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Effect of Palm Oil Fuel Ash (POFA) Nano Particles on the Viscosity of the Trans-esterified Neem Oil

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Abstract

Biodiesel is being increasingly recognized as a promising alternative fuel amid concerns over the depletion of fossil fuels and environmental pollution. However, despite its well-known advantages, biodiesel still faces challenges such as high viscosity, density, and surface tension, which limit its direct use in unmodified diesel engines. This study investigates the impact of Palm Oil Fuel Ash (POFA) on the viscosity of transesterified neem oil. The physical and chemical properties of POFA were examined using scanning electron microscopy (SEM) and X-ray fluoroscopy (XRF). The process involved purifying the crude neem oil, followed by transesterification, with varying concentrations (0.1 wt%, 0.2 wt%, 0.3 wt%, 0.4 wt%, and 0.5 wt%) of POFA used as a heterogeneous catalyst. The viscosity of the resulting samples was then measured. SEM analysis revealed the presence of dispersed particles on POFA, while XRF analysis indicated that POFA primarily consists of silicon oxide. It was observed that the viscosity of the samples decreased with increasing temperature and with higher concentrations of POFA. For instance, the viscosity decreased from 60.45 mPas at 20°C for crude neem oil to 29.33 mPas at 60°C for transesterified neem oil with 0.5 wt% POFA. Importantly, the viscosities measured were found to consistent with ASTM standards. This research demonstrates the potential of using neem oil as a biodiesel feedstock with the addition of POFA as a catalyst.

Keywords: *Palm Oil Fuel Ash (POFA), Neem Oil, SEM, XRD, Viscosity.*

1. Introduction

From the early nineteenth century, the primary fuels used for transportation have been derived from petroleum. Predictions of imminent depletion of fossil fuels and heightened concerns over pollution hazards, as raised by health agencies, have spurred efforts to seek renewable sources of fuel for the transportation sector (Avinash et al., 2014). The combination of high demand and the negative global environmental impacts of fossil fuel usage raises questions about its reliability for sustainable economic growth (Ismail et al., 2022). As a solution, there is a global shift towards relying more on renewable sources to ensure energy supply meets the extensive demands for economic growth, improved living standards, and population expansion (Fadhil et al., 2018). Biodiesel considered as an eco-friendly, biodegradable, non-toxic, and carbon-neutral fuel, can be produced from both edible and non-edible oil feedstocks (Muhammad et al., 2023). The production of biodiesel depends on the availability of specific feedstocks and the cost of raw materials (Jiang et al., 2022). According to Isiaka & Olufolahan (2018), biodiesel comprises long-chain mono-alkylic esters derived from fatty acids obtained from renewable resources, suitable for use in diesel

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engines. Blends with diesel fuel are denoted as "Bx", with 'x' representing the percentage of biodiesel in the blend. For instance, "B5" indicates a blend containing 5% biodiesel and 95% diesel fuel. Consequently, B100 denotes pure biodiesel (Romano et al., 2011). According to ASTM, biodiesel is defined as "a fuel comprised of mono alkyl esters of long-chain fatty acids derived from vegetable oils or animal fats, designated B100". Biodiesel contains no petroleum but can be blended with mineral diesel fuel at any level to create a biodiesel blend. The vegetable and animal-derived feedstocks used for biodiesel production are referred to as triacylglycerides (TAGs), or simply triglycerides (Fadhi et al., 2017).

Viscosity is the resistance of a fluid (liquid or gas) to change its shape or movement of neighboring portions relative to one another (Jamo et al., 2023). It characterizes the resistance to the flow of liquid and is inversely proportional to fluid velocity (Jamo et al., 2023). Higher viscosity indicates more unfavorable use; sedimentation in equipment, poor atomization, and difficult evaporation are consequences of high viscosity or maintaining incorrect temperature (Hoang, 2021). Typically, biodiesel viscosity is slightly higher than that of conventional diesel fuel at 40°C. However, at lower temperatures, biodiesel viscosity increases significantly, especially below 25°C. Various methods exist to reduce biodiesel viscosity, such as preheating, blending, or using additives (Nguyen & Kim, 2017).

The most widely recognized method for producing biodiesel is transesterification, a chemical process involving consecutive reversible reactions between the triglyceride segment of vegetable oil and an alcohol in the presence of a catalyst. This process produces ester (i.e., biodiesel) and a by-product (i.e., glycerin) (Uba et al., 2024). The triglyceride is stepwise converted to diglyceride, monoglyceride, and finally glycerol, with 1 mol of alkyl ester removed at each step. The formation of alkyl ester from monoglyceride is believed to determine the reaction rate, as monoglyceride is the most stable intermediate compound. Common alcohols used in transesterification include methanol, ethanol, propanol, and butanol (Musa et al., 2022). Neem, scientifically known as Azadirachta indica, is a tree in the family Meliaceae that grows in various parts of the world, including Nigeria. Its seeds contain 40% oil, making them highly suitable for biodiesel production. Neem oil has a higher molecular weight, viscosity, density, and flash point than diesel fuel. It is typically light to dark brown, bitter, and has a strong odor reminiscent of peanuts and garlic (Zakari et al., 2024). In a study by Jamo et al. (2023), it was found that the viscosity of samples decreases with increasing temperature, but at 0.2 wt% of eggshell, it shows variation at standard intervals for changes in temperature. This suggests a potential catalytic behavior of eggshell as a heterogeneous catalyst at 0.2 wt%. The researchers concluded that biodiesel produced using jatropha as feedstock and eggshell as a heterogeneous catalyst at 0.2 wt% via transesterification can be used in diesel engines, indicating that eggshell influences the viscosity of transesterified jatropha oil at 0.2 wt%.

Literature search has revealed no results regarding the effect of Palm Oil Fuel Ash (POFA) on the viscosity of transesterified neem oil. Therefore, the aim of this paper is to investigate the effects of POFA on the viscosity of transesterified neem oil.

2. Materials and Method

2.1 Chemicals

The materials and reagents utilized in conducting this research comprise crude neem oil (Azadirachta indica) with 8% concentration, palm oil fuel ash (POFA), 8% sodium hydroxide (NaOH), distilled water, 64% citric acid ($C_6H_8O_7$) with a purity of 99.7%, activated carbon, methanol, silicon reagent, and acetone.

2.2 Equipment

The instruments utilized in this study include a magnetic stirrer with a thermostatically controlled rotary hot plate model (IKA C-MAG HS10), a Brookfield Digital viscometer model (Brookfield, RVDV-I), a thermometer, beakers, conical flasks, measuring cylinders, a Cheng Sang Vacuum oven model (MA 0 -30L), a Digital weight balance

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(AND model GT2000 EC), 24 cm filter paper, a funnel, a Digital stopwatch, a spatula, a calorimeter, an electric heater, a lighter, sampling bottles, an FTIR machine, and an SEM machine.

2.3 Methodology

2.3.1 Catalyst preparation

The palm husk waste was gathered from Market Street, subjected to combustion until ash formation, and subsequently sifted to eliminate any coarse particles, resulting in the production of Palm Oil Fuel Ash (POFA) (Nura et al., 2023). Following the analytical procedure outlined by Nura et al. (2023), the POFA was then further characterized for its structural and chemical properties.

2.3.2 SEM characterization of Palm Oil Fuel Ash (POFA)

The multipurpose Scanning Electron Microscope (PHENOM PRO X MVE01570775 model) was utilized for the characterization of Palm Oil Fuel Ash (POFA). Approximately 1g of POFA was subjected to electron beam irradiation within the machine. Through this process, the electrons interacted with atoms within the POFA, generating diverse signals that provided insights into its surface topography and composition. The electron beam was systematically scanned in a raster pattern, with the position of the beam synchronized with the intensity of the detected signals, ultimately resulting in the formation of an image depicting the POFA (Stokes, 2008).

2.3.3 XRF Characterization of POFA

The X-ray fluorescence (XRF) characterization of Palm Oil Fuel Ash (POFA) was conducted using the ARL QUANT'X EDXRF Analyzer (S/N 9952120) model. In this process, X-rays emitted by the XRF machine interacted with the atoms of 1g of POFA, causing the displacement of electrons from their inner orbitals. This resulted in the excitation of the atoms and the emission of high-energy radiation, including photons and electrons. Subsequently, the emitted lines were detected and integrated to determine their intensity levels. Finally, these intensities were converted into elemental concentrations, which were then displayed on the monitor for analysis (Haschke, 2014).

2.3.4 Purification of Neem Crude oil

The crude neem oil underwent a purification process as follows: Initially, 200 ml of neem oil was measured using a measuring cylinder and then heated to 70°C using a hot magnetic stirrer equipped with a thermometer. Subsequently, 0.5g of citric acid was dissolved in 1.5ml of distilled water and added to the heated oil sample, which was continuously heated and stirred for 15 minutes at 70°C. Additionally, 4 ml of 8% NaOH solution (prepared by dissolving 8g NaOH in 100 ml of distilled water) was added to the oil and subjected to continuous heating and stirring for another 15 minutes at the same temperature. The resulting mixture was transferred to a vacuum oven and heated at 85°C for 30 minutes. It was then returned to the hot magnetic stirrer, where 2g of silicone reagent was added and allowed to heat and stir for an additional 30 minutes. The temperature was subsequently raised to 85°C, and 4g of activated carbon was added to each 100 ml of the oil sample, followed by another 30 minutes of heating and stirring. Finally, the mixture was separated using a separating funnel (Ismail et al., 2022).

2.3.5 Transesterification of Neem Oil

Approximately 60g of neem oil was measured and placed in a 250 ml conical flask, where it was heated and stirred to reach a temperature range of 60 – 65°C on a hot magnetic stirrer plate. Additionally, 0.6g of NaOH was measured using an electronic weight machine and dissolved in 21 ml of methanol before being added to the mixture. The solution was then allowed to heat for 60 minutes with continuous stirring on the hot magnetic plate. After maintaining a uniform stirring and heating for the specified time at 65°C, the mixture was carefully transferred into a separating funnel using a glass funnel. Subsequently, the mixture was left to cool for approximately 40 minutes. Upon cooling, it was observed to separate into two distinct liquid layers, with the upper layer identified as biodiesel and the lower layer as triglycerol fatty acid (Ismail et al., 2022; Musa et al., 2022).

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2.3.6 Transesterification of Neem Oil Using POFA as Catalyst

Approximately 60g of neem oil was weighed using an electronic weighing machine and transferred into a 250ml conical flask. It was then heated and stirred using a hot plate with a magnetic stirrer until it reached a temperature range of 60 – 65°C. A mixture of 0.6g of NaOH and 0.1wt% POFA was prepared by dissolving them in 21ml of methanol, which was subsequently added to the heated sample. The mixture was allowed to heat for 60 minutes before being transferred into a separating funnel. After this time, the sample was left to cool for about 40 minutes, during which it separated into two distinct layers, with the upper layer identified as biodiesel and the lower layer as triglycerol fatty acid. The same procedure was repeated for samples containing 0.2 wt%, 0.3 wt%, 0.4 wt%, and 0.5 wt% POFA (Ismail et al., 2022).

2.3.7 Measurement of viscosity

Viscosity measurements were conducted using a Brookfield viscometer model DV-II+PRO (S/N 621-216), operating at a speed range of 50 rpm with a spindle size of 2. The crude neem oil was placed into a beaker, and the viscometer was initiated with the appropriate angular speed selected. The viscometer provided readings of the viscosity of the crude neem oil, which were recorded. The same procedure was repeated for the purified neem oil, transesterified neem oil, and transesterified neem oil with POFA catalyst. The viscosity of each sample was measured accordingly (Jamo et al., 2023).

3. Results And Discussions

3.1 Scanning Electron Microscopy (SEM) of POFA

Figure 1 depicts the SEM image of POFA, revealing the presence of dispersed particles with a porous and mountainous structure at a magnification of 500X (537µm and 10KV). This finding aligns with the results obtained by Jamo et al. (2018).

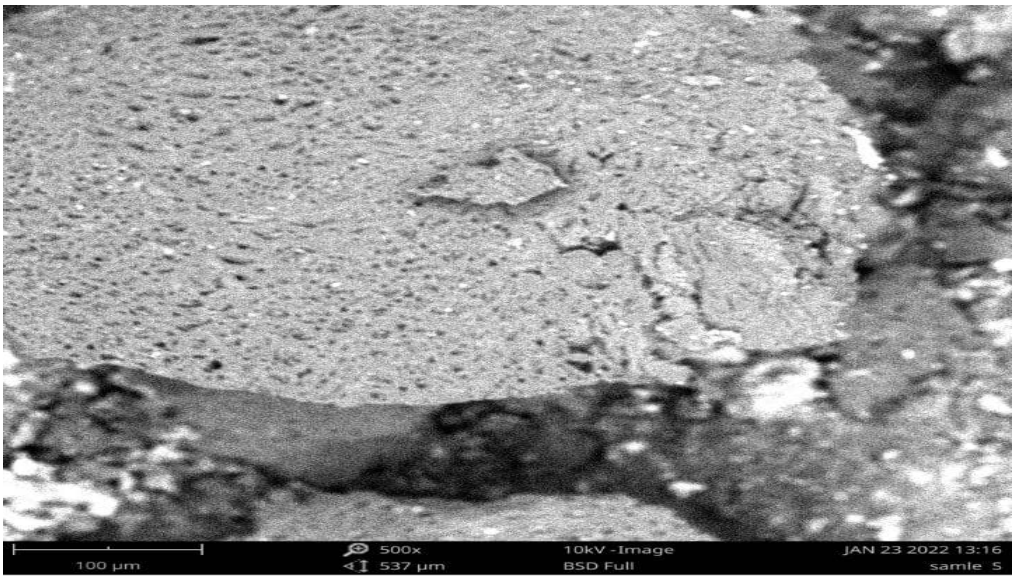

Figure 1: SEM of POFA at the magnification of 500X (537µm and 10KV).

3.2 XRF of POFA

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Table 1 presents the XRF analysis of POFA, detailing the compounds present and their corresponding percentage concentrations. According to the Table 1, SiO_2 accounts for 73.23% of the composition, followed by CaO at 8.34%, K2O at 5.45%, SrO at 3.15%, Al_2O_3 at 2.33%, MgO at 1.23%, P_2O_5 at 1.33%, SO_3 at 1.29%, Fe_2O_3 at 0.91%, with the remaining compounds collectively constituting 1.40%, and a loss of ignition at 1.34%. It's noteworthy that SiO_2 exhibits the highest concentration among the compounds, while Fe_2O_3 shows the lowest. This suggests that POFA primarily consists of silicon oxide. Consequently, POFA can be utilized as a catalyst without posing environmental concerns. These findings are consistent with the results reported by Aboustait et al. (2016) and Nura et al. (2022).

Table 1. X-ray Fluorescent of Palm Oil Fuel Ash (POFA).

S/N	Compound	Percentage concentration (%)
1	SiO_2	73.23
2	CaO	8.34
3	K_2O	5.45
4	SrO	3.15
5	Al_2O_3	2.33
6	MgO	1.23
7	P_2O_5	1.33
8	SO_3	1.29
9	Fe_2O_3	0.91
10	Lost of ignition	1.34
11	Others	1.40

3.3 Viscosities of the Samples

Table 2 presents the viscosity values of crude, purified, and transesterified neem oil in mPa·s. It can be observed that the viscosity of crude neem oil decreases from 60.45 mPa·s at 20°C to 45.33 mPa·s at 60°C, while the purified oil decreases from 55.44 mPa·s at 20°C to 37 mPa·s at 60°C. Similarly, the viscosity of the transesterified oil decreases from 48.22 mPa·s at 20°C to 32.21 mPa·s at 60°C. This decrease in viscosity can be attributed to the increase in temperature and the removal of residue and glycerol present in the crude neem oil. The viscosity values of the transesterified oils in Table 2 comply with ASTM standards and are consistent with results reported by Samuel et al. (2019) and Jamo et al. (2023). Furthermore, Figure 1 provides a graphical representation of the data presented in Table 2, depicting the viscosities (mPa·s) of crude, purified, and transesterified neem oil against temperature (°C). As depicted, the viscosity of the oil decreases as the temperature increases, a trend attributed to the purification and transesterification processes Jamo et al. (2023).

Table 2. Viscosity Of The Crude, Purified and Transesterified Neem Oil

Temperature (^oC)	Viscosity of Neem oil (mPa·s)		
	Crude Neem oil	Purify	Transesterified
20	60.45	55.44	48.22
30	56.33	51.49	45.67
40	50.43	47.33	40.32
50	48.35	43.48	37.34
60	45.33	37.00	32.21

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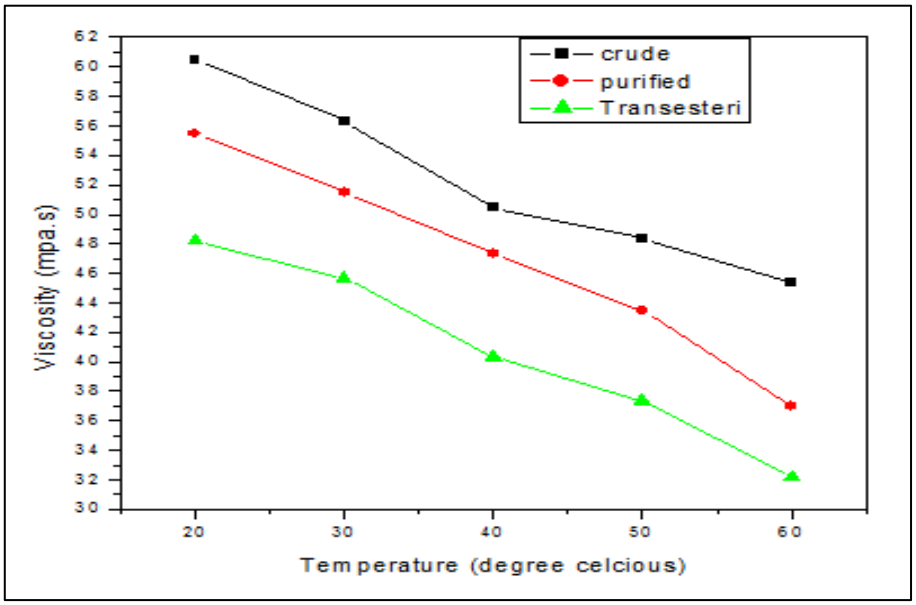

Figure 2: Viscosity of crude, purified and transesterified neem oil against temperature.

Table 3 presents the viscosities (mPa·s) of transesterified neem oil with varying concentrations of POFA (0.1 wt%, 0.2 wt%, 0.3 wt%, 0.4 wt%, and 0.5 wt%) as a catalyst. It is evident that the viscosities decrease with increasing temperature. Specifically, the viscosity of transesterified neem oil with 0.1 wt% POFA decreases from 44.30 mPa·s to 35.10 mPa·s over a temperature range of 20°C to 60°C. Similarly, the viscosities of neem oil transesterified with 0.2 wt%, 0.3 wt%, 0.4 wt%, and 0.5 wt% POFA decrease from 41.34 mPa·s to 33.21 mPa·s, 39.21 mPa·s to 31.78 mPa·s, 40.23 mPa·s to 28.34 mPa·s, and 39.27 mPa·s to 29.33 mPa·s, respectively. These decreases in viscosity are attributed to the reaction between neem oil and POFA. Notably, transesterification with 0.3 wt% POFA exhibits a consistent decrease in viscosity across the temperature range. However, it is observed that the viscosity increases beyond 0.3 wt% POFA, indicating that an excessive application of POFA leads to increased viscosity. The viscosities reported in Table 3 comply with ASTM standards and are consistent with findings reported by Musa et al. (2022) and Jamo et al. (2023).

Figure 3 represent the graphical form of the viscosity (mPa.s) of transesterified neem oil with 0.1 wt%, 0.2 wt%, 0.3 wt%, 0.4 wt%, and 0.5 wt% of POFA as catalyst against the temperature. Figure 3 illustrates the inverse relationship between viscosity and temperature, with viscosity decreasing as temperature increases. However, at 0.3 wt%, viscosity remains within a standard range of values. This suggests that biodiesel produced using neem oil as a feedstock and POFA as a homogeneous catalyst at 0.3 wt% can be effectively utilized as diesel fuel in various regions worldwide, including both temperate and tropical climates.

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Table 3: Viscosity of transesterified Neem oil with 0.1 wt%, 0.2 wt%, 0.3 wt%, 0.4 wt%, and 0.5 wt% of POFA as catalyst

Temperature (°C)	Viscosity of transesterified (mPa·s) in (wt%)				
	0.1	**0.2**	**0.3**	**0.4**	**0.5**
20	44.30	41.34	39.21	40.23	39.27
30	42.21	39.24	37.56	35.44	37.56
40	40.33	38.25	35.43	32.48	33.43
50	39.22	35.67	33.45	30.12	30.79
60	35.10	33.21	31.78	28.34	29.33

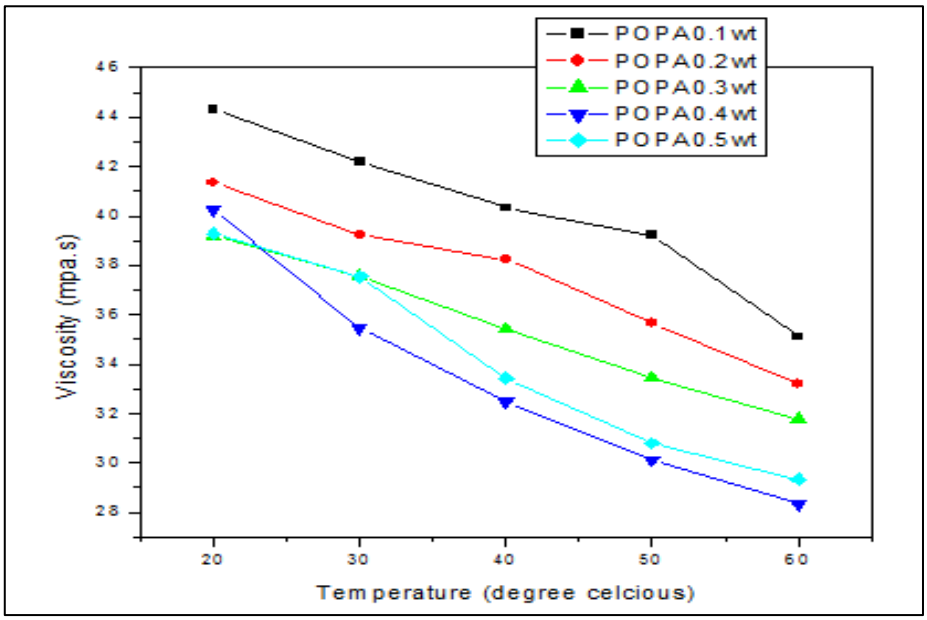

Figure 3: Variation of viscosity of transesterified neem oil with temperature.

4. Conclusion

In conclusion, biodiesel was successfully produced through trans-esterification and purification techniques. The dynamic viscosities of the samples, including crude, purified, and trans-esterified neem oil, were measured at different temperatures, revealing a consistent decrease as temperature increased. Notably, at 0.3 wt%, significant variation in viscosity was observed with rising temperature. Thus, it can be inferred that the sample containing 0.3 wt% POFA exhibited the most favorable properties among the eight samples tested. Further research is recommended to explore the impact of POFA on the viscosity of other transesterified oils, both as an additive and a catalyst.

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