2^4 Factorial Design Optimization of Process Variables for Biodiesel Production from Waste Cooking Oil

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ABSTRACT

The increase of global population, leads to greater energy demand, causing resource depletion, increased emissions, and environmental degradation. The production of biodiesel from Waste Cooking Oil (WCO) offers a promising solution. However, optimizing WCO biodiesel often requires costly and complex processes such as advanced photocatalysts, microwave heating, or membrane reactors. This study uses a 2^4 factorial experimental design to improve WCO biodiesel production based on ASTM specifications, resulting in a 96% biodiesel output at the following conditions: 40 °C, 30 min transesterification, 0.5% catalyst, and a 12:1 alcohol-to-oil molar ratio. The measured fuel properties—specific gravity, kinematic viscosity (at 40 °C), acid number, sulfated ash content, cloud point, flash point, distillation temperature, copper corrosion resistance, carbon residue content, iodine value, methyl ester content, monoglyceride content, free glycerol content, and total glycerol content—show that high-quality biodiesel can be produced from WCO under simple and economically viable conditions.

Keywords-biodiesel; energy; optimization; transesterification; waste cooking oil

I. INTRODUCTION

The ongoing global energy crisis, driven by high energy consumption and rapid economic growth, emphasizes the need for sustainable energy alternatives in light of the environmental and health consequences of fossil fuels [1]. The transportation sector and certain power plants, in particular, are heavily reliant on petroleum products such as gasoline and diesel [2]. More than 75% of all Greenhouse Gas (GHG) and almost 90% of all CO_2 emissions come from fossil fuels, making them the most significant cause of climate change [3]. Promoting renewable energy is a crucial strategic concern for transitioning energy systems and environmental protection. To ensure economic development and sustainability, it is important to reduce the reliance on non-renewable energy sources and increase the use of renewable energy sources [4, 5]. Biofuels constitute a solution for overcoming the energy crisis, because of the abundant availability of waste feedstocks and the variety of chemical and biological conversion technologies for production [6, 7]. Biodiesel, in particular, is receiving increasing attention due to its favorable properties and chemical characteristics [8], along with its compatibility with conventional diesel engines; little to no modifications are required for operation [9-11]. Biodiesel–diesel blends can significantly impact combustion behavior and emission characteristics in compression-ignition engines under different operating conditions [12]. Authors in [13] examined a B30 biodiesel blend and revealed a reduction in hydrocarbon (HC) and carbon monoxide (CO) emissions, indicating substantial emission benefits compared to conventional diesel operation. Authors in [14] indicated that biodiesel–diesel blends can reduce CO, HC, and smoke emissions, though trade-offs in NO_x emissions may occur depending on operating conditions and blend composition. Authors in [15] showed that the properties of biodiesel fuel can be accurately predicted from the composition of Fatty Acid Methyl Esters (FAMEs) using regression-based models, while authors in [16] found that the choice of biodiesel feedstock significantly affects GHG emissions and energy balance, with

Jatropha and Calophyllum inophyllum exhibiting the most favorable performance. Biodiesel consists of monoalkyl esters of long-chain fatty acids [17] and the most common method of production is transesterification, in which triglycerides (the primary components of vegetable oils and animal fats) react with short-chain alcohols (such as methanol or ethanol) to produce FAMEs and glycerol as byproducts [18, 19]. Biodiesel is a promising substitute for conventional diesel due to its extraction from renewable triglycerides found in vegetable oils [20-24]. A significant advantage of using WCO for biodiesel synthesis is the low cost of production and its availability [25, 26] in restaurants, cafeterias, and domestic kitchens. Current research focuses on converting WCO into biodiesel. However, the elevated Free Fatty Acid (FFA) content in WCO causes several undesirable side reactions during production. The biodiesel obtained from this process is tested for its characteristics and compared with fuel standards.

II. METHODOLOGY

WCO is collected from restaurants and households in Surabaya, Indonesia. After collection, the oil is filtered to remove food residue, and the basic properties are determined through analysis. To optimize biodiesel production from WCO, an esterification step is crucial for reducing the FFA content, preventing soap formation, and improving product quality and biodiesel yields. The esterification process was catalyzed by H_2SO_4. Transesterification was conducted using different catalyst concentrations: 0.5 wt% and 1.5 wt% NaOH. The process was examined at two different reaction temperatures: 40°C and 65°C, and two reaction times: The alcohol-to-oil molar ratio was consistently maintained at 6:1 and 12:1, and the reaction times were 30 min and 90 min. The acid number was determined using a specific procedure. First, a 5 g oil sample was weighed into a 250 mL Erlenmeyer flask. Then, 20 mL of isopropyl alcohol was added. The mixture was stirred with a magnetic stirrer at 50 °C for 30 min. After cooling, three to four drops of phenolphthalein indicator were added to the solution, which was then thoroughly mixed. Titration with potassium hydroxide (KOH) was performed until a persistent pink color appeared, and the volume of KOH consumed was recorded. The acid number was then calculated using [27]:

$$acid\ number = \frac{volume\ KOH\ x\ N\ x\ BM}{oil\ weight} \quad (1)$$

where the volume of KOH (mL) is the amount of KOH solution consumed during titration, N is the molarity of the KOH solution (0.1 N), BM is the molecular weight of KOH (56.1 g/mol), and oil weight (g) is the mass of the analyzed oil sample. Separately, measured quantities of sodium hydroxide and methanol were combined in a conical flask and allowed to dissolve. The resulting methoxide solution was then added to the preheated oil sample. The reaction mixture was subsequently placed in a water bath equipped with a shaker and maintained at the specified temperature and time with a constant stirring speed of 200 rpm to facilitate the transesterification reaction. After the specified reaction time, the mixture was transferred to a separating funnel and left to settle overnight to allow for phase separation of the biodiesel and glycerol. Then, multiple washing cycles were conducted until a clear ester layer was observed. The pH of the wash water and biodiesel was continuously monitored to ensure a neutral pH (pH 7). Finally, the biodiesel was heated to eliminate any remaining moisture. After the reaction, the unreacted oil settled to the bottom of the biodiesel layer and was drained. The purified biodiesel then underwent rotary evaporation to eliminate residual methanol. The biodiesel yield was then determined using:

$$yield\% = \frac{Amount\ of\ biodiesel\ produced}{Amount\ of\ oil} x100 \quad (2)$$

The optimization of variables affecting the synthesis of methyl esters from waste vegetable oil was performed using a 2^4 factorial design. The selected factors were reaction temperature, reaction time, catalyst concentration, and the alcohol-to-oil ratio. Two levels were studied to optimize the oil conversion process, as depicted in Table I. Figure 1 shows the overall methodology used for producing biodiesel from WCO.

TABLE I. EXPERIMENTAL AND PREDICTED RESPONSES OF DEPENDENT VARIABLE (BIODIESEL YIELD) (Y)

Temperature (^{0}C)	Time (min)	Catalyst concentration (mole)	Mole ratio	Methyl ester yield
				Experimental results
40	30	0.5	6:1	90
65	30	0.5	6:1	85
40	90	0.5	6:1	92
65	90	0.5	6:1	87
40	30	1.5	6:1	90
65	30	1.5	6:1	85
40	90	1.5	6:1	92
65	90	1.5	6:1	87
40	30	0.5	12:1	96
65	30	0.5	12:1	95
40	90	0.5	12:1	94
65	90	0.5	12:1	95
40	30	1.5	12:1	93
65	30	1.5	12:1	93
40	90	1.5	12:1	92
65	90	1.5	12:1	90

To ensure product quality, the raw biodiesel is purified to remove residual alcohol, catalyst, glycerol, soap, and other impurities. The purification process primarily consists of washing and drying the methyl esters. After purification, the main characteristics of the biodiesel are established through comprehensive characterization according to the ASTM standard for biodiesel.

III. RESULTS AND DISCUSSION

Due to its high FFA content, pretreatment of used cooking oil is significant before biodiesel processing. Additionally, the oil's other properties, including viscosity and water content, were evaluated and found to exceed the prescribed standard limits.

A. Equation Optimization of Process Variables

A 2^4 full factorial experimental design was used to quantify the effects of reaction temperature, transesterification time, catalyst loading, and the methanol-to-oil molar ratio on the yield of biodiesel. The optimum biodiesel yield of 96% was achieved with the following process variable levels: 40°C reaction temperature, 30-minute reaction time, 0.5 wt% NaOH,

and a 12:1 methanol-to-oil molar ratio. The methanol-to-oil molar ratio strongly influenced the ester yield. Increasing the ratio from 6:1 to 12:1 consistently increased the biodiesel yield to approximately 95–96%. This reflects the equilibrium-controlled nature of the transesterification reaction and the beneficial effect of excess alcohol in shifting the reaction toward ester formation. However, further increases beyond a 12:1 ratio may lead to increased glycerol solubility and separation difficulties. Therefore, a 12:1 ratio is a practical upper limit under the present conditions. Reaction temperature also played a crucial role. Across the factorial runs, higher conversion rates were obtained at 40°C than at 65°C, despite the expectation that higher temperatures accelerate reaction rates. This behavior can be attributed to methanol evaporation at 65°C, which reduces the available alcohol in the liquid phase, thus lowering the overall conversion.

Fig. 1. Flowchart of the biodiesel production from WCO.

The influence of transesterification time was less significant than that of the molar ratio and temperature. Increasing the reaction time from 30 min to 90 min did not significantly increase the yield; in some cases, it led to slightly lower values. Once near-complete conversion is reached, a prolonged reaction can favor reverse or side reactions, which may intensify soap formation in the presence of FFA. However, this does not contribute to further yield improvement. Therefore, the factorial results indicate that 30 min is sufficient to achieve high conversion under optimized conditions. Catalyst loading had a non-monotonic effect on biodiesel production. A catalyst concentration of 0.5 wt% produced higher yields than a concentration of 1.5 wt% when all other conditions were identical. Excess NaOH promotes the saponification of residual FFA in WCO, increases soap formation, and stabilizes emulsions. This complicates phase separation and reduces the apparent biodiesel yield. Even under optimal conditions (40 °C, a 12:1 molar ratio, and 30 min), increasing the catalyst concentration to 1.5 wt% decreased the yield. This confirms that moderate catalyst loading is preferable for WCO transesterification. The 2^4 factorial design shows that a low temperature (40°C), short reaction time (30 min), moderate catalyst loading (0.5 wt%), and a 12:1 methanol-to-oil ratio are ideal for maximizing biodiesel yield from WCO while minimizing side reactions and separation issues. This design-of-experiments approach is more reliable for understanding factor interactions than the one-factor-at-a-time studies commonly reported in WCO biodiesel research.

B. Characterization of Biodiesel Produced

The essential properties of the biodiesel processed from WCO were evaluated by comparing them to relevant standards, as portrayed in Table II. Excessive specific gravity is a key determinant of fuel injection system performance and is linked to increased exhaust emissions, which contribute to environmental pollution and global warming. In Table II, the density of the produced biodiesel was measured at 858.0 kg/m³ using the ASTM D1298 hydrometer method [28]. The kinematic viscosity at 40 °C was 4.422 mm²/s, within the ASTM limit of 2.3–6.0 mm²/s, confirming that the transesterification process reduced the raw oil's high viscosity and improved the engine fuel system's flow characteristics. The produced biodiesel's acid number was 0.15 mg KOH/g, which is well below the ASTM maximum of 0.5 mg KOH/g. This indicates very low residual FFA content, suggesting good oxidation stability and a reduced risk of corrosion in the fuel system. The sulfated ash content was 0.02%, satisfying the ASTM limit and implying low levels of inorganic contaminants that could otherwise contribute to deposit formation. The cloud point of 18°C is consistent with ASTM guidance for similar fuels, indicating that the biodiesel is suitable for use in mild and warm climates where ambient temperatures do not fall significantly below this value.

With a measured flash point of 152 °C, this fuel significantly exceeds the ASTM minimum of 100 °C, enhancing safety during handling, transport, and storage. The distillation temperature at 90% recovery was 348 °C, which is below the ASTM maximum of 360 °C. This result confirms that the fuel meets the required volatility and distillation profile for biodiesel. The copper strip corrosion rating of 1A satisfies the ASTM specification and indicates negligible corrosive effects on metallic components in contact with the fuel. The iodine value of 63.84 g I_2/100 g is lower than the ASTM upper limit and reflects a moderate degree of unsaturation, balancing oxidative stability and cold-flow behavior. The methyl ester content of 97.93% exceeds the minimum requirement of 96.5%, demonstrating an efficient transesterification process and high triglyceride-to-ester conversion. The measured contents of monoglyceride (1.35%), free glycerol (0%), and total glycerol (0.26%) are within or near ASTM limits. This indicates that undesirable glyceride species and residual glycerol are adequately controlled. This is important for preventing deposits and injector fouling. Taken together, these results confirm that biodiesel produced from WCO under optimized conditions fully complies with ASTM biodiesel specifications and is suitable as a diesel substitute or blend component. The combination of a high production of 96% and ASTM-compliant properties under relatively mild and simple operating conditions highlights the technical feasibility and practical relevance of WCO-based biodiesel production. Furthermore, the 2^4 factorial design approach used in this study provides detailed operating guidelines that can serve as a reference for process design, optimization, and scale-up in future WCO biodiesel applications.

TABLE II. PROPERTIES OF THE BIODIESEL PRODUCED FROM WCO

Properties	**Unit (s)**	**Experimental results**	**Biodesel ASTM standard**
Specific gravity	Kg/m^3	858.0	850 - 890
Kinematic viscosity, 40°C	mm^2/s	4.422	2.3 – 6.0
Acid number	mg KOH/g, max	0.15	0.5
Sulfated ash	%-mass, max	0.02	0.02
Cloud point	°C, max	18	18
Flash point	°C, min	152	100
Distillation temperature, (90% recovered)	°C, max	348.0	360
Copper corrosion (3 h at 50°C)	Class	1a	1
Carbon residue	%-mass, max	0.04	0.05
Iodine value	gI_2/100g, max	63.84	115
Methyl ester	%-mass, min	97.93	96.5
Monoglyceride	%-mass, max	1.35	0.8
Free glycerol	%-mass, max	0	0.02
Total glycerol	%-mass, max	0.26	0.24

IV. CONCLUSIONS

This study used a 2^4 full factorial design to optimize the production of biodiesel from Waste Cooking Oil (WCO). The process variables were reaction temperature, transesterification time, catalyst loading, and the methanol-to-oil molar ratio. The optimum biodiesel yield of 96% was achieved with the following process variable levels: 40°C reaction temperature, 30-min transesterification time, 0.5 wt% NaOH catalyst loading, and a 12:1 methanol-to-oil molar ratio. Factorial analysis revealed that a higher methanol-to-oil ratio (up to 12:1), relatively low temperature, short reaction time, and moderate catalyst loading favor maximizing biodiesel yield while minimizing soap formation and separation issues. The produced biodiesel exhibited physicochemical properties under

these optimized conditions that were in good agreement with ASTM biodiesel specifications, including specific gravity, kinematic viscosity at 40°C, acid number, sulfated ash, cloud point, flash point, distillation temperature, copper corrosion, carbon residue, iodine value, methyl ester content, monoglyceride, free glycerol, and total glycerol. These results confirm the technical feasibility of using WCO as a feedstock and demonstrate that high-quality biodiesel can be produced under relatively simple, economically viable operating conditions.

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