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Full Length Research Article

THE EFFECT OF ADDITION OF FLY ASH ON PHYSICAL PROPERTIES OF TRANSETERERIFIED CALABASH OIL AS SOURCE OF BIODIESEL

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ABSTRACT
The change in climatic patterns, depletion of fossils fuels and environmental issues have led to the search of alternative energy sources. One of these renewable sources of energy is biodiesel. This work investigates the effects of the addition of fly ash on the physical properties of transesterified calabash oil. The physical and chemical properties of fly ash were studied using scanning electron microscopy (SEM) and Xray fluoroscopy (XRF) characterizations. The crude calabash oil was purified, transeterified and fly ash were dispersed in the trans-esterified oil with concentration ranging from 0.1% to 0.4% in the interval of 0.1% Fourier Transform Infrared spectra (FTIR) was used to examine the structures of the samples were ester presence. SEM characterizations indicate the presence of dispersed particles with irregular shape on the nanoparticles. The pour point, fire point and the flash point of the samples were studied. It was found out among other things that small amount of fly ash (0.3%) in the oil could improve the physical properties of the fluid. The Nano fluid with 0.3% concentration of fly ash appears to have optimum physical property.

Keywords: Calabash oil, Flash point, Fly ash, Pour point, SEM

INTRODUCTION

The use of fossil fuels (petroleum, natural gas and coal) has dominated the sources of global energy supply for decades. But they are non-renewable and their reserves are rapidly being depleted; couples with the incessant environmental problems such as air pollution and global warming that are associated with them, efforts are increasingly being made to harness sustainable energy from renewable resources. Renewable energy sources provide 25% of the total global energy, of which 14-16% is provided from biomass (Itodo, 2007). Interestingly, alternative fuels, such as biodiesel, biohydrogen, biomethane and bioethanol are increasingly being generated from biomass in several places around the world. 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. It is strongly recommended to be used in place of petroleum-based diesel in diesel engines. Unlike the other biofuels which require some form of modifications before they can be effectively utilized in their respective areas, biodiesel can be used in diesel engines with little or no modifications. Moreover, vegetable oils, the precursor of biodiesel are biodegradable because they have both lipid soluble and water soluble properties (Koh *et al,.* 2011). Edible vegetable oils such as sunflower, soyabean, rapeseed, corn, and canola have been used for biodiesel production. Non-edible vegetable oils like Jathropha curcas and others have also been found suitable. So far, more than 95% of the global biodiesel production comes from edible oil resources, particularly from the agricultural industry. This has led to the concern that the knock-on effect of this practice would be a global adverse food shortage (i.e. food versus fuel dispute) (Koh *et al.,* 2011). But non-edible vegetable oils have been observed to be suitable for biodiesel production in developing countries because of food crises (Mustafa, 2011).

Biodiesel is defined strictly as the mono alkyl ester (usually methyl ester) of renewable fats and oils (Howell, 2007; Chouhananad *et al., 2011*). It consists primarily along chain fatty acid esters, produced by the transesterification reaction of vegetable oils with short chain alcohols. Distinct advantages of biodiesel include a high flash point of over 100°c, excellent lubricity, a BTU content comparable to that of petrol diesel, and virtually no suffer or aromatic content. Above all biodiesel is nontoxic and biodegradable (Gashaw *et al.,* 2014).

Biodiesel is produced via esterification of free fatty acid or by Tran's esterification of an oil or fat with a small alcohol (methanol or ethanol) in the present of a catalyst. The reaction is performed using chemical (acid or base) or enzymatic (mainly lipase) catalyst. (Sokoto *et al.,* 2020).

Ecological concern and intensive diesel oil consumption investigate the need to reasonable substitute fuels. Consumable vegetables oils are more expensive and hence, there is an inspiration to create biodiesel from non – edible vegetables oils such as calabash oil and waste cooking oil (Ibrahim *et al.*, 2016).

Calabash is classified to the kingdom of Plantae, division of magnoliophyta, class of Magnoliopsida, order of curcurbitacles, family of cucurbitaceous, genus of Lagenaria and species of lagenariasinceraria. Calabash is one of the first cultivated plants in the world, grown not for food and as container (Fran and will, 2008). It is found in many parts of northern Nigeria. It can be harvestested in 100 – 120 days after sowing (Osukoya *et al., 2021*). The calabash oil was found mostly in storage organ such as seeds and occur mainly in a dipose tissue that have to be extracted (Tolulope *et al.,* 2022; Dandago *et al,* 2009).

Researchers have shown that the properties of biodiesel may vary significantly depending on their chemical compositions and fatty acid composition, which give obvious effect on engine performance and emissions. Therefore, when considering a specific biodiesel a measurement of its properties (such as pour point, flash point, fire point, density, refractive index and soon) is required (Atabani *et al.,* 2013).s

Fly ash is a by-product produced by the combustion of coal and heavy/crude oil as a fuel in power production plants. Coal fly ash can be categorized into two distinct class i.e. F and C. class F fly ash is produced as a result of burning anthracite or sometimes bituminous coal. On the other hand, class C fly ash is produced by burning sub-bituminous coal or lignite (Das *et al; 2021*). The classification is based on the sum of the major oxides present in the fly ash such as silicon dioxide (SiO_2), ferroxide (Fe_2O_3), aluminium oxide (Al_2O_3) and MgO. In class F fly ash, these oxides represent more than 70% of the product, while in class C they represent about 50%. However, fly ash produce from heavy or crude oil consists of similar oxides than those of coal fly ash but a similar oxide than those of coal fly ash but a smaller concentration. Burning heavy or crude fuel oil yields around 3kg of ash per kiloliter of oil (Amran *et al.,* 2021).

Research conducted by (Jamo *et al.,*2019). Shows that addition of 0.5% of CeO_2 appears to be more suitable for insulation as the sample was observed to have lowest vicosity. They further explain that increasing the concentration of CeO_2 nanoparticle above 0.5% in the transterified Jatropha oil was observed to have initiated an increase in viscosity. The aim of this paper is to investigate the effect of the addition of fly ash on the physical properties of transesterified calabash oil as sources of biodiesel.

MATERIAL AND METHODS

Chemicals

The chemicals and materials used in carrying out this research are; crude calabash oil, sodium hydroxide (NaOH), fly ash and methanol.

Equipment

The equipment used in carrying out this study are: 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, Fourier transform infrared spectroscopy (FTIR) machine SHIMADZU FTIR-8400S, X-ray fluoroscopy machine ARL QUANT'X EDXRF Analyzer (S/N 9952120) and scanning electron microscope (SEM) machine PHENOM PROX MVE01570775.

METHODOLOGY

SEM Characterization of Addition (fly ash)

SEM characterization of fly ash was done at Umaru Musa Yaraduwa University Katsina central laboratory using multipurpose Scanning Electron Microscope machine operated at 15 kV which employed secondary signals which revel the surface morphology of the fly ash.

XRF Characterization of Addition (fly ash)

XRF characterization of fly ash was done at Umaru Musa Yaraduwa University Katsina central laboratory using XRF machine where by X-rays has been directed towards the fly ash which leads to the formation of diffraction. This diffraction is later sent through focusing slit to analyze the fly ash. Photons are diffracted with various wavelengths from the fly ash crystal and collected by the detector which transfers them to the computer for analysis.

Purification of Calabash oil

The crude calabash oil was purified through the following procedure; 200 ml of the calabash oil was measured using measuring cylinder; the oil was pre-heated to 70^{o}C using hot magnet stirrer with thermometer. 1.5 ml citric acid was measured and added to the heated oil sample and continuously heated and stirred for 15 minutes at 70^{o}C .4g of 8% NaOH (by dissolving in 100 ml of distilled water) was then be added to the oil and continuously heated and stirred for 15 minutes at 70^{o}C. The mixture was then transferred to the vacuum oven where it was heated at 85^{o}C for 30 minutes. Then the mixture was taken back to hot magnetic stirrer and heated to 70^{o}C after which a 2 g 0f silicone reagent was added while it was being heated and stirred for 30 minutes. Then the temperature was increased to 85^{o}C and 4 g of activated carbon was added to each 100 ml of the oil sample, heated and stirred for 30 munities. Then the mixture was separated using filter paper

Transesterification of Calabash Oil

60g of the Calabash 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 minutes of uniform stirring and heating on the hot magnetic plate maintaining a temperature of 65℃, then it has been poured into the separating funnel through a glass funnel. The mixture has been allowed to cool for about 40 minutes. 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.

Nano-fluids Preparation

The fly ash has been made locally from burning of fire wood. It is then removed impurities by using sieving material. Nano-fluids are prepared by two step process. The volume concentration of 0.1%, 0.2%, 0.3% and 0.4% of fly ash and transesterified calabash oil was made respectively. To make the fly ash more stable and remain more dispersed, each sample was stirred for 3-4 hours using magnetic stirrer, then the samples were taken for analysis.

Infrared Spectral Analysis of Transesterified Calabash Oil

The FTIR spectral analysis was done at Umaru Musa Yaradua University Katsina central laboratory using FTIR machine which revealed the functional group of the sample.

During the analysis, the sample in a form of thin film was placed between two potassium bromide discs made from single crystals, then a drop of the liquid is placed on one of the discs and the other is placed on top it which leads to the spreads of the sample into a thin film.

The source which is located at the FTIR machine generates radiation which passes through the sample and interferometer and finally reaches the detector. Then the signal is amplified and then converted to digital signal by the amplifier and analog to digital converter respectively. Finally, the signal is transferred to a computer in which Fourier transform is carried out.

Pour Point

Using an improvised method, the experimental procedures of pour point measurement for crude, purified and trans-esterified oils are enumerated below; the cylindrical test tube was filled with the crude calabash oil to a specific level mark (5ml). The test tube was clamped with a wooden clamp carrying the thermometer then placed in a bath of crushed ice (ice bath) and allowed to cool at a specified rate interval of 3ºC for flow characteristics the lowest temperature at which the movement of the oil is observed within 5 s is taken as pour point on the thermometer. ASTM 1999, D 97. The same procedure was repeated for the purified and trans-esterified calabash oils.

Flash Point

The flash point for crude, purified and trans-esterified oils was also measured; A 100 ml conical flask was filled to a specific mark level (10 ml) with crude calabash oil and heated at 14 t0 17 ºC / min (25 to 30 ºF / min) on the hot plate until the temperature is 56 ºC (100 ºF) below the expected flash point, the rate of temperature changes was then reducing to 5 to 6 ºC /min (9 to 11 ºF/ min) and the test flame was applied for every 2 ºC (5 ºF) until the oil burn for at least 5s. The flash point was taken at the lowest temperature when an application of the flame test caused the vapor above the sample to ignite. ASTM 1999, D 92. The same procedure was repeated for the trans-esterified calabash oils.

Fire Point

The fire point is the temperature at which bio lubricant combustion would be sustained for at least five minutes after the ignition source has been removed.

Density

The weight of a small beaker is determined using an electronic weighing balance. 2ml of the oil poured into it and the weight noted. Density is calculated using equation

Density = mass of oil / volume of oil weight

RESULTS AND DISCUSSION

Surface Morphology of fly ash

Figure 1 revealed the SEM of the fly ash. It indicates the presence of dispersed particle and irregular shapes of the particles at the magnification of 500X (100µm and 10KV). This is similar to the result obtained by (Joseph *et al.,* 2022).

Figure 1: SEM of fly ash

Elemental Analysis of fly ash

The XRF chemical constituents of fly oxide is presented in table 1. Based on the results, SiO_2 and Al_2O_3 have the highest percentage in terms of the oxide concentration on the fly ash.

Table1: percentage concentration of element

S/N	Compound	% Concentration Of Compound
1	SiO_2	58.26%
2	CaO	13.88%
3	SrO	10.29%
4	$Al2O_3$	6.03%
5	K_2O	3.32%
6	MgO	2.77%
7	P_2O_5	1.31%
8	SO_3	1.79%
9	Fe_2O_3	0.71%
10	Cl	0.34%
11	Others	1.30%

FT-IR Spectral Result

The band with peaks 650 to 1400 cm^{-1}represent C-O bond then 1500 to 1800cm^{-1} represent C=O bond while 2700 to 3000 cm^{-1} described C-H stretching from 3000 to 3700cm^{-1}described OH bond. Here C-O and C=O signify the presence of ester or ether group in the sample (Junior et al., 2018).

Figure 2, illustrates the FTIR spectrum plotted for transmittance against the wave number (cm−1) based on the amount of light absorbed by specific molecules present in the transesterified Calabash oil, the ester is at 723.179934, 964.191125, 1162.970700, 1237.582158, 1461.180168, 1744.461940, 2676.298215, 2855.168112 and 2922.256195 peaks. These indicate the presence of biodiesel in the sample.

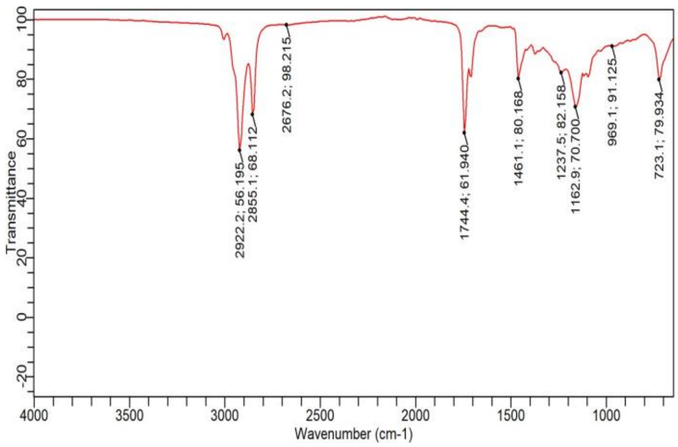

Figure 2: : FT-IR Spectra of Transesterified Calabash oil

Table 2: Pour point, Flash point and Fire point Results of Calabash

S/N	Samples	Pour point (°C)	Flash Point (°C)	Fire Point (°C)
1.	Crude Calabash Oil	10	115	135
2.	Purified Calabash Oil	7	117	152
3.	Trans – esterified	5	126	157
4.	Trans - esterified (0.1w% fly-ash)	3	127	160
5.	Trans - esterified (0.2w% fly-ash)	5	128	164
6.	Trans - esterified (0.3w% fly-ash)	4	129	168
7.	Trans - esterified (0.4w% fly-ash)	6	124	155
8.	Biodiesel standard	-15 to 10	100 to 170	>130
9.	Diesel standard	-15 to 5	60 to 80	60 to 80

The pour point of lubricating fluid gives an indication of the temperature below which it may not be possible to pour or remove the oil from its container. It was found that the pour point of the crude is 10°C while that of the purified oil is 7°C, for Transesterified is 5°C and for Trans - esterified (0.3w% fly ash) is 4°C. The pour point decreases as the fly ash concentration is increases. The Transesterified (0.3w% fly ash) oil has lower pour point due to the removal of gums from the crude oil, it is well known that lubricating fluid with low pour point is better especially in a cool whether condition.

The flash point test is sensitive in the study of lubricating fluid, because it gives the indication of the flammability of an oil. The flash point of the crude, purified, Trans – esterified and Trans - esterified (0.3w% fly ash) oils are 115°C, 117°C, 126°C and 129°C respectively, the higher temperatures of the purified oil indicates its usefulness for lubrication and it may be as a result of removing the flammables materials from the crude oil. The fire point test is very important in the study of lubricating fluid. The fire point of the crude, purified, Trans – esterified and Trans - esterified (0.3w% fly ash) oils are 135°C, 152°C, 157°C and 168°C respectively, this indicates that the fire point increases after purification, transesterification and transesterification with addition of fly ash. All are within the ASTM standard.

Table 3: Result of density

S/N	Sample	Density (g/cm^3)	Density (kg/ m^3)
1.	Crude calabash oil	0.861	861
2.	Purified calabash oil	0.872	872
3.	Trans-esterified calabash oil	0.874	874
4.	Trans-esterified (0.1w% fly ash)	0.875	875
5.	Trans-esterified (0.2w% fly ash)	0.876	876
6.	Trans-esterified (0.3w% fly ash)	0.877	877
7.	Trans-esterified (0.4w% fly ash)	0.881	873
8.	Biodiesel standard	0.88	880.00
9.	Diesel standard	0.85	850.00

The density of the samples is calculated as describe in the methodology and the result is summarized on table 4.3. It can be seen that the crude oil has low density than the purified oil, transesterified oil. But transesterified (0.3w% fly ash) has the highest density. But all are within the limit of biodiesel standard and that of diesel standard. This is similar to the result obtained by (Kanti *et al*., 2022).

CONCLUSION

This work investigate the influence of fly ash as additive on the physical properties of transesterified calabash oil. The mophorlogical characteristics of fly ash studied using scanning electron microscope indicate the presence of dispersed particle with irregular shape. The chemical consituent of fly ash studied through Xray flouroscopy revealed that the fly ash comprises of high percentage of SiO_2. The experimental results shows that the Transesterified nanofluid with (0.3w% fly ash) appears to be more suitable for insulation as the sample was observed to have lowest pour point of 4℃ and higher flash point of 129℃ as well as fire point of 169℃ together with higher density. This is due to the removal of gums in form of phospholipids from the crude oil. Hence, therefore, the result of the study shows that the trans-esterified nanofluid calabash oil with 0.3% oil is potential candidate for the production of lubricating fluid.

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