CHAPTER III
METHODOLOGY

3.1 Materials

Homogeneous catalysts used in this research are sodium carbonate (anhydrous) supplied by Riedel-de Haen and sodium hydroxide obtained from Lab Scan. Moreover, this research used heterogeneous solid catalysts, which are magnesium oxide (96.0% (AR)) and calcium oxide(96.0% (AR)) supplied by UNILAB, barium oxide (95% (purified)) and zirconium oxide (pure) supplied by Riedel-de Haen, and strontium oxide (99.9% (purified)) supplied by Aldrich.

3.2 Equipment

3.2.1 Reactor.

A 250-ml three-necked flask is equipped with a condenser, a thermometer and a sampling port was used in the experiment. The custom made furnace to fit with the three-necked flask was used to heat up and the temperature was digitally controlled. The nitrogen gas is used to purged the system and provide the inert atmosphere during reaction. The magnetic stirrer was used to provide agitation. The experimental set-up was shown in Figure 3.1

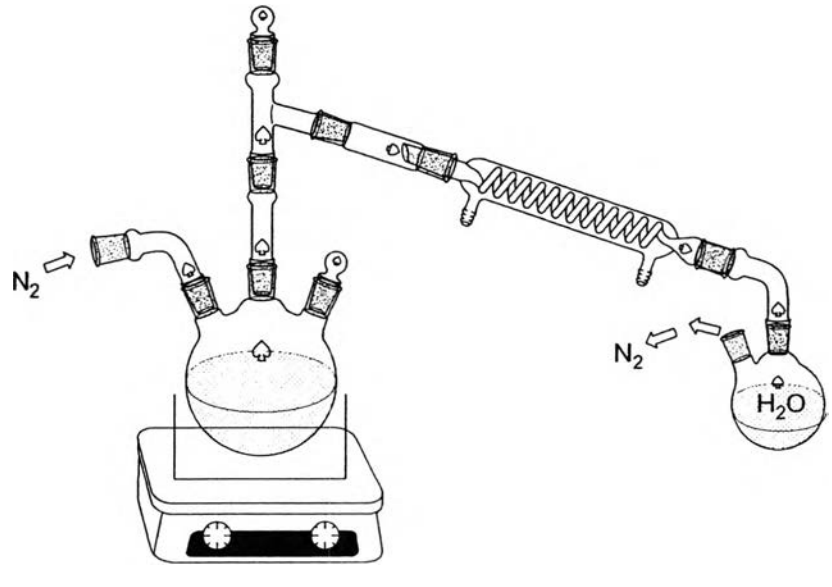

Figure 3.1 Experimental set-up used for synthesis polyglycerols.

3.2.2 High Performance Liquid Chromatography (HPLC)

The Perkin Elmer Series 200 high pressure liquid chromatography with refractive index Series 200 detector was used to analyze diglycerol product samples. The chromatographic column was ZORBAX SAX (4.6 mm×150 mm×5 μm). The mobile phase was acetonitrile/water mixture (80:20 vol/vol) at a flow rate 1.0 ml/min. The column temperature was at ambient temperature (27°C). The pump pressure was operated in the range of 300 to 600 psi. The polyglycerols samples were diluted with water and the injection volume was 20 μl. High Performance Liquid Chromatography (HPLC) was shown in Figure 3.2

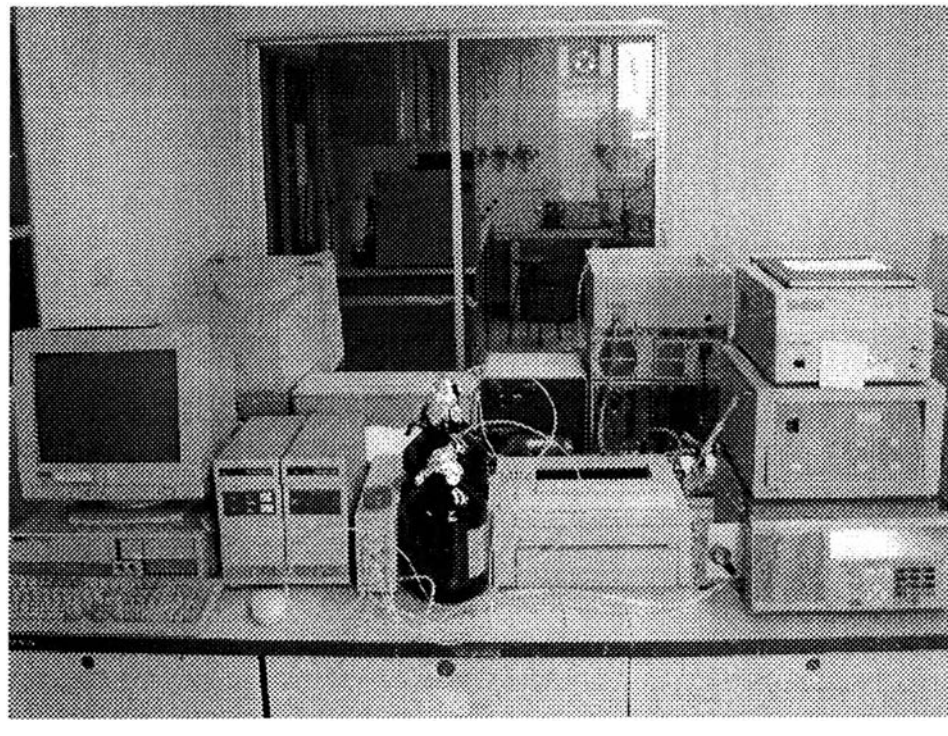

Figure 3.2 High Performance Liquid Chromatography (HPLC).

3.3 Methodology

3.3.1 Experimental

- **Polymerization of Glycerol by using Homogeneous Catalysts**

Fifty grams of glycerol was weighed and placed in a 3-neck round bottom flask. The flask was then heated to temperature 150°C under nitrogen atmosphere. After 30 minutes, the flask was heated to temperature 240°C and the desired amount of catalyst was weighed and mixed with glycerol in the reactor. The reaction was carried out until it reached the desired reaction time. The reactor was then cooled down to room temperature.

The homogeneous catalysts were sodium carbonate and sodium hydroxide. The polymerization reaction was studied at the reaction temperature 240 °C under nitrogen atmosphere and stirrer speed of 500 rpm.

- **Polymerization of Glycerol by using Heterogeneous Catalysts**

The investigation of heterogeneous catalysts was done by fixing the reaction temperature at 240 °C under nitrogen atmosphere and stirrer speed 500 rpm. The heterogeneous catalysts were calcium oxide, magnesium oxide, strontium oxide, barium oxide, and zirconium oxide.

The catalysts were studied in the effect of the reaction time (1, 2, 3, 4, 5 hrs), catalyst loading (1.0, 2.0, 4.0, 8.0, 12.0 wt%) and reaction temperature (220, 230, 240, 250, 260 °C).

3.3.2 Products Analysis

Analysis of the products was performed by using high-performance liquid chromatography (HPLC), a Perkin Elmer Series 200 LC-pump and a refractive index Series 200 detector. The system was controlled by a PC with a software package (Perkin Elmer Turbochrom Navigator). ZORBAX SAX column (4.6 mm×150 mm×5 μm) was used and the mobile phase was acetonitrile/water mixture (80:20 vol/vol) at a flow rate 1.0 ml/min. The column temperature was at ambient temperature (27°C). The pump pressure was operated in the range of 300 to

600 psi. The polyglycerols samples were diluted with water and the injection volume was 20 µl.

The glycerol conversion was defined as shown in Equation (3.1). In the first step, the weight of used glycerol was calculated from the approximately fifty gram of sample (from experimental part) subtract with the remaining of glycerol that calculate from HPLC chromatogram (from peak area convert to amount of glycerol in grams).

$$\text{Glycerol conversion (wt\%)} = \frac{\text{Weight of used glycerol}}{\text{Weight of starting glycerol}} \times 100 \qquad (3.1)$$

The diglycerol selectivity was defined as a ratio of weight of diglycerol, which was determined by using HPLC, to weight of product (except remaining glycerol) as shown in Equation (3.2).

$$\text{Diglycerol Selectivity (wt\%)} = \frac{\text{Weight of diglycerol}}{\text{Weight of product}} \times 100 \qquad (3.2)$$

The diglycerol yield was defined as a ration of weight of diglycerol, which was determined by using HPLC, to weight of starting of glycerol as shown in Equation (3.3).

$$\text{Diglycerol Yield (wt\%)} = \frac{\text{Weight of diglycerol}}{\text{Weight of starting glycerol}} \times 100 \qquad (3.3)$$