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PREPARATION OF BIODIESEL FROM SOYBEAN OIL BY TRANSESTERIFICATION

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Abstract
Biodiesel fuels were prepared from vegetable oil soybean and studied through viscosity, pour point, flash point and Fourier Transform Infrared (FTIR). The crude soybean oil was purified, trans-esterified and nanoparticles were dispersed in the trans-esterified oil with concentration ranging from 0.25% to 1.0% in 0.25% interval. Fourier Transform Infrared spectra (FTIR) was used to examine the structures of the samples, The viscosity, pour point and the flash point were studied. It was found out among other things that the optimal biodiesel volume was obtained at a temperature of 80 ºC and a time of 63 munities, also it was found that small amount of CuO(0.25%) nanoparticles in the oil could improve the physical properties of the fluid. The nanofluid with 0.25% concentration of CuO appears to have optimum physical property.

Key words: FTIR,Pour Point, Soybean Oil, Transesterification.

Introduction

Lubricants are being utilized in all sectors of industry for lubricating their machines and materials. Reports indicate that nearly 39 million metric tons of lubricants were used globally in 2017, with a projected increase of 1.2% over the next decade [1]. Approximately 85% of lubricants being used around the world are petroleum-based oils. Enormous use of petroleum based oils, created many negative effects on environment. The major negative effect is particularly linked to their inappropriate use, which results in surface water and groundwater contamination, air pollution, soil contamination, and consequently, agricultural product and food contamination. Stronger environmental concerns and growing regulations over contamination and pollution will increase the need for renewable and biodegradable lubricants [2].

An annual growth rate of 7–10% for environmentally favorable lubricants is expected on the market over the next few years compared to a rate of only 2% for the overall lubricant market [3]. Vegetable oils are a viable and renewable source of environmentally favorable oils. The vegetable oils provides qualities desirable in a lubricant. Long, polar fatty acid chains provide high strength lubricant films that interact strongly with metallic surfaces, reducing both friction and wear. The strong intermolecular interactions are also resilient to changes in temperature providing a more stable viscosity, or high viscosity coefficient. The entire base oil is also a potential source of fatty acids. But the oxidative stability, freezing point and viscosity of the oil were the primary obstacle to the industrial application [4-16].

The addition of nanoparticles into a lubricating oil is expected to enhance significantly the oxidative stability, reduces the friction coefficient and increases the load-bearing capacity of the friction parts in mechanical systems. A variety of mechanisms have been proposed to explain the lubrication enhancement of the nanoparticle suspended lubricating oil (i.e., nano-oil), including the ball bearing effect [5], protective film mending effect and polishing effect . These mechanisms can be mainly classified into two groups. The first is the direct effect of the nanoparticles on lubrication enhancement. The nanoparticles suspended in lubricating oil play the role of ball bearings between the friction surfaces. In addition, they also make a protective film to some extent by coating the rough friction surfaces [6]. The other is the secondary effect of the presence of nanoparticles on surface enhancement. The nanoparticles deposit on the friction surface and compensate for the loss of mass, which is known as mending effect. And also the roughness of the lubricating surface is reduced by nanoparticle-assisted abrasion, which is known as a polishing effect.

However, paper wishes to investigate the effects of CuO Nano particles on rheological properties of biodiesel extracted from soy bean. The addition of small amount of CuO Nano particles in biodiesel is expected to bring about boost some of the physical properties of the biodiesel.

Materials and Methods

Materials

Chemicals

The materials and reagents used in carrying out the research are as follows: crude soy bean oil (Azadirachta indica), 8 % sodium hydroxide (NaOH), 64 % citric acid ($C_6H_8O_7$, purity: 99.7 %), silicon (SiO_2) reagent, activated carbon, acetone, and distilled water (H_2O).

Equipment

The equipment's used in carrying out this study are: magnetic stirrer with thermostatically controlled rotary hot plate (IKA C-MAG HS10), Brookfield Digital viscometer {Brookfield,RVDV-I}, thermometer, measuring cylinder, Cheng Sang Vacuum oven (MA 0-30L), Digital weight balance (AND model GT2000 EC), beaker, 24 cm filter paper, funnel, Digital stop watch, sampling bottles, spatula.

Methodology

Sample Purification

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

Trans-esterification

60g of the crude soy bean oil was measured in 250ml of conical flask and was heated and stirred to a temperature of 80, 70, 55, 40 and 32 ℃ respectively on a hot magnetic stirrer plate, 0.6g of NaOH was measured using the electronic weight machine and allowed to dissolve in 21ml of methanol and then allowed to heat for 80,63, 60 and 57 minutes respectively with the stirrer on the hot magnetic plate. After uniform stirring and heating on the hot magnetic plate. Each sample was then poured into the separating funnel through a glass funnel and separated. The mixture was allowed to cool for about 40 minute. Afterwards, it was observed that they separated into two liquid layers. The upper layer is the biodiesel and the lower layer is triglycrol fatty acid. The biodiesel was then separated from its byproduct. The effect of reaction time and temperature ware analysed.

Nano-fluids Preparation

The CuO Nano-particles powder was purchased from Sky Spring Nano materials, Inc., U. S. A, the size of the nano-particles is 10-20nm and the surface was modified with Epoxy Group and it's dispersible as mentioned by the company. Nano-fluids are prepared by two step process. The volume concentration of 0.25%, 0.5%, 0.75% and 1% of powdered nano-particles trans-esterified crude soy bean oil was made respectively. To make the nano-particles more stable and remain more dispersed, each sample was stirred for 3-4 hours using magnetic stirrer, then the samples were taken for analysis.

Viscosity

Viscosity was measured using Brookfield viscometer in a speed range of 50 rpm with spindle size of 2 since a small quantity of the sample is to be measured. The following are the detailed procedure for viscosity measurement;the sample was poured into a beaker, the spindle was fixed and the machine was started, the angular speed was selected on the viscometer and the viscosity was read and recorded the same procedure was repeated for the purified and trans-esterified soy bean oil.

Pour point

Using an improvised method, the experimental procedures of pour point measurement for both crude and purified oil are enumerated below;The cylindrical test tube was filled with the soy bean oil to a specific level mark (5 ml). 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 3 ºC 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 neem oil.

Flash point

The flash point for both crude and purified neem oil was also measured;

A 100 ml conical flask was filled to a specific mark level (10 ml) with crude soy bean 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 reduce 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 5 s. 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 purified and trans-esterified oil.

Pour point, Flash Point and Fire Point Result of Soy Bean

Pour point; 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 found that the pour point of the crude is 10 ℃ while that of the purified oil is 9℃, and Trans – esterified is 8℃, consequently the pour point Trans - esterified (0.25w% CuO) is 5℃. This implies that the Trans - esterified (0.25w% CUO) is 5℃ oil has lower pour point due to the removal of gums in form of phospholipids from the crude oil , its well known that lubricating fluid with low pour point is better especially in a cool whether condition.

Flash point; The flash point test is sensitive in the study of lubricating fluid, because its gives the an indication of the flammability of an oil. They may also be used to provide a quantitative indication of contamination with more flammable materials. the flash point of both the crude, purified, Trans – esterified and Trans - esterified (0.25w% CuO) oils are 167 ℃, 125 ℃, 119 and 134 ℃ respectively. the higher temperatures of the purified oil indicate .its usefulness for lubrication and it may be as a result of removing the flammables materials from the crude oil.

Results and Discussion

It was found that some variables effect the production of biodiesel from vegetable oils, the variables are: (1) free fatty acid and moisture content for based catalyst transesterification, free fatty level should be less then 3% and water free. (2) reaction time and temperature. The citric acid used during the purification was convert the free fatty acid into insoluble salt while the based sodium hydroxide was added to convert the insoluble salts into soluble one finally the heating process was done to evaporates all the water moisture in the samples

During the transesterification, different reaction time and temperature ware used in order to obtain the optimal temperature and time for the biodiesel production. The results are summarized in a table 1 and 2.

Time variation (min)	80	63	60	57
Biodiesel volume (cm^3)	24.5	25.1	24.3	20.7

Table 1: Volume concentration yield for time variation

Temperature variation (ºC)	80	70	55	40	32
Biodiesel volume (cm^3)	27	25.2	22.1	15.2	17.5

Table 2: Volume concentration yield for temperature variation

Based on the above result it was clearly indicated that, the optimal volume of biodiesel was obtained at 63 min and 80 ºC

So also, Figure 1 presents the plots of viscosity versus temperature of soybean oil. As expected, the viscosity decreased with the temperature. It can be noted a pseudoplasticity, followed by a Newtonian behavior at lower temperature. Nevertheless, it is worth to point out that the extent of pseudoplasticity decreases with the increase in temperature. The viscosity of crude samples has higher viscosity than the pure and transesterified biodiesel. High viscosity of crude soybean oil is due to high molecular mass and large chemical structure. The viscosity of pure vegetable oils is 10–15 times greater than the viscosity of diesel. Transesterification of oils produces esters with a viscosity of approximately twice that of diesel. Vegetable oils have high molecular weights in the range 600–900, which are three or more times higher than diesel fuels [15]. Free fatty acids or compounds with hydroxy groups possess significantly higher viscosity [16]. Similar result was obtained by Geng et al., [17].

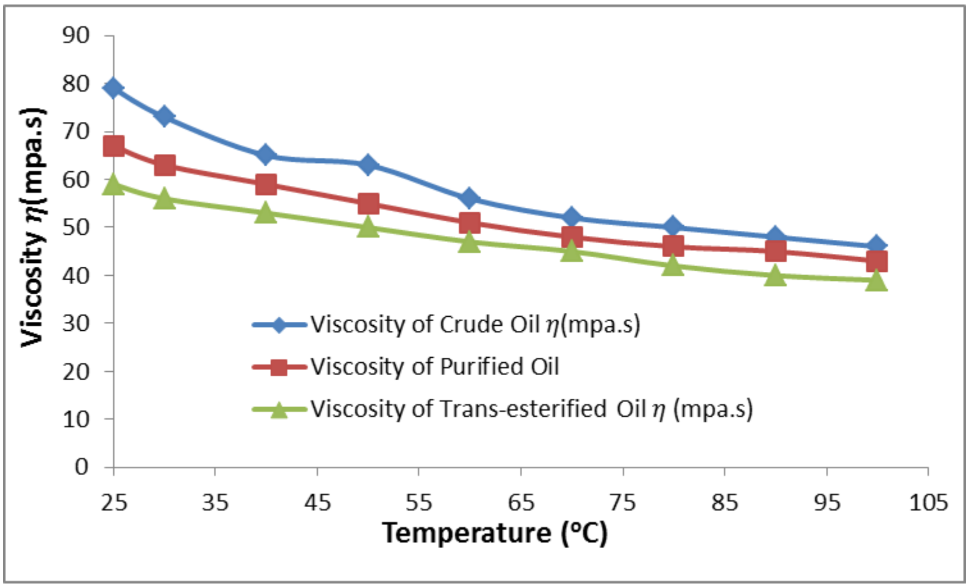

Figure 1: Graph of Viscosity versus Temperature of Soybean Oil

Good heat transfer and fluid flow are characteristics of insulating fluid, as a result of low viscosity which necessitated the additional study of this physical property. To be assured of the effective cooling of transformer cores and windings, capacitors, regulators, pumps, etc. The viscosity of the oil was systematically studied as shown in Figure 2. From the Figure, it is observed that the viscosity is decreasing with increase in temperature. The viscosity of the nanofliud containing 0.25% nano particles oil was observed to be lower than that of transesterified. After adding the nanoparticles above 0.25% the viscosities were found to increases with an increase in temperature. Similarly, the percentage concentration shows a significant effect in the enhancement of the viscosity of the fluid.

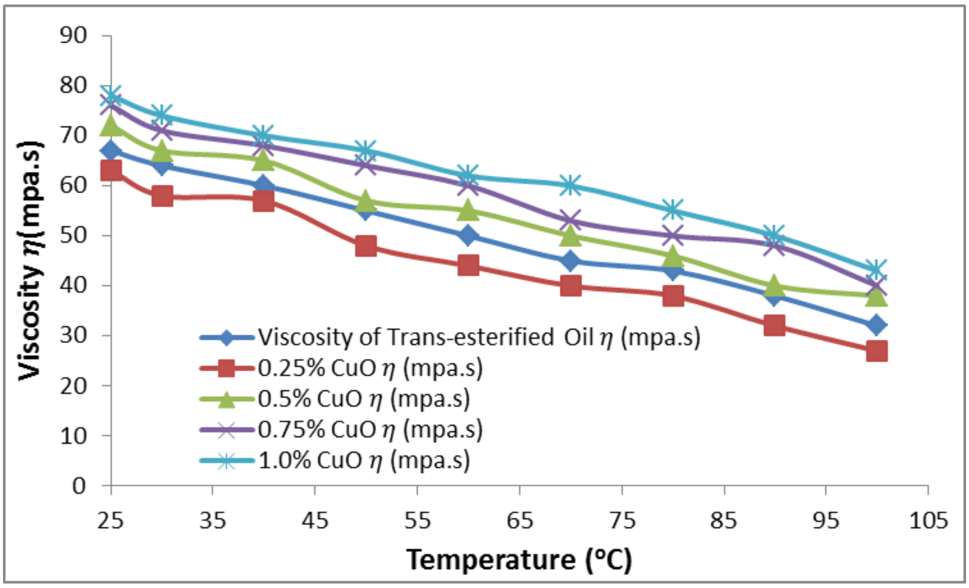

Figure 2: Graph of Viscosity versus Temperature of CuO Nano -Soybean fluid

The FTIR analysis is employed in order to identify the presence of CuOnanoparticles in the based trans-esterified castor oil, the FTIR spectra of all the samples were obtained using SHIMADZU FTIR-8400S Spectrophotometer at National Research Institute For Chemical Technology (NARICT) Zaria. It is an established fact that the fundamental vibrations of solids particle (fingerprint) are localized in the low frequency region (<1200 cm^{-1}) of the midrange (400-4000cm^{-1}) of the infrared spectrum. So the FTIR spectra of purified neem and nano-fluid are presented below.

The FTIR spectra shown in Figures 3- 6, displayed the typical bands that exist in natural esters. The band with a peak at 2924 cm^{-1} describes C-H stretching. The bands with peaks at 1674 cm^{-1} and 1033 cm^{-1} are vibration of C=O and C-O. These are typical bands that describe esters. The bands with peaks at 1442 cm^{-1} and 840 cm^{-1} are due to methylene scissoring and rocking. The addition of CuOnanoparticles is expected to change the FTIR spectra of the fluid. Meanwhile no visible change was observed when the concentration of the nanoparticle in the fluid was 0.25%and 0.5%, a band was observed around 864 cm^{-1}.

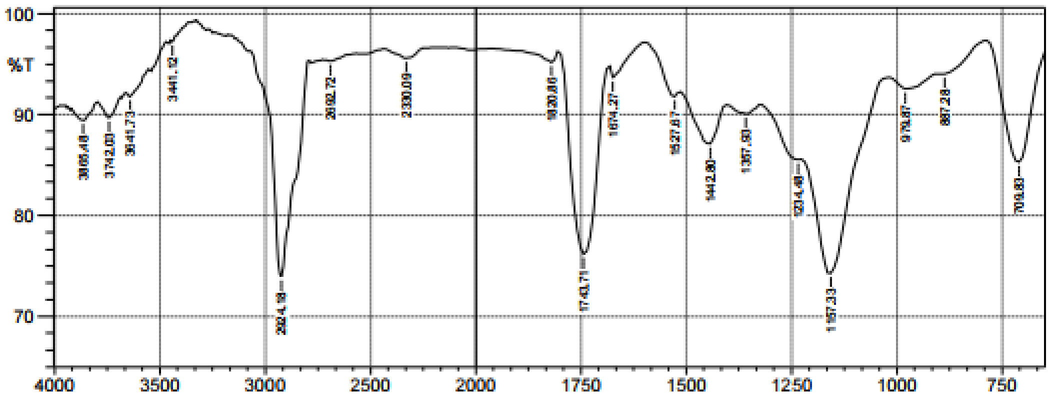

Figure 3: FT-IR Spectra of crude soy bean oil

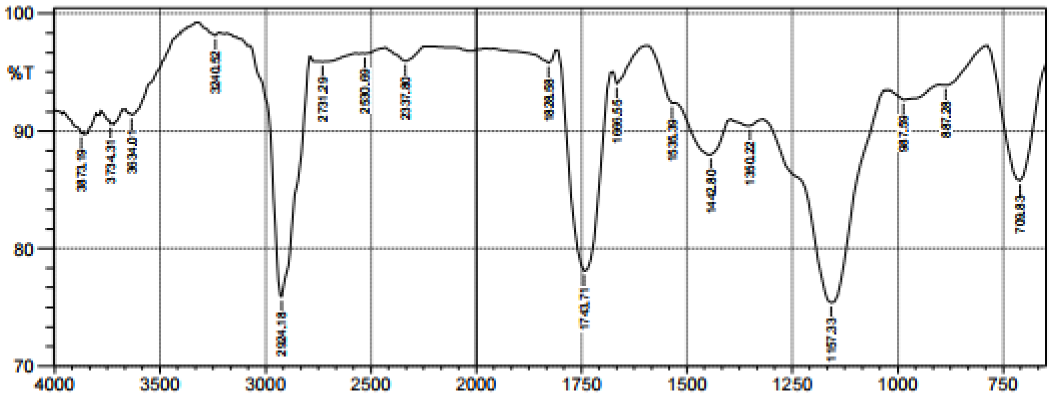

Figure 4: FT-IR Spectra of purified soy bean oil

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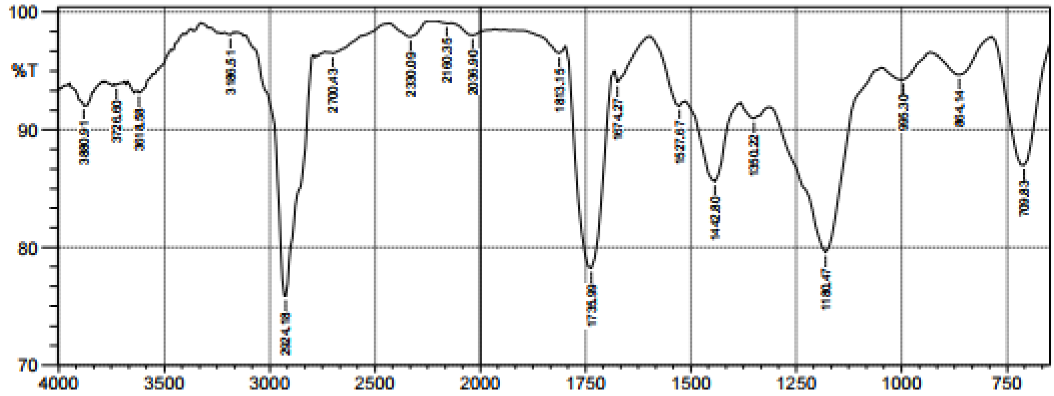

Figure 5: FT-IR Spectra of purified soy bean oil

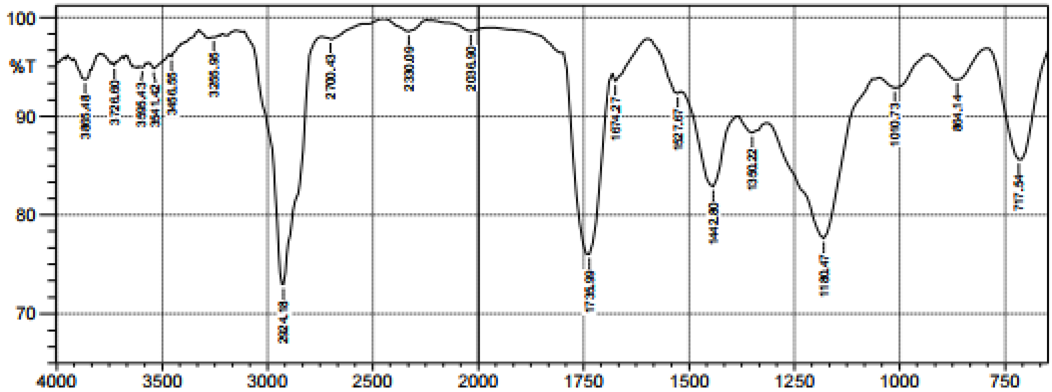

Figure 6: FT-IR Spectra of 0.25% nanoparticles concentration in castor oil

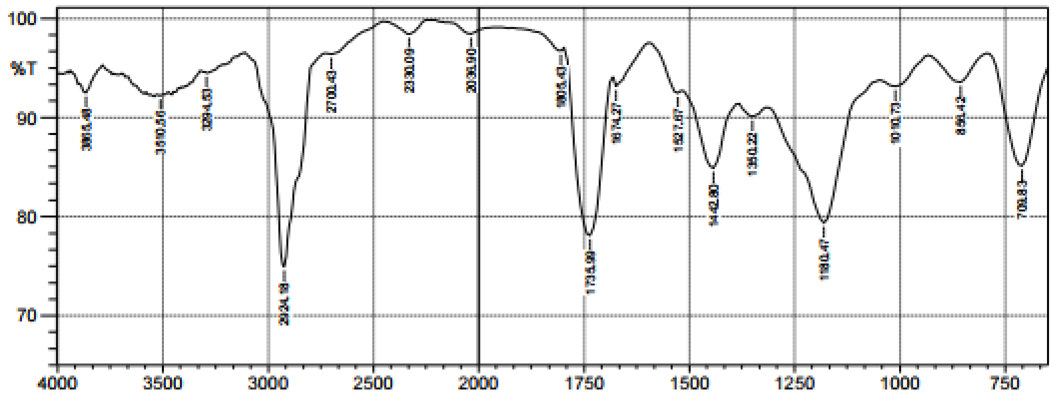

Figure 5: FT-IR Spectra of 0.5% nanoparticles concentration in castor oil

Conclusion

The results shows that the samples has viscosity lower than conventional insulating fluid. Just like most insulating fluids, the dynamics viscosity decrease with increase in temperature. The addition of nanoparticles (0.25w% CuO) resulted in decrease in viscosity. The viscosity change may have contributed to the observed increase in with temperature. The Trans - esterified nanofluid with (0.25w% CuO) appears to be more suitable for insulation as the sample was observed to have lowest pour point of 2°Cand higher flash point of 145°C. 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 nano fliud castor oil with 0.25% oil is potential candidate for the production of lubricating fluid.

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