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Full Length Research Article

INFLUENCE OF FLY ASH AS ADDITIVE ON THE VISCOSITY OF TRANSESTERIFIED NEEM OIL

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ABSTRACT
Biodiesel as the renewable source of energy has become one of the alternative solutions towards the problems associated with the use of fossil fuels in energy generation. Most of the biodiesel produced via tansesterification process are associated with viscosity that is above diesel limit. This problem can be mitigated through the use of solid materials as additive. This paper investigates the influence of fly ash as additive on the viscosity of transesterified neem oil. The morphology and chemical constituent of fly ash were studied using Scanning Electron Microscopy (SEM) and Xray Fluoroscopy (XRF). The crude neem oil was purified; transesterified and 0.1 wt%, 0.2 wt%, 0.3wt%, 0.4wt% and 0.5wt% of fly ash were used as additive. The viscosity of crude, purified, transesterified and transesterified with 0.1 wt%, 0.2 wt%, 0.3wt%, 0.5wt% and 0.5wt% of fly ash as additive were measured using brookfield digital viscometer. SEM shows that the mophorlogy of fly ash was dispersed particle with cloudy structure while XRF shows that the major compound presence on fly ash was silicon oxide. The viscosity of the samples decreases as the temperature increases and with the addition of fly ash. But on the addition of 0.3wt% it shows significant variation at equal range of temperature. This indicated that the fly ash can be used as biodiesel additive on neem oil as it decreases the level of viscosity presence on the oil.

Keywords: Fly ash, Neem oil, Scanning electron microscope, Xray flouroscope, Viscosity

INTRODUCTION
Fossil fuels, particularly petroleum, natural gas and coal have been the dominant sources of global energy sources for decades. These energy sources are non-renewable and are associated with environmental problems such as air pollution and global warming. (Awulu *et al.,* 2015). Renewable sources of energy such as solar, biofuel, hydro, geothermal etc. tend to provide solution towards the above problems (Ismail *et al.,* 2022). Biodiesel has taken the centre stage in several biomass and biofuel-based studies because it is produced from vegetable oils, which have the advantage of renewability (carbon neutral), biodegradability, safety, portability, non-toxicity, high heat content (about 88% of diesel fuel), better lubricity, lower sulphur and aromatic contents (Koh & Ghazi, 2011; Musa *et al.,* 2022).
The most adopted approach used in producing biodiesel is transesterification; which is the reaction between oil and an alcohol, usually methanol or ethanol to obtained alkyl-esters (often methyl or ethyl) and glycerol. This process reduces the molecular weight of the oil, its viscosity, density and increases the volatility to bring them closer to those of diesel fuel. The reaction is reversible, so an excess of alcohol is used to shift the balance in the direction of the products (esters and glycerol) formation. A molar ratio alcohol-oil of 6:1 is often used in the industrial process to achieve higher conversion rate to 98% of the oil (Djibri *et al.,* 2015; Jamo *et al.,* 2019).
Vegetable oils, however, are the most feedstock used in the biodiesel production. Edible vegetable oils for example rapeseed oil, soybean oil, sunflower oil, palm oil etc., constitute the first generation of feedstock for biodiesel. However, the use of inedible oils for energy purposes can avoid conflicts between food and energy security. That is why the research is now directed towards the use of non-edible oils which form the second generation of biodiesel feedstock. Thus, the neem seeds oil could be an interesting feedstock for biodiesel the production (Djibri *et al.,* 2015; Muhammad *et al.,* 2023).
Neem is a tree in the family '*maliaceae*' which grows on the various parts of the world including Nigeria. Its scientific name was 'Azadirachta indica'. The evergreen tree is large, reaching 12 to 18 meters in height with a girth of up to 1.8 to 2.4 meters. The seeds have 40% oil which has high potential for the production of biodiesel. It has a higher molecular weight, viscosity, density, and flash point than diesel fuel. Neem oil is generally light to dark brown, bitter and has a strong odor that is said to combine the odors of peanut and garlic (Ali *et al.,* 2013; Musa *et al.,* 2022).
Viscosity is the resistance of liquid to flow which is determined by measuring the amount of time taken for a given measure of oil to pass through an orifice of a specified size. There are two types of viscosity which include kinematic viscosity (cSt) and dynamic viscosity (mm^2/s or mPa.s) (Gashaw and Lakachew, 2014). viscosity is the most important property of biodiesel since it affects the operational performance of fuel injection equipment, particularly at low temperatures (Atabani *et al.,* 2013). It has been shown that the viscosity oil methyl esters decrease sharply after transesterification processes of the oil (Abbas *et al.,* 2010); (Atabani *et al.,* 2013); (Gashaw and Lakachew, 2014). In a research conducted by Raja *et al.,* (2011) found out the viscosity of Jatropha biodiesel decreases from 57 to 4.73cSt of the oil. Ali *et al.,*(2013) found the viscosity of neem oil biodiesel as 5.9cSt and similarly Djibri *et al.,* (2015) found the viscosity of neem oil to be $35.8mm^2/s$ at 40^0C. Musa *et al.,* (2022) found that the viscosity of transesterified neem oil decreases from 96mpa.s at the temperature of 10℃ to 24mpa.s at the temperature of 100℃.
The high values of viscosity associated with the biodiesel produced give rise to poor fuel atomization, incomplete combustion, and carbon deposit on the injectors (Ismail *et al.,*2022). These problems give rise to operational problems like difficulty in engine starting, unreliable ignition and deterioration in thermal efficiency (Gashaw and Lakachew, 2014). Different solid materials (fly ash, calcium oxide, silicon oxide, eggshell, crab shell) was used as additive in

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order to improve the quality of biodiesel which eventually reduced the amount of viscosity presence on the biodiesel (Ismail *et al.,*2022). But the use of fly ash as additive on transesterified neem oil has not been reported in the literature.

Fly ash, a by-product of coal combustion, is one of the most complex and abundant of anthropogenic materials. Fly ash consists of predominantly silt-size particles ranging from grey to tan to reddish brown. Analytical texture data from various investigators (Adriano *et al.,* 2015; Gupta *et al.,* 2016) indicates that, in general, fly ash has a silt loam texture. Fly ash is finer than 0.010 mm, depending on the type of removal system and furnace (Ahmaruzzaman, 2010); (Blissett and Rowson 2012); (Vassilev *et al.,* 2015). The experimental results show that fly ash had excellent activity and stability as additive on transesterified calabash oil (Nura *et al.,* 2023). The aim of this research is to study the influence of fly ash as additive on the viscosity of transesterified neem oil.

THEORETICAL BACKGROUND

This research is based on the characterization of fly ash (SEM and XRF), characterization of biodiesel produced (FTIR) and the measurement of viscosities of the samples.

The scanning electron microscope (SEM) is one of the most versatile methods available for the examination and analysis of the microstructure morphology and chemical composition characterizations (Zhou *et al.,* 2006). The technique utilizes the wavelength of light specifically absorbed by the samples which can best be described by equation 1 (Mäkinen *et al.,* 2006).

$$\Delta E = \frac{hc}{\lambda} \quad (1)$$

where E is the energy absorbed in eV, λ is the wavelength in m, h is the plank constant which is given by 6.67 x 10^{-34}Js and c is the speed of light given by 3.0 x $10^{9} ms^{-1}$.

Beer's formula relates light absorption and analyzes concentration as in equation 2 (Mäkinen *et al.,* 2006);

$$\log_{10}\frac{I_0}{I_t} = a \times b \times c \quad (2)$$

Where I_o = incident light intensity, I_t = transmitted light intensity, a = absorption coefficient (absorptivity), b = length of absorption path, c = concentration of absorbing atoms.

X-Ray fluorescence (XRF) is an established analytical method for the examination of the compound composition of bulk material and also for the characterization of coating systems (Haschke, 2014). According to Baranowsk *et al.,* (2001), If X-ray with quanta of energy E and an intensity of I_o passed through a layer of material, then the ray emerging from behind the iron layer will only be left with the intensity $I < I_o$ as a result of the absorption. The relationship between I and I_o after the transition through the layer of thickness x is given by:

$$I = I_0 e^{-\mu x} \quad (3)$$

where μ = linear absorption coefficient

Viscosity is the physical property that characterizes the flow resistance of simple fluids and is based on the Newton's law of viscosity which defines the relationship between the shear stress and shear rate of a fluid subjected to a mechanical stress. It states that the ratio of shear stress to shear rate is a constant, for a given temperature and pressure. The viscosity is independent of the shear rate and is defined by George & Qureshi (2013) as;

$$Coefficient\ of\ viscosity(\eta) = \frac{\text{Shear stress}}{\text{shear rate}} = Constant \quad (4)$$

where,

$$\text{Shear stress} = \frac{\text{Force (F)}}{\text{Area (A)}} \quad (5)$$

and the shear rate is defined as;

$$\text{Shear rate} = \frac{\text{Stain}}{\text{Time}} \quad (6)$$

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 (C 6H8O7, purity: 99.7%), Silicon reagent, activated carbon, acetone and distilled water (H_2O).

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, digital viscometer, sampling bottles, spatula, XRF machine and SEM machine.

Methodology

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 (Stokes, 2008).

XRF Characterization of Fly Ash

X-ray fluoroscopy characterization of fly ash was done using ARL QUANT'X EDXRF Analyzer (S/N 9952120) where by the incoming X-rays from an XRF machine knock the electron of 1g of fly ash atom out of the inner orbital. This results in the excitation of the atom and the production of high-energy radiation (photons, protons, electrons, etc.). The next process involves the detection and integration of characterized emitted lines to give varying levels of intensity. Finally, the detected line intensities are converted to elemental concentrations which is then displaced on the monitor (Haschke, 2014).

Purification of Neem Crude oil

The crude neem oil has been purified through the following procedure; 200 ml of the neem oil has been measured using measuring cylinder which is then freely-heated to $70^0\ C$ using hot magnet stirrer with thermometer. 0.5g of citric acid has been measured and dissolved into 1.5ml of distilled water and then added to the heated oil sample which is continuously heated and stirred for 15 minutes at $70^0\ C$. 4 ml of 8 % NaOH (by dissolving 8g NaOH in 100 ml of distilled water) has also been added to the oil and continuously heated and stirred for 15 minutes at $70^0\ C$.The mixture has been transferred to the vacuum oven where it has been heated at $85^0\ C$ for 30 minutes.Then it has been taken back to the hot magnetic stirrer and heated at $70^0\ C$ after which a 2g of silicone reagent has been added while it was being heated and stirred for 30 minutes. Then the temperature has been increased to $85^0\ C$ and 4 g of activated carbon has been added to each 100 ml of the oil sample, heated and stirred for 30 minutes. Then the mixture has been separated using separating funnel (Ismail *et al.,*2022).

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Transesterification of Neem Oil
60g of the neem oil has been measured in 250ml of conical flask and then heated and stirred to a temperature of 60-65℃ on a hot magnetic stirrer plate, 0.6g of NaOH has been measured using the electronic weight machine and allowed to dissolve in 21ml of methanol and then added to the mixture and allowed it to heat for 60 minutes with the stirrer on the hot magnetic plate. After 60 minute of uniform stirring and heating on the hot magnetic plate maintaining a temperature of 65°C, then it has been poured into the separating funnel through a glass funnel. The mixture has been allowed to cool for about 40 minute. Afterwards, it has been observed to separate into two liquid layers. The upper layer is the biodiesel and the lower layer is triglycrol fatty acid (Ismail *et al.,*2022).

Nano Fluid Preparation
The fly ash powder of concentrated 0.1wt%, 0.2wt%, 0.3wt%, 0.4wt% and 0.5wt% respectively were dispersed in to the 10g of transesterified neem oil and stirred for 2-3 hours using magnetic stirred which is then subjected to analysis (Musa *et al.,* 2022).

Measurement of Viscosity
Viscosity was measured using Brookfield viscometer DV-II+PRO (S/N 621-216) with an operational speed range of 50 rpm with spindle size of 2. The crude neem oil was poured into a beaker then the viscometer was started and angular speed was selected on it. The Viscometer revel the viscosity of the crude neem oil which has been read and recorded. The same procedure has been applied to the purified, transesterified and transesterified neem oil with the addition of fly ash concentration of 0.1wt%, 0.2wt%, 0.3wt%, 0.4wt% and 0.5wt% respectively (Nura *et al.,* 2023).

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.,* 2022).

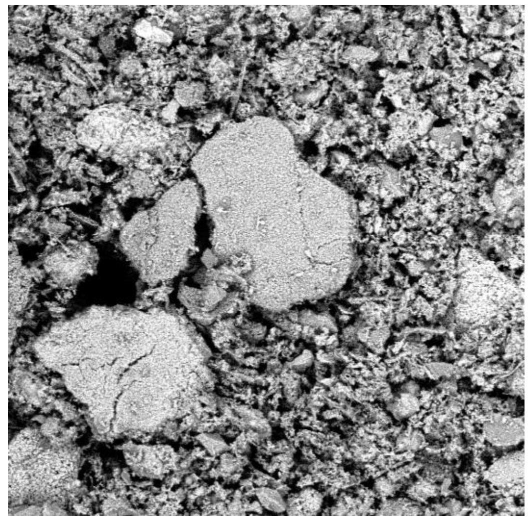

Plate 1: SEM of Fly Ash

XRF of Fly Ash
The XRF of fly ash were presented in Figure 1 in terms of compound percentage concentration against the compounds.

RESULTS AND DISCUSSIONS

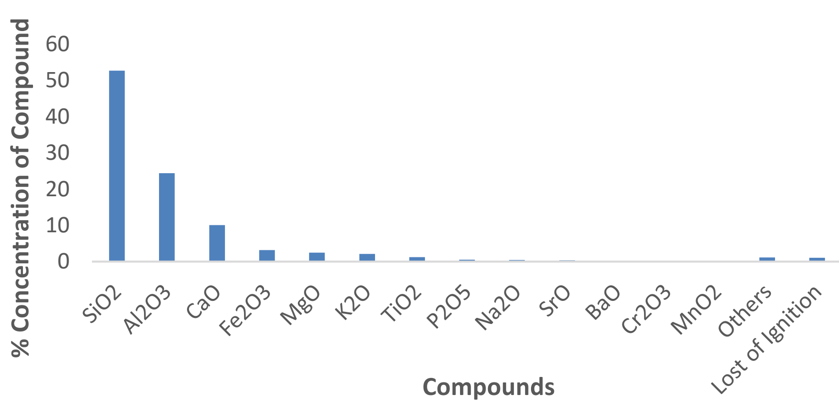

Figure 1: Percentage Concentration of Compounds

Based on Figure 1 SiO_2 was 52.63%, Al_2O_3 was 24.41%, CaO was 10.04%, Fe_2O_3 was 3.20%, MgO was 2.51%, K_2O was 2.10%, TiO_2 was 1.21%, P_2O_5 was 0.52%, Na_2O was 0.40%, SrO was 0.33%, BaO was 0.21%, Cr_2O_3 was 0.12%, MnO_2 was 0.11%,

Others were 1.18% and Lost of Ignition was 1.03%. It can be observed that SiO has the highest compound concentration in terms of percentage while MnO_2 has the lowest percentage concentration. This indicate that fly ash comprises mainly of silicon oxide. Due to this it can be used as additive without environmental problem. This result was similar to the result obtained by Somerset *et al.,* (2004); Yang *et al.,* (2014); Aboustait *et al.,* (2016) and Nura *et al.,* (2022).

Viscosity of the Samples
Figure 2 is the graph of viscosities (mpa.s) of crude, purified and

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trans-esterified neem oil against temperature (°C**).**

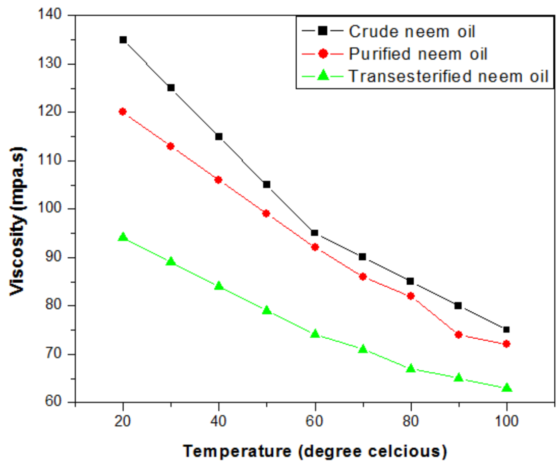

Figure 2: Graph of Viscosity of crude neem oil, purified neem oil and trans-esterified neem oil

It can be seen from figure 2 that the viscosity of crude neem oil decreases from 135mpa.s at the temperature of 20°C to 75mpa.s at a temperature of 100°C with a specific pattern, similarly the viscosity of purified neem oil decrease from 120mpa.s at 20°C to 72mpa.s at 100°C and finally that of trans-esterified neem oil decreases from 94mpa.s at 20°C to 63mpa.s at 100°C within a regular interval from 20°C to 60°C. Generally the viscosities of all the three samples decrease with the increase in temperature which is similar to the result obtained by Raja *et al.,* (2011) And Jamo *et al.,* (2022).

Figure 3 shows the graph of viscosities (mpa.s) of trans-esterified neem oil with 0.1wt%, 0.2wt%, 0.3wt%, 0.4wt%, and 0.5wt% fly ash against the temperature (°C**).**

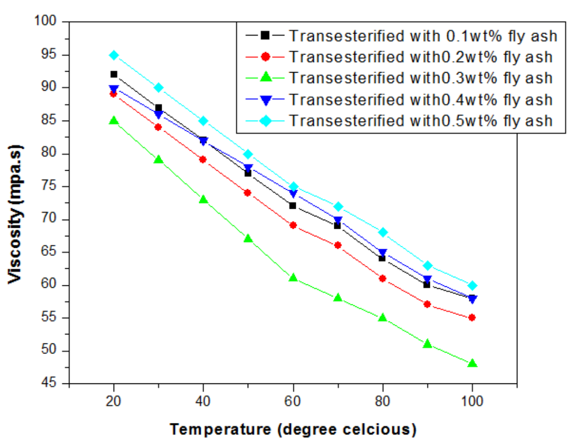

Figure 3: Viscosity of Trans-esterified Neem Oil with 0.1wt%, 0.2wt%, 0.3wt%, 0.4wt%, and 0.5wt% fly ash with Temperature

Figure 3 indicate that the viscosity of trans-esterified neem oil with 0.1wt% fly ash decrease from 92mpa.s at 20°C to 58mpa.s at 100°C then for 0.2wt% it decreases from 89mpa.s at 20°C to 55mpa.s at 100°C, then that of 0.3wt% decreases from 85mpa.s at 20°C to 48mpa.s at 100°C, equally for 0.4wt% it shows decreases from 90mpa.s at 20°C to 58mpa.s at 100°C and finally 0.5wt% fly ash decrease from 95mpa.s at 20°C to 60mpa.s at 100°C. It can be observed that the viscosities of the samples decreases with increase in the amount of fly ash from 0.1wt% to 0.3wt% e.g from 92mpa.s to 85mpa.s at 20°C then it increases after the addition of 0.4wt% fly ash and 0.5wt% fly ash i.e from 90mpa.s to 95mpa.s at 20°C. It can also be seen that 0.3wt% the viscosity decreases with increase in temperature at regular interval from 85mpa.s at 20°C to 61mpa.s at 60°C and then it decreases at irregular interval. This was as a result of the interaction between the amount of fly ash added and the neem oil. It can be deduced that the biodiesel produced using neem oil as a feedstock and fly ash as additive especially 0.3wt% can be used as diesel fuel at different parts of the world (temperate and tropic regions). The viscosities of figure 3 were within the ASTM limit and equally similar to the results obtained by (Raja *et al.,* 2011); (Jamo *et al.,* 2022).

CONCLUSION

This work investigate the influence of fly ash as additive on the viscosity of transesterified neem oil. The viscosity of the samples decreases as the temperature increases and with the addition of fly ash, but at 0.3wt% shows variation at standard interval for the change in temperature. This indicate a potential behaviour of fly ash as additive on transesterified neem oil for both insulation and energy generation. It is recommended to investigate the influence of fly ash as additive on other fuel properties of transesterified neem oil apart from the viscosity in oder to fully determine the additive behaviour of fly ash.

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