# 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. The solid catalysts used in this study are magnesium oxide (96.0% (AR)) and calcium oxide (96.0% (AR)) obtained from UNILAB and barium oxide (95% (purified)) purchased from Riedel-de Haen.

## 3.2 Equipment

### 3.2.1 Reactor

A 250-ml three-necked flask equipped with a reflux 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 supply heat and the temperature was digitally controlled by a temperature controller equipped with a thermo couple. The nitrogen gas was continuously purged the system to provide the inert atmosphere during reaction and to carry the water that is form during the raction out from the reaction. The magnetic stirrer was used to provide agitation. The experimental set-up was shown in Figure 3.1

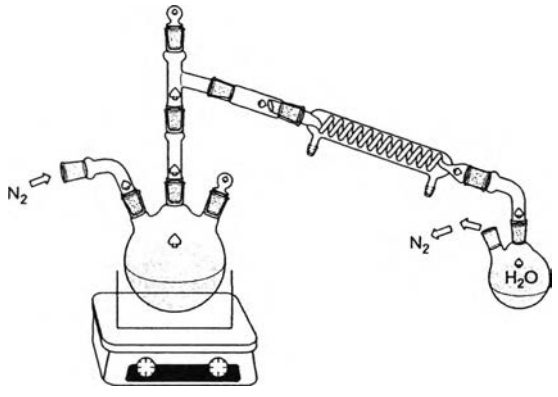


**Figure 3.1** Experimental set-up used for synthesis polyglycerols.

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. High Performance Liquid Chromatography (HPLC) was shown in Figure 3.2

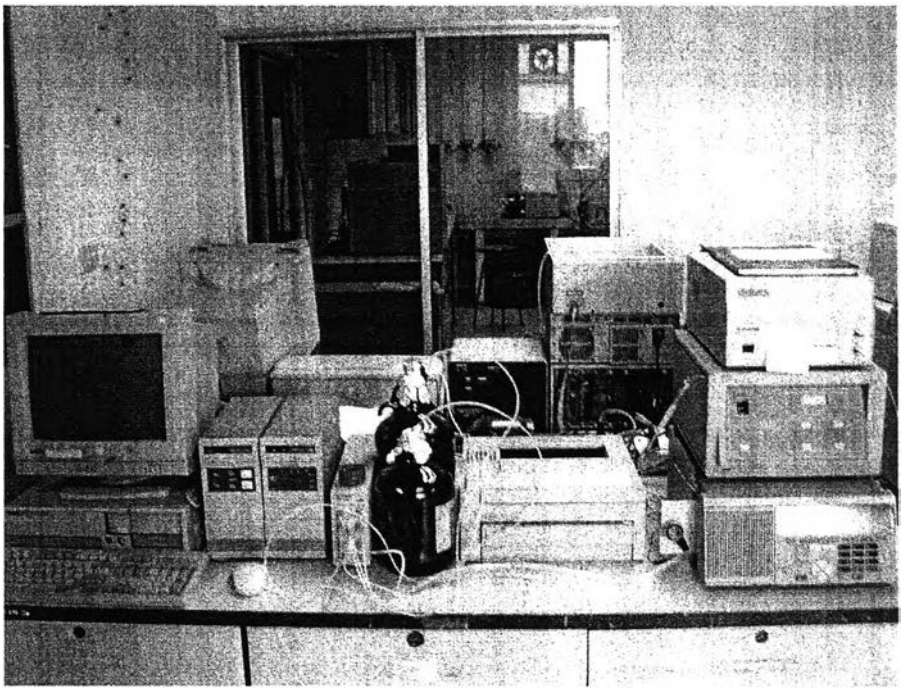

**Figure 3.2** High Performance Liquid Chromatography (HPLC).

**3.3 Methodology**

3.3.1 Reaction

Fifty grams of glycerol is weighed and placed in a 3-neck round bottom flask. The flask is then heated to temperature 150°C under nitrogen atmosphere. After 30 minutes, the flask is heated to desired reaction temperature and 1 g of catalyst was added in the reactor. The investigation of heterogeneous catalysts

is done under nitrogen atmosphere and stirrer speed is 500 rpm. The reaction is carried out until it reaches the desired reaction time. The reactor is then cooled down to room temperature. The catalysts are studied in the effect of the reaction time (1, 2, 3, 4, 5 hrs) and reaction temperature (220, 230, 240, 250°C).

3.3.2 Products quantification

Analysis of the products is performed by using HPLC,The column temperature is at ambient temperature (27°C). One gram of product was diluted with water in 25-ml volumetric flask prior to inject to HPLC and the injection volume was 20 µl.

The amount of glycerol and diglycerol are quantified by comparing the RID signal for each glycerol and diglycerol of the HPLC chromatogram of polyglycerols product with the RID signal of each glycerol and diglycerol standard.

The glycerol conversion is defined as shown in Equation (3.1). In the first step, the weight of glycerol used 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).

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

The diglycerol selectivity is defined as a ration 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 is 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).

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