## Catalytic Epoxidation of Rice Bran Oil *via* an *In Situ* Generated Hybrid Peracids Mechanism

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#### Abstract

In this study, *in-situ* formed peracid was used to generate epoxidised rice bran oil. The following were found to be the ideal epoxidation reaction parameters: (1) 85 °C reaction temperature; (2) 0.8 g catalyst loading hybrid oxygen carrier; (3) 450 rpm string speed; and (4) 1.1 molar ratio of hydrogen peroxide to rice bran oil. Under these conditions, the highest oxirane content achieved was 49 %. A mathematical model was developed in MATLAB using the fourth-order Runge-Kutta method and simulated annealing optimisation to describe the reaction's kinetic behaviour accurately. By turning rice bran oil into a product with added value, this study helps reduce dependency on petroleum-based resources and offers important insights into reaction dynamics, which are crucial for industrial applications.

#### Keywords

Epoxidation, rice bran oil, oxirane content, biomass conversion

## 1 Introduction

The depletion of oil reserves coupled with rising global energy prices due to economic growth, has made energy consumption efficiency an increasingly challenging goal.<sup>1</sup> Additionally, petroleum serves as a primary source for hydrocarbon production.<sup>2</sup> These concerns have drawn researchers' attention to renewable resources for epoxide production by epoxidation of vegetable oil. Apart from being renewable, vegetable oils contain a high percentage of unsaturated fatty acids, such as oleic, linoleic, and linolenic acids, making them environmentally friendly and readily available at a lower cost.<sup>3</sup> The fatty acid composition of rice bran oil is crucial for the epoxidation process, as it determines the number of double bonds available for conversion into epoxy groups. Rice bran oil is known for its diverse fatty acid profile, which includes both saturated and unsaturated fatty acids.4

Epoxidations have greater reactivity towards highly substituted alkenes than hydrogenation reactions, which generally favour less-substituted alkenes.<sup>5</sup> In hydrogenations, selectivity arises from a mix of steric and thermodynamic factors. The mechanism of epoxidation in vegetable oils occurs chemically at the double bonds.<sup>6,7</sup> Many researchers focused on the production of polyols, the use as lubricants, or the use as intermediates to produce polyurethanes, have investigated the epoxide ring opening reaction of epoxidised vegetable oils.<sup>8,9</sup> Epoxidation converts an alkene into an epoxide, a three-membered ring with oxygen.<sup>10</sup> This reaction is significant in organic chemistry and industrial applications, such as the synthesis of epoxy resins and other valuable chemicals.<sup>11</sup> Carboxylic acids transfer oxygen from hydrogen peroxide to alkenes, forming epoxides. In this study, the peracid is synthesised in situ from carboxylic acid and hydrogen peroxide. While effective, the use of formic acid, particularly in oxidising environments, poses a risk of corrosion due to its strength as an acid. This can lead to degradation of materials and equipment used in the process, necessitating careful consideration and mitigation strategies.<sup>12</sup> To address the corrosion issue, a novel method has been proposed that involves using a hybrid oxygen carrier solution combining both formic acid and acetic acid. This hybrid approach aims to balance the benefits of both acids while minimising the corrosive effects associated with formic acid alone. Therefore, the objective of this study was to (1) investigate the effect of process parameters on the epoxidation of rice bran oil, and (2) to develop a kinetic model for epoxidation of rice bran oil with applied oxygen carrier.

## 2 Materials and methods

#### 2.1 Materials

The following materials were used in the epoxidation process: formic acid 75 %, 30 % hydrogen peroxide, crystal violet, 48 % hydrogen bromide, and 100 % glacial acetic acid for the titration. Crystal violet served as an indicator

KUI-14/2025 Original scientific paper Received October 18, 2024 Accepted March 19, 2025

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https://doi.org/10.15255/KUI.2024.045

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in titration to determine oxirane content. Amberlite was used as a catalyst during the epoxidation of rice bran oil, facilitating the reaction between formic acid, hydrogen peroxide, and the rice bran oil ensuring efficient oxirane ring formation. All the material were used as received and supplied by BT Sciences Sdn. Bhd.

#### 2.2 Epoxidation of rice bran oil

The epoxidation process was conducted by mixing 100 g of rice bran oil with formic acid in a 500-ml beaker under continuous stirring at 300 rpm. The mixture was then heated to a temperature of 65-85 °C. Once the temperature stabilised, hydrogen peroxide was introduced into the system dropwise, while maintaining specific molar ratios of hydrogen peroxide to rice bran oil (1 : 1, 2 : 1, and 3 : 1). The reaction time was recorded from the moment hydrogen peroxide addition began. The dropwise addition of hydrogen peroxide ensured controlled reaction kinetics and minimised the risk of side reactions. The molar ratio and reaction parameters were optimised to maximise oxirane content while maintaining process efficiency.

#### 2.3 Determination of relative conversion to oxirane

Relative conversion to oxirane (RCO) quantifies the epoxidation efficiency by comparing the amount of epoxide produced to the initial double bonds in the reactant.<sup>13,14</sup> The RCO equation incorporates both theoretical and empirical OOC values, as shown in Eqs. (1)–(3).

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

$$OOC_{theor} = \left\{ \frac{X_0}{A_i} / \left[ 100 + \frac{X_0}{2A_i} \cdot A_0 \right] \right\} \cdot A_0 \cdot 100$$
 (2)

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

where  $X_0$  is the initial iodine value,  $A_i$  is iodine molar mass,  $A_{0}$  is oxygen molar mass, N is HBr normality, V is HBr volume, and W is sample weight.

#### 2.4 Numerical kinetic model

There are several assumptions in this model, which are as stated in Eq. (4)–(6). The epoxidation occurs in a single phase with constant volume to avoid distribution between aqueous and oil phases.

$$\mathsf{HCOOH} + \mathsf{H}_2\mathsf{O}_2 \underset{k_{12}}{\overset{k_{11}}{\longleftrightarrow}} \mathsf{HCOOOH} + \mathsf{H}_2\mathsf{O} \tag{4}$$

$$HCOOOH+ RBO \xrightarrow{k_{21}} EPOXY+HCOOH$$
(5)

$$EPOXY+ H_2O \xrightarrow{k_{31}} DHSA$$
(6)

where, RBO, EPOXY, and DHSA stand for rice bran oil, epoxidised rice bran oil, and dihydroxystearic acid, respectively. The rate equations are modelled as a set of simultaneous differential equations in Eqs. (7)–(13).

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

$$+k_{12}[HCOOOH][H_2O] + k_{21}[HCOOOH][RBO]$$

$$\frac{d[H_2O_2]}{dt} = -k_{11}[HCOOH][H_2O_2] + k_{12}[HCOOOH][H_2O] (8)$$

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

$$-k_{12}[HCOOOH][H_2O] - k_{21}[HCOOOH][RBO]$$

$$\frac{d[H_2O]}{dt} = k_{11}[HCOOH][H_2O_2] - (10)$$

$$-k_{12}[HCOOOH][H_2O] - k_{31}[H_2O][RBO]$$

$$\frac{d[RBO]}{dt} = -k_{21}[HCOOOH][RBO] (11)$$

$$\frac{d[EPOXY]}{dt} = k_{21}[HCOOOH][RBO] - k_{31}[EPOXY][H_2O] (12)$$

$$\frac{dDHSA}{dt} = k_{31}[EPOXY][H_2O] (13)$$

### 3. Results and discussion

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#### 3.1 Effect of temperature

Based on Fig. 1, the RCO at 85 °C peaks at about 40 % after 400 min. At 75 °C, the RCO increases to about 30 %, while at 65 °C, it remains the lowest at around 28 %. The RCO at 85 °C begins to decline after 30 min, suggesting that oxirane rings may degrade at higher temperatures. After peaking at 85 °C within 20 to 30 min, the RCO declines sharply to about 10 % by the 50-minute mark, before gradually increasing to 15 % by 60 min. At 75 °C, the RCO exhibits a second peak around 40 min before declining and stabilising at 15–20 % by 60 min. At 65 °C, the RCO also shows a second peak at 40 min and then declines, remaining quite high at 30 % at 60 min. The drop in RCO at higher temperatures could be attributed to side reactions that cause the breakdown of oxirane rings.

The maximum RCO was achieved at 45 min across all temperature conditions, indicating the importance of reaction time in maximising RCO. However, the RCO percentage fluctuated throughout the 45 min reaction period due to the reversible nature of the formic acid reaction.<sup>15</sup> Higher temperatures accelerated these reversible reactions involving formic acid, leading to an increased epoxide degradation rate and shifting the reaction equilibrium position toward the reverse direction, as described by Le Chatelier's Principle.<sup>16</sup> This shift led to a decrease in the overall percentage of RCO.



*Fig.* 1 – Impact of temperature on RCO. Condition: stirring speed of 350 rpm, catalyst loading of 0.8 g, and hydrogen peroxide molar ratio of 1 : 1.

#### 3.2 Effect of stirring speed

To ensure that the epoxidation reaction occurs in a kinetically controlled regime and remains free from external mass transfer limitations, it is crucial to examine the effect of stirring speed on the percentage relative conversion to oxirane (RCO). Stirring speed plays a vital role in ensuring that the reaction proceeds without being hindered by external mass transfer limitations.<sup>17</sup> By varying the stirring speed, the point at which the reaction is free from these limitations can be identified, ensuring that the reaction kinetics are accurately measured. As shown in Fig. 2, higher stirring speeds, such as 450 rpm, lead to a rapid initial increase in RCO but also cause faster degradation, resulting in a lower final RCO. In contrast, a moderate stirring speed (350 rpm) provides a balanced approach, achieving both a high and prolonged RCO. At lower stirring speeds (250 rpm), the epoxidation process proceeds more slowly but steadily. Stirring speed also influences the optimal reaction time. At 350 rpm, the ideal reaction duration for achieving maximum RCO appears to be around 30 min. At 450 rpm, a shorter reaction time leads to an increased RCO, but faster degradation follows. Furthermore, higher



*Fig.* 2 – Impact of speed on RCO. Conditions: temperature of 85 °C, catalyst loading of 0.8 g, and hydrogen peroxide molar ratio of 1 : 1.

stirring speeds cause instability and accelerate epoxide ring degradation, indicating the need for precise control of both stirring duration and speed to optimise RCO without epoxide ring degradation.

#### 3.3 Effect of catalyst loading

As shown in Fig. 3, the RCO for 0.8 g of Amberlite increases from 0 % to approximately 15 % within the first 20 min. Following this, the RCO drops to around 1 % before gradually rising again to reach about 49 %. With 0.9 g of Amberlite, RCO rapidly reaches about 15 % in the first 20 minutes, showing a similarly high initial reaction rate as with 0.8 g. The RCO then remains relatively steady towards the end of the process. For the maximum amount of Amberlite used, 1.0 g, the RCO increases to about 24 % within the first 10 min. After an initial decline, the RCO gradually rises over the next 50 min before experiencing a 16 % drop thereafter. The effect of catalyst loading on a chemical reaction, such as the epoxidation of rice bran oil, can significantly influence the reaction rate, yield, and overall efficiency.<sup>18</sup>



*Fig.* 3 – Impact of catalyst loading on RCO. Conditions: temperature of 85 °C, stirring speed of 350 rpm, and hydrogen peroxide molar ratio of 1 : 1.

#### 3.4 Effect of the molar ratio of hydrogen (HP) peroxide to RB0

Fig. 4 demonstrates that the ratio of HP to RCO increased to 49 % within 30 min, at which point the ring opened and the epoxidation of rice bran oil began. Among the tested ratios, the 1 : 1 ratio of hydrogen peroxide to oil yields the best results for the epoxidation reaction. In contrast, the HP 1 : 1 ratio yielded the 2 : 1 and 3 : 1 ratios resulted in lower RCO values, as excess hydrogen peroxide will damage the ring opening. The HP 2 : 1 and HP 3 : 1 ratios show only modest increases in RCO at the 30-minute mark, rather than the more pronounced increase observed with HP 1:1. High concentrations of hydrogen peroxide are hazardous and can pose safety risks, such as oxidative burns or decomposition explosions. It is essential to carefully control the amount used in the reaction to ensure safety and prevent hazardous situations.<sup>19</sup>



Fig. 4 – Impact of hydrogen peroxide molar ratio on RCO. Conditions: temperature of 85 °C, stirring speed of 350 rpm, and catalyst loading of 0.8 g.

#### 3.5 Characterisation

Based on Fig. 5, the C=O stretching vibration is observed at approximately 1740 cm<sup>-1</sup>. The ester carbonyl groups found in triglycerides are indicated by this peak. The C-H stretching vibration was observed in the FTIR spectrum between approximately 2850 and 2920 cm<sup>-1</sup>. The symmetric and asymmetric stretching vibrations of methylene (CH<sub>2</sub>) groups are represented by these peaks. Oxirane ring formation was observed at peaks around 1250 and 950 cm<sup>-1</sup>.<sup>20</sup> The C=O stretching vibration was observed around 1740 cm<sup>-1</sup>, and due to the modification, a shift or variation in intensity may occur in the carbonyl stretching region. C–H stretching has peaks at around 2850 and 2920 cm<sup>-3</sup>, like raw rice bran oil. The analysis focused on detecting epoxide groups and the disappearance of =CHstretching peaks. Spectra were recorded in the mid-infrared range (400–4000 cm<sup>-1</sup>) using a PerkinElmer Spectrum One FTIR spectrometer.



Fig. 5 – FTIR spectrum for raw and epoxidised rice bran oil

#### 3.6 Numerical kinetic model

The reaction rates (k) obtained from the simulation are presented in Table 1. The reaction rate indicates how quickly a reaction occurs, with higher values showing faster kinetics.<sup>21</sup> Notably, the concentration of hydrogen peroxide emerges as the primary factor influencing the reaction rate across all samples.

Table 1 – Estimated reaction rate constants from simulations

Reaction rate constant	Value/mol I min <sup>-1</sup>
k <sub>11</sub>	0.012
	14.730
	29.142
	0.177
Correlation coefficient, $r = 0.9$	

Fig. 6 shows an initial increase in reaction rate from 0 to 15 min, followed by a decrease from 15 to 25 min. This pattern aligns with the expected behaviour of undergoing the degradation process of epoxidation after the initial reaction period. It is noted that there are slight differences between the simulation and experimental data. Simulation relies on chemical equations and reactions occurring simultaneously, while experimental data may involve various parameters and conditions that could influence the results differently.



*Fig.* 6 – Comparison of the oxirane content between simulation and experiment

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## 4 Conclusion

The optimum conditions for the epoxidation reaction were identified as follows: a reaction temperature of 85 °C, a hydrogen peroxide to rice bran oil unsaturation molar ratio of 1 : 1.5, 0.8 g catalyst loading, and 450 rpm agitation speed. Under these conditions, a maximum epoxide yield of 49 % was achieved. This optimisation not only enhances the economic viability of the process but also supports the long-term use of rice bran oil in the production of valuable industrial chemicals. The results highlight the potential of rice bran oil as a sustainable feedstock for producing bio-based epoxides, which can serve as precursors for biopolymers, plasticisers, and stabilisers in various industrial applications.

#### ACKNOWLEDGEMENTS

This work was supported by the Fundamental Research Grant Scheme, Ministry of Education Malaysia, Ref: FRGS/1/2023/TK09/UITM/03/2.

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## SAŽETAK

# Katalitička epoksidacija ulja rižinih mekinja pomoću *in situ* generiranih hibridnih peroksikiselina

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Za nastanak epoksidiranog ulja rižinih mekinja, u ovom je istraživanju upotrijebljena *in situ* generirana peroksikiselina. Određeni su optimalni uvjeti reakcije: (1) temperatura od 85 °C, (2) 0,8 g katalizatora, (3) brzina miješanja od 450 okr min<sup>-1</sup> te (4) molarni omjer vodikova peroksida i ulja rižinih mekinja 1 : 1. U tim uvjetima postignut je maksimalni sadržaj oksirana od 49 %. Da bi se točno opisala kinetika reakcije, razvijen je matematički model u MATLAB-u primjenjujući metodu Runge-Kutta četvrtog reda i optimizaciju simuliranim kaljenjem. Pretvorbom ulja rižinih mekinja u proizvod dodane vrijednosti, ovo istraživanje pridonosi smanjenju ovisnosti o naftnim derivatima te donosi važan uvid u dinamiku reakcije, što je iznimno bitno za industrijsku primjenu.

#### Ključne riječi

Epoksidacija, ulje rižinih mekinja, sadržaj oksirana, pretvorba biomase

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Izvorni znanstveni rad Prispjelo 18. listopada 2024. Prihvaćeno 19. ožujka 2025.