CHAPTER III

EXPERIMENTAL METHOD

3.1 Materials

Polyurethane foam used in this experiment is rigid polyurethane foam for construction provided by Pacific Plastics (Thailand) Ltd. (DOW). Diethanolamine (M&B) and sodium hydroxide (J.T. Baker) were used as received. Bisphenol A type epoxy resin (Epiclon 850) was provided by Siam Chemical Industry Co., Ltd.

3.2 Instruments

The Infrared (IR) spectra were recorded on a Nicolet FTIR spectrophotometer. The Nuclear Magnetic Resonance (NMR) spectra were recorded on a Bruker Fourier Transform NMR spectrometer Model ACF 200. The Differential Scanning Calorimetry (DSC), Thermal Gravimetric Analysis (TGA) and Dynamic Mechanical Analysis (DMA) thermograms were recorded on Perkin-Elmer DSC 7, Perkin-Elmer TGA 7 and Netzsch DMA 242, respectively. Gel Permeation Chromatogram (GPC) was obtained by using the Millipore GPC Waters 400 E. Vicat softening point was measured by the HDT-VICAT model A/3 M. Hardness of cured epoxy resin was measured by the Shore Durometer Type D

3.3 Glycolysis of Foam

Glycolysis was carried out under nitrogen atmosphere in a four-neck round bottom flask equipped with a reflux condenser, a thermometer and a stirrer. Rigid polyurethane foam in cubic shape of about 1 cm^3 was added portionwise into this flask containing diethanolamine and pellet NaOH at temperature of 130 to 150°C. The decomposition reaction proceeded until the foam was completely dissolved. The glycolyzed products were then filtered when they were still hot by fine thin cloth of 1,600 mesh.

Three types of glycolyzed products were prepared as shown in Table 1.

Table 1 Three types of glycolyzed products

Glycolyzed Product	Weight of diethanolamine (g)	Weight of foam (g)	Weight of NaOH (g)
GP 11	300	300	0.4
GP 21	300	600	2.7
GP 31	150	450	1.8

GP11 was produced using only 0.4 g of NaOH in the glycolysis process. Before using GP11 to be a curing agent of epoxy resin, more amout of NaOH (1.4 g) was then dissolved in GP11 in order to be a catalyst in the curing reaction. Total amount of NaOH used in production of GP11 is 0.3% by weight of the glycolyzed product.

Unlike GP11, GP21 and GP31 were prepared using NaOH 0.3% by weight of glycolyzed products so obtained in the glycolysis process and they can be used to cure epoxy resin directly without adding more NaOH.

Because foam has to be added portionwise, so more foam leads to more feeding time. Consequently, the reaction time of the glycolyzed products is different and depends on the feeding rate and the amount of foam. Typically, a complete glycolysis reaction in this investigation took 50 to 60 hours.

3.4 Sample Preparation

Bisphenol A type epoxy resin was mixed with the glycolyzed product in a round bottom flask at different ratios. Vacuum was then applied in order to remove bubbles generated during mixing. The mixtures were heated in an oven at temperature of 150°C for 100 minutes. The moulds were made of aluminum foil which can be peeled out after curing. Subsequently, the cured resins were cooled down in a freezer and cut into required dimensions.

Test specimens for measuring hardness are at least 6 mm in thickness. The measurements were done in an area of at least 12 mm from any edge (ASTM D2240-86)

Test specimens for Vicat softening point measurement are flat, between 3 and 6.5 mm thick and at least 10x10 mm in area. At least two measurements were carried out for each sample (ASTM D1525).

Names of different types of cured epoxy resin are represented by P with four digits where first two digits indicate types of the glycolyzed products used and the last two digits show weight ratios of the glycolyzed products to the epoxy resin.

Specimens for DMA are approximately 1x5.5 cm in dimensions and 2 to 3 mm in thickness. All specimens were prepared to be flat and uniform in thickness.

Samples used in Infrared Spectroscopy were prepared by filtering them through Teflon® membranes. Heating was required in order to decrease sample viscosity to assist the filtration. Filtered samples were then applied, as thin as possible, on a KBr plate before a measurement was carried out. ^{1}H and ^{13}C NMR measurements were performed by using $CDCl_3$ as a solvent.

In the case of Gel Permeation Chromatography, glycolyzed products were dissolved in tetrahydrofuran. The solutions were filtered through the membranes made of Teflon® and then injected into the instrument. Polymer standards used are polystyrene which have molecular weights of 418, 456, 500, 2630, and 2980 g/mole.