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Catalytic Epoxidation of Waste Palm Cooking Oil Using *In Situ* Generated Peracid and a Hybrid Catalyst System

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Abstract

This study investigates the catalytic epoxidation of waste palm cooking oil *via* an *in situ* generated peracid mechanism, employing a hybrid homogeneous catalyst system comprising sulphuric and nitric acid to enhance efficiency and sustainability. The research focuses on optimising key reaction parameters, which include temperature, catalyst loading, hydrogen peroxide-to-oleic acid molar ratio, and formic acid-to-oleic acid molar ratio. The optimised conditions identified were: (1) reaction temperature of 65 °C, yielding the highest oxirane content while minimising side reactions; (2) a catalyst loading of 0.5, achieving the best balance of conversion efficiency and reaction stability; (3) a hydrogen peroxide-to-oleic acid molar ratio of 2 : 1, maximising oxirane yield without promoting degradation reactions; and (4) a formic acid-to-oleic acid molar ratio of 1 : 1, providing the optimal acid concentration for efficient epoxidation. These findings support the potential of using waste-derived oils in biopolymer production, aligning with sustainable development and circular economy principles. This optimised approach enhances waste valorisation and contributes to the synthesis of eco-friendly materials.

Keywords

Epoxidation, waste cooking oil, oxirane content, hybrid catalyst, eco-friendly

1 Introduction

Waste palm cooking oil (WPCO) is a significant by-product generated in large quantities by the food industry, particularly in regions where palm oil is a staple, such as Southeast Asia. Improper disposal of WPCO poses serious environmental challenges, including water pollution and soil degradation, while representing a lost opportunity for material recovery and valorisation.[1,2] As WPCO contains triglycerides with unsaturated fatty acid chains, it is a promising feedstock for producing bio-based chemicals and materials, contributing to waste reduction and sustainable resource use. The conversion of WPCO into valuable products aligns with sustainable development goals by transforming waste into renewable resources for various industries.[3,4]

Epoxidation, a key chemical modification, introduces oxirane rings into unsaturated bonds in oils, converting them into epoxidised derivatives suitable for industrial applications.[5,6] Epoxidised oils are valuable as bio-based plasticisers, stabilisers, and precursors for polyols, which serve in producing environmentally friendly polymers and coatings.[7,8] Various epoxidation methods exist, including the use of peracetic acid, performic acid, and hydrogen peroxide. Among these, *in situ* generated peracid using hydrogen peroxide and an organic acid is preferred for its efficiency, safety, and reduced environmental impact.[9] This method not only enhances product selectivity but also minimises the formation of unwanted by-products, making it an ideal approach for sustainable chemical synthesis.[10,11]

Catalysts play a crucial role in the efficiency and selectivity of the epoxidation process; with strong mineral acids such as sulphuric acid being commonly used.[12] Sulphuric acid is highly effective in catalysing the *in situ* generation of peracids from hydrogen peroxide; however, its use presents notable challenges. Sulphuric acid's corrosive nature presents significant risks to equipment and personnel, requiring meticulous handling and disposal to mitigate potential environmental impacts.[13] Additionally, prolonged exposure to sulphuric acid can degrade equipment, increasing maintenance costs and complicating process sustainability. These disadvantages underscore the need for alternative or modified catalytic systems that retain catalytic efficiency while minimising corrosive risks.[13,14]

To address these challenges, this study proposes a hybrid homogeneous catalyst system that combines sulphuric acid with nitric acid for the epoxidation of WPCO. The incorporation of nitric acid aims to mitigate the corrosiveness of sulphuric acid, potentially reducing equipment degradation and improving overall process safety. Additionally, nitric acid enhances the oxidative environment, which can increase the efficiency of the epoxidation reaction while maintaining product quality. Hybrid catalysts that incorporate multiple acids such as sulphuric acid and hydrochloric acid[15] have shown potential for achieving process stability and reducing environmental impact without sacrificing catalytic performance, making them promising alternatives for sustainable chemical production. The novelty of this

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study lies in its development and application of a hybrid homogeneous catalyst for WPCO epoxidation, a concept not widely explored in current literature. By using a combination of sulphuric and nitric acids, this research aimed to optimise the balance between catalytic efficiency and environmental safety, contributing to greener industrial practices. The study's findings offer valuable insights into waste resource utilisation and underscore the potential of bio-based materials for reducing environmental footprints.

2 Materials and methodology

2.1 Materials

The following chemicals were sourced from Merck Sdn. Bhd. and used in this study: 30 % hydrogen peroxide (H_2O_2), 85 % formic acid (FA), 75 % oleic acid, 95 % sulphuric acid, glacial acetic acid, crystal violet solution, hydrogen bromide, and distilled water.

2.2 Epoxidation procedure

The epoxidation process was conducted in a 1-litre beaker with a stirring rate of 350 rpm. Initially, 100 g of waste palm cooking oil were combined with formic acid in specified molar ratios added simultaneously to a beaker and continuously stirred. The mixture was gradually heated to 70 °C, at which point hydrogen peroxide was added incrementally. The reaction time was measured from the initial addition of hydrogen peroxide, and 4-ml samples were taken every 10 min to minimise formic acid vaporisation and prevent runaway reactions. The oxirane oxygen content (OOC) was assessed following the AOCS Tentative Method Cd 9-57.[16] For each sample, 10 ml of acetic acid was added in an Erlenmeyer flask and shaken, followed by the addition of two drops of crystal violet indicator. The solution was titrated with hydrogen bromide until a stable bluish-green colour persisted for at least 30 s, as described in Eq. (1).

$$\mathrm{OOC}_{\mathrm{exp}} = 1.6 \cdot N \cdot \frac{(V-B)}{W} \tag{1}$$

N is the normality of HBr, *V* is the blank volume, *B* is the titration volume, and *W* is the weight of sample. The relative percentage conversion to oxirane content (RCO) can be calculated using Eqs. (2) and (3), where $\mathrm{OOC}_{\mathrm{exp}}$, $\mathrm{OOC}_{\mathrm{theo}}$, IV_{o}, A_{i}, A_{o} are experimental oxirane oxygen content, theoretical oxirane oxygen content, initial iodine value, molar mass of iodine and molar mass of oxygen, respectively.

$$\mathrm{RCO} = \frac{\mathrm{OOC}_{\mathrm{exp}}}{\mathrm{OOC}_{\mathrm{theo}}} \cdot 100\ \% \tag{2}$$

$$\mathrm{OOC}_{\mathrm{theo}} = \left[\frac{\frac{\mathrm{IV}_{\mathrm{o}}}{2A_{\mathrm{i}}}}{100 + \left(\frac{\mathrm{IV}_{\mathrm{o}}}{2A_{\mathrm{i}}} \right) A_{\mathrm{o}}} \right] A_{\mathrm{o}} \cdot 100 \tag{3}$$

3 Results and discussion

3.1 Effect of temperature on epoxidation of waste palm cooking oil

The impact of temperature on the epoxidation of waste palm cooking oil is evident from the observed trends in relative conversion to oxirane (RCO%) at 65 °C, 75 °C, and 85 °C. Initially, RCO% increased across all temperatures, reaching a peak before gradually declining. At 65 °C, RCO% peaked around 30 min, indicating an optimal balance between reaction rate and stability of the epoxide product. In contrast, higher temperatures of 75 °C and 85 °C resulted in lower maximum RCO% values, suggesting that elevated temperatures may not enhance conversion rates but instead promote side reactions or degradation processes.

These findings align with recent studies emphasising the critical role of temperature control in optimising epoxidation conditions. For instance, research on the epoxidation of soybean oil demonstrated that higher temperatures could lead to increased side reactions, reducing the overall efficiency of the process.[17] Similarly, a study on the epoxidation of cottonseed oil found that elevated temperatures accelerated ring-opening reactions, thereby decreasing the yield of the desired epoxide product.[18] Therefore, maintaining moderate temperatures, such as 65 °C, is crucial for maximising epoxide yield and minimising side reactions during the epoxidation of waste cooking oil. This approach ensures higher selectivity and product stability, as excessive heat may promote undesirable reactions that compromise the efficiency of the epoxidation process.

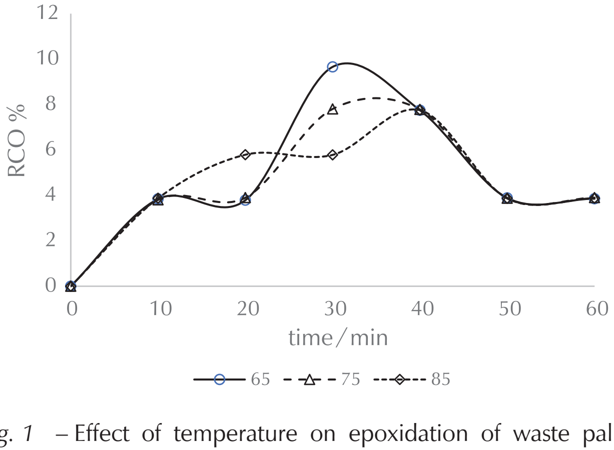

Fig. 1 – Effect of temperature on epoxidation of waste palm cooking oil

3.2 Effect of catalyst loading epoxidation of waste palm cooking oil

Fig. 2 illustrates the effect of various catalyst loadings (0.3, 0.5, and 0.7) on the relative conversion to oxirane (RCO%) over time in the epoxidation process. As seen, the RCO% increased initially for all catalyst loadings, reaching a peak before declining. The catalyst loading of 0.5 achieved the highest peak RCO% at around 30 min, suggesting that this loading level provided an optimal balance of catalytic activity, maximising conversion efficiency without promoting side reactions or deactivating the catalyst.

The decreased RCO% at both lower (0.3) and higher (0.7) catalyst loadings points to the need for optimisation in catalyst quantity to achieve desired epoxidation outcomes. Insufficient catalyst loading (0.3) may fail to drive the reaction efficiently, resulting in lower RCO% values due to a limited number of active sites. On the other hand, excessive catalyst loading (0.7) could lead to unintended side reactions, such as ring-opening reactions or polymerisation, as observed in other epoxidation studies.[19] High catalyst concentration can also cause deactivation over time due to the accumulation of by-products that block active sites, leading to reduced overall RCO%.

These findings underscore the importance of precise catalyst optimisation to balance reaction kinetics and selectivity, thereby maximising epoxidation yield. The optimal catalyst loading is critical to maintain a high conversion rate while avoiding side reactions or deactivation, as noted in recent studies on vegetable oil epoxidation.[20,19]

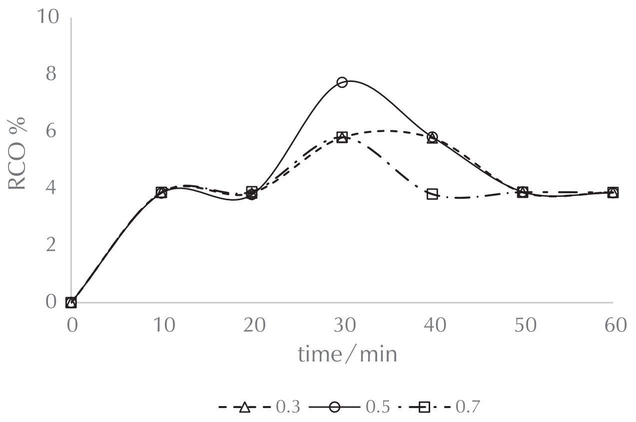

Fig. 2 – Effect of catalyst loading on epoxidation of waste palm cooking oil

3.3 Effect of hydrogen peroxide molar ratio on epoxidation of waste palm cooking oil

Fig. 3 displays the influence of different hydrogen peroxide-to-waste palm cooking oil molar ratios (1 : 1, 2 : 1, and 3 : 1) on the relative conversion of carbonyl oxygen (RCO%) over time during the epoxidation process. The 2 : 1 ratio achieved the highest peak RCO%, reaching approximately 10 % around the 25-minute mark, which suggested that this molar ratio provided an optimal balance for efficient epoxidation.

The lower performance at a 1 : 1 molar ratio indicated insufficient oxidising power, as seen in its gradual increase in RCO% without a prominent peak. This aligns with findings in recent studies, where a suboptimal molar ratio of oxidising agent limited the extent of epoxidation due to a shortage of available reactive oxygen species, leading to incomplete conversion.[21]

In contrast, the 3 : 1 molar ratio reached a peak earlier but declined rapidly, which may have been due to excess hydrogen peroxide. Excess oxidant can increase the likelihood of side reactions, such as ring-opening or polymerisation, as excess radicals or oxidative intermediates can destabilise the system.[22] This rapid decline in RCO% at higher oxidant levels has been documented in recent literature, where excess hydrogen peroxide can reduce product stability and lead to inefficient epoxidation.[23]

The findings underscore the importance of optimising the molar ratio to maximise epoxidation efficiency while minimising side reactions and maintaining stability. The 2 : 1 molar ratio emerged as the most favourable, delivering a high RCO% peak without rapid degradation, which is crucial for achieving high yields in an industrially viable process.

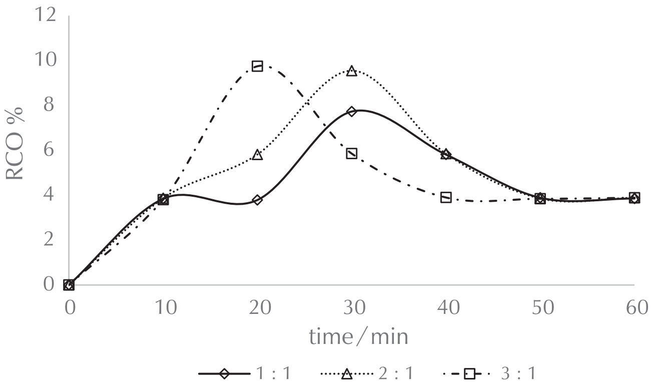

Fig. 3 – Effect of hydrogen peroxide molar ratio on epoxidation of waste palm cooking oil

3.4 Effect of formic acid molar ratio on epoxidation of waste palm cooking oil

Fig. 4 demonstrates the impact of different formic acid-to-waste palm cooking oil molar ratios (1 : 1, 2 : 1, and 3 : 1) on the relative conversion to oxirane (RCO%) over time in the epoxidation process. The data revealed that the 1 : 1 molar ratio achieved the highest peak RCO% at around 7 % at approximately 30 min, suggesting that this ratio may be optimal for maximising epoxidation efficiency.

The lower RCO% peaks observed at 2 : 1 and 3 : 1 molar ratio indicated potential limitations with higher formic acid concentrations. The 2 : 1 ratio resulted in a moderate peak but declined quickly, implying that it may not have provided stable reaction conditions. At a 3 : 1 ratio, the RCO% reached its peak earlier but dropped sharply, suggesting that excess formic acid could lead to instability in the reaction system. Excess formic acid can promote side reactions, such as ring-opening of the oxirane, which decreases the overall yield, a phenomenon observed in recent studies.[24]

The observed findings align with existing literature emphasising the critical role of formic acid as a co-reactant in epoxidation processes. Careful optimisation of its concentration is essential to prevent side reactions and ensure stability. For instance, research on the epoxidation of soybean oil with formic acid and hydrogen peroxide has shown that excessive formic acid can lead to side reactions, reducing the selectivity and stability of the epoxidised product.[25] Similarly, investigations into the epoxidation of vegetable oils have indicated that optimal formic acid concentrations are crucial for maximising epoxide yield while minimising by-products.[26] These studies underscore the importance of precise control over formic acid concentration to enhance the efficiency and selectivity of the epoxidation process.

Thus, the results indicated that a lower formic acid ratio (1 : 1) is effective for maximising epoxide yield, minimising side reactions, and ensuring reaction stability. This emphasizes the importance of precise control over formic acid concentration in epoxidation processes to optimise product quality.

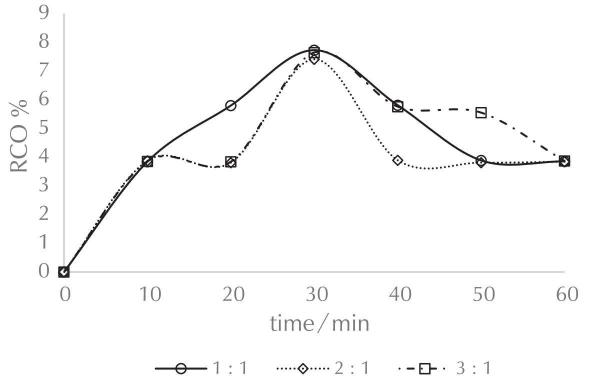

Fig. 4 – Effect of formic acid molar ratio on epoxidation of waste palm cooking oil

4 Conclusion

This study successfully optimised the epoxidation of waste palm cooking oil using an *in situ* peracid method with a hybrid catalyst system. The findings demonstrated that a reaction temperature of 65 °C, a catalyst loading of 0.5, a hydrogen peroxide-to-oleic acid molar ratio of 2 : 1, and a formic acid-to-oleic acid molar ratio of 1 : 1 yielded the highest oxirane content while minimising side reactions. These optimal conditions not only maximised conversion efficiency but also promoted reaction stability, making the process more viable for industrial applications. The study highlights the potential of utilising waste-derived oils for sustainable biopolymer production, aligning with the principles of waste valorisation and circular economy. This approach offers a promising pathway toward synthesising eco-friendly materials and supports the advancement of green chemistry in industrial applications.

Ethics approval

This study did not involve human and/ or animal subjects. No ethical approval was required.

Competing interests

The authors declare that they have no competing interests.

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Availability of data and materials

The datasets generated and/or analysed during the current study are available from the corresponding author upon reasonable request.

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SAŽETAK

Katalitička epoksidacija otpadnog palmina ulja za kuhanje primjenom *in situ* generiranog peroksikiselinskog sustava i hibridnog katalizatorskog sustava

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Ovo istraživanje ispituje katalitičku epoksidaciju otpadnog palmina ulja za kuhanje putem mehanizma *in situ* generirane peroksikiseline, primjenjujući hibridni homogeni katalitički sustav koji se sastoji od sumporne i dušične kiseline, s ciljem poboljšanja učinkovitosti i održivosti procesa. Istraživanje je usmjereno na optimizaciju ključnih reakcijskih parametara, uključujući temperaturu, količinu katalizatora, molarni omjer vodikov peroksid prema oleinskoj kiselini te molarni omjer mravlja kiselina prema oleinskoj kiselini. Određeni su optimalni uvjeti, (1) temperatura reakcije od 65 °C, pri kojoj se postiže najveći sadržaj oksirana uz minimalne nusreakcije, (2) količina katalizatora od 0,5, čime se ostvaruje ravnoteža između učinkovitosti pretvorbe i stabilnosti reakcije, (3) molarni omjer vodikov peroksid prema oleinskoj kiselini od 2 : 1, kojim se maksimizira prinos oksirana bez poticanja razgradnih reakcija, i (4) molarni omjer mravlja kiselina : oleinska kiselina = 1 : 1, koji osigurava optimalnu koncentraciju kiseline za učinkovitu epoksidaciju. Dobiveni rezultati potvrđuju potencijal uporabe ulja dobivenih iz otpada u proizvodnji biopolimera u skladu s principima održivog razvoja i kružnog gospodarstva. Taj optimizirani pristup pridonosi vrednovanju otpada i razvoju ekološki prihvatljivih materijala.

Ključne riječi

Epoksidacija, otpadno ulje za kuhanje, sadržaj oksirana, hibridni katalizator, ekološki prihvatljivo

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