 equipment. Nitrogen was used as a purge gas. The thermograms were obtained in the temperature range of 30 to 180 °C at 15°C/min. The storage modulus (E'), loss modulus (E"), and loss tangent (tanδ) were recorded in the thermograms. The glass transition temperature was determined by the maximum point of loss modulus curve.

4.12 Differential Scanning Calorimeter (DSC)

Thermal characteristics of each specimen were determined by Perkin-Elmer Differential Scanning Calorimeter, Diamond DSC. Approximately 5-9 mg of samples was sealed in aluminum pans and was tested using the temperature ramp rate of 10°C/min from room temperature to 180°C under nitrogen atmosphere. Glass transition temperature of samples was determined as the midpoint temperature of the change in specific heat in the transition region.

4.13 Contact Angle Measurement

The polymer matrices surfaces were characterized by the measurement of water contact angle measurement. The water contact angle is an indicator of the polarity and fiber wettability of the matrices which was measured by a sessile drop method at room temperature using an optical bench-type contact angle goniometer (CAM-PLUS MICCRO). Purified water droplet (2μl) was dropped on the sample surface by a micro-syringe and then the contact angle was measured immediately after

placement on the polymer surface. Each reported value was averaged from 5-10 measurements.

4.14 Solvent Extraction

The composites were extracted with chloroform, which effectively dissolves polymer matrices, to measure the weight ratio between Kevlar fiber and polymer matrix in the composites. 10 g of each composite was placed in glass container and then 150 ml of chloroform was added into the container to submerge the composites for 48 hours. After that, the residual fiber was filtrated and leached with chloroform twice times and dried in an air oven for 24 hours before weighing.

4.15 Density Measurement

The density of PC/ABS blends and their composites were measured by water displacement method according to ASTM D729-(Method A).

The density was calculated using the following equation

$$\rho = \left(\frac{A}{A-B}\right) \times \rho_o$$

where ρ = Density of the specimen (g/cm^3)

A = Weight of the specimen in air (g)

B = Weight of the specimen in liquid (g)

ρ_o = Density of the liquid at the given temperature (g/cm^3)

4.16 Thermo Gravimetric Analysis (TGA)

The degradation temperature (T_d) and char yield of the PC/ABS blends at various compositions and their composites with different matrix composition were studied using a Perkin Elmer Instrument Technology (SII Diamond TGA/DTA) thermo gravimetric analyzer at heating rate of 20°C/min. The temperature was

scanned from room temperature to 900°C under nitrogen atmosphere. The purge nitrogen gas flow rate was 100 ml/min. The measured sample was approximately 8-12 mg. Weight loss of the samples were measured as a function of temperature. The degradation temperature (T_d) were reported at 5% weight loss and the char yield were reported at 900 °C.

4.17 Ballistic Impact Test

The ballistic impact test was preformed to evaluate the most appropriate composition of the matrix used for ballistic composite and ballistic to determine the suitable number of layers of Kevlar composite which passes ballistic impact test level III-A following NIJ standard. The tested composites were approximately 12.5 x 12.5 cm and their thickness varied with the number of layers of Kevlar cloth used.

The first ballistic evaluation was performed using a 9 mm hand gun with a standard gain of a round lead projectile and lead outer coating. The composites used in this experiment consisted of two panel of 10 piles composites.

The second evaluation was performed using a test weapon having impact velocity following NIJ standard of level III-A. The composites used in this experiment have combined thicknesses of 40, 50, and 60 piles, respectively. The descriptions of all composites used in these experiments were listed in Table 4.1 and 4.2.

Table 4.1 Description of composites laminates used for ballistic impact evaluation.

Number of piles	Panel arrangement	Type of matrix
20	10+10 10+10 10+10 10+10 10+10 10+10	ABS 25/75 PC/ABS 40/60 PC/ABS 60/40 PC/ABS 75/25 PC/ABS PC

Table 4.2 Description of composites laminate used for determining the suitable number of layers of composites which exceed level III-A of NIJ standard

Number of piles	Panel arrangement	Type of matrix
40	30+10 40+0	40/60 PC/ABS 40/60 PC/ABS
50	20+10+10+10 30+10+10 40+10 50+0	40/60 PC/ABS 40/60 PC/ABS 40/60 PC/ABS 40/60 PC/ABS
60	30+10+10+10	40/60 PC/ABS