Research Article

Thi Hong Tran*, Quy Diem-Do, Quyen Huynh, and Minh Tan Phan

A green approach in the biological base oil process

Abstract: This article presents a green approach in the biological base oil synthesis from catfish fat by-product using the hydrodynamic cavitation technique. The effects of parameters on the yield of reaction were investigated. The application of the cavitation technique has improved the process efficiency significantly. The catfish polyester oil yield reached at high value (94.6%) at the inlet pressure of 60 psi, the molar ratio of acetic anhydride/epoxy of 1.25/1, 85°C, and only 10 min, and also under these reaction conditions, magnetic stirring technique could only give the limited yield of catfish oil polyester of 12%. The results of Fourier transform infrared resonance, ^{1}H nuclear magnetic resonance (NMR), and ^{13}C NMR analyses and characteristic properties of materials and products have demonstrated that the chemical structure of polyester catfish oil was triglyceride chains containing saturated alkyl R chains and branched by methyl ester functional groups and the polyester catfish oil has met the quality standards of SN150 and SN500 mineral base oils. It could be said that the biological base oil process from catfish fat by-products under the assistance of cavitation techniques would develop sustainably.

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1 Introduction

The research on biological base oil production comes from the risks of global environmental problems, such as the depletion, low renewable capacity of fossil resources, and the severe environmental pollution by the arising wastes from the use of petroleum products [1]. Vegetable oils are high-molecular-weight compounds with the main structure being triglyceride ester chains [2], so they have properties such as high flash point, high viscosity, and high index viscosity [3], which are consistent with the basic features of mineral base oils. It is also because vegetable oils are high-molecular-weight compounds and contain highly unsaturated components, so their low-temperature performance and oxidation stability can be lower than those of mineral base oils. Therefore, to increase the alternative use of vegetable oils, the chemical conversion of vegetable oils can be really necessary. A process consisting of successive reactions of epoxidation of the unsaturated component and opening of the epoxy ring with nucleophile agents, such as alcohols, carboxylic acids, and derivatives, such as acid anhydrides, has been widely used to chemically convert vegetable oils to bio-based oils. Turco et al. [4] converted soybean oil to polyol oil through epoxidation and ring-opening of epoxy with alcohols and soybean polyol oil product has been used as bio-lubricants. Similarly, with soybean oil, Nguyen et al. synthesized bio-lubricant through the epoxy reaction and the ring-opening reaction of epoxy with an H_2O agent [5]. Mohd et al. [6] used palm oil epoxidation reaction and opening reaction of the epoxy ring with oleic acid. The results showed that the ability to work at cold temperatures and the oxidation stability of polyester oil product were significantly increased. Sharma et al. [7] and Somidi et al. [8] synthesized bio-lubricants from canola oil through the epoxidation reaction and the ring-opening reaction of epoxy with acetic anhydride. The products of canola polyester oil

could be used as bio-based oils or bio-lubricants because they had low pour point, high thermal oxidation stability, and high lubricity and they could be predicted to have high biodegradability. For the purpose of synthesizing bio-lubricants, Le and Nguyen performed the epoxidation reaction of catfish fat and the ring-opening reaction of epoxy with acetic anhydride. The results showed that compared with the catfish fat starting material, the catfish polyester oil (CES) product has not only higher lubricity but also much higher oxidation resistance [9].

It can be said that the obtained products from the process of vegetable oil epoxidation and opening the epoxy ring with nucleophile agents have not only high lubricating properties, high working at cold temperature ability, and high oxidation stability but are also highly biodegradable and the process of epoxidation and the ring-opening reaction with acetic anhydride were highly selective because of the irreversible nature of the reaction. However, the use of vegetable oil as a material in the bio-based oil production may cause a shortage of vegetable oil supply in meeting the nutritional needs and quality of meals of all families. While Vietnam is one of the seafood exporters with a large market share in the world, catfish fillet meat is one of the main products. In 2018, Vietnam's catfish processing output reached 1.42×10^6 tons, and according to the Vietnamese government's plan, by 2020, Vietnam will process $1.8–1.9 \times 10^6$ tons of catfish [10]. Catfish fat is a by-product of catfish processing, accounting for the majority of by-products [11], so Vietnam can supply catfish fat in large, stable, and low-cost quantities. The results of our group's previous studies [12,13] have shown that the chemical structure and the characteristic physicochemical properties of catfish fat were similar to those of vegetable oils. Therefore, catfish fat was completely suitable as a material for the production of bio-based oils. Besides it, using catfish fat as a raw material in the production of bio-based oil will bring about high-economic efficiency and create added value for the catfish farming and processing industry in Vietnam.

On the other hand, vegetable oils and catfish fat with high molecular weight and high viscosity could have hindered the diffusion of the two reaction phases. This was detrimental to the reactivity to occur. Meanwhile, in previous studies, the reactions were carried out under the support of a conventional stirrer, such as magnetic stirrer and mechanical stirrer, so the reaction consumed a lot of chemicals and a lot of energy, and the reaction time response lasted many hours. In addition, the low agitation could cause uneven heat distribution in the reaction zone, and this could cause some side reactions, making it difficult to purify the products, reducing the product purity, and reducing reaction yield.

The hydrodynamic cavitation device, with its simple and easy design, could be widely used in heterogeneous reactions. According to Chuah et al. [14], the operating principle of the hydrodynamic cavitation reactor could be described as the flow of fluid passing through the obstacle as a venture, single-hole, and multi-hole diaphragm, which will be depressurized due to an increase in the local velocity under the effect of cross-sectional area and when the pressure decreases below the vapor pressure will produce air bubbles in the liquid medium. Another study by Chuah et al. [15] observed that during the lifetime, the air bubble will gradually increase in size due to more gas infiltration and increase in the surface area along with the decrease in external pressure. The size of the air bubbles could increase from 3 to 400 times, depending on their initial size of them. When the air bubbles moved to a large differential pressure area, they will burst and create a high-pressure area, which could reach at 1,000–2,000 bars and the high temperature in the range of 4,000–6,000 K, depending on the local fluid. Although the temperature in this area was very high, the heat was dispersed very quickly and evenly due to the very small area. The heating and cooling rates in the cavitation zone could be estimated to be about 10^{6}°C·s^{-1}. Therefore, at any given time, the temperature of the entire liquid was always equal to the temperature of the surroundings.

High-performance mixing techniques based on ultrasonic techniques [16] and hydrodynamic cavitation techniques [17] have been used in the reactions to eliminate phase contact interference between heterogeneous reagents. In which, mixing techniques based on cavitation technique have been applied with many outstanding advantages [18]. Using the cavitation technique has created a homogeneous reaction zone due to high mixing efficiency. The cavitation took place in the sliding stage of the liquid, so reducing the possibility of wear on the pipe walls. Besides, the productivity and efficiency of the device could be easily adjusted and selected through the selection of operating parameters and conduction device configuration to optimize cavitation formation conditions [19,20]. The outstanding advantages of hydrodynamic cavitation technique evaluated based on the observarted yield of heterogeneous reactions in previous studies and the easy applicability of industrial production scale of cavitation technique have been demonstrated.

The obtained results in the research of Mohan et al. [21] showed that biodiesel production from cottonseed oil and methanol was more convenient when the reaction was performed in a ultrasonic cavitation reactor. The yield of the reaction reached 96.4% in only 30 min. Whereas the reaction was performed in a mechanical stirring reactor, the yield reached 90.7% in 110 min with the higher consumption reactants. Chuah et al. [17] carried out a heterogeneous reaction of methanol and waste

cooking oil; alkaline catalysis was carried out on a mechanical stirrer and a hydrodynamic cavitation device at the same reaction conditions. In the case of time of 15 min, reaction performed in hydrodynamic cavitation reactor resulted in the conversion of 98.1%, whereas performing the reactor under the assistance of mechanical stirrer gave a limited conversion at 19%. The observed results in the study of Khan et al. [22] showed that performing the heterogeneous reaction under the support of the hydrodynamic cavitation technique greatly reduced the reaction time compared to carrying out the reaction under the support of a mechanical stirrer. Namely, the optimal time for the transesterification of *Cannabis sativa* L. oil and methanol, alkaline catalysis, was 20 min for the hydrodynamic cavitation approach and it was 90 min for the mechanical stirring approach. The research results of Sun et al. [18] showed that the hydrodynamic cavitation technique has created many advantages for the process in sonochemistry technology as well as in industrial-scale applications. Therefore, the limitations of the conventional mixing technique have been overcome by the intensification mixing cavitation technique.

In this study, from an abundant, readily available, inexpensive, and nontoxic by-product of catfish fat, an environmentally friendly bio-based oil product was produced. On the other hand, the chemical conversion of catfish fat has applied the cavitation technique, which cavitation technique is considered a green chemical technique because it can shorten the reaction time significantly, reduce the reaction temperature, reduce chemical consumption, and improve process efficiency. The utilizing catfish fat by-product as materials in bio-based oil production with the assistance of cavitation techniques has been identified as the main objective of the study. In this study, the biological base oil synthesis reaction performed under the support of a magnetic stirrer was also conducted to compare with the efficiency of the reaction carried out with the assistance of a hydrodynamic cavitation device. The utilization of catfish fat by-product as material for bio-based oil production could not only contribute to increasing economic value for catfish farmer and to sustainable economic development but also contribute to reducing the dependence on petroleum resources and to energy security and reducing the environmental pollution.

2 Materials and methods

2.1 Materials

Catfish fat raw material (CFF) was supplied by the Phuoc Thanh Agricultural Company Limited, An Giang province,

Viet Nam. CFF was put into the vacuum filter device with a filter cloth with a hole diameter of 30 μm. A mechanical stirrer was installed, a gentle stirring speed was set and the catfish fat was heated to a temperature of about 30–35°C for 1 h to reduce the viscosity of the catfish fat. The vacuum filtration was taken place over a period of 3–4 h. Then, the blow-down valve was opened to collect catfish oil (CFO), which was used as a starting feedstock in the chemical conversion process. CFO was stored in the darkened can and stored away from direct light. Hydroperoxide (30 wt%), acetic acid (99 vol%), and sulfuric acid (99.9 vol%) were made in Viet Nam. Acetic anhydride (assay ≥99.9 vol%) was originated in Spain. The chemicals and solvents in analysis were procured by Merck (Germany).

2.2 Experimental process

From the results of studies on the influence of the device geometry on the performance of the hydrodynamic cavitation reactor [23,24], the cavitation device is an orifice plate type, 1 hole with a diameter of 2 mm, 10 L capacity in the previous works [12,13] was used as the reactor of the chemical conversion process in this study. The cavitation system consists of a cylindrical container heated or cooled by an inner coil and an outer jacket, which is connected to a centrifugal pump with a shoulder pressure when injecting the reaction material mixture through the stopper to the cavitation zone. The inlet pressure is regulated using valve V3 on the main line and valve V4 on the bypass. The device system diagram is shown in Figure 1.

First, materials and reagents were placed in container 2. Next, valve V2 and valve V4 were opened so that the reaction mixture was stirred and circulated for about 1 min. Valve V4 was closed and at the same time valve V3 was opened so that the reaction mixture was pumped by pump 1 into the cavitation zone. Temperature and pressure values were monitored through the display on the manometer-P and thermometer-T. After the reaction was finished, valve V1 was opened to collect the raw product mixture, and then the product mixture was purified. Finally, the power, water, and the entire cavitation system were turned off.

The chemical conversion of catfish oil to polyester bio-based oil was proposed to include two reaction stages, which were epoxidation of catfish oil with peracetic acid agent (formed from a mixture of hydroperoxide and acetic acid) and ring-opening reaction of catfish epoxy oil (CEP) with acetic anhydride agent, catalyzed by sulfuric acid. The schematic diagrams of chemical reactions are shown in Figure 2.

The catfish oil epoxidation reaction was performed under the appropriate conditions, which were identified in our previous study [13], as the inlet pressure of 50 psi, the molar ratio of $H_2O_2/CH_3COOH/C{=}C$ of 3.75/1.25/1, the
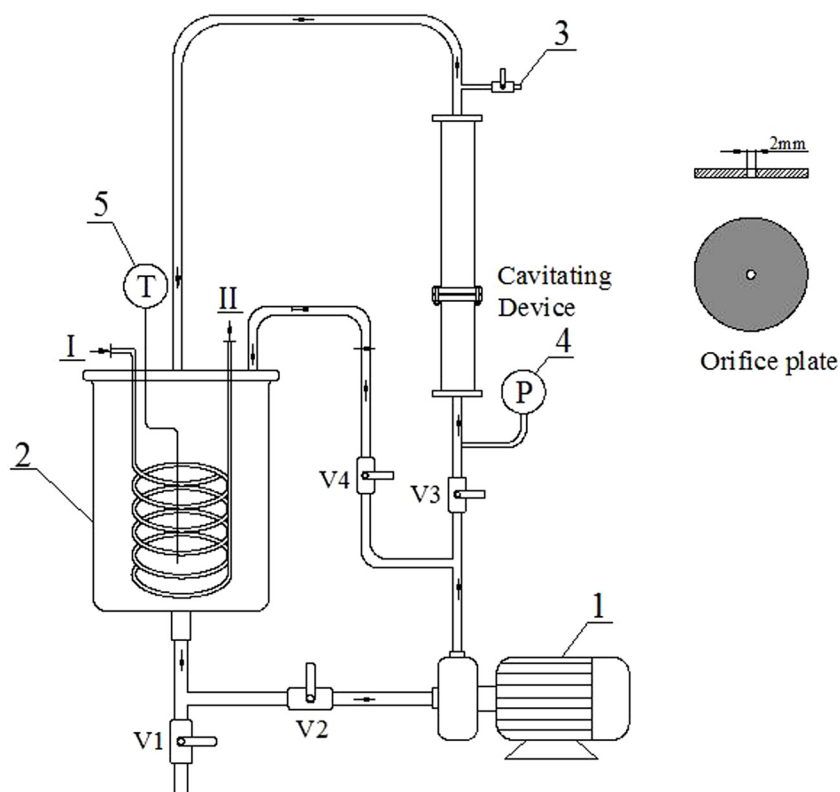
Figure 1: Schematic representation of cavitation device system; (1) pum, (2) tank, (3) sampling valve, (4) pressure gauge, (5) thermometer.

temperature of 45°C and the time of 5 min [13]. The ring-opening reaction of CEP with acetic anhydride was carried out in the investigated parameters, which included the inlet pressure of 40–75 psi, the molar ratio of acetic anhydride/epoxy ring of 0.75–1.75, the temperature of 65–100°C, and the time of 4–10 min [25].

In the catfish epoxidation reaction stage, the raw product mixtures were washed with water several times until the acid was gone and the water was removed with anhydrous Na_2SO_4, collecting CEP as feedstock for the opening reaction of epoxy ring with acetic anhydride agent. Similarly, the obtained raw product mixtures from the epoxy ring-opening reaction were washed with water several times until the acid was gone and the water was removed with anhydrous Na_2SO_4, collecting CES. CES determined the ester value according to the ASTM D5558-95 to evaluate the efficiency of the reaction.

2.3 Detection method

The functional groups of samples were determined by Fourier transform infrared resonance (FT-IR) and ^1H nuclear magnetic resonance (NMR) and ^{13}C-NMR. The FT-IR ana-

lysis was performed on the FT/IR Agilent, serial number C019561788. The wavenumber was in the range of 400–4,000 cm^{-1} and the ^1H NMR and ^{13}C NMR analyses were carried out on the NMR Bruker 500 MHz, $CDCl_3$ solvent. The oxidation stability of samples was determined by the thermogravimetric analysis (TGA) and the Rancimat test.
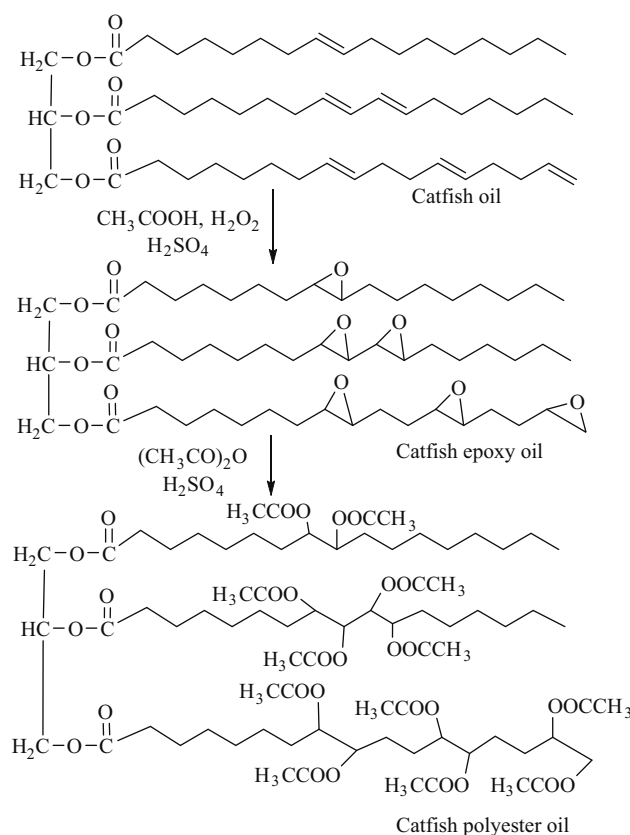
2.4 The yield calculation of reaction

The yield of ring-opening reaction y (%) was calculated by Eq. 1:

$$y = \frac{EV_{exp}}{EV_{theo}} \times 100\% \tag{1}$$

where EV_{exp} and EV_{theo} are the ester values of experiment and theory (mg KOH·g^{-1}). According to the ASTM D5558-95, EV_{exp} and EV_{theo} were calculated by Eqs. 2 and 3:

$$EV_{exp} = \frac{V_0 - V_1}{w} \times 0.1 \times 28.05 \tag{2}$$

$$EV_{theo} = \frac{10E}{43} \times 28.05 \tag{3}$$

Figure 2: The synthesis reaction scheme of CES from catfish oil.

where V_0 and V_1 (mL) are the volumes of titration of blank samples; w (g) is the weight of samples and; E (wt%) is the epoxy content of epoxy catfish oil, which was calculated according to the ASTM D1652.

3 Results and discussion

CEP determined the epoxy content (E) according to the standard of ASTM D1652 and E was reached 10.7 wt%.

The experiments to investigate the influence of inlet pressure, loading reagents, temperature, and time on the reaction efficiency were conducted with the assistance of the hydrodynamic cavitation equipment to determine the optimal conditions for the ring-opening reaction of CEP with an acetic anhydride agent, catalyzed sulfuric acid.
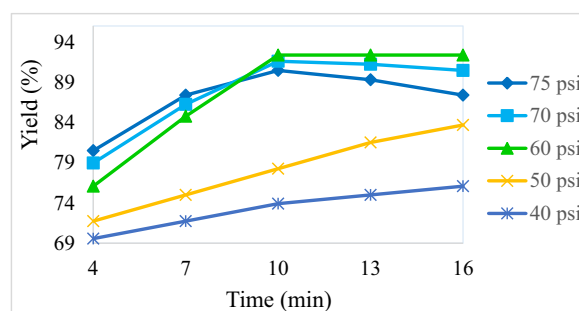
3.1 Effect of inlet pressure

Experiments to investigate the influence of inlet pressure on the yield of ring-opening reaction of CEP with acetic

anhydride agent were carried out at the molar ratio of acetic anhydride/epoxy (An/Ep) of 1.25/1 at 80°C for a period from 4 to 16 min. The results are presented in detail in Figure 3.

From the presented data in Figure 3, it can be seen that the inlet pressure has a great influence on the reactivity. As the pressure increased from 40 to 50 psi, the reaction yield gradually increased in the surveyed time. When the pressure increased to 60 psi, the reaction yield increased rapidly. The yield reached the maximum value of 92.4% at 10 min and after 10 min, the reaction yield was unchanged. A subsequent increase in inlet pressure to 70 and 75 psi resulted in a slight decrease in reaction yield, considering at 10 min the yield decreased from 92.4% to 90.5%.

The theory of reaction engineering in hydrodynamic cavitation systems has suggested that an increase in inlet pressure can lead to an increase in cavitation efficiency because inlet flow increases with inlet pressure increased. This can lead to an increase in the number of cycles of the flow through the orifice, which increases the retention time of the flow in the cavitation zone, thereby increasing the cavitation efficiency. This can happen when the inlet pressure is increased within a certain limit, which is called the supercavitation pressure or chocked pressure ($P_{chocked}$). When the inlet pressure increases beyond the value of $P_{chocked}$, the reaction efficiency hardly increases but tends to be reduced due to having the obstruction of flow through the orifice of the cavitation system [26]. In this study, increasing pressure between 40 and 60 psi resulted in an increase in the flow velocity through the hole, which led to the increase in the amount of liquid passing through the cavitation zone and the increase in the formation of air bubbles in the reaction mixture. The increase in liquid passing through the hole could lead to the decrease in the pressure in the behind area of the hole. This could enhance the burst strength of the air bubbles, resulting in chaotical

Figure 3: Effect of inlet pressure on the CES yield in surveying time of 4–16 min.

agitation in the reaction mixture. As a result, the phase contact surface of the reagents increased, and the capacity of heat transfer and mass transfer increased, so the reactivity has increased significantly. The inlet pressure was increased to 70 and 75 psi, for the time of 4–7 min, and the cavitation efficiency increased so the reaction efficiency increased. After 10 min, the downstream zone of the hole could appear localized area to be filled with a large number of air bubbles, which have tended to coalesce to form larger air bubbles and this caused a blockage of the flow through the orifice, causing that the pressure in the behind area of the hole did not decrease further. Thus, the amount of liquid passing through the hole did not increase and the bursting of the air bubbles could be inhibited. As a result, the reactivity was not increased but decreased.

It could be said that, when the inlet pressure increased, the ability for the ring-opening reaction of CEP with acetic anhydride agent increased, but this was only happened when the pressure increased to a certain value called the chocked pressure ($P_{chocked}$). If the inlet pressure was increased beyond $P_{chocked}$, the reaction rate was reduced again. This has also been studied and presented similar results in the study of Sarvothaman et al. [27]. A similar result was also reported by Fatt et al. in the study on the effect of inlet pressure on the biodiesel synthesis reaction from cooking oil and methanol, alkaline catalysis, performed in the hydrodynamic cavitation device [28].

In this study, the inlet pressure at 60 psi could be the *chocked* pressure value and it was selected as the pressure value to investigate the influence of the remaining factors.

3.2 Effect of loading reactant

The influence of the amount of loading reagents on the reaction efficiency of ring-opening of CEP was investigated through the molar ratio of An/Ep in the range of 0.75/1–1.75/1. The experiments were carried out at the inlet pressure of 60 psi, 80°C in the time of 4–16 min, and the results are shown in Figure 4.

The presented data in Figure 4 showed that as the molar ratio of An/Ep increased from 0.75 to 1.0, the reaction yield gradually increased in during the investigation time. When the molar ratio of An/Ep further increased to 1.25, the reaction yield increased rapidly and reached at a maximum of 92.4% in a reaction time of 10 min. The molar ratio of the reactants increased to higher values, 1.5 and 1.75, and even prolonging the reaction time
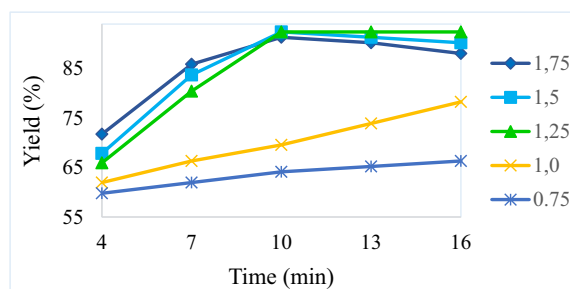
Figure 4: Effect of molar ratio of acetic anhydride/epoxy on the CES yield in surveying time of 4–16 min.

beyond 10 min, the reaction yield did not increase further and it tended to decrease slightly.

According to the theory of epoxy ring-opening reaction with acetic anhydride agent, the molar ratio of An/Ep has a value of 1/1. The results of the study by Wang et al. [29] confirmed that soybean epoxidized oil has a high viscosity. Similarly, Silviana et al. [30], have also determined that epoxy oil synthesized from waste cooking oil has a very high viscosity. So to increase the diffusion capacity of CEP into anhydride agent acetic acid and increase the mass transfer as well as the heat transfer in the reaction mixture, an amount of reactant with a molar ratio of An/Ep is required in higher than 1/1. So when the molar ratio of An/Ep increased from 0.75 to 1.25, the reaction yield increased gradually.

According to the reaction theory performed in the hydrodynamic cavitation equipment system, when the amount of acetic anhydride agent increased, the reaction mixture was diluted; thus, the fluid velocity and the flow velocity increased through the diaphragm; this increased the number of air voids. Then, the pressure in the downstream of the hole is further reduced, leading to an increase in the bursting strength of the air bubbles and an increase in the agitation in the reaction mixture. This could create high diffusion and high contact between the reactant phases. Therefore, the capacity of heat transfer and mass transfer in the reaction mixture was increased and, as a result, the reaction rate was increased; but if the reaction mixture was more diluted, the formation of air bubbles was increased, but they tend to clump together to occupy the area downstream behind the hole, so that the pressure drop did not decrease further, thus strengthening. The bursting rate of the air bubbles could be reduced, causing the reaction rate not to increase further, but it reduced slightly. Therefore, when the reaction was carried out with a higher amount of acetic anhydride, corresponding to the molar ratio of An/Ep at 1.5 and 1.75, the reaction yield was slightly decreased [31]. In addition, in these cases, an excess of acetic anhydride

may be present, and under the intense agitation of the hydrodynamic cavitation system, this excess of acetic anhydride could be formed emulsions with the H_2SO_4 catalyst and polyester catfish oil products; this could make it difficult to refine the product, so the yield was also slightly reduced.

A similar result was presented by Chuah et al. in the study of the influence of the methanol/oil molar ratio on the conversion of heterogeneous reaction of biodiesel synthesis, performed in a hydrodynamic cavitation device [17]. The results of the study of heterogeneous reaction of *Cannabis sativa* oil and methanol, alkaline catalysis, performed in the hydrodynamic cavitation device of Khan et al. have shown that, as the molar ratio of methanol/oil increased, the amount of air bubbles formed in the reaction mixture was increased and subsequently the bursting intensity of the bubbles was increased, creating a high-phase contact surface between reactants, so the reaction yield was increased [22]. Similarly, the optimization of the heterogeneous reaction between castor oil and methanol, performed in the hydrodynamic cavitation device of Mistry et al. [32], confirmed that the methanol/oil molar ratio could be one of the major factors and it was surveyed in the range of 6/1–10/1. The biodiesel yield reached the highest value at the methanol/oil molar ratio of 6/1 under other optimal reaction conditions.

3.3 Effect of temperature

Investigating experiments of the effect of temperature on the yield of ring-opening reaction of CEP with acetic anhydride were performed at the inlet pressure of 60 psi and the molar ratio of An/Ep of 1.25/1, and the results are shown in Figure 5.

The data in Figure 5 have shown that temperature could be one of the parameters in strong influence on
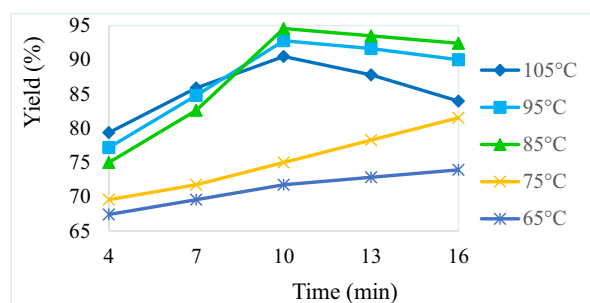
Figure 5: Effect of temperature on the CES yield in surveying time of 4–16 min.

reactivity. Namely, as the reaction temperature increased from 65°C to 75°C, the yield increased significantly during the investigation time of 4–16 min. When the temperature increased to 85°C, the reaction yield increased and reached a maximum in 10 min. As the temperature increased to higher values, 95°C and 100°C, the reaction yield increased for the first time, 4–7 min, and the yield tended to decrease, even though the reaction time was extended to 10, 13, and 16 min.

The results could be because the temperature was increased, the viscosity of CEP was decreased, favoring the formation of cavitation in the reaction mixture, and then the bursting intensity of the air bubbles was increased. This created a strong agitation in the reaction mixture, leading to the dispersion of acetic anhydride agents into the oil phase increased, so the heat transfer and mass transfer in the reaction mixture were enhanced and the reaction rate was increased. The temperature was increased to 85°C, the fluid viscosity was also reduced much more, the flow rate or flow through the hole was increased much more, as well as the bursting of air bubbles was increased significantly in the first time (4–10 min), but after 10 min, the bursting strength of the air bubbles was reduced because they could have clumped together to form a larger array of air bubbles that inhibited this bursting, so the reaction velocity in these cases was reduced. Further increasing the temperature to 95°C and 105°C values, the reaction yield variation tended to be similar to the case of 85°C reaction temperature. It means that in the initial period (4–7 min), the cavitation device efficiency was enhanced and therefore the reaction efficiency increased but the reaction efficiency was decreased with increasing time reaction to 13 min and 16 min.

Menzl et al. [33] suggested that the ability of a fluid to form cavitation depended on the surface tension and viscosity of the fluid. The effect of temperature on cavitation system activity or heterogeneous reaction efficiency has also been reported and explained similarly by Mohod et al. [34] in the study on the heterogeneous biodiesel synthesis reaction, carried out in a hydrodynamic cavitation device. At other fixed conditions, when the temperature was increased from 40°C to 50°C, the reaction yield was increased, but the yield was decreased when the temperature was increased to 60°C.

It could be concluded that the inlet pressure of 60 psi, temperature of 85°C, the molar ratio of acetic anhydride/epoxy of 1.25/1, time reaction of 10 min, were considered to be the optimal conditions of reaction and the reaction yield reached at 94.6%.

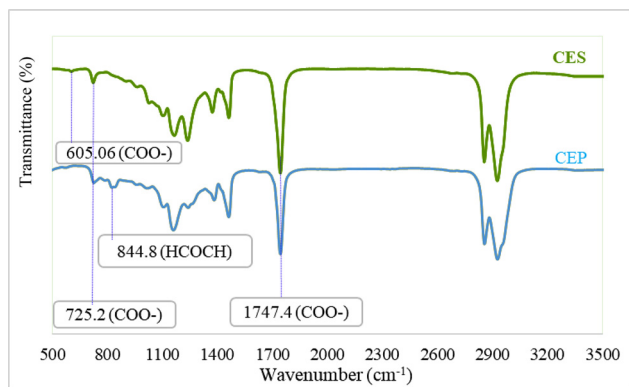It could be said that after the CEP ring-opening process with acetic anhydride, carried out under the

Figure 6: FT-IR spectra of CEP and CES product.

assistance of cavitation equipment, the epoxy rings were opened and formed methyl ester (CH₃COO−) branches in the chains alkyl R of the triglyceride structure. The obtained results have shown the advantages of the hydrodynamic cavitation technique and the outstanding advantages of the nucleophile agent acetic anhydride were also shown. It may be the irreversible nature of the reaction and the heat stability of the agent that limits some of the side reactions. And as a result, process efficiency has increased dramatically. These results were also elucidated by the results of FT-IR, ^{1}H NMR, and ^{13}C NMR analyses detailed in Figures 6–8.

3.4 FT-IR analysis

The FT-IR spectrum of CEP appeared the characteristic peaks for its major functional groups. Peaks appeared at 1,376.93, 1,464.67, and 2,852.20, 2,919.70 cm^{-1} could verify the presence of CH₃, CH₂, and CH groups. The CO and C=O of the ester functional group (O−C=O) could be determined through the appearing peaks at 1,112.73, 1,160.94, and 723.18, 1,741.41 cm^{-1}. The FT-IR spectrum of CEP appeared to very clearly peak at 844.8 cm^{-1}, which was typical for the epoxy ring (HCOCH). Similarly, the FT-IR spectra of CES appeared characteristic peaks for the major functional groups of CEP, but here the appearance of the characteristic peak for the epoxy ring was not found, and instead the strong appearance of a new peak at 605.6 cm^{-1}, which was typical for the ester functional group (O−C=O), the strengthening of peaks at 725.2 and 1,747.4 cm^{-1} also characterized the ester functional group. This could confirm the presence of the methyl ester group (CH₃COO−) in the structure of the CES product.

The results of the FT-IR analysis in this study were reported and explained similarly in other studies on the ring-opening reaction of epoxy oils based on vegetable oils and fats with an acetic anhydride agent. Analytical results in the study of Sharma et al. [7] showed that the appearing peaks at 3,007 and 738 cm^{-1} in the FT-IR spectrum of canola oil demonstrated the presence of HC=CH
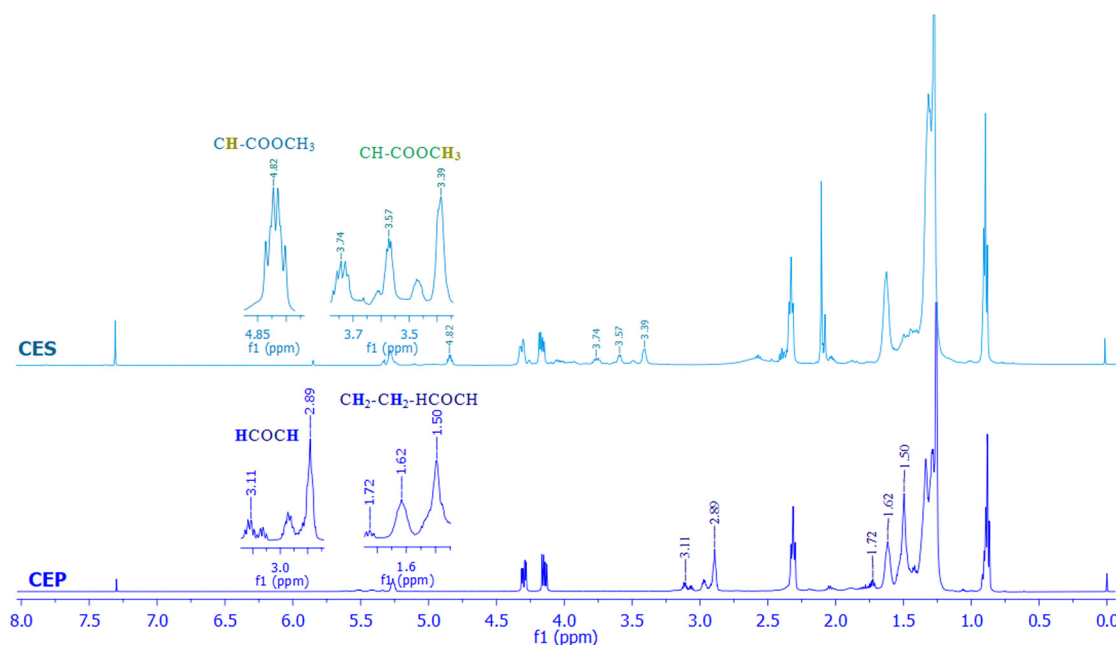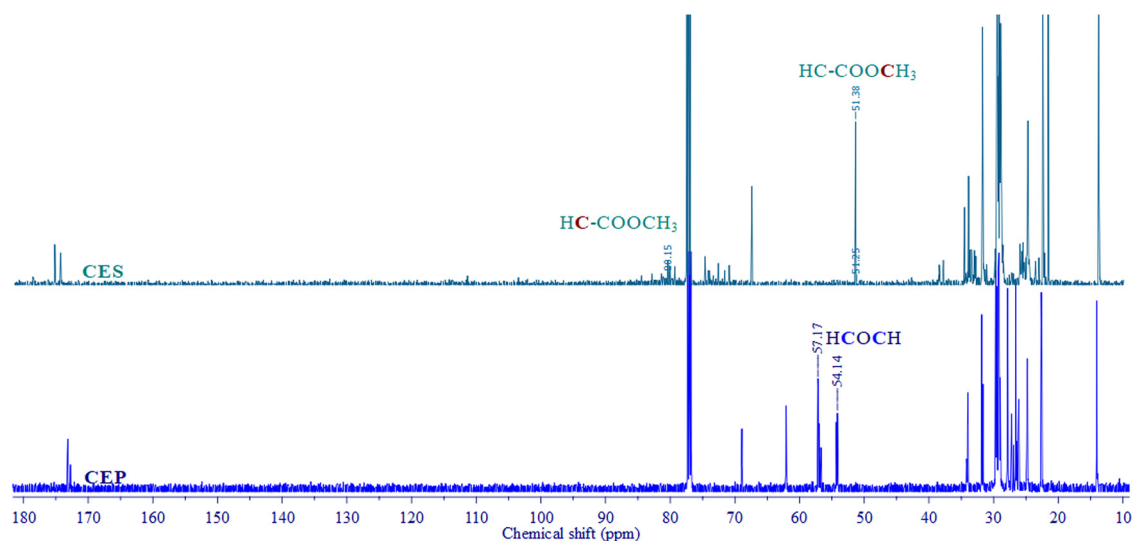

Figure 7: ^{1}H NMR spectra of CEP and CES product.

Figure 8: ^{13}C NMR spectra of CEP and CES product.

double bonds in it. In the FT-IR spectra of the canola epoxy oil, no characteristic peaks for the double bond HC=CH were found, and instead the appearance of peak at 831 cm^{-1} demonstrated the presence of an epoxy ring in the canola epoxy oil structure. Similarly, the FT-IR spectra of the canola polyester oil did not find a characteristic peak for the epoxy ring, but instead the appearance of new peaks at 725 and 604 cm^{-1} along with an increased intensity peak at 1,750 cm^{-1}. This confirmed the presence of the methyl ester group in the product. In the study by Le and Nguyen [9], the FT-IR spectra of the catfish fat appeared to peak at 1,655 and 3,006 cm^{-1}, which proved the presence of the HC=CH double bond in the structure of the catfish fat. The FT-IR spectra of the CEP did not find characteristic peaks for the double bond, instead the appearance of the peak at 824 cm^{-1}, which demonstrated the presence of an epoxy ring in the epoxy oil structure. Similarly, the FT-IR spectra of the CES did not find a characteristic peak for the epoxy ring. This could be confirmed that the epoxy rings of CEP were opened with an acetic anhydride agent.

3.5 NMR analysis

In Figure 7, characteristic peaks for the major protons of the CEP appeared in the ^{1}H-NMR spectrum of CEP. Namely, the protons of **CH$_3$** and **CH$_2$** of the R chains were confirmed by the appearance of peaks at 0.87–0.89 and 1.26–1.30 ppm. The protons of β-**CH$_2$** and α-**CH$_2$** of the R chain linked to the ester group (–COO) could be verified by the presence of peaks at 1.61–2.02 and 2.29–2.32 ppm. The appearance of signal

peaks at 4.13–4.16 and 4.28–4.31 ppm could be specific to the protons of **CH** and **CH$_2$** of the glycerol wires. The presence of chemical signals at 7.27 ppm could determine the protonation of the solvent **CHCl$_3$**. The ^{1}H NMR spectrum of CEP clearly showed signals at 2.89–3.11 and 1.5–1.72 ppm, which could be specific to the protons of the epoxy ring (**HCOCH**) and β-**CH$_2$** and α-**CH$_2$** linked to the epoxy ring in the structure of CEP.

The characteristic peaks of protons of the main functional groups of CEP appeared in the ^{1}H NMR spectrum of the CES product, but the appearance of the characteristic proton peaks of the epoxy ring was not found, and instead the strong appearance of new peaks at 3.39–3.74 and 4.82 ppm, which could be characterized for the represents of the protons of the methyl ester group (**CH$_3$**COO–) and the proton linked to the methyl ester group (**HC**–COOCH$_3$) in the structure of CES.

^{13}C NMR spectrum of CEP in Figure 8 showed the characteristic peaks for the major carbons of CEP. Namely, the carbons of **CH$_3$** and **CH$_2$** of the R chains and the carbon of **CH$_2$** in the R chains linked to the ester group (–COO) were confirmed by the appearance of peaks at 14.03–14.07, 22.66–31.90, and 34.01–34.18 ppm.

The appearance of signal peaks at 62.09–68.91 and 172.78–173.19 ppm could be specific to the carbon of **CH** and **CH$_2$** of glycerol chains and the carbon of the ester functional group (**C**OO). The presence of chemical signals at 76.78–77.29 ppm could determine the carbonation of the solvent **C**HCl$_3$. The ^{1}H NMR spectrum of CEP clearly showed signals at 54.14–57.17 ppm, which could be specific to the carbons of the epoxy ring (H**C**OC**H**) in the structure of CEP.

The characteristic carbon peaks of the main functional groups of CEP appeared in the ^{13}C NMR spectrum of the CES product, but the appearance of the characteristic carbon peaks of the epoxy ring was not found, and instead the strong appearance of new peaks at 51.38 and 80.15 ppm, which could be determined for representing the carbons of the methyl ester group ($\textbf{C}$H$_3$COO–) and the carbon linked to the methyl ester group (H$\textbf{C}$–COOCH$_3$) in the structure of CES.

The results of the ^{1}H NMR and ^{13}C NMR analyses in this study were reported and explained similarly in other studies on the ring-opening reaction of epoxy oils based on vegetable oils and fats with an acetic anhydride agent. Analytical results in the study of Sharma et al. [7] showed that the appearing peaks at 5.2–5.4 ppm in the ^{1}H NMR spectrum of canola oil demonstrated the presence of protons of HC=CH double bond in it. In the ^{1}H NMR spectra of the canola epoxy oil, no characteristic peak for protons of the double bond HC=CH was found, and instead the appearance of peak at 2.7–3.1, 1.9, and 1.6 ppm, which demonstrated the presence of protons of the epoxy ring, α-CH$_2$ and β-CH$_2$ linked to epoxy ring in the canola epoxy oil structure. Similarly, the ^{1}H NMR spectra of the canola polyester oil did not find a characteristic peak for protons of the epoxy ring, but instead the appearance of new peaks at 3.7 ppm. This confirmed the presence of protons of the methyl ester group in the product. The ^{13}C NMR spectrum of canola oil demonstrated the presence of carbons of C=C double bond in it via the peaks shown at 120–140 ppm. The ^{13}C-NMR spectrum of the canola epoxy oil did not find the characteristic peaks for the carbons of the C=C double bond and new peaks appeared at 53–58 ppm, which were typical for the epoxy ring carbons in the structure of canola epoxy oil. Similarly, the ^{13}C-NMR spectra of the canola polyester oil did not find a characteristic peak for carbons of the epoxy ring, but instead the appearance of new peaks at 51 ppm. This confirmed the presence of carbons of the methyl ester group in the product. In the study by Le and Nguyen [9], the ^{1}H NMR spectra of the CEP appearance the peaks at 2.9–3.1, 1.7–1.9, and 1.4–1.6 ppm, which demonstrated the presence of protons of the epoxy ring, α-CH$_2$ and β-CH$_2$ linked to epoxy ring in the epoxy oil structure. The ^{1}H NMR spectra of the CES did not find characteristic peaks for protons of the epoxy ring. This could be confirmed that the epoxy rings of CEP were opened with an acetic anhydride agent.

The results of the ^{1}H NMR and ^{13}C NMR spectral analyses of the samples were consistent with the results of the FT-IR spectral analysis and they could also be consistent with the obtained results in the survey on the influence of parameters on the ability of CEP ring-opening reactivity to the acetic anhydride agent, performed under the assistance of cavitation equipment. From these results, it could be said that the ring-opening reaction of CEP with acetic anhydrite, catalyzed by sulfuric acid, carried out under the assistance of hydrodynamic cavitation equipment has completely occurred.

3.6 Comparison of the performance of the hydrodynamic cavitation device and the magnetic stirring

At the determined optimal reaction conditions, the ring-opening reaction of CEP with acetic anhydride agent catalyzed by sulfuric acid was carried out with the assistance of a hydrodynamic cavitation device and magnetic stirrer to compare and evaluate the effect of applying the cavitation technique to the reaction process. The results are shown in Figure 9.

The data in Figure 9 show that mixing based on the cavitation technique led to a much higher reactivity than the case using a magnetic stirrer. Considering the reaction time of 10 min, the reaction efficiency reached 94.6% while the reaction using a magnetic stirrer resulted in a limited yield of 12%. The obtained results could be explained because the cavitation-based mixing technique has created a chaotic agitation leading to a higher-phase contact surface between the reactants. This could be effective in eliminating mass transfer resistance during the reaction.
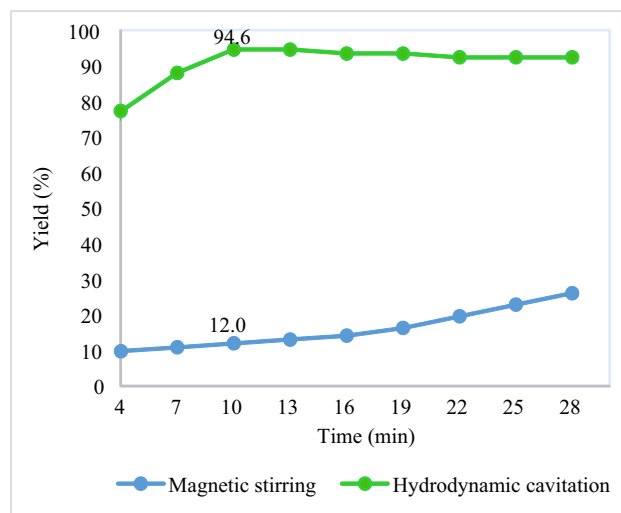
Figure 9: Comparison of the performance of the hydrodynamic cavitation device and the magnetic stirring.

In addition, the hydrodynamic cavitation reactor generates internal heat and the heat could be evenly distributed throughout the entire reaction mixture with the fastest possible heat transfer rate and, thus, reaction efficiency reached a higher value. While performing the reaction under the help of a magnetic stirrer, the phase contact between the CEP and the acetic anhydride agent mainly depended on the stirrer being magnetic, which could lead to some limits on the diffusivity of the reacting phases with each other and thus produces poor heat and mass transfer between reactants, resulting in slow reaction rates.

It can be seen that the efficiency of the reaction process has increased significantly when applying the cavitation technique. The reaction efficiency was increased with reduced reagent consumption, reduced reaction temperature, and shortened reaction time greatly. This has also been confirmed when compared with the results of other studies on the ring-opening reaction of epoxy oils based on vegetable oils or fish fat with acetic anhydride agent, acid catalysis was carried out under the support of a heated magnetic stirrer. Sharma et al. [7] performed the ring-opening reaction of 30 g of epoxidized canola oil with 45 g of acetic anhydride, a solid acid catalyst of 10 wt% of epoxidized oil, in the three-neck round bottom flask and a heated magnetic stirrer at 130°C, and the reaction time lasts up to 15 h. Somidi et al. [8] studied the ring-opening reaction of vegetable epoxy oils with acetic anhydride agent, MoO_3/Al_2O_3 catalyst, carried out under the support of a magnetic stirrer and the optimal conditions of the reaction were determined at molar ratio of anhydrie acetic/epoxy of 2/1, 85°C, loading catalyst of 12 wt% of epoxidized oil and reaction time of 4 h. The optimal conditions of the ring-opening reaction of epoxy oil were established at a molar ratio of anhydride acetic/epoxy of 2/1, 85°C, the loading catalyst of 12 wt% of epoxidized oil, and the reaction time of 4 h. Le and Nguyen synthesized bio-lubricant from catfish fat through epoxidation and epoxy ring-opening reactions, carried out with the

support of a heated magnetic stirrer. The conversion efficiency of the epoxy ring-opening reaction reached the highest value under the conditions including the mass ratio of acetic anhydride/epoxy of 1.5/1, the *p*-toluenesulfonic acid catalyst consumption of 0.1125 wt% epoxy oil, 85°C, for 5 h reaction time [9].

3.7 The characteristic properties

The specific properties of the catfish oil (CF) starting material and the CES product were determined to evaluate the efficiency of a chemical process. For CES to be used as a bio-based oil, it must meet the quality requirements of a mineral base oil. Therefore, the basic properties of CES have been determined based on the quality standards of mineral base oils SN150 and SN500, and the results are presented in Table 1.

The observed results in Table 1 show that the basic properties of polyester catfish oil product (CES) have been significantly improved compared to the starting catfish oil material (CF). Specifically, the density and open cup flash point increased from 0.916, 292°C, to 0.996, 322°C. The pour point was reduced from 20°C to as low as −2°C. Similarly, the kinematic viscosity at 40°C and 100°C and the viscosity index increased, respectively, from 95.26, 11.30, and 105.2 cst to 157.17, 20.72, and 154.33 cst. In particular, the thermal stability and oxidation stability of CES reached high values, 249–310°C and 11.71 h, while catfish oil was decomposed at a lower temperature, 174–245.5°C, and oxidation stability time was also lower, only approximately 0.2 h.

Obtained results could be explained that CF material has the main structure of triglycerides with straight-chain alkyl R chains, so it could tend to easily form a crystalline structure at cold temperature by homogenous superposition of the structure. Once formed, the crystals grow and

Table 1: The basic properties of catfish oil material (CF), CES product, and mineral base oils SN150 and SN500 (SN150, SN500)

Properties	CF [12,36]	CES	SN150	SN500 [12,36]	Analysis method
Density	0.916	0.996	0.880	0.889	ASTM D1298
Flash point (°C)	292	322	210	250	ASTM D92
Pour point (°C)	20	−2	−6	−6	ASTM D97
Kinematic viscosity (cst)					ASTM D445
40°C	95.26	157.17	39	96.03	
100°C	11.30	20.72	6	11.59	
Index viscosity	105.2	154.33	95.91	109.07	ASTM D2270
Thermal stability (°C)	174−245.5	249−310	265.5−340	258.5−318	TGA
Oxidative stability (h)	≥0.20	≥11.71	≥30	≥29.52	Rancimat

increase in size, which restricts the flow due to the loss of kinetic energy of the individual molecules. This could be limited the ability of catfish oil to operate at low temperatures. After conducting the epoxidation and epoxy ring-opening process, the main structure of CES was still the triglyceride chains but the C=C double bonds in the R chains were saturated and branched by methyl ester groups (CH_3COO-), so it could take more energy to burn and this could lead a higher flash point. This structure was also creating a steric barrier around the individual molecules, preventing the self-stacking of the molecules, and hindering the crystallization of the molecules. This could contribute to its low pour point. Moreover, the triglyceride chain structure with C=C of R alkyl chains was saturated and branched with methyl ester (CH_3COO-) functional groups, which could form a network of intermolecular hydrogen bonds. This could lead the CES products to have increased thermal stability and oxidation stability. This was similarly explained by Sharma et al. [7] in the study of synthesizing bio-lubricant from canola oil through the reactions of epoxidation and opening the epoxy ring with an acetic anhydride agent. In the study of Abdullah et al. [35], triester oil was synthesized from starting material oleic acid through successive reactions including epoxidation, epoxy ring opening with oleic acid, and acylation with acetic anhydride, and the synthetic triester oil product has a low pour point value (−73°C), high onset temperature (260°C), and high viscosity index. The authors explained that the increase in chain length and branching by ester groups in the structure of the products could lead to a lowered pour point, increased thermal stability, and increased viscosity index. In our previous study [36], the properties, such as flash point, melting point, viscosity, viscosity index, and oxidation stability of polyol oil product synthesized from catfish oil material through reactions of epoxidation and ring-opening of epoxy with *iso*-propanol, have been significantly improved because the structure of the product is triglyceride chains with branching by hydroxyl and *iso*-propyl groups.

When compared with mineral base oil SN150, SN500 (SN150, SN500) could be generally found that CES was able to meet most of the properties according to the quality standards of the SN150 and SN500. Regarding the higher pour point and lower thermal oxidation stability of CES compared with SN150, SN500 could be fixed by adding an amount of additives of pour point depressant and anti-oxidation to the formulation of bio-lubricant blending from polyester catfish oil.

It could be said that CES could meet the quality standard of SN150, SN500 mineral-based oil for lubrication, friction reduction, anti-wear, and ability of cooling, sealing,

and so on. Therefore, it has a very high potential to be used as an alternative to mineral base oils.

4 Conclusion

The use of the cavitation technique in the reaction process has led to a very high yield of the polyester catfish oil (94.6%) at reaction conditions as inlet pressure of 60 psi, the molar ratio of acetic anhydride/epoxy ring of 1.25/1, 85°C, and the time of only 10 min. Meanwhile, using magnetic stirring could lead to a limited yield of 12%. The analysis results showed that the basic properties of polyester catfish oil have been significantly improved compared to the raw catfish oil and it could be used as an alternative to mineral base oils. The utilization of catfish fat by-products as raw materials for bio-based oil production along with the application of cavitation techniques could create the great potential for sustainable development for biological base oil production and industrial scale-up of the process could be predicted to be easily accomplished. Analysis and assessment of the possibility of using as an alternative to mineral base oil of CES products are necessary to further study. In addition, the formulation of bio-lubricant from CES and the quality assessment of biolubricant according to the quality standards of mineral lubricants should also be conducted in further studies.

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