

## CHAPTER III EXPERIMENTALS

### 3.1 Materials

2,2'-Azobisisobutyronitrile (AIBN), a commercial grade, was supplied by Pan Asia Industrial Co., Ltd., Thailand.

2,2'-Azobis-(2,4-dimethyl valeronitrile) (ADVN); a commercial grade, was supplied by Pan Asia Industrial Co., Ltd., Thailand.

Acetone; an analyze grade from Labscan Asia Co., Ltd., Thailand; %Purity = 99.5%; Boiling point = 56.1 °C.

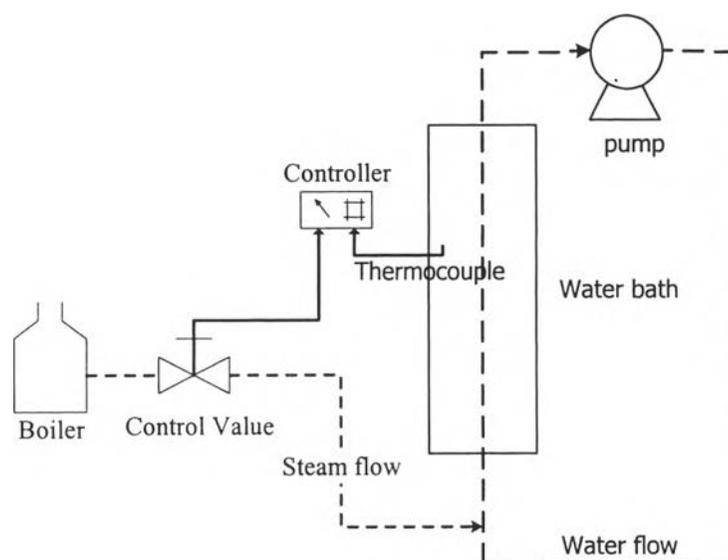
Methanol; an analyze grade from Labscan Asia Co., Ltd. Thailand; %Purity = 99.8%; Boiling point = 64.5 °C.

Methyl methacrylate (MMA) monomer; a commercial grade, was supplied from PAN Industrial Co., Ltd., Thailand.

### 3.2 Equipments

#### 3.2.1 Heating-Water Bath

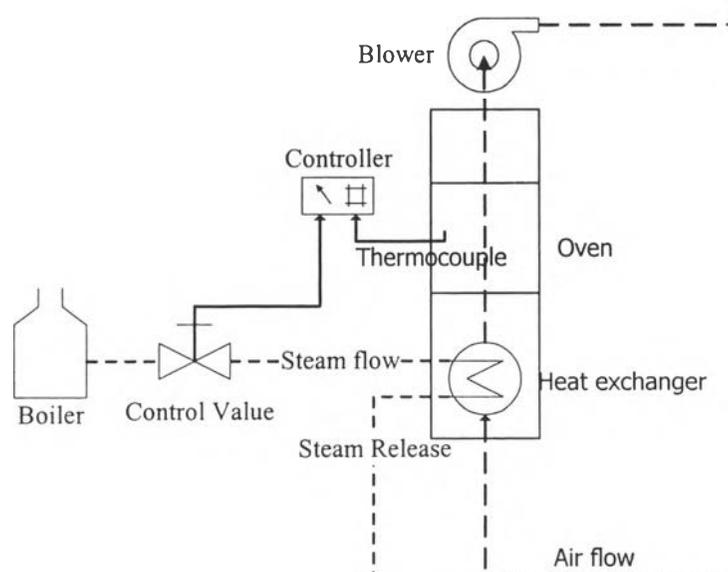
A pump was used for the circulation of water in a bath. The steam, heated source, was mixed with the water in the bath. In order to polymerize samples were put in the water bath. The diagram of the water bath system is shown in Figure 3.1.



**Figure 3.1** The diagram of water bath.

### 3.2.2 Heating-Air Oven

A blower circulated the heated air in an oven. The heated source of hot air was steam from the boiler in which the amount of steam was controlled by electronic controlled valve. Samples were placed in the oven during polymerization. The diagram of the oven system is shown in Figure 3.2.



**Figure 3.2** The diagram of hot-air oven.

### 3.2.3 Glass Molds and PVC Gaskets

Glass molds were the temper glass from the Thai-Asahi Co., Ltd. and PVC gaskets were produced by extruder in PAN Asia Co., Ltd.

### 3.2.4 Temperature Data Logger

In order to observe the temperature of reaction in the samples the temperature data logger was used.

### 3.2.5 Zwick Pendulum Impact Tester

Impact properties of the PMMA casted sheet were performed by Zwick pendulum impact tester model 5113. Using pendant load of 2.7 N, and the release angle was 124.4 degrees. The specimen size was 62 mm long and 12.7 mm width.

### 3.2.6 Hardness Measurement

Surface hardness of the PMMA casted sheet was measured by Rockwell Hardness tester in scale-M (Ball ¼ inch and load 100 kg).

### 3.2.7 Gel Permeation Chromatography (GPC)

Number average molecular weight ( $\overline{M}_n$ ), weight average molecular weight ( $\overline{M}_w$ ), z-average molecular weight ( $\overline{M}_z$  and  $\overline{M}_{z+1}$ ), and molecular weight distribution (*MWD*) of the PMMA casted sheets were measured by Waters Gel Permeation Chromatography Machine model Waters 150-CV attached with two columns: PL gel 10 µm mixed B and MW resolving range is 500-10,000,000.

## 3.3 Methodology

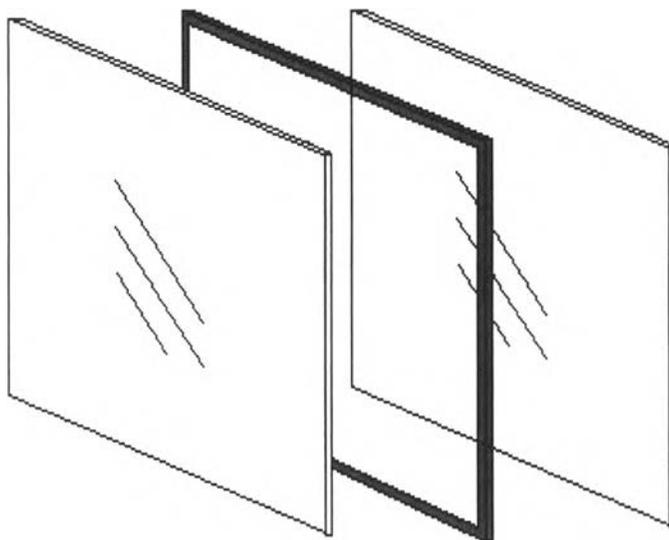
### 3.3.1 Preparation of Poly(methyl methacrylate) Syrup Solution

The PMMA syrup was prepared by mixing MMA monomer with 0.0018 %wt of azo-bisisobutyronitrile (AIBN) in a batch reactor and stirred for 40-45

minutes at polymerization temperature of 80°C. The syrup was controlled a percent conversion of about 7-10 % by the viscous time at 50 seconds measurements and it was filtrated and dried under vacuum to eliminate contaminations and bubbles before using for the starting materials.

### 3.3.2 Preparation of Glass Mold

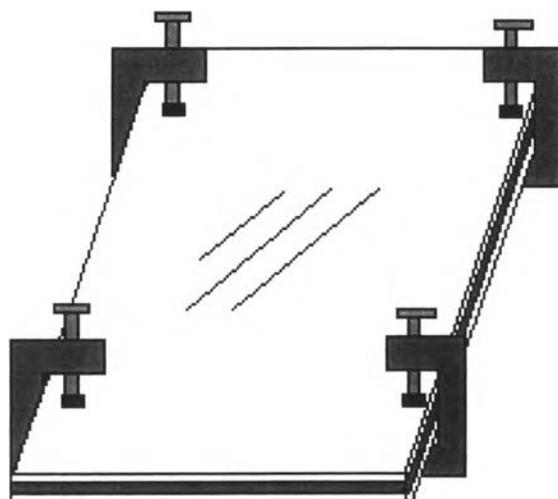
Glass molds were polished and inspected some defects such as scratches before use while PVC gaskets were prepared suitable thickness by using extruder. The PVC gasket and two glass molds were assembled that they were like a sandwich, as shown in Figure 3.3.



**Figure 3.3** The molds and the PVC gaskets before clamping.

### 3.3.3 Preparation of Poly(methyl methacrylate) Casted Sheet

The experiment was done to compare reaction time of polymerization process and the properties of the PMMA sheet from various thickness, pigments of PMMA sheet and initiator concentration.



**Figure 3.4** The mold after assembly.

PMMA syrup (from 3.3.1.) was, firstly, mixed with ADVN and additive such as pigment, UV stabilized, and others for 20 minutes. Secondly, the mixture was mixed into the glass mold in a certain dimension and it was clamped by C-clamps, shown in Figure 3.4. The mold was put into the heated source for 2-7 hours (depend on thickness and types of PMMA sheet) at specified temperature and annealed at temperature range from 105 to 120°C. After that, PMMA sheet was taken out from the mold by releasing the clamps. The physical and chemical conditions of each sample preparation were given in Table 3.1. The cast mould dimension was 300 mm long, 120 mm wide, and 3-10 mm thick

**Table 3.1** Physical and chemical conditions used for preparing PMMA sheets

Varying Parameter	Thickness or Pigments	Polymerization Temperature (°C)	Annealing Temperature (°C)	Initiator Concentration (%)
Thickness				
5 mm	000	60	120	0.022, 0.018, 0.015
8 mm				0.013, 0.011, 0.008
10 mm				0.008, 0.006, 0.004
Pigments				
000, 018	3 mm	60	120	0.15 (AIBN) 0.038 (ADV N)
102, 115				
136, 137				
202, 212				
235, 302				
327, 348				
362, 373				
402, 422K				
433S, 502				
522, 531				
814, 993				

### 3.4 PMMA Casted Sheet Characterization

#### 3.4.1 PMMA Yield Measurement

After polymerization, the sample was taken out of the glass mold, if the sample was still liquid, it was weighed to 5 g and poured into 125 mL Erlenmeyer flask and if the sample is solid, the sample was weight to 0.5 g. Sample was dissolved by 30 mL acetone at room temperature until sample was dissolved all.

After that, the 70 mL of methanol was added to the solution at room temperature to precipitate PMMA part, and the excess monomer and initiator were washed out.

After filtrated, purified PMMA samples were dried in vacuum drier at least 6 hrs to exclude residual solvent and were weighed. The percent yield of PMMA was calculated using equation 3.1:

$$\% \text{ Yield of PMMA} = \frac{W_1 \times 100}{W_2} \quad <3.1>$$

where  $W_1$  = weight of PMMA before dissolving,  
 $W_2$  = weight of PMMA after precipitation.

#### 3.4.2 Mechanical Characterization

To investigate mechanical properties of the blends, the final PMMA cast sheet from each condition was cut into specific size for each measurement. Zwick pendulum impact tester using 2.7 Joules striker measured impact resistance using unnotched Izod test method following ASTM D256. Surface hardness of the PMMA sheet was measured using Rockwell Hardness Tester following ASTM D2240.

#### 3.4.3 Molecular Weight and Molecular Weight Distribution Determination

The molecular weight ( $\overline{M}_n$ ,  $\overline{M}_w$ ,  $\overline{M}_z$ , and  $\overline{M}_{z+1}$ ) and MWD of selected PMMA cast sheet were determined by using GPC technique as described in 3.2.7.

### 3.5 Modeling of the PMMA Sheet-Casting process

Studied all equation that concern with the process such as kinetic equation of PMMA, equation of the Trommsdorff effects. Then solve all equation to investigate the temperature profile, conversion profile and molecular weight profile by using computer program.