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# Sustainable Epoxidation of Unsaturated Fatty Acid through Peracid Mechanism with Amberlite Resin as a Catalyst

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**Abstract**

The growing global transition from non-renewable to renewable raw materials has spurred significant interest in sustainable alternatives. Nevertheless, the utilisation of amberlite resin for catalysing the production of epoxidised palm oil remains under-explored. This study investigates the effects of varying the molar ratio of formic acid to palm oil (0.5 : 1, 1 : 1, and 2 : 1) and different stirring speeds (150 rpm, 250 rpm, and 350 rpm) on the resulting oxirane value. Findings indicate that a molar ratio of 1 : 1 between formic acid and palm oil results in a 62 % oxirane value within 30 min, surpassing the outcomes of other tested ratios. A stirring speed of 350 rpm produced the highest oxirane value, at 68 %, compared to stirring speeds of 250 rpm and 150 rpm. The experiment successfully optimised the 1 : 1 molar ratio of formic acid to palm oil and a stirring speed of 350 rpm to increase epoxidation of palm oil. The Runge-Kutta Fourth Order method, in conjunction with genetic algorithm optimisation for numerical integration, was used to establish a mathematical model.

**Keywords**

*Peracid mechanism, epoxidation, oxirane content, palm oil, genetic algorithm*

## 1 Introduction

In recent years there has been a significant rise in interest in bio-based materials, largely due to the rapid consumption and non-renewable nature of petroleum resources. In various industries, like the production of fine chemicals and polyurethane, conventional polyols generated from petroleum have been partially or completely replaced by vegetable oil-based polyols due to their affordability, environmental friendliness, and renewable nature.[1] Palm oil, derived from the *Elaeis* genus is the second most significant source of edible oil worldwide, following soybean oil. It accounts for approximately 20 % of the world's total production of oils and fats.[2] The rising cost of mineral oils, along with growing environmental concerns, has led to increased interest in renewable alternatives across numerous sectors in recent years.[3] Epoxidation is a straightforward and efficient method for transforming double bonds into new and reactive functional molecules, which is a key aspect in the chemical modification of polyolefin.[4] Epoxidation of olefinic molecules can be accomplished in four ways, with the most common being epoxidation using percarboxylic acid.[5] This can be achieved using either organic or inorganic peroxides. Another approach involves the use of molecular oxygen.[6] The most commonly used method for the epoxidation of unsaturated molecules involves using organic peracids, which may be pre-formed or generated *in situ*.[7] Epoxidation is a crucial functional reaction for converting double bonds into stable functional groups, thereby enhancing the oxidative stability of plant oils.[8]

Homogeneous acid catalysts, like sulphuric acid, are widely employed in epoxidation reactions; however, they come with significant disadvantages, including challenges in separation, environmental concerns, and corrosive properties. In contrast, Amberlite ion exchange resins are favoured in industrial-scale epoxidation due to their excellent selectivity, ease of handling, stability, and reusability,[9] making Amberlite a more sustainable and practical choice for industrial-scale applications. The adoption of Amberlite in the epoxidation of palm oil signifies progress toward greener catalytic systems. This approach emphasises efficiency and sustainability, addressing gaps in existing studies where such solid catalysts remain less explored for palm oil epoxidation processes. In the epoxidation process, formic acid performs two essential functions: it catalyses the formation of the oxirane ring and participates as a reactant in the hydrolysis of that ring, making it vital to the entire process.[10] Achieving the highest oxirane oxygen content requires precise adjustment of the formic acid quantity to effectively balance its role in catalysing the oxirane ring formation with its involvement in hydrolysis. Maintaining this equilibrium is crucial for optimising the epoxidation outcome. The mechanism of electrophilic addition is typically involved in the epoxidation.[11] In this process, peroxyacids use syn stereochemistry to transfer an oxygen atom to an alkene, creating both C−O bonds on the same side of the double bond in a direct, single-step process without intermediate stages.[12] A nucleophile subsequently attacks this site, displacing the carboxylate, which acts as an effective leaving group.[13]

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Historically, fossil fuel-derived petroleum has been the dominant raw material in the production of oil-based products. Its negative environmental impact, including pollution, sea level rise, harmful chemical emissions, and global warming, has led to increasing concern. Since petroleum is non-renewable and cannot be readily replenished, transitioning to renewable alternatives such as vegetable oils has become a vital strategy for sustainable development. Therefore, in this study, vegetable oil was used as a renewable substitute for petroleum-derived resources.[14] To date, limited studies have investigated the specific effects of the formic acid to palm oil molar ratio and agitation speed on the epoxidation of palm oil, particularly using Amberlite resin as a solid catalyst. This research aimed to address that gap. The main objectives of this study were: (1) to assess the impact of the molar ratio of formic acid to palm oil on relative conversion to oxirane (RCO), and (2) to examine the influence of stirring speed on RCO.

## 2 Materials and method

### 2.1 Materials

The raw material used was palm oil (Lam Soon, Buruh brand). Formic acid (85 %) and hydrogen peroxide (30 %) were utilised as donors and carriers. The epoxy content was determined *via* titration using glacial acetic acid (100 %) and hydrobromic acid (45 %). Amberlite was employed as the catalyst. Crystal violet dye was used as an indicator.

### 2.2 Method/synthesis

In a 500-ml beaker, 50 g of palm oil, 4.07 g of formic acid, and 12.04 g of hydrogen peroxide were combined to initiate the reaction. Subsequently, the beaker was placed in a water-filled pot, acting as an insulator to maintain a consistent reaction temperature, similar to a water bath. A magnetic stirrer set on a heated plate was used to agitate the mixture at 350 rpm. The initial conditions for the experiment are shown in Table 1. Temperature was measured by inserting a thermometer directly into the beaker. A burette, fitted with a retort stand clip, was filled with 25 ml of hydrobromic acid solution. The timing for oxirane oxygen content (OOC) measurement began once 0.5 g of amberlite was added to the solution and the temperature reached 70 °C. Samples were collected every 5 min from the beaker using a syringe, with 2 g transferred into a smaller beaker for titration. To stop the reaction, 10 ml of glacial acetic acid and a drop of crystal violet solution were added, followed by gentle stirring for a brief period. Hydrobromic acid was then added to the solution and titrated until the endpoint – a yellowish-green colour – was observed.

*Table 1* – Initial experimental conditions

| Parameters | Conditions |
|---|---|
| temperature | 70 °C |
| stirring speed | 350 rpm |
| molar ratio of palm oil : formic acid : hydrogen peroxide | 1 : 1 : 1 |

### 2.3 Analytical procedure

The process involved taking samples every 5 min to measure the oxirane oxygen content (OOC), which was then used to compute the relative conversion to oxirane (RCO%) based on both the experimental oxirane oxygen content ($OOC_{exp}$), determined through titration, and the theoretical oxirane oxygen content ($OOC_{theo}$). Formulas for calculating RCO, $OOC_{theo}$, and $OOC_{exp}$ are provided in Eqs. (1)–(3).

$$RCO = (OOC_{exp} / OOC_{theo}) \cdot 100 \quad (1)$$

$$OOC_{theo} = \{(X_0 / A_i)/ [100 + (X_0 / (2A_i)) (A_0)]\} \cdot A_0 \cdot 100 \quad (2)$$

$$OOC_{exp} = 1.6 \cdot N \cdot (V-B) / M \quad (3)$$

$N$ is the HBr normality, $B$ is the volume of the HBr solution used for the blank in millilitres, $V$ is the volume of the HBr solution used for titration, $M$ is the sample weight, and $X_0$ is the preliminary iodine value. $A_i$ is the iodine molar mass and $A_o$ is the oxygen molar mass.

### 2.4 Fourier transform infrared (FTIR) analysis

Fourier transform infrared (FTIR) spectroscopy is a valuable technique for identifying both organic and inorganic functional groups in a sample. In this study, FTIR analysis was conducted using a Spectrum One spectrometer (Perkin-Elmer, USA) to characterise the functional groups present in the raw palm oil and optimised epoxide. The analysis aimed to confirm the presence of epoxide groups and the disappearance of absorption peaks corresponding to =CH stretching vibrations, indicating successful epoxidation. The FTIR spectra were obtained using the Perkin Elmer spectrum and recorded in the range of 3900–700 $cm^{-1}$.

### 2.5 Numerical kinetic modelling

Simulations were conducted using MATLAB Version IV (The MathWorks Inc., USA) to determine the reaction rate constants. This model is based on several key assumptions: it assumes that the epoxidation process occurs within a single phase, thus avoiding the need for distribution constants typically required for systems with separate aqueous and oil phases. It further assumes that the volume of each phase remains unchanged throughout the reaction. Additionally, the model considers all reactions to be homogeneous, as outlined in Eqs. (4) and (5).

$$HCOOH + H_2O_2 \underset{k_{12}}{\overset{k_{11}}{\rightleftarrows}} HCOOOH + H_2O \quad (4)$$

$$HCOOOH + \text{oleic acid} \xrightarrow{k_{21}} \text{epoxide} + HCOOH \quad (5)$$

A genetic algorithm was developed to solve ordinary differential equations (ODEs). This self-learning system oper-

ates in parallel and is well-suited to handling both linear and nonlinear equations. Genetic algorithms are pivotal in artificial intelligence and machine learning, particularly in optimisation and kinetic studies. The kinetic model for the epoxidation process and epoxide ring degradation can be formulated using rate constants such as $k_{11}$, $k_{12}$, and $k_{21}$. The model incorporated a set of simultaneous differential equations, as described in Eqs. (6)–(11).

$$\frac{d[HCOOH]}{dt} = -k_{11}[HCOOH][H_2O_2] + \\ + k_{12}[HCOOOH][H_2O] + k_{21}[HCOOOH][\text{oleic acid}] \quad (6)$$

$$\frac{d[H_2O_2]}{dt} = -k_{11}[HCOOH][H_2O_2] + k_{12}[HCOOOH][H_2O] \quad (7)$$

$$\frac{d[HCOOOH]}{dt} = k_{11}[HCOOH][H_2O_2] - \\ - k_{12}[HCOOOH][H_2O] - k_{21}[HCOOOH][\text{oleic acid}] \quad (8)$$

$$\frac{d[H_2O]}{dt} = k_{11}[HCOOH][H_2O_2] - k_{12}[HCOOOH][H_2O] \quad (9)$$

$$\frac{d[\text{oleic acid}]}{dt} = -k_{21}[HCOOOH][\text{oleic acid}] \quad (10)$$

$$\frac{d[\text{epoxide}]}{dt} = k_{21}[HCOOOH][\text{oleic acid}] \quad (11)$$

## 3 Results and discussion

### 3.1 Effect of different molar ratios of formic acid

Epoxide production was carried out using three different molar ratios of formic acid to palm oil: 0.5 : 1, 1 : 1, and 2 : 1, at constant temperature and an agitation speed of 350 rpm. Fig. 1 clearly shows that the highest amount of epoxidised palm oil was achieved at varying molar ratios of formic acid to palm oil. Specifically, at a 0.5 : 1 ratio, the maximum relative conversion to oxirane (RCO%) reached 33 % after 30 min of reaction time. For the 2 : 1 ratio, the RCO% peaked at 62 %, representing the highest conversion observed. Meanwhile, at a 1 : 1 ratio, the RCO% reached a maximum of 45 %, also recorded at the 30-minute mark.

Furthermore, as the concentration of formic acid increased, both the reaction rate and the yield of epoxides increased. This was because a higher amount of formic acid leads to increase *in situ* formation of performic acid upon reaction with hydrogen peroxide, which acts as the actual epoxidising agent. The greater availability of performic acid accelerated the transfer of oxygen atoms to the double bonds in palm oil, thus enhancing epoxide formation. Similar observations were reported by *Sobhan et al.*,[15] where increased formic acid concentration led to improved epoxidation efficiency due to greater peracid availability. However, at a formic acid to palm oil molar ratio of 2 : 1, a slight decrease in epoxide yield was observed, suggesting that the higher concentration of formic acid leads to the breakdown of oxirane rings. While formic acid serves as a crucial oxygen carrier necessary for supplying sufficient epoxides in the aqueous phase, it also plays a significant role in the hydrolysis of oxirane rings.[16]

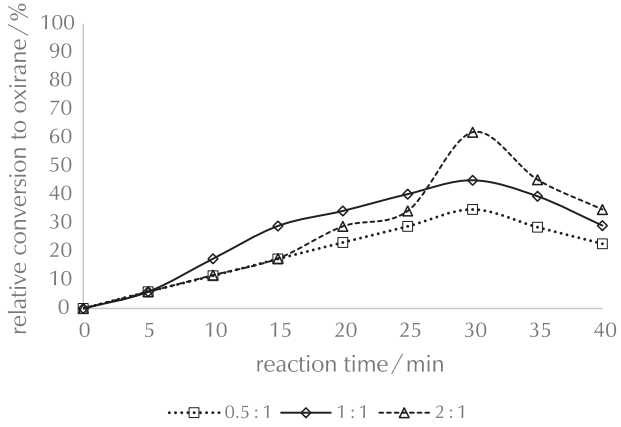



*Fig. 1* – RCO values for different molar ratios of formic acid and palm oil

### 3.2 Effect of different agitation speeds

Epoxidation experiments were conducted at three agitation speeds: 150 rpm, 250 rpm, and 350 rpm at a temperature of 70 °C. According to Fig. 2, the RCO percentage increased with reaction time and reached a maximum at the 30-minute mark for all agitation speeds. At 150 rpm, the maximum RCO% was approximately 32 %. At 250 rpm, the conversion increased further, peaking at around 45 %, while the highest RCO% was achieved at 350 rpm, reaching 68 % in 30 min. These results indicate that increasing agitation enhances the epoxidation efficiency, with 350 rpm producing the most significant oxirane conversion.

Based on the RCO% measurements, 350 rpm appeared to be the optimal stirring speed. The results are consistent with previous findings, which also identified 350 rpm as the optimum stirring speed for achieving high epoxidation efficiency in similar reaction systems.[17] This stirring speed likely enhances mass transfer between phases without causing excessive shear that could lead to side reactions or oxirane ring degradation. To fully understand this behaviour, it is necessary to assess the contributions of both primary (epoxidation) and secondary responses. Secondary responses may explain the decrease observed in RCO values. The epoxidation reaction rate rose when the formic acid or double bond molar ratio increased, and the secondary reaction increased the water content, compromising epoxy degradation selectivity. The formation of the oxirane ring in palm oil was accelerated by increasing the stirring speed from 150 to 350 rpm. The maximum RCO observed was 68 %, reached at a stirring speed of 350 rpm. It was assumed that under the given conditions, the reaction would not be limited by mass transfer resistance.[18] Therefore, subsequent investigations were conducted using a stirring speed of 350 rpm.

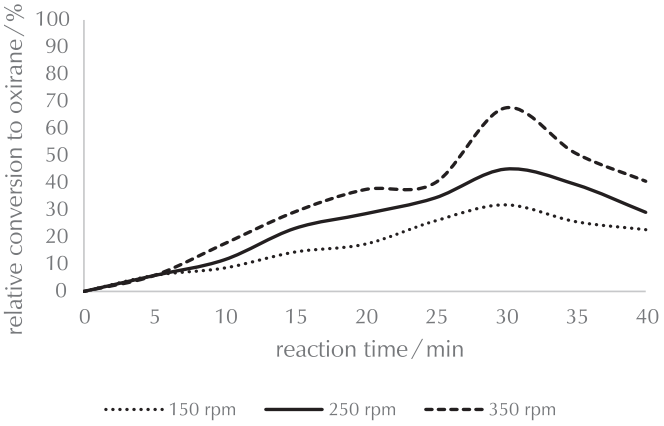



*Fig. 2* – RCO values for different agitation speeds

### 3.3 Fourier transform infrared (FTIR) spectroscopy

Fourier transform infrared (FTIR) spectra are essential for detecting and identifying characteristic groups within a material. Fig. 3 shows the FTIR spectra of palm oil and epoxidised palm oil under optimum conditions. The transformation of functional groups during the epoxidation process is evident from key changes in the spectral regions.

In the spectrum of raw palm oil, the distinct absorption peak observed around 1650 $cm^{-1}$, corresponds to the C=C stretching vibration of the alkene group (unsaturated fatty acid chains). This peak is characteristic of the olefinic double bonds present in triglycerides derived from vegetable oils.[8,12] After epoxidation, this peak significantly reduced in intensity, indicating the successful conversion of C=C double bonds into epoxide rings. In addition, the presence of a double band between 850 and 1300 $cm^{-1}$ indicated the existence of epoxy groups in the epoxidised palm oil, consistent with findings reported by other researchers.[19–21]

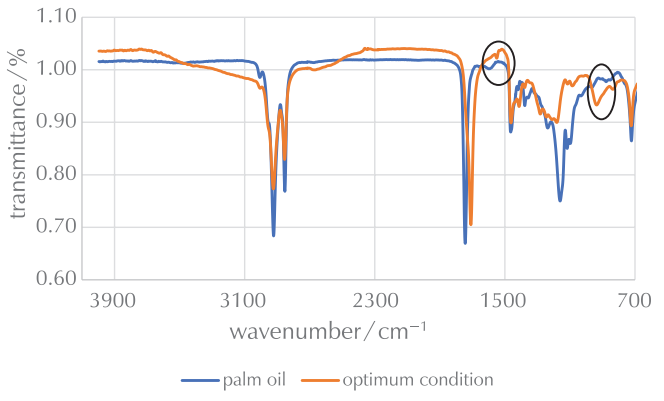



*Fig. 3* – FTIR analysis for optimum conditions and raw palm oil

### 3.4 Numerical modelling of epoxidation of palm oil

Simulations were conducted using MATLAB Version IV (The MathWorks Inc., USA) to determine the reaction rate constants, *k*. Kinetic studies of epoxidation are essential for understanding the reaction rates and the factors influencing them. In experimental studies of palm oil epoxidation, it was observed that the reaction between formic acid and hydrogen peroxide progressed slowly, resulting in the formation of epoxidised palm oil. This slow progression was attributed to the partitioning of formic acid between the oil phases and the insolubility of hydrogen peroxide in the oil. Table 2 reveals that the coefficient $k_{11}$ is notably lower than $k_{12}$, indicating that the latter represents a faster reaction. This suggests that controlling the loss of performic acid during the addition of oleic acid to the *in situ* formed performic acid, to produce an epoxide, accelerates the reaction rate while reducing the energy required for molecule interactions. Fig. 4 presents a comparison between simulation results and experimental data based on the kinetic reaction outlined in Table 2. Generally, the oxirane content increases during the initial 0–35 min of reaction time, before decreasing due to epoxide ring degradation. As expected, a minor discrepancy exists between the simulation and experimental data, which can be attributed to the assumption in the chemical equations that all reactions occur simultaneously.

*Table 2* – Rate constants for the epoxidation of palm oil under optimised process parameters

| Reaction rate constant | Value / $M^{-1}\,min^{-1}$ |
|---|---|
| $k_{11}$ | 0.057 |
| $k_{12}$ | 35.28 |
| $k_{21}$ | 4.95 |

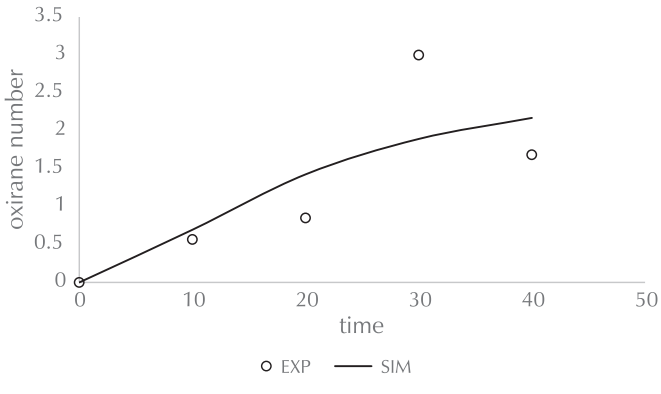



*Fig. 4* – Oxirane number between experimental and simulation

## 4 Conclusion

In conclusion, the highest relative conversion to oxirane – 68 % – was observed at a stirring speed of 350 rpm. Meanwhile, the highest conversion, at 62 %, was achieved with a 1 : 1 molar ratio of formic acid to palm oil. This study demonstrates that higher stirring speeds and optimal molar ratios of formic acid to palm oil led to increased relative conversion to oxirane. However, increasing the molar ratio of formic acid beyond a certain point may lead to degradation of oxirane rings. The investigation into the epoxidation of palm oil, along with the analysis of how thermal agitation speed and the molar ratio of formic acid to palm oil affect the relative conversion to oxirane (RCO) were successfully accomplished. These findings validate the feasibility of Amberlite as an alternative to conventional homogeneous acid catalysts, offering advantages such as ease of separation, reusability, and reduced environmental impact. The implications of these results extend to broader applications in the development of bio-based materials, particularly in the synthesis of bio-polyols and other epoxide-derived products used in polymers, coatings, and adhesives. The use of Amberlite catalyst aligns with green chemistry principles and supports the transition toward more environmentally friendly chemical processes. Moreover, this methodology may be adapted for the epoxidation of other unsaturated vegetable oils, especially those derived from non-edible or waste sources, thereby contributing to circular economy practices in the oleochemical industry.

### Ethics approval

Not applicable.

### Competing interests

The authors declare no competing interests.

### Author contribution:

Siti Mazlifah Ismail: Experimental work, Writing; Siti Nadia Abdullah: Writing; Nurul Izzah Md Zain: Experimental work, Siti Juwairiyah A Rahman: Writing; Mohd Jumain Jalil: Data curation, Writing; Intan Suhada Azmi: Conceptualisation and methodology.

### Availability of data and materials

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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## SAŽETAK

### Održiva epoksidacija nezasićenih masnih kiselina perkiselinskim mehanizmom s amberlitnom smolom kao katalizatorom

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Rastuća globalna tranzicija s neobnovljivih na obnovljive sirovine potaknula je značajan interes za održivim alternativama. Primjena amberlitne smole za kataliziranje proizvodnje epoksidiranog palminog ulja nedovoljno je istražena. Ova studija istražuje utjecaje promjene molarnog omjera mravlje kiseline i palminog ulja (0,5 : 1, 1 : 1 i 2 : 1) te različitih brzina miješanja (150, 250 i 350 okretaja u minuti) na rezultirajući udio oksirana. Nalazi pokazuju da molarni omjer 1 : 1 rezultira s 62 % oksirana unutar 30 minuta te nadmašuje rezultate drugih ispitanih omjera. Brzina miješanja od 350 okretaja u minuti proizvela je najviše oksirana (68 %). Da bi se povećala epoksidacija palminog ulja provedena je optimizacija procesa. Optimizacija je provedena kombinirajući metodu Runge-Kutta četvrtog reda s genetičkim algoritmom.

**Ključne riječi**
*Prekiselinski mehanizam, epoksidacija, sadržaj oksirana, palmino ulje, genetički algoritam*

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