MOLEKUL
eISSN: 2503-0310

Articles
https://doi.org/10.20884/1.jm.2026.21.1.12176

Optimization of the epoxidation reaction of *Calophyllum inophyllum* oil-based epoxy

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Received July 16, 2024; **Accepted** March 03, 2026; **Available** online March 20, 2026

ABSTRACT. Epoxy is synthesized through an epoxidation reaction, namely the oxidation of double bonds by active oxygen to form an oxirane ring. Generally, epoxy is synthesized from petroleum-derived raw materials which are non-renewable energy sources. Vegetable oils which contain unsaturated bonds can be used as alternative raw materials for producing epoxy. In this research, *Calophyllum inophyllum* oil (the local name is nyamplung), which is a non-edible and non-commercial vegetable oil, was used as a feedstock in the preparation of epoxy. Epoxidation takes place in situ, namely performic acid which reacts with nyamplung oil is produced from the reaction between HCOOH and H_2O_2, and is catalyzed by sulfuric acid simultaneously. The indicators in this epoxidation reaction are the oxirane oxygen content (OOC) and the relative conversion to oxirane (RCO). The optimum epoxidation conditions obtained are duration of epoxidation of 6 h, ratio of oil:HCOOH:H_2O_2 is 1:3:4 (mole/mole), and epoxidation temperature of 45 °C. The product was characterized using FTIR and NMR.

Keywords: *Calophyllum inophyllum* oil; epoxidation; epoxy; oxirane oxygen content

INTRODUCTION

Over the past few decades, globally, dependence on petroleum as a raw material for petrochemical products has increased. This high dependency on petroleum is not accompanied by sufficient availability and even tends to decline because petroleum is a non-renewable resource. In addition, the price of petroleum is unstable and its derivatives are not environmentally friendly. These conditions have caused, recently, research to focus on the production of oleochemicals as a substitute for petrochemicals. Vegetable oil is used as a substitute for petroleum and its derivatives because of its high biodegradability, abundant availability, and environmental friendliness (Freire et al., 2023; Sabnis & Kaikade, 2023). Conversion of vegetable oils into bio-fuels and oleochemicals usually goes through the processes of transesterification or alcoholysis, esterification, epoxidation and hydrotreatment (Foo et al., 2022). The largest component of vegetable oil is triacylglycerol (triglyceride), which is an ester of fatty acids and glycerol. The hydrocarbon chains of fatty acids that form triglycerides usually have 14 to 22 carbon chains. Unsaturated fatty acids from vegetable oils generally contain 1 to 3 unsaturated bonds. Modification of unsaturated bonds of fatty acid chains from vegetable oils can produce new chemicals or intermediate compounds or even monomers to produce polymers.

Epoxy is a cyclic ether called an oxirane ring. Epoxy which is synthesized from vegetable oil, bio-based epoxy, is usually used as a plasticizer and stabilizer in the polyvinyl chloride (PVC) industry (Cai et al., 2020; Yang et al., 2023). Modification of double bonds in vegetable oils is the most widely used method for producing epoxidized vegetable oils. Generally, epoxy is synthesized through a chemical epoxidation reaction, namely the oxidation reaction of double bonds in vegetable oils by peroxy acid. Organic acids act as oxygen acceptors, while those that act as oxygen donors are peroxides, usually the peroxide used is hydrogen peroxide. Peroxy acid, which is formed due to the reaction of organic acids with peroxide, will oxidize the double bonds in vegetable oil to form epoxy groups/ oxirane rings (Janković et al., 2020; Hernández-Cruz et al., 2021; Balakrishnan et al., 2023).

Currently, vegetable oil is the main choice as raw material for producing epoxy because vegetable oil is a renewable oil source, non-toxic and environmentally friendly (Feofilovs et al., 2023). Vegetable oils that have unsaturated bonds in their fatty acid chains, such as soybean, cottonseed, olive, palm, rapeseed, and sunflower oils were used commercially as feedstocks to produce epoxy (Marriam et al., 2023). However, these oils are used commercially as food ingredients, so replacing these oils with oils from plants that are not utilized, but whose oils contain high amounts of unsaturated fatty acids, must be investigated. One of them is *Calophyllum inophyllum* oil (hereinafter referred to as nyamplung oil), which is oil produced from the *Calophyllum inophyllum* tree that grows widely in the coastal region of West Nusa Tenggara, Indonesia. This oil has advantages compared to oils from other productive plants, such as *Areca catechu* L. (palm), *Helianthus annuus* L. (sunflower) and *Jatropha curcas* L, including long productivity, high oil content and high potential for survival in nature. *Calophyllum inophyllum* seeds contain oil of around 40-73% (Jain et al., 2018; Suhendra et al., 2019; Ponnusami & Arumugam, 2019). The fatty acids that make up nyamplung oil consist of unsaturated fatty acids, which are palmitoleic (0.1-0.2%), linolenic (0.2-0.3%), arachidic (0.8-0.9%), linoleic (24-25%) and oleic acid (46-47%), and saturated fatty acids, namely stearic (13-14%) and palmitic acid (14-15%) (Suhendra et al., 2019; Damanik et al., 2017). The data above shows that nyamplung oil can be used as a feedstock for producing epoxy, this is because its unsaturated fatty acid content is more than 70%.

The effectiveness of epoxidation of nyamplung oil is the main discussion in this article. This is due to, during the epoxidation process, the oxirane ring formed can undergo ring reopening. The reason is that the position of the three atoms in the oxirane ring is an equilateral triangle. This position causes the bonds between the three atoms to become tense, so that the three atoms become very reactive. This situation can cause the breaking of one of the bonds in the epoxy groups. The reopening of oxirane ring is lead to several factors, including the catalyst used, temperature, epoxidation duration, and the ratio of oil, peroxide and organic acids used (Ramírez et al., 2022; Meng et al., 2021; Azmi et al., 2022). This article explains several parameters that influence the epoxidation process, including the effect of epoxidation duration, the influence of ratio of H_2O_2:formic acid:nyamplung oil, and the effect of epoxidation temperature. The goal is to produce high percentage of RCO or efficiency of the epoxidation reaction.

EXPERIMENTAL SECTION

Materials

Extraction and purification of nyamplung oil is carried out using the procedures described in our previous article (Gunawan et al., 2023). *Calophyllum inophyllum* oil was extracted from *Calophyllum inophyllum* seeds using hexane as a solvent. All chemicals/ reagents used are produced by Merck, Germany which have high purity, analytical grade. The chemicals/ reagents were formic acid (98%), hydrobromic acid (47%), hydrogen peroxide (30%), crystal violet indicator, phenolphthalein indicator, sodium hydroxide, acetic acid glacial, methanol 98%, ethanol absolute, sodium chloride, Na_2SO_4 anhydrate, n-hexane, silica gel 60 G, hydrochloric acid and sodium bicarbonate.

Basic Epoxidation Procedure

The general procedure of epoxidation refers to our previous publication (Gunawan et al., 2023). Into a three-neck flask, put approximately 8 g of nyamplung oil. Then, organic acid (formic acid) and H_2O_2 were added drop by drop. Then the mixture was stirred at a speed of ±100 rpm and H_2SO_4 was added slowly. After the epoxidation process is complete, separation and purification of the product is carried out by transferring the mixture into a separating funnel and then adding $NaHCO_3$, NaCl and distilled water. Then, the product/epoxy formed is separated and purified. The purified epoxy is used for further processing, characterization and determination of the percentage of OOC and RCO.

The Epoxidation Optimization

The three main parameters of the epoxidation reaction that were optimized in this study were the ratio of nyamplung oil:organic acid:peroxide, epoxidation duration and temperature of epoxidation. The method used is one parameter at time, that is, one parameter is fixed while other parameters are varied. Variations in the ratio of the amount of nyamplung oil:formic acid:H_2O_2 were 1:7:2, 1:6:3, 1:5:4, 1:4:5, 1:3:6, and 1:2:7 (mole/ mole). The epoxidation temperatures varied were 35, 45, 55, and 65 °C. Meanwhile, the variations in epoxidation duration were 1, 2, 3, 4, 5, 6, and 7 hours.

Measurements

Important parameters in vegetable oil epoxidation are percentage of OOC, iodine value (IV) and percentage of RCO. IV is a quantity which indicates the number of unsaturated bonds in a fat or oil. Therefore, the breaking of double bonds in fats or oils is indicated by a decrease in the IV value. The IV value of the product/ epoxy and nyamplung oil is determined using ASTM D 5554-95 procedure. Based on this procedure, IV is calculated using the following equation:

$$IV = \frac{12.69 \; x \; N \; x \; (V_1 - V_2)}{W} \qquad (1)$$

where:

- 12.69 = conversion factor of $Na_2S_2O_3$ to the mass of iodine,
- N = normality of the $Na_2S_2O_3$ solution,
- V_1 = $Na_2S_2O_3$ volume (mL) for blank titration,
- V_2 = $Na_2S_2O_3$ volume (mL) for sample titration,
- W = sample weight (g).

Another parameter is the percentage of OOC, which is defined as the amount of oxirane rings formed during the epoxidation process. The OOC (%) is determined based on the AOCS Cd 9-57 method, namely titration of the HBr solution on the sample, and is calculated based on the following equation:

$$OOC\ (\%) = \frac{V \times N \times 1.6}{W} \qquad (2)$$

where W = weight of sample, N = HBr normality, and V = HBr volume.

Quality of the oxirane ring/ epoxy groups or efficiency of epoxidation process is expressed as a percentage of RCO, which was calculated using equation below (Rahman et al., 2023):

$$RCO\ (\%) = \frac{OOC_e}{OOC_p} \times 100 \quad (3)$$

where OOCe is experimental OOC, while OOCp is predicted OOC. The OOCp is calculated using the equation below,

$$OOC_p = \left[\frac{\left(\frac{IV_0}{2A_i}\right)}{100+\left(\frac{IV_0}{2A_i}\right)(A_0)}\right] \times A_0 \times 100 \quad (4)$$

where: A_0 = oxygen atomic weight, A_i = iodine atomic weight, and IV_0 = nyamplung oil IV.

Characterization of the product

Product characterization was carried out based on analysis of the NMR and FTIR spectra. NMR spectra (^{1}H and ^{13}C) were recorded using a 500 MHz Agilent DD2 NMR Spectrometer, Agilent Technologies, Inc. - United States. Meanwhile, the FTIR spectrum was recorded using a Frontier FTIR Spectrometer, PerkinElmer Inc. - United States.

RESULTS AND DISCUSSION

The Influence of Epoxidation Temperature

Epoxidation temperature has a major influence on the formation of oxirane ring. Temperature affects the oxidation process, namely the breaking of unsaturated bonds in oil followed by the binding of oxygen. Thus, the epoxidation temperature is also closely related to the percentages of OOC and RCO. At an epoxidation temperature of 35-45°C the OOC value increased, however, the OOC value tended to decrease as the epoxidation temperature increased (**Figure 1**). Increasing the epoxidation temperature causes increased collisions between reactant molecules. The performic acid formed as a result of this collision breaks the double bonds of nyamplung oil and then binds oxygen to form an oxirane ring/ epoxy group (Bohórquez et al., 2022). The addition of a catalyst, sulfuric acid, causes an exothermic reaction that can accelerate the epoxidation process (Bohórquez et al., 2020). Therefore, increasing the reaction temperature and adding a catalyst can reduce the OOC value due to increasing the overall reaction temperature. High temperatures and sulfuric acid catalysts can cause hydrolysis reactions in the oxirane ring or reopening of the oxirane ring. High temperature also causes a reduction in catalytic activity (Wai et al., 2019). In this study, the highest OOC value was obtained at an epoxidation temperature of 45 °C, therefore, it was used as an epoxidation temperature parameter in subsequent experiments.

The Influence of Ratio of H_2O_2 and Formic Acid (HCOOH)

Study on the influence of ratio of H_2O_2:HCOOH:nyamplung oil was carried out at a fixed concentration of nyamplung oil, while the concentration of HCOOH:H_2O_2 (mole/mole) varied (7:2; 6:3; 5:4; 4:5 ; 3:6 and 2:7). **Figure 2** shows that increasing H_2O_2 concentration and decreasing of the concentration of HCOOH causes an increase in the percentage of OOC. This is because H_2O_2 is an oxygen donor, so increasing the concentration of H_2O_2 also increases the amount of oxygen bound to the double bonds of the nyamplung oil thus formed epoxy group/ oxirane ring. Meanwhile, formic acid acts as an oxygen carrier. However, high concentrations of HCOOH can cause the oxirane ring to reopen and form undesirable hydroxyl or glycol compounds (Rahman et al., 2023).

Figure 2 also shows that the optimum concentration ratio of H_2O_2 and formic acid is obtained at a ratio of 6:3 or 2:1, therefore this ratio is used in subsequent experiments. This ratio is similar to the epoxidation study of methyl esters of Jatropha oil conducted by Derahman et al. (2019).

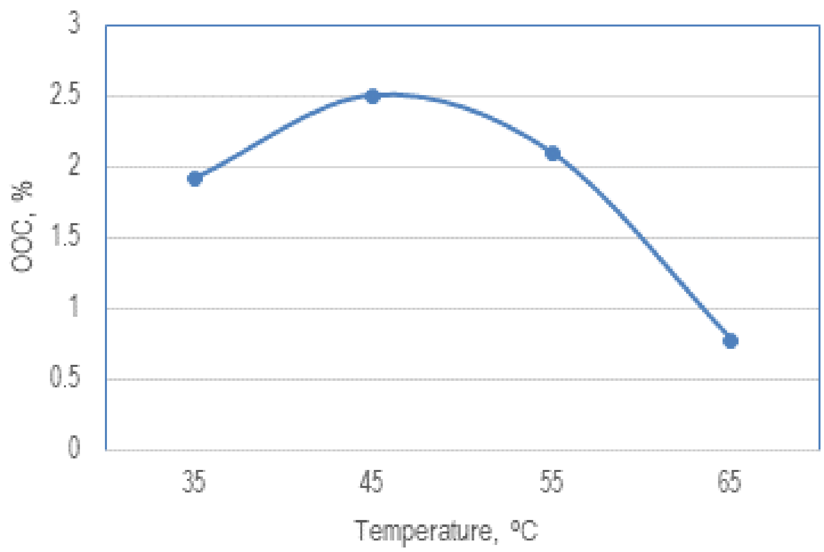

Figure 1. The OOC value (%) of epoxidation temperature effect (nyamplung oil : HCOOH:H_2O_2 is 1:5:4); duration of epoxidation is 2 hours)

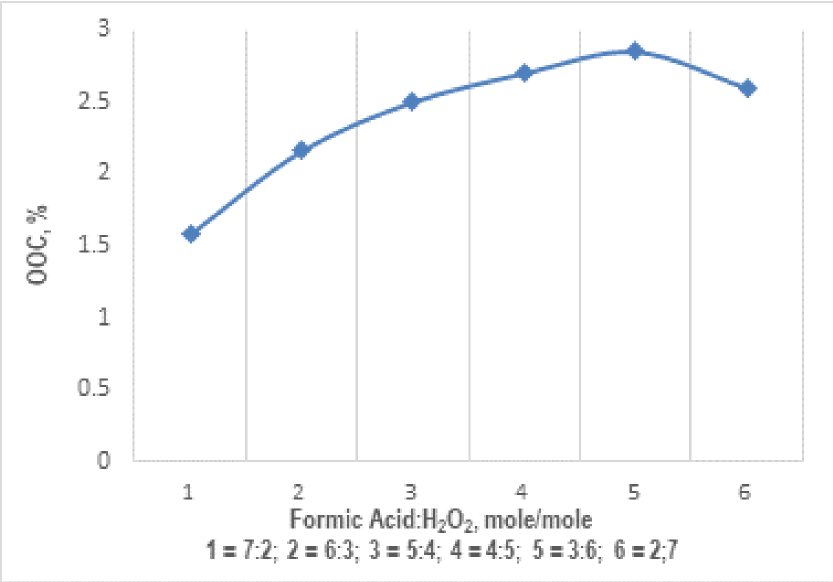

Figure 2. The OOC value (%) of influence of ratio of formic acid:H_2O_2 (epoxidation temperature of 45 °C; duration of epoxidation in 2 h)

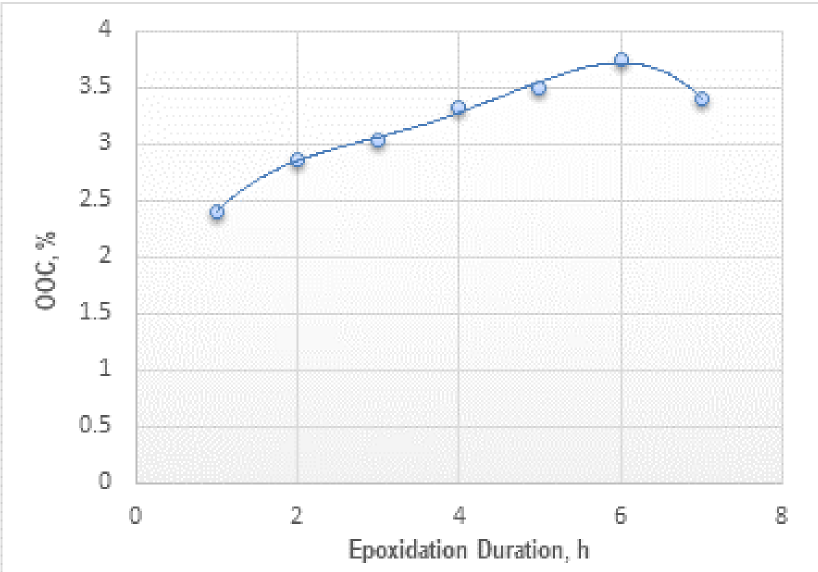

Figure 3. The OOC value (%) of influence of epoxidation duration (epoxidation temperature of 45 °C; ratio of formic acid:H_2O_2 is 1:2)

Influence of Epoxidation Duration

Figure 3 shows the OOC percentage which increases with increasing epoxidation duration. Increasing the epoxidation duration causes the opportunity for collisions between molecules to become greater, so that more double bonds break and turn into oxirane rings. The percentage OOC continues to increase until the epoxidation duration reaches 6 hours. After 6 hours, the percentage of OOC continued to decrease. Possible causes are (1) The reaction for the formation of performic acid is a reversible reaction (Farinelli et al., 2022), therefore increasing the epoxidation duration causes the formed peracetic acid to decompose again, (2) three-atom bonds or oxirane rings can undergo degradation/ reopening caused by long intermolecular collisions, (3) the formation of diols is a result of degradation or reopening of epoxy groups. **Figure 3** also shows that the epoxidation duration of 6 hours is the optimum epoxidation duration, therefore it is used as a parameter in subsequent experiments.

The optimum epoxidation duration of 6 hours in this research was longer than the epoxidation duration of tung oil, which was 4 hours, using the same catalyst, sulfuric acid (Budiyati et al., 2020). This difference in epoxidation duration is likely due to differences in the types and amounts of unsaturated fatty acids that make up the two oils. The unsaturated fatty acids in nyamplung oil are around 71% (Jain et al., 2018), whereas in tung oil it reaches 98% (Xu et al., 2017).

Meanwhile, the epoxidation reaction of *Ximenia americana* oil, which contains almost the same unsaturated fatty acids as nyamplung oil, has an epoxidation duration of around 4 hours, but produces a lower OOC percentage (Shagal et al., 2013). However, the epoxidation duration of 6 hours is better compared to research conducted by Kurańska & Niemiec (2020), which obtained an epoxidation duration of 7 hours for cooking oil epoxidation. They used the ion exchange resin Amberlite IR-120, a solid catalyst. In solid catalysts, the products formed can cover the pores of the catalyst. The closure of the catalyst pores causes the active site of the catalyst to be blocked, thereby slowing down the epoxidation rate (Wai et al., 2019).

Step Up of the Reaction

The step up reaction is to increase the amount of reactant 10 times. This step up stage is a simulation for a larger reaction scale, namely industrial scale. The optimum conditions obtained in the epoxidation optimization stage are used in this step up stage. The epoxidation conditions used are epoxidation duration of 6 hours, ratio of nyamplung oil:formic acid:H_2O_2 is 1:3:4 (mole/mole), and epoxidation temperature of 45ºC. The percentages of OOC and RCO of the product/ epoxy synthesized using the optimum epoxidation conditions were 3.7 and 76.8%, respectively. This percentage of RCO is better compared to the percentage of RCO of epoxidation of Jatropha oil, which is 69% (Hazmi et al., 2013).

The epoxidation reaction takes place in situ, that is, the entire epoxidation process runs simultaneously. The peroxy acid used is performic acid which is obtained by reacting a peroxide, H_2O_2, with an organic acid, HCOOH, and a sulfuric acid catalyst. The mechanism of the epoxidation reaction (**Figure 4**) begins with the reaction of peroxy acid formation. Furthermore, the peroxy acids formed oxidize unsaturated bonds in the oil to form epoxy groups.

Characteristic of the Product

Physical Properties

The physical properties measured were IV and viscosity of nyamplung oil and its products (**Table 1**). The IV of nyamplung oil is greater than the IV of its product (**Table 1**). This indicates that the unsaturated bonds in the oil have been broken and epoxy groups have been formed However, the product is semi-solid and the presence of product IV is an indication that the epoxidation process did not break all the double bonds. According to Shagal et al. (2013) in the epoxidation process, the reduction in the IV value of the oil is an indication that the oil double bonds have been oxidized and epoxy groups have been formed. However, reopening the formed oxirane ring did not change the IV value of the product. Therefore, to measure the epoxidation efficiency, the percentage of RCO is determined.

In addition to IV, another indicator that can be used to identify whether unsaturated bonds in oil have been broken during the epoxidation process is the ratio of the viscosity of the oil and its products. Viscosity is a measure of the thickness of a liquid. In vegetable oils, the more unsaturated bonds and the longer the carbon chains of the constituent fatty acids, the more dilute the oil. Table 1 shows that the viscosity of nyamplung oil is smaller than the viscosity of the product/epoxy. Increasing product viscosity during the epoxidation process is an indication that some of the unsaturated bonds in the oil have been broken. This is in accordance with the statement of Azmi et al (2022), the RCO value of the epoxidation process is directly proportional to the viscosity of its product.

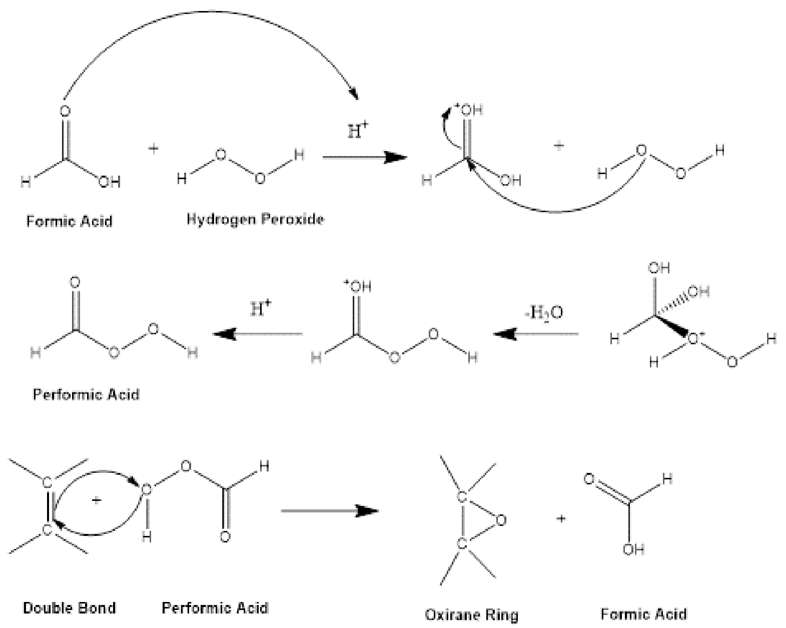

Figure 4. The mechanism reaction of epoxidation (Gunawan et al., 2023).

Table 1. Physical properties of the nyamplung oil and the product

Parameter	Value
IV of nyamplung oil, gram iodine/ 100 grams	81.22
IV of product, gram iodine/ 100 grams	13.32
Nyamplung oil viscosity, cP	15.75
Product viscosity, cP	30.56

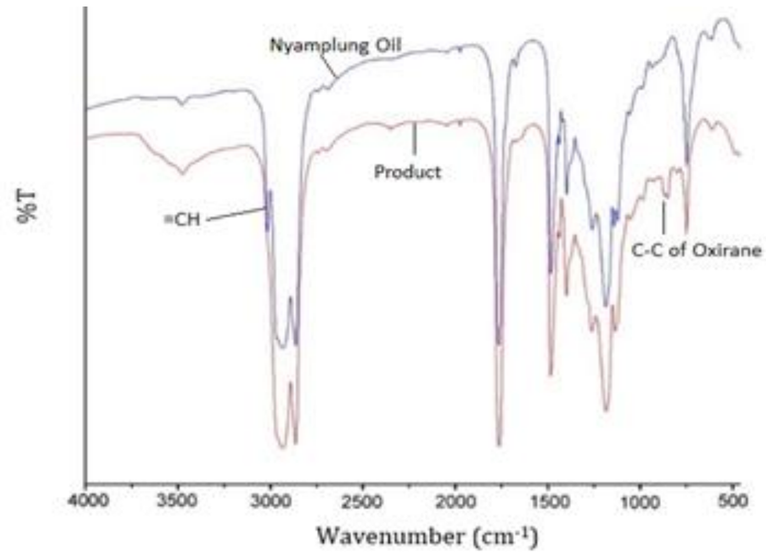

Figure 5. Infrared spectrum of nyamplung oil and its products

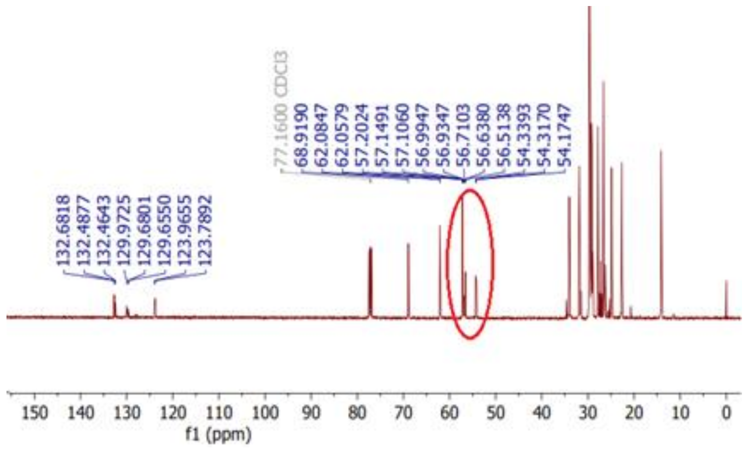

Figure 6. Spectrum of ^{13}C NMR of the product

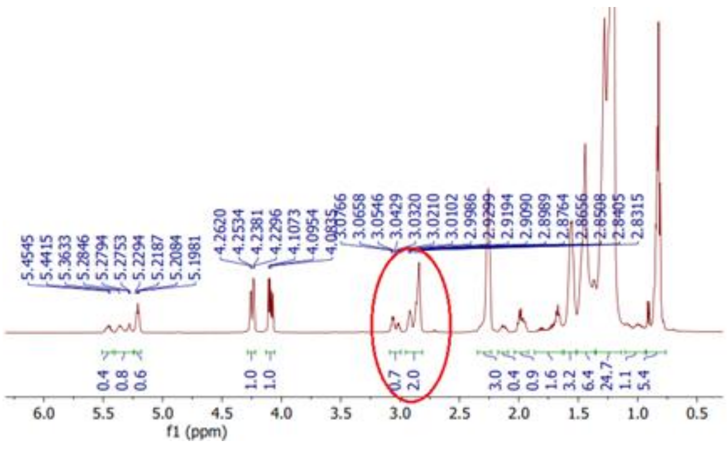

Figure 7. ^{1}H NMR Spectrum of the epoxy/ product

NMR and FTIR Spectrum Analysis

The nyamplung oil spectrum in **Figure 5** identifies the presence of absorptions at wave numbers 3007 cm^{-1} and 1655 cm^{-1} which are typical absorptions of C=C-H strain. In the product spectrum, the absorptions at this wave number are no longer visible. This means that the C=C bonds of the fatty acids that make up nyamplung oil have been broken and formed epoxy groups/ oxirane rings. The product spectrum also shows a typical absorption of the C-C epoxy group at a wave number of 824 cm^{-1}. The three absorptions mentioned above are an indication of breaking the unsaturated bonds in the oil and forming epoxy groups/oxirane rings. Gunawan et al. (2023) and Derahman et al. (2019), which respectively carried out epoxidation of ketapang oil and methyl ester of jatropha oil, also reported absorption at 825 – 824 cm^{-1}.

Meanwhile, the ^{13}C NMR spectrum (**Figure 6**) of the product shows a chemical shift at 54, 56 and 57 ppm which is the C-O bond of the epoxy group. These results are in accordance with research by Martínez et al. (2021), on the epoxidation of linseed (*Linum usitatissimum*) oil, identified the C-O bonds of the epoxy group at 40 – 80 ppm. Identification of the presence of epoxy groups in a compound can also be done using the ^{1}H NMR spectrum. The ^{1}H NMR spectrum of the epoxidation product of nyamplung oil (**Figure 7**) shows a chemical shift at 2.8–3.1 ppm which is believed to be an epoxy group. This result is identical to Gong et al. (2023) who obtained a chemical shift of the epoxy group at 2.6 – 3.3 ppm.

CONCLUSIONS

Optimizing the chemically epoxidation of nyamplung oil has been successfully carried out. The oxidizing agent used is performic acid which is synthesized in situ by reacting formic acid (HCOOH) and hydrogen peroxide (H_2O_2), and is catalyzed by sulfuric acid (H_2SO_4). The epoxidation parameters to produce epoxy that has the highest percentage of RCO are epoxidation duration of 6 hours, ratio of nyamplung oil:HCOOH:H_2O_2 is 1:3:4 (mole/mole), and epoxidation temperature of 45 °C. Under these reaction conditions, the percentage of RCO was 76.8%. The products have been characterized using the FTIR spectrophotometer and the ^{13}C-NMR and ^{1}H-NMR spectrophotometers.

ACKNOWLEDGEMENTS

This work was financially supported by the Ministry of Education, Culture, Research and Technology of Republic of Indonesia (Grant Number: 2348/ UN18.L1/PP/2023).

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