CHAPTER III
EXPERIMENTAL

3.1. Materials

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

2,2'-Azobisisobutylonitrile (AIBN), a commercial grade, was supplied by Pan Asia Industrial Co., Ltd., Thailand, Half-life is approximately 1000 min at 60°C.

2,2'-Azobis-(2,4-dimethyl valeronitrile) (ADVN); a commercial grade; was supplied by Pan Asia Industrial Co.,Ltd., Thailand; its half-life is approximately 200 min at 60°C.

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

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

Tetrahydrofuran (THF); HPLC grade from Labscan Asia Co., Ltd. Thailand; Assay = 99.9%.

All chemicals were used as received.

3.2 **Equipment**

3.2.1 Heating-Air Reactor

The heated air in the reactor was circulated by using a blower. The temperature of hot-air was controlled by steam from the boiler in which the amount of steam was controlled by electronic controlled valve. Samples were placed in the reactor during polymerization. The diagram of the reactor is shown in figure 3.1.

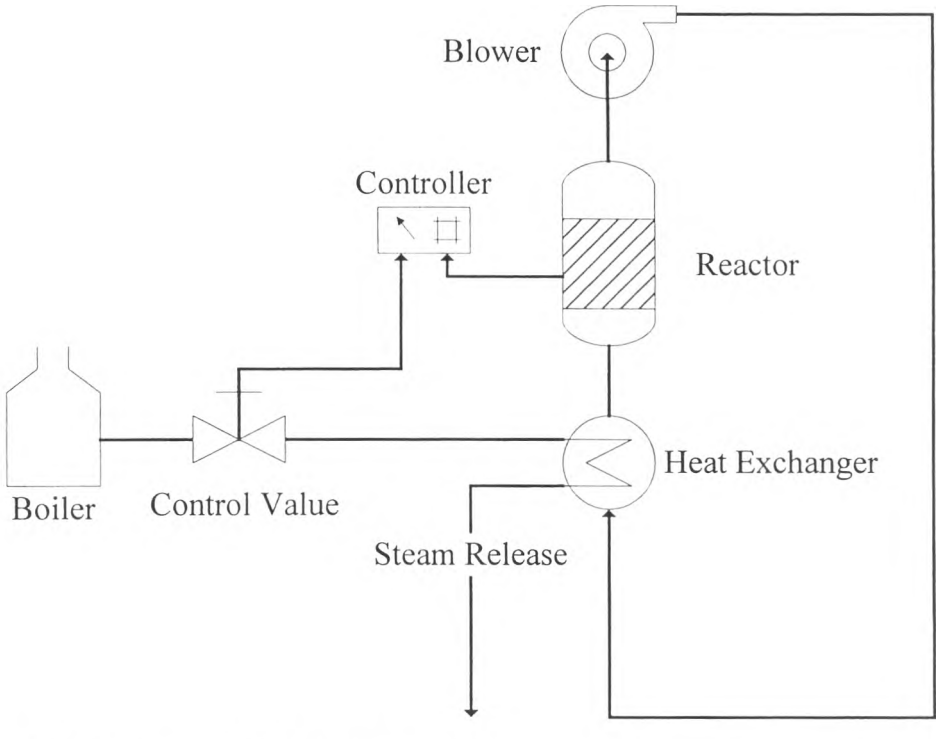

Figure 3.1 A diagram of hot-air reactor.

3.2.2 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 sample size was 3 mm, 63.5 mm long, 12.7 mm width, and 2.5 mm notch grooved.

3.2.3 Hardness Measurement

Surface hardness of PMMA casted sheets were measured by Zwick shore D durometer model 3100.

3.2.4 Gel Permeation Chromatography (GPC)

Number average molecular weight ($\overline{M}_n$), weight average molecular weight ($\overline{M}_w$), and molecular weight distribution (MWD) of PMMA casted sheets were measured by Waters Gel Permeation Chromatography Machine model 600E attached with RI (Waters) 410 and UV (Waters 486) detectors. Three columns, Styragel HR0.5, HR4E, and HR5E were connected in series and THF (HPLC grade) was used as an eluent. The flow rate was maintained at 1 ml/min throughout the experiment.

3.3. Methodology

3.3.1 Preparation of Poly(methyl methacrylate) Syrup Solution

PMMA syrup solution was prepared by mixing MMA monomer with 10 ppm of azo-bisisobutylonitrile (AIBN) in a batch reactor and stirred for 30 minutes at polymerization temperature of 80 °C. The syrup will have a percent conversion of about 7 % and it was used as the starting material to make PMMA casted sheet.

3.3.2 Preparation of Glass Mold

Firstly, two glass plates were cleaned in order to get very smooth surface. Secondly, a cylindrical PVC extrudate with a diameter of 5 mm. was attached to one glass plate. Thirdly, another glass plate was placed on top of the prior one. Finally, clamps were used to tighten the glass mold.

3.3.3 Preparation of Poly(methyl methyacrylate) Casted Sheet

The experiment was done to compare the properties of the PMMA sheet from various polymerization temperatures (either isothermal or programmed temperature profiles) and initiator concentration.

PMMA syrup (from 3.3.1.) was, firstly, stirred with ADVN for 20 minutes. Secondly, the mixture was poured into the glass mold in a certain dimension. The mold was put into the heating oven for 4 hours at specified temperature. After 4 hours, PMMA casted 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 mm thick.

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 weighted to 5 g and pour into 125 ml Erlenmeyer flask, if the sample is solid, the sample was cut into a $3\times10\times15$ mm^3 diameter. Sample was dissolved in 30 ml acetone at room temperature for 24 hr. 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.

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

Varying Parameter	Casting Temperature (°C)	Initiator Concentration (wt%)	Polymerization Time (min)
Isothermal casting temperature	50, 58, 60, 62, 70	0.038	240
Initiator concentration	60	0.019, 0.038, 0.076	240
Non-isothermal temperature profiles	- 60 for 150 min, 120 for 90 min - 62 for 150 min, 120 for 90 min	0.038	240

After that purified PMMA samples were dried in vacuum drier for 4 hr to exclude residual solvent and were weighed. The percent yield of PMMA was calculated using this equation:

$$\% \text{ yield of PMMA} = \frac{W_2 \times 100}{W_1} \tag{3.1}$$

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 casted sheet from each condition was cut into specific size for each measurement. Impact resistance was measured using ASTM D256 test method by Zwick impact tester using 2.7 Joules striker. Surface hardness of the PMMA sheet was measured using Shore D Hardness Durometer following ASTM D2240.

3.4.3 Molecular Weight and Molecular Weight Distribution Determination

The molecular weight ($\overline{M}_n$ and $\overline{M}_w$) and MWD of selected PMMA casted sheet were determined by using GPC technique as described in 3.2.5.