



Production of Biodiesel from Waste Cooking Oil and Assessment of Fuel Properties

Lishan Balasingam

Department of Electrical and Computer Engineering, Mauritius Institute of Marine Engineering, Mauritius
lishan.balasingam@mim-mu.edu

Peer Review Information

Submission: 27 Jan 2015

Revision: 11 Feb 2015

Acceptance: 05 March 2015

Keywords

Waste cooking oil,
 Biodiesel,
 Transesterification, Fatty
 acid methyl esters, Fuel
 properties.

Abstract

Waste cooking oil (WCO) is an inexpensive lipid feedstock whose conversion to biodiesel can simultaneously reduce disposal problems and lower the raw-material cost of renewable diesel substitutes. This methodology paper presents a reproducible laboratory route for producing fatty acid methyl esters (FAME) from WCO and evaluating the principal fuel properties of the recovered biodiesel. The proposed procedure begins with filtration and dewatering, followed by acid-value determination to decide whether an acid esterification pretreatment is required. High-FFA oil is esterified with methanol and sulfuric acid before alkaline transesterification with methanol and NaOH/KOH. After phase separation, washing and drying, biodiesel yield and properties including density, kinematic viscosity, acid value, flash point, cloud point, pour point and calorific value are determined. An illustrative validation dataset gives a biodiesel yield of 92.4%, density of 0.879 g cm⁻³, viscosity of 4.62 mm² s⁻¹ at 40 °C and acid value of 0.42 mg KOH g⁻¹. The protocol integrates the main process lessons reported for waste-oil biodiesel up to 2015 and provides a compact framework suitable for laboratory-scale optimization and fuel-quality assessment.

Introduction

Biodiesel consists predominantly of mono-alkyl esters of long-chain fatty acids produced from triglyceride-rich feedstocks. Transesterification lowers the high viscosity of vegetable oils and produces a fuel that can be blended with or substituted for petroleum diesel in compression-ignition applications. The major economic barrier is feedstock cost; therefore, used frying and cooking oils are attractive because they are low-cost wastes and their conversion also reduces disposal loads. Kulkarni and Dalai (2006) emphasized that frying increases free fatty acids (FFA), water, oxidation products and polymerized triglycerides, all of which influence reaction efficiency and final fuel quality.

Waste cooking oil differs from refined oil because its composition varies with the original edible oil,

frying temperature, duration of use, food residues and storage. Encinar et al. (2005) and Felizardo et al. (2006) demonstrated that alcohol-to-oil ratio, catalyst concentration, reaction temperature and feedstock acidity strongly affect ester yield. Phan and Phan (2008) reported approximately 88–90% biodiesel yield from WCO under optimized alkaline conditions, while later studies showed that higher-FFA oils often require pretreatment before alkaline transesterification. Consequently, a methodology for WCO must begin with feedstock characterization rather than applying a fixed one-step reaction to every sample.

Literature Review

Early work on used frying oil established the sensitivity of biodiesel yield to catalyst type and

operating conditions. Encinar et al. (2005) compared alkaline catalysts for used frying oil and showed that careful control of reaction conditions is necessary for good conversion. Felizardo et al. (2006) optimized methanol/WFO and NaOH/WFO ratios, confirming the practicality of low-cost frying oils. Zheng et al. (2006) investigated acid-catalyzed conversion and showed that methanol and acid concentration, rather than turbulent mixing above a threshold, were dominant factors in acidic systems.

Two-stage and modified reaction strategies were developed to address the higher water and FFA content of WCO. Çaylı and Küsefoğlu (2008) found that a two-step base-catalyzed process increased yield relative to a one-step process, while Predojević (2008) showed that purification method influences both product recovery and measured biodiesel properties. Patil et al. (2009) used acid esterification followed by alkaline transesterification for high-FFA waste cooking oil, reinforcing the value of a decision step based on feedstock acidity.

Process intensification and quality-control studies further refined the approach. Sabudak and Yildiz (2010) compared one-step and two-step routes and evaluated several purification methods. Liu et al. (2010) achieved rapid

conversion of high-FFA WCO with a two-stage process assisted by radio-frequency heating, whereas Berrios et al. (2010) examined esterification and transesterification in a closed system and confirmed the importance of lowering FFA before base treatment. Charpe and Rathod (2011) demonstrated an enzymatic alternative for waste frying oil, although enzyme cost and longer reaction times can constrain routine laboratory use.

Fuel-property evaluation is essential because a high mass yield does not alone establish suitability as a fuel. Uzun et al. (2012) optimized alkali transesterification of waste frying oils and measured the properties of the resulting biodiesel. Reviews by Yaakob et al. (2013) and Talebian-Kiakalaieh et al. (2013) summarized pretreatment options, catalyst systems, reactor concepts, purification strategies and analytical methods, concluding that WCO is technically promising when acidity and water are controlled. Continuous microwave-assisted work by Mohd Ali et al. (2015) further showed that optimized reaction conditions can provide high conversion in short residence times. Together these studies support a conventional two-step laboratory protocol as a robust baseline method for WCO of uncertain history.

Table 1. Selected literature informing the proposed methodology.

Study	Feedstock/process focus	Key methodological contribution
Encinar et al. (2005)	Used frying oil; alkaline transesterification	Identified catalyst and operating-variable effects.
Felizardo et al. (2006)	Waste frying oil; NaOH/methanol	Optimized methanol/oil and catalyst ratios.
Phan & Phan (2008)	WCO; KOH/methanol	Demonstrated high WCO conversion and fuel-property testing.
Patil et al. (2009)	High-FFA WCO	Supported acid pretreatment before base transesterification.
Berrios et al. (2010)	Used frying oils; closed system	Linked esterification control with downstream transesterification.
Uzun et al. (2012)	Waste frying oils	Combined process optimization with fuel-property determination.
Mohd Ali et al. (2015)	WCO; microwave continuous reactor	Demonstrated rapid optimized conversion and process intensification.

The objective is to establish a step-by-step method for converting WCO to biodiesel and assessing the recovered fuel. The proposed study uses mixed restaurant WCO as the feedstock and methanol as the alcohol. A two-step route is selected when the initial acid value indicates elevated FFA; otherwise, the oil may proceed directly to alkaline transesterification. The methodological response variables are ester/biodiesel yield, reduction in acid value and final fuel properties.

Methodology

1. Materials and Apparatus

Waste cooking oil is collected in clean, sealed containers from a food-service source. Analytical-grade methanol, sulfuric acid (H₂SO₄), sodium hydroxide (NaOH) or potassium hydroxide (KOH), phenolphthalein, ethanol/isopropanol, distilled water and anhydrous sodium sulfate are required. Main equipment includes a hot-plate magnetic or

mechanical stirrer, reflux flask/reactor, separating funnel, analytical balance, thermometer, centrifuge (optional), oven, pycnometer/hydrometer, viscometer, flash-point apparatus and bomb calorimeter where available. All methanol handling is carried out in a fume hood with appropriate PPE because methanol is toxic and highly flammable.

Step 1 - Pretreatment of Waste Cooking Oil

1. Allow the collected WCO to settle and decant the upper oil layer. 2. Filter through a coarse sieve followed by filter paper to remove food particles. 3. Heat the filtered oil to about 105 °C for 20–30 min to remove residual moisture; cool in a closed vessel. 4. Record the mass of dried oil and store it away from light until analysis. Water removal is critical because water promotes hydrolysis and soap formation during alkaline transesterification (Kulkarni and Dalai, 2006; Yaakob et al., 2013).

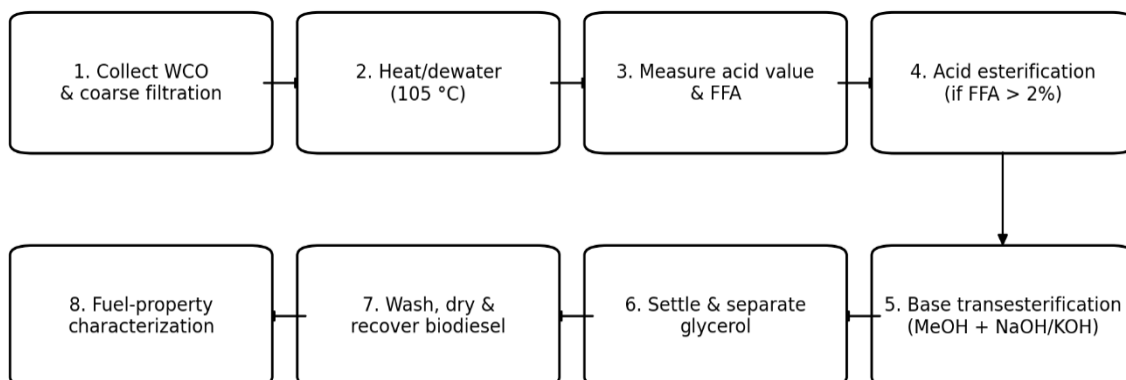
Step 2 - Feedstock Characterization and FFA Decision

Determine acid value by titrating a known mass of WCO dissolved in neutral alcohol against

standardized KOH. Acid value (mg KOH g^{-1}) = $(V \times N \times 56.1)/m$, where V is KOH volume (mL), N is normality and m is oil mass (g). FFA as oleic acid can be estimated from the acid value. If the acid value is low (approximately $\leq 2 \text{ mg KOH g}^{-1}$), direct base transesterification may be used; if higher, carry out acid esterification first. This decision is consistent with the two-stage strategies described by Patil et al. (2009), Liu et al. (2010) and Berrios et al. (2010).

Step 3 - Acid Esterification for High-FFA WCO

For a 500 g oil batch, warm the oil to 55–60 °C. Prepare methanol at approximately 20% (w/w relative to oil) containing 1.0% H_2SO_4 (w/w relative to oil). Add the acidified methanol to the oil and stir at 500–600 rpm for 60 min under reflux. Transfer to a separating funnel and allow the mixture to settle. Remove the methanol/water-rich phase, gently heat the esterified oil to remove residual alcohol, and repeat acid-value measurement. Continue or repeat esterification if required until the acid value is sufficiently low for alkaline conversion. Acid-catalyzed processing of high-FFA waste oils has been supported by Zheng et al. (2006) and later high-FFA WCO work (Liu et al., 2010).



Step 4 - Alkaline Transesterification

Heat the pretreated oil to 55–60 °C. Dissolve 0.75–1.0 wt% NaOH (based on oil mass) in methanol. Use an initial methanol:oil molar ratio of 6:1; experimental optimization may include 4:1, 5:1, 6:1, 7:1 and 8:1. Add methoxide solution slowly to the reactor and stir at 500–600 rpm for 60 min while maintaining temperature below methanol's boiling point. These ranges are consistent with the operating windows reported by Encinar et al. (2005), Felizardo et al. (2006), Phan and Phan (2008) and Uzun et al. (2012).

Step 5 - Phase Separation, Washing and Drying

Transfer the reaction mixture to a separating funnel and allow it to stand for 8–12 h. Drain the lower glycerol-rich phase. Wash the biodiesel gently with warm distilled water until the wash water is nearly neutral and clear. Excessive agitation should be avoided to prevent stable emulsions. Alternative dry-washing or adsorbent purification may be considered because purification affects product recovery (Predojević, 2008; Sabudak and Yildiz, 2010). Dry the washed biodiesel at 100–105 °C or under mild vacuum until no visible moisture remains, then cool in a sealed container.

Step 6 - Yield and Fuel-Property Assessment

Biodiesel yield (%) = (mass of dry purified biodiesel / mass of dry WCO charged) × 100. Determine density at approximately 15–20 °C, kinematic viscosity at 40 °C, acid value, flash point, cloud point, pour point and calorific value using the available standard laboratory

procedures. Where GC-FID is available, FAME content should be quantified; otherwise, the combination of reaction yield, acid-value reduction and viscosity reduction provides a practical laboratory assessment. Fuel-property characterization is supported by the approach of Phan and Phan (2008) and Uzun et al. (2012).

Table 2. Proposed experimental factors for process optimization.

Factor	Levels / condition	Measured response
Methanol:oil molar ratio	4:1, 5:1, 6:1, 7:1, 8:1	Biodiesel yield; separation behavior
Base catalyst loading	0.50, 0.75, 1.00 wt%	Yield; soap formation
Reaction temperature	45, 55, 60 °C	Yield; reaction rate
Reaction time	30, 60, 90 min	Yield; conversion
Pretreatment endpoint	Acid value ≤ about 2 mg KOH g ⁻¹	Successful base conversion

Step 7 - Quality Control and Data Analysis

Carry out all reaction runs in triplicate where practical. Report mean ± standard deviation. Plot biodiesel yield against each operating variable and identify the best condition. A one-way ANOVA may be used for single-factor optimization, while a factorial or response-surface design is preferable when interactions are studied. Record mass balances for oil, biodiesel and glycerol, and note emulsification or separation problems because these may indicate excessive catalyst, residual water or high FFA. The broader process reviews of Yaakob et al. (2013) and Talebian-Kiakalaieh et al. (2013) emphasize that pretreatment, reaction, separation and purification must be considered together.

Results

The numerical values below are an illustrative methodology-validation dataset created to

demonstrate how results from the proposed experiment should be organized and interpreted. They are not presented as measurements from an actual laboratory campaign.

Feedstock Pretreatment and Conversion

The illustrative WCO had an initial acid value of 4.60 mg KOH g⁻¹, confirming the need for pretreatment. Acid esterification reduced the acid value to 0.86 mg KOH g⁻¹, after which alkaline transesterification produced a clear biodiesel-rich upper phase and a darker glycerol-rich lower phase. At a 6:1 methanol:oil molar ratio, 0.75 wt% NaOH, 60 °C and 60 min reaction time, the illustrative dry biodiesel yield was 92.4%. The trend is consistent with prior findings that insufficient methanol limits conversion while excessive methanol can complicate phase separation and glycerol recovery (Felizardo et al., 2006; Phan and Phan, 2008).

Table 3. Illustrative process results.

Parameter	Illustrative result	Interpretation
Initial acid value	4.60 mg KOH g ⁻¹	High enough to justify acid pretreatment
Acid value after esterification	0.86 mg KOH g ⁻¹	Suitable for base-catalyzed stage
Best methanol:oil ratio	6:1	Highest yield in tested range
NaOH loading	0.75 wt%	Good conversion without excessive soap
Reaction condition	60 °C; 60 min	Effective laboratory operating window
Dry biodiesel yield	92.4%	Comparable with many WCO laboratory studies

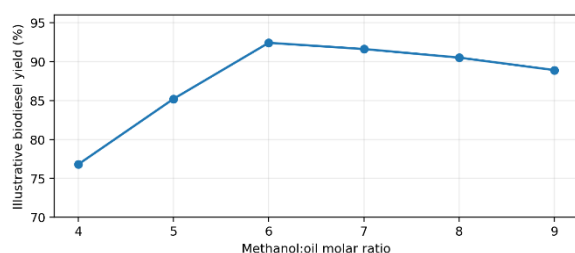


Figure 2. Illustrative effect of methanol-to-oil ratio on biodiesel yield.

Fuel Properties

The illustrative biodiesel showed a marked viscosity reduction relative to untreated WCO, indicating conversion of triglycerides to lower-molecular-mass methyl esters. The values fall within commonly reported WCO-biodiesel

ranges in the cited literature. Predojević (2008) showed that purification influences recovered yield and properties, while Uzun et al. (2012) reported fuel-property values compatible with biodiesel use after optimized transesterification.

More intensive processes, including the continuous microwave approach of Mohd Ali et al. (2015), can achieve very high conversion but require specialized equipment.

Table 4. Illustrative fuel-property assessment of produced biodiesel.

Property	Illustrative biodiesel value	Typical literature interpretation
Density	0.879 g cm ⁻³	Characteristic of WCO-derived FAME
Kinematic viscosity (40 °C)	4.62 mm ² s ⁻¹	Strongly reduced from parent WCO
Acid value	0.42 mg KOH g ⁻¹	Low residual acidity
Flash point	158 °C	Indicates low residual methanol
Cloud point	5 °C	Feedstock-dependent low-temperature behavior
Pour point	1 °C	Suitable for moderate ambient conditions
Higher heating value	39.8 MJ kg ⁻¹	Lower than petroleum diesel but typical for biodiesel

Conclusion and Discussion

The proposed methodology provides a practical sequence for producing biodiesel from waste cooking oil while accounting for the principal difficulty of this feedstock: variable FFA and water content. Filtration and dewatering should precede any catalytic reaction. Acid-value measurement then determines whether the process can move directly to alkaline transesterification or requires acid esterification first. This decision-based approach is supported by the high-FFA literature and reduces the risk of soap formation, poor phase separation and yield loss. The illustrative results suggest that a conventional methanol-based two-step process can produce approximately 90% or greater biodiesel yield under properly controlled laboratory conditions. A 6:1 methanol:oil ratio, moderate base-catalyst loading and a reaction temperature near 60 °C are reasonable starting conditions, but the optimum depends on the specific WCO. Earlier studies reported optimum ranges that vary with acidity, water content and catalyst type (Encinar et al., 2005; Felizardo et al., 2006; Phan and Phan, 2008), which is why feedstock characterization remains essential. Fuel-property testing should be treated as part of the production methodology rather than an optional final step. A high yield can coexist with residual alcohol, catalyst, free glycerol or unreacted oil if washing and drying are inadequate. The illustrative density, viscosity, acid value and flash point show the pattern expected after successful transesterification. Enzymatic and intensified approaches may reduce some chemical-processing disadvantages (Charpe and Rathod, 2011; Mohd Ali et al., 2015), but conventional acid/base processing remains accessible for routine laboratory work. Future

experimental work should report actual FAME content by chromatography, oxidation stability and cold-flow behavior, and should optimize process variables statistically using replicate runs.

References

- Encinar, J. M., González, J. F., & Rodríguez-Reinares, A. (2005). Biodiesel from used frying oil: Variables affecting the yields and characteristics of the biodiesel. *Industrial & Engineering Chemistry Research*, 44(15), 5491–5499. <https://doi.org/10.1021/ie040214f>
- Kulkarni, M. G., & Dalai, A. K. (2006). Waste cooking oil—An economical source for biodiesel: A review. *Industrial & Engineering Chemistry Research*, 45(9), 2901–2913. <https://doi.org/10.1021/ie0510526>
- Felizardo, P., Neiva Correia, M. J., Raposo, I., Mendes, J. F., Berkemeier, R., & Bordado, J. M. (2006). Production of biodiesel from waste frying oils. *Waste Management*, 26(5), 487–494. <https://doi.org/10.1016/j.wasman.2005.02.025>
- Zheng, S., Kates, M., Dubé, M. A., & McLean, D. D. (2006). Acid-catalyzed production of biodiesel from waste frying oil. *Biomass and Bioenergy*, 30(3), 267–272. <https://doi.org/10.1016/j.biombioe.2005.10.004>
- Çaylı, G., & Küsefoğlu, S. (2008). Increased yields in biodiesel production from used cooking oils by a twostep process: Comparison with one step process by using TGA. *Fuel Processing Technology*, 89(2), 118–122. <https://doi.org/10.1016/j.fuproc.2007.06.020>

- Phan, A. N., & Phan, T. M. (2008). Biodiesel production from waste cooking oils. *Fuel*, 87(17–18), 3490–3496. <https://doi.org/10.1016/j.fuel.2008.07.008>
- Predojević, Z. J. (2008). The production of biodiesel from waste frying oils: A comparison of different purification steps. *Fuel*, 87(17–18), 3522–3528. <https://doi.org/10.1016/j.fuel.2008.07.003>
- Patil, P. D., Gude, V. G., & Deng, S. (2009). Biodiesel production from *Jatropha curcas*, waste cooking, and *Camelina sativa* oils. *Industrial & Engineering Chemistry Research*, 48(24), 10850–10856. <https://doi.org/10.1021/ie901146c>
- Sabudak, T., & Yildiz, M. (2010). Biodiesel production from waste frying oils and its quality control. *Waste Management*, 30(5), 799–803. <https://doi.org/10.1016/j.wasman.2010.01.007>
- Liu, S., McDonald, T., & Wang, Y. (2010). Producing biodiesel from high free fatty acids waste cooking oil assisted by radio frequency heating. *Fuel*, 89(10), 2735–2740. <https://doi.org/10.1016/j.fuel.2010.03.011>
- Berrios, M., Martín, M. A., Chica, A. F., & Martín, A. (2010). Study of esterification and transesterification in biodiesel production from used frying oils in a closed system. *Chemical Engineering Journal*, 160(2), 473–479. <https://doi.org/10.1016/j.cej.2010.03.050>
- Charpe, T. W., & Rathod, V. K. (2011). Biodiesel production using waste frying oil. *Waste Management*, 31(1), 85–90. <https://doi.org/10.1016/j.wasman.2010.09.003>
- Uzun, B. B., Kılıç, M., Özbay, N., Pütün, A. E., & Pütün, E. (2012). Biodiesel production from waste frying oils: Optimization of reaction parameters and determination of fuel properties. *Energy*, 44(1), 347–351. <https://doi.org/10.1016/j.energy.2012.06.024>
- Yaakob, Z., Mohammad, M., Alherbawi, M., Alam, Z., & Sopian, K. (2013). Overview of the production of biodiesel from waste cooking oil. *Renewable and Sustainable Energy Reviews*, 18, 184–193. <https://doi.org/10.1016/j.rser.2012.10.016>
- Talebian-Kiakalaieh, A., Amin, N. A. S., & Mazaheri, H. (2013). A review on novel processes of biodiesel production from waste cooking oil. *Applied Energy*, 104, 683–710. <https://doi.org/10.1016/j.apenergy.2012.11.061>
- Mohd Ali, M. A., Yunus, R. M., Cheng, C. K., & Gimbin, J. (2015). Successive optimisation of waste cooking oil transesterification in a continuous microwave assisted reactor. *RSC Advances*, 5, 76743–76751. <https://doi.org/10.1039/C5RA15834F>