Exploring Oil Palm Fruit Pulp for Direct Biodiesel Production via In-Situ Transesterification

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Abstract

Conventional biodiesel production from palm oil requires separate extraction and transesterification steps, leading to increased costs and complexity. This study introduces an innovative in-situ transesterification method utilizing oil palm pulp, eliminating the need for oil extraction and simplifying the production process, which ultimately reduces costs. The effects of catalyst type, methanol-to-pulp ratio, and hexane addition on biodiesel yield were systematically evaluated. Gas chromatography-mass spectrometry (GC-MS) was employed to confirm the biodiesel purity and assess the composition. Results showed that sulphuric acid (H_2SO_4) outperformed sodium hydroxide (NaOH) due to reduced soap formation, which hindered phase separation. The highest biodiesel yield of 38.79% was achieved at 75°C, using 3 wt% sulphuric acid, a 2:1 methanol-to-pulp ratio (ml:g), and a 24-hour reaction time, with no hexane addition. The presence of hexane as a co-solvent had minimal impact on biodiesel yield. This study demonstrates a cost-effective, simplified process for biodiesel production from oil palm pulp, offering significant potential for scaling up production. Future research could focus on conducting a detailed cost analysis and exploring the scalability of the in-situ process to validate its commercial viability.

Keywords: *In-situ transesterification, biodiesel, extraction, palm oil pulp, co-solvent*

1. Introduction

Biodiesel is emerging as a promising alternative to fossil fuels, particularly given the current shortage of conventional energy sources. Its renewable nature makes it a viable option for sustainable energy. Additionally, biodiesel produces fewer greenhouse gas emissions, contributing to a cleaner energy future. Conventionally, biodiesel is produced through transesterification using refined vegetable oils and methanol in the presence of a catalyst [1], [2]. However, this method involves multiple steps, including oil extraction and purification, which increase processing costs and energy consumption [3]. In-situ biodiesel production, also known as direct transesterification, simplifies the process by simultaneously extracting and converting the oil within the biomass into biodiesel [4]. This method is particularly attractive for palm oil fruit, one of the most abundant and high-yielding oil sources [5].

Palm oil is primarily extracted from the mesocarp of oil palm (*Elaeis guineensis*) fruit through mechanical pressing and solvent extraction. The conventional refining process involves several stages, including sterilization, digestion, pressing, and purification. The extracted crude palm oil undergoes

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refining to remove free fatty acids, impurities, and moisture before being used for biodiesel production [5]. However, these processing steps require significant energy input and generate waste by-products, raising concerns about sustainability. In contrast, in-situ biodiesel production eliminates the need for pre-extracted oil, allowing direct conversion of oil-bearing biomass into biodiesel, thereby reducing processing complexity and costs.

Several studies have explored in-situ transesterification as a cost-effective and energy-efficient approach to biodiesel production [4,6-10]. Researchers have investigated various biomass sources, including microalgae, jatropha seeds, and soybeans, demonstrating that direct transesterification can improve process efficiency by reducing solvent and catalyst consumption [4]. Tarigan et al., (2019) reported that a one-step reactive extraction–transesterification method for converting wet spent coffee grounds into biodiesel, using hexane as a co-solvent, achieved up to 97% conversion of fatty acids to FAME within 30 minutes and reduced energy consumption by more than 38% [7]. Kim & Yeom (2020) reported in their study on biodiesel production from waste coffee grounds that the one-step direct process (OSDP) reduced methanol and hexane usage by 69.7% and 67.2%, respectively, compared to the conventional two-step process, without compromising the biodiesel yield [8]. Al-Humairi et al., (2022) reported that biodiesel can be produced directly from the freshwater microalgae *Chlorella vulgaris* using an in-situ reactive extraction technique catalyzed by sulfuric acid, demonstrating that prior lipid extraction is not required, even with high water content [9].

While in-situ transesterification has been explored as a cost-effective approach to biodiesel production, most previous studies have focused on crude palm oil rather than directly utilizing oil palm pulp as the feedstock. In this study, oil palm pulp is selected not only for its oil content but primarily to reduce the number of processing steps typically required in conventional biodiesel production. By using the pulp directly, the process eliminates the need for separate oil extraction and purification prior to transesterification, thereby simplifying production and potentially lowering overall costs and energy consumption.

A study conducted by Jairurob et al., (2013) demonstrated the feasibility of using palm fruit fiber as a biodiesel feedstock with an alkaline catalyst (KOH), investigating reaction parameters such as catalyst loading, reaction time, and the methanol-to-oil molar ratio [11]. However, the impact of different catalyst types and the role of co-solvents in optimizing biodiesel yield from oil palm pulp remain underexplored. This study investigates several factors that affect biodiesel production from oil palm pulp. It compares the performance of acid and base catalysts and examines the impact of varying methanol-to-oil palm pulp ratios on yield. The study also explores how adding hexane as a co-solvent influences the overall biodiesel yield.

2. Methodology

2.1. Reagents and material

Fresh oil palm fruits were sourced from a local farm near Kulai in Johor, Malaysia. They were used as the primary feedstock for the study. Upon arrival at the laboratory, the fruits were thoroughly washed to remove any dirt and impurities before processing. The oil palm fruit pulp, also known as the fibrous mesocarp, was carefully separated from the kernel using a sharp knife.

After separation, the fibrous mesocarp was crushed using a high-speed blender, resulting in a homogeneous mixture that is optimal for the in-situ transesterification process. This blending step is critical as it increases the surface area of the biomass, thereby enhancing the efficiency of the oil extraction and conversion process.

The chemical reagents used in this study were of analytical grade. Methanol, hexane, sulphuric acid, and sodium hydroxide, each with a purity of above 99%, were purchased from Fisher Scientific. The use of these high-purity chemicals was crucial in minimizing side reactions and ensuring the

integrity of the transesterification process, which ultimately contributes to the reliability of the biodiesel yield data.

2.2. Experimental procedure.

In this study, approximately 25 grams of blended fibrous mesocarp (BFM) were measured and combined with 100 mL of methanol, followed by the addition of a catalyst - either sulphuric acid or sodium hydroxide - at a concentration of 3 wt% relative to the oil palm fruit weight. The 3 wt% catalyst concentration was selected based on the recommendation by Kim & Yeom (2020) who found it to be effective for biodiesel production in a similar one-step process [8]. The reaction was carried out using a 500 mL three-neck round-bottom flask and heated under reflux at 75°C for 24 hours as shown in Figure 1. Magnetic stirring was employed throughout the reaction to ensure proper mixing and contact between the pulp, methanol, and catalyst. After the reaction, the mixture was allowed to cool to room temperature and then filtered under vacuum to remove any insoluble materials. The filtrate was then decanted using a separating funnel and left undisturbed for 6 hours to achieve complete biodiesel separation. The filtrate consisted of two distinct layers of glycerol and biodiesel, which were then separated. The biodiesel was then washed with water and heated at 100^{o} C for 1 hour for the removal of methanol and the remaining impurities. Biodiesel produced from each test was analyzed using a GCMS. The yield of biodiesel was calculated as in equation 1. To assess reproducibility, one experiment was repeated in triplicate, while the remaining experiments were conducted once due to resource constraints.

The experiments were repeated with different ratios of methanol to oil palm pulp using sulfuric acid as a catalyst. Hexane was used as a solvent in the extraction of oil. Further experiments were conducted to investigate the effect of hexane addition to methanol. The range of methanol to hexane volume ratio was 10:0 to 10:5

$$\%\, Yield\ of\ biodiesel = \frac{mass\ of\ product\ (g)}{mass\ of\ mesocarp\ used\ (g)} \text{ x } \%\ of\ FAME\ in\ product \text{ x } 100\ \% \qquad (1)$$

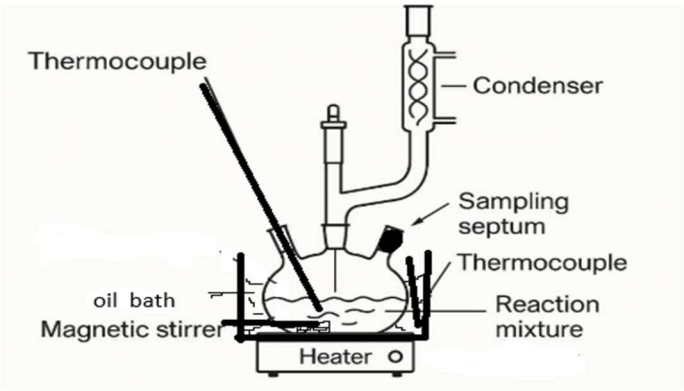

Figure 1. Reactor configuration for in-situ biodiesel production from palm oil pulp

3. Results and discussion

3.1. Comparative performance of sulphuric acid and sodium hydroxide as catalysts

The product from the experiment was analyzed using GC-MS to verify that it was biodiesel. The GC-MS analysis identified the presence of methyl esters, the main components of biodiesel, and assessed the purity of the product. The analysis was used to calculate the biodiesel yield, as described in Equation 1. The two main types of catalysts used in this process were acid catalysts (e.g., sulphuric acid, H_2SO_4) and base catalysts (e.g., sodium hydroxide, NaOH), each having distinct effects on the reaction and biodiesel yield.

In this study, the use of NaOH as a base catalyst resulted in the absence of a clear phase separation between biodiesel and glycerol. This was due to the formation of a large quantity of soap, which significantly hindered the separation process and reduced biodiesel yield. NaOH is highly reactive with free fatty acids (FFA) and water present in the oil, leading to saponification - a side reaction that produces soap instead of biodiesel. Consequently, no distinct layers were observed, making it difficult to isolate the biodiesel product. This outcome aligns with previous studies, which highlight that base-catalyzed transesterification is efficient for low-FFA oils but problematic for feedstocks with high moisture or FFA content due to unwanted soap formation [12], [13]

Conversely, the use of sulphuric acid (H_2SO_4) as an acid catalyst resulted in a biodiesel yield of 31.05%, with no significant soap formation. Acid catalysts are more effective for high-FFA feedstocks because they promote esterification, converting FFA into biodiesel instead of forming soap. This is particularly beneficial in the case of oil palm fruit pulp, which contains a substantial amount of FFA and water.

3.2. Influence of the methanol-to-blended fibrous mesocarp (BFM) ratio

The effect of methanol to blended fibrous mesocarp ratio (BFM) is shown in Figure 2. A significant increment in biodiesel yield was obtained when the ratio of methanol to BFM was increased from 1:1 to 2:1 but showed a decreasing trend at higher ratios. The trend was identical to results obtained by Zakaria and Harvey (2012) on in-situ transesterification of rapeseed [14]. It was reported that at a higher methanol-to-oil molar ratio, the separation of the ester and glycerol phases becomes increasingly difficult. Furthermore, excessive methanol may dilute the reaction mixture, potentially shifting the equilibrium away from biodiesel formation and leading to lower conversion. Excessive methanol can lead to reduced separation efficiency, lower biodiesel yield, and additional processing costs for methanol removal and recycling. Therefore, optimizing the methanol-to-BFM ratio is crucial for achieving high biodiesel yield, efficient separation, and economic feasibility of the in-situ transesterification process. The ability to directly convert oil-rich biomass such as BFM into biodiesel without a prior oil extraction step simplifies the process and reduces both equipment and solvent requirements. Methanol served both as a solvent and a reactant - facilitating lipid extraction from the BFM and enabling the transesterification reaction to proceed simultaneously. This dual function likely enhanced mass transfer between phases and contributed to the biodiesel yield observed. However, the detailed effects of methanol on mass transfer and reaction kinetics were not specifically examined in this study.

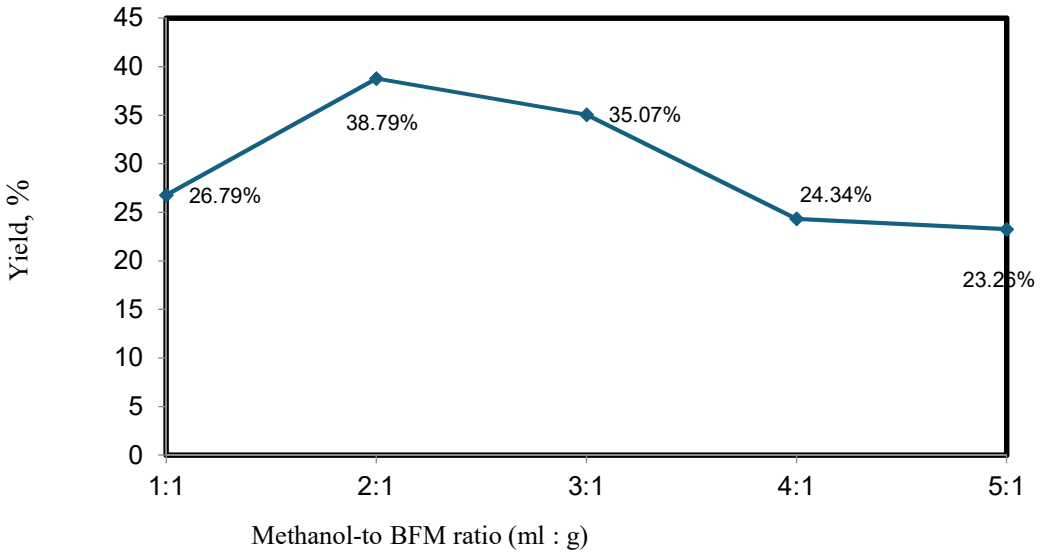

Figure 2. Biodiesel yield variation with different methanol-to-blended fibrous mesocarp (BFM) ratios

3.3. Impact of the methanol-to-hexane ratio on biodiesel yield

Hexane, a nonpolar solvent, is used to enhance oil extraction from the biomass by dissolving lipids that are otherwise less accessible. Figure 3 presents the effect of varying the methanol-to-hexane ratio on biodiesel yield. A slight increase in biodiesel yield was observed as the methanol-to-hexane ratio increased, reaching a maximum yield of 37.46% at a ratio of 10:3. However, further addition of hexane beyond this ratio led to a decreasing trend in biodiesel yield, with the yield dropping to as low as 27.23%.

This trend can be attributed to the dual role of hexane in the in-situ transesterification process. At lower concentrations, hexane acts as a co-solvent, improving oil extraction from the fibrous mesocarp and enhancing the reaction between triglycerides and methanol. This intensification effect facilitates better mass transfer, allowing for more efficient biodiesel production. However, excessive hexane dilutes the oil phase, leading to a lower concentration of reactants and subsequently reducing the reaction rate. As a result, excessive hexane may hinder effective contact between methanol and oil, thereby negatively impacting biodiesel yield.

Previous studies have reported similar observations, highlighting that only a small quantity of co-solvent is necessary to enhance extraction and intensify the transesterification process [9,15]. Excessive hexane not only lowers biodiesel yield but may also increase processing costs due to the additional energy required for solvent recovery and separation.

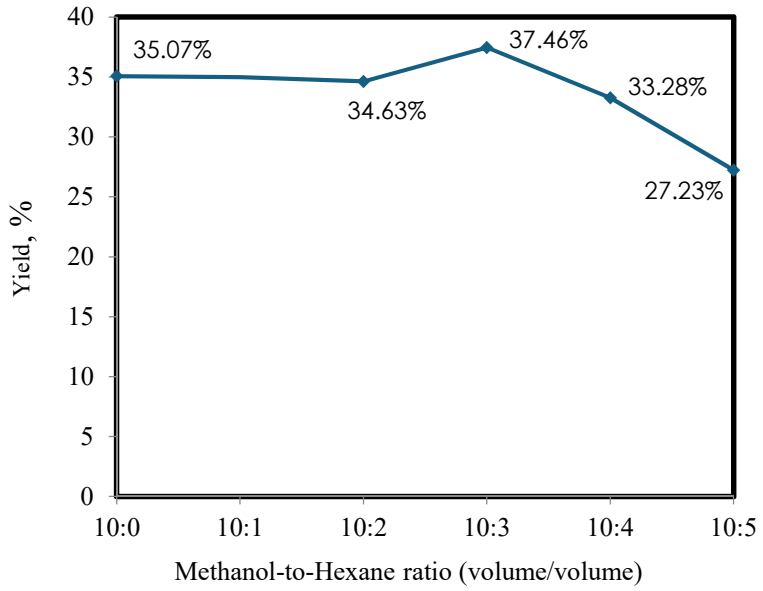

Figure 3. Biodiesel yield variation with changes in the methanol-to-hexane ratio

4. Conclusion

This study demonstrates that sulfuric acid (H_2SO_4) outperforms sodium hydroxide (NaOH) as a catalyst in the direct transesterification of palm fruit pulp for biodiesel production. The use of NaOH led to excessive soap formation, hindering phase separation and reducing biodiesel yield. The highest biodiesel yield of 38.79% was obtained at a methanol-to-oil palm pulp ratio of 2:1 (ml:g) with 3 wt% sulfuric acid catalyst, under reflux at 75°C for 24 hours. Notably, the addition of hexane as a co-solvent did not enhance the biodiesel yield, as it diluted the oil phase and slowed the reaction rate. These findings emphasize the importance of selecting the appropriate catalyst and process conditions for optimizing biodiesel yield from oil palm pulp.

Future research could focus on optimizing the in-situ transesterification process for large-scale biodiesel production and exploring alternative co-solvents or techniques to improve yield. Additionally, cost analysis would be valuable for assessing the commercial feasibility of this method.

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Conflict of interest

We declare no conflict regarding the publication of the study.

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