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OPTIMIZATION OF CATALYST CONCENTRATION IN BIODIESEL PRODUCTION FROM USED PALM OIL VIA ACID-CATALYZED TRANSESTERIFICATION

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ABSTRACT

The global energy crisis and environmental concerns have intensified the search for sustainable fuels like biodiesel. This study presents a systematic optimization of sulfuric acid (H_2SO_4) catalyst concentration for biodiesel production from used palm oil. The effect of H_2SO_4 concentration within the range of 0.25% to 2.0% (w/w) on biodiesel yield and key fuel properties was investigated. Results demonstrated that the biodiesel yield increased significantly with catalyst concentration, reaching a maximum of 96.44 ± 0.38% at an optimum of 1.5% (w/w) H_2SO_4. A further increase to 2.0% resulted in no significant yield improvement, indicating a plateau effect. The biodiesel produced at this optimum condition was analyzed for critical quality parameters. The measured values, acid value (0.20 mg KOH/g), flash point (235 °C), water content (0.04%), and density (0.89 g/cm³), were found to be in full compliance with the ASTM D6751 standard. The study conclusively establishes that a 1.5% (w/w) H_2SO_4 catalyst concentration is optimal for the efficient and economical production of high-quality, standard-compliant biodiesel from used palm oil, offering a viable waste-to-energy pathway for regions with abundant palm oil waste.

Keywords: Biodiesel, Palm Oil, Transesterification, Catalyst.

1. INTRODUCTION

The escalating global demand for energy, driven by population growth and rapid industrialization, continues to exert immense pressure on finite fossil fuel reserves. This, coupled with the volatile economics of petroleum-based fuels and their well-documented environmental impacts, has underscored the urgent need for sustainable and renewable energy alternatives (Changmai et al., 2020, Lamba et al., 2024). Among these alternatives, biodiesel has emerged as a promising substitute for conventional diesel due to its renewability, biodegradability, and superior emission profile, including significant reductions in carbon monoxide, unburned hydrocarbons, and particulate matter (Maulidiyah et al., 2021, Abdulkareem & Nasir, 2024).

Biodiesel is chemically defined as fatty acid alkyl esters, primarily Fatty Acid Methyl Esters (FAME), produced via the transesterification of triglycerides derived from vegetable oils, animal fats, or waste cooking oils with a short-chain alcohol, typically methanol, in the presence of a catalyst (Changmai et al., 2020, Cerón Ferrusca et al., 2023). The American Society for Testing and Materials (ASTM D6751) standard provides stringent specifications to ensure biodiesel quality and engine compatibility. While feedstocks like soybean, rapeseed, and sunflower oil are commonly used, the high productivity and widespread cultivation of oil palm make it a significant resource. In this context, used palm oil presents a particularly attractive, low-cost, and sustainable feedstock. Its utilization not only reduces production costs but also addresses waste disposal issues, especially in regions with high palm oil consumption (Lamba et al., 2024, Ooi et al., 2024).

The transesterification process is critically influenced by several parameters, among which the type and concentration of the catalyst are paramount. Homogeneous acid catalysts, such as sulfuric acid (H_2SO_4), are highly effective for feedstocks with high Free Fatty Acid (FFA) content, as they simultaneously catalyze both esterification of FFAs and transesterification of triglycerides, thereby minimizing soap formation (Asaad et al., 2023, Obidike et al., 2022). However, the catalyst concentration must be meticulously optimized. An insufficient amount leads to incomplete conversion and low yields, while an excess can foster side reactions, cause equipment corrosion, increase production costs, and complicate downstream purification (Asaad et al., 2023, Onyia et al., 2024).

Although extensive research exists on biodiesel production from various waste cooking oils, a review of the literature reveals a distinct knowledge gap. While many studies compare different catalyst types (e.g., NaOH vs. H_2SO_4 or homogeneous vs. heterogeneous), few have undertaken a systematic, quantitative investigation of the effect of incremental H_2SO_4 catalyst dosage specifically on the conversion of used palm oil, a feedstock often characterized by high FFA content (Maulidiyah et al., 2021, Obidike et al., 2022). Moreover, many studies report yield outcomes without a concurrent, comprehensive evaluation of how these incremental changes impact multiple critical fuel properties as per international standards.

This study is therefore designed to address this gap. The primary objective is to rigorously investigate the impact of varying sulfuric acid catalyst concentrations (0.25% to 2.0% w/w) on both the yield and the quality of biodiesel produced from used palm oil. The specific aims are:

1. To determine the optimal H_2SO_4 concentration that maximizes biodiesel yield.
2. To evaluate key fuel properties (acid value, flash point, water content, and density) of the biodiesel produced at the optimal condition against the ASTM D6751 standard.
3. To provide a clear, empirical correlation between catalyst dosage and process efficiency for this specific feedstock, contributing to the development of cost-effective and sustainable biodiesel production protocols relevant to regions with abundant palm oil waste.

2. MATERIALS AND METHODS

The used palm oil (UPO) feedstock was collected from various local restaurants in Nekede, Imo State, Nigeria. Methanol (CH_3OH, 99.8% purity) and concentrated sulfuric acid (H_2SO_4, 98% purity) were used as the alcohol and homogeneous acid catalyst, respectively. Potassium hydroxide (KOH), phenolphthalein indicator, and ethanol for titration were of analytical grade. All chemicals were used as received without further purification.

The collected UPO was first filtered through a muslin cloth to remove suspended food particles and solid impurities. It was then heated to 110°C for 15 minutes in a heated beaker to evaporate any residual moisture. The initial Free Fatty Acid (FFA) content of the pre-treated oil was determined to be 2.8% (equivalent to an acid value of 5.6 mg KOH/g) via a standard titration method (ASTM International, 2018), confirming the necessity of an acid-catalyzed process to avoid saponification.

Biodiesel production was carried out via a one-step acid-catalyzed transesterification process. For each experimental run, a fixed mass of 50 g of pre-treated UPO was measured into a 250 mL conical flask and heated to 60°C on a magnetic hotplate stirrer. This temperature was selected to balance reaction kinetics against methanol reflux (Ojiego et al., 2014).

Separately, a methanolic solution of H_2SO_4 was prepared by dissolving various masses of the catalyst into 40 g of methanol (corresponding to a methanol-to-oil molar ratio of approximately 1:8) (Tsaoulidis et al., 2023). The catalyst concentration was varied at 0.25%, 0.5%, 1.0%, 1.5%, and 2.0% (w/w of the oil). The methanolic H_2SO_4 solution was then added to the pre-heated oil. Using a heating mantle with magnetic stirrer, the reaction mixture was continuously stirred at a constant rate for 1 hour, maintaining the temperature at 60°C.

After the reaction, the mixture was transferred to a separatory funnel and allowed to settle for 24 hours for phase separation. The lower glycerol-rich layer was drained off, and the upper biodiesel layer was recovered. To purify the biodiesel, it was washed with warm distilled water (approximately 50°C) several times until the wash water became clear, indicating the removal of residual catalyst, methanol, and soaps. The washed biodiesel was then dried by heating to 110°C to evaporate any traces of water. All experiments were conducted in duplicate, and the reported yields represent the mean values.

The percentage yield of biodiesel was calculated using the formula:

% Yield = (Mass of Biodiesel Produced / Mass of UPO Used) × 100

Biodiesel Quality Analysis

The biodiesel produced at the optimal catalyst concentration (1.5% H_2SO_4) was analyzed for key fuel properties according to relevant ASTM standard methods.

Acid Value: Determined by potentiometric titration according to the ASTM D664 method (ASTM International, 2023).

Flash Point: Measured using a Pensky-Martens closed cup apparatus as per ASTM D93 (ASTM International, 2020a).

Water Content: Analyzed by volumetric Karl Fischer titration following ASTM D6304 (ASTM International, 2020b).

Density: Measured at 15°C using a digital densimeter in accordance with ASTM D4052 (ASTM International, 2022).

3. RESULTS AND DISCUSSION

Effect of Catalyst Concentration on Biodiesel Yield

The effect of sulfuric acid (H_2SO_4) catalyst concentration on the yield of biodiesel from used palm oil is presented in Table 1. The results represent the mean values of duplicate experiments, demonstrating high reproducibility.

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Table 1: Effect of H_2SO_4 Catalyst Concentration on Biodiesel Yield (n=2)

H_2SO_4 Catalyst (% w/w)	Mass of Biodiesel Produced (g)	Biodiesel Yield (%)	Standard Deviation
0.25	32.61	65.22	±0.85
0.50	40.60	81.20	±0.92
1.00	45.13	90.26	±0.45
1.50	48.22	96.44	±0.38
2.00	48.19	96.38	±0.42

The data reveal a clear positive correlation between catalyst concentration and biodiesel yield within the range of 0.25% to 1.5% w/w H_2SO_4. The yield increased substantially from 65.22% at 0.25% catalyst to a maximum of 96.44% at 1.5% catalyst. This trend can be attributed to the increased availability of H^+ ions at higher catalyst concentrations, which protonate the carbonyl group of triglycerides and free fatty acids, thereby enhancing the reaction kinetics for both transesterification and esterification (Asaad et al., 2023, Onyia et al., 2024).

However, a further increase in catalyst concentration to 2.0% w/w resulted in no significant improvement in yield (96.38%), indicating a plateau effect. The slight, statistically insignificant decrease suggests that the reaction had reached its completion point at 1.5% catalyst. Beyond this optimum, excess acid catalyst may promote minor side reactions, such as ether formation or slight dehydration of methanol, or increase the solubility of glycerol in the biodiesel phase, which can impede separation and slightly reduce the measured yield (Asaad et al., 2023, Obidike et al., 2022). Therefore, 1.5% w/w H_2SO_4 is identified as the optimal catalyst concentration, providing the best trade-off between high conversion efficiency and economical chemical usage.

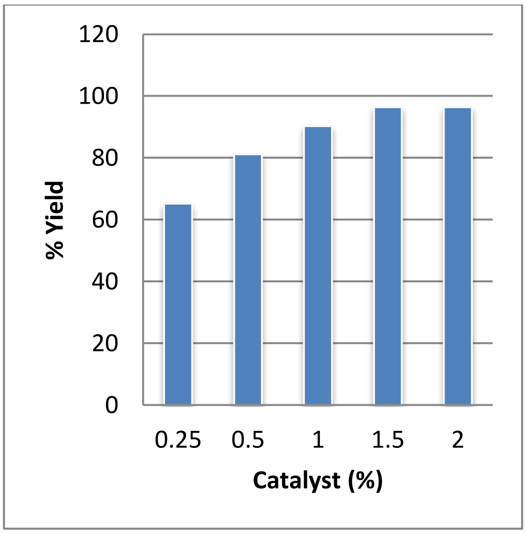

Figure 1: Variation of % Yield with Catalyst Dosage

Fuel Quality Assessment at Optimal Conditions

The biodiesel produced at the optimal catalyst concentration of 1.5% w/w H_2SO_4 was subjected to a comprehensive quality analysis. The results of key fuel properties, along with their corresponding ASTM D6751 standard limits, are presented in Table 2.

Table 2: Quality Parameters of Biodiesel Produced at 1.5% H_2SO_4 vs. ASTM D6751 Standards

Quality Parameter	Measured Value	ASTM D6751 Standard	Compliance
Acid Value (mg KOH/g)	0.20	≤ 0.50 (max)	Yes
Flash Point (°C)	235	≥ 130 (min)	Yes
Water Content (% vol)	0.04	≤ 0.05 (max)	Yes
Density at 15°C (g/cm³)	0.89	0.86 - 0.90	Yes

Acid Value: The measured acid value of 0.20 mg KOH/g is well below the maximum limit specified by ASTM D6751 (0.50 mg KOH/g). A low acid value indicates a minimal content of free fatty acids and other acidic compounds in the final product. This is crucial for preventing corrosion in engine fuel systems and storage tanks, and for minimizing deposit formation, thereby ensuring long-term engine durability (Obidike et al., 2022, ASTM International, 2023).

Flash Point: The flash point of the produced biodiesel was 235°C, which significantly exceeds the minimum safety requirement of 130°C. A high flash point indicates a low concentration of volatile and flammable substances (like residual methanol), making the biodiesel much safer to handle, store, and transport compared to petroleum diesel (ASTM International, 2020a).

Water Content: The water content was determined to be 0.04%, which is below the strict ASTM limit of 0.05%. Low water content is essential for preventing several operational issues, including microbial growth in storage tanks, hydrolysis of the biodiesel (which would increase the acid value over time), and corrosion. It also ensures that the fuel's calorific value is not compromised (ASTM International, 2020b).

Density: The density of the biodiesel was 0.89 g/cm³, falling perfectly within the ASTM-specified range of 0.86–0.90 g/cm³. Density is a critical parameter as it influences the operation of fuel injection systems. A density within the standard range ensures proper atomization of the fuel in the engine cylinder, which is vital for efficient combustion and optimal engine performance (ASTM International, 2022).

The comprehensive quality analysis confirms that the biodiesel produced from used palm oil under the optimized condition of 1.5% w/w H_2SO_4 is not only high-yielding but also a high-quality fuel that meets all the critical specifications of the ASTM D6751 standard, ensuring its compatibility with conventional diesel engines.

4. CONCLUSION

This study successfully demonstrates the critical importance of optimizing catalyst concentration in the acid-catalyzed transesterification of used palm oil for biodiesel production. The systematic investigation reveals a clear correlation between sulfuric acid (H_2SO_4) concentration and biodiesel yield, with a well-defined optimum at 1.5% (w/w). At this concentration, a maximum biodiesel yield of 96.44% was achieved, beyond which no significant improvement occurred, indicating catalyst saturation and potential onset of counterproductive side reactions at higher concentrations.

The comprehensive quality assessment of biodiesel produced at the optimal condition confirms its exceptional fuel properties. With an acid value of 0.20 mg KOH/g, flash point of 235°C, water content of 0.04%, and density of 0.89 g/cm³, the fuel fully complies with the stringent requirements of the ASTM D6751 standard. This confirms the technical viability of the produced biodiesel as a safe, efficient, and engine-compatible alternative to conventional diesel.

The findings provide a clear, empirically-derived optimization guideline for converting used palm oil—a prevalent waste stream in many developing regions—into high-quality biodiesel. The identification of 1.5% H_2SO_4 as the optimal catalyst concentration offers a practical pathway for cost-effective and sustainable biodiesel production, contributing to waste valorization and renewable energy generation.

While homogeneous H_2SO_4 is effective, further research should explore heterogeneous acid catalysts (e.g., sulfonated carbon, zeolites) or enzyme-based catalysts. These could offer advantages such as easier separation, reusability, reduced corrosion, and elimination of washing steps, thereby reducing environmental impact and operational costs.

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