IJFAC

Indonesian Journal of
Fundamental and Applied Chemistry

Article http://ijfac.unsri.ac.id

Synthesis of Epoxide as Intermediate Compounds for Biolubricant Production from Crude Palm Oil

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Abstract

The use of biolubricant must be encouraged to reduce environmental pollution from non-biodegradable lubricants by utilizing crude palm oil. This study aims to synthesize epoxide compounds from crude palm oil (CPO) as intermediate compounds to produce Biolubricant and determine the kinetic reaction and the enthalpy of the epoxide reaction. The process of synthesizing epoxide was carried out in a stirred reactor. The epoxidation reaction occurred at 60, 70, and 80 °C for 30 – 180 min with 30 min intervals using 1, 2, and 3% H_2SO_4 as catalysts. The highest conversion was obtained at a reaction temperature of 70 °C at 90 min using 2% H_2SO_4 of 87.59%. The characteristics of the epoxide compound produced have a density of 0.951 g/cm^3, a kinematic viscosity of 44.388 CSt, an acid number of 4.32 mg NaOH/g oil, and oxirane number of 5.731 g/cm^3. The reaction kinetic constant and enthalpy were 0.0104 mol/mL min and 10494.762 J/mol. The synthesized epoxide from crude palm oil was proven to be a suitable intermediate compound for biolubricant production.

Keywords: Biolubricant, Biodegradable, Epoxide, Crude palm oil

Abstrak (Indonesian)

Penggunaan biolubricant harus didorong untuk mengurangi pencemaran lingkungan dari pelumas non-biodegradable dengan memanfaatkan minyak kelapa sawit. Penelitian ini bertujuan untuk mensintesis senyawa epoksida dari minyak kelapa sawit sebagai senyawa antara untuk menghasilkan biolubricant dan menentukan reaksi kinetik dan entalpi reaksi epoksida. Proses sintesis epoksida dilakukan dalam reaktor berpengaduk. Reaksi epoksidasi terjadi pada suhu 60, 70, dan 80 °C selama 30 – 180 menit dengan interval 30 menit menggunakan katalis 1, 2, dan 3% H_2SO_4. Konversi tertinggi diperoleh pada suhu reaksi 70 °C pada 90 menit menggunakan 2% H_2SO_4 sebesar 87,59%. Karakteristik senyawa epoksida yang dihasilkan memiliki densitas 0,951 g/cm^3, viskositas kinematik 44,388 CSt, bilangan asam 4,32 mg NaOH/g minyak, dan bilangan oksiran 5,731 g/cm^3. Konstanta kinetika dan entalpi reaksi adalah 0,0104 mol/mL min dan 10494.762 J/mol. Epoksida hasil sintesis dari minyak kelapa sawit terbukti menjadi senyawa antara yang cocok untuk produksi biolubricant.

Kata Kunci: Pelumas nabati, Biodegradable, Epoksida, Minyak kelapa sawit

Article Info

Received 18 April 2022
Received in revised 15 May 2022
Accepted 24 May 2022
Available online 25 June 2022

INTRODUCTION

The increase of global population with industrialization and modernization has led to increased energy consumption. Fossil fuels have produced CO_2 which has caused global warming. Fuel and lubricants are increasingly being used for power generation, mining, agriculture, forestry activities, transmission oil, engine oil, hydraulic oil, and transportation activities [1]. The use of production machines in various industries and motorized vehicles has increased in direct proportion to the production of engine lubricants required to maintain engines and motor vehicles. Lubricants on the market today are generally synthesized from raw materials derived from petroleum derivatives.

About 85% of lubricants used worldwide are made from crude oil [2]. The use of lubricants in the automotive sector in 2017 reached 39 million tons and

DOI: 10.24845/ijfac.v7.i2.42

continues to increase by 2% every year [3], [4]. In Indonesia, lubricants are produced from crude oil as much as 60% for domestic needs by PT. Pertamina, while 40% are still imported [5]. The manufacture of biolubricant from palm oil (CPO) is one of the pathways for the downstream oleochemical industry. The upstream agro-industry development program is focused on industries, one of which is the oleochemical industry, namely biolubricant (rolling oils). This statement follows regulations from the Ministry of Industry of the Republic of Indonesia and Presidential Regulation Number 2 of 2018 concerning the 2015-2019 national industrial policy.

The increasing use of lubricants has caused the pollution of B3 and non-biodegradable waste to the environment. Exposed used lubricants not only contaminate the soil, but also pollute the air. Meanwhile, lubricants discharged into waters can enter wells, agricultural areas, and drinking waters [6], [7]. This can be overcome by replacing non-biodegradable lubricants with biolubricant. Besides being cheap and environmentally friendly, lubricants from vegetable oils show good lubrication properties with a high viscosity index. However, vegetable oils have several drawbacks that limit their application. One of the drawbacks is the low oxidative stability due to the presence of bis-allylic protons in the plant oil structure, which are highly susceptible to free radical attack [8], [9]. Therefore, oxidative degradation can form polar oxygen compounds [10].

Oxidative weakness in vegetable oils can be overcome by chemical modification of the molecular structure. The double bonds and reactivity can allow the addition of functional reactions to unsaturated fatty acids. One of the functional reactions of double bonds to increase the oxidative stability of vegetable oils is in the epoxidation process. In addition, the advantages of using biolubricants from vegetable oils are renewable, heat stable, high viscosity index, easy flow, low emission, stable evaporation, high additive solubility, and easily soluble in other liquids, biodegradable, non-toxic, and environmentally friendly.

Biolubricant can be produced from various types of plants, such as oil palm, soybean, sunflower, castor and others. As one of the largest CPO production countries in the world, Indonesia produced approximately 36.96 million tons with 12.76 million ha in 2018 [11], [12]. Thus, Indonesia can produce biolubricant in large quantities. Research on biolubricant synthesis has been carried out by previous researchers. Zin et al. [13] synthesized biolubricant from the epoxidation process of crude palm kernel oil with the conversion of oxirane number achieved was 65.59%. Biolubricant production from soybean oil has been carried out through epoxidation and hydroxylation processes. The highest conversion of epoxy compounds achieved was 53.90% [14].

Synthesis of biolubricants from castor oil using a saponification process followed by esterification has been carried out by Pathmasiri et al. [15]. The kinematic viscosity of 79.4 cSt was obtained 40 °C. Furthermore, a study on the selective epoxidation of sesame oil by Musik and Milchert [16] resulted in a relative conversion of 78.1 % of oxirane. Based on these searches, epoxide synthesis as an intermediate compound in the biolubricant production from crude palm oil is limited. Therefore, this study aims to obtain epoxide compounds from crude palm oil as intermediate biolubricant compounds at various temperatures, times and catalyst concentrations. The quality of the epoxide compounds was also evaluated in this work.

MATERIALS AND METHODS

Materials

Crude palm oil (CPO) as the primary raw material was obtained from PT. Golden Oilindo Nusantara in Ogan Ilir, South Sumatra. The characteristics analysis results of CPO are presented in **Table 1**. Glacial acetic acid, sulfuric acid, and Sodium Bicarbonate were purchased from Merck, and hydrogen Peroxide 30% was purchased from Bratachem. Peroxylic acid as a reactant is obtained from acetic acid and hydrogen peroxide reactions. Sulfuric acid is fed into the reactor as a catalyst.

Table 1. Characteristics of crude palm oil compared to other vegetable oils

Parameter	**Unit**	**Value**		
		CPO	**Soybean oil**	**Castor seed oil**
Density	g/mL	0.948	0.836	0,955
Kinematic viscosity	CSt	37.978	471	218.18
Acid number	mg NaOH/ mg CPO	8.320	0.44	2
Oxirane number	%/100 g	0.099	-	-

Crude palm oil has a density value that is not much different from soybean oil and castor bean oil but has a significant difference in kinematic viscosity and acid value. Even though it has physicochemical differences, palm oil can still be used as a raw material for making biolubricant.

Epoxide Synthesis

The epoxide synthesis was begun by mixing 200 mL of crude palm oil (CPO) with 20 wt% acetic acid to be fed into the reactor. The epoxidation reaction took place in a stirred reactor equipped with heating, cooling and temperature control systems. The reaction took place at three different temperatures, namely 60, 70, and 80 °C. After the desired temperature is reached, 60 wt% hydrogen peroxide is added to the mixture. The mixture formed was then added with sulfuric acid with variations of 1, 2, and 3 wt% dropwise for 30 – 180 min with 30 min intervals. The resulting epoxide compound was cooled and separated using a separatory funnel to then add a saturated solution of sodium bicarbonate (200 mL) and aquadest (200 mL) accompanied by stirring. This process is repeated several times to neutralize the mixture. After neutral pH, the mixture was allowed to stand for 30 min to separate the epoxide, water and other compounds. The characteristics of the epoxide compound produced were determined from the density, kinematic viscosity, acid number and oxirane number.

Epoxide compound analysis

Crude palm oil (CPO) and epoxide compounds consisted of density using the SNI-06-4085-1996 method, kinematic viscosity using the ASTM 445 method, and the acid number using the AOAC 1995, and oxirane number using the ASTM D1652 method.

RESULTS AND DISCUSSION

Epoxide compound density

The effect of epoxide compounds on variations in reaction time, reaction temperature, and catalyst concentration has been investigated on the density of the epoxide compounds (**Figure 1**). In the process using 1% H_2SO_4 catalyst, the longer the reaction time and the higher the temperature, the density of the epoxide compound increases from 0.872 to 0.971 gr/cm^3. The density of epoxy compounds at constant reaction time increases with temperature [14]. Meanwhile, when the 2% – 3% H_2SO_4 catalyst was applied, the longer the reaction time and the higher the reaction temperature, the density value decreased from 0.971 – 0.796 gr/cm^3. The highest density value was obtained at 80 °C for 60 min of 0.971 gr/cm^3. With increasing reaction temperature, the epoxide compound will expand, which causes the volume to increase, resulting in a smaller density.

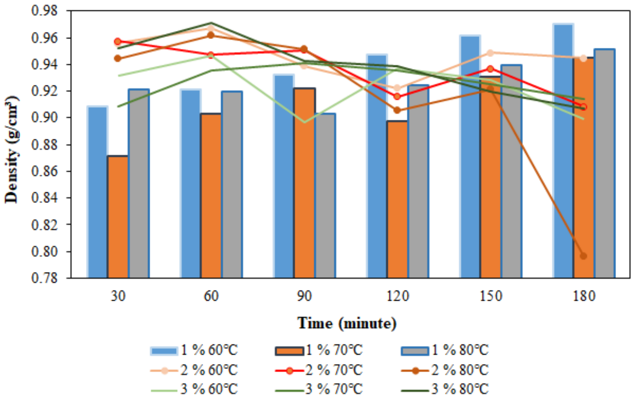

Figure 1. The effect of variations in reaction time, temperature, and catalyst concentration on the density of the epoxide compound

Epoxide compound viscosity

The results of the kinematic viscosity analysis of the epoxide compound are shown in **Figure 2**. The higher the reaction temperature and the length of the reaction time, the synthesis process using 1% and 3% H_2SO_4 resulted in a decrease in the kinematic viscosity coefficient from 46.525 to 36.229 CSt. Meanwhile, using 2% H_2SO_4 produces fluctuating kinematic viscosity coefficient values. The lowest viscosity was obtained at 36.229 CSt at 60 °C for 180 min with a catalyst concentration of 3%. The highest viscosity was achieved at 80 °C for 120 min and 2% catalyst concentration of 57.792 CSt. These results are the effect of the unstable reaction temperature during the epoxidation process and the large density of the resulting epoxy compound.

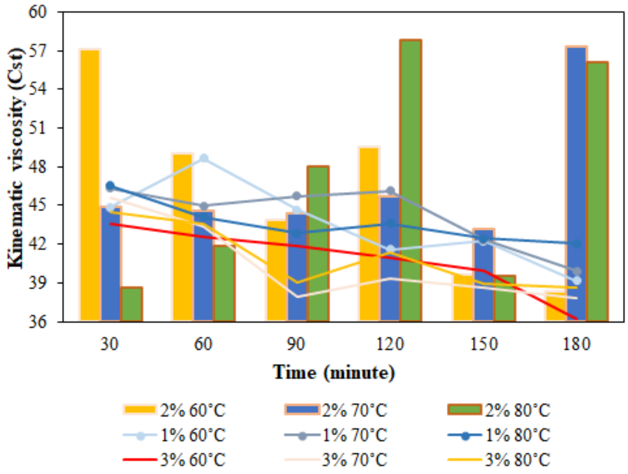

Figure 2. Kinematic viscosity analysis of epoxide compounds

Epoxide compound acid number

Figure 3 shows the epoxide acid number that tends to increase and is directly proportional to the increase in temperature, time, and catalyst concentration. Using 1, 2, and 3% catalysts resulted in acid number values in the range of 15.28 – 34.48, 4.32 – 24.64, and 13.60 – 29.12 mg NaOH/g oil, respectively. The lowest acid value was obtained using 2% H_2SO_4 at 70 °C and 120 min at 4.32 mg NaOH/g Oil. This condition indicated that the longer the reaction time and the higher the temperature, the value of the acid number did not decrease and even continued to increase, as shown in **Figure 3**.

As the reaction temperature, reaction time, and catalyst concentration increase, the oxidation process will be faster and more reactive between oxygen and fatty acids. This oxidation reaction will cause an increase in the acid number in the reaction. In addition, because the reaction is reversible, there will also be a re-formation of the acetic acid compound from the side reaction, increasing the acid number. Although the oxidation process advances, it will reduce the oxirane compound formed because the oxirane ring is degraded, which will result in a hydroxylation reaction that can form polyol compounds.

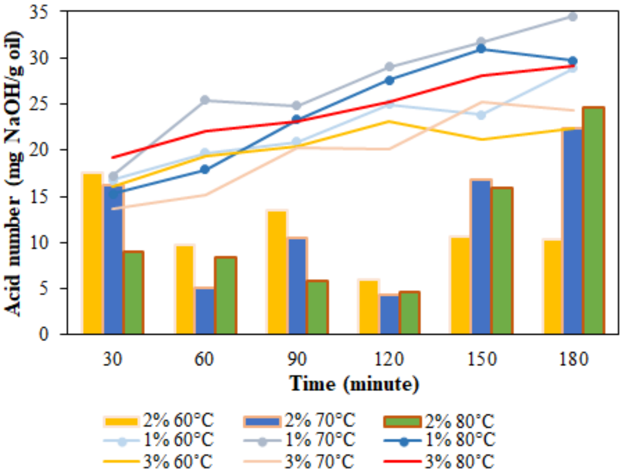

Figure 3. The effect of variations in reaction time, temperature, and catalyst concentration on the acid number of epoxide compounds

Effect of temperature on oxirane number of epoxide compounds

The effect of reaction temperature on the oxirane number can be seen in **Figure 4**. The highest oxirane number was obtained at 70 °C of 5.7312 gr/cm^3. At lower temperatures (60 °C), the reaction is slow and takes a long time to get high conversion. Also, it takes a longer time to complete the double bond conversion [17]. Compaction tends to occur and can reduce the possibility of reactant interface interactions [18]. The oxygen concentration of the oxirane increases with reaction time. It takes a long time to achieve high yields without depletion [19].

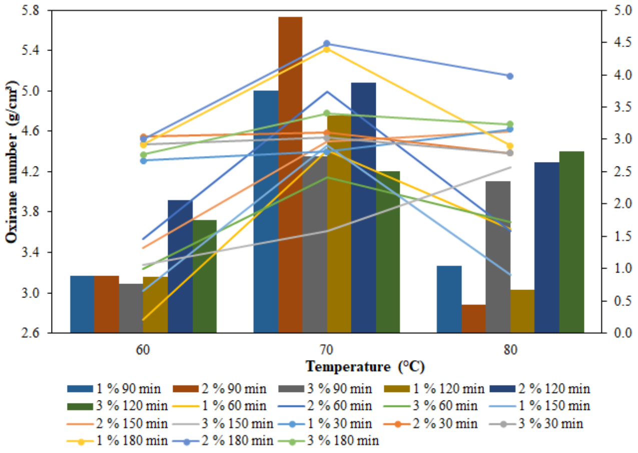

Figure 4. Effect of temperature on the oxirane number of epoxide compounds

Effect of reaction time on oxirane number of epoxide compounds

The longer the reaction time, the greater the value of the oxirane number. This is indicated by the results presented in **Figure 5**. The highest oxirane number was achieved at 90 min of 5.731 gr/cm^3. The longer the reaction time, the higher the reaction rate of the epoxide compound so that the chance of the reactant molecules colliding with each other is heightened. In addition, the double bonds contained in the oil undergo oxidation of the opening of the double bonds by peroxy carboxylic acid [20], [21].

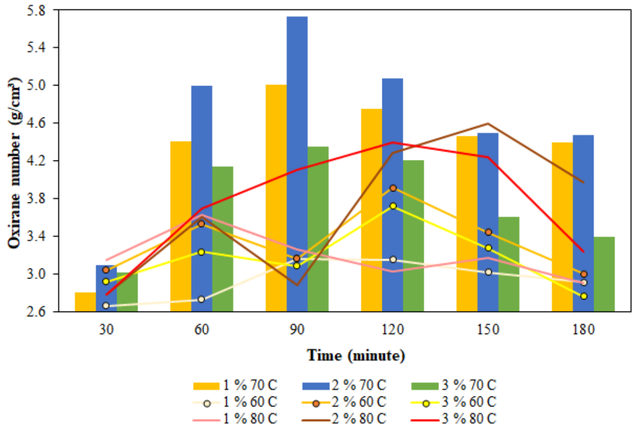

Figure 5. Effect of reaction time on the oxirane number of epoxide compounds

The degradation of the oxirane group can occur during the epoxidation reaction caused by the presence of acetic acid from the reaction of the formation of reversible peroxyacetic acid. Acetic acid and sulfuric acid can also produce side reactions. Thus, the longer the reaction time, the greater the degradation of the oxidant [22]. The presence of benzene in the reaction will minimize the opening of the oxirane/epoxide ring to form more epoxide compounds [23].

Effect of catalyst concentration on oxirane number of epoxide compounds

The effect of catalyst concentration on the oxirane number can be seen in **Figure 6**. The greater the catalyst concentration, the higher the oxirane number obtained. In using 2% catalyst, the oxirane number obtained was 5.731 g/cm^3. This is made possible by the rate of formation of the oxirane becoming faster with the increase in the amount of catalyst. The catalyst affects the conversion of the double bond and the formation of the desired product [24]. The extra hydrogen ions from the external acid catalyst can influence the increase in the formation of the oxirane ring [25]. At higher concentrations, sulfuric acid and peracetic acid come into contact with the epoxide formed, increasing the likelihood of the epoxy ring being hydrolyzed or exposed by temperature. At higher concentrations, sulfuric acid and peracetic acid come into contact with the epoxide formed, increasing the possibility of the epoxy ring being hydrolyzed or opened by temperature to achieve high conversion [26]. The addition of sulfuric acid will increase the conversion of oxirane number in fatty acid epoxidation [27].

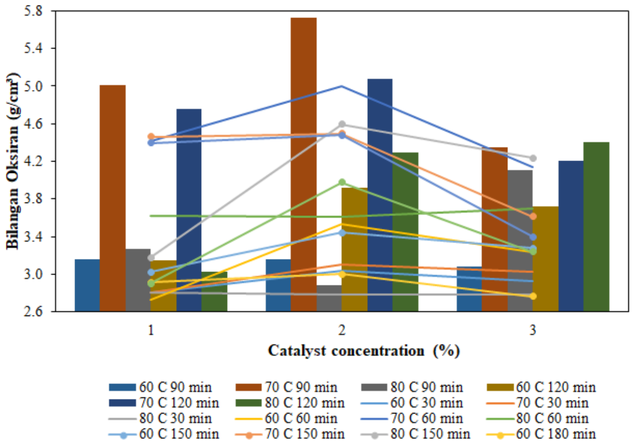

Figure 6. Effect of catalyst concentration on the oxirane number of epoxide compounds

The standard deviation (SD) for density, viscosity, acid number and oxirane number were 0.0129–0.061, 1.6188–8.3994, 2.5315–7.5801, and 0.2102–0.8859, respectively (**Table 2**). The standard deviation value obtained is low. It was indicated that the data from this study is close to the average value, and the range of data variations is getting smaller.

Table 2. Standard Deviation

Parameter		**Analysis**			
Catal (%)	**Temp (°C)**	**Density**	**Viscosity**	**AD**	**ON**
1	60	0.02	3.29	4.28	0.21
	70	0.02	2.56	6.09	0.77
	80	0.0167	1.61	6.43	0.24
2	60	0.0152	7.04	3.87	0.35
	70	0.0198	5.26	7.14	0.88
	80	0.0610	8.39	7.58	0.73
3	60	0.0203	2.59	253	0.33
	70	0.0129	3.25	4.69	0.52
	80	0.0228	2.55	3.72	0.62

Effect of temperature, time, and catalyst concentration on epoxide conversion and reaction kinetics

An epoxidation reaction is a step reaction that is reversible and irreversible. In the initial stage, the reaction is initiated by feeding triglycerides from CPO with peroxylic acid (Peracetic). This epoxidation reaction will produce an epoxy compound in the form of the desired product and acetic acid as a by-product according to the reaction scheme in **Figure 7**.

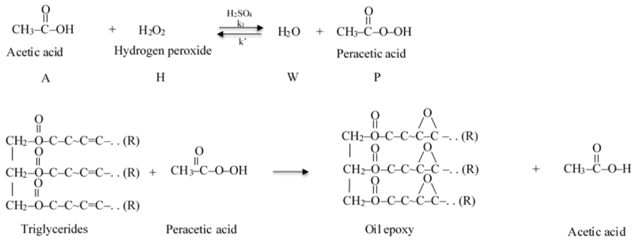

Figure 7. Reaction scheme for epoxidation reaction

Based on the reaction in **Figure 7**, the epoxidation reaction kinetics can follow equation 1 as follow:

$$\frac{dC_P}{dt} = k_1 \cdot C_A \cdot C_H - k_2 \cdot C_P \cdot C_T \quad (1)$$

$$\frac{dC_E}{dt} = k_2 \cdot C_P \cdot C_T \quad (2)$$

Where:

C_P = Peroxylic acid
C_A = Acetic acid
C_H = Hydrogen peroxide acid
C_T = Triglycerides (CPO)
C_E = Epoxide

Substitute equation 2 into equation 1 to obtain equation 3.

$$\frac{dC_P}{dt}=k_1 \,.\, C_A \,.\, C_H - \frac{dC_E}{dt} \qquad (3)$$

Because it is assumed that the change in the concentration of peracetic acid with time is very small compared to the concentration of the oil epoxide so that peracetic acid becomes intermediate, then:

$$\frac{dC_P}{dt} = 0 \text{ , that } \frac{dC_P}{dt} \ll \frac{dC_E}{dt}$$

The equation 4 is obtained:

$$\frac{dC_E}{dt}=k_1 \,.\, C_A \,.\, C_H \qquad (4)$$

with $C_A = C_{A0}$, thus the equation 5 become:

$$\frac{dC_E}{dt}=k_1 \,.\, C_{A0} \,.\, C_H \qquad (5)$$

From the mass balance, the equation 6 derived:

$$C_H = C_{H0} - C_E \qquad (6)$$

C_H is the limiting reagent.

Substitute equation 6 into equation 5 to get equation 7-8.

$$\frac{dC_E}{dt}=k_1 \,.\, C_{A0} \,.\, (C_{H0}-C_E) \qquad (7)$$

$$k' = k_1 \,.\, C_{A0}$$

$$\frac{dC_E}{dt}=k' \,.\, (C_{H0}-C_E) \qquad (8)$$

Equation 8 is integrated to obtain equation 9.

$$\int_0^{C_E} \frac{dC_E}{(C_{H0}-C_E)} = k' \int_0^t dt \qquad (9)$$

Example: $C_{H0} - C_E = U$

$$dU = -dC_E$$

$$dC_E = -dU$$

$$= -\int_0^{C_E} \frac{dU}{U} = k' \int_0^t dt$$

$$= -\ln U \Big|_0^{C_E} = k' \,.\, t \Big|_0^t$$

$$= -\ln (C_{H0} - C_E) \Big|_0^{C_E} = k' \,.\, t \Big|_0^t$$

$$= -\ln (C_{H0} - C_E) \Big|_0^{C_E} = k' \,.\, t \Big|_0^t$$

$$= -\ln(C_{H0} - C_E) + \ln C_{H0} = k'.(t) \qquad (10)$$

Equation 10 can be simplified to equation 11.

$$\ln (C_{H0}) - \ln(C_{H0} - C_E) = k'.(t)$$

$$\ln \left[\frac{(C_{H0})}{(C_{H0}-C_E)}\right] = k' .\, (t) \qquad (11)$$

Afterwards, substitute equation 6 into equation 11 to generate equation 12.

$$\ln \left(\frac{C_{H0}}{C_H}\right) = k'.(t) \qquad (12)$$

It is known that the concentration equation corresponding to the conversion is equation 13-16:

$$C_H = C_{H0} \,.\, (1 - X) \qquad (13)$$

$$\ln \frac{C_{H0}}{C_H} = k'.(t) \qquad (14)$$

$$\ln \frac{C_{H0}}{C_{H0}\,(1-X)} = k'.(t) \qquad (15)$$

$$\ln \frac{1}{(1-X)} = k'.(t) \qquad (16)$$

Equation 16 is used to determine the reaction constant.

From the epoxide reaction scheme, the conversion is calculated based on the oxirane number obtained from each variable in the epoxide reaction process shown in Figure 8.

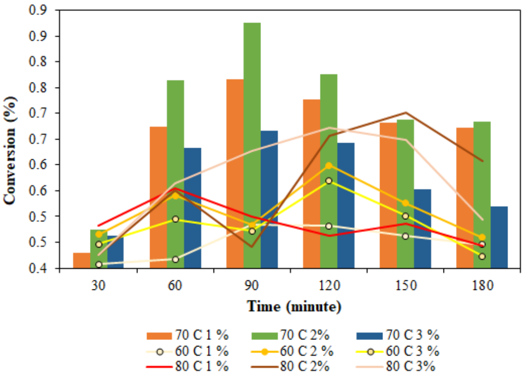

Figure 8. Effect of temperature, time, and catalyst concentration on the conversion of epoxide compounds

The resulting conversion is more significant with the higher the reaction temperature, the length of reaction time, and the concentration of the catalyst used (**Figure 8**). The highest conversion obtained was 87.59% at 70 °C for 90 min using 2% H_2SO_4. These operating conditions were marked as optimum conditions for the production of epoxide compounds. The lowest conversion was 40.79% at 60 °C for 30 min using 2% H_2SO_4. After passing the optimum conditions, the conversion value decreased.

The constant rate of the epoxidation reaction at a specific temperature is determined from the relationship curve ln (1/(1-x)) versus time (t) so that a linear regression line is obtained, which has the slope of the curve as a slope. The results of the reaction rate kinetics calculation are summarized in **Table 3**.

Table 3. k value of the epoxidation process

H_2SO_4 (%)	Temperature (°C)	k (mol/ml min)
1	60	0,0047
	70	0,0091
	80	0,005
2	60	0,0056
	70	0,0104
	80	0,0073
3	60	0,0051
	70	0,0068
	80	0,007

The highest reaction rate constant was 0.0104 mol/mL min at 70 °C using 2% catalyst, while the lowest reaction rate constant was obtained at 60 °C. The difference in the value of the reaction rate constant is caused by temperature variations that affect the conversion. Then, the reaction rate constant at 80 °C was slightly lower. The reaction equilibrium at 80 °C has dropped as the temperature exceeded the equilibrium limit. As a result, the conversion was more down and produced more by-products because the reaction is reversible.

The epoxidation reaction rate constant is used to calculate the activation energy and enthalpy at each temperature of the epoxidation reaction. The activation energy was calculated using the Arrhenius equation as follows (Eq. 17):

$$k=Ae^{-E/RT} \tag{17}$$

Eq. (17) is converted into logarithmic form into Eq. 17.

$$\ln k = \ln A - E/RT \tag{18}$$

By plotting ln k against 1/T on the curve, the activation energy (-E/R) and frequency factor (ln A) can be solved from the slope and intercept in the curve. The calculated values of activation energy (E), frequency factor (A), and enthalpy were summarized in **Table 4**.

Table 4. Activation energy, frequency factor and enthalpy of the epoxide synthesis reaction

Acid (%)	Temp (°C)	E_a (J/mol)	Frequency Factor (mol/L min)	ΔH (J/mol)
1	60	3603.953	0.021	835.391
	70			752.251
	80			669.111
2	60	13346.464	0.812	10577.902
	70			10494.762
	80			10411.622
3	60	15514.755	1.442	12746.193
	70			12663.053
	80			12579.913

In **Table 4**, it can be seen the effect of reaction temperature and sulfuric acid catalyst concentration. The higher the reaction temperature and catalyst concentration, the greater the activation energy, frequency factor, and reaction heat. This showed that although considerable activation energy can slow down the reaction, adding the catalyst concentration will speed up the reaction that occurs. The frequency factor value is close to one, which means that all particles have sufficient energy to react, although reacting requires a greater heat reaction. Under the optimum conditions for the epoxide reaction (70 °C, 90 min, and 2% H_2SO_4), the resulting enthalpy was 10494.762 J/mol.

CONCLUSION

The synthesis of epoxide compounds from crude palm oil has been successfully carried out. Variables that affect the yield of epoxide compounds have been investigated. The higher the reaction temperature, reaction time, and catalyst concentration, the greater the oxirane number and the resulting conversion. The highest conversion achieved was 87.59% at 70 °C, for 90 min and 2% H_2SO_4, with a reaction rate constant of 0.0104 mol/mL min and enthalpy of 10494.762 J/mol. The epoxide compound has characteristics with the density of 0.951 g/cm^3, Kinematic Viscosity of 44.388 CSt, an acid number of 4.32 mg NaOH/g oil, and an oxirane number of 5.731 g/cm^3.

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