

Efficacy of Fly Ash as Additive on the Physical Properties of Biodiesel Extracted from Neem Oil

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ABSTRACT

Biodiesel is regarded as a sustainable alternative to conventional fossil fuels. This study investigated the influence of fly ash as a fuel additive on selected physical properties of biodiesel produced from neem (*Azadirachta indica*) oil through alkaline transesterification. The chemical composition of the fly ash was determined using X-ray fluorescence (XRF), while Fourier Transform Infrared (FTIR) spectroscopy confirmed the formation of fatty acid methyl esters (FAME). Fly ash was incorporated into the biodiesel at concentrations of 0.1–0.5 wt%, and its effects on pour point, flash point, fire point, and density were evaluated. XRF analysis showed that the fly ash consisted mainly of SiO₂ (52.63%), Al₂O₃ (24.41%), and CaO (10.04%), indicating the presence of mineral oxides capable of modifying fuel properties. FTIR spectra verified the successful conversion of neem oil into biodiesel through characteristic ester functional groups. The pour point decreased from 12°C for crude neem oil to 3°C at 0.3 wt% fly ash, indicating improved low-temperature flow behaviour. Flash point and fire point also decreased from 214°C and 222°C to 115°C and 133°C, respectively, at the same concentration. The density declined from 920 kg m⁻³ for crude neem oil to 870 kg m⁻³ at 15°C for biodiesel containing 0.5 wt% fly ash. Overall, fly ash effectively modified the physical properties of neem biodiesel, with 0.3 wt% providing the best balance between improved cold flow performance and acceptable fuel safety, demonstrating its potential as an economical and environmentally friendly biodiesel additive.

Keywords:

Fly ash,
FTIR,
Neem oil,
Pour point,
Fire point.

INTRODUCTION

The environmental degradation associated with the extensive use of fossil fuels has raised significant concerns regarding their long-term sustainability for economic development (Ismail *et al.*, 2022). Consequently, there is a growing need to transition toward renewable energy sources, particularly biodiesel, to meet the increasing global energy demand while minimizing environmental impacts (Wang *et al.*, 2020; Zakari *et al.*, 2024).

Among the various methods available for biodiesel production, transesterification remains the most commonly employed technique. This process involves a chemical reaction between vegetable oil or animal fat and

an alcohol, usually methanol or ethanol, resulting in the formation of fatty acid alkyl esters (biodiesel) and glycerol as a by-product. The reaction effectively reduces the molecular weight and viscosity of the oil, making it suitable for use as a diesel fuel substitute (Musa *et al.*, 2022).

Although edible vegetable oils have traditionally dominated commercial biodiesel production, their utilization has generated considerable concerns regarding food security, land competition, and production costs. Consequently, attention has shifted toward non-edible oils that can serve as sustainable feedstocks without competing with food resources. Among these feedstocks, neem (*Azadirachta indica*) oil has attracted increasing

interest because of its abundance, wide availability in tropical regions, high oil yield, and favourable physicochemical properties for biodiesel production (Onukwuli *et al.*, 2021; Dash *et al.*, 2021; Idris *et al.*, 2024).

Neem (*Azadirachta indica*) is a large evergreen tree widely distributed across tropical and subtropical regions, including Nigeria. The seeds of neem contain approximately 40% oil, making them a promising non-edible feedstock for biodiesel production. This suitability is attributed to their favorable physicochemical characteristics, such as high molecular weight, viscosity, density, and flash point (Musa *et al.*, 2022).

The suitability of biodiesel for commercial utilization depends largely on its physicochemical properties. Parameters such as density, flash point, fire point, viscosity, and pour point directly influence fuel atomization, ignition quality, storage safety, cold-flow behaviour, and engine performance. Consequently, these properties are routinely evaluated to determine compliance with internationally accepted fuel quality specifications such as ASTM D6751 and EN 14214 (ASTM International, 2023).

The pour point is defined as the lowest temperature at which a fuel remains capable of flowing. It occurs when sufficient wax crystals are formed within the fuel, causing it to gel and restrict fluid movement (Ismail *et al.*, 2026). Previous studies have reported varying pour point values for neem oil biodiesel. For instance, Taiwo *et al.* (2020) and Onukwuli *et al.* (2021) recorded pour points of 8.3°C and 4.0°C, respectively. Similarly, Nura *et al.* (2023) observed that the pour point of transesterified calabash oil decreased from 10°C to 6°C following purification, transesterification, and the incorporation of fly ash as an additive.

Flash point refers to the minimum temperature at which a fuel produces sufficient vapour to ignite momentarily when exposed to an external ignition source such as a flame or spark. It is inversely related to the volatility of the fuel (Abubakar *et al.*, 2025). Studies by Sekhar *et al.* (2019), Taiwo *et al.* (2020), and Onukwuli *et al.* (2021) reported flash point values of 128°C, 157.52°C, and 160°C, respectively, for neem oil biodiesel. These values fall within the ASTM biodiesel specification range of 100–170°C (Jamo *et al.*, 2023).

Fire point is the lowest temperature at which the vapour of a fuel continues to burn for at least five seconds after ignition (Babu & Anand, 2019). Sekhar *et al.* (2019) reported a fire point of 135°C for neem oil biodiesel. Likewise, Onukwuli *et al.* (2021) and Dash *et al.* (2021) obtained fire point values of 163°C and 173°C, respectively.

Density is a physical property that describes the relationship between the mass and volume of a substance and is commonly expressed in kilograms per cubic metre (kg/m³) for liquid fuels (Abubakar *et al.*, 2025). Nura *et*

al. (2023) reported changes in the density of calabash oil biodiesel following purification, transesterification, and the addition of fly ash. Similarly, Mallah and Sahito (2020) determined the density of neem oil biodiesel to be 910 kg/m³, while Taiwo *et al.* (2020) reported a value of 890 kg/m³.

Recently, considerable research has focused on improving biodiesel properties through the incorporation of additives. Metallic oxides, nanoparticles, bio-based additives, and industrial waste-derived materials have demonstrated the potential to modify combustion characteristics, thermal stability, oxidation resistance, and cold flow behaviour of biodiesel fuels (Sajjadi *et al.*, 2022; Yatim *et al.*, 2024). Among these materials, fly ash generated during coal combustion has attracted attention because it is inexpensive, abundantly available, and contains significant quantities of silica, alumina, calcium oxide, iron oxide, and other mineral oxides that possess catalytic and adsorptive properties. Utilising fly ash as a biodiesel additive also provides an environmentally sustainable pathway for converting industrial waste into value-added energy materials (Naskar & Sharma, 2025). Previous studies have investigated the application of fly ash in biodiesel systems. For example, Nura *et al.* (2023) reported that fly ash improved selected physical properties of calabash oil biodiesel. Similarly, recent investigations have explored the influence of different mineral-based additives on biodiesel derived from castor, olive, Jatropha, and other non-edible oils (Gwadabe *et al.*, 2024; Idris *et al.*, 2024). However, despite the promising characteristics of neem oil biodiesel, limited information is available regarding the influence of fly ash on its physical properties, particularly its effects on pour point, flash point, fire point, and density.

Therefore, this study investigates the influence of fly ash addition on selected physical properties of biodiesel produced from neem oil.

MATERIALS AND METHODS

Chemicals and Equipment

The chemicals, reagent and materials used in carrying out this research were; crude neem oil, sodium hydroxide (NaOH), fly ash, methanol, 64 % citric acid (C₆H₈O₇, purity: 99.7%), Silicon reagent, activated carbon, acetone and distilled water (H₂O).

The equipment used in carrying out this research were: magnetic stirrer with thermostatically controlled rotary hot plate (IKA C-MAG HS10), thermometer, measuring cylinder, Digital weight balance (AND model GT2000 EC), beakers, conical flask, 24 cm filter paper, funnel, Digital stop watch, sampling bottles, spatula, XRF machine and FTIR machine.

Methodology

XRF Characterization of Fly Ash

The elemental composition of the fly ash was determined using X-ray Fluorescence (XRF) spectroscopy with an ARL QUANT'X EDXRF Analyzer (S/N 9952120). In this technique, a 1 g sample of fly ash was exposed to X-rays, causing excitation of the constituent atoms and the emission of characteristic secondary radiation. The emitted radiation was detected, processed, and quantified by the instrument software, which converted the signals into elemental concentrations and displayed the chemical composition of the fly ash on the monitor (Durumin-Iya *et al.*, 2024).

SEM characterization of Fly Ash

The Scanning Electron Microscope [SEM] characterization of fly ash was done using multipurpose Scanning Electron Microscope [SEM] PHENOM PRO X MVE01570775. 1g of fly ash was scanned with a focused beam of electrons supplied by the machine.. The electrons interact with atoms in the fly ash, producing various signals that contain information about the surface topography and composition of the fly. The electron beam is scanned in a raster scan pattern, and the position of the beam is combined with the intensity of the detected signal which finally produces the image of fly ash (Ismail *et al.*, 2022; Uba *et al.*, 2024).

Purification of Crude Neem Oil

The crude neem oil was purified through a series of treatment processes. Initially, 200 mL of crude neem oil was heated on a magnetic hot-plate stirrer, after which 0.5 g of citric acid dissolved in 1.5 mL of distilled water was added. The mixture was continuously heated and stirred for 15 minutes. Subsequently, 4 mL of an 8% sodium hydroxide (NaOH) solution was introduced, and the mixture was further heated and stirred for another 15 minutes. The treated oil was then transferred into a vacuum oven and maintained for 30 minutes. Thereafter, 2 g of silicone reagent was added, and the mixture was heated and stirred for an additional 30 minutes. Finally, activated carbon was added at a concentration of 4 g per 100 mL of oil, followed by heating and stirring for 30 minutes. The purified oil was then separated using a separating funnel (Zakari *et al.*, 2024).

Transesterification of Neem Oil

A quantity of 60 g of purified neem oil was measured into a 250 mL conical flask and heated to a temperature range of 60–65°C using a magnetic hot-plate stirrer. Meanwhile, 0.6 g of NaOH was dissolved in 21 mL of methanol to prepare the methoxide solution. The methoxide was then added to the heated oil, and the resulting mixture was stirred and maintained at 65°C for 60 minutes. After the reaction, the mixture was allowed to cool for 40 minutes and transferred into a separating funnel. Two distinct

layers were formed: the upper layer consisted of biodiesel, while the lower layer contained glycerol and other reaction by-products (Auwalu *et al.*, 2025).

Preparation of Fly Ash Biodiesel Nanofluid

Fly ash powder was dispersed into 10 g of transesterified neem oil at concentrations ranging from 0.1 wt% to 0.5 wt%, with an increment of 0.1 wt%. The mixtures were continuously stirred using a magnetic stirrer for 2–3 hours to ensure uniform dispersion of the particles. The prepared nanofluids were subsequently subjected to further analyses (Gwadabe *et al.*, 2024).

Fourier Transform Infrared (FTIR) Spectral Analysis

Fourier Transform Infrared (FTIR) spectroscopy was carried out using a SHIMADZU FTIR-8400S spectrometer to identify the functional groups present in the samples. A thin film of the sample was prepared by placing a drop between two potassium bromide (KBr) discs. The sample was then exposed to infrared radiation, and the transmitted radiation was detected by the instrument. The resulting signal was amplified, digitized, and processed through Fourier transformation to obtain the infrared spectrum for functional group identification (Ismail *et al.*, 2022).

Determination of Pour Point

The pour point of crude neem oil was determined using a cylindrical test tube, thermometer, retort stand, and ice bath. The sample was cooled at a controlled rate of approximately 3°C min⁻¹ in the ice bath. The lowest temperature at which the oil remained capable of flowing within 5 seconds was recorded as the pour point. The same procedure was repeated for the purified neem oil, transesterified neem oil, and transesterified neem oil containing fly ash additive (ASTM International, 2021; Ismail *et al.*, 2026).

Determination of Flash Point

The flash point of crude neem oil was measured by heating 10 mL of the sample in a conical flask at a rate of 14–17°C min⁻¹ until the temperature reached 56°C. Thereafter, the heating rate was reduced to 5–6°C min⁻¹. A test flame was introduced at intervals of every 2°C rise in temperature until ignition of the vapour was observed. The lowest temperature at which the vapour ignited was recorded as the flash point. This procedure was similarly applied to the purified neem oil, transesterified neem oil, and transesterified neem oil blended with fly ash additive (ASTM International, 2022).

Determination of Fire Point

The fire point was determined by heating 10 mL of crude neem oil in a conical flask to 56°C and then gradually increasing the temperature. A test flame was applied at every 2°C increment until the vapour ignited and

sustained combustion for at least 5 seconds. The lowest temperature at which this occurred was recorded as the fire point. The same procedure was employed for the purified neem oil, transesterified neem oil, and transesterified neem oil containing fly ash additive (Nura *et al.*, 2023).

Determination of Density

Density meter machine was switched on and the stability of temperature was ensured. The measuring cell was cleaned and dried using little quantity of n-Hexane and then Acetone. The test sample was then injected into the density meter using a 10ml plastic syringe. It is important to ensure that the filled-in sample is homogenous and free

of gas bubbles. And the syringe was left in the filling position to prevent the filled-in sample from leaking. The upper hose was connected for evacuation and the lower hose for injection. And the upper density value was taken when reading was stable. Finally the sample of biodiesel from the density meter was evacuated and flushed with n-Hexane and then Acetone (ASTM International, 2023; Ayodeji *et al.*, 2023).

RESULTS AND DISCUSSION

XRF OF Fly Ash

The XRF of fly ash conducted at Umaru Musa Yaradua University Katsina Central laboratory were presented in table 1 in terms of compound percentage concentration.

Table 1: XRF of Fly Ash

S/N	Compound	% Concentration of Compound
1	SiO ₂	52.63
2	Al ₂ O ₃	24.41
3	CaO	10.04
4	Fe ₂ O ₃	3.20
5	MgO	2.51
6	K ₂ O	2.10
7	TiO ₂	1.21
8	P ₂ O ₅	0.52
9	Na ₂ O	0.40
10	SrO	0.33
11	BaO	0.21
12	Cr ₂ O ₃	0.12
13	MnO ₂	0.11
14	Others	1.18
15	Loss of Ignition	1.03

Table 1 shows elemental oxide composition of fly ash analyzed using X-ray fluorescence (XRF) analysis. From the results, it can be seen that fly ash contains mainly silica (SiO₂, 52.63%) and alumina (Al₂O₃, 24.41%), forming more than 77% of the total composition of fly ash. It is known that SiO₂ and Al₂O₃ have high thermal stability and adsorptive properties hence making fly ash ideal as fuel additives. This finding corroborates the findings reported by Naskar & Sharma, (2025); Shirsath, (2026). Calcium oxide (CaO, 10.04%) in fly ash is important since CaO is a catalytic agent and might help improve the quality of biodiesel due to improved interaction between the fuel and catalyst. Other metal oxides in smaller concentrations present in fly ash include iron oxide (Fe₂O₃, 3.20%), magnesium oxide (MgO, 2.51%), potassium oxide (K₂O, 2.10%), and titanium oxide (TiO₂, 1.21%). The metal oxides in the fly ash play an important role in the performance of biodiesel, in terms

of thermal stability and reduction of contaminants. This concurs with findings by Doucet, (2026); Vignesh & Sreevalli, (2026). The lower value of the loss on ignition (1.03%) reflects the presence of little or no unburnt carbon or any volatiles, implying that the fly ash is of high purity. Hence, the XRF test result confirms the presence of large amounts of mineral oxides which can be used to change or improve the physical characteristics of neem biodiesel. The findings support the results obtained by Nura *et al.* (2023); Doucet, (2026); Vignesh & Sreevalli, (2026).

SEM of Fly Ash

Plate 1 show the scanning electron microscope (SEM) of the fly ash at the magnification of 1000X (80µm and 10kV). It indicates the presence of dispersed particle with irregular shapes and cloud structure. This was similar to the result obtained by (Somerset *et al.*, 2004); (Chindaprasirt *et al.*, 2009) (Nura *et al.*, 2023).

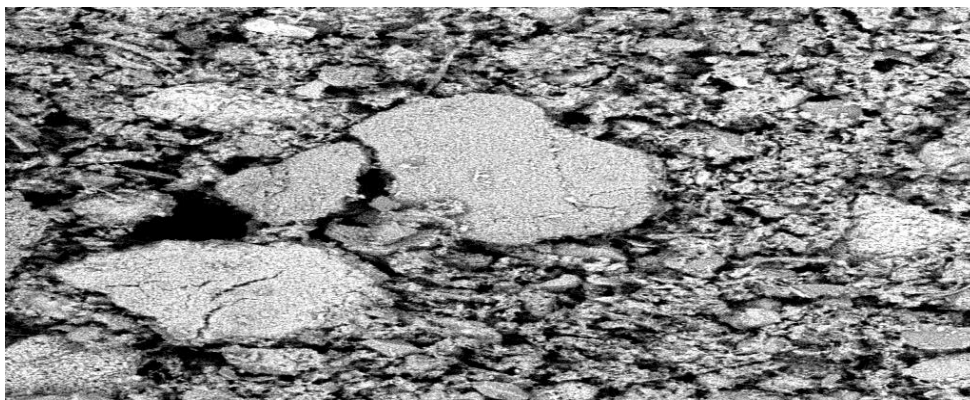


Figure 1: SEM of Fly Ash

FT-IR Spectra of Transesterified Neem Oil

Figure 1 presents the Fourier Transform Infrared (FTIR) spectrum of transesterified neem oil (biodiesel). The FTIR

spectrum serves as evidence of the successful conversion of neem oil into biodiesel through the transesterification process.

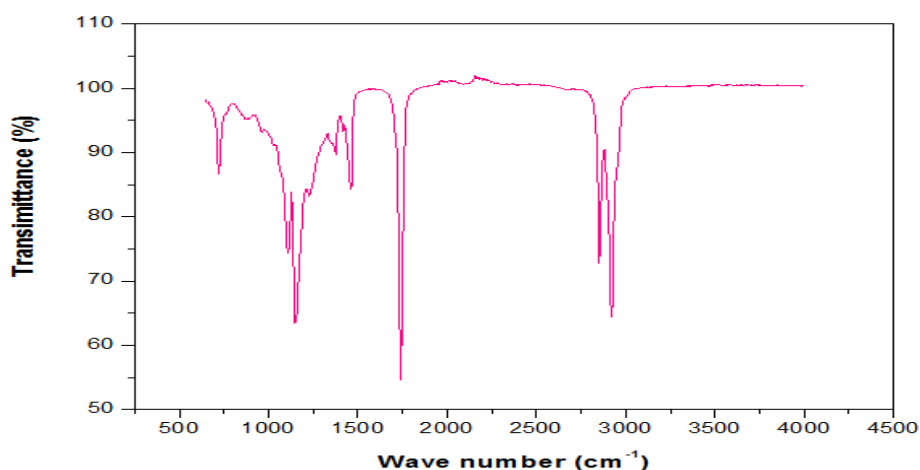


Figure 2: FT-IR Spectra of Trans Esterified Neem Oil

The spectrum of Figure 1 has the following characteristic absorptions bands, which correspond to fatty acid methyl esters (FAME). They constitute the main components of biodiesel. The important peaks observed include; Strong absorption band at 1740 cm^{-1} indicating ester carbonyl ($\text{C}=\text{O}$) stretching vibration showing the formation of methyl esters. Peaks between $2850\text{--}2950\text{ cm}^{-1}$ indicating C-H stretching vibrations from aliphatic hydrocarbons. Absorption bands ranging $1160\text{--}1240\text{ cm}^{-1}$ indicating C-O stretching vibrations of ester group. Peaks appearing at 1460 cm^{-1} and 1370 cm^{-1} representing CH_2 and CH_3 bending vibrations. The occurrence of these functional

groups signifies that the neem oil has successfully undergone transesterification reactions to produce biodiesel molecules hence proving the formation of biodiesel. According to the study carried out by Sridevi *et al.* (2026); Simao *et al.* (2026) and Chhikara *et al.* (2026) this was observed.

Pour Point, Flash Point and Fire Point of the Samples

Table 2 reveals the results of Pour point of crude neem oil, purified neem oil, trans-esterified neem oil and trans-esterified neem oil with addition of fly ash generated during the experiment.

Table 2: Pour Point, Flash Point and Fire Point of Crude Neem Oil, Purified Neem Oil, Trans-Esterified Neem Oil and Trans-Esterified Neem Oil with Addition of Fly Ash

S/N	Neem oil	Pour Point (°C)	Flash Point (°C)	Fire Point (°C)
1.	Crude	12	214	222
2.	Purified	10	190	210
3.	Trans-esterified	9	145	160
4.	Trans-esterified (0.1wt% fly ash)	7	135	151
5.	Trans-esterified (0.2wt% fly ash)	5	125	142
6.	Trans-esterified (0.3wt% fly ash)	3	115	133
7.	Trans-esterified (0.4wt% fly ash)	4	120	135
8.	Trans-esterified (0.5wt% fly ash)	6	134	145
9.	Biodiesel standard	-15 to 10	100 to 170	—
10.	Diesel standard	-15 to 5	60 to 80	60 to 80

As can be observed in Table 2, the pour point reduced continuously from 12°C for crude neem oil to 9°C after transesterification, suggesting enhanced low-temperature flow characteristics. When fly ash was added, there was a further reduction in pour point, with the lowest value of 3°C obtained at 0.3 wt% fly ash followed by an increase above this concentration. It means that fly ash plays a role in enhancing cold flow characteristics in biodiesel up to an optimum concentration of 0.3 wt%. At concentrations higher than 0.3 wt%, particle agglomeration might lower its efficiency. All values were recorded within the ASTM biodiesel standard limit ranging between -15°C and 10°C. According to a study by Nura *et al.* (2023); Ismail *et al.* (2026) and Sridevi *et al.* (2026)

In the same manner, there was a drop in the flash point of the biodiesel from 214°C for the crude neem oil to 145°C in the case of the transesterified biodiesel, as a result of the generation of lighter and highly combustible methyl esters. However, with the addition of fly ash at 0.3 wt%, the flash point was dropped to 115°C, thereby increasing fuel volatility and ignitability by increasing the concentrations higher than 0.3 wt%, which leads to the

lower efficiency of the fly ash. Although a decrease occurred, the resulting flash points are within the permissible range of ASTM biodiesel specification (100-170°C). This agrees with the results of Musa *et al.* (2022); Jamo *et al.* (2023); Ismail & Durumin Iya, (2025); Sarhan *et al.* (2026).

A similar trend was observed for the fire point, which decreased from 222°C for crude neem oil to 160°C after transesterification and further to 133°C at 0.3 wt% fly ash. This reduction indicates that the biodiesel requires less energy to sustain combustion. Importantly, the fire point at 0.3 wt% fly ash demonstrate acceptable combustion safety characteristics. This tally with the study conducted by Jamo *et al.* (2023) and Auwalu *et al.* (2025).

Densities of Samples

Table 3 were the result of density of crude neem oil, purified neem oil, trans-esterified neem oil and trans-esterified neem oil with addition of fly ash measured at 15°C.

Table 3: Density of Crude Neem Oil, Purified Neem Oil, Trans-Esterified Neem Oil and Trans-Esterified Neem Oil with Addition of Fly Ash

S/N	Neem oil	Density (kgm ⁻³)
1.	Crude	920
2.	Purified	903
3.	Trans-esterified	890
4.	Trans-esterified (0.1wt% fly ash)	885
5.	Trans-esterified (0.2wt% fly ash)	880
6.	Trans-esterified (0.3wt% fly ash)	875
7.	Trans-esterified (0.4wt% fly ash)	878
8.	Trans-esterified (0.5wt% fly ash)	884
9.	Biodiesel standard	870 to 900
10.	Diesel standard	850

The density of crude neem oil was determined as 920 kg m⁻³, and there was a reduction to 903 kg m⁻³ in density after purifying the oil. It is evident that the reduction in density could have been as a result of reduction in impurities, gums, free fatty acids, and other suspended

particles in the crude oil. As such, there was an increased refinement in the oil through purification process leading to reduction in its density. Thereafter, the density was found to reduce further to 890 kg m⁻³ after transesterification. This is due to the reduction in the

molecular weight and intermolecular forces when the triglycerides are transformed to methyl esters (ME). Further, the incorporation of fly ash led to another reduction in density of the biodiesel. With increasing fly ash content from 0.1, 0.2, and 0.3wt% up to 885, 880 and 875 kg m⁻³ respectively. The reduction may be associated with the adsorption of residual impurities and moisture by the fly ash particles, leading to a more homogeneous fuel with enhanced physicochemical characteristics. Furthermore, by increasing the concentration of fly ash above 0.3wt%, the density tend to increase which is as a result of the over addition of fly ash. The measured densities of biodiesel containing fly ash fall within the ASTM biodiesel standard range of 870–900 kg m⁻³, indicating compliance with international fuel quality requirements. The results were similar to the result obtained by Hamadou *et al.* (2020); Yatim *et al.* (2024); Ismail & Durumin Iya, (2025); Sarhan *et al.* (2026).

CONCLUSION

The efficiency of fly ash as an additive in terms of improving the physical properties of biodiesel obtained from neem oil has been successfully determined in this study. XRF studies showed that fly ash possessed high contents of silica, alumina, and calcium oxide, all possessing good adsorption and catalytic capabilities. FTIR spectrum indicated the formation of neem oil biodiesel via the presence of functional groups of fatty acid methyl esters.

The study showed that the use of purification, transesterification, and addition of fly ash had increased the physical properties of biodiesel obtained from neem oil. Pour point was reduced from 12°C for crude neem oil to a minimum of 3°C at 0.3 wt% fly ash. This showed that the biodiesel possesses better cold-flow properties. Flash point and fire point were also lowered to 115°C and 133°C, respectively. This means that the fuel has better ignition and combustion behaviors without violating ASTM biodiesel specifications. The density of biodiesel also lowered gradually from 920 kg m⁻³ in crude neem oil to 875 kg m⁻³ at 0.3 wt% fly ash.

The findings demonstrate that fly ash has the potential to modify the measured physical properties of neem biodiesel at the concentration of 0.3wt%. Nevertheless, additional studies such as the measurement of engine performance, combustion, emission and rheological has to be employed so that the fly ash has to be fully characterized as a biodiesel additive.

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