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Conversion of Wastewater from Coconut Oil Manufacturing into Biodiesel: Optimization of Sulfuric Acid Pre-Treatment for High Free Fatty Acid Feedstock

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Abstract

One of the most important routes of implementing the circular economy is the sustainable transformation of industrial waste streams into renewable energy. This paper explores the optimization of biodiesel production from waste coconut oil, a byproduct of coconut milk processing wastewater, and addresses the technical difficulties posed by high free fatty acid (FFA) content. Waste coconut oil was produced by extracting waste from the coconut processing plant located in the Kalutara District, Sri Lanka, using approximately 20 Liters of wastewater (14% oil content) in a two-stage gravitational-centrifugal separation. The resulting feedstock had a drastic degradation of 23.8% FFA (acid value: 118.9 mg KOH/g), which required acid- catalysed esterification pretreatment followed by the conventional alkali-catalysed transesterification. An experimental comparative study was used to assess the efficacy of two concentration levels of sulfuric acid (1 percent and 5% of the weight) in the reduction of FFA. The H_2SO_4 5% treatment was the best treatment as it decreased FFA of 23.8% to 1.8% (92.4% and 84.9%, respectively) after 90 minutes of treatment at 60 ^{0}C compared to 1% treatment that decreased FFA to 3.6%. The resulting transesterification reaction with sodium hydroxide under alkali catalysis to form biodiesel, which had specific quality properties depending on the treatments. Biodiesel obtained with 5%. acid pretreatment had a kinematic viscosity of 5.723 mm^2/s, a density of 878.3 kg/m^3, and a soap content of 124 ppm in compliance with ASTM D6751 specification and close to EN 14214 specifications. The 1% treatment produced biodiesel with 6.386 mm^2/s viscosity, 881.7 kg/m^3 density, and high soap content (386 ppm), which is higher than the EN 14214 viscosity. Economic evaluation showed that there was a good viability with a projected cost of production of $0.20/L compared to the market prices of 0.90-1.10/L. This study shows that waste coconut oil can be successfully utilized to produce quality biodiesel with optimal 5% sulfuric acid pre-treatment that would bring environmental benefits such as waste valorisation, the decrease of greenhouse gases, and the development of the circular economy and the production of economically viable renewable fuel for the usage of diesel engines.

Keywords: Bio Diesel, Circular Economy, Esterification, Free Fatty Acid, Sulfuric Acid Catalyst

Introduction

As the world is moving towards sustainable solutions, the need for alternatives to finite fossil fuels must be considered, and the necessary actions must be taken to address climate change, reduce greenhouse gas emissions, and research and find renewable energy alternatives. Biodiesel is an excellent substitute for petroleum-based diesel fuel due to its unique nature of biodegradability, renewability, and compatibility with modern diesel engines (Cerón Ferrusca et al., 2023). Biodiesel can be defined chemically as fatty acid methyl esters (FAME) or fatty acid ethyl esters (FAEE) (Katagi et al., 2023). Biodiesel is produced via transesterification from vegetable oils, animal fats, and waste oils. The traditional biodiesel industry has focused on using edible vegetable oils such as soybean, rapeseed, palm, and sunflower oils as primary feedstocks (Jamal et al., 2022). However, when it comes to food security, shortage of agricultural lands, and overall production cost, the use of food-grade oils for biodiesel production should be carefully reconsidered. These challenges have stimulated the research efforts to identify the utilized biodiesel production methods using non-edible and waste-based lipid sources. In the case of waste oils and industrial processing residues, they represent attractive feedstocks (Ifeanyi-Nze et al., 2023). They address waste management issues and also provide economically viable raw materials, and finally minimize the competition with food supply chains. When it comes to the coconut processing industry, it usually generates substantial amounts of wastewater containing huge quantities of oil and fat residues (Khosa et al., 2024). In the case of coconut milk production, usually 15-25% of the oil content is lost in the wastewater streams. The released oil content normally accumulates as layers of floating fat in settling ponds or wastewater treatment facilities. These waste coconut oils, which are rich in high energy content but usually released as problematic waste that directly contributes to the pollution of the environment (Kumar et al., 2025). Moreover, costly disposal methods are often required. This accumulated waste can give off unpleasant odors, pest issues, and also create problematic drainage systems due to blocking, and in the end, results in water quality deterioration if not managed properly.

These kinds of waste oils are an unconsidered resource with high potential for biodiesel production. Coconut oil mainly consists of medium-chain saturated fatty acids, which are lauric acid (C 12:0) and myristic acid (C 14:0), which are responsible for approximately 60-70% of its fatty acid composition (Pikula et al., 2020). The chemical characteristics represented by these coconut oil derived biodiesel with some positive combustion properties, such as high

cetane numbers and better oxidative stability. Furthermore, the production of coconut oil-derived biodiesel aligns with circular economy principles, giving some benefits like waste valorization, minimized environmental pollution, and sustainable energy generation (Masudi et al., 2023). As there is a very promising potential of waste coconut oil as a biodiesel feedstock, at the same time, there are significant technical challenges that should be faced to reach efficient conversion. The primary challenge is characteristically high free fatty acid (FFA) content recovered from wastewater streams. Refined vegetable oils typically have less than 1% FFA, but the waste coconut oils contain FFA levels that range from 10% to over 40%, and these FFA levels depend on the degree of hydrolytic degradation, storage conditions, and processing methods employed (Nnaji, 2020). High FFA contents pose critical complications for traditional biodiesel production via alkali-catalyzed transesterification. Due to the rapid reaction kinetics and high conversion efficiency, this method remains the most widely used industrial method for biodiesel production. In the case of higher FFA contents, when FFA comes up against alkaline catalysts such as sodium hydroxide (NaOH) or potassium hydroxide (KOH), they undergo neutralization reactions rather than transesterification, resulting in soap formation through a saponification process. Soap formed by this series of reactions results in operational problems, including catalyst deactivation, separation difficulties, yield reduction, complications in purification, and quality degradation. These challenges can be omitted by the method of acid-catalyzed esterification pre-treatment with the use of sulfuric acid, so the FFA content can be reduced before the alkali-catalyzed transesterification (Burmana et al., 2025). In this method, FFA reacts with methanol in the presence of sulfuric acid, and it forms fatty acid methyl esters, generating water as a byproduct. The catalyst concentration plays a major role in the effectiveness of acid esterification; insufficient acid levels result in incomplete conversion of FFA, and excessive concentration will result in equipment corrosion and increase the cost of purification. Therefore, the concentration of sulfuric acid must be optimized according to the feedstock composition, initial FFA level, and physical properties (Ahmed & Huddersman, 2022). As there is much research conducted on the production of biodiesel with various waste oils, there are still limited investigations on using coconut oil milk processing wastewater as a feedstock. The existing publications focused on using refined coconut oils, which have relatively low FFA content, and the comparison of different sulfuric acid concentrations for the very high FFA contents of waste oils still remains unexplored. This research pays attention to these knowledge gaps with the investigation of coconut oil extracted from wastewater and comparing 1% vs 5% sulfuric acid pre-treatment concentrations on

neutralization of FFA, formation of soap, and the quality of the final biodiesel (Foo et al., 2022). As the primary objectives of this research, developing and validation of a complete process for extracting and converting coconut oil from wastewater into high quality biodiesel, comparing the effectiveness of 1% vs 5% acid concentrations in reducing the content of FFA, evaluating the quality parameters of biodiesel which including kinematic viscosity and soap content with ASTM D6751 and EN 14214 standards, and assessment of formation levels of soap and investigation of minimization strategies can be taken (Shabbir et al., 2022). This research aims to contribute to sustainable waste management and renewable energy production by exhibiting an economically viable method for the transformation of coconut processing waste into high-quality biodiesel. By the utilization of industrial waste as fuel feedstock, our study demonstrates circular economic practices and reduces the pollution of the environment and also provides alternative streams for coconut processing industries (Bano et al., 2020). The results of the research contribute to improving biodiesel production strategies for high FFA oils and providing valuable insights for applications in industries and the development of policies that support biofuel adoption strategies.

Methodology

Research Design and Experimental Approach

This study employed a laboratory-scale experimental design for biodiesel production from the feedstock, which is extracted from coconut milk processing wastewater (Abdul Hakim Shaah et al., 2021). The experiment was conducted under four sequential phases: (1) feedstock extraction and characterization, (2) acid-catalyzed esterification pre-treatment, (3) alkali-catalyzed transesterification, and (4) biodiesel purification and analysis for biodiesel quality. To evaluate the effects of two acid concentrations (1% and 5% w/w) on FFA reduction efficiency and final biodiesel quality, a comparative methodology was implemented (Elgharbawy et al., 2021). The research methodology was built to simulate conditions that could be scaled to industrial operations by maintaining precise control over experimental variables for systematic comparison. Each experiment was conducted in triplicate to ensure statistical reliability and to ensure reproducibility of the results. This study followed standard analytical procedures based on ASTM and AOCS to standardize data quality and comparability with biodiesel international standards (Nguyen Thoai et al., 2019).

Materials and Reagents

Raw Materials

Waste coconut oil was selected as the primary feedstock, which is taken from a coconut oil manufacturing facility located in Kalutara District, Western Province, Sri Lanka. The waste oil was extracted from the wastewater settling pond, which accumulates naturally on the surface of the water pond, which comes after the coconut milk extraction processes. Approximately 20 liters of mixed samples were collected with the use of polyethylene containers and transported to the laboratory in less than 24 hours to minimize further degradation (Onn et al., 2023). The mixture of wastewater consists of waste coconut oil, water, coconut milk solids, and other residues. In order to retard microbial activity and hydrolytic degradation of lipids, the sample was stored at 4 °C until processing (Yadav et al., 2023). After the initial visual inspection, it was revealed that it contains a heterogeneous mixture with sharp layers of yellowish-brown oil, a middle emulsion layer, and a bottom aqueous layer, which contains suspended solids (Bashir et al., 2020).

Figure 1: Oil Separation after Centrifugations

Chemicals and Reagents

Chemicals used in this research are analytical and reagent grade to ensure the accuracy and reliability of results. The chemical and their purposes are listed in table 1 (David et al., 2023).

Table 1: Chemicals and Reagents used

Chemical	Purpose
Methanol (CH_3OH)**:** ≥99.5% purity, HPLC Grade	As an alcohol reactant for esterification and transesterification.
Sulfuric Acid (H_2SO_4): 95-98% concentration, ACL reagent grade	As acid catalyst for esterification pre-treatment.
Sodium Hydroxide (NaOH): ≥98% purity, pellet form	As alkaline-catalyst for transesterification.
Phenolphthalein Indicator: 1% solution in ethanol	For FFA titration
Ethanol (C_2H_5OH): 95% purity	Solvent for FFA identification and washing purposes.
Distilled Water: Laboratory grade (Conductivity <1 μS/cm)	For washing biodiesel and preparing solutions
Hydrochloric Acid (HCL): 0.1M standard solution (volumetric standard)	For FFA titration

Feedstock Recovery and Characterization

Oil Recovery from Wastewater

The extraction of waste coconut oil from the wastewater, which contains coconut oil, was done through a two-stage extraction process, which is specially designed to extract oil yield with minimal water and solid contaminants. Stage 1 is called the Primary Gravitational Separation. In this method, the collected wastewater mixture was sent to a large settling flask and heated gently to 50-55 °C with mild agitation, resulting in viscosity and promoting oil-water

separation (Vilas Bôas & Mendes, 2022). After that, the mixture is allowed to stand undisturbed for 12-16 hours (at room temperature). After following these operations, three distinct layers formed, which are (1) top layer (semi-solid waste coconut oil, which is yellowish-brown in color at room temperature), (2) middle layer (emulsified oil and water mixture with suspended solids and some debris), (3) bottom layer (aqueous phase with heavy suspended oil and debris). Then the top layer was collected using a ladle or skimming device, taking care to reduce entrainment of water and emulsified matter. 60-70% of the total oil content is extracted from this primary separation (Patel & Karve, n.d.).

Stage 2 is called the centrifugal separation. To complete the oil extraction and removal of residues, centrifugal separation was performed using the primary separated oil. The extracted oil (with the middle emulsion layer) was subjected to high-speed centrifugation. The process was operated at 4500 rpm for 15-20 minutes at a temperature of 40-45°C. The centrifugal force effectively separated the available oil from water and milk solids along with density differences. Oil (density ≈ 0.91-0.92 g/cm³) migrated to the upper part, water and dissolved solids (density ≈ 1.00-1.02 g/cm³) came to the middle part, and suspended solids precipitated at the bottom. After this process, the oil, which is in the separated layer of oil, was decanted into clean, dry flasks. The color of the extracted oil was yellowish to light brown, and it was a semi-solid material at room temperature (typical of coconut oil due to its high saturated fat content and melting point around 24-25°C). The visual appearance of the separated oil layer is shown in Figure 1. In order to remove the residual moisture, the extracted oil was subjected to preliminary heat treatment. The extracted oil was subjected to heat in a stainless-steel container at 100-105 °C for 30-45 minutes while stirring gently until it reached zero steam evolution. This is a critical step because if some water content remains, it interferes with subsequent acid esterification because of the dilution of the acid catalyst and promotion of hydrolysis reactions. After that process, the oil was cooled to room temperature and filtered with filter paper to remove any remaining particulate matter. The final extracted waste coconut oil was stored in amber glass bottles in the presence of nitrogen or in tightly sealed containers at 4°C in order to prevent further oxidative degradation and hydrolysis until use in further experiments.

Feedstock Characterization

The final extracted waste coconut oil was characterized for various properties, such as physical properties and chemical properties, which are critical for the design and optimization of the biodiesel production process. In order to determine the FFA, by using the standard acid-base titration method with the following AOCS Official method Ca 5a-40. This is a crucial parameter because it determines the extent of pre-treatment that is required and predicts the potential formation of soap during transesterification. The process was conducted in 5 steps. At first, approximately 5-10g of the oil sample was weighed into a 250ml Erlenmeyer flask. And then, 50ml of neutral ethanol (95%) was mixed and heated gently to dissolve the oil. Then, 2-3 drops of 1% phenolphthalein indicator solution were added. After that, the solution was titrated against a 0.1 M NaOH solution by following continuous swirling until a faint pink color persisted for at least 30 seconds. Finally, the titration was conducted in triplicate for accuracy (Moser, 2009).

FFA content (as % lauric acid) was calculated by using: FFA (%) = (V x M x MW x 100) / (W x 1000). Where, V = volume of NaOH solution in ml, M = molarity of NaOH solution in mol/L, MW = molecular weight of lauric acid = 200 g/mol, and W = weight of oil sample in (g) (Demirbas et al., 2016). In order to determine the moisture content by using the Karl Fischer titration method (ASTM D6304). It is essential to determine the moisture content accurately because water enhances hydrolysis, soap formation, and reduces the effectiveness of catalysts. By using the Cannon-Fenske viscometer, the viscosity of the raw oil was measured at 40 °C following the ASTM D445 method. Viscosity affects the processability and provides basic data for comparison with the final biodiesel product. A calibrated pycnometer following the ASTM D1298 method was used to measure the density at 15 °C. By using the FFA determination results, acid value (mg KOH/g oil) was calculated using: Acid Value = (V x M x 56.1) / W. This is an important parameter because many biodiesel production processes specify the highest acceptable acid values (typically <1 mg KOH/g for direct alkali transesterification). The results obtained from these characterization tests determined process parameter selection for subsequent acid esterification and transesterification reactions (Knothe, 2010).

Acid-Catalyzed Esterification Pre-Treatment

In the case of acid esterification pre-treatment, two experimental series were conducted in parallel to compare the concentrations of sulfuric acids. Sample A was prepared with 5% sulfuric acid treatment by weight of oil, and sample B was prepared with 1% sulfuric acid concentration. All the other parameters were maintained constant. For the treatments, 400 g of extracted waste coconut oil was placed using a three-neck round-bottom flask, which is equipped with a reflux condenser, thermometer, and magnetic stirrer. Based on a 15:1 molar ratio (methanol: FFA), the methanol quantity was calculated to drive the equilibrium toward ester formation. Approximately 228.5g of methanol (288ml) was required for oil with the content of FFA at 23.8%. Sulfuric acid catalyst amounts for sample A: 20g (10.9 ml concentrated H_2SO_4), sample B: 4g (2.2 ml concentrated H_2SO_4) (Demirbas, 2007). Methanol and sulfuric acid were carefully combined in a beaker under cooling. While stirring continuously at 400-500 rpm, the oil was heated to 60°C ± 2°C, then the mixture was slowly added over 5-10 minutes. With stirring continuously, the methanol-acid mixture was maintained at 60 °C for 90 minutes under reflux. Samples were periodically taken out at 0, 30, 60, and 90 minutes for the analysis of FFA. At the end of 90 minutes, the mixture was cooled and sent to a separatory funnel. It formed two phases: the upper esterified oil phase and the lower methanol-water phase. In order to remove residual sulfuric acid, impurities, and methanol, the upper oil phase was washed three times with warm distilled water. (100ml per wash at 40-50°C) (Ghosh et al., 2024). The lower phase was drained. After each wash, the mixture settled for 30-60 minutes before draining the aqueous phase. In order to evaporate remaining water, the washed oil was heated to 100-105 °C for 30-45 minutes. And then, cooled, filtered, and stored until transesterification (Canakci & Van Gerpen, 2001).

Alkali-Catalyzed Transesterification

In order to convert the remaining triglycerides from esterified oils from both treatments into biodiesel, alkali-catalyzed transesterification was conducted. Using a three-neck flask with a reflux condenser, thermometer, and magnetic stirrer results in removing residual catalyst, soap, glycerol, and methanol. After each wash, the mixture was subjected to settling for 30-60 minutes before draining the aqueous layer. After following each step, the final biodiesel was dried at 105 °C for 30 minutes to evaporate residual moisture, then subjected to filtering with Whatman No. 1 filter paper and stored in amber bottles. Pretreated oil (300 g) was heated to 60 °C (Zulqarnain et al., 2021). By using methanol calculated for a 6:1 molar ratio with oil

(approximately 54ml for 300g oil) and 1.0% NaOH catalyst by weight (3g), with NaOH dissolved in methanol, a methanol-catalyst solution was prepared. After the oil reached 60 °C, the methanol-NaOH solution was added, and then it was maintained at 60 °C with stirring at 400-600 rpm for 60 minutes (Osman et al., 2024). After that, the mixture was separated into a separatory funnel, and settling was done for 2-4 hours. After forming the two phases (the upper phase, which is the crude biodiesel phase, and the lower phase, which is the glycerol phase), the glycerol phase was drained. After that, the crude biodiesel is subjected to washing three to four times with warm distilled water with a volume of 100ml per wash at a temperature of 40-50 °C (Kumawat & Rokhum, 2022).

Fuel Property Testing

The final stage is fuel property testing. In order to measure critical quality parameters of biodiesel, it was measured following standard methods. In order to measure Kinematic viscosity, the sample was measured at 40 °C using Cannon-Fenske viscometer tubes in a temperature bath which had a constant temperature at 40.0 ± 0.1°C following ASTM D445. Viscometer tubes, which were clean and dry, were charged with filtered biodiesel samples, equilibrated in the bath for 15 minutes, and the flow time was measured as samples passed between timing marks (Samuel et al., 2024). For the calculation of Kinematic viscosity, the equation $\nu = C \times t$ was used, where ν = kinematic viscosity (mm^2/s), C = viscometer constant, and t = flow time (s). The readings were performed in triplicate to enhance accuracy (Maroa & Inambao, 2021). With a calibrated pycnometer following ASTM D1298 at 15 °C, the density was measured. A clean, dry pycnometer was weighed (W1), filled with biodiesel, placed in a 15 °C water bath for 30 minutes, wiped, and weighed (W2). With $\rho = (W2 - W1) / V$, where V = pycnometer volume, density was calculated. In order to determine the soap content, a modified AOCS method was followed, where 10 g of biodiesel was dissolved in 90ml of warm isopropanol and titrated with 0.01 M HCl using bromophenol blue indicator. Soap content was expressed as ppm sodium soap (Jan et al., 2023).

Results and Discussion

The extracted waste coconut oil showed characteristics of degraded coconut oils with increased FFA content. FFA (expressed as lauric acid equivalent) content of 23.8% exhibited in the oil, corresponding to an acid value of 118.9 mg KOH/g oil, validating that direct alkali-catalyzed

transesterification would not be practical without pre-treatment because conventional practice dictates streams which were exposed to moisture and microorganisms, elevated temperatures while processing that increased hydrolysis, availability of natural lipase enzymes that catalyze triglyceride hydrolysis, and shortage of antioxidants. Physical properties were a yellowish-brown semi-solid nature at room temperature (24-26 °C), kinematic viscosity of 28.7 mm^2/s at 40 °C, density of 0.918 g/cm^3 at 15°C, moisture content of 0.42%, saponification value of 252 mg KOH/g, and iodine value of 9.2 g I_2/100g. The highly saturated nature of coconut oil, predominantly composed of medium-chain saturated fatty acids (lauric acid 47-52%, myristic acid 18-21%, palmitic acid 8-10%), was confirmed by the low iodine value, suggesting favorable oxidative stability for derived biodiesel. 2.8 kg of recoverable waste coconut oil was obtained from approximately 20 liters of oil-wastewater mixture, representing approximately 14% oil content, which aligns with reported oil losses during coconut milk manufacturing. With primary gravitational separation recovering 1.9 kg (68%) and secondary centrifugal separation recovering 0.9 kg (32%), the two-stage separation process proved that it achieved an overall efficiency of recovery exceeding 95%. Feedstock FFA levels should not exceed 0.5–1.0% for a successful base-catalyzed production of biodiesel. The higher FFA levels resulted from prolonged storage in wastewater.

Clear differences between the two treatments demonstrated the substantial effectiveness of acid-catalyzed esterification in reducing FFA content. When it comes to sample A (5% sulfuric acid treatment), reduced FFA from an initial 23.8% to a final 1.8%, achieving 92.4% FFA reduction with a final acid value of 9.0 mg KOH/g. Sample B (1% sulfuric acid treatment) reduced FFA from 23.8% to 3.6%, achieving 84.9% FFA reduction with a final acid value of 18.0 mg KOH/g. These results indicate that higher sulfuric acid concentration achieved greater FFA conversion. When it comes to 5% treatment, it has reduced FFA content to 1.8%, which is in the acceptable range (2-3%) for the alkali-catalyzed transesterification. When it comes to 1% treatment, it leaves a final content of 3.6%, which represents a borderline level that results in the formation of soap. When the FFA content was monitored at different time intervals, it revealed important kinetic information. When it comes to 5% H_2SO_4 treatment: 0 minutes = 23.8% FFA, 30 minutes = 12.4% FFA, 60 minutes = 3.2% FFA, 90 minutes = 1.8%, while for 1% H_2SO_4 treatment: 0 minutes = 23.8% FFA, 30 minutes = 16.1% FFA, 60 minutes = 5.8% FFA, 90 minutes = 3.6% FFA. By referring to these data, it can be determined that FFA reduction follows typical pseudo-first-order kinetics with respect to FFA concentration when

there is excessive availability of methanol. As evidenced by the steeper initial decline for sample A, the increased catalyst concentration gradually increases the reaction rate. From the superior performance of sulfuric acid at 5% treatment, it can be determined that sulfuric acid serves dual roles: making carbonyl carbon more electrophilic and susceptible to nucleophilic attack by methanol by protonating the carbonyl oxygen of FFA, and catalyzing the dehydration step that drives the reaction forward by forming water as a leaving group. Accelerating both forward esterification and water removal from intermediate species was provided by higher acid concentrations. However, there are some practical limitations when it comes to increasing the acid concentration. Increased acid concentrations can enhance undesirable side reactions, which include charring, polymerization, and increased equipment corrosion. The FFA reduction performance for both treatments is summarized in Table 2.

Table 2: FFA Reduction Performance

FFA Reduction Performance	Sample A	Sample B
Initial FFA	23.8% (as lauric acid)	23.8% (as lauric acid)
Final FFA after esterification	1.8%	3.6%
FFA reduction	92.4%	84.9%
Final acid value	9.0 mg KOH/g	18.0 mg KOH/g

Alkali-catalyzed transesterification success was directly influenced by the effectiveness of acid pre-treatment. A smooth reaction with minimal foaming and clear phase separation was shown by Sample A. After settling time for 2 hours, biodiesel yielded at 88.3%, with a clear brown glycerol layer relatively free of emulsion, and also with minimal visible soap formation, with a slight cloudiness at the interface. In the case of sample B, it showed a moderate foaming during the reaction. The phase separation required 4-5 hours with a biodiesel yield of 82.1%, a darker, more viscous glycerol layer with emulsified material, and more soap formation with a thick emulsion layer at the interface. Direct correlation can be seen in the improved

transesterification performance of sample A, with its lower residual FFA content after acid pre-treatment, with only 1.8% FFA remaining, and soap formation during base-catalyzed transesterification was minimal. In the case of sample B, it resulted in higher residual FFA content 3.6% in more extensive soap formation that consumed approximately 3.6% of the catalyst by weight, reducing its availability for desired transesterification.

When it comes to kinematic viscosity, it directly affects the characteristics of the fuel injection, quality of atomization, and the efficiency of the combustion, which are the crucial parameters of biodiesel. When it comes to kinematic viscosity ranges, ASTM D6751 and EN14214 specify a range of 1.9-6.0 mm^2/s and 3.5-5.0 mm^2/s, respectively. In the case of sample A, which has 5% H_2SO_4 pre-treatment, it measured an average viscosity of 5.723 mm^2/s (measurements: 5.698, 5.731, 5.741 mm^2/s; standard deviation 0.022; coefficient of variation 0.38%). And the sample B, which has 1% H_2SO_4 pre-treatment, measured average viscosity of 6.386 mm^2/s (measurements: 6.362, 6.395, 6.400 mm^2/s; standard deviation 0.020; coefficient of variation 0.31%). Statistical analysis revealed a difference in means of 0.663 mm^2/s, t-statistic of 42.8, and p-value <0.001, indicating the difference is highly statistically significant. The samples A and B both fall within the ASTM D6751 acceptable range (1.9-6.0 mm^2/s), while sample B is in the upper limit. However, when it comes to the EN 14214 specification (3.5-5.0 mm^2/s), sample B exceeded the upper limit, while sample A also marginally exceeded but was much closer to compliance. When it comes to viscosity differences of samples, a value of 0.663 mm^2/s (approximately 11.6% higher in sample B) which is operationally significant and can be results in higher residual FFA content, incomplete esterification which leads to leave more un-esterified polar compound, incomplete transesterification leaving higher levels of mono-, di-, and triglycerides (which have considerable higher viscosity than FAME), and samples with more problematic phase separation retaining slightly more moisture. Increased fuel system pressure requirements, poorer atomization leading to larger droplet sizes, incomplete combustion resulting in higher particulate emissions, increased injector coking and deposit formation, and reduced engine power output and efficiency can be seen due to biodiesel with viscosity close to or exceeding 6.0 mm^2/s. By looking at these results, it can be determined that they have strong support for using a higher sulfuric acid concentration (5%) during the process of pre-treatment in order to achieve better biodiesel quality with optimized viscosity characteristics.

Table 3: Kinematic Viscosity Results

Sample Name	Measurement 1 (mm^2/s)	Measurement 2(mm^2/s)	Measurement 3(mm^2/s)	Average viscosity	Standard deviation	Coefficient of variation
Sample A	5.698	5.731	5.741	5.723	0.022	0.38%
Sample B	6.362	6.395	6.400	6.386	0.020	0.31%

Density plays a crucial role as an important biodiesel, which directly affects fuel injection timing, energy content, and characteristics of the combustion. When it comes to EN14214 standard, the density value must be required to be in the range of 860-900 kg/m^3 at 15°C. Sample A has a density of 878.3 kg/m^3, and sample B has a density of 881.7 kg/m^3. Both samples are within the EN 14214 specification. As both the samples met the density requirements according to the EN 14214 standard, Sample B shows a density slightly higher than that of Sample A. The difference is 3.4 kg/m^3. Sample B is consistent with a higher content of residual polar compounds (FFAs, soap traces, glycerides), which have a relatively higher density than pure FAME. Due to the predominance of medium-chain saturated fatty acids, the density values are typical for coconut oil biodiesel, which tends to fall in the lower-middle range of specification.

Soap can be taken as a crucial purity parameter that significantly affects the quality and the usability of the final biodiesel. Sample A and sample B had soap contents of 124 ppm and 386 ppm (as sodium soap), respectively. Both the samples fell below the specification limit of 500 ppm, while sample B approached a concern level. When the two values are compared (124 ppm vs. 386 ppm) sample B represents a threefold higher soap content, which the difference of sample A and B represents a qualitatively significant difference with practical implications. Primarily, the soap is produced from a reaction between FFAs and alkaline catalyst during transesterification (FFA + NaOH → Sodium soap + H_2O), if the conditions are excessively alkaline or water is present, with additional soap which potentially forms from saponification of triglycerides. After acid pre-treatment, the higher soap content in sample B directly reflects

its higher residual FFA content (3.6%-1.8%). These FFAs reacted to form approximately twice as much soap during subsequent transesterification with NaOH catalyst. Filter plugging (at lower temperatures, soap can crystallize, forming deposits that clog fuel filters), injector deposits (combustion of soap will results it deposits of ash which accumulates on injector nozzles), storage instability (soap is hygroscopic and also can absorb water which results in phase separation and microbial growth), corrosion (corrosion of fuel system components can occur due to sodium soap particularly with moisture present), and increased emission (increased particulate emissions and potential catalyst poisoning with exhaust after-treatment systems due to sodium soap). When the measured properties of biodiesel compared with international standards are revealed, in the case of kinematic viscosity, sample A and sample B both meet ASTM D6751 (1.9-6.0 mm^2/s). The sample B was noticed near the upper limit, but neither fully met EN 14214 (3.5-5.0 mm^2/s), though sample A was much closer to compliance. When it comes to density, sample A and sample B both meet EN 14214 (860-900 kg/m^3) with allowable margins. In the case of soap content, sample A and sample B both contained acceptable levels (<500 ppm), with sample A showing a superior purity. When the partial non-compliance with EN 14214 viscosity specifications proposes further optimization is required for European market applications. However, biodiesel from 5% acid pre-treatment would be acceptable for markets governed by ASTM D6751 (including the United States), pending verification of additional parameters. A comprehensive comparison of measured biodiesel properties against international standards is presented in Table 4, which forms the primary basis for interpreting the results.

Table 4: Comparison of Measured Biodiesel Properties with International Standards

Property	ASTM D6751	EN 14214	Sample A (5% H_2SO_4)	Sample B (1% H_2SO_4)
Kinematic Viscosity (40°C, mm^2/s)	1.9-6.0	3.5-5.0	5.723	6.386
Density (15°C,	Not specified	860-900	878.3	881.7

kg/m³)				
Soap Content (ppm)	<500 typical	<500 typical	124	386

which reduces viscosity and weakens emulsion stability, salt addition (addition of 5-10g of NaCl per liter assists breaking emulsions by increasing ionic strength), increasing settling time (allowing 6-8 hours instead of 2-4 hours), attentive decanting using a separatory funnel, and brief centrifugation (2000-3000 rpm for about 10 minutes). Soap formation can be taken as a significant challenge throughout biodiesel production, even with optimized pre-treatment. Optimal pre-treatment reducing FFA to 2% following effective acid esterification (most crucial), precise catalyst measurement to prevent excess alkalinity, anhydrous conditions which ensure dry oil and dry methanol to prevent base-catalyzed hydrolysis, careful addition of catalyst-methanol mixture to prevent localized alkalinity, optimal temperature (60°C) which prevents excessive saponification which leads to ensure good reaction rates, through washing multiple times, and acidified washing with final wash using dilute citric acid solution (0.1%) to neutralize residual alkali and dissolve soap can be taken as effective strategies for soap formation reduction.

When it comes to transforming waste coconut oil from the non-usable waste coconut oil from wastewater streams into usable biodiesel provides multiple environmental advantages which includes: waste reduction (minimizes approximately 14% of wastewater amount from disposing, eliminates oil-contaminated wastewater which requires treatment, reduction of biochemical oxygen demand and chemical oxygen demand in released wastewater), emission reductions (biodiesel which produced from coconut oil minimizes CO_2 emissions by approximately 50-70% when compared with the lifecycle of petroleum diesel, reduced particulate matter emissions, minimized sulfur dioxide emissions as the product is essentially sulfur-free),resource conservation (biodiesel is a good replacement with fossil fuel with renewable energy from wastewater, no additional land use which needed unlike purpose-grown biofuel crops, minimized competition with food production), and especially circular economy benefits (converts waste into value-added product, closes resource loops in coconut processing industry, creates additional value from existing operations). When it comes to economic analysis, it reveals promising viability. When it comes to production costs, it estimates per liter

of biodiesel includes: free waste coconut oil (disposal cost avoided), methanol (0.24L and $0.40/L) $0.096, sulfuric acid (5% concentration) $0.020, NaOH $0.030, others (water/electricity) $0.050, which comes as a total of $0.20 per liter in variable costs. When compared with petroleum diesel, it typically costs $0.80-1.20 per liter, but the commercial biodiesel costs $0.90-1.40 per liter. From small to medium scale (100-1000 L/day), biodiesel production from waste coconut oil demonstrates promising economics with the calculated production cost of $0.20/L, market price of $0.90-1.10/L, gross margin of $0.70-0.90/L, and potential net margin of $0.30-0.50/L after accounting for labor, equipment depreciation, and overhead. For a production plant that can process 500/L per day: the annual production cost of 182,500L, annual revenue ($1.00/L) of $182,500, variable costs annually of $36,500, leads to a potential gross profit of $146,000. When it comes to scale increasing and fixed costs are amortized over larger volumes, these economics become increasingly favorable.

Conclusion

This study established the technical and economic viability of transforming waste coconut oil in the coconut milk processing wastewater into high-quality biodiesel by optimal acid-catalyzed esterification pre-treatment. The study fills important knowledge gaps in biodiesel production using high free fatty acid (FFA) feeds and develops principles of the circular economy in the coconut processing sector. The two-step gravitational-centrifugal separation process was effective in recovering 2.8 kg of waste coconut oil using 20 liters of wastewater, which indicates 14% of oil content and a recovery rate exceeding 95%. Characterization showed high levels of degradation of feedstock with 23.8% FFA content and acid value of 118.9 mg KOH/g, which supports the fact that pre-treatment must be done prior to conventional alkali-catalyzed transesterification. The high FFA was attributed to prolonged exposure to moisture, microorganisms, and enzymatic hydrolysis when stored in the wastewater, which was the case with the industrial waste oil streams. Relative comparison of the content of sulfuric acid (1%: 5% by weight) showed a significant discrepancy in FFA reduction rates and the quality of biodiesel obtained. The H_2SO_4 (5% solution) treatment allowed a reduction in FFA (23.8 to 1.8) to 92.4% in 90 minutes with a 15:1 methanol-FFA molar ratio to effectively neutralize the acid value to 9.0 mg KOH/g. Contrastingly, 1% treatment had an 84.9% decrease, leaving residual FFA of 3.6% that had an acid value of 18.0 mg KOH/g. The kinetic analysis showed both a pseudo-first-order FFA reduction behavior at greater catalyst concentrations, exhibiting steeper initial decline rates, and that sulfuric acid has two catalytic

roles: carbonyl activation and dehydration. The performance of alkali-catalyzed transesterification was directly affected by the activity of acid pre-treatment. Sample A (5% pre-treatment) showed minimal foaming and clear phase separation within 2 hours, with no or low soap formation. Sample B (1% pre-treatment) was moderate in foaming,

The overall fuel property testing showed that there were notable quality variations among treatments. The 5% acid pre-treatment biodiesel had a kinematic viscosity of 5.723mm^2/s at 40 0 C, which is within the ASTM D6751 range (1.9-6.0 mm^2/s) but slightly higher than the EN 14214 range (3.5-5.0 mm^2/s). Biodiesel after 1% treatment had a viscosity of 6.386 mm^2/s, which is ASTM but a significantly higher level than EN 14214. The difference in the viscosity of 0.663 mm^2/s (11.6%) (statistically significant 0.001) is because of the incomplete esterification and the preservation of un-trans esterified glycerides. Analysis of the soap content showed a triple difference between treatments (124 ppm and 386 ppm), both of which are below specification limits (less than 500 ppm), but with Sample B nearly reaching the alarming levels capable of leading to filter plugging, injector deposits, and storage instability. Environmental analysis reveals that waste-to-biodiesel conversion has significant advantages associated with it such as an eradication of 14% wastewater oil content that has to be disposed of at a high cost, a decrease in biochemical oxygen demand and chemical oxygen demand in discharged wastewater, a lifecycle CO_2 emission reduction of 50-70% relative to petroleum diesel and conservation of resources that do not necessitate an increase in land use or competition with food supply. The process is an example of the concepts of a circular economy by turning problematic waste into value-added renewable fuel. Economic analysis demonstrates that the company is economically viable with a projected variable cost of production of $0.20/L and the estimated price of commercial bio-diesel at $0.90-1.10/L, which is comparatively cheaper with potential gross margins of $0.70-0.90/L. In the case of 500 L/day operations, which result in 182,500 L per year, the estimated gross profit is $146,000, and the economies of scale at higher levels show amortization of fixed costs, allowing substantial increases in gross profits. especially in tropical regions where coconut processing is significant. Future work should explore continuous flow reactor designs to improve scalability, investigate alternative acid catalysts, optimize the methanol-to-oil molar ratio, and evaluate additional EN 14214 and ASTM D6751 quality parameters, including flash point, cetane number, and cold filter plugging point. and show the concepts of sustainable development in which the waste management process overlaps with renewable energy generation.

Data Accessibility Statement

The data generated and analyzed during this study are available from the corresponding author upon reasonable request. Raw experimental data including FFA titration measurements, viscosity readings, density measurements, and soap content determinations are retained by the authors and will be provided on reasonable request.

Ethics and Consent Statement

This study did not involve human participants, animal subjects, or sensitive personal data. Ethical approval was therefore not required. All experimental work was conducted using industrial wastewater and chemical reagents in a laboratory setting at Sabaragamuwa University of Sri Lanka, in compliance with institutional safety and environmental guidelines.

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Competing Interests Statement

The authors declare that there are no competing interests. No financial or personal relationships have influenced the work reported in this paper.

Authors' Contributions

Hasith Perera and Shen Hosan conceptualized the study and designed the experimental framework. Hasith Perera., Samith Randika., and Dhanuka Jayawardhana conducted the laboratory experiments. Dasith Wijesekara and Shakya Abeysinghe contributed to analytical measurements and data collection. Vimukthi Vithanage and Chanaka Galpayage performed data analysis and statistical evaluation. Hasith Perera drafted the manuscript. Prasad Amarasinghe and Kaveenga Koswattage reviewed, edited

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