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Glycerolysis of free fatty acids in red palm oil using potassium hydroxide as a homogeneous catalyst

Tata Shelvynar Meilinda[1], Yanna Syamsuddin[1], Muhammad Dani Supardan[1*]

[1] Department of Chemical Engineering, Universitas Syiah Kuala, Jalan Tengku Syech Abdur Rauf No. 7, Banda Aceh, Indonesia
* Corresponding author's e-mail: m.dani.supardan@usk.ac.id

ABSTRACT

Glycerolysis is an effective method for lowering free fatty acid (FFA) levels in vegetable oils, including red palm oil (RPO), thereby improving overall oil quality. This study examined the effects of key process variables—molar ratio of FFA to glycerol, reaction temperature, stirring speed, and catalyst loading—on the glycerolysis of RPO using potassium hydroxide (KOH) as a catalyst. Best conditions were achieved at an FFA-to-glycerol molar ratio of 1:4, a reaction temperature of 80 °C, a stirring speed of 300 rpm, and a catalyst loading of 0.7% (w/w oil), resulting in 96% FFA conversion within 90 minutes. Under these conditions, the FFA content was reduced from 13.69% to 0.55%. The glycerolysis process effectively lowers the FFA, peroxide, and saponification values of the oil, while having no significant impact on its density, viscosity, or fatty acid composition.

Keywords: free fatty acid, glycerolysis, potassium hydroxide, red palm oil.

INTRODUCTION

Red palm oil (RPO) is a potential raw material whose quality can be enhanced through glycerolysis. Unlike crude palm oil (CPO), RPO does not undergo a bleaching process, and the processing temperature is generally lower to preserve its nutritional components. Characterized by its distinctive red color, RPO is particularly rich in carotenoids and β-carotene (Gao et al., 2023). RPO has been utilized across diverse sectors, including health, skincare, and food (Sumarna et al., 2022). In the food industry, it has been reported to serve as an emulsifier (Gao et al., 2023), antioxidant (Qatrinada et al., 2021), and milk fat substitute (Zaini et al., 2023). Several countries have incorporated RPO into a wide range of food products, such as fortified biscuits, suji halwa, Indian besan laddhu sweets, spring rolls, pastries, donuts, curries, cakes, instant noodles, brown sugar, and dry bread. These applications highlight the importance of accurately determining the physicochemical properties and nutritional profile of RPO to support its broader utilization (Marliyati et al., 2021). Despite its high nutritional value, RPO is susceptible to thermal oxidation during heating or frying, resulting in the formation of degradation products such as aldehydes, ketones, acids, alcohols, and polymers, which reduce its quality and stability (Hidayati et al., 2025). The bioactive compounds, particularly carotenoids and vitamin E, are also unstable and sensitive to light and temperature, leading to significant nutrient loss during storage (Azhari et al., 2025). Elevated levels of free fatty acids (FFA) further accelerate oxidative deterioration, as FFAs easily oxidize into primary oxidation products, increasing the peroxide value and reducing shelf life (Kvangarsnes et al., 2021; Lin and Chiu, 2010). Therefore, reducing FFA content is crucial for improving the oxidative stability and preserving the nutritional quality of RPO, which contains valuable lipophilic compounds such as carotenoids, vitamin E, phytosterols, and polyunsaturated fatty acids (Teh et al., 2023). The improved oxidative stability and purity of RPO offer potential advantages for the food and cosmetics industries, where extended shelf life, safety, and product consistency are vital.

One promising approach for reducing FFA levels is glycerolysis, which is typically applied when the FFA content exceeds 5% (Mamtani et al., 2021). Compared with esterification, glycerolysis offers several advantages: it does not require an excess of alcohol, and the amount of base catalyst required is lower than the acid catalyst used in esterification. This process effectively reduces FFA while simultaneously increasing the glyceride fraction. In the glycerolysis process, glycerol directly interacts with FFA to generate glyceride compounds, which consequently enhance the quality of the oil (Elgharbawy et al., 2021). Both chemical and enzymatic glycerolysis are highly affected by key operating parameters, including temperature, reaction duration, catalyst type and concentration, the molar ratio of reactants, and the presence of solvents (Subroto et al., 2021). The adoption of glycerolysis for RPO is especially relevant to the sustainable engineering practices. This approach not only optimizes the quality of natural resources (such as palm oil) but also contributes to reducing the environmental footprint of the palm oil industry.

Multiple studies have demonstrated the efficacy of glycerolysis in reducing FFA content. Setyaningsih et al. (2021) investigated the glycerolysis process employing palm fatty acid distillate (PFAD) as the raw material, with para-toluenesulfonic acid (pTSA) serving as the catalytic agent. Under best conditions, the FFA content was successfully reduced from 81.67% to 6.36%. However, the use of an acid catalyst in glycerolysis has several drawbacks, such as the high cost of the catalyst, the excess glycerol required, difficulty in catalyst separation, corrosive properties, and the high reaction temperature. The high temperature also causes the resulting product to undesirable taste and color change can occur (Suriaini et al., 2021). A similar study was conducted by Panji et al. (2019), using CPO as the feedstock with free lipase enzyme catalyst and immobilized lipase enzyme. The main finding of this study was that the free lipase enzyme catalyst reduced the FFA content from 7.62% to 1.91%. Meanwhile, the use of immobilized lipase enzyme on $CaCO_3$ successfully lowered the FFA content from 2.30% to 2.24%. Nevertheless, the elevated cost of enzymes continues to present substantial challenges in attaining large-scale production (Wang et al., 2024).

As an alternative, the use of homogeneous base catalysts such as KOH and NaOH can be considered. Catalysts play a crucial role in glycerolysis reactions and in the conversion of FFA. Although several catalysts, including eggshells-derived CaO, have exhibited satisfactory performance in previous studies (Idris et al., 2019), whereas others, such as MgO, have shown minimal influence on the reaction (Ngatirah et al., 2024). Among these, KOH demonstrates distinct advantages owing to its low cost, high chemical stability, and excellent solubility in glycerol. Moreover, KOH exhibits strong catalytic activity, particularly in facilitating the abstraction of hydrogen atoms from the hydroxyl groups of alcohols such as glycerol, ethanol, and methanol (Elgharbawy et al., 2021). Accordingly, this study aims to evaluate the best conditions for the glycerolysis reaction in reducing FFA in RPO using KOH as a homogeneous catalyst, with particular emphasis on the influence of FFA to glycerol molar ratio, reaction temperature, stirring rate, and catalyst loading.

MATERIALS AND METHOD

Materials

RPO was sourced from Anugerah Kebun Ku, Lampung, Indonesia, and stored under standard laboratory conditions, containing a free fatty acid (FFA) level of 13.69%. Technical-grade glycerol (85%) and analytical-grade potassium hydroxide (99%) were obtained from Merck (Darmstadt, Germany).

Glycerolysis reaction

The glycerolysis reaction was performed in a 250 mL three-neck round-bottom flask fitted with a magnetic stirrer, condenser, and thermometer, as illustrated in Figure 1. A predetermined amount of glycerol was mixed with potassium hydroxide and stirred under heating to prepare the potassium-glycerol solution. Separately, RPO was preheated on a hotplate, after which the potassium-glycerol solution was added and the mixture was maintained under the designated reaction conditions. The response variable was FFA conversion, while the independent variables included the molar ratios of FFA to glycerol (1:2, 1:3, and 1:4), reaction temperature (60, 70, and 80 °C), stirring speed (200, 250, and 300 rpm), and catalyst loading (0.5, 0.7, and 1% w/w oil). Reaction samples were collected at 5 min during the first 15 min

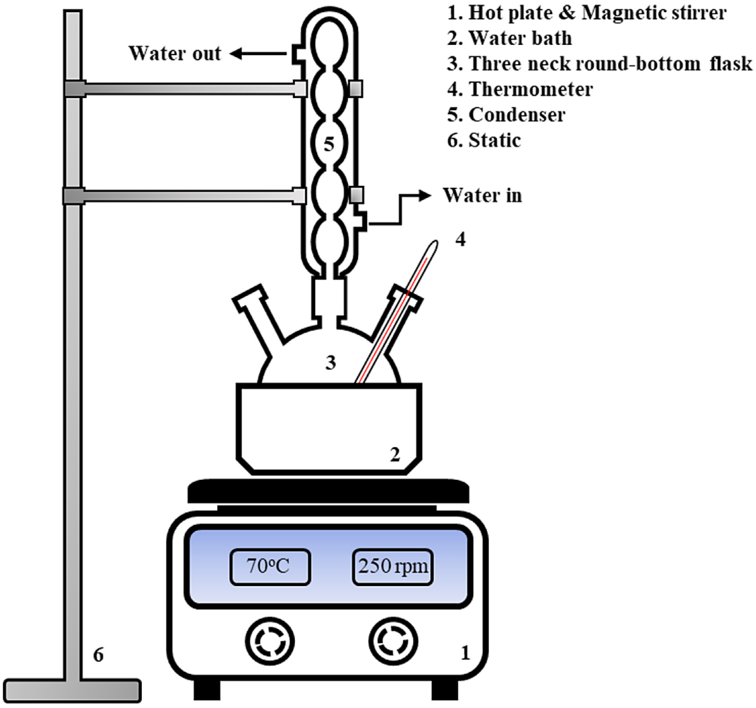

Figure 1. Experimental setup

and subsequently every 15 min until the reaction was terminated at 90 min. FFA conversion was subsequently determined for each sample.

The progress of the glycerolysis reaction under various experimental conditions was assessed based on FFA conversion. The FFA content of the samples was determined using an acid–base titration method, and the conversion was calculated from the change in acid value. The FFA conversion (X) was determined according to Equation 1.

$$X\,(\%) = \frac{[FFA]_0 - [FFA]_t}{[FFA]_0} \times 100 \quad (1)$$

where: $[FFA]_t$ is the concentration of FFA at time t (mg KOH/g), and $[FFA]_0$ – is the initial concentration of FFA (mg KOH/g).

Analysis method

The physicochemical properties of RPO—including density, peroxide value, saponification value, and FFA content—were determined following the official AOCS protocols (AOCS Cc 10c–95, Cd 8–53, Cd 3–25, and Ca 5a–40, respectively). Viscosity was measured at 40 °C using a ostwald viscometer (Middleboro, MA, USA). Carotenoid concentration was quantified by UV–Vis spectrophotometry, in which 25 mg of sample was dissolved in 10 mL of petroleum ether and scanned across 370–550 nm with a T80 spectrophotometer (PG Instruments, Wibtoft, UK). Color attributes (L*a*b*) of RPO were also assessed. The chemical composition of glycerolysis products was analyzed using gas chromatography–mass spectrometry (GC–MS, Shimadzu QP 2010 Plus, Japan) under the following conditions: split injection (1:20 ratio), DB5-MS capillary column (30 m × 0.25 mm ID, 1 μm film thickness, 5% phenyl–95% polydimethylsiloxane), helium as the carrier gas at 3 mL min^{-1}, and a programmed oven temperature of 50–180 °C (8 °C min^{-1}), 180–230 °C (5 °C min^{-1}), and 230–310 °C (20 °C min^{-1}), followed by a 15 min isothermal hold. Compound identification was achieved by correlating the mass spectra with entries from the NIST library, and quantification was expressed as relative peak area percentages using integrated data analysis software. Furthermore, FT-IR spectra of both upper and lower phases were collected at room temperature with a PerkinElmer UATR spectrometer in the 4000–500 cm^{-1} range.

RESULTS AND DISCUSSION

Effect of process parameters

The results of glycerolysis at different molar ratios of free fatty acid (FFA) to glycerol are presented in Figure 2. The conversion of FFA

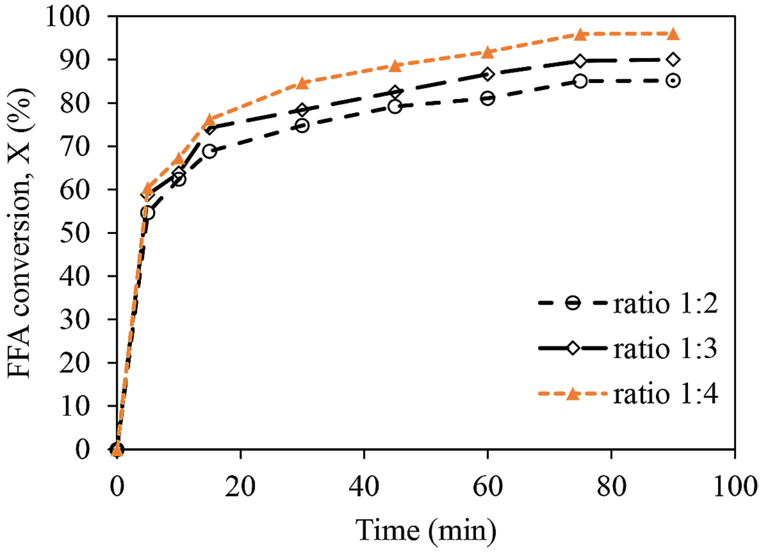

Figure 2. Effect of FFA to glycerol molar ratio on FFA conversion

gradually increased with reaction time and approached a near-equilibrium state at around 60 minutes. Comparable findings were reported by Suriaini et al. (2021), who demonstrated that increasing the molar ratio of glycerol to crude palm oil (CPO) significantly reduced FFA content. However, excess glycerol can impede the reaction progress due to an imbalance of reactants (Mamtami et al., 2021). According to Yencham et al. (2025), glycerolysis is a reversible reaction in which one mole of triglyceride reacts with two moles of glycerol to yield three moles of monoglyceride. While a slight excess of glycerol is required to drive the equilibrium toward product formation, an overly large amount should be avoided to ensure optimal reaction efficiency.

Figure 3 illustrates the effect of reaction temperature on FFA conversion a molar ratio FFA to glycerol of 1:4, a stirring speed of 300 rpm, and a catalyst loading of 0.7% (w/w of oil). After 90 minutes of reaction, the highest conversion was achieved at 80 °C, while the lowest conversion was observed at 60 °C. Notably, the conversions obtained at 70 °C and 80 °C were comparable. As suggested by Idris et al. (2021), when two temperatures result in similar conversions, the best condition can be determined based on energy efficiency and reaction duration. Accordingly, 70 °C was considered the most favorable

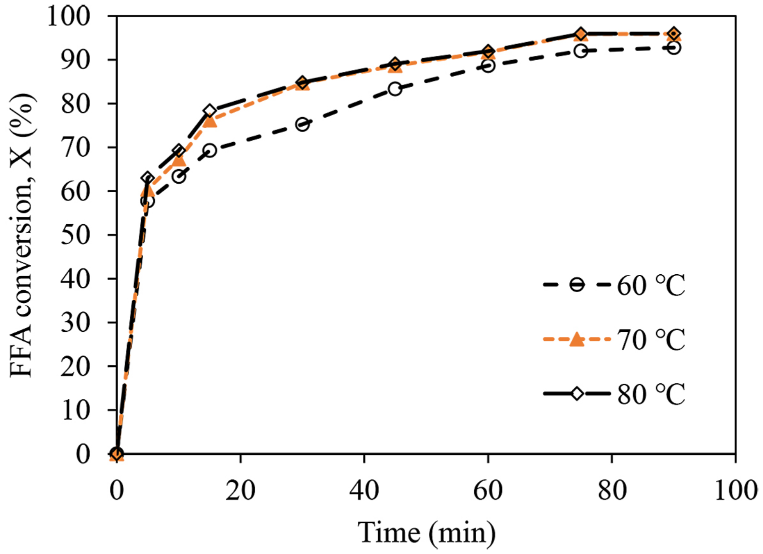

Figure 3. Effect of temperature on FFA conversion

reaction temperature. This finding is consistent with the results of Suriaini et al. (2021), who reported maximum FFA conversion from CPO at 75 °C. The observed effect of temperature can be explained by the behavior of RPO during heating. According to Hidayati et al. (2025), the FFA content in RPO decreases when the rate of FFA formation is slower than its degradation or conversion into volatile compounds. The presence of β-carotene, a natural antioxidant, also contributes to suppressing FFA formation during heating process. The unstable double bonds in the β-carotene structure render it highly reactive toward free radicals, thereby delaying FFA accumulation. However, the decomposition of β-carotene and the subsequent formation of volatile compounds under thermal conditions may accelerate FFA degradation. The glycerolysis reaction itself is time-dependent, and increasing the reaction temperature not only enhances the rate of conversion but also reduces the time required to achieve equilibrium (Chetpattananondh et al., 2024). More broadly, elevated temperatures accelerate reaction kinetics by facilitating the attainment of activation energy (Miyuranga et al., 2022).

High stirring speeds facilitate efficient liquid-phase mass transfer and ensure system homogeneity, thereby enhancing the effectiveness of reactant conversion (Muanruksa et al., 2021). Figure 4 illustrates the effect of stirring speeds of 200, 250, and 300 rpm on FFA conversion under fixed conditions of a 1:4 molar ratio, a reaction temperature of 70 °C, and a catalyst loading of 0.7% (w/w of oil). The results show that FFA conversion increased progressively with reaction time, with the highest conversion observed at 90 minutes when stirring at 300 rpm, whereas the lowest conversion at 90 minutes occurred at 200 rpm. Consistent with these findings, Miyuranga et al. (2022) reported that increasing the stirring speed to the range of 300–400 rpm in CPO significantly reduced FFA levels. In contrast, at 200 rpm, the reduction in FFA content occurred at a slower rate, particularly noticeable during the initial sampling at 5 minutes. The role of agitation is crucial, as it enhances the interfacial contact area between substrates, thereby improving mass transfer and accelerating the overall reaction rate (Subroto et al., 2021). Moreover, agitation increases molecular mobility between reactants, facilitating more effective collisions and promoting conversion (Suriaini et al., 2021).

Figure 5 presents the effect of varying catalyst loadings (0.5, 0.7, and 1% w/w oil). After 90 minutes of reaction, the highest FFA conversion was obtained with a catalyst loading of 0.7% (w/w oil), while the lowest conversion was observed at 0.5% (w/w oil). Similar findings were reported by Suriaini et al. (2021) in the glycerolysis of CPO, where a catalyst loading of 0.5% (w/w oil) resulted in the least reduction of FFA content. The enhancement in FFA conversion with higher catalyst loading is primarily attributed to the increased availability of active catalytic sites that promote the reaction (Suriaini, 2021).

As a homogeneous catalyst, KOH is considered more effective under relatively

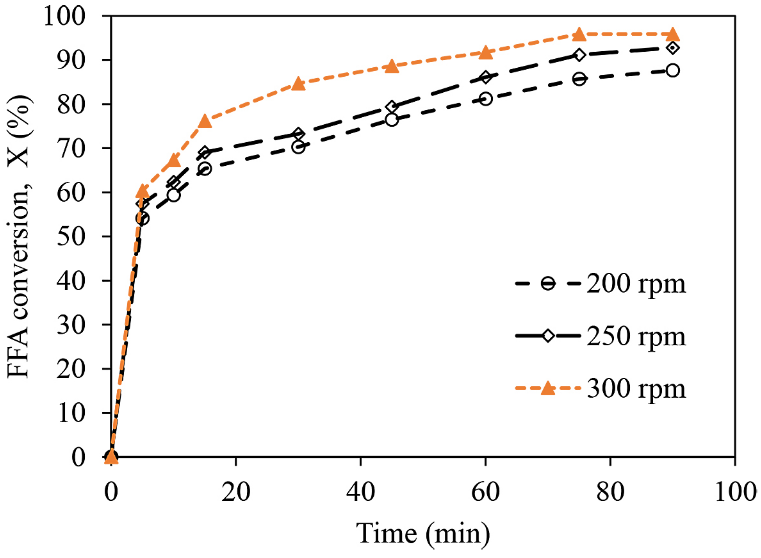

Figure 4. Effect of stirring speeds on FFA conversion

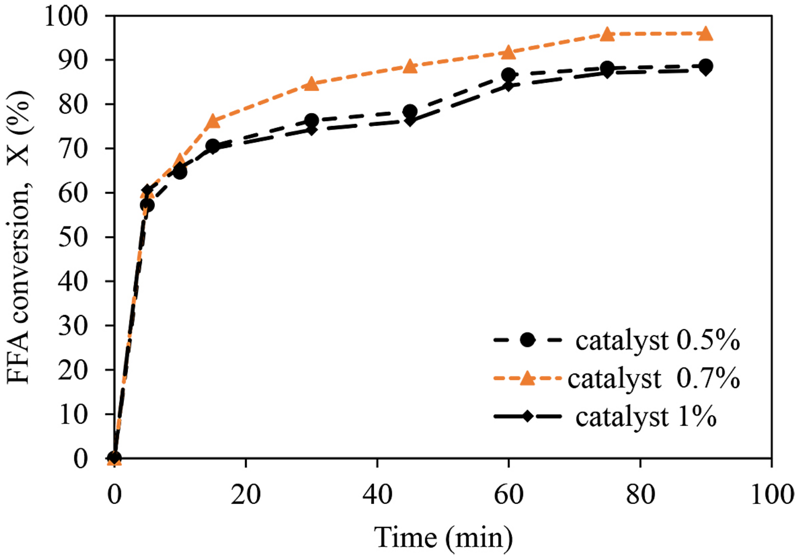

Figure 5. Effect of catalyst loading on FFA conversion

low-temperature conditions, making it well-suited for glycerolysis processes (Subroto et al., 2021). Additionally, KOH is capable of extracting hydrogen atoms from the hydroxyl groups of alcohols such as methanol, ethanol, and glycerol, thereby facilitating the reaction mechanism (Miyuranga et al., 2022). For comparison, Table 1 summarizes the FFA reduction achieved in glycerolysis reactions of fatty acids as reported by previous studies. However, the use of homogeneous KOH presents challenges in catalyst recovery and product purification, which may limit its scalability for industrial applications. To enhance its applicability, future research should explore the

Table 1. Comparison of operating conditions and FFA reduction in glycerolysis

Oil	Catalyst	Operating condition	FFA (%)			References
			Initial	Final	Conversion	
Red palm oil	KOH	Temperature = 80 °C FFA to glycerol = 1:4 Catalyst = 0.7 w/w% Reaction time = 90 min	13.69	0.55	96	This work
Palm fatty acid distillate	CaO derived from chicken eggshells	Temperature = 180 °C FFA to glycerol = 1:2 Catalyst = 1.5 w/t% Reaction time = 360 min	81	1.5	98	(Idris et al., 2021)
Palm fatty acid distillate	Commercial CaO	Temperature = 200 °C FFA to glycerol = 1:2 Catalyst = 1.5 w/t% Reaction time = 360 min	81	3.7	95	(Idris et al., 2021)
Crude palm oil	KOH	Temperature = 75 °C FFA to glycerol = 1:4 Catalyst = 0.75 w/t% Reaction time = 90 min	6.15	0.28	95	(Suriaini, Arpi, et al., 2021)
Low FFAs oil	KOH	Temperature = 65 °C FFA to glycerol = 1:2 Catalyst = 1.6 w/t% Reaction time = 20 min	7.6	0.13	98	(Elgharbawy et al., 2021)
Moderate FFAs oil	KOH	Temperature = 65 °C FFA to glycerol = 1:2 Catalyst = 1.6 w/t% Reaction time = 30 min	11.97	0.47	96	(Elgharbawy et al., 2021)
Palm fatty acid distillate	Para-toluene sulfonic acid	Temperature = 150 °C FFA to glycerol = 1:6 Catalyst = 1.5 w/t% Reaction time = 90 min	81.67	0.51	99	(Setyaningsih et al., 2021)

use of heterogeneous KOH-based catalysts and continuous-flow or fixed-bed reactor systems.

Physicochemical properties of red palm oil

The physicochemical properties of RPO are presented in Table 2. The results indicate that glycerolysis significantly influenced the FFA content, peroxide value, and saponification value of RPO. The observed decrease in peroxide value can be attributed to the high β-carotene content in RPO, which acts as a natural antioxidant and effectively inhibits the formation of peroxide compounds (Marliyati et al., 2021). In contrast, glycerolysis showed no significant effect on the density and viscosity of RPO, suggesting that these parameters remain relatively stable during the reaction.

Carotenoids are among the key compounds largely preserved during the specialized refining process, imparting the characteristic orange-red hue to RPO (Tan et al., 2021). Figure 6 shows a visual comparison between RPO and the glycerolyzed RPO. Overall, no notable changes in color were detected. In theory, the use of an alkaline catalyst in glycerolysis may reduce carotenoid levels, since alkaline conditions and high temperatures can accelerate the degradation of thermolabile pigments (Da Silva et al., 2021). Nevertheless, the present study shows that glycerolysis did not significantly decrease carotenoid content, likely due to the relatively mild reaction conditions at 80 °C. This result contrasts with the findings of Da Silva et al., (2021) on buriti oil, where base-catalyzed glycerolysis at 210 °C led to almost complete carotenoid degradation within only two hours of reaction. Color is a key factor influencing the visual attractiveness and acceptability of the oil. The color parameters were measured using the *Lab* color system, in which *L* represents lightness (0 = black, 100 = white), *a* corresponds to the red-green axis, and *b* indicates the yellow-blue axis (Satriana et al., 2024).

Table 2. Physicochemical properties of RPO

Property	Red palm oil	*After glycerolysis
Density (g/cm^3)	0.88	0.89
Viscosity (cP)	12.21	12.80
FFA content (%)	13.69	0.55
Peroxide value (meq O_2/kg)	2	4
Saponification value (mg KOH/g)	224.40	210.38
Caroten (ppm)	465.20	473.01
Color value		
L	40	43
a	45	44
b	54	52

Note: * molar ratio FFA to glycerol of 1:4, a reaction temperature of 80 °C, a stirring speed of 300 rpm, and a catalyst loading of 0.7%.

Gas chromatography mass spectra (GC-MS) analysis of red palm oil

Table 3 presents the fatty acid composition of RPO and its glycerolysis products. The compositions are relatively similar, indicating no significant change in fatty acid profiles after glycerolysis. During the reaction, glycerol reacts with FFA to produce monoglycerides, while the overall fatty acid profile of the oil remains unchanged. These findings are consistent with Marliyati et al. (2021), have reported that oleic acid constitutes the major fatty acid in RPO, with its fatty acid composition showing an almost equal distribution between saturated

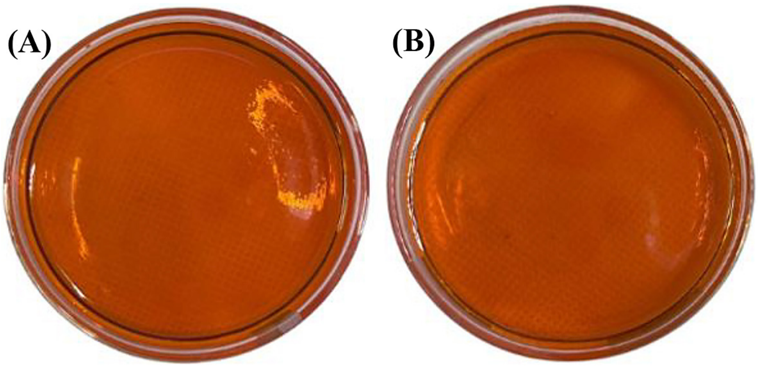

Figure 6. Visual aspect of the samples: (A) RPO; and (B) glycerolyzed RPO

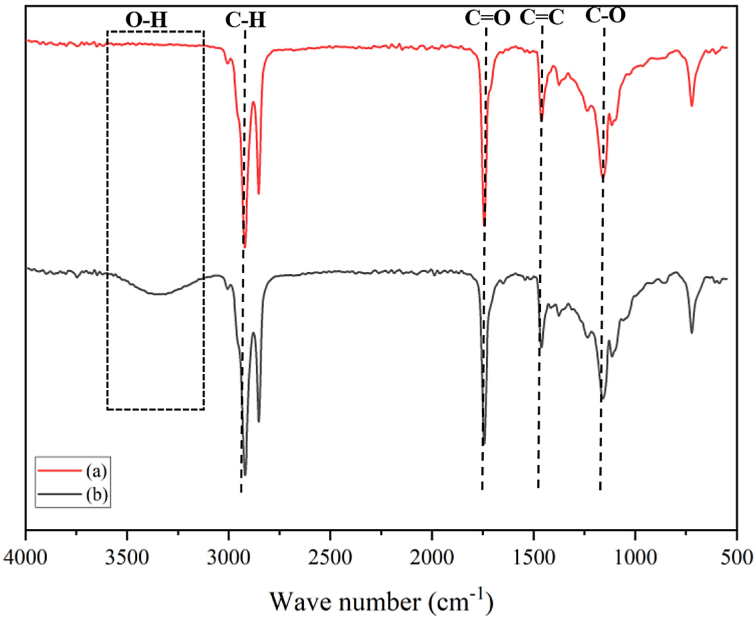

Figure 7. FTIR spectra of (a) RPO and (b) glycerolyzed RPO

Table 3. Fatty acid composition of the oil

Property	Red palm oil (%)	After glycerolysis* (%)
Lauric acid	0.13	1.6
Myristic acid	0.81	1.3
Palmitic acid	41.15	38.73
Palmitoleic acid	0.11	0.16
Stearic acid	3.54	3.54
Oleic acid	43.88	43.92
Linoleic acid	9.73	10.07
Linolenic acid	0.23	0.27
Arachidic acid	0.28	0.34
Gadoleic acid	0.11	0.08

Note: *molar ratio FFA to glycerol of 1:4, a reaction temperature of 80 °C, a stirring speed of 300 rpm, and a catalyst loading of 0.7%.

fractions (primarily palmitic acid) and unsaturated fractions (dominated by oleic acid). This balance contributes to the semi-solid nature of RPO at room temperature and boosts its resistance to lipid oxidation, making it more stable than other vegetable oils high in monounsaturated fatty acids (Tan et al., 2021).

Fourier transform infrared spectroscopy (FTIR) analysis of red palm oil

The FTIR spectra displaying the principal absorption bands and corresponding functional groups are presented in Figure 7.

The functional groups can be categorized into five main spectral regions based on their wavelength ranges: 1000–1300 cm^{-1} (C–O stretching), 1500–1650 cm^{-1} (C=C stretching), 1650–1800 cm^{-1} (C=O stretching), 2800–3000 cm^{-1} (C–H stretching), and 3000–3600 cm^{-1} (–OH stretching) (Satriana et al., 2024). The main functional groups identified with their corresponding wavenumbers include 3005 cm^{-1} for C=C bending, 2921 and 2852 cm^{-1} for symmetric and asymmetric CH_2 stretching, 1743 cm^{-1} for C=O stretching, 1461 cm^{-1} for CH_2 scissoring, 1159 cm^{-1} for C–O stretching, and 721 cm^{-1} for $(CH_2)_4$ rocking vibrations (Suriani et al., 2021). These groups were found in both RPO and glycerolyzed RPO, suggesting that the glycerolysis process preserves the main components of RPO and does not lead to the formation of undesirable compounds (Suriaini et al., 2021). The absorption band at 1711 cm^{-1} corresponds to the carbonyl group (C=O) of carboxylic acids. In glycerolyzed RPO, however, the intensity of this peak appeared weaker due to the reduced FFA content compared with crude RPO (Japir et al., 2017). Moreover, the broad absorption band at 3347 cm^{-1} corresponds to intermolecular hydrogen (–OH) stretching originating from the glycerolyzed RPO (Maulida et al., 2020).

CONCLUSIONS

In this study, the free fatty acid (FFA) content in red palm oil (RPO) was effectively reduced through the glycerolysis process using glycerol as a reactant. The application of a KOH base catalyst significantly accelerated the reaction between FFA and glycerol, enabling efficient conversion. Under best conditions a molar ratio FFA to glycerol of 1:4, a reaction temperature of 80 °C, a stirring speed of 300 rpm, and a catalyst loading of 0.7% (w/w of oil) the initial FFA content of 13.69% was successfully reduced to 0.55%. The experimental results further indicated that glycerolysis did not significantly affect the oil's density, viscosity, carotenoid content, or fatty acid composition. These findings demonstrate that glycerolysis is a promising and efficient method for lowering FFA levels in red palm oil, thereby improving its quality and potential applications. The process could be made more environmentally friendly and cost-effective by optimizing catalyst reuse, minimizing wastewater generation, and improving reactor design to reduce energy and operational costs in the industry.

Acknowledgments

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