

# The Effect of Ethylene Glycol to Soy Polyurethane Foam Classifications

Flora Elvistia Firdaus

**Abstract**—Soy polyol obtained from hydroxylation of soy epoxide with ethylene glycol were prepared as pre-polyurethane. The two step process method were applied in the polyurethane synthesis. The blending of soy polyol with synthetic polyol then simultaneously carried out to TDI (2,4): MDI (4,4') (80:20), blowing agent, and surfactant. Ethylene glycol were not taking part in the polyurethane synthesis. The inclusion of ethylene glycol were used as a control. Characterization of polyurethane foam through impact resilience, indentation deflection, and density can visualize the polyurethane classifications.

**Keywords**—Ethylene glycol, polyurethane foam, soy polyol, synthetic polyol

## I. INTRODUCTION

THE production of polyurethane foams is based on the reaction of organic isocyanates with polyols. Both starting materials are mostly derived from petrochemical resources. As natural resources become scarce, many researchers are beginning to investigate and utilize various renewable resources; the abundant and cheap vegetable oils [1]-[2].

In polymer industry, vegetable oils have been utilized as an alternative feedstock for monomers. Oils such as soybean oil, castor oil, and rapeseed oil based polyols have been used to make polyurethane foams as well [3].

Vegetable oils have a number of excellent properties which can be utilized in producing valuable polymeric materials. The oils are characterized for their hydroxyl value and fatty acid composition. The modified oils have higher hydroxyl values and lower levels of unsaturated acids than regular unmodified oils. The modified, soybased, vegetable oil polyols can be incorporated as a replacement for conventional polyols, reacting with isocyanates to produce flexible slabstock polyurethane foams (PUF), elastomers and coatings [4]-[7].

Soybean is a triglyceride with two dominant fatty acids, linoleic acids SBO is a triglyceride with two dominant fatty acids, linoleic acid (ca. 50%, two double bonds) and oleic acid (ca. 25%, one double bond), fielding an average number of 4.6 double bonds per molecule. The average molecular mass (MW) of SBO of American origin is 874 and despite variations in composition from molecule to molecule, it is virtually monodisperse. ESO can be prepared by the epoxidation of SBO using peroxyacetic acid generated in situ in the presence of sulphuric acid as the catalyst [8].

Standard industrial production is based on in-situ epoxidation, in which peracid is generated by reacting acetic or formic acid with hydrogen peroxide in the presence of strong mineral acids such as  $\text{H}_2\text{SO}_4$  and  $\text{H}_3\text{PO}_4$  [9]-[10].

Typical conversions of double bonds to epoxy groups are about 90% owing to the partial consumption of epoxy groups in side reactions.

It has been reported that epoxidized soybean oil alone when used in a 50:50 blend with a petroleum based polyol had similar density versus compressive strength properties with foams made from 100% petroleum based polyol [11]. Tu *et. al* reported that rigid polyurethane foams are formed by the reaction of alcohol with two or more reactive hydrogen functional groups per molecule (diols or polyols) and isocyanates group per molecule [12]. And Guo and Javni reported that rigid foam formulation typically requires a high hydroxyl value (OH number) in the 400-500 mgr KOH/gr range to obtain the necessary rigidity [13] or otherwise reduction of oxirane number.

The objectives of this study were to evaluate an approach for preparing a polyurethane foam having a good Impact Resilience, Indentation Force Deflection (IFD) polyol, density which were characterized into flexible polyurethane.

## II. EXPERIMENTAL PROCEDURES

### A. Materials

The refined soybean oil was provided by Sione darby edible products Ltd, Singapore, Iodine Value 53.89 gr Iod/100 gr sample with viscosity at 27°C is 44,3007 cp. Epoxidized soybean oil was prepared according to our previous work with oxirane number 4.7 mgr KOH/gr under optimized conditions with temperature of 70°C and for 90 minutes of reaction. Glacial acetic acid (96%), sodium carbonate, sodium hydroxide, hydrogen peroxide (30%), sulphuric acid, ethylene glycol, ethanol, acetic anhydride in pyridine, o-phosphoric acid 85%. All were purchased from PT. Harum Kimia Jakarta.

### B. Methods

A two step process was applied for the preparation of soybean oil based polyol; an intermediate product for polyurethane synthesis. Ethylene glycol (EG) is with the reaction from epoxide to polyol. Ethylene glycol inclusion in the polyurethane synthesis were used as a control. Generally, several studies have reported the use of chain extension in the reaction has much successful in forming hard segments of polyurethane.

The first step of the reaction is to react the unsaturated fatty acids in triglycerides with peracetic acid to soy epoxide using sulphuric acid as the catalyst. In the second step the epoxides were converted into polyols using o-phosphoric acid 85% as the catalyst. The reaction has made triglyceride transformed into oxirane ring.

According to Wang, directly reacting diisocyanate and polyol were still much doubt because generally still has very poor physical properties and often does not exhibit microphase separation [14].

Flora Elvistia Firdaus is with the Department of Chemical Engineering, Jayabaya University, Jl. Pulomas Kav 23 Jakarta-13210 Indonesia (phone: +62218583591; fax: +62218583591; (e-mail: flora\_elvistia@yahoo.com).

### C. Preparation of Polyol

The polyol synthesizing was made by mixing epoxide to ethylene glycol in stoichiometric calculations. The ratio of was established from the previous work of epoxide/ethylene glycol was at 1:1; 1:5; 1:7 and 1:9 (mol/mol) under three temperature reactions which are 50,60, and 70°C.

The experiments were carried out in a 500 ml three necked round bottom flask which where equipped with a thermometer, and mechanical stirrer. The whole apparatus was placed in a waterbath to maintain the temperature at range  $110 \pm 5^\circ\text{C}$  which measured inside the flask. The obtained product was allowed to cool to room temperature.

The viscosity of the sample polyol were 560.431 cp was measured at  $50^\circ\text{C}$  using viscometer Ostwald.

TABLE I  
SOY POLYOL FORMULATION

Symbol of sample	Epoxide/EG (mol/mol)	H <sub>3</sub> PO <sub>4</sub> 85% (v/v)	Temp. C	Acid number of polyol (mgr KOH/gr)
1	1:01	0.145	50	2.9
2	1:01	0.145	60	2.9
3	1:01	0.145	70	2.2
4	1:05	0.231	50	2.9
5	1:05	0.231	60	2.2
6	1:05	0.231	70	1.8
7	1:07	0.274	50	2.2
8	1:07	0.274	60	2.2
9	1:07	0.274	70	1.8
10	1:09	0.316	50	2.2
11	1:09	0.316	60	2.2
12	1:09	0.316	70	1.8

vol. of H<sub>3</sub>PO<sub>4</sub> is based on total volume of polyol

The acid number of polyol were determined at different temperature conditions which are 50,60, and 70°C. It was found the acid number with rising temperature lowered linearly the acid number. It was clear that all of these value gave a range of polyol and polyurethanes, which can impart to the final properties of polyurethane products. Otherwise, temperature conditions at 50°C mostly have higher density than 60 and 70°C for all concentrations of epoxide/EG, 1:1; 1:5, and 1:7 except for 1:9 (mol/mol).

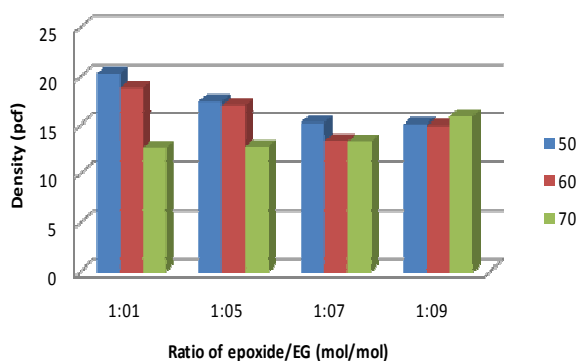


Fig. 1 Density of Polyurethane

Each bar representing the temperature reaction: 50,60, and 70°C

### D. Foaming

The foaming process is known as a free rise in molding. The polyurethane system freely expanded in horizontal direction were poured into the mold with volume of 300 ml.

The typical formulation of the polyurethane synthesis were made in twelve formulas which then exist as Polyurethane foam (PUF); PUF1 to PUF12.

Toluene diisocyanate (TDI) with 4,4: Methylene diamine isocyanate (MDI) (70:30) 5 ml, synthetic polyol 20 ml, soy polyol 5 ml, and EG 0 ml.

One formula was used as a control, the concentration of EG was 4 ml, synthetic polyol was 0 ml, and soy polyol was 8 ml.

The characterization of polyurethane foam were observed by its elasticity and firmness. Tests were applied to the foam which are (a) Impact Resilience (IR, ball rebound) is a measure of elasticity, bounce, or springiness of foam and is expressed as % of return, or % resilience (b) Indentation Force Deflection (IFD), is one of the test method to determine load bearing capacity; firmness or stiffness, and is expressed in pounds/inch<sup>2</sup>.

TABLE II  
POLYURETHANE FOAM ELASTICITY

Symbol of sample	Initial foam height (cm)	Foam Height after loading (cm)	Elasticity (%)
PUF1	4	0.4	90
PUF2	3.5	0.2	94.3
PUF3	4.5	0.6	86.7
PUF4	4	0.4	90
PUF5	4.5	0.8	82.2
PUF6	3	0.4	86.66
PUF7	6.5	0.8	87.69
PUF8	4.5	0.5	88.88
PUF9	4	0.4	90
PUF10	NA	NA	0
PUF11	5	0.7	86
PUF12	4	0.3	92.5

The polyurethane PUF 10 is found to be the formula can be neglected because the foams were not rising perfectly, so the elasticity of the foam were 0%. This property is related to the foam firmness which was the ability the foam to reform to its original height.

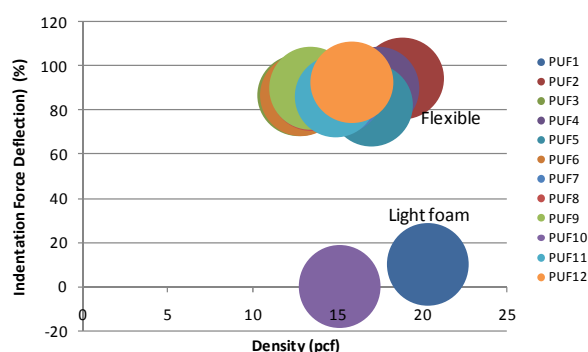


Fig. 2 Polyurethane classification by IFD test

Classification of polyurethane foam from twelve formulas above can be determine through a combination of its IFD and IR test. It is found that PUF 5 and PUF 10 were categorized into light PUF, otherwise PUF1, PUF2, PUF3, PUF4, PUF6, PUF7, PUF8, PUF9, PUF11, and PUF12 were flexible.

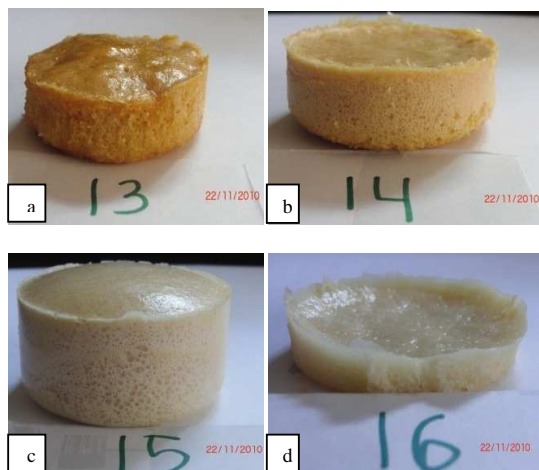


Fig. 2 Differentiation ingredients of Ethylene glycol in Polyurethane synthesis

a.Soypolyol with ethylene glycol b.Mixed soy-synthetic polyol c.Soy polyol without ethylene glycol d. Synthetic polyol

The formula use as a control was based on ethylene glycol inclusion into polyurethane synthesis. This can be a challenge into the future work because it replaced 100% of the synthetic polyol which means 0 ml of synthetic in the formula to the combination with soy polyol, also result a flexible polyurethane foam.

### III. CONCLUSION

Overall, temperature reaction in the epoxide hydroxylation contributes to the ultimate properties of polyurethane foam products. The Ethylene glycol inclusion in the polyurethane synthesis can be a desirable formula which can replace the used of synthetic polyol.

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### REFERENCES

- [1] A. Guo, Y.J Cho, Z.S. Petrovic, Structure and properties of halogenated and non halogenated soy based polyols. *J Polym Sci Pol Chem*, 2000, 38:3900-3910;
- [2] Y.H Hu, Y. Gao, D.N. Wang, C.P. Hu, S. Zu, L. Vanoverloop, D. Randall, Rigid polyurethane foam prepared from rapeseed oil based polyol. *J Appl Polym Sci*, 2002, 84:591-597
- [3] A.Guo, I.Javni, Z. Petrovic, Rigid polyurethane foams based on soybean oil. *J Appl Polym Sc*, 2000, 77:467-473)
- [4] J.John, B. Mrinal and B.T. Robert, *J. Appl Polym Sci.*, 86, 2002, p. 3097
- [5] I. Javni, Z.S. Petrovic, A. Guo and R. Fuller, *J. Appl. Poly. Sci.*, 77, 2000, p. 1723
- [6] P. Tran, D. Graiver and Narayan, *J. Am. Oil Chem. Soc.*, 82, 2005, p.653
- [7] M. He, X. Wang and J. Liu, *Chinese J. Appl. Chem.* 15, 1998, p. 117
- [8] Z.S. Petrovic, I. Javni and A. Guo, *Proc. Polyurethanes EXPO'98*, SPI, Polyurethane Divisions, Dallas, TX, 1998, p.559
- [9] Z.S. Petrovic, A. Zlatanic, C.C. Lava, and S. Sinadinovic- Fiser, Epoxidation of Soybean Oil in Toluene with Peroxoacetic and Peroformic Acids- Kinetics and Side reactions, *Eur. J. Lipid Sci. Technol.* 1014: 2002, pp. 293-299
- [10] A.E., Gerbase, J.R. Gregorio, M.Martinelli, M.C. Brasil, and A.N.F. Mendes, Epoxidation of Soybean by the Methyltrioxorhenium-CH<sub>2</sub>Cl<sub>2</sub>/H<sub>2</sub>O<sub>2</sub> Catalytic Biphaseic System, *J.Am Oil Chem.Soc.* 79: 2002, pp. 179-181
- [11] C.S. Lee, T.L. Ooi, C. H. Chuah, S. Ahmad, *Journal of the American Oil Chemist Society*, 84, 2007, p.1161
- [12] Y.C. Tu. P.P. Kiatsimkul, G.J.Suppes, F.H. Hsieh, *Journal of Applied polymer Science*, 105, 2007, p.453
- [13] A. Guo, I. Javni, Z. Petrovic, *Journal of Applied polymer Science*, 77, 2001, p.467
- [14] F.Wang Polydimethylsiloxane modification of segmented thermoplastic polyurethanes and polyureas, *Dissertation*, Virginia polytechnic institute and state university, 1998, p.14