CHAPTER III
EXPERIMENTAL

3.1 Materials

A commercial grade of 2,2'-Azobisisobutylonitrile (AIBN) was supplied by Pan Asia Industrial Co., Ltd., Thailand.

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

An analytical grade of acetone (99.5% purity and the boiling point of 56.1°C) was purchased from Labscan Asia Co., Ltd., Thailand.

An analytical grade of methanol (99.8% purity and the boiling point of 64.5°C) was purchased from Labscan Asia Co., Ltd., Thailand.

A commercial grade methyl methyacrylate (MMA) monomer was supplied by Pan Asia Industrial Co.,Ltd., Thailand.

3.2 Equipment

3.2.1 Heating-Water Bath

A pump was used for the circulation of water in a bath. The steam, heating source, was mixed with the water in the bath. For polymerization reaction to occur, samples were immersed in the water bath. The diagram of the heating water bath system is shown in Figure 3.1.

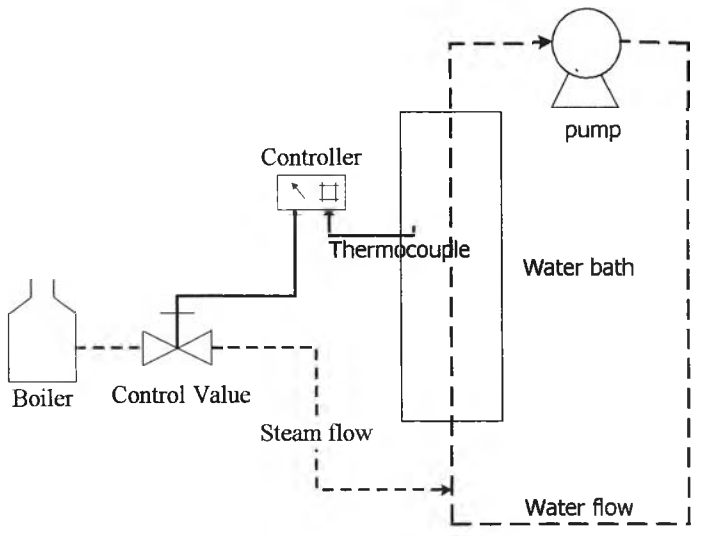

Figure 3.1 Diagram of heating water bath.

3.2.2 Heating-Air Oven

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

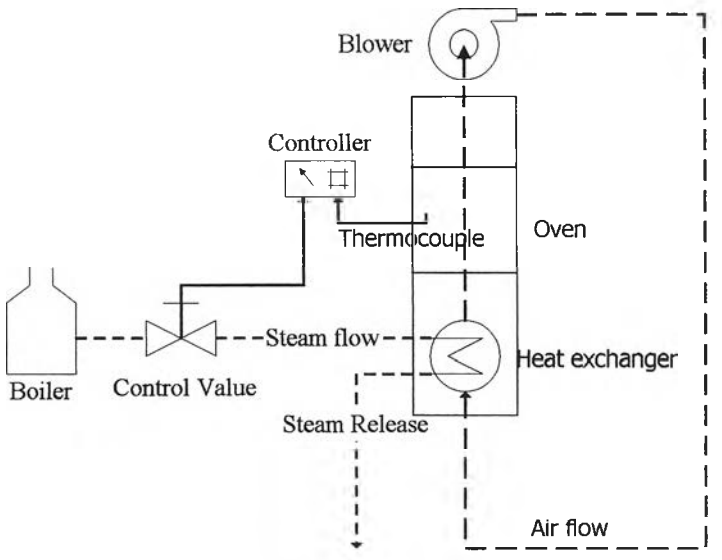

Figure 3.2 Diagram of hot-air oven.

3.2.3 Glass Molds and PVC Gaskets

Glass molds were the temper glass from the Thai-Asahi Co.,Ltd. and PVC gaskets were melt-extruded.

3.2.4 Temperature Data Collector

In order to observe temperature evolution in the samples during the reaction, a hand-made temperature data collector was used.

3.2.5 Zwick Pendulum Impact Tester

Impact properties of the PMMA casted sheets were performed by Zwick pendulum impact tester model 5113, using pendant load of 2.7 N and the release angle of 124.4 degrees. The specimen size was 3 mm in thickness, 63.5 mm in length, and 12.7 mm in width.

3.2.6 Hardness Measurement

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

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 10 ppm of azo-bisisobutylonitrile (AIBN) in a batch reactor and stirred for 30 minutes at the polymerization temperature of 80°C. The monomer conversion of the syrup was

controlled to be about 7 to 10% by simply controlling the viscosity of the syrup. The syrup was then filtrated and degassed before further use.

3.3.2 Preparation of Glass Mold

Glass molds were polished and inspected for some defects such as scratches before use while PVC gaskets were melt-extruded. A PVC gasket and two glass molds were assembled in a the fashion schematically shown in Figure 3.3. The mold dimension was 300 mm in length, 120 mm in width, and 3 mm in thickness.

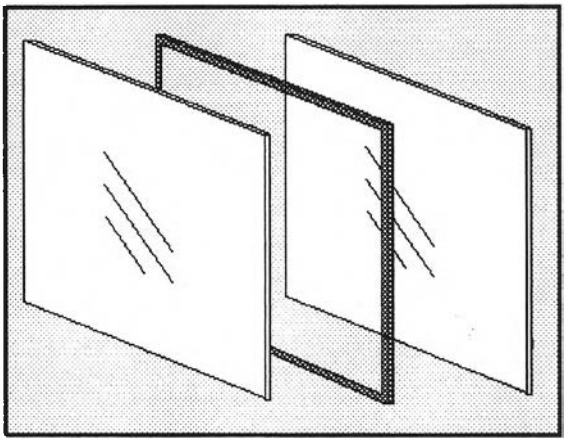

Figure 3.3 Assembly of molds and a PVC gasket before clamping.

3.3.3 Preparation of Poly(methyl methacrylate) Casted Sheet

The experiment was carried out to compare the properties of PMMA sheets prepared from various polymerization conditions (according to either the one-step isothermal or two-step isothermal process) and initiator concentrations.

PMMA syrup (see Section 3.3.1.) was first stirred with ADVN for 20 minutes. The mixture was then poured into the as-prepared glass mold and it was clamped by C-clamps, as shown in Figure 3.4. The molding was later put into an appropriate heating source (use either water or air as the heat transfer medium) for 4 hours at a specified temperature. After 4 hours, the PMMA sheet was taken out from the mold. Numerous PMMA sheets were prepared according to the experimental conditions shown in Table 3.1.

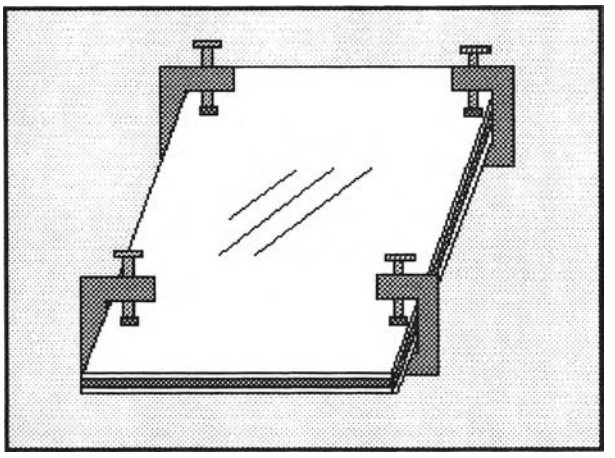

Figure 3.4 Mold after assembly.

Table 3.1 Experimental conditions for preparing PMMA sheets

Process	System	Polymerization Temperature (°C)	Initiator Concentration (%w/w)	Polymerization Time (min.)
One-step Isothermal Process	Water, hot air	60, 62, 65, 68, 70	0.030, 0038	240
Two-step Isothermal Process	Water-air,	60-105, 60-115, 60-125, 63-105, 63-115, 63-125, 65-105, 65-125	0030, 0038	240
	Air	60-105, 60-115, 60-125, 63-105, 63-115, 63-125	0030, 0038	240

3.4 Characterization of PMMA Sheets

3.4.1 PMMA Yield Measurement

After polymerization, the sample was taken out of the glass mold. If the sample was still in liquid form, 5 g of the sample was poured into 125 mL

Erlenmeyer flask. If the sample is in solid form, the sample, cut into a rectacgular shape of $3\times10\times15$ mm^3, was dissolved by 30 mL acetone at room temperature for 24 hrs. Then, 70 mL of methanol was added to the solution at room temperature to precipitate out PMMA. The excess monomer and initiator supposedly stayed in the solution.

After filtration, purified PMMA samples were dried in a vacuum drier for at least 6 hrs to rid of solvent and were weighed. The yield percentage of PMMA conversion was calculated according to Equation 3.1:

$$\% \text{ Yield of PMMA} = \frac{W_2 \times 100}{W_1} \qquad (3.1)$$

where W_1 = weight of PMMA before dissolving,

W_2 = weight of PMMA after precipitation.

3.4.2 Mechanical Characterizations

Only the samples attaining ultimate yield for each experimental condition were tested for their impact resistance and surface hardness. Impact resistance of the as-prepared samples was measured according to ASTM D256 by a Zwick impact tester equipped with the 2.7-Joules striker. However notch did not apply on the samples in this method because the equipment could not read the impact resistance value. Surface hardness of the as-prepared samples was measured using a Shore D Hardness Durometer according to ASTM D2240.

3.4.3 Molecular Weight and Molecular Weight Distribution Determination

The molecular weight averages ($\overline{M}_n$, $\overline{M}_w$, $\overline{M}_z$, and $\overline{M}_{z+1}$) and the molecular weight distribution (MWD) of selected as-prepared PMMA sheets were determined by size-exclusion chromatography (SEC) or gel-permeation chromatography (GPC) technique, as previously described in Section 3.2.7.