

## CHAPTER IV

### EXPERIMENTS

In this chapter, the materials, equipments, sample preparations and experimental procedure that have been used in this research will be briefly explained.

#### 4.1 Materials

##### 4.1.1 Poly(bisphenol A carbonate), PC

There were three different molecular weights of poly(bisphenol A carbonates), PC, used in this work. They were purchased from Aldrich Chemical Company, Inc. The melt flow index are 7, 10, 12 respectively by method in ASTM D1238. They are noted as PCM7, PCM10, and PCM12. The viscosity average molecular weight of these polymer are 26208, 25223 and 22865 as determined by Cannon Fenske Viscometer. All of these polymers are transparent pellet as received.

The glass transition temperature ( $T_g$ ) of PC is approximately at 150°C and the melting temperature ( $T_m$ ) of PC is approximately at 230 °C [Charrier, 1991]. The average  $T_g$  of PCs used in this experiment are 151.55, 147.91, 148.59 for PCM7, PCM10 and PCM12 respectively as detected by DSC. The density of all polymers are 1.5860 g/cm<sup>3</sup> (20 °C). The structure of poly (bisphenol A carbonates) is shown in Figure 4-1.

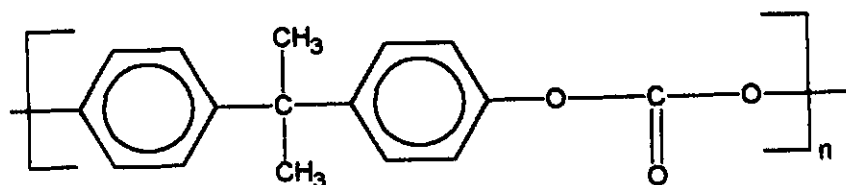


Figure 4-1 The structure of poly(bisphenol A carbonate)

The fourth PC used in this work was supported by Bayer Company. Its structure is the same as Figure 4-1. The  $T_g$  of this polycarbonate is 146.14 °C as detected by DSC. The viscosity average molecular weight is 19323 determined by the same method previously used. This polycarbonate is one of the commercial grades.

#### 4.1.2 Liquid Crystal

Low molar mass thermotropic liquid crystals were used in this work. They were manufactured by Merck under the trade name of "LICRISTAL". Two low molar mass thermotropic liquid crystals in cyclohexyl-biphenylcyclohexanes (CBC) group were chosen. But they are different from each other by chain end. Each one is used to blend with poly(bisphenol A carbonates).

##### 4.1.2.1 CBC33

CBC33 is in the form of a white powder as received. Its properties are shown in Table 4-1 and its structure is shown in Figure 4-2.

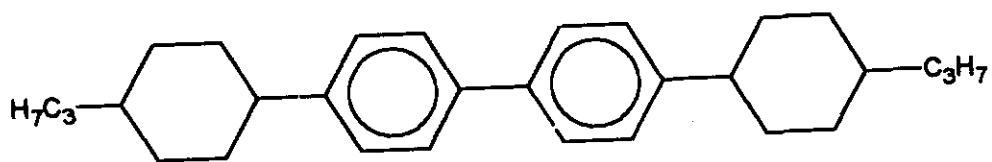


Figure 4-2 The structure of CBC33

Table 4-1 Properties of CBC33

Melting point	158.0 °C
S→N transition temperature	223.0 °C
Clearing point	327.0 °C
Melting enthalpy	13 kJ/mol
Molecular mass	420.67 g/mol

#### 4.1.2.2 CBC53

CBC53 is in the form of a white powder as received. Its structure are shown in Figure 4-3 and its properties are shown in Table 4-2

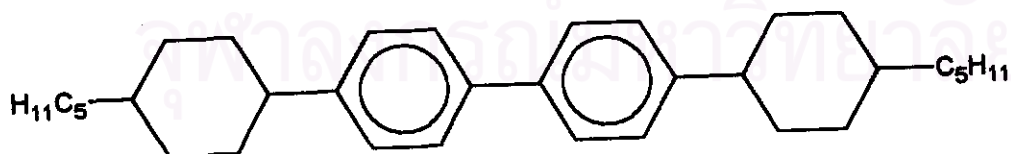


Figure 4-3 The structure of CBC53

Table 4-2 Properties of CBC53

Melting point	64.0 °C
S→N transition temperature	260.0 °C
Clearing point	317.0 °C
Melting enthalpy	8.0 kJ/mol
Molecular mass	430.72 g/mol

Two low molar mass liquid crystals in phenyl-cyclohexylbenzoates group (HP35 and HP5N) were chosen to compare the effects of nematic orientation in shear viscosity. Their properties are shown in Table 4-3.

Table 4-3 Properties of HP35 and HP5N.

Properties	HP35	HP5N
Melting point	80.0 °C	111 °C
S→N transition temperature	87.0°C	-
Clearing point	176.0 °C	226.0 °C
Melting enthalpy	25.1 kJ/mol	21.4 kJ/mol
Molecular mass	392.59 g/mol	375.52 g/mol

## 4.2 Equipments

### 4.2.1 Digital Hot Plate Stirrer

A Cole-Parmer digital hot plate stirrer was used in this study for preparing or mixing the samples. This hot plate stirrer is programmable. All functions are settable from a digital front panel keyboard and display. The plate temperature, the stirrer speed, and time are controllable.

### 4.2.2 Hot Press

A hot press used in this experiment is made in Thailand. The maximum working temperature of this machine is 600 °C and the maximum pressure is 350 kg/cm<sup>2</sup> (5000 psi)

### 4.2.3 Capillary Rheometer

Capillary rheometer is the best method of measuring processability. It directly simulates real processes. In this study, the capillary of Rosand Precision Ltd. model RH-74 was used. The die diameter of 1 mm with capillary of length-to-diameter ratio (L/D) 16/1 was fixed to obtain viscosity data. The temperature was set constant at 290 °C (between nematic region of liquid crystal and above melting temperature of PC). The piston speeds were 60, 80, 100, 120, 160, 200 and 280 mm/min corresponding to the shear rate of 1800, 2400, 3000, 3600, 4800, 6000 and 8400 s<sup>-1</sup> respectively. Capillary with converging entrance angles 180° (flat entrance) was used.

#### 4.2.4 Tensile Testing Machine

The tensile tests were conducted to obtain the stress-strain behavior by stretching pure PC and blends at a constant rate. Tensile testing was performed on LLOYD 2000R. The test methods and procedure set in the ASTM D822. The test methods cover the determination of tensile properties of plastics in form of thin sheet, including film.

Each specimen was mounted on the mechanical testing machine as shown in Figure 4-4. The forces necessary to elongate and to break the specimen were measured by the load cell in the machine. The stress occurred at the yield point was designated as the tensile strength at yield. The stress occurred at break was designated as the tensile strength at break. The mean value of ten measurements was used. In this study, the crosshead speed was set constant at 100 mm/min and a full scale load of 1 kN.

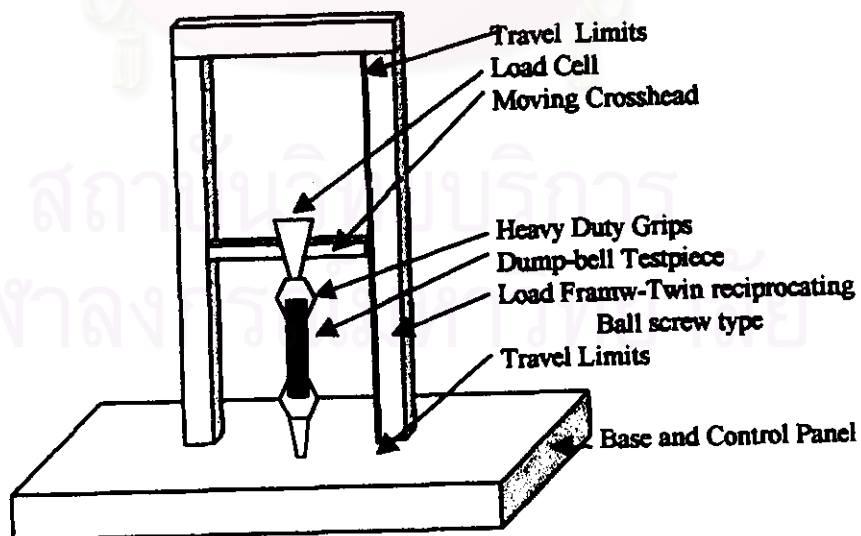


Figure 4-4 Tensile Testing Machine

#### 4.2.5 Differential Scanning Calorimeter (DSC)

The glass transition temperatures ( $T_g$ ) of each sample were determined by using a differential scanning calorimeter (DSC). A Dupont DSC model 2910 was used in this work. Indium ( $T_m=156.4\text{ }^\circ\text{C}$ ) was used as a standard for calibration.

DSC is a thermal analysis technique which measures heat flow into or out of a sample and a reference as a function of time or temperature. The schematic representation of DSC are shown in Figure 4-5.

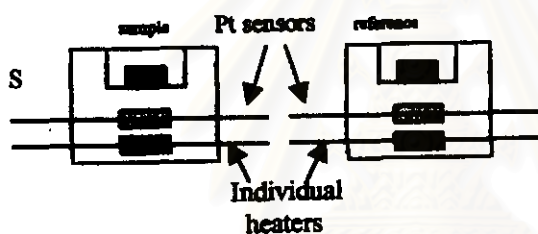


Figure 4-5 Schematic representation of DSC. S and R denote a sample and a reference, respectively [Gedde, 1995].

#### 4.2.6 Cannon Fenske Viscometer

The Cannon Fenske viscometer of Schott Gerate company was used for determining the viscosity average molecular weight. The size number was 25 corresponding to the capillary diameter of 1 mm.



### 4.3 Mixing and Sample Preparation

In this study, melt mixing was performed for sample preparation. Samples were prepared for measuring melt viscosity by using capillary rheometer and for measuring tensile properties by tensile testing machine. All samples were prepared on digital hot plate stirrer.

#### 4.3.1 Mixing for measuring melt viscosity

Before testing the melt viscosity, pure polymers which have different molecular weight were blended with liquid crystals. Blends of 0.25, 0.5 and 1 % by weight were prepared on this study. The polymers were weighed in a beaker about 40 g. And the liquid crystals were weighed corresponding to the desired blend compositions. Each blend was placed on a teflon sheet and put on the digital hot plate stirrer. The hot plate was set at the temperature of 260 °C. Polymer and liquid crystal were mixed together for 10 minutes by mechanical stirring polymer melt using a screw driver in order to have a uniform mixture. After that, the blend was split into 6 pieces.

Each piece was put on the teflon sheets and then compressed for 3 minutes on the hot press which set at the temperature of 290 °C. The polymer was air cooled for 5 minutes before removing from the teflon sheet.



### 4.3.2 Tensile specimen preparation

The tensile specimen in this work was prepared in form of sheet, with the thickness of 0.1-1 mm. Two compositions were studied. The polymers of Bayer were weighed about 1.7 g. And the liquid crystals were weighed corresponding to the desired blend compositions. Each blend was placed on a teflon sheet and put on the digital hot plate stirrer. The hot plate was set at the temperature of 260 °C. Polymer and liquid crystal were blended together by mechanical stirring melt polymer using a screw driver for 10 minutes to obtain a homogeneous blend.

The blend was put on the stainless steel of 0.45 cm in thickness which covered with aluminium foil and then heat for 3 minutes on the hot press at the temperature of 290 °C. The polymer was stirred by screw driver for 3 minutes. It was then heated for about 6 minutes until the polymer nearly melt. Then it was followed with hot compression at 320 kg/in<sup>2</sup> about 2 minutes. The polymer was cooled in water bath at room temperature before removing from the teflon sheet.

For each blend composition, the same procedure was made and also for pure polymer. The obtained polymer sheets which have the thickness about 0.15-0.3 mm were cut into the rectangular shape of 20x90 mm<sup>2</sup>. The specimens tested on the tensile testing machine to obtain the tensile properties.

## 4.4 Method of Determination

### 4.4.1 Melt viscosity

Rheological measurements were carried out using the capillary rheometer. The procedures for measuring melt viscosity at various shear rate were as follows. Firstly, switch on the rheometer testing instrument, the computer, and the temperature control unit. The rheometer temperature was set at 290 °C (for PC) on the control unit. The type of test was programmed using the set up program and entering data. The piston speed profiles were 60, 80, 100, 120, 160, 200 and 280 mm/min to give the shear rate profiles at 1800, 2400, 3000, 3600, 4800, 6000 and 8400 respectively. Make sure the empty rheometer barrel is perfectly clean by passing a thin rod covered with clean rag through the barrel. Also the capillary was checked that it was perfectly clean. The capillary was placed inside the barrel, from the bottom and screwed in the capillary holder.

The polymer was charged on the top of the heated rheometer barrel. Care was needed in order to avoid trapping air inside the barrel. The plunger was jogged down until it contacted with the molten polymer and waited for 5 minutes for the plunger and polymer to reach the barrel temperature. The rheometer was automatically extruded the polymer on the bottom of the barrel. When the test ended, the crosshead stopped and the test results were automatically displayed on the screen.

#### 4.4.2 Tensile properties

The tensile properties of ASTM specimens (50 mm gauge length) were determined with the tensile testing machine operated at 100 mm/min cross head speed. The roller grips were used.

#### 4.4.3 Glass transition temperatures

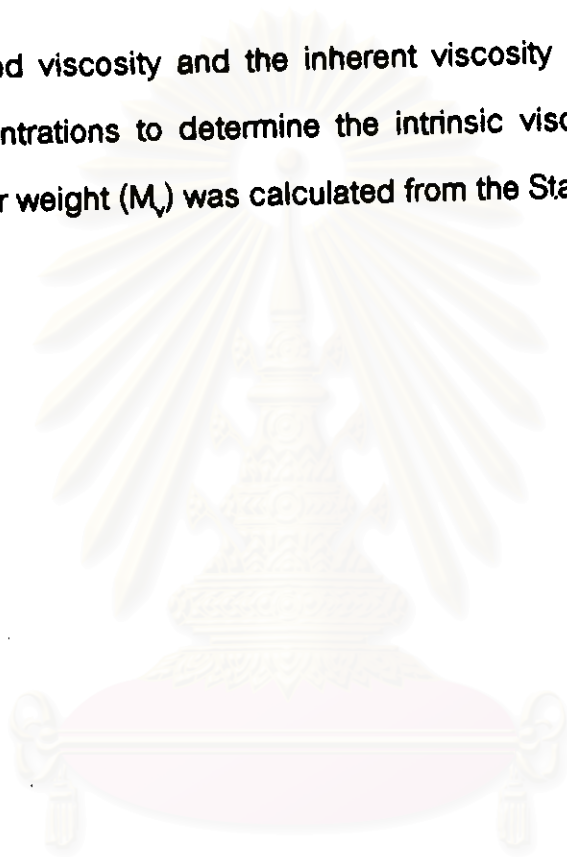
The glass transition temperatures of the pure polymer, the blends and liquid crystals were measured by DSC. Indium was used as a standard for calibration. The samples of 10-20 mg which weighed on DSC pan were scanned at the heating rate of 10 °C/min starting from 30 °C and ending at 300 °C. The DSC thermogram of heat flow versus temperature was analyzed. The glass transition is taken to be the inflection point of the curve. Data reported here were taken from second heating runs made after cooling with an ice/water.

#### 4.4.4 Viscosity Average Molecular weight

The molecular weights of polymers were measured by dilute solution technique. The Cannon Fenske viscometer was used. The procedure was followed by ASTM D2515. The unknown molecular weight polymer or blend was weighed about 2 g and dissolved in 100 ml of dichloromethane in a volumetric flask. The stock solution was allowed to equilibrate at 25 °C in a water bath. Pipette the stock solution to achieve the dilute solutions with have 4 different concentrations in 50 ml volumetric flask. The dilute solution was steeped in a water bath for at least 15 minutes before each measurement. For measuring the viscosity time, pipette 10 ml of

solution into viscometer. Each solution concentration was measured the time which the solution level passed the marked line. Each solution concentration was measured 4 times. The average time was noted and the relative viscosity, specific viscosity, inherent viscosity, reduced viscosity were calculated.

The reduced viscosity and the inherent viscosity were plotted as a function of concentrations to determine the intrinsic viscosities. Viscosity average molecular weight ( $M_v$ ) was calculated from the Staudinger equation.



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