



Synthesis of Ester from Waste Cooking Oil (WCO) and Pentaerythritol via Dean-Stark Distillation for Potential Use as Biolubricant

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ABSTRACT

Waste cooking oil (WCO) is a group of oils or fats that have been used for cooking or frying repeatedly and are no longer suitable for use. Repeated heating of cooking oil will cause the formation of trans fatty acids that trigger coronary heart disease (CHD). This study aims to synthesize esters as biolubricant candidates from WCO. WCO was purified using 0.05% (v/v) of H₃PO₄ and 2% (w/v) of bleaching earth, then saponified with ethanolic NaOH and hydrolyzed with 25% H₂SO₄. The fatty acids obtained were then esterified with pentaerythritol using a dean-stark distillation apparatus with 2% H₂SO₄ catalyst and toluene solvent at a temperature of 170-180°C for 6 hours. The results of the acid number analysis showed an ester conversion of 83.08%. Furthermore, the FT-IR spectra showed the presence of C=O (1730 cm⁻¹) and C-O-C (1178 cm⁻¹) groups, indicating the formation of esters. These results indicate that esters from used cooking oil have the potential to be environmentally friendly biolubricants.

Keyword: Biolubricant, Dean-Stark Distillation, Pentaerythritol, Synthesis, Waste Cooking Oil.

ABSTRAK

Minyak goreng bekas (WCO) merupakan kelompok minyak atau lemak yang sudah digunakan untuk memasak atau menggoreng secara berulang dan tidak layak untuk digunakan lagi. Pemanasan berulang pada minyak goreng akan menyebabkan terbentuknya asam lemak trans yang memicu penyakit jantung koroner (PJK). Penelitian ini bertujuan mensintesis ester sebagai kandidat biopelumas dari WCO. WCO dimurnikan menggunakan H₃PO₄ 0,05% (v/v) dan *bleaching earth* 2% (b/v), lalu disaponifikasi dengan NaOH etanolik dan dihidrolisis dengan H₂SO₄ 25%. Asam lemak yang diperoleh kemudian diesterifikasi dengan pentaeritritol menggunakan alat destilasi *dean-stark* dengan katalis H₂SO₄ 2% dan pelarut toluena pada suhu 170-180°C selama 6 jam. Hasil analisis bilangan asam menunjukkan konversi ester sebesar 83,08%. Selanjutnya, spektra FT-IR menunjukkan adanya gugus C=O (1730 cm⁻¹) dan C-O-C (1178 cm⁻¹), menandakan terbentuknya ester. Hasil ini menunjukkan bahwa ester dari minyak goreng bekas berpotensi sebagai biopelumas ramah lingkungan.

Kata Kunci: Alat Destilasi *Dean-Stark*, Biopelumas, Minyak Goreng Bekas, Pentaeritritol, Sintesis.

1. Introduction

Lubricants are fluids that reduce the frictional resistance between moving metal surfaces [1]. They are widely utilized across various industries, including drilling, automotive, and manufacturing [2]. Conventional lubricants are typically derived from fossil fuels [1]. However, these petroleum-based products pose significant risks to human health and the environment due to their toxicity and poor biodegradability [2]. In contrast, vegetable oil-derived lubricants, known as biolubricants, offer several advantages, including biodegradability, non-toxicity, minimal adverse effects on human health, and cost-effectiveness [3]. Biolubricants are employed



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in the form of fatty acid ester compounds, which provide superior lubrication properties due to the polarity of ester groups that facilitate durable adhesion in forming effective protective layers [4]. Current biolubricant production still relies on vegetable oils as raw materials, potentially creating conflicts with their use as food sources. A highly promising alternative source of fatty acids is waste cooking oil, which represents an underutilized resource.

Waste Cooking Oil (WCO) comprises oils or fats that have been repeatedly used for frying in household, commercial, or industrial food processing activities. The properties of waste cooking oil can vary depending on frying conditions, such as temperature and cooking duration. Waste cooking oil is made up of many kinds of vegetable oils, such as sunflower, palm, rapeseed, soya, etc]. Improper disposal of WCO leads to numerous environmental issues, including water and soil contamination, human health concerns, and disruption of aquatic ecosystems. Repeated use of cooking oil can trigger various health problems, including hypertension, atherosclerosis (potentially leading to coronary heart disease), and osteoporosis [6]. Contaminants present in WCO also render it unsuitable for food applications [7].

Despite limitations such as high free fatty acid and water content, WCO is considered one of the most promising bio-oil feedstocks [8, 9]. Common industrial applications of waste cooking oil are related to energy production through direct combustion [10, 11] or biofuel synthesis [12, 13]. WCO also represents an attractive alternative for biolubricant production due to its biodegradability and renewability [14]. Furthermore, WCO consists of saturated and unsaturated fatty acids, making it resistant to low temperatures and stable at high temperatures when used as biolubricant [15]. However, the content and properties of impurities in WCO limit its utilization through chemical, catalytic, biochemical, or thermochemical transformations. Consequently, various methods have been evaluated for WCO pretreatment prior to valorization [6]. In this research, WCO purification was conducted through the addition of H_3PO_4 and bleaching earth, which function to adsorb metal ion particles on the surface [16]. Biolubricants typically utilize high molecular weight alcohols to enhance stability, with pentaerythritol being one such alcohol.

Pentaerythritol is an organic compound with multiple hydroxyl groups (-OH). It is employed as a fundamental component in the synthesis of compounds such as resins, lubricants, antioxidants, polyurethanes, and explosives [17]. Incorporating polyhydric alcohols like pentaerythritol into fatty acid significantly improves the thermal and oxidative stability of the lubricants, particularly at elevated temperatures [18]. Pentaerythritol esters as lubricants exhibit several physicochemical characteristics, including low saturated vapor pressure [19], biodegradability [20], and high thermal-oxidative stability [21]. The presence of four -OH groups in the molecule provides opportunities for varied ester synthesis depending on the type of acid fragment or mixture [22].

In previous research, [23] synthesized complex pentaerythritol esters through esterification using pentaerythritol and dibasic acid catalyzed by tetrabutyl titanate. The reaction was conducted under nitrogen protection and gradually heated to 220°C for 8 hours. Separation was also performed through vacuum distillation. In other research, the use of Dean-Stark distillation has emerged as a promising method for esterification due to its efficiency in equipment and time. The Dean-Stark trap is used to collect water as a by-product in esterification. Since esterification is reversible, removing water is crucial to increase product yield in accordance with Le Chatelier's principle [24]. Based on the above discussion, the researchers are interested in synthesizing ester compounds from waste cooking oil with pentaerythritol using sulfuric acid catalyst, toluene solvent, and Dean-Stark apparatus, which have potential as biolubricants. The acid value determination was performed by alkalimetric titration to determine the conversion of fatty acids to esters. Functional group changes were determined using FT-IR spectrophotometry.

2. Materials and Methods

2.1. Equipments

The equipments and instruments used in this research were hot plate, vacuum erlenmeyer 500 ml, vacuum pump, buchner vacuum, burette 25 ml, beaker glass 100 ml, beaker glass 1 L, erlenmeyer 100 ml, drop pipette, measuring flask 250 ml, separating funnel, three-neck flask, condenser, glass stir rod, analytical balance, clamp and clamp, magnetic bar, Heidolph Rotary Evaporator, and Agilent Cary 630 Spectrophotometer FT-IR.

2.2. Materials

The chemicals and reagents during this research were waste cooking oil from fried food seller in Medan, pentaerythritol, H_3PO_4 , bleaching earth, filter paper, NaOH, distilled water, phenolphthalein indicator, isopropyl alcohol, ethanol, methanol, toluene, H_2SO_4 , ethyl acetate, NaHCO_3 , NaCl, and Na_2SO_4 anhydrous.

2.3. Refinery of Waste Cooking Oil

500 mL of WCO was mixed with H_3PO_4 0.05% and bleaching earth 2% in a vacuum Erlenmeyer. Then, the solution was heated at 105°C and stirred at a constant speed for 90 mins in vacuum conditions. Subsequently, the mixture was filtered to remove the adsorbent from the solution. Finally, the acid value of refined WCO was analyzed by alkalimetry titration.

2.4. Hydrolysis of Refined Waste Cooking Oil

In this step, 250 g of refined WCO was mixed with NaOH ethanolic 12% in a beaker glass 1 L. The mixture was stirred and heated at 57°C for 75 mins. Then, the mixture was cooled until the formation of fatty acid salt. After that, the remaining ethanol and unsaponifiable oil were separated from the salt with a vacuum pump. The filter cake was washed with 400 ml of ethanol and added with H_2SO_4 25% until the $\text{pH}=3$ to hydrolyze the fatty acid salt and the mixture was stirred for 30 mins. Furthermore, the mixture was dropped into a separating funnel to separate fatty acid and the mixture of ethanol and H_2SO_4 25%. Additionally, the fatty acid was washed with warm water until the water was neutral. Finally, the fatty acid was filtered and evaporated to separate the remaining water on a hot plate at 110°C . The acid value of WCO fatty acid was conducted to analyze the effectiveness of the hydrolysis process.

2.5. Synthesis of Ester from Waste Cooking Oil Fatty Acid and Pentaerythritol

Approximately 10.8256 g fatty acid derived from waste cooking oil (WCO) and 1.36 g of pentaerythritol were placed into a three-neck flask. Subsequently, 100 ml of toluene and H_2SO_4 2% were introduced as solvent and catalyst. The reaction was conducted at $170\text{--}180^\circ\text{C}$ for 6 hours utilizing Dean-Stark distillation apparatus under continuous stirring and refluxing. Then, cool the mixture in room temperature. Toluene was subsequently extracted from the mixture via rotary evaporator at 100°C . Additionally, 25 ml of ethyl acetate and 10 ml of saturated NaHCO_3 solution were introduced to the mixture, after which the mixture was dropped into a separating funnel and shake it vigorously. Separate the organic layer and wash it with saturated NaHCO_3 solution three times, followed by wash the organic layer with 10 ml of saturated NaCl solution and 10 ml of distilled water two times. The washing procedure was sustained with distilled water until a neutral pH of 7 was attained. The remaining toluene and ethyl acetate were eliminated via a rotary evaporator. Anhydrous Na_2SO_4 was included into the ester to retain the remaining water. The acid value of ester was analyzed to ascertain the % conversion of ester, while the ester functional groups were characterized using FT-IR Spectroscopy.

2.6. Determination of Acid Value for Refined Waste Cooking Oil, Fatty Acid, and Ester

Acid value is amount of alkali (NaOH or KOH) which is needed to neutralize acids in 1 g of sample mixture [25]. Determination of acid value was based on the AOCS method Te 1a-64. Approximately 10 g of WCO or 0.2 g fatty acid/ester was mixed with 50 ml of solvents (Isopropyl Alcohol:Ethanol, 1:1) followed by 3 drops of phenolphthalein. Then, the mixtures were boiled for five minutes and titrated with NaOH 0,1 N until a pale pink color was appeared. The titration was carried out for three times and the determination of acid value was by using the Equation (1).

$$AV = \frac{V_t \times N_{\text{NaOH}} \times 56,1}{W} \quad (1)$$

Where:

V_t = volume of titrant (mL)
 N_{NaOH} = normality of NaOH
 56,1 = molecular weight of KOH
 W = weight of sample (g)

3. Results and Discussion

3.1. Refinery of Waste Cooking Oil

The Waste Cooking Oil (WCO) utilized in this study was collected from commercial food vendors in Medan following multiple frying cycles, containing significant levels of thermal degradation products and food residues. Purification of the WCO was conducted to remove these contaminants and enhance its suitability

for subsequent esterification reactions. This purification step is essential as thermally degraded WCO contains various detrimental compounds including oxidized triglycerides, polymeric material, and polar compounds that can significantly impede catalytic activity and reaction efficiency during esterification [26, 27].

Previous studies have demonstrated that untreated WCO with high levels of impurities results in lower conversion rates, increased side reactions, and inferior product properties when synthesizing biolubricants [15]. Furthermore, [10] reported that free fatty acids and moisture content in unpurified WCO can deactivate catalysts and promote hydrolysis reactions that compete with the desired esterification pathway. The refinement process involved treating the WCO with 0.05% (w/w) H_3PO_4 and 2% (w/w) bleaching earth under vacuum conditions at controlled temperature ($85 \pm 2^\circ\text{C}$), optimizing the adsorption of polar compounds and suspended particulates. This approach aligns with methodologies established by [28], who demonstrated that acid-activated bleaching earth effectively reduces peroxide values and removes trace metals that could catalyze oxidative degradation of the final biolubricant product. To enhance separation efficiency and filtration kinetics, vacuum-assisted filtration was implemented, yielding a purified substrate as illustrated in the process schematic (Figure 1).

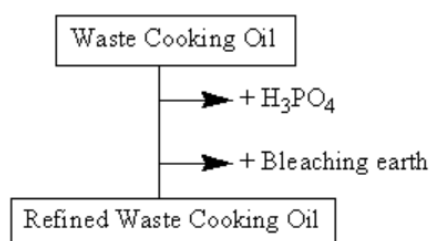


Figure 1. Refinery of Waste Cooking Oil

Acid value determination was performed on the refined WCO to establish baseline acidity parameters and enable quantitative comparison with the subsequently produced WCO fatty acid fraction. The differential acid values between refined WCO and its fatty acid derivative provided a critical metric for evaluating the efficacy of the hydrolysis procedure. Analytical measurements indicated that the refined WCO exhibited a mean acid value of 22.49 mg KOH/g, establishing the initial carboxylic acid concentration prior to pentaerythritol esterification. Average acid value of refined WCO as calculation below:

$$AV_1 = \frac{19.9120 \times 0.1003 \times 56.1}{5.0401} = 22.23 \text{ mg KOH/g}$$

$$AV_2 = \frac{22.1230 \times 0.1003 \times 56.1}{5.5402} = 22.47 \text{ mg KOH/g}$$

$$AV_3 = \frac{20.7380 \times 0.1003 \times 56.1}{5.1219} = 22.78 \text{ mg KOH/g}$$

$$\text{Average AV} = \frac{22.23 + 22.47 + 22.78}{3} = 22.49 \text{ mg KOH/g}$$

3.2. Hydrolysis of Refined Waste Cooking Oil

Hydrolysis of triglyceride components in the refined WCO was conducted to generate free fatty acids required for subsequent esterification with pentaerythritol. While conventional industrial hydrolysis of triglycerides typically employs extreme conditions ($250\text{--}260^\circ\text{C}$, 4–8 MPa) as described by [29], this study implemented a two-step approach to facilitate the hydrolysis under milder laboratory conditions. The initial step involved saponification of the refined WCO using 12% (w/v) ethanolic NaOH, followed by acidification of the resultant fatty acid salts with 25% (v/v) H_2SO_4 . This methodological approach circumvents the high energy demands of direct hydrolysis, consistent with findings by [30], who demonstrated that alkaline saponification followed by acidification achieves comparable fatty acid yields to direct hydrolysis while requiring substantially reduced thermal input. The reaction mechanism for this two-step hydrolysis process is depicted in Figure 2.

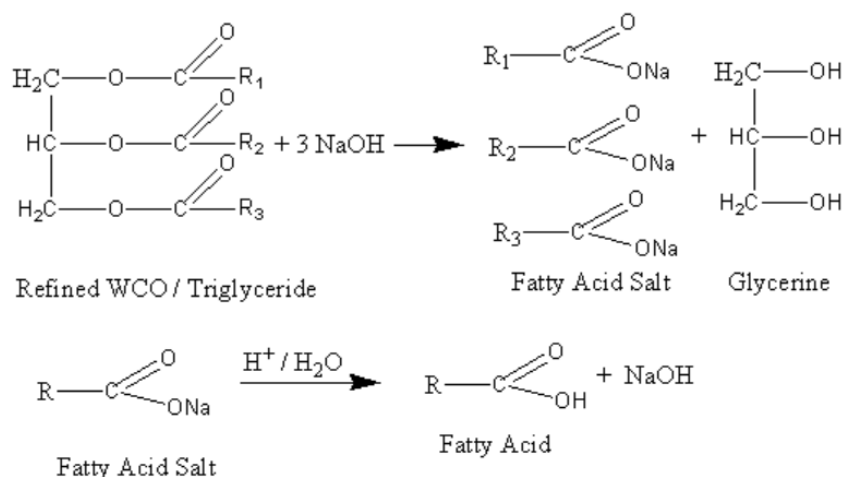


Figure 2. Hydrolysis of Refined Waste Cooking Oil

The saponification reaction proceeded efficiently at moderate temperatures (65-70°C), while the subsequent acidification to liberate free fatty acids was conducted at ambient temperature with vigorous agitation, eliminating the need for additional thermal energy input. This strategic modification to the conventional hydrolysis protocol proved particularly advantageous for laboratory-scale operations, aligning with green chemistry principles outlined by [31] for sustainable biolubricant synthesis. The resulting WCO-derived fatty acids manifested as a pale-yellow semisolid material with a measured acid value of 152.27 mg KOH/g. Average acid value of fatty acid as calculation below:

$$AV_1 = \frac{6.6580 \times 0.1003 \times 56.1}{0.2462} = 152.17 \text{ mg KOH/g}$$

$$AV_2 = \frac{5.8640 \times 0.1003 \times 56.1}{0.2166} = 152.34 \text{ mg KOH/g}$$

$$AV_3 = \frac{5.4594 \times 0.1003 \times 56.1}{0.2017} = 152.30 \text{ mg KOH/g}$$

$$\text{Average AV} = \frac{152.17 + 152.34 + 152.30}{3} = 152.27 \text{ mg KOH/g}$$

The substantial increase in acid value from the refined WCO (22.49 mg KOH/g) to the liberated fatty acids (152.27 mg KOH/g) provides quantitative confirmation of successful triglyceride hydrolysis, consistent with theoretical calculations based on triglyceride molecular weight distributions in WCO as reported by [32]. The isolated fatty acid fraction served as the primary reactant for subsequent esterification with pentaerythritol to synthesize the target biolubricant.

3.3. Synthesis of Ester from Waste Cooking Oil Fatty Acid and Pentaerythritol

Ester from fatty acid and pentaerythritol is potentially as biolubricant [1]. Tabel 1 shows several publications describing synthesis of esters as biolubricants using fatty acids and some kinds of alcohol. All researchers used polyols such as neopentylglycol (NPG), trimethylolpropane (TMP), or pentaerythritol (PE). Polyol esters have extreme stability due to their high molecular weight [2].

Table 1. Several biolubricants from esterification of fatty acids with polyols

Fatty Acid	Polyol	Method	References
10-undecylenic acid	PE	Dean-Stark apparatus and reflux condenser	[1]
Palm Fatty Acid Distillate (PFAD)	TMP or PE	Reflux	[3]
n-pentanoic acid	PE	Reflux under nitrogen protection	[22]
Crude Palm Oil (CPO) fatty acid	NPG	Dean-Stark apparatus	[33]

Esterification of the WCO-derived fatty acids with pentaerythritol was executed employing Dean-Stark distillation apparatus to facilitate continuous water removal and drive reaction equilibrium toward product formation. The reaction was conducted utilizing 2% (w/w) H_2SO_4 as a catalyst and toluene as an azeotropic solvent at 170-180°C for 6 hours, optimizing both kinetic and thermodynamic parameters. This temperature range was selected based on findings by [34], who identified 175°C as the optimal temperature balancing reaction rate and minimizing thermal degradation of fatty acid chains. The reaction mechanism for the formation of pentaerythritol tetraester is illustrated in Figure 3.

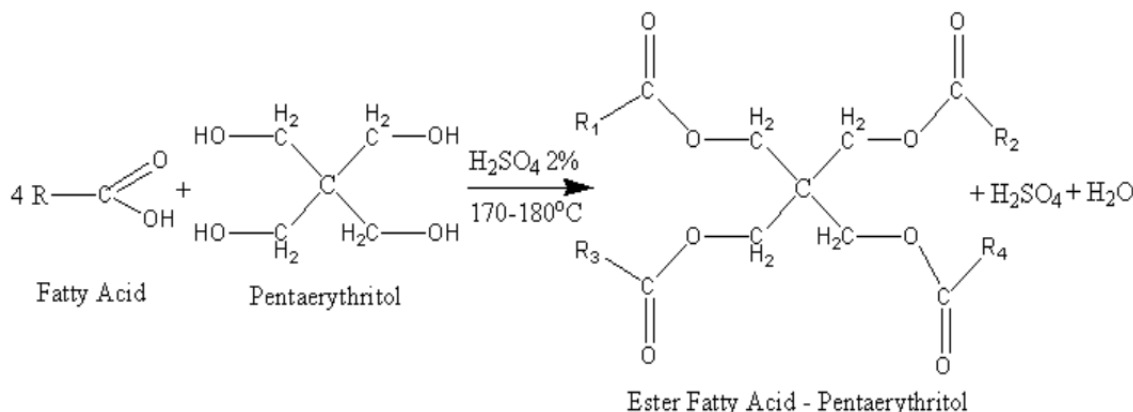


Figure 3. Esterification of WCO Fatty Acid with Pentaerythritol

Following synthesis, the crude product underwent sequential purification protocols including solvent extraction by ethyl acetate, saturated NaHCO_3 solution, saturated NaCl solution, and distilled water to remove unreacted substrates and catalyst residues. The purified pentaerythritol fatty acid ester presented as a pale-yellow liquid with substantially reduced acid value compared to the starting material. Acid value determination was strategically employed to quantitatively assess the extent of esterification, as residual carboxylic acid functionalities directly correlate with unconverted fatty acids, consistent with analytical approaches described by [35]. Triplicate acid value measurements yielded a mean value of 25.82 mg KOH/g for the synthesized ester, representing an 83.08% conversion efficiency as calculated via Equation 2.

$$\text{AV}_1 = \frac{15.0870 \times 0.1003 \times 56.1}{3.2503} = 26.12 \text{ mg KOH/g}$$

$$\text{AV}_2 = \frac{16.0400 \times 0.1003 \times 56.1}{3.5211} = 25.63 \text{ mg KOH/g}$$

$$\text{AV}_3 = \frac{15.140 \times 0.1003 \times 56.1}{3.3124} = 25.72 \text{ mg KOH/g}$$

$$\text{Average AV} = \frac{26.12 + 25.63 + 25.72}{3} = 25.82 \text{ mg KOH/g}$$

$$\% \text{ Conversion} = \frac{\text{Average AV of WCO fatty acid} - \text{Average AV of ester}}{\text{Average AV of WCO fatty acid}} \times 100\% \quad (2)$$

$$\% \text{ Conversion} = \frac{152.27 - 25.82}{152.27} \times 100\%$$

$$\% \text{ Conversion} = 83.08\%$$

This conversion rate significantly exceeds previously reported values of approximately 60% by [36] under comparable reaction conditions, likely attributable to the optimized water removal strategy employed in the present study. The enhanced conversion efficiency is particularly significant as higher molecular weight esters with minimal residual carboxylic acid functionality exhibit superior tribological properties. This improvement can be attributed to the increased polar ester functionalities that promote adhesion to metal surfaces while

maintaining appropriate molecular spacing, as elucidated in comprehensive structure-property relationship studies by [37] thereby enhancing boundary lubrication performance in high-load applications.

Further analysis was done for the ester by Spectrophotometer FT-IR to strengthen the evidence that formation ester was occurred. The scanning wavenumber were from 650 cm^{-1} to 4500 cm^{-1} . The difference spectrum of fatty acid and ester was shown in Figure 4.

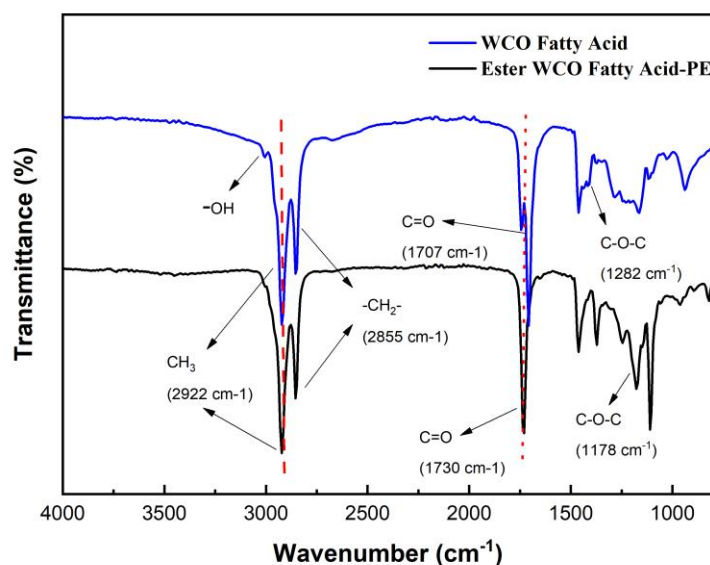


Figure 4. The Difference FT-IR Spectrum of WCO Fatty Acid and Ester

The FTIR spectral comparison between waste cooking oil (WCO) fatty acid and its corresponding ester (WCO fatty acid-PE) provides definitive evidence of successful esterification. This transformation represents a significant valorization pathway for waste oils, converting problematic waste into value-added products. The most compelling evidence of esterification is observed in two key spectral regions. First, the hydroxyl (-OH) stretching band ($3000\text{--}3500\text{ cm}^{-1}$) present in the WCO fatty acid spectrum shows significant reduction in the ester product. This indicates consumption of carboxylic acid groups during the reaction, as documented by [38]. Second, the characteristic carbonyl (C=O) stretching vibration shifts from 1707 cm^{-1} in the fatty acid to 1730 cm^{-1} in the ester. This 23 cm^{-1} increase aligns with [39], who reported similar shifts upon esterification of vegetable oils. The enhanced C-O-C stretching vibrations at 1282 cm^{-1} and 1178 cm^{-1} in the ester spectrum further confirm successful conversion. These bands represent the ester linkage formation and match the findings of [40], who observed comparable peaks in biodiesel produced from waste oils.

The alkyl group vibrations (CH_3 at 2922 cm^{-1} and CH_2 at 2855 cm^{-1}) remain largely unchanged between the spectra, consistent with expectations since the hydrocarbon chain structure is preserved during esterification. Similar observations were reported by [41] in their comprehensive review of biodiesel production techniques. This spectroscopic evidence suggests high conversion efficiency in the esterification process. The results align with [42], who demonstrated that proper esterification of WCO can yield products with properties comparable to those derived from virgin oils. Future work should focus on quantifying the conversion rate through peak intensity ratio analysis and correlating spectral features with physicochemical properties of the produced esters.

4. Conclusion

This study successfully synthesized pentaerythritol fatty acid esters from waste cooking oil through three integrated steps: purification, hydrolysis, and esterification. Purification with 0.05% H_3PO_4 and 2% bleaching earth effectively removed contaminants, producing oil with an acid value of 22.49 mg KOH/g . Two-step hydrolysis—alkali saponification and acid hydrolysis—successfully decomposed triglycerides under moderate conditions, producing fatty acids with an acid value of 152.27 mg KOH/g . Esterification using Dean-Stark distillation resulted in a high conversion of 83.08% , exceeding previous reports ($\sim 60\%$). This approach offers

a sustainable method for converting waste cooking oil into high-performance biolubricants, supporting the principle of a circular economy and potentially applying in the environmentally friendly lubrication industry.

5. Acknowledgments

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6. Conflicts of Interest

There is no conflict of interest linked with this work.

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