



CHAPTER IV

EXPERIMENT

This experiment consists of two parts. The first part is depolymerization of polyethylene terephthalate (PET). This part is to study the effect of the conditions of depolymerization on hydroxyl value of the glycolized product. The second part concerns polyurethane manufacturing. In this part, the glycolized product was used as the chain extender for polyurethane synthesis. The mechanical properties of the polyurethane with the glycolized product as the chain-extender and those of the polyurethane with ethylene glycol as the chain-extender were compared. The details of the experiment are in the following sections :

4.1 Depolymerization of PET

PET was depolymerized by excess ethylene glycol. The part of depolymerization of PET consists of the following headings :

- 4.1.1 Chemical and raw material preparation
- 4.1.2 Apparatus
- 4.1.3 Procedure
- 4.1.4 The scheme of depolymerization of PET
- 4.1.5 Determination of hydroxyl value
- 4.1.6 Identification of functional group of the glycolized product
- 4.1.7 Viscosity of the glycolized product to determine \overline{M}_v

4.1.1 Chemical and Raw material preparation

a. PET (Polyethylene terephthalate)

In this work, PET from post-consumer drinking water bottles was used. The trademark of the PET bottles is POLY. The PET bottles were crushed by a grinder. Average size of crushed PET were smaller than 5×5 square mm.

b. EG (Ethylene glycol) CH₂OHCH₂OH

A chemical grade of ethylene glycol was purchased without further purification. Ethylene glycol was produced by Ajax Chemical, 9 Short Street Auburn N.S.W. 2144 Australia.

c. Zinc acetate (CH₃COO)₂Zn . 2H₂O

A chemical grade of zinc acetate was purchased without further purification. Zinc acetate was produced by Ajax Chemical, 9 Short Street Auburn N.S.W. 2144 Australia.

4.1.2 Apparatus

The apparatus for depolymeriation of PET consisted of the following units:

- a. Three of necked resin reaction kettle, with three standard tappers ; one of 14/26 joint and the others of 24/29 joints.
- b. Thermometer of temperature range from 200 to 300 °C.
- c. Reflux condenser, with standard - tapper 14/26 joint, 12 ft., used to prevent ethylene glycol releasing and control pressure in the kettle at normal.
- d. Clamp
- e. Electromantle
- f. Glass cork
- g. Flask 250 ml.

4.1.3 Procedure

The crushed PET was depolymerized by glycolysis of ethylene glycol with zinc acetate as the catalyst. The crushed PET was accurately weighed, then transferred to three-necked resin reaction kettle. Zinc acetate was accurately weighed at 0.5 % of PET weight. The catalyst was mixed together with PET flakes into the kettle. Suitable measured ethylene glycol was poured in the kettle. All components in the kettle were vigorously mixed. The thermometer, the reflux condenser and the glass cork were fitted to the neck of the kettle. The kettle was placed in the heating mantle. The temperature of the heating mantle was set.

Depolymerization runs were done at the temperature between 190 to 200°C. When depolymerization finished at the appointed time, the mixture was divided into two parts. The first one was poured on a salver and the mixture became solid at the temperature around 50 to 60°C. Some solid mixtures were taken to proof hydroxyl value before ethylene glycol extraction. The solid mixture were called glycolized product before ethylene glycol extraction.

The other was poured into the flask with 250 ml. of the distilled water to extract ethylene glycol. The unreacted ethylene glycol can dissolve in water. The powdered solid was separated from the solution. The ethylene glycol - dissolved water was poured off and the new distilled water was added to the powdered solid again and stirred vigorously. After stirring, the flask was set still to allow phase separation to occur. The solid powder sank at the bottom of the flask. The solid powder was washed by water 10 times, while filtration was aided by suction method. The solid powder was placed in the vacuum oven at the temperature of 60°C for 24 hours to release water. The dry solid powder was taken to determine its hydroxyl value after ethylene glycol extraction. The dry solid powder was called glycolized product after ethylene glycol extraction.

4.1.4 The scheme of depolymerization of PET

1. The first series of is to study the effect of PET/EG ratio on hydroxyl value of glycolyzed product before and after ethylene EG extraction. The two conditions of the experiment were controlled. The temperature was controlled at 190°C and the depolymerized time was controlled at 4 hours. PET/EG ratios were varied in this experiment. Table 4.1 displayed the weight of PET and zinc acetate and volume of ethylene glycol, that were used in each batch of the experiment.

2. The second series of experiment is to study the effect of depolymerization times on hydroxyl value of glycolyzed product after ethylene glycol extraction. The PET/EG ratio was controlled at 1:1.5 ,1:2.5 and 1:4. The temperature was controlled at 190°C. Depolymerized times were varied from 1 hours to 9 hours. Sample were taken at the end of each hour. Table 4.2 displayed the weight of PET and zinc acetate and volume of ethylene glycol, that were used in the various depolymerized times.

3. The third series of experiment is to study the effect of temperature on hydroxyl value of glycolyzed product after ethylene glycol extraction. The PET/EG ratio was controlled at 1:1.5 and the depolymerized time was controlled at 4 hours. The temperature of the experiment were varied. Table 4.3 displayed the weight of PET and zinc acetate and volume of ethylene glycol, that were used in the temperature range from 190 °C to 250 °C.

Table 4.1 Quantity of PET, ethylene glycol and zinc acetate used in depolymerization at controlled condition I.

Control Condition I Temperature 190 °C

Depolymerization time 4 hours

Varying Condition I PET / Ethylene glycol ratio

Molar ratio of PET / EG	Code	Weight of PET (g)	Weight of Zinc acetate (g)	Weight of Ethylene glycol (g)
1: 1.5	GP(1,5,4,190)	48	0.24	14
1: 2	GP(2,4,190)	48	0.24	19
1: 2.5	GP(2,5,4,190)	48	0.24	23
1: 3	GP(3,4,190)	48	0.24	28
1: 3.5	GP(3,5,4,190)	48	0.24	33
1: 4	GP(4,4,190)	48	0.24	37

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Table 4.2 Demonstration of depolymerization times and quantity of PET, ethylene glycol and zinc acetate used in depolymerization at controlled condition II.

Control Condition II PET/Ethylene glycol ratio used 1:1.5,1:2.5,1:4

Temperature 190° C

Varying Condition II Depolymerization time (hours)

Molar ratio of PET / EG	Code	Weight of PET (g)	Weight of Zinc acetate (g)	Weight of Ethylene glycol (g)
1:1.5	GP(1.5, hrs. , 190)	48	0.24	14
1:2.5	GP(2.5, hrs. , 190)	48	0.24	23
1:4	GP(4, hrs. , 190)	48	0.24	37

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Table 4.3 Demonstration of temperature and quantity of PET, ethylene glycol and zinc acetate used in depolymerization at controlled condition III.

Control Condition III PET/Ethylene glycol ratio used 1:1.5

Depolymerization time 4 hours

Varying Condition III Temperature (°C)

Temperature (°C)	Code	Weight of PET (g)	Weight of Zinc acetate (g)	Weight of Ethylene glycol (g)
190	GP(15,4,190)	48	0.24	14
210	GP(15,4,210)	48	0.24	14
230	GP(15,4,230)	48	0.24	14
250	GP(15,4,250)	48	0.24	14

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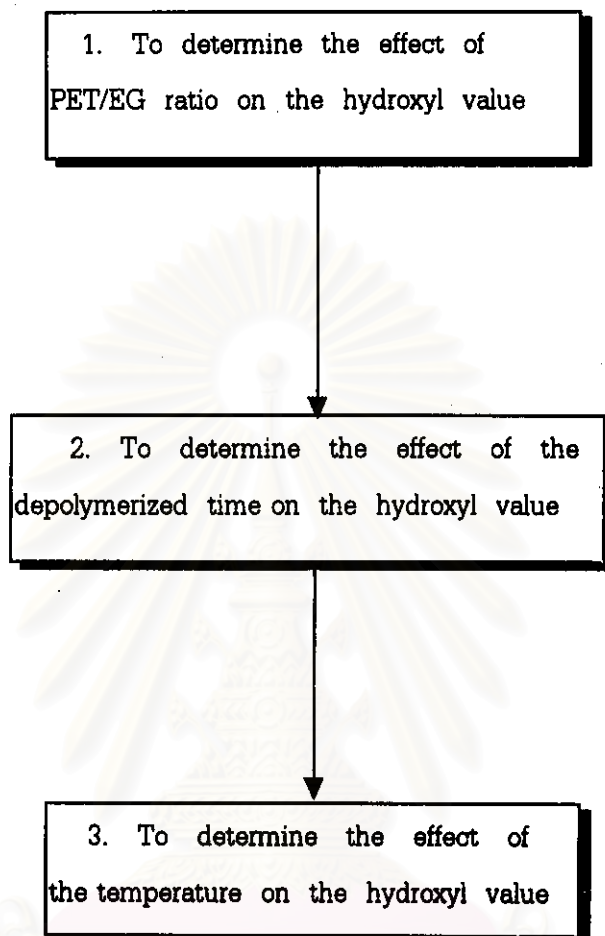


Figure 4.1 The scheme of depolymerization of PET

4.1.5 Determination of hydroxyl value (ASTM D2849 Method A)

The hydroxyl value of the glycolized products before and after ethylene glycol extraction were analyzed according to ASTM D2849 Method A. This section consisted of five headings :

4.1.5.A Chemicals

4.1.5.B Reagent preparation

4.1.5.C Apparatus

4.1.5.D ASTM D2849 Method A

4.1.5.E Calculation

4.1.5.A Chemicals

a. Pyridine C_5H_5N : A chemical grade of pyridine was purchased from the market without further purification. Pyridine was produced by Ajax chemical, Short Street Auburn N.S.W. 2144 Australia.

b. Acetic anhydride $C_4H_6O_3$: A chemical grade of acetic anhydride was purchased from the market without further purification. Acetic anhydride was produced by Riedel de Haen.

c. Phenolphthalein $C_8H_6O_4$: A chemical grade of phenolphthalein was purchased from the market. Phenolphthalein was supplied by Merck.

4.1.5.B Reagent Preparation

The reagent preparation is described as follows.

Acetylating Solution

Pyridine and acetic anhydride was mixed by using 88 volumetric percent of pyridine and 12 volumetric percent of acetic anhydride. The solution was prepared in a hood for avoiding danger.

Acetylating Indicator

Phenolphthalein 1 g. was dissolved in 100 ml. of pyridine, to obtain acetylating indicator solution. The solution was prepared in a hood for avoiding danger.

Potassium Hydroxide Solution (KOH solution)

Solid potassium hydroxide was accurately weighed 28.06 g. The weighed solid potassium hydroxide was dissolved in 1 liter of distilled water to make 0.5 N. KOH solution.

4.1.5.C Apparatus

The apparatus for determining hydroxyl value consisted of the following units :

- a. Reflux condenser, with standard taper 24/29 joint, 14 ft.
- b. Thermometer of temperature range 0 to 100°C.
- c. Flask, 250 ml., with standard taper 24/29 joint.
- d. Buret, the total capacity of buret was 100 ml.
- e. Hot plate, with variable resistance for temperature control.
- f. Water bath, maintained at 92 ± 2 °C

4.1.5.D ASTM D2849 Method A

This method is described as follows. About 0.5 g. of sample was accurately weighed and added to 10 ml. of acetylating solution, in a 250 ml. round bottom flask. The flask was fitted with a vertical reflux condenser and heated with stirring in boiling water bath for about 2 hours. The mixture was cooled to room temperature and hydrolyzed by distilled water. The resulting solution was titrated against 0.5 N KOH standard using phenolphthalein as indicator.

A blank run was performed without the sample. The 10 ml. of acetylating solution was added to a 250 ml. round bottomed flask, then fitted the flask with the vertical reflux condenser. The flask was heated by boiling water bath for about 2 hours. After cooling flask to room temperature, the mixture in the flask was hydrolyzed by distilled water. The hydrolyzed mixture was titrated against 0.5 N. KOH standard using phenolphthalein as an indicator.

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4.1.5.E Calculation

ASTM D2849 Method A uses an equation to calculate hydroxyl value. The SI units was used in the equation. The equation is :

$$(4.1) \quad \text{Hydroxyl Value} = \frac{(V_{\text{blank}} - V_{\text{sample}}) [\text{KOH}]}{W_{\text{sample}}}$$

Where :

V_{blank} is the volume of 0.5 N. KOH that reacted against the hydrolyzed blank.

V_{sample} is the volume of 0.5 N. KOH that reacted against the hydrolyzed mixture.

[KOH] is the concentration of potassium hydroxide solution.

W_{sample} is the weight of sample taken.

From the equation, the quantity of the hydroxyl value was obtained. The unit of the hydroxyl value is mg. of KOH/g. of sample. As the unit of the volume of the 0.5 N. KOH is milliliters (ml.). Normal (N.) is the unit of the KOH concentration. The unit of the weight of sample taken is gram (g.).

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4.1.6 Identification of functional group of the glycolyzed product

NICOLET FT-IR Impact 400 spectroscopy at Chemical Engineering Laboratory , Chulalongkorn University, was used to identify the functional group of glycolyzed product, i. e. , BHET , GP(1.5,4,190) without EG ,GP(4,8,190) without EG.

BHET

BHET was the final products of PET depolymerization . GP(4,8,190) without EG was mixed with distilled water. It was suspension. The suspension was heated , BHET dissolved in boiling water. The suspension filtration was aided by suction method , when the suspension was still boiling. The filtrate contained BHET and distilled water. The filtrate was heated again to concentrate the BHET solution , after that poured the concentrated BHET solution into the beaker set temperature at 0 °C, to crystallize BHET . The crystalline BHET occurred and was separated from the solution by used suction method. The method to produce BHET shows in figure 4.2.



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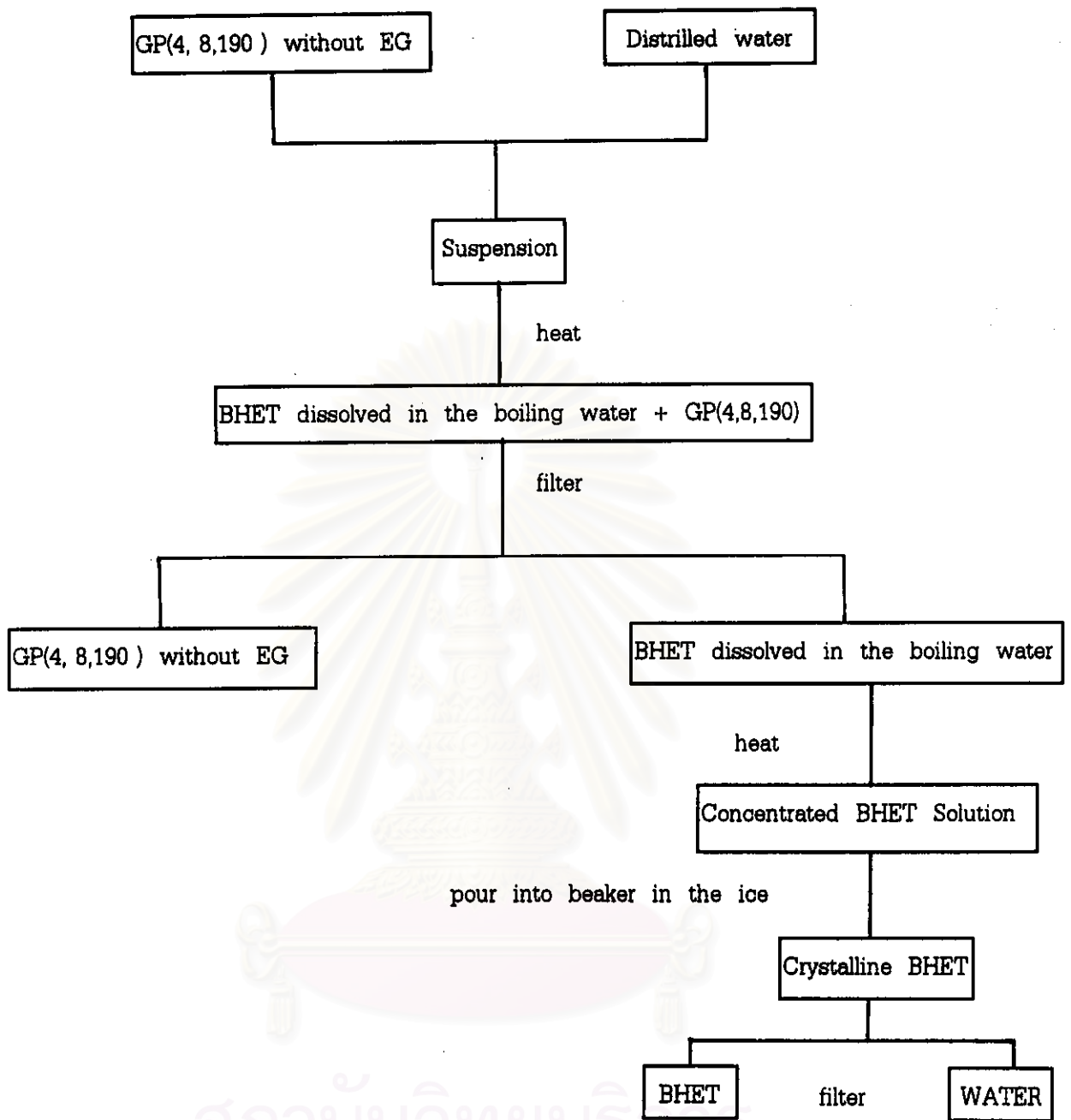


Figure 4.2 Method to produce BHET from GP(4,8,190) without EG



4.1.7 Viscosity of the glycolized product solution determined \overline{Mv}

The viscosity of the glycolized product was calculated from the relative viscosity. The relative viscosity was determined by using ASTM D4603, measuring the flow time of a solution of the glycolized product concentration and the flow time of the pure solvent in a capillary viscometer at a fixed temperature. The relative viscosity value is calculated from t/t_0 , t is the average glycolized product solution flow time (second, s.) and t_0 is the average solvent flow time (second, s.). The relative viscosity was used to calculate the intrinsic viscosity, using Billmeyer relationship. The equation 4.2 is Billmeyer relationship.

$$(4.2) \quad \eta = 0.25 (\eta_r - 1 + 3 \ln \eta_r) / C$$

where :

η is the intrinsic viscosity, (deciliters / gram, dL./g.)

η_r is the relative viscosity.

C is the concentration of glycolized product solution.
(gram/deciliters, g./dL.)

The intrinsic viscosity was used to calculate the viscosity average molecular weight of the polymer (\overline{Mv}). The equation of Mark-Houwink equation presents the relation of the intrinsic viscosity and \overline{Mv}^a . The equation 4.3 is Mark-Houwink equation.

$$(4.3) \quad \eta = K \overline{Mv}^a$$

where :

η is the intrinsic viscosity, (deciliters/gram, dl./g.)

K and a are the constants for given polymer type, solvent, temperature, and have been determined and tabulated in the handbook of polymer. From the handbook of polymer, 'K' of PET in phenol/tetrachloroethane(40/60 by weight) at 25 °C is 1.4×10^5 dl./g. and 'a' is 0.64.

4.2 Polyurethane Manufacturing

The glycolized products from depolymerization of PET(in part 4.1) was used as the chain extender in polyurethane manufacturing. Polyurethane in this thesis was produced by batch experiment. The aims of the experiment was to produce polyurethane with three kinds of chain extender from depolymerization of PET (GP(1.5,4,190),GP(4.8,190), and GP(4.8,190) without EG) and then, to compare their mechanical properties with those of polyurethane with EG. The part of polyurethane manufacturing consisted of the following headings :

4.2.1 Chemicals and raw material preparation

4.2.2 Apparatus

4.2.3 Polymerization

4.2.4 Post curing

4.2.5 Mechanical properties analysis

4.2.1 Chemicals and raw material preparation

a. Polyol

Two kinds of polyols that are used in polyurethane manufacturing, are polyester polyols and polyether polyols. In this work polyester polyol was used to produce polyurethane elastomer. The polyester polyol was donated by Thai Polyurethane Industry Co.,Ltd.. The tradename of polyester polyol is F113. The specifications of F113 are presented in table 4.4 .

b. Diisocyanate

There are two types of diisocyanate that are commonly used in polyurethane manufacturing, TDI and MDI. This work used polymeric MDI. The polymeric MDI was supplied by Thai Polyurethane Industry Co. Ltd.. The tradename of polymeric MDI is Raypol C 900. The specifications of Raypol C 900 are presented in table 4.5.

Table 4.4 The specifications of polyester polyol (F113)

Specifications	Polyester polyol(F113)
Acid number(mg.KOH/g.)	0.5 - 0.8
Hydroxyl number (mg.KOH/g.)	54 -58
Viscosity at 60°C	1050 - 1200
Color (APHA)	<100
Density at 25°C	1.16
Water content (%)	<0.05

Table 4.5 The specification of polymeric MDI (Raypol C 900)

Specification	Polymeric MDI (Raypol C 900)
Physical state at room temperature	Liquid
Color	Fawn to dark brown
Odor	None to aromatic at room temperature
Density at 25 °C	1.24
Viscosity at 25 °C	200 -500
%Free NCO (by weight)	31.5
Flash point (°C)	> 200

c.Chain - extender

Four kinds of chain extender were used in polyurethane manufacturing. There were EG, GP(1.5,4,190), GP(4,8,190) and GP(4,8,190) without EG. The aim of the

experiment was to compare the properties of the polyurethane with different chain extenders.

GP(1.5,4,190)

GP(1.5,4,190) containing unreacted ethylene glycol was the glycolized product from that produced by depolymerization of PET. Depolymerization was run at temperature 190°C. The ratio of PET/EG was 1:1.5. The time for depolymerization was 4 hours. After depolymerization, GP(1.5,4,190) with unreacted ethylene glycol was taken to determine the hydroxyl value. The hydroxyl value was used to calculate the quantity of GP(1.5,4,190) to be added in the polyurethane manufacturing. In this work GP(1.5,4,190) was used in polyurethane manufacturing because the cost of the investment was cheap.

GP(4,8,190)

GP(4,8,190) was the glycolized product that was depolymerized at temperature 190°C for 8 hours. The PET/EG ratio was 1:4. The cost of GP(4,8,190) which containing more EG was more expensive than GP(1.5,4,190), but hydroxyl value of GP(4,8,190) was higher than GP(1.5,4,190). The constituents of the glycolized product from depolymerization were the oligomer of PET such as, BHET, dimer, trimer and unreacted ethylene glycol mixed with the oligomer.

GP(4,8,190) without EG

GP(4,8,190) was the glycolized product that was depolymerized at temperature 190°C for 8 hours. The PET/EG ratio was 1:4. From depolymerization, the glycolized product consisted of the PET oligomer and unreacted ethylene glycol. This chain extender was prepared by removing EG from GP(4,8,190). For depolymerization of 96 g. of PET, the extraction used 5000 ml. of water. The glycolized product from depolymerization was poured into water, then the mixture was vigorously stirred and the mixture was set still for 48 hours. After 48 hours, the mixture was filtered. The solid glycolized product obtained was GP(4,8,190) without ethylene glycol which was taken to dry in the vacuum oven at temperature 60°C for 120 hours. The product was taken to determination the hydroxyl value.

Ethylene glycol

Ethylene glycol was the chain extender to be used as reference for with the chain extenders from depolymeriation in mechanical properties comparison. The chemical grade of ethylene glycol was purchased from the market. The structure and specifications of ethylene glycol are presented in figure 4.3 and table 4.6 .

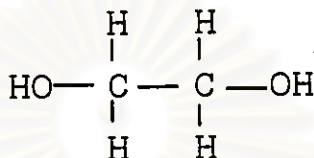


Figure 4.3 Structure of ethylene glycol

Table 4.6 Specification of ethylene glycol

Specifications	Ethylene glycol
Boiling range (95 % min.) (°C)	195 to 199
Density at temperature 20° C (g./ml.)	1.112 to 1.115
Reflective index at temperature 20 °C	1.434 to 1.432
Maximum Limit of impurities(%)	
Sulphated Ash	0.01
Acidity	0.1 mil. H
Sulphate (SO ₄)	0.005
Total chlorine	0.006
Molecular weight	62.07
Functionality	2
Hydroxyl Value (mg. KOH / g.)	1801

4.2.2 Apparatus

The apparatus for polyurethane manufacturing consisted of the following unit :

- a. mechanical stirrer
- b. beaker 250 ml.
- c. aluminum mold
- d. vacuum oven
- e. hot plate

4.2.3 Polymerization

In this part of the experiment, four different kinds of chain extender : GP(1.5,4,190), GP(4,8,190), GP(4,8,190) without EG, EG were used in polyurethane manufacture. When EG was used, the process is as follow. At first, the polyol was preheat at 100 ° C to remove moisture. EG was added into the polyol when the polyol was at the room temperature. It was followed by adding MDI to polyol mixture. Polyol mixture and MDI was mixed for 6 minutes.

After mixing, the liquid polyurethane was poured onto aluminum mold which was coated with silicone. The quantity of the polyol,MDI and the chain extender used in polyurethane mixing was demonstrated in table 4.7,table 4.8,table 4.9 and table 4.10.

With either GP(1.5,4,190), GP(4,8,190) or GP(4,8,190) without EG was used as chain-extender, it was added in the polyol when the polyol was heated at 190 °C. MDI was added in the polyol mixture after the temperature of the mixture was cooled to 140 °C. The polyol mixture and MDI were mixed for a few seconds, the liquid polyurethane obtained was poured into as describe above.

Before polyurethane manufacture, all the reactants , i.e. , polyol , MDI , and chain-extender, were placed in the vacuum oven maintaining at 60 °C in order to remove excess moisture.

4.2.4 Post curing

The finished polyurethane was release out of the mold. In order to complete the cross-linking, polyurethane was placed in the oven to at 80 °C for 24 hours.

4.2.5 Properties Analysis

4.2.5.A Hardness

Hardness is defined as the resistance of a material to deformation, particularly permanent deformation, indentation or scratching. The hardness testing method was carried out using a Durometer Instrument test which complied with ASTM D2240 (Shore or Durometer hardness). There are three types of pin-shaped indentors that give measurement in A,B,C,D,DO,O and OO scales as shown in figure 4.5. The A scale was used for this study. The thickness of specimen was 6 mm. The testing took place at the standard condition of 23 ± 2 °C and 50 ± 5 % relative humidity. The hardness measurement was performed at 8 positions on specimen surface. The average values were reported. Figure 4.5 shows the Durometer, indenter and spring force combination.

4.2.5.B Tensile test

The tensile properties are mechanical properties. The mechanical properties are the reactions that result when a force or a load is applied to a material. The tensile properties are found by measuring of the ability of polyurethane elastomer to withstand pulling stress. The tensile specimens were prepared by cutting the finished polyurethane in the dumbbell shape. The shape and dimension of the specimen for tensile test are show in figure 4.4 and table 4.11.

Table 4.7 Molar ratio and quantity of reactants
with EG as chain-extender at NCO/OH ratio 1:1

Molar ratio	Quantity of polyol : MDI : EG(pbw)
1 : 2 : 1	100 : 27.48 : 3.11
1 : 3 : 2	100 : 41.23 : 6.22
1 : 4 : 3	100 : 54.96 : 9.33
1 : 5 : 4	100 : 68.70 : 12.43
1 : 6 : 5	100 : 82.44 : 15.54

Table 4.8 Molar ratio and quantity of reactants
with GP(1.5,4,190) as chain-extender at NCO/OH ratio 1:1

Molar ratio	Quantity of polyol : MDI : GP(1.5,4,190) (pbw)
1 : 2 : 1	100 : 27.48 : 9.95
1 : 3 : 2	100 : 41.23 : 19.90
1 : 4 : 3	100 : 54.96 : 28.84
1 : 5 : 4	100 : 68.70 : 39.80
1 : 6 : 5	100 : 82.44 : 49.75

Table 4.9 Molar ratio and quantity of reactants
with GP(4,8,190) as chain-extender at NCO/OH ratio 1:1

Molar ratio	Quantity of polyol : MDI : GP(1.5,4,190) (pbw)
1 : 2 : 1	100 : 27.48 : 3.75
1 : 3 : 2	100 : 41.23 : 7.5
1 : 4 : 3	100 : 54.96 : 11.25
1 : 5 : 4	100 : 68.70 : 15.00
1 : 6 : 5	100 : 82.44 : 18.75

Table 4.10 Molar ratio and quantity of the polyurethane
with GP(4,8,190) without EG as chain-extender at NCO/OH ratio 1:1

Molar ratio	Quantity of polyol : MDI : GP(1.5,4,190) without EG (pbw)
1 : 2 : 1	100 : 27.48 : 11.38
1 : 3 : 2	100 : 41.23 : 22.76
1 : 4 : 3	100 : 54.96 : 34.14
1 : 5 : 4	100 : 68.70 : 45.52
1 : 6 : 5	100 : 82.44 : 56.90

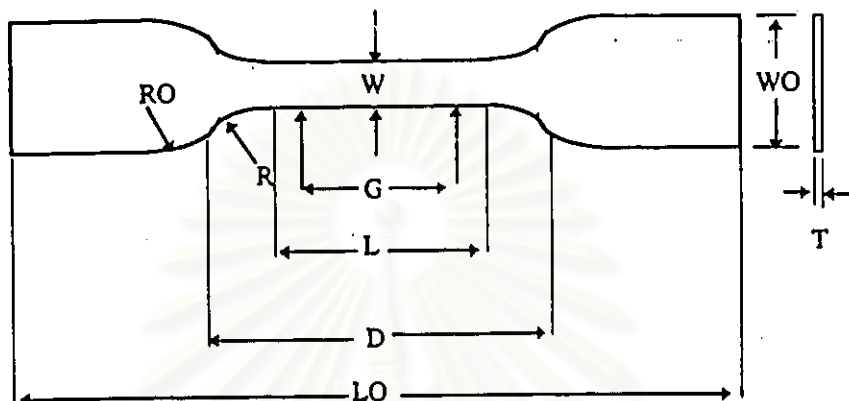


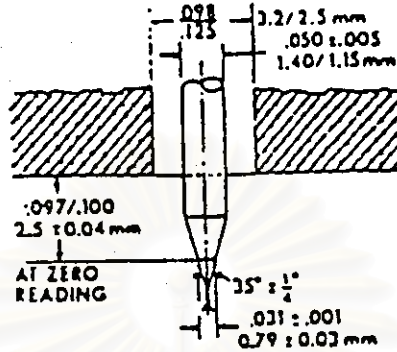
Figure 4.4 The illustration of the shape and the dimension of the tensile specimen

Table 4.11 The dimension of the specimen
(ASTM standard D638 type IV)

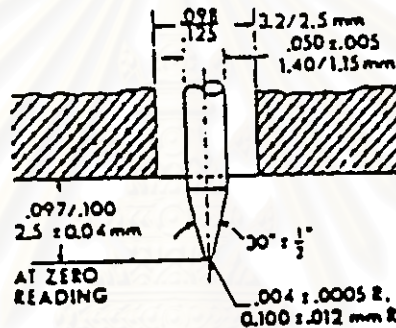
Dimension(see drawing)	ASTM standard D638 type IV
	in. (mm.)
W - Width Of narrow section	0.25(6)
L - Length of narrow section	1.30(33)
WO - Width over-all , min.	0.75(19)
LO - Length over-all , min	4.5(115)
G - Gage length	1.00(25)
D - Distance between grips	2.5(64)

NOTE 2—Durometer scale comparison chart only. This is not and cannot be used as a conversion reference.

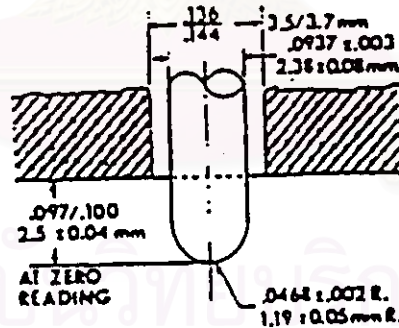
Type A	10	20	30	40	50	60	70	80	90	100
Type B	10	20	30	40	50	60	70	80	90	100
Type C	10	20	30	40	50	60	70	80	90	100
Type D	10	20	30	40	50	60	70	80	90	100
Type DO	10	20	30	40	50	60	70	80	90	100
Type O	10	20	30	40	50	60	70	80	90	100
Type OO	10	20	30	40	50	60	70	80	90	100



(a) Indenter for Type A and C Durometers



(b) Indenter for Type B and D Durometers



(c) Indenter for Type DO, O, and OO Durometers

NOTE—Spring Force Combinations:

$$\text{Force, } N = 0.550 + 0.075 H_a$$

where H_a = hardness reading on Type A, B and O durometers.

$$\text{Force, } N = 0.4445 H_c$$

where H_c = hardness reading on Type C, D and DO durometers.

$$\text{Force, } N = 0.203 + 0.00908 H_{oo}$$

where H_{oo} = hardness reading on Type OO durometers.

Figure 4.5 Durometer, indenter and spring force combination