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# Epoxidation of Oleic Acid Using Acetic Acid and Citric Acid as Hybrid Organic Oxygen Carriers

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**Abstract**

This study investigates the epoxidation of oleic acid employing a hybrid oxygen-carrier system consisting of acetic acid and citric acid. Conventional peracid-based epoxidation pathways often suffer from instability and undesirable side reactions, which restrict their broader application. To overcome these challenges, the influence of three key parameters, namely reaction temperature, hydrogen peroxide, and the oxygen carrier molar ratio, was systematically investigated. The results indicate that moderate reaction conditions (55–65 °C) enhanced oxirane formation and maintained epoxide stability, while both hydrogen peroxide and oxygen carriers at a stoichiometric ratio (1 : 1) achieved the highest epoxide yield of approximately 21 %. Increasing either parameter beyond the optimum promoted oxirane ring-opening and degradation reaction, leading to reduced conversion. A first-order kinetic model was established and optimised using MATLAB, and the predicted values showed excellent agreement with experimental data ($R^2 = 0.9927$), confirming the reliability of the developed model.

Overall, the use of acetic-citric acid hybrid oxygen carriers improved epoxide stability, minimised secondary reactions, and provided a greener alternative for producing high-quality epoxidised oleic acid with potential application in sustainable polymer and bio-based chemical industries.

**Keywords**

*Epoxidation, oleic acid, hybrid oxygen carrier, kinetic model*

## 1 Introduction

Epoxidised vegetable oils (EVOs) have attracted increasing attention as a sustainable alternative to petroleum-based intermediates because they are derived from renewable, sustainable resources and are environmentally friendly.[1,2] Owing to their high oxirane content and the reactivity of the epoxide functional group, EVO serves as a versatile intermediate for producing polyols, glycols, lubricants, and polymer plasticisers.[3–5] Industrially, epoxidation is commonly carried out using *in situ*-generated peracids formed from organic acids such as formic or acetic acid in the presence of hydrogen peroxide. During this process, the carbon-carbon double bonds of unsaturated fatty acid chains are converted into epoxide (oxirane) groups through electrophilic oxygen-transfer reactions.[6,7]

Despite their widespread application, conventional single-acid peracid systems often suffer from limited stability of the generated peroxycarboxylic acids and the formation of strongly acidic reaction environments, which promote undesirable oxirane ring-opening reactions and reduce epoxide selectivity. These limitations highlight the need for improved oxygen-carrier systems capable of providing controlled peracid generation while maintaining sufficient oxidative activity. Reaction parameters such as oxidant molar ratio, temperature, stirring rate, and catalyst loading are also known to strongly influence epoxide yield and selectivity, making systematic optimisation essential for achieving efficient epoxidation performance.[8,9]

Vegetable oils remain highly attractive substrates for epoxidation because of their wide availability, structural tenability, and favourable physicochemical properties, including high lubricity, biodegradability, and low toxicity.[10–13] Epoxide produced from these oils is useful for creating various fine compounds such as PVC plasticisers,[14] coatings,[15] and biolubricants.[16] In previous studies[16–18], epoxidation parameters have been investigated to maximise the relative conversion to oxirane (RCO) using vegetable oils as the feedstock. For instance, the epoxidation of jatropha oil has been reported to achieve a yield of approximately 87 %, whereas palm kernel oil produces about 85 % yield, and epoxidation of palm olein can reach epoxy yields of nearly 89 %.[19]

Conventional epoxidation of oleic acid often suffers from low selectivity and significant ring-opening side reactions when using single peracid systems. The instability of peracetic acid and reliance on strong mineral acids also limit process sustainability and industrial application. This study presents a hybrid oxygen carrier system combining acetic acid and citric acid, which can generate peracids more steadily while enhancing oxygen transfer. Therefore, the objectives of this study were (1) to determine the effects of oxygen carrier ratio, temperature, and hydrogen peroxide molar ratio, and (2) to develop a kinetic model for the epoxidation of oleic acid via an *in situ* hybrid oxygen carrier. The approach is novel in balancing reactivity and stability, reducing side reactions, and providing a greener alternative for high-yield epoxidation of oleic acid.

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## 2 Materials and method

### 2.1 Materials

Oleic acid ($C_{18}H_{34}O_2$) was used as the reactant for the epoxidation reaction and was supplied by Chemiz (M) Sdn. Bhd. Acetic acid (glacial) was supplied by R & M Chemical Sdn. Bhd., while hydrogen peroxide (50 %) and hydrobromic acid (48 %) were obtained from QReC Sdn. Bhd. Citric acid (95–97 %) was sourced from Merck Sdn. Bhd. All chemicals were of analytical grade and were used as received without further purification.

### 2.2 Experimental setup

The epoxidation of oleic acid (50 g, $MW = 282.46\ \mathrm{g\,mol^{-1}}$) was conducted in a 250-ml beaker equipped with a magnetic stirrer and thermometer, which was subsequently immersed in a thermostatic water bath. The oleic acid was first heated to 70 °C under continuous stirring at 300 rpm. A pre-prepared oxygen-carrier mixture consisting of acetic acid and citric acid at the desired molar ratio was then introduced into the reactor and allowed to homogenise with the substrate.

Hydrogen peroxide (50 wt%) was subsequently added dropwise at the required molar ratio relative to oleic acid while maintaining constant stirring to ensure controlled *in situ* formation of peracids and to prevent localised overheating. Evaporation losses were minimised by covering the beaker with perforated aluminium foil throughout the reaction, allowing safe pressure release while limiting exposure to ambient conditions. The reaction temperature was maintained at 70 °C for the specified reaction time to promote epoxidation while minimising hydrogen peroxide decomposition.

### 2.3 Determination of oxirane oxygen content (OOC)

The equation of relative conversion to oxirane (RCO) (Eq. (1)) incorporates both the theoretical OOC value (Eq. (2)) and the experimental OOC value (Eq. (3)).[20] The theoretical value was derived from the reaction stoichiometry, whereas the experimental value was determined from actual measurements of the reaction mixture. The experiments were performed in duplicate to improve the accuracy and reliability of the results.

$$\mathrm{RCO}(\%) = \frac{\mathrm{OOC_{exp}}}{\mathrm{OOC_{theor}}} \cdot 100 \tag{1}$$

$$\mathrm{OOC_{theor}} = \frac{\left(\frac{IV_0}{2A_i}\right)}{100 + \left(\frac{IV_0}{2A_i}\right)A_0} \cdot A_0 \cdot 100 \tag{2}$$

$$\mathrm{OOC_{exp}} = 1.6 \cdot N \cdot \frac{(V - B)}{W} \tag{3}$$

where $X_o$ is the initial iodine value, $A_i$ is the iodine molar mass, $A_o$ is the oxygen molar mass, $N$ is the HBr normality, $V$ is the volume of HBr solution used for the blank in ml, $V$ is the volume of HBr solution used for titration, and $W$ is the weight of epoxidised oleic acid.

### 2.4 Fourier-transform infrared spectroscopy (FTIR)

Fourier transform infrared (FTIR) spectroscopy was employed to identify the chemical functional groups present in the epoxide and polyol products formed during the epoxidation of oleic acid. FTIR spectra were recorded using a Perkin Elmer Spectrum 100 FTIR spectrometer over the wavenumber range of 4000–500 $\mathrm{cm^{-1}}$. For the analysis, 2 g of the reaction mixture was transferred into a clean vial, ensuring all residues were removed to prevent contamination. This technique allows the detection and comparative assessment of functional groups based on the characteristic adsorption bands in the sample spectra.

### 2.5 Kinetic modelling of the *in situ* epoxidation of oleic acid

The kinetic model for epoxidation assumed a single-phase reaction, constant phase volumes, and negligible heat transfer. It involved the *in situ* formation of peracids (PA) (Eq. (4)), and the formation of ESO (Eq. (5)), with ring opening shown in Eq. (6).

$$\mathrm{HO + HP} \underset{k_{12}}{\overset{k_{11}}{\rightleftarrows}} \mathrm{PA + water} \tag{4}$$

$$\mathrm{PA + OA} \xrightarrow{k_2} \mathrm{EOA + HA} \tag{5}$$

$$\mathrm{EOA + water} \xrightarrow{k_3} \mathrm{ring\ opening} \tag{6}$$

where HO, HP, OA, and EOA are acetic acid + citric acid, hydrogen peroxide, oleic acid, and epoxidised oleic acid. The rate equations were modelled, resulting in simultaneous differential Eqs. (7)–(13).

$$\frac{\mathrm{d[HO]}}{\mathrm{d}t} = -k_{11}[\mathrm{HO}][\mathrm{HP}] + k_{12}[\mathrm{PA}][\mathrm{water}] + k_2[\mathrm{PA}][\mathrm{OA}] \tag{7}$$

$$\frac{\mathrm{d[HP]}}{\mathrm{d}t} = -k_{11}[\mathrm{HO}][\mathrm{HP}] + k_{12}[\mathrm{PA}][\mathrm{water}] \tag{8}$$

$$\frac{\mathrm{d[PA]}}{\mathrm{d}t} = +k_{11}[\mathrm{HO}][\mathrm{HP}] - k_{12}[\mathrm{PA}][\mathrm{water}] - k_2[\mathrm{PA}][\mathrm{OA}] \tag{9}$$

$$\frac{\mathrm{d[water]}}{\mathrm{d}t} = +k_{11}[\mathrm{HO}][\mathrm{HP}] - k_{12}[\mathrm{PA}][\mathrm{water}] - k_3[\mathrm{EOA}][\mathrm{water}] \tag{10}$$

$$\frac{\mathrm{d[OA]}}{\mathrm{d}t} = -k_{21}[\mathrm{PA}][\mathrm{OA}] \tag{11}$$

$$\frac{d[EOA]}{dt} = +k_2[PA][OA] - k_3[EOA][water] \quad (12)$$

$$\frac{d[ring\ opening]}{dt} = +k_3[EOA][water] \quad (13)$$

## 3 Results and discussion

### 3.1 Effect of temperature

At 55 °C, the relative conversion to oxirane (RCO) increased steadily and reached its highest value of approximately 20 % at around 40 min before gradually declining, as shown in Fig. 1. The slower rate of increase suggests that the reaction was more controlled under this condition, with oxirane groups formed and preserved for a longer duration. This indicates that lower temperatures are beneficial for maintaining oxirane stability and delaying the onset of secondary reactions.[21] At 65 °C, the reaction trend was slightly different. Although the RCO increased rapidly in the early stage and reached approximately 15 % at around 30 min, the decline occurred earlier than at 55 °C. This behaviour reflects the higher susceptibility of the oxirane ring to decomposition at elevated temperatures. Ring-opening and hydrolysis reactions likely became more significant, reducing the overall epoxide yield despite the faster initial formation. At 75 °C, the effect of temperature was more pronounced. The RCO rose sharply during the first 30 min but then dropped drastically to below 10 % by the end of the reaction. This sharp decline suggests that, although higher temperatures accelerate epoxidation, they also promote side reactions that destabilise the oxirane ring. Overall, the results suggest that moderate conditions in the range of 55–65 °C provide a more favourable balance between oxirane generation and stability when acetic acid and citric acid are used as hybrid oxygen carriers. These findings are in good agreement with the observations reported in the previous study.[22,23]

### 3.2 Effect of hydrogen peroxide molar ratio

At the lower hydrogen peroxide ratio (HP 0.5), the relative conversion to oxirane (RCO) increased rapidly and reached a peak of nearly 20 % at around 30 min, as shown in Fig. 2. The RCO then declined sharply, reflecting the limited stability of the oxirane ring under these conditions. This pattern suggests that when hydrogen peroxide is present in smaller amounts, epoxidation occurs rapidly in the early stage; however, the imbalance between oxidant and substrate promotes ring-opening and other competing reactions once the maximum conversion is reached.

Under the stoichiometric condition (HP 1), the system behaved more steadily. The RCO continued to increase until about 40 min, reaching the highest value of approximately 20 %. The decline after this point was more gradual compared to HP 0.5, indicating that this condition provided a better balance between oxirane formation and degradation. These results demonstrate that the stoichiometric level of hydrogen peroxide is the most suitable for sustaining epoxidation activity when hybrid organic oxygen carriers are applied.[24]

At higher hydrogen peroxide loading (HP 1.5), the maximum RCO achieved was lower, at approximately 15 %. This reduced yield can be attributed to the excess oxidant, which is known to accelerate side reactions such as oxirane ring cleavage and the production of diols or glycols. In addition, surplus hydrogen peroxide tends to decompose more rapidly, generating reactive species that destabilise the epoxide structure. This outcome points to the autocatalytic nature of excess oxidants; higher concentrations do not enhance performance but instead compromise epoxide stability, thereby emphasising the need for precise control of oxidant levels.

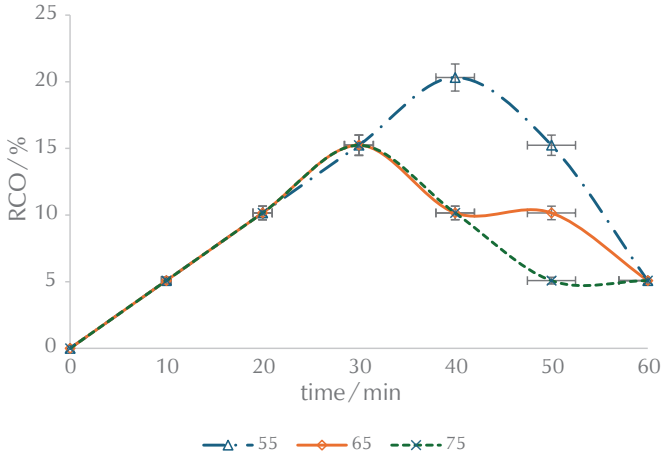


*Fig. 1* – Effect of reaction temperature (55, 65, and 75 °C) on the relative conversion to oxirane (RCO) during the epoxidation of oleic acid using acetic acid–citric acid hybrid oxygen carriers, molar ratio of oil : HP (1 : 1), and molar ratio of oil: OC (1 : 1)

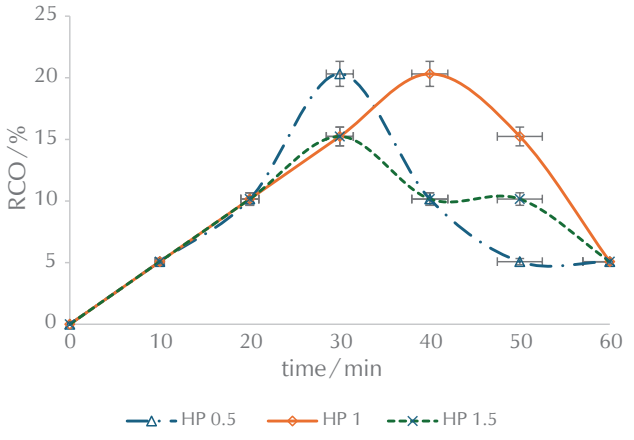


*Fig. 2* – Influence of hydrogen peroxide molar ratio (0.5, 1.0, and 1.5) on the relative conversion to oxirane (RCO) in the epoxidation of oleic acid with hybrid oxygen carriers, reaction temperature 65 °C, and molar ratio oil : OC (1 : 1)

### 3.3 Effect of hybrid oxygen carrier molar ratio

At the lower molar ratio (OC 0.5), the relative conversion to oxirane (RCO) rose gradually and reached approximately 10 % after 30 min before declining with time, as shown in Fig. 3. This shows that, although epoxidation occurred, the yield was restricted by the limited supply of the oxygen carrier. With less oxidant available, the generation of peracid was insufficient, and only a portion of the double bonds in oleic acid could be converted into oxirane groups. When the stoichiometric ratio was applied (OC 1), the reaction gave the best outcome. The RCO increased sharply, peaking close to 21 % around 30 min, followed by a slight decrease. This indicates that a balanced oxygen carrier ratio promotes efficient peracid formation, producing a higher epoxide yield and better oxirane stability.[25] The more controlled decline compared with OC 0.5 suggests that side reactions were minimised when the oxidant was well matched to the substrate.

At the highest ratio (OC 1.5), the RCO remained low throughout the reaction, never exceeding about 5 %. The suppressed performance at this level was likely due to an excessive amount of oxidant, which encouraged ring-opening and other degradation pathways. An oversupply of oxygen carriers can also cause faster hydrogen peroxide breakdown, generating reactive species that destabilise the epoxide. These results suggest that, while the oxygen carrier is crucial for epoxidation, excessive loading does not improve the process and instead reduces efficiency, making the stoichiometric ratio the most suitable condition.

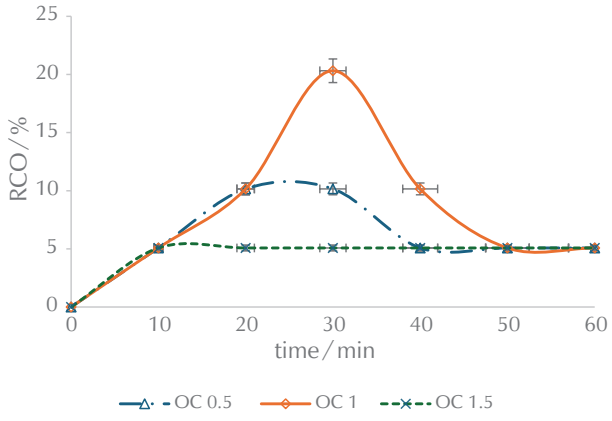


*Fig. 3* – Effect of oxygen carrier molar ratio (0.5, 1.0, and 1.5) on the relative conversion to oxirane (RCO) during the epoxidation of oleic acid using acetic acid–citric acid hybrid oxygen carriers, reaction temperature 65 °C, and molar ratio oil : HP (1 : 1)

### 3.4 FTIR spectra of oleic acid and epoxidised oleic acid

The FTIR spectra of oleic acid and its epoxidised product using acetic acid and citric acid as hybrid organic oxygen carriers provide clear evidence of epoxidation, as illustrated in Fig. 4. The disappearance of the olefinic bands in epoxidised oleic acid at ~3008 $cm^{-1}$ (=C−H) and ~1650 $cm^{-1}$ (C=C) indicates the consumption of double bonds, while the appearance of a new absorption band at ~800–900 $cm^{-1}$, corresponding to the asymmetric C−O−C stretching vibration of the oxirane ring, confirms the formation of epoxide. The carbonyl stretching band observed at ~1740 $cm^{-1}$ remained essentially unchanged, demonstrating that the carboxylic functionality was preserved throughout the reaction. Similar spectral features, particularly the disappearance of olefinic bands together with the appearance of oxirane-related absorption, have been consistently reported in recent studies,[13,18,19] further supporting the success of the epoxidation observed in this work.

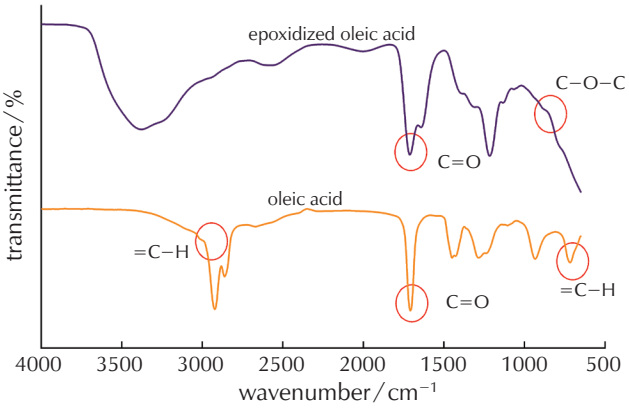


*Fig. 4* – FTIR spectra of oleic acid and epoxidised oleic acid

### 3.5 Kinetic model of epoxidation of oleic acid

Fig. 5 shows the model prediction of epoxy concentration as a function of time based on a first-order kinetic model, numerically solved and optimised in MATLAB using a hybrid optimisation approach (Genetic Algorithm + fmincon). The model prediction demonstrates excellent agreement with the experimental data, particularly at 0, 30, and 40 min, where the predicted and measured values nearly coincide. The maximum value at approximately 30 min represents the point of highest epoxide conversion rate, suggesting that this is the optimal reaction duration for achieving maximum epoxide yield under the studied conditions. The slight deviation observed at 50 min may be attributed to secondary reactions, catalyst deactivation, or minor experimental error.

The goodness-of-fit statistics further validate the accuracy of the model: SSE = $1.11 \cdot 10^{-4}$, RMSE = $5.28 \cdot 10^{-3}$, and $R^2$ = 0.9927, confirming that over 99 % of the variation in the data is captured by the first-order kinetic model. Figs. 6 and 7 present the residual analysis, combining residuals *versus* time and residual distribution. The residual values remain very low (within ± 0.01) and are randomly scattered around zero, indicating the absence of systematic bias. The distribution of residuals is nearly symmetric and centred at zero, which confirms that model errors are random and homoscedastic. The largest positive residual appears at 40 min (the model slightly underestimates epoxy concentration), while the negative residual at 50 min suggests a slight overestimation at longer times.

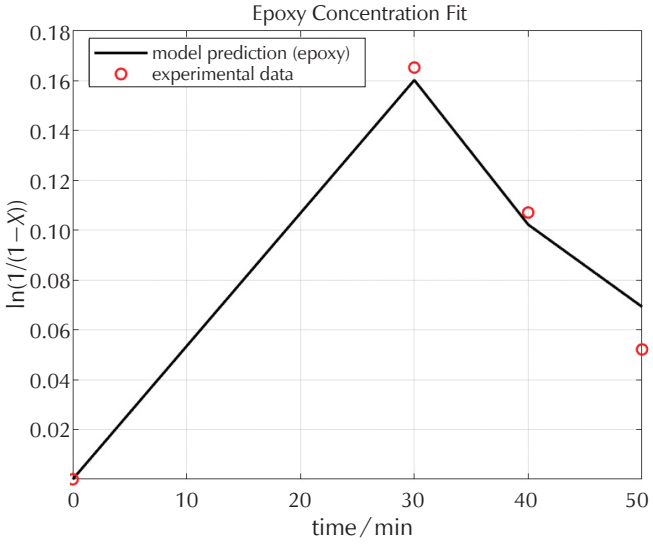


*Fig. 5* – Model prediction of epoxy concentration versus time using first-order kinetic numerical modelling in MATLAB (GA + fmincon optimisation) compared with experimental data

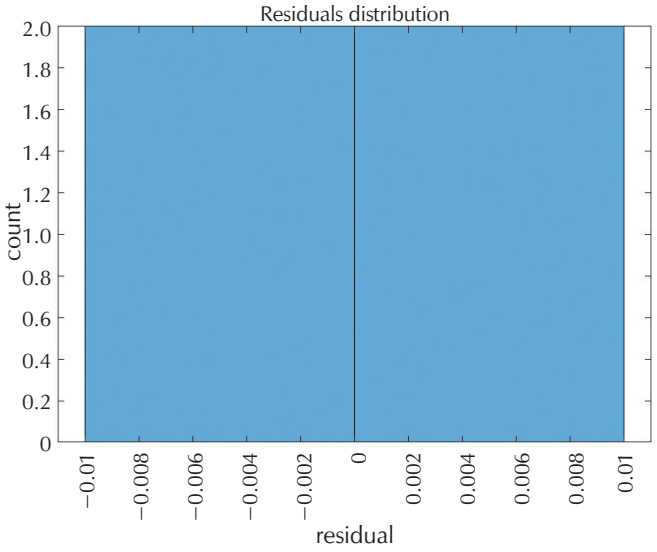


*Fig. 7* – Residual distribution demonstrating near-symmetry and absence of systematic bias

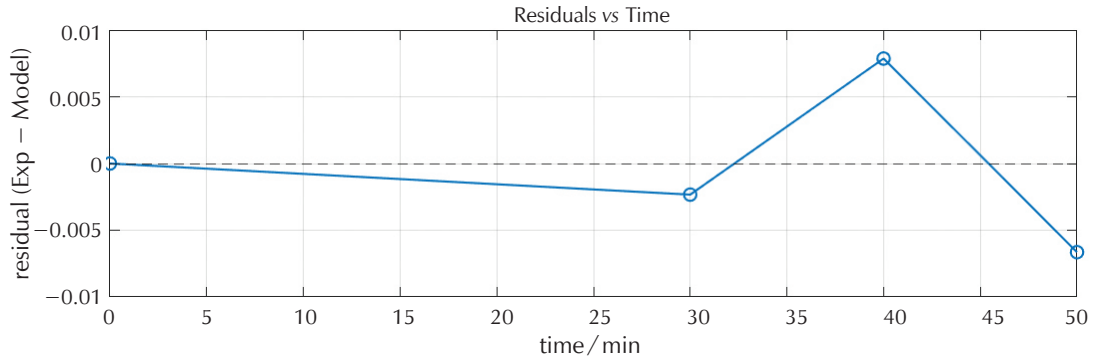


*Fig. 6* – Residuals *versus* time for the first-order kinetic model showing random scatter around zero

## 4 Conclusion

The results demonstrate that the combination of acetic and citric acids functions effectively as a hybrid oxygen-carrier system for epoxidation of oleic acid. The optimal condition was achieved at 55–65 °C with equimolar hydrogen peroxide and oxygen carrier ratios, producing the highest oxirane conversion at 21 %. Deviation from these optimal conditions, particularly the use of excess oxidant or oxygen carrier, promoted side reactions that destabilised the oxirane ring and subsequently reduced reaction efficiency. The proposed first-order kinetic model showed good agreement with the experimental results, supporting the validity of the reaction mechanism and predictive capability of the kinetic model.

Overall, the hybrid oxygen-carrier system provides a more balanced control between oxidative reactivity and epoxide stability compared with the conventional single-acid system, making it a promising pathway for greener epoxidation processes. The present study focuses on oleic acid as a model compound to systematically evaluate the reaction kinetics and the hybrid oxygen-carrier behaviour, while investigations involving different vegetable oil derivatives will be pursued in future works to assess the broader applicability of the proposed system. The implementation of this approach may contribute to sustainable production of renewable intermediates for polymers, coatings, and other bio-based chemical materials.

### Availability of data and materials

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

### Authors' contributions

Nur Azmina Asyiqin Binti Norazmi: Experimental work and manuscript drafting. Intan Suhada Azmi and Mohammad Aathif Addli: Data curation. Mohd Jumain Jalil and Intan Suhada Azmi: Conceptualisation and methodology. Aishah

Derahman, Mohd Jumain Jalil and Mohammad 'Aathif Addli: Review and editing of the manuscript.

### Competing interests

The authors declare no competing interests.

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## SAŽETAK

### Epoksidacija oleinske kiseline primjenom octene i limunske kiseline kao hibridnih organskih prijenosnika kisika

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U ovom radu istražena je epoksidacija oleinske kiseline primjenom hibridnog sustava prijenosnika kisika koji se sastoji od octene i limunske kiseline. Konvencionalni postupci epoksidacije temeljeni na peroksikiselinama često su ograničeni njihovom nestabilnošću i pojavom neželjenih sporednih reakcija, što umanjuje mogućnost njihove šire primjene. Kako bi se prevladali navedeni nedostaci, sustavno je ispitan utjecaj triju ključnih procesnih parametara: reakcijske temperature, količine vodikova peroksida i molarnog omjera prijenosnika kisika. Rezultati su pokazali da umjereni reakcijski uvjeti (55 – 65 °C) pogoduju nastajanju oksiranskog prstena i očuvanju stabilnosti epoksida. Najveći prinos epoksida, približno 21 %, postignut je pri stehiometrijskom omjeru vodikova peroksida i prijenosnika kisika (1 : 1). Povećanje količine vodikova peroksida ili prijenosnika kisika iznad optimalnih vrijednosti potaknulo je otvaranje oksiranskog prstena i razgradne reakcije, što je rezultiralo smanjenjem stupnja konverzije. Za opis kinetike procesa razvijen je kinetički model prvoga reda, koji je optimiran primjenom programskog paketa MATLAB. Predviđene vrijednosti pokazale su izvrsno slaganje s eksperimentalnim rezultatima ($R^2$ = 0,9927), čime je potvrđena pouzdanost razvijenog modela.

Općenito, primjena hibridnog sustava prijenosnika kisika na bazi octene i limunske kiseline doprinijela je povećanju stabilnosti epoksida, smanjenju sekundarnih reakcija te predstavlja održiviju alternativu za proizvodnju visokokvalitetne epoksidirane oleinske kiseline s potencijalnom primjenom u proizvodnji održivih polimera i biobaziranih kemikalija.

**Ključne riječi**

*Epoksidacija, oleinska kiselina, hibridni prijenosnik kisika, kinetički model*

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