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

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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 ($HSbF_6{\cdot}6H_2O$) 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 present in epoxidized palm oil (EPO) at 833 cm^{-1} was disappeared in the synthesized polyol while the hydroxyl group present with the emergence of peak at 3522 cm^{-1} in the FTIR spectrum. Based on NMR, the disappearance of epoxy ring group at 2.8-3.0 ppm has indicated the completely reacted oxirane ring with the presence of signal at 3.7 ppm corresponded to hydroxyl group.*

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 material 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 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 diicocyanate [4]. The obtained PU will then be applied in high performance elastomers including flexible foams, coatings and binders [5].

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Based on previous studies, polyol from vegetable oil has been done on 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] have reported to synthesize 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) has been 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 $HSbF_6 \cdot 6H_2O$, a superacid as catalyst. The proposed reaction scheme is shown in Figure 1 as below.

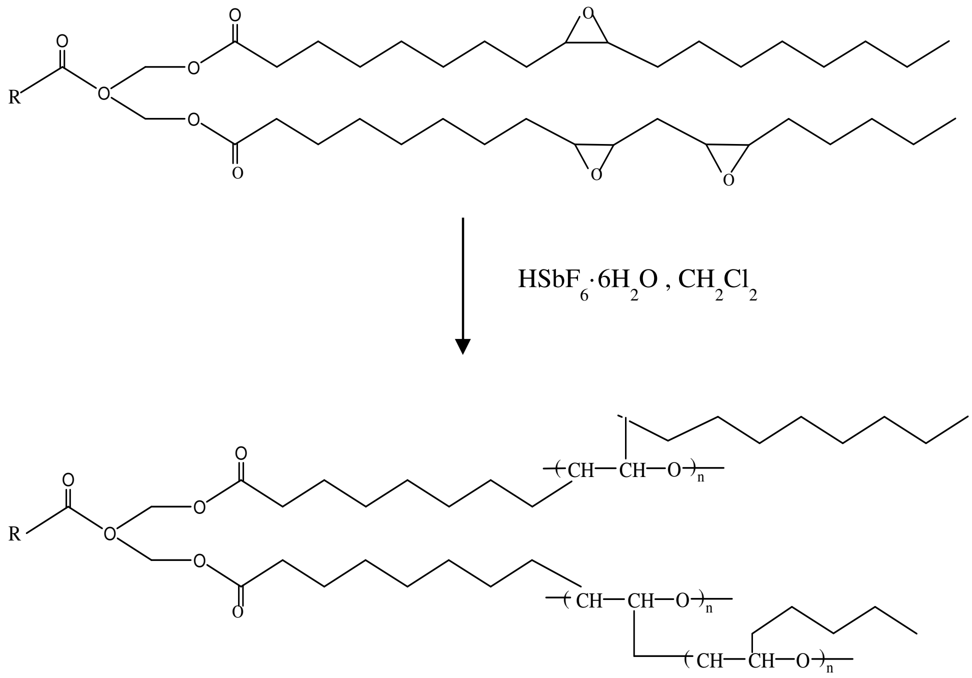

Figure 1: Synthesis of PEPO polymers scheme.

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 from Budi Oil Sdn. Bhd. Fluoroantimonic acid hexahydrate ($HSbF_6 \cdot 6H_2O$) 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 fluoroantimonic acid hexahydrate as catalyst for 3 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.

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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 cm^{-1} showed that the opening of epoxy ring has occurred. Meanwhile, the appearance of the broad OH band at 3522 cm^{-1} in PEPO is clearly observed. The formation of peak suggested that hydroxyl group was formed in the synthesized polyol.

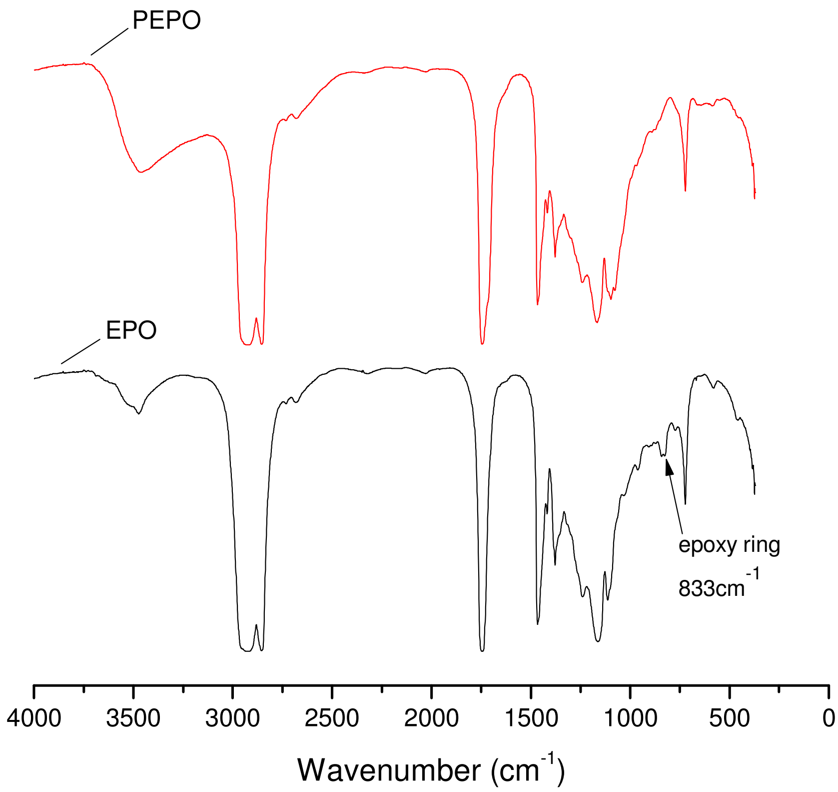

Figure 2: IR spectra for EPO and PEPO.

3.2 NMR Analysis

NMR was done to confirm the formation of polyol. Figures 3 and 4 exhibit the ^{1}HNMR spectrum of EPO and PEPO respectively. From Fig. 3, the highest peak at 1.00 – 1.30 ppm belongs to the CH_2 proton in the fatty acid moiety. The presence of epoxy ring was detected by the peaks at 2.8-3.0 ppm. Meanwhile, the peaks that appeared at 2.7-2.9 ppm were corresponding to the proton adjacent to epoxy cyclic ring groups. Fig. 4 shows the ^{1}HNMR spectrum of PEPO produced. The proton peaks at epoxy cyclic ring group at 2.8-3.0 ppm disappeared and a new small signal at 3.7 ppm has emerged which indicated the presence hydroxyl group of the opened epoxy ring.

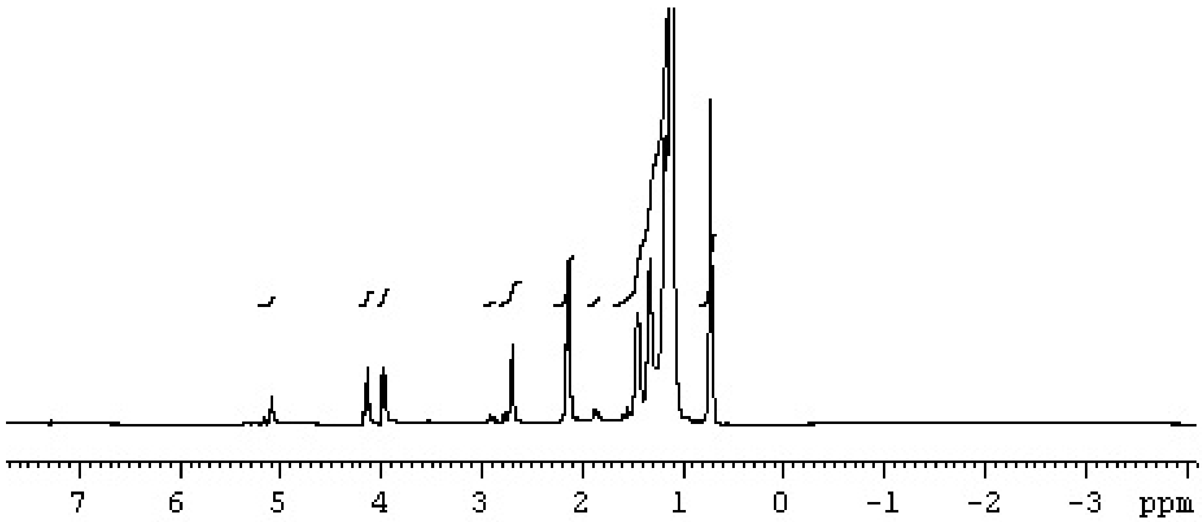

Figure 3: [1]HNMR spectra of EPO.

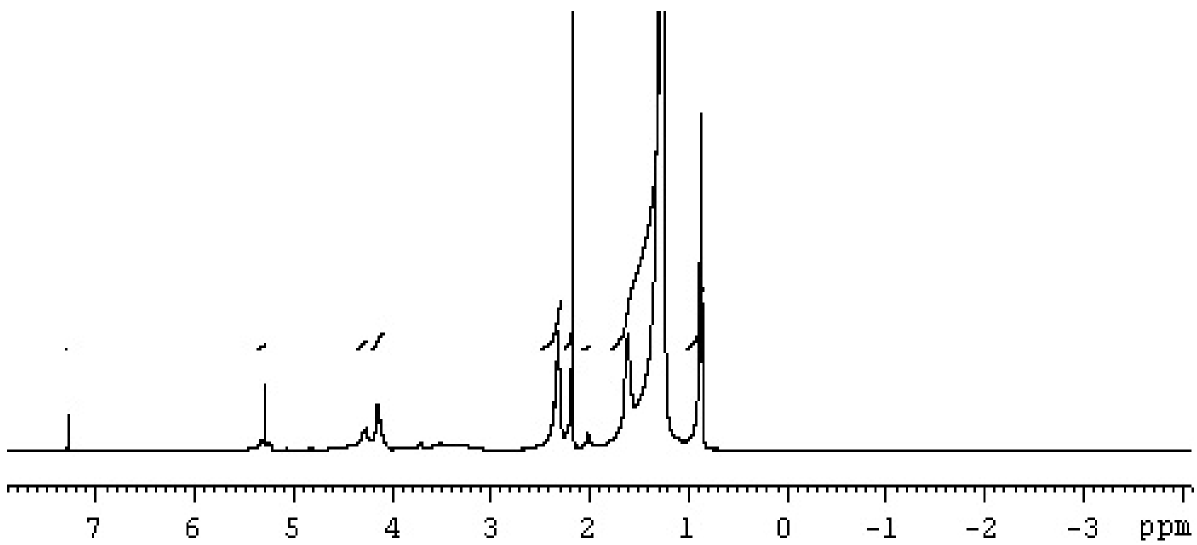

Figure 4: HNMR spectra of PEPO.

4.0 CONCLUSSION

Palm oil based polyol has been synthesized from EPO by using the super acid, $HSbF6\cdot6H_2O$ catalyst in methylene chloride solution by ring opening polymerization method. The material structural properties and characteristics 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.

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