

# Characterization of Polyol Synthesized from Epoxidized Palm Oil Using Fourier Transform Infra-Red and Nuclear Magnetic Resonance

W. N. Wan Tajulruddin<sup>a</sup>, A. R. Rahmat<sup>\*b</sup>, Z. A. Mohd Yusof<sup>c</sup>, A. Fakhari<sup>d</sup>

Polymer Engineering Department, Faculty of Chemical Engineering, Universiti Teknologi Malaysia, 81310 Johor, Malaysia

<sup>a</sup>wnhayati87@gmail.com, <sup>\*b</sup>k-razak@cheme.utm.my, <sup>c</sup>zainalabidinm@unikl.edu.my,

<sup>d</sup>alirezafakhari26@yahoo.com

**Abstract** – In this present study, ring opening polymerization (ROP) method has been used to prepare a bio-based polyol (PEPO) from epoxidized palm oil (EPO). This reaction was carried out in the presence of fluoroantimonic acid hexahydrate (HSbF<sub>6</sub>·6H<sub>2</sub>O) as a catalyst at certain concentration. The synthesized polyol was characterized with regard to its structural properties by fourier transform infra-red (FTIR) and nuclear magnetic resonance (NMR). The findings of this study revealed that the epoxy group presents in epoxidized palm oil (EPO) at 833 cm<sup>-1</sup> disappeared in the synthesized polyol while the hydroxyl group presents with the emergence of peak at 3522 cm<sup>-1</sup> in the FTIR spectrum. Based on NMR, the disappearance of epoxy ring group at 2.8-3.0 ppm indicates the completely reacted oxirane ring with the presence of signal at 3.7 ppm corresponded to hydroxyl group. **Copyright © 2015 Penerbit Akademia Baru - All rights reserved.**

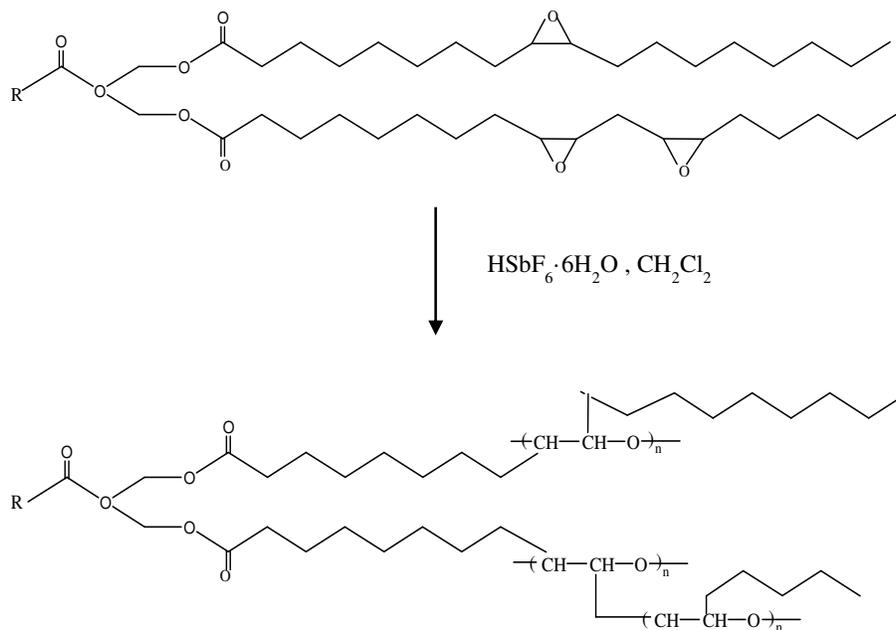
**Keywords:** Polyol, Epoxidized Palm Oil, Ring Opening Polymerization

## 1.0 INTRODUCTION

Utilization of epoxidized vegetable oil has become a potential growth for polymer development in many industries parallel to green technology application. The sustainability of vegetable oil makes it suitable as a renewable source to be used as raw materials for green polymer. In vegetable oil, there are unsaturation sites present which can be chemically modified to a value added product by a complicated reaction called epoxidation. Normally, epoxidation of vegetable oil is carried out by using peracids such as m-chloroperbenzoic, performic, and peracetic which can be generated in situ [1]. This value added product will provide an oxirane ring which has a high reactivity that is important for the synthesis of various polymers such as glycols, stabilizer, and plasticizer including polyols [2].

Bio-based polyol is mostly obtained from vegetable oil via several different synthesis routes. Some of the most popular methods are glycerolysis, hydroformylation, amidification, hydroxylation and epoxidation followed by ring opening polymerization [3]. Generally, polyol is needed as building blocks for further polyurethane (PU) synthesis by a reaction with diisocyanate [4]. The obtained PU will then be applied in high performance elastomers including flexible foams, coatings and binders [5].

Based on previous studies, polyol from vegetable oil has been prepared using different methods and catalysts. Yahya et al. [6] applied hydroxylation method by using glycerol as hydroxyl source to prepare a palm oil based polyol. Besides, Lee and Lee [7] reported synthesizing polyols by reacting EPO with a series of dicarboxylic acid for PU production. Apart from that, the formation of polyol also can be done by using superacid as studied by Liu and Biswas [8]. In their work, the epoxidized soybean oil (ESO) was used as a starting material and reacted with superacid as catalyst. Therefore in this study, pertaining to EPO as a potential starting material, the polyol was synthesized using ring opening polymerization in the presence of  $\text{HSbF}_6 \cdot 6\text{H}_2\text{O}$ , a superacid as catalyst. The proposed reaction scheme is shown in Figure 1 below.



**Figure 1:** Synthesis of PEPO polymers scheme.

## 2.0 METHODOLOGY

### 2.1 Material

EPO with the range of 2.7 - 2.9 % of oxirane oxygen content (OOC), 1049 g/mol of molecular weight, 0.58 eq/100 g of iodine value was supplied by Budi Oil Sdn. Bhd. Fluoroantimonic acid hexahydrate ( $\text{HSbF}_6 \cdot 6\text{H}_2\text{O}$ ) was purchased from Sigma Aldrich Inc. Methylene chloride was obtained from Qrec Chemical Co.Ltd.

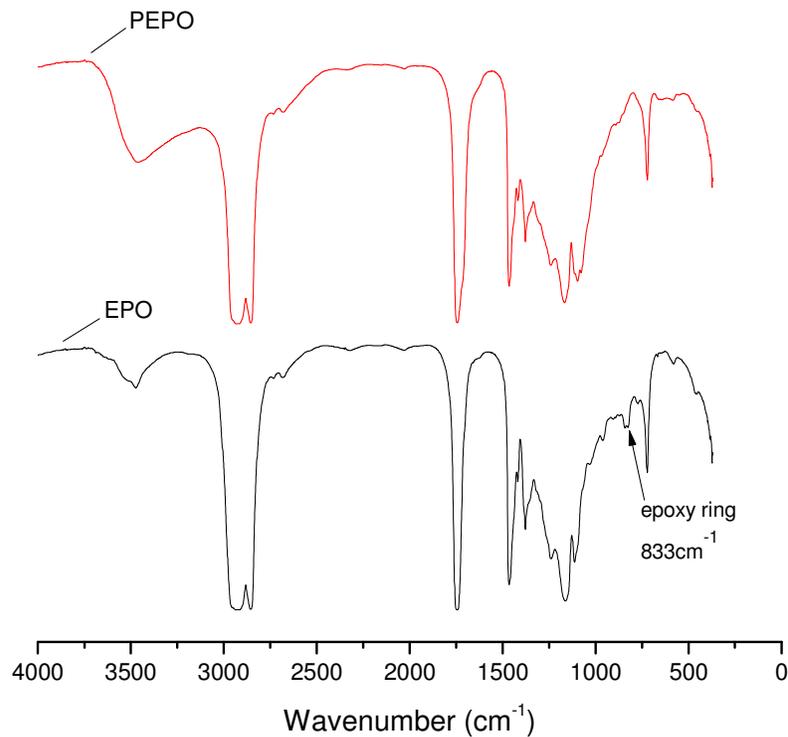
### 2.2 Sample Preparation

The sample was prepared by ring opening polymerization. In ring opening reaction, EPO was reacted with methylene chloride as solvent with the presence of fluoroantimonic acid hexahydrate as catalyst for three hours at room temperature with nitrogen flow to obtain polyol. Methylene chloride was removed by using rotary evaporator and the resulting product was dried in vacuum oven until constant weight was obtained.

### 3.0 RESULTS AND DISCUSSION

#### 3.1 FTIR Analysis

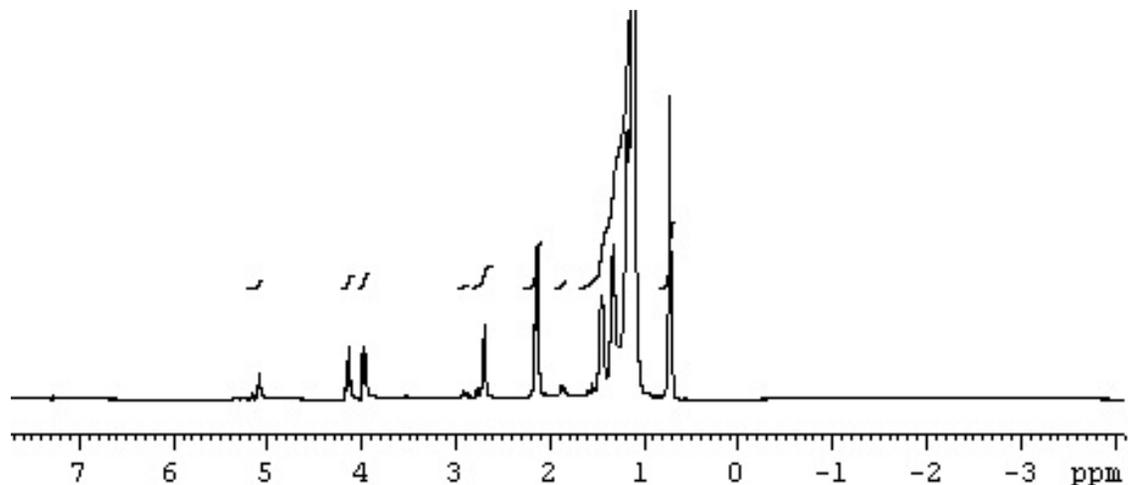
FTIR spectra of the unreacted EPO and PEPO are presented in Fig. 2. In comparison with the spectrum of EPO, the disappearance of epoxy groups at  $833\text{ cm}^{-1}$  shows that the opening of epoxy ring has occurred. Meanwhile, the appearance of the broad OH band at  $3522\text{ cm}^{-1}$  in PEPO is clearly observed. The formation of peak suggests that hydroxyl group was formed in the synthesized polyol.



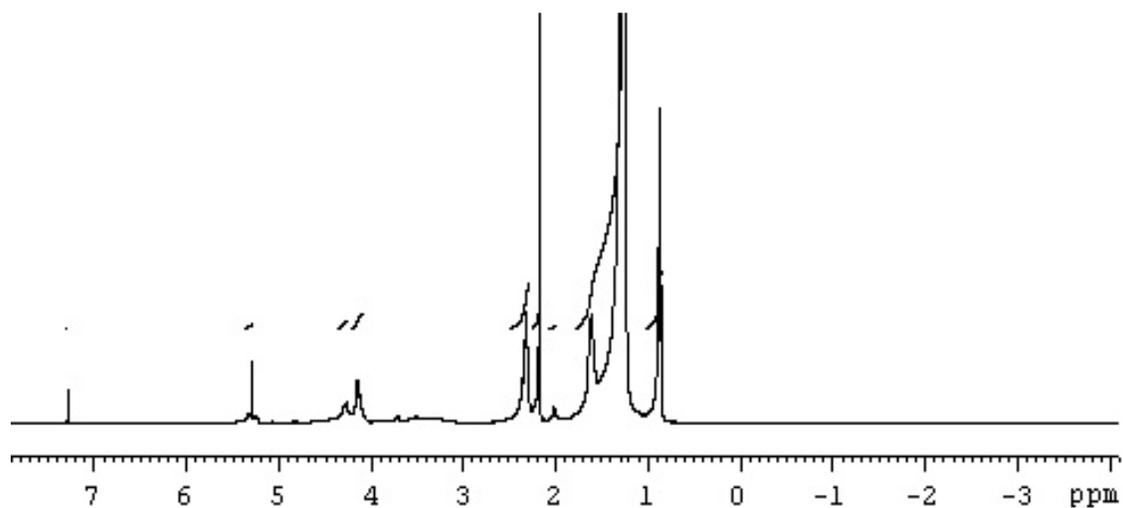
**Figure 2:** IR spectra for EPO and PEPO.

#### 3.2 NMR Analysis

NMR was done to confirm the formation of polyol. Figures 3 and 4 exhibit the  $^1\text{H}$ NMR spectrum of EPO and PEPO respectively. From Fig. 3, the highest peak at  $1.00 - 1.30\text{ ppm}$  belongs to the  $\text{CH}_2$  proton in the fatty acid moiety. The presence of epoxy ring was detected by the peak at  $2.8-3.0\text{ ppm}$ . Meanwhile, the peaks that appeared at  $2.7-2.9\text{ ppm}$  were corresponding to the proton adjacent to epoxy cyclic ring groups. Fig. 4 shows the  $^1\text{H}$ NMR spectrum of PEPO produced. The proton peaks at epoxy cyclic ring group at  $2.8-3.0\text{ ppm}$  disappeared and a new small signal at  $3.7\text{ ppm}$  has emerged which indicates the presence hydroxyl group of the opened epoxy ring.



**Figure 3:** <sup>1</sup>H NMR spectra of EPO.



**Figure 4:** <sup>1</sup>H NMR spectra of PEPO.

#### 4.0 CONCLUSION

Palm oil based polyol was synthesized from EPO by using the super acid, HSbF<sub>6</sub>·6H<sub>2</sub>O catalyst in methylene chloride solution by ring opening polymerization method. The material structural property and characteristic results of the obtained polyol were verified using FTIR and NMR. The analysis confirmed by the formation of hydroxyl group with the disappearance of epoxy group in PEPO shows that the reaction has successfully occurred.

**REFERENCES**

- [1] H. Patil and J. Waghmare, Catalyst for Epoxidation of Oil: A Review, *Discovery* 3 (2013) 10-14.
- [2] S. Vinay and P.P. Kundu, Addition Polymers from Natural Oils - A Review, *Progress in Polymer Science* 31 (2006) 983-100.
- [3] M. Desroches, M. Escouvois, R. Auvergne, S. Caillol and B. Boutevin, From Vegetable Oils To Polyurethanes: Synthetic Routes to Polyols And Main Industrial Products, *Polymer Reviews* 52 (2012) 1-96.
- [4] Z.S. Petrović, Polymers from Biological Oils, *Contemporary Materials I-1* (2010) 39-50.
- [5] C. Sylvain, D. Myriam, B. Gilles, L. Cédric, R. Rémi and B. Bernard, Synthesis of New Polyester Polyols from Epoxidized Vegetable Oils and Biobased Acids, *European Journal of Lipid Science and Technology* 114 (2012) 1447-1459.
- [6] S.M. Yahaya, A.F. Mohd and R. Mohamed, Synthesis and Characterization of Palm Oil Based Polyol, *Advanced Materials Research* 812 (2013) 275-280.
- [7] C.S. Lee and S.C. Lee, Preparation of Polyester Polyol from Epoxidized Palm Olein, *Chinese Journal of Chemistry* 29 (2011) 840-846.
- [8] Z. Liu and A. Biswas, Fluoroantimonic acid hexahydrate (HSbF<sub>6</sub>·6H<sub>2</sub>O) Catalysis: The Ring-Opening Polymerization of Epoxidized Soybean Oil, *Applied Catalysis A: General* 453 (2013) 370-375.