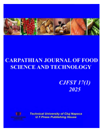

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Research article

CATALYTIC PERFORMANCE OF ACTIVATED MGO IN THE GLYCEROLYSIS OF PALM KERNEL OIL FOR TAG CONVERSION

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Abstract

The use of heterogeneous catalysts in synthesizing mono- and diacylglycerols has increased significantly, with metal oxides favoured for their thermal stability, large surface area, and easy purification. Activation is crucial for enhancing their reactivity and optimizing catalytic performance. This study aimed to evaluate the effect of the catalyst activation method in the glycerolysis reaction. Mono- and diacylglycerol (MDAG) products were synthesized via glycerolysis of coconut oil. The process used glycerol as the substrate, with a molar ratio of 1:2.3 and 2% magnesium oxide (MgO) as a catalyst. The optimal product was achieved at a reaction temperature of 175°C using water-impregnated MgO. This resulted in a triacylglycerol (TAG) conversion of 68.73%, a monoacylglycerol (MAG) content of 4.81%, a diacylglycerol (DAG) content of 26.46%, a slip melting point range of 22.8–24.7°C, and a free fatty acid (FFA) level of 0.78%.

1. Introduction

Coconut oil is a promising raw material for glycerolysis due to its high content of medium-chain fatty acids, particularly lauric acid, and its favorable physical and chemical properties (Swastawati et al., 2019). Its significant triglyceride content makes it an ideal candidate for conversion into mono- and diacylglycerols (MAGs and DAGs), valuable intermediates for food and pharmaceutical applications (Zha et al., 2014). Additionally, coconut oil offers potential health benefits, including antimicrobial properties and positive metabolic effects (Ngatirah et al., 2022). Glycerolysis of coconut oil produces products with desirable emulsifying and thickening properties, enhancing the functionality and nutritional value of food formulations, particularly in applications like ice cream and confectionery, thus improving texture and mouthfeel.

The glycerolysis reaction process is influenced by several factors, including the reaction temperature, the type and concentration of the catalyst, the molar ratio of glycerol and oil, the mixing intensity, and the reaction time (Kombe et al., 2013). The glycerolysis reaction can run fast in the presence of a catalyst. Catalysts that can be used are enzyme catalysts, base catalysts, and acid catalysts. Enzyme catalysts have the drawback that they cannot operate at high temperatures and are also costly (Al-Baarri et al., 2018; Wibawanti et al., 2021). The weakness of acid catalysts is the slow reaction rate, the catalyst is difficult to separate from the reaction, washing the catalyst causes contamination of the reaction products (Mazubert et al., 2013). Alkaline catalysts have advantages such as fast glycerolysis reaction rates, low prices, and easy to separate catalysts. In this study using heterogeneous base catalyst. Heterogeneous base catalysts are more advantageous because they are easily separated from the reaction products and can be used several times with almost the same efficiency (Faba et al., 2012).

Triana (2014) investigated MDAG synthesis via glycerolysis using FHPKO substrates with glycerol (mole ratios of 1:1.5 and 1:2.3) and a 1% NaOH catalyst, yielding MAGs of 47.72% and 47.02%. The 1:1.5 mole ratio substrate showed a 3.70% increase in MAG, while the 1:2.3 ratio showed a larger increase of 23.09%. Hapsari (2020) further optimized MDAG synthesis by using a blend of FHPKO and coconut oil, achieving the best results with a 1:2.3 mole ratio of FHPKO to glycerol and a 1.97% NaOH catalyst, which maximized MAG and DAG production, with no detectable TAG fraction. Ngatirah et al. (2022) studied MDAG synthesis from palm kernel olein and stearin using a 1:4 glycerol ratio, 40°C, and a 10% Lypozime RM IM as catalyst, yielding a final product with an HLB value of 5.92, and a melting point of 35.56°C.

Magnesium oxide (MgO) has emerged as a promising heterogeneous catalyst for glycerolysis due to its unique physicochemical properties and ability to facilitate transesterification (Belelli et al., 2015). As a solid base catalyst, MgO offers advantages such as high stability, ease of recovery, and reusability, making it attractive for industrial applications. Its high surface area and basicity enhance catalytic activity, promoting the efficient glycerolysis of triglycerides into valuable mono- and diacylglycerols (MAGs and DAGs) (Díez et al., 2011). Additionally, the solid-state nature of MgO allows for easy separation from the reaction mixture, enabling catalyst recovery and reuse in subsequent cycles. Moreover, the versatility of MgO as a catalyst can be enhanced through modification or activation methods, such as doping with other metal oxides or combining it with alkaline solutions (Manríquez-Ramírez et al., 2013a). Consequently, MgO is suitable for various glycerolysis applications, including biodiesel production and the synthesis of food emulsifiers and functional ingredients (Anggoro et al., 2019a; Rafati et al., 2019). This study aims to examine the effect of reaction temperature and different activated MgO catalysts on the conversion rate of TAG into MAG and DAG. In addition, this study also evaluated the effect of these factors on the FFA value and slip melting point.

2. Materials and methods

2.1. Materials

Magnesium oxide (MgO) was obtained from Merck KGaA (Darmstadt, Germany). Coconut oil was purchased from PT. Barco (Jakarta, Indonesia). Glycerol (technical grade) was obtained from P&G Chemicals (Kuala Lumpur, Malaysia). Hexane, ethyl acetate, KOH, benzoic acid, ethanol, and commasie brilliant blue (CBB) were obtained from Merck KGaA (Darmstadt, Germany). All these chemical materials were of analytical grade.

2.2. Methods

2.2.1. MgO Catalyst Preparation

The root powder (2 g) was extracted with the assistance of pectinase. Extraction of phenolic compounds was tested at different material/solvent ratios (1/5, 1/7, 1/9, 1/11 and 1/13, w/v), pectinase concentrations (0.1, 0.2, 0.3, 0.4 and 0.5%, v/w), extraction times (40, 60, 80, 100 and 120 minutes), pH (3.5, 4, 4.5, 5 and 5.5) and extraction temperatures (30, 40, 50, 60 and 70C). The mixture was filtered through Whatman No.4 filter paper in vacuum and TPC and AC were then analyzed. The glycerolysis process used three catalyst types: non-activated MgO, water-impregnated MgO ($MgO\text{-}H_2O$), and KOH-impregnated MgO (MgO-KOH). Catalyst activation involved suspending 10 g of MgO in 50 mL of distilled water for water impregnation, or in 50 mL of 2M KOH solution for KOH impregnation. Each mixture was heated and stirred at 80°C for 2 hours using a magnetic stirrer, and it was then filtered. The filtered catalysts were dried in an oven for 6 hours and subsequently calcined at 500°C for 4 hours (Manríquez-Ramírez et al., 2013b).

2.2.2. Coconut Oil Glycerolysis with Heterogeneous Catalysts

The glycerolysis reactions were carried out in a stirred tank reactor (STR). A substrate mixture of 100 g, containing coconut oil (CNO) and glycerol in a molar ratio of 1:2.3, was introduced into the STR and heated to 165°C and 175°C. Catalysts at 2% w/w of the oil weight were then added. The reaction was carried out for 6 hours at a stirring speed of 300 rpm using a magnetic bar. Samples were taken every 60 minutes to analyze the concentrations of monoacylglycerols (MAG), diacylglycerols (DAG), and triacylglycerols (TAG) throughout the reaction. Subsequently, the reaction product was cooled to 70°C. The product mixture was separated from glycerol and the catalyst via centrifugation at 2000 rpm for 10 minutes.

2.2.3. Characterization of Heterogeneous Catalysts

The functional groups of the catalyst granules were analyzed using Fourier Transform Infrared (FTIR) spectroscopy with a Perkin Elmer UATR (USA). X-ray diffraction (XRD) analysis was conducted using a Shimadzu XRD 7000 S/L (Japan) with Cu radiation at 30 kV and 30 mA, scanning samples in the range of $2\theta = 10–90°$ at a rate of 4°/min (Malyani, 2015). The analysis of catalyst basicity was conducted following the method of Rahul et al. (2011). A 1-gram sample of the catalyst was added to 10 mL of distilled water and stirred for 1 hour at 125 rpm using a magnetic stirrer. The catalyst was then filtered, and the resulting solution was mixed with a drop of phenolphthalein (PP) indicator until a pink color developed. Titration was performed using 0.01 M benzoic acid until the solution became colorless, and the titration volume was subsequently calculated.

2.2.4. Acylglycerol Concentration Analysis

The concentrations of MAG, DAG, and TAG in the product were analyzed using thin-layer chromatography (TLC) on G60 F254 plates. A developer solution mixture of hexane, ethyl acetate, and acetic acid in a ratio of 9:1:0.1 (v/v/v) was used. The TLC plate was activated by heating at 100°C for 1 hour, and then 1 µL of the sample was applied with a micropipette, ensuring a spot distance of 15 mm. The plate was placed in the TLC chamber until the developer reached the marked line, then air-dried at room temperature. Coomassie blue (0.02% w/v), dissolved in a mixture of acetic acid, methanol, and distilled water (1:3:6), was used for staining. The plate was scanned using a Camag Automatic TLC Scanner with WinCATS software at a wavelength of 629 nm, and MAG and DAG concentrations were quantified based on the peak area in the chromatogram.

2.2.5 Free Fatty Acid Analysis and Slip Melting Point

Free fatty acid analysis was performed according to AOCS Official Method Ca 5a-40 (2003), by calculating the percentage based on the dominant fatty acid in the sample (i.e. lauric acid). Slip melting point analysis using AOCS Cc 3-25 (AOCS, 2003) by inserting the sample into a capillary tube and kept in a freezer for 16 hours, then heated gradually (approximately 1°C per minute) until the sample melts completely.

3. Results and discussions

3.1. Characteristics of catalyst functional groups

FTIR spectroscopy was employed to characterize the functional groups present in the MgO catalysts. The spectral range analyzed spanned from 450 cm⁻¹ to 4000 cm⁻¹, as illustrated in Figure 1. The FTIR analysis revealed absorption peaks at 3699 cm⁻¹, corresponding to O-H groups (indicative of hydrogen bonding). These O-H groups are associated with various functional groups, including alcohols, carboxylic acids, esters, and ethers (Jeevanandam et al., 2018). The stretching of the O-H bonds occurs within a highly polarized hydrogen bond network. This O-H stretching is attributed to water that is physically adsorbed onto the catalyst (Nayebzadeh et al., 2017). The absorption peak at 1428 cm^{-1} shows the bending vibration of the OH bond (Moorthy et al., 2015). the peak at 450 cm^{-1} absorption region characterises the strain vibrations derived from Mg-O (Sutapa et al., 2018). The absorption peak of 671 cm^{-1} and 879 cm^{-1} indicates the presence of Mg-O and Mg-O-Mg vibrations, respectively (Kandiban et al., 2015).

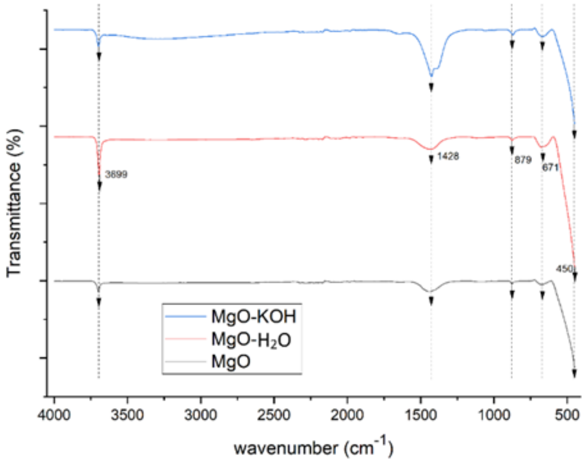

Figure 1. FTIR spectrum of activated MgO

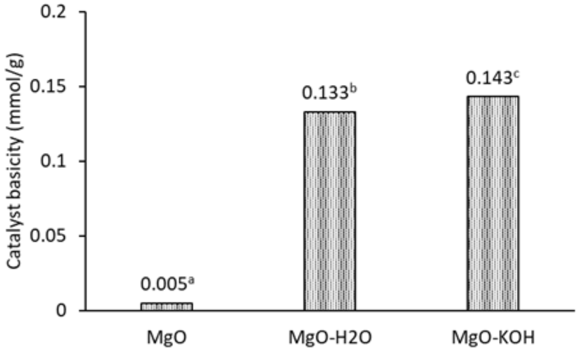

Figure 2. Basicity value of non-activated and activated MgO catalyst

Taslim et al. (2019) identified a peak at 887.26 cm⁻¹ as potassium due to KOH impregnation in natural zeolite, indicating potassium oxide presence. Similarly, the KOH-impregnated catalyst in this study likely shows a comparable peak. Munandar et al. (2014) associated the 770–803 cm⁻¹ range with K-O functional groups. The MgO-KOH catalyst displays a deeper spectral indentation than MgO and MgO-H_2O at 1428 cm⁻¹, likely due to an OH group, caused by hydroxyl group decomposition and adsorbed water. Strong hydrogen bonding by the OH and its presence suggests that MgO-

KOH is more basic than the other catalysts (Roni & Legiso, 2021).

3.2. Catalyst basicity

Catalyst alkalinity plays a crucial role in determining catalytic activity and can be enhanced through impregnation techniques. Figure 2 illustrates the basicity values for the activated MgO catalyst and their corresponding standards. These findings align with studies by Anggoro et al. (2019b), which reported that increasing the active metal content during impregnation, as well as optimizing calcination conditions, significantly enhances catalyst basicity and, consequently, its performance in catalytic reactions.

The incorporation of KOH into MgO increases the basicity of the catalyst. MgO-KOH exhibits a basicity of 0.143 mmol/g, higher than that of MgO-H_2O at 0.133 mmol/g and MgO alone at 0.005 mmol/g. Basic cations increase the electron density within the oxygen framework, thereby promoting its basic properties (Sahu et al., 2015). The basicity of the catalyst can be increased by impregnation, with KOH infiltration into the pores of the MgO catalyst increasing the alkalinity (Oko & Feri, 2019). Furthermore, catalyst basicity increases proportionally with the concentration of KOH applied during the impregnation process (Buchori et al., 2018).

Research by Anggoro et al. (2018) found that the addition of 2% Ca to MgO catalysts increased the basicity to 0.095 mmol/g, while a 3% Ca concentration resulted in a decrease in alkalinity to 0.035 mmol/g. This decrease in basicity at higher Ca concentrations may be due to oversaturation, where smaller Ca ions replace Mg ions within the crystal lattice. Lintang (2020) reported that MgO has a limited total basicity of 0.39 mmol/g, whereas MgO synthesised with H_2O has a higher total basicity of 2.05 mmol/g. At calcination temperatures below 900ºC, the OH group on the MgO surface remains partially intact, likely contributing to the weak base sites on the MgO-H_2O catalyst (Puriwat et al., 2010). This weak base site effect may explain the lower alkalinity of the MgO-H_2O catalyst compared to KOH.

3.3. Effect of using activated MgO catalyst on TAG conversion

The difference in TAG conversion values in the glycerolysis reaction using activated MgO catalyst and different reaction temperatures can be seen in Figure 3. During the glycerolysis process, TAG is converted to MAG and DAG. Triacylglycerol acts as a limiting reactant which will react with excess amounts of glycerol (Arwani, 2017). TAG conversion analysis that has been carried out produces a significant difference between the two temperature treatments ($P<0.05$). A temperature of 175ºC produces a higher triglyceride conversion than a temperature of 165ºC. A higher reaction temperature can decompose more TAGs (Mostafa et al., 2013). Elevated reaction temperatures increase the molecular transformation rate by providing atoms and molecules with greater kinetic energy. This energy boost leads to more frequent intermolecular collisions, making it easier for intermolecular bonds to break (Okullo & Temu, 2015). Excessive use of glycerol will maximize the conversion of TAG to MAG and DAG.

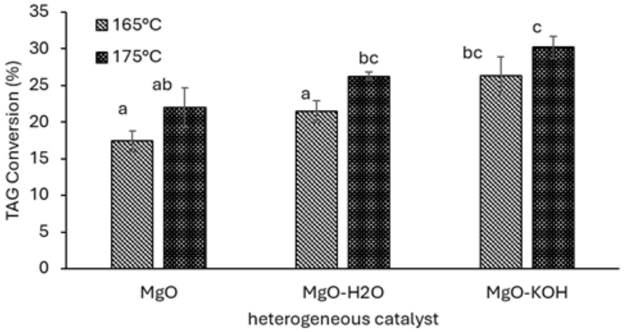

Figure 3. TAG conversion in glycerolysis reaction using different types of activated MgO catalysts (different letters indicated significantly different values ($p < 0.05$))

Figure 3 demonstrates that the use of MgO-KOH resulted in the highest TAG conversion of 30.21% at 175°C, demonstrating its superior catalytic performance compared to MgO and MgO- H_2O. These findings indicate that the alkaline properties of the catalyst significantly enhance the rate of the glycerolysis reaction (Knothe et al., 2015). Under alkaline conditions, the catalyst can attracts H^+ ions from glycerol, forming glycerolate anions, which actively catalyze the glycerolysis reaction. This observation aligns with previous studies by Buchori et al. (2019), which highlighted the effectiveness of optimized heterogeneous catalysts like KF/CaO-MgO in achieving high yields of monoglycerides through controlled reaction conditions.

3.4. Acylglycerol concentration

The results of the analysis of MAG content from the glycerolysis reaction can be seen in Figure 4. Elevating the reaction temperature facilitates the interaction between TAG molecules and glycerol, enhancing the diffusion of reactants with the catalyst's active surface. Azis et al. (2013) demonstrated that higher temperatures increase intermolecular motion, resulting in a higher kinetic energy of reacting molecules. This increased kinetic energy leads to more frequent collisions between reactant molecules, thereby increasing the reaction conversion rate. According to Moquin et al. (2006), MAG molecules can form through multiple reaction pathways, including reactions between TAG and glycerol, DAG and glycerol, as well as between glycerol and free fatty acids. Wangi et al. (2023) also mentioned that the glycerolysis reaction occurs in 6 stages (equation 1-6).

$$\text{TAG} + \text{glycerol} \rightleftharpoons \text{MAG} + \text{DAG} + 3\text{H}_2\text{O} \tag{1}$$

$$\text{DAG} + \text{glycerol} \rightleftharpoons 2\text{MAG} + 3\text{H}_2\text{O} \tag{2}$$

$$\text{TAG} + \text{MAG} \rightleftharpoons 2\text{DAG} \tag{3}$$

$$\text{TAG} + \text{H}_2\text{O} \rightleftharpoons \text{DAG} + \text{FFA} \tag{4}$$

$$\text{DAG} + \text{H}_2\text{O} \rightleftharpoons \text{MAG} + \text{FFA} \tag{5}$$

$$\text{MAG} + \text{H}_2\text{O} \rightleftharpoons \text{glycerol} + \text{FFA} \tag{6}$$

Different activation methods of MgO catalysts also have a significant effect on MAG content ($p<0.05$). Catalysts with higher basicity values can enhance MAG production, as they facilitate more effective interactions between glycerol and other molecules, providing a favorable environment for MAG formation. The MgO-KOH catalyst yielded a higher MAG fraction than other catalysts, reaching 4.31%.

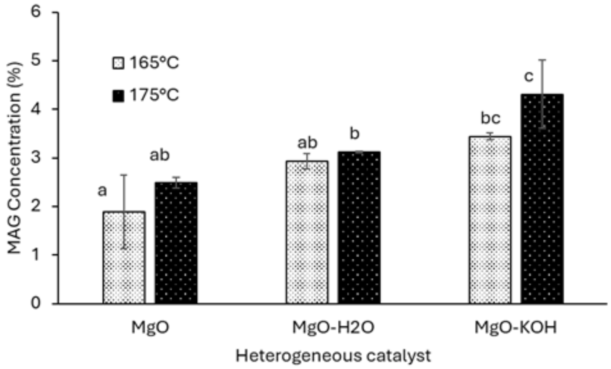

Figure 4. MAG concentration in glycerolysis reaction with different reaction temperatures and activated MgO catalysts (different letters indicated significantly different values ($p < 0.05$))

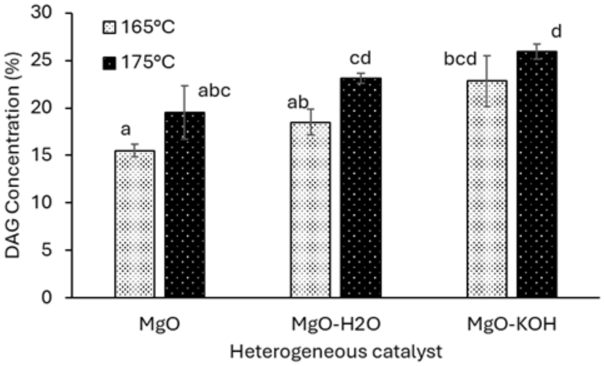

Figure 5. DAG concentration in glycerolysis reaction with different reaction temperatures and activated MgO catalysts (different letters indicated significantly different values ($p < 0.05$))

The DAG fraction had the same results as the MAG fraction where the treatment temperatures of 165ºC and 175ºC were significantly different (Figure 5). The temperature treatment of 175ºC produces a higher value than the temperature of 165ºC. The higher the reaction temperature, the greater the kinetic energy possessed by the reacting molecules. So that it has an impact on more collisions between reactant molecules so that the reaction conversion increases (Azis et al., 2013). Statistical analysis revealed that the application of MgO, MgO-H_2O, and MgO-KOH catalysts had a significant impact on the glycerolysis reaction using coconut oil as the substrate ($p<0.05$). This study found that a reaction temperature of 175°C with the MgO-KOH catalyst produced the highest diacylglycerol (DAG) fraction at 25.9%, attributed to the increased rate constant of the glycerolysis reaction at elevated temperatures (Wangi et al., 2023). These findings align with previous studies where the use of KF/CaO-MgO catalysts resulted in significant increases in monoglyceride yields under optimized reaction conditions. The combination of high surface area and strong basicity of the catalyst enhances reaction efficiency (Buchori et al., 2020).

In comparing MAG and DAG fractions, the DAG fraction was higher than MAG due to the elevated temperature and molar ratio used. During glycerolysis, hydroxyl groups interact with fatty acids, increasing DAG content (Dijkstra, 2020). A short reaction time can limit fatty acid binding to MAG, resulting in lower MAG yields. Both MAG and DAG increase during glycerolysis but eventually decrease after reaching a peak, influenced by temperature (Affandi et al., 2017). Excess FFAs in solution promote DAG formation from MAG and FFA reactions. TAG undergoes partial hydrolysis, and MAG esterifies to form DAG, which the catalyst can further convert to MAG until equilibrium is reached. Lower temperatures affect glycerol solubility, and continued glycerolysis may lead to reverse reactions, reducing MAG and raising TAG in MDAG products (Chetpattananondh & Tongurai, 2008).

3.5. Effect on free fatty acid (FFA) val

Figure 6 shows the value of FFA contained in the MDAG product produced at different reaction temperatures and activated MgO catalysts. The MDAG produced at 175°C exhibited higher free fatty acid (FFA) levels than at 165°C. This increase may occur during glycerolysis, where water formation leads to the hydrolysis of TAG, DAG, and MAG. Melwita et al. (2015) found that water in the reaction promotes hydrolysis, causing oil to form FFAs and glycerol, thus raising FFA levels that would otherwise be reduced. The increase in free fatty acid (FFA) content can also result from product degradation, influenced by oxidation or reverse reactions during glycerolysis (Zhang et al., 2015). High temperatures lower solution viscosity (Pratama et al., 2018), enhancing diffusivity between reactants, leading to increased molecular collisions and product formation, thereby reducing FFA levels in the emulsifier. However, reaction temperature has an optimal point; beyond this, collisions between FFA and glycerol decrease, reducing reaction efficiency and causing FFA levels to rise again (Melwita et al., 2015). Elevated FFAs may further trigger saponification if the fatty acids react with the catalyst, forming soap and water. The lowest FFA content, 0.42%, was observed in the MDAG product produced at 165°C using the MgO-KOH catalyst. This low FFA level is likely due to FFA molecules formed during the reaction promptly interacting with glycerol to produce MAG and DAG.

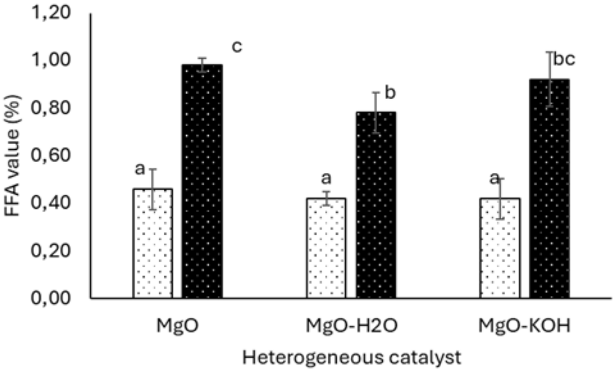

Figure 6. FFA Value of MDAG product (different letters indicated significantly different values ($p < 0.05$))

3.6 Slip melting point of MDAG product

The results of the melting point of MDAG products can be seen in Table 1. Based on the analysis results, the coconut oil used in this study has a slip melting point in the range of 23.6-24.1. Products with high MAG and DAG content exhibit higher SMP values compared to those with low MDAG content. This is evidenced by the MDAG product generated in the glycerolysis reaction at a temperature of 175°C using the MgO-KOH catalyst, which resulted in SMP values ranging from 24.3 to 25.9. The melting point of the MDAG product is influenced by several factors, namely the length of the fatty acid chain, the unsaturation ratio, the position of the fatty acid in the glycerol group (Karabulut et al., 2004).

Generally, MDAG products exhibit a higher SMP value than oil due to their increased hydroxyl groups, which enable the formation of hydrogen bonds among MDAG molecules. However, some SMP values of MDAG products in this study were lower than those of the oil, likely due to the influence of glycerol present in the mixture. The high content of free glycerol in the product lowers the melting point, as glycerol has a melting point of 17.8°C, further reducing the liquid point of the product.

Table 1. Slip melting point (SMP) of MDAG product

Temperature	**Catalyst**	**SMP (°C)**
165°C	MgO	22.5-22.8
	MgO-H$_2$O	22.2-23.4
	MgO-KOH	21.9-22.1
175°C	MgO	22.8-24.7
	MgO-H$_2$O	22.8-24.7
	MgO-KOH	24.3-25.9

4. Conclusions

The MDAG product can be synthesized through a glycerolysis process using coconut oil and glycerol as substrates at a mole ratio of 1:2.3, with the addition of 2% catalyst. A reaction temperature of 175°C resulted in higher fractions of MAG, DAG, and TAG compared to 165°C, as the basicity of the catalyst enhances the rate constant of glycerolysis. The use of the MgO-KOH catalyst yielded the highest conversion rates: 30.21% for TAG, 25.9% for DAG, and 4.31% for MAG, with a melting point range of 22.8–24.7°C and free fatty acid content of 0.7812%. The optimal product was obtained using a treatment temperature of 175°C and the MgO-KOH catalyst activation method. Therefore, activating the MgO catalyst with a KOH solution effectively produces coconut oil-based MDAG products. These products have potential applications in various food products, including ice cream and confectionery.

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