Epoxidation of Unsaturated Oleic Acids
via in Situ Catalytic Performic Acid

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ABSTRACT: *Studies on the epoxidation of fatty acids have garnered much interest in recent years due to the rising demand for eco-friendly epoxides derived from vegetable oils. This study aims to optimize the process parameters of epoxidation of palm oleic acid via an in situ peracid mechanism with an applied homogenous catalyst. Oleic acid was epoxidized using performic acid-generated in situ through the reaction between hydrogen peroxide and formic acid when sulfuric acid was applied as a catalyst. The optimum reaction condition of epoxide was at the temperature of 45°C, the molar ratio of formic acid to oleic acid at 1.64:1 and the molar ratio of hydrogen peroxide to oleic acid at 2:1. Lastly, a mathematical model was developed using the numerical Runge Kutta-4th Order method. In the model, the method was applied with a genetic algorithm optimization to determine the process model that fit the experimental data using MATLAB software. After 100 iterations, the reaction rate constant for epoxidized oleic acid production was: $k_{11} = 1.9305$ L/mol. min, $k_{12} = 15.2284$ L/mol. min, $k_{21} = 0.0570$ L/mol. min, $k_{31} = 0.0106$ L/mol. min. Overall, epoxidized oleic acid was successfully produced by in situ performic acid mechanism with 80% relative conversion to oxirane.*

KEYWORDS: *Epoxidation; Oxirane content; Optimization; Kinetic study; Hydrolysis; Oleic acid; Performic acid.*

INTRODUCTION

In today's world, the consumption of raw materials has changed from non-renewable materials-based to renewable materials-based. The previous production of epoxide was from petroleum oil. But now, the production of epoxide was made using a renewable source, namely vegetable oil. The cost-effectiveness and environmentally friendly material traits of vegetable oil made it to be chosen as an alternative to producing epoxide. Epoxy is a pre-polymer with low molecular weight and consists of more than one epoxide group. The formation of epoxide groups occurs when there is a chemical reaction that converts the double bonds by the addition of oxygen atoms [1]. Several methods can be used to produce epoxidized vegetable oil, such as epoxidation with halohydrins and molecular hydrogens [2], epoxidation with organic and inorganic oxidants [3], as well as epoxidation with per carboxylic acid generated with in situ or preformed [4]. In terms of application, it is used as a plasticizer, stabilization in polyvinyl chloride, and intermediates product, such as for polyols production [5]. The highest composition of oleic acid is present in vegetable oil, hence, it is suitable and has a better performance than produced epoxidized oleic acid. The reaction mechanism to obtain epoxides from oleic acids was shown in Equations (1, 2).

$$H_2O_2 + HCOOH \xrightarrow{H_2SO} HCO_2OH + H_2O \qquad (1)$$

$$R-CH-CH-R' + HCO_2OH \leftrightarrow \qquad (2)$$
$$R-CH-O-CH-R + HCOOH$$
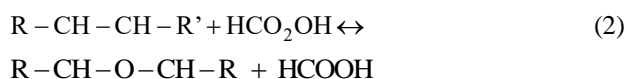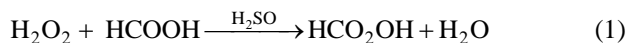
In epoxidation reactions, formic acids work as an oxygen carrier while hydrogen peroxide is an oxygen donor [6]. The reaction between formic acid and hydrogen peroxide results in the formation of performic acid. Formic acid is preferred as an oxygen carriers over acetic acid because it has high reactivity and requires no catalyst to speed the formation of peracids [7]. According to *Aguilera et al.* [8], epoxidation is usually performed with organic peracid or peroxides since hydrogen peroxide (which is used as an oxygen donor in the epoxidation process) is insoluble in oil. Peracid is more favourable as it is inexpensive and thus, it is widely used for epoxidation in the industry. Peracid is usually prepared using *in situ* methods due to the handling issues associated with preformed peracid as preformed peracid is unstable and explosive.

In the past, epoxides were mainly produced from petroleum-based sources. However, many problems were encountered regarding this material in terms of cost and environmental impacts [9]. Based on previous studies the production of polyols can be produced from vegetable oil [10]. However, details on the influence reaction parameter and reaction optimization associated with the oxirane ring formation should be further investigated. Secondly, to date, epoxidized oleic acid production was mainly from sunflower [11] and soybean oil [12], while in this research, palm oil was used since each of the analysed vegetable oils has a specific oleic acid purity and iodine value, depending on their plant sources.

To date, there is limited published work on the kinetics of the production of epoxidized oleic acid by homogenous catalyst based on optimization of epoxidized oleic acid. The kinetic investigation of this reaction is important for the design of an industrial-scale reactor for the desired reaction to proceed to the required degree of conversion. The rate of reaction caused solely by chemical reaction is known as intrinsic reaction rate whereas the rate of reaction that is affected by chemical reaction and transport properties is termed global reaction rate. In this research, the intrinsic reaction rate was studied to obtain a reaction rate for a chemical reaction by assuming the system to behave like a homogeneous system. Therefore, this paper aimed to study the effect of temperature, unsaturation molar ratio of formic acid to oleic acid, and hydrogen peroxide to oleic acid on the formation of epoxidized oleic acid with the determination of its physicochemical properties. Lastly, the main contributions of this study were the significant improvement in the epoxidation process and the application of epoxidized oleic acid.

EXPERIMENTAL SECTION
Materials

The chemicals involved were hydrogen peroxide (30%), formic acid (85%), oleic acid (75% purity), sulphuric acid (95%), glacial acetic acid, crystal violet solution, hydrogen bromide, and distilled water.

Epoxidation procedure

The epoxidation was carried out in a 250 mL beaker equipped with a magnetic stirrer and thermometer. Oleic acid, formic acid and sulphuric acid were added simultaneously into the flask. The mixture was stirred

Table 1: Reaction parameter and range for epoxidation of oleic acid.

Reaction parameter	Range	Units
Temperature	45-95	°C
Agitation speed	1000	Rpm
Reaction time	45	Minutes
Catalyst (sulfuric acid)	0.2-0.5	Gram

continuously at a constant speed and reaction time. It was gradually heated until the desired temperature was achieved. The setting temperature was located on the hot plate with a thermometer placed inside the beaker. Hydrogen peroxide will be added drop by drop during stirring after achieving the desired temperature. Table 1 summarises the material and experimental parameters that were set up for the epoxidation of oleic acid. All experiments were repeated three times.

Determination of experimental Oxirane Oxygen Content (OOC)

The procedure to obtain the OOC value can be explained as follows: 10 mL of acetic acid was added to the sample weight in an Erlenmeyer flask and the mixture was shaken. Next, two drops of crystal violet indicator were dropped into the mixture. Then, the mixture was titrated while stirring with hydrogen bromide until the bluish-green color is obtained and stayed at least 30 [13]. The equation to calculate (OOC_{exp}) in moles per 100 g is shown as Equation 3.

$$OOC_{exp} = \frac{T \times N_{HBR} \times 1.6}{W} \tag{3}$$

In which, the T volume of hydrogen bromide (mL) is required to titrate the sample, N_{HBR} is the normality of the hydrogen bromide, and W is the weight of the sample (g). The method to determine OOC can be found in the appendix. From Equation 2, the Relative Conversion To Oxirane (RCO) content can be calculated using Equations 4 and 5. In which OOC_{theo} is the theoretically maximum oxirane oxygen content.
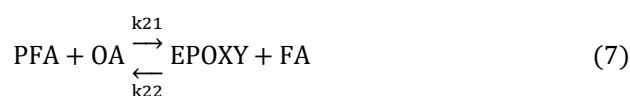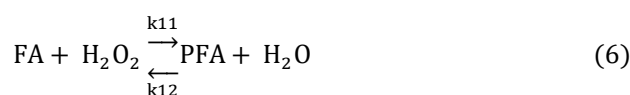
$$RCO = \frac{OOC_{exp}}{OOC_{theo}} \times 100\% \tag{4}$$

$$OOC_{theo} = \left[\frac{\frac{IV_o}{2A_I}}{100 + \left(\frac{IV_o}{2A_I}\right)A_o} \right] A_o \times 100 \tag{5}$$

Fourier Transform InfraRred (FT-IR) analysis

Fourier Transform InfraRed (FT-IR) spectroscopy is the most useful method to identify chemicals that are either organic or inorganic, and it can be utilized quantitatively for some components of an unknown mixture [14]. It can be applied to the analysis of liquids, including oil. The involvement of functional groups in sample uptake was evaluated by FT-IR (Perkin Elmer Spectrum One, USA). Oleic acid and epoxidized oleic acid were analyzed using FT-IR to determine the presence of the epoxy group and the disappearance of stretching vibration peak of =CH oleic acid stretching, respectively. The spectra were recorded by FT-IR from a wavelength ranging between 400-4000cm^{-1}, which is mid-infrared corresponding to fundamental molecular vibration mode.

Kinetic study of degradation of epoxidation palm oleic acid

The development model in the kinetic study of the epoxidation process is based on the assumptions that; 1) The epoxidation process takes in a single phase to avoid distribution constant for the qualification of different species in aqueous and oil phases, 2) Each phase's volume remains constant throughout the process, 3) All reactions involved are homogeneous process, 4) The reactions are away from the interface. *In situ* epoxidation is characterized by two main reactions involving the formation of performic acid and epoxide, as illustrated in Equation 6 and Equation 7, respectively. The degradation of epoxidation is described in Equation 8.

$$FA + H_2O_2 \underset{k_{12}}{\overset{k_{11}}{\rightleftharpoons}} PFA + H_2O \tag{6}$$

$$PFA + OA \underset{k_{22}}{\overset{k_{21}}{\rightleftharpoons}} EPOXY + FA \tag{7}$$

$$EPOXY + H_2O \underset{k_{32}}{\overset{k_{31}}{\rightleftharpoons}} DHSA \tag{8}$$

Where FA, H_2O_2, PFA, H_2O, OA, EPOXY and DHSA are formic acid, hydrogen peroxide, water, oleic acid, epoxide vegetable oil and dihydroxy stearic acid, respectively. Note that, k_{22} and k_{32} are zero since the process is a forward reaction. Meanwhile, k_{11}, k_{12}, k_{21}, k_{22}, k_{31}, and k_{32} are the kinetic constants with respect to Equations 9 until 15. From the given epoxidation and

degradation process, the differential equations described for each species are further derived from Equations 9 to 15.

$$\frac{d[FA]}{dt} = -k_{11}[FA][H_2O_2] + k_{12}[PFA][H_2O] + \quad (9)$$
$$k_{21}[PFA][OA] - k_{22}[EPOXY][FA]$$

$$\frac{d[H_2O_2]}{dt} = -k_{11}[FA][H_2O_2] + k_{12}[PFA][H_2O] \quad (10)$$

$$\frac{d[PFA]}{dt} = k_{11}[FA][H_2O_2] - k_{12}[PFA][H_2O] - \quad (11)$$
$$k_{21}[PFA][OA] + k_{22}[EPOXY][FA]$$

$$\frac{d[H_2O]}{dt} = k_{11}[FA][H_2O_2] - k_{12}[PFA][H_2O] - \quad (12)$$
$$k_{31}[H_2O][EPOXY] + k_{32}[DHSA]$$

$$\frac{d[OA]}{dt} = -k_{21}[PFA][OA] + k_{22}[EPOXY][FA] \quad (13)$$

$$\frac{d[EPOXY]}{dt} = k_{21}[PFA][OA] - k_{22}[EPOXY][FA] - \quad (14)$$
$$k_{31}[EPOXY][H_2O] + k_{32}[EPOXY][H_2O]$$

$$\frac{d[DHSA]}{dt} = k_{31}[EPOXY][H_2O] - k_{32}[DHSA] \quad (15)$$

To determine the rate coefficient numerically, parametric studies were conducted. There are two computing processes involved, i.e., solving a set of differential equations (Equation 9 – Equation 15) numerically as well as computing the errors between experimental and simulation. The ODE45 function from MATLAB was used to solve the differential equation by integrating numerically using the fourth-order Runge-Kutta method. The model was measured by minimizing the error, *e,* between simulation with the experimental data. The error function *e* follows Equation 16.

$$e = \sum_{i=1}^{n} \frac{\left| EPOOA_i^{sim} - EPOOA_i^{exp} \right|}{n} \quad (16)$$

where $EPOOA_i^{sim}$ and $EPOOA_i^{exp}$ with the estimated and experimental epoxy concentrations, i is the ith data point, and n is the total number point. The near experiment and simulation were considered good.

RESULTS AND DISCUSSION
The influence of reaction temperature

To investigate the effect of temperature, an epoxidation reaction was carried out with a constant molar ratio of oleic acid to formic acid and hydrogen peroxide, i.e., 1:1.64 and 1:2, respectively. The studied temperatures for this research were 45°C, 70°C and 95°C. The samples were taken in five minutes intervals for 45 minutes. Generally, a lower temperature will contribute to a high rate of epoxidation, depending on the method of adding hydrogen peroxide. Initially, hydrogen peroxide was poured at once. However, later on in this research, hydrogen peroxide was added dropwise to prevent the solution from decomposing due to the sudden accumulation of hydrogen peroxide [15]. Referring to Fig. 2, the percentage of RCO was gradually increased at 45°C, starting from 5 minutes up to 30 minutes of reaction time. Then, 80% of RCO was achieved at this temperature.

Furthermore, a lower epoxidation rate was observed at the initial step but it increased the rate of ring-opening. Furthermore, the percentage of RCO at 70°C and 95°C were 42% and 43%, respectively. The degradation rate of epoxide will increase if the operating temperature is high. Therefore, the percentage of RCO formation will decrease[16]. This experiment showed that reaction time is a crucial part of the attainment of maximum RCO. The maximum percentage of RCO was obtained at 25 minutes of reaction time for all temperature conditions. As shown in Fig. 2, the percentage of RCO increased and decreased along the 45 minutes reaction time, which is not constant. This condition happened due to the reversible reaction of formic acid. Even though a high reaction temperature will increase the rate of a reversible reaction, it can also shift the reaction's equilibrium position to the reverse (Le Chateleir Principle) [17].

The Influence of the unsaturation mole ratio of formic acid to oleic acid

The effect of formic acid on oleic acid unsaturation mole ratio of 1:1, 1.5:1 and 2:1 was studied. Meanwhile, the temperature was constant at 45ºC, and the molar ratio of hydrogen peroxide was 1:2. The amount of formic acid is vital because a double bond of conversion will be achieved with a high mole ratio of formic acid content in the solution. In addition, it is acted as an oxygen carrier in the epoxidation reaction [18]. Fig. 3 shows the effect of formic acid on all molar ratios.

Referring to Fig. 3, the highest percentage of RCO is 80% at a 1.00-mole ratio and then 72% at 1.64%. Meanwhile, the lowest percentage was found at the highest mole ratio of formic acid. The highest RCO was achieved
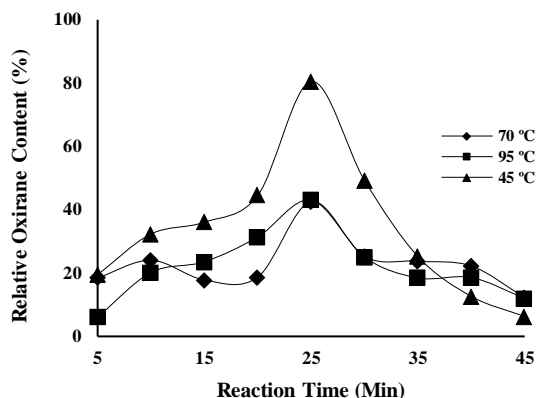
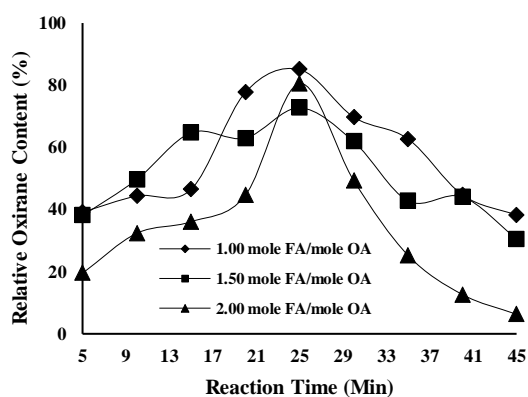**Fig. 2: Effect of temperature on epoxidation of oleic acid.**

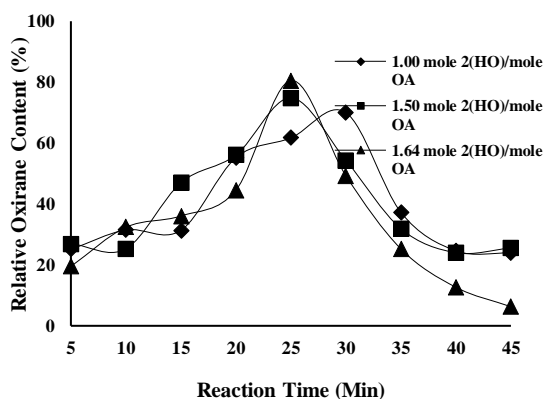**Fig. 3: Effect of unsaturation molar ratio formic acid on epoxidation of oleic acid.**

Fig. 4: The effect of molar ratio hydrogen peroxide on epoxidation of oleic acid.

at 25 minutes of reaction time. In terms of epoxidation rate, it will be increased with an increased mole ratio of formic acid [19]. However, higher formic acid content can lead to

an unstable oxirane ring [20]. It also tends to promote hydrolysis of the epoxide, decreasing the percentage of RCO. Other than that, a deficiency of formic acid can increase or decrease the RCO% along the reaction time due to the instability of the reaction [4].

The influence of the unsaturation mole ratio of hydrogen peroxide to oleic acid

Various mole ratios of hydrogen peroxide to oleic acid (1:1, 1:1.5 and 1:1.64) were studied to form epoxidized oleic acid, as shown in Fig. 4. Hydrogen peroxide acted as an oxygen donor in the formation of an oxirane ring. The results showed that the increase in hydrogen peroxide content led to an increase in the RCO percentage. Meanwhile, the lowest mole ratio of hydrogen peroxide to oleic acid, which was 1:1, gave poor stability to the oxirane ring. According to a previous study, the effect of degradation rate occurring at a low concentration of hydrogen peroxide will increase in the formation of diol and α-glycol as a side product. On top of that, a higher concentration of hydrogen peroxide to oleic acid (1:1.6) achieved a high percentage of RCO, which was 80%. This optimum conversion of oxirane of oleic acid to epoxide can be attained after the reaction time was achieved 25 minutes. As highlighted by the previous study, the yield of epoxidized oleic acid becomes constant at the maximum point with the other process parameters (temperature, agitation speed, concentration of catalyst, formic acid and oleic acid) at optimum conditions [21].

According to a previous study, 30% of aqueous H_2O_2 with mole ratio to oleic acid 1:0.75 gives the highest epoxidation rate [22]. However, the hydrogen peroxide becomes unstable as the highest rate of ring degradation occurs, which led to the production of diol and α-glycol as side products [23]. Meanwhile, a mole ratio of 1:1 can increase the complete conversion of oxirane. Furthermore, the same concentration of hydrogen peroxide with a mole ratio of 1:1 can convert epoxidation oleic acid at 25 minutes reaction time where about 88% of conversion was achieved. Furthermore, a high concentration of hydrogen peroxide can cause an increase in oxirane conversion rate, but it is not good because it can affect the stability of oxirane[24].

### *Characterization of Epoxidized Oleic Acid*

Based on the observation, oleic acid appeared as a crystal-clear liquid at room condition, while epoxidized oleic acid and polyol were in the form of semi-solid.

Table 2: Chemical/physical properties of raw and epoxidized oleic acid.

	Oleic acid	Epoxidized oleic acid
State of matter	liquid	Semi solid
Viscosity	4.5	10.2

Table 3: Optimum reaction conditions for the production epoxidized oleic acid.

Reaction conditions	Optimum value
Formic acid to oleic acid molar ratio	1: 1.00
Hydrogen peroxide to oleic acid molar ratio	1:1.64
Reaction temperature (°C)	45
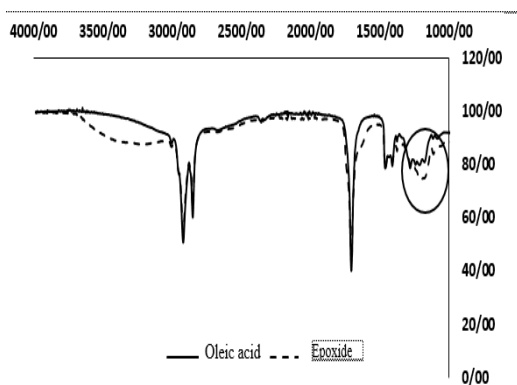
Fig. 5: FT-IR combination of oleic acid and epoxidized oleic acid.

The properties of each chemical were examined by FT-IR spectroscopy. This technique was used to determine the C=C double bonds in the oleic acid and the formation of an oxirane ring in epoxidized oleic acid. In addition, the formation of alcohol groups (OH groups), long-chain hydrocarbon groups and the opening oxirane content in the polyol were identified. The FT-IR spectrum for these chemicals is shown in Fig. 5. From the FT-IR, the double bond of oleic acid can be detected at wavenumber 2980 cm⁻¹, where this double-bond disappeared when epoxide groups were formed. The functional groups of oxirane ring-opening (C-O-C) were detected at wavenumber 1340 cm⁻¹. Furthermore, there is the presence of alcohol groups (OH) and groups of polyols that were observed at wavenumber 3440 cm⁻¹ and 816 cm⁻¹.

Furthermore, the physical form clearly showed that raw oleic acid is liquid while epoxidized oleic acid is in semi-solid form. This is due to the addition of formic acid and hydrogen peroxide to the oleic acid solution, where they reacted. It is supported with the result of viscosity as shown in Table 2, where viscosity of oleic acid is much lower than epoxidized oleic acid due to their different form.

Optimum reaction condition of epoxidized oleic acid

Looking at the previous result where the effect of reaction conditions can be summarized and presented, the optimum process can be shown in Table 3. It can be used by other researchers to produce epoxidized oleic acid *via* a homogeneous catalyst.

Comparison between simulation and experimental data

During the epoxidation process, the reaction between formic acid and hydrogen peroxide occurs for 15-30 minutes to form aqueous performic acid. Then, it slowly started increasing to form epoxide due to the diffusion of a different phase of the aqueous phase and the oil phase of performic acid reacted with oleic acid to form epoxidized oleic acid. Furthermore, an optimization tool in MATLAB simulation which is the genetic algorithm method was used to fit the experimental data and optimized the reaction. Runge Kutta Fourth Order Method was applied using the ode45 tool to solve the complex system of differential equations. The initial concentration of hydrogen peroxide, formic acid and oleic acid have used the value from the previous experiment as a reference to find the value of the kinetic rate constant, k. The kinetic rate constant for this experiment was k_{11} = 1.9305 L/mol. min, k_{12} =15.2284 L/mol. min, k_{21} = 0.0570 L/mol. min, k_{31} = 0.0106 L/mol. min with the lowest error compared to other experiments, which was only 0.1960. Fig. 6 illustrates the comparison between the epoxy concentration from the lab experiments and the simulation data. The operation in MATLAB simulation did not consider the heat transfer and heat loss during the reaction occur.

CONCLUSIONS

This study found that the optimum oxirane yield obtained at 45 °C is 80%. The functional group form in the product, analyzed by FT-IR, was consistent with the proposed chemical structure from the previous study, and therefore, the finding of this study is comparable with the results obtained from literatures. This study also found that
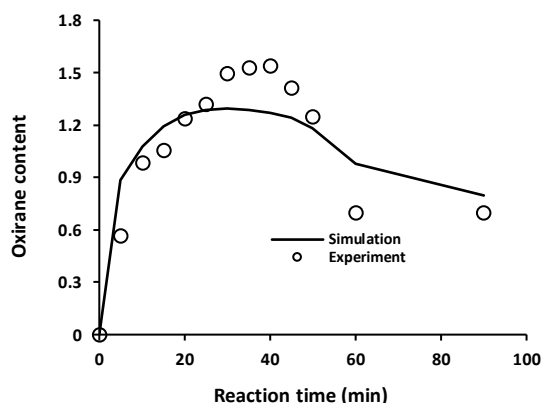
Fig. 6: Numerical modelling- Epoxidation.

the kinetic model proposed for the epoxidation reaction of palm oil successfully represents the experimental data. Furthermore, each of the reactions involved in the epoxidation and degradation process was successfully simulated using MATLAB simulation while the minimum error for the simulation was 0.1960, which is within the acceptable limit.

Acknowledgements

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