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EFFECTS OF ZINC OXIDE NANOPARTICLES ON VISCOSITY OF TRANSESTERIFIED NEEM OIL

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ABSTRACT
Efforts have been made worldwide to find alternative fluids for industrial applications. Vegetable oil appears to be a perfect alternative, but using most of the vegetable oil as a feedstock made its use for industrial purposes challenging. The recent trend is to develop coolants/lubricants from non-edible seed oil. This work investigates the effects of zinc oxide nanoparticles on viscosity of transesterified neem oil. The crude neem oil was purified, transesterified and nanoparticles were dispersed in the transesterified oil at concentrations ranging from 0.0% to 1.0% at 0.2% intervals. Fourier Transform Infrared (FTIR) spectra were used to examine the structures of the samples and Scanning Electron Microscopy (SEM) analyses were used to examine the surface morphology of the samples. Viscosity were examined. Among other things, it was found that a small amount of ZnO (0.6%) nanoparticles in the oil could improve the viscosity of the fluid. The nanoliquid with a ZnO concentration of 0.6% appears to have optimal properties.

Keywords: FTIR, Neem Oil, SEM, XRF, ZnO

INTRODUCTION
Fossil fuels are still produced today by subterranean heat and pressure; they are consumed faster than they are produced insufficient quantities or unreasonably priced petroleum fuels worries us deeply, while renewable energies offer a promising alternative because they are clean and environmentally friendly(Zheng *et al.*, 2020). Due to petroleum fuels, pollution and accelerated energy consumption have already impacted landmass aquariums and Earth's biodiversity(Saxena *et al*., 2013). Because petroleum diesel and gasoline are mixtures of hundreds of different chemicals with different hydrocarbon chains, many of these are dangerous and toxic. Carbon monoxide (produced when there is sufficient or incomplete combustion), (Ali *et al*., 2013) nitrogen oxides (produced when combustion occurs at very high temperatures), sulfur oxides (produced when elemental sulfur is present in the fuel) and particulate matter usually produced during combustion, other specific emissions are relevant. Therefore, it is time to look for alternative fuels where we have several alternative fuel sources such as vegetable oils, biogas, biomass and primary alcohols, all of which are inherently renewable. Among these fuels, vegetable oils seem to be of paramount importance as they are renewable and widely used, biodegradable, non-toxic and environmentally friendly. The alternative fuel that is much closer to the diesel engine is biodiesel (Dhar *et al*., 2012; Shaaban and Abd, 2020; Mohadesi *et al*., 2020).
Biodiesel is a clean-burning diesel fuel made from vegetable oils, animals or fat. Its chemical structure is that of fatty acid alkyl esters (FAAE). Biodiesel as a fuel causes far fewer toxic air emissions than fossil diesel. In addition, it burns cleaner and has lower sulphur levels, reducing emissions. Due to its origin from renewable raw materials, it is more likely to compete with petroleum products in the future. However, the International Energy Agency defines renewable energy as energy from natural processes that is constantly replenished and comes in its various forms, directly or indirectly, from the sun or from heat generated deep within the earth. (Van Gerpen *et al*., 2021).
Biodiesel is made from animal fat or the chemical reaction of vegetable oil as an alternative fuel for diesel engines that require alcohol as methanol. A base is required as a catalyst, usually a sodium or potassium hydroxide, after which a chemical compound called methyl ester known as biodiesel is produced. Biodiesel has been reported to be a processed fuel made from a biological source, which is also like petro diesel. (Pandya *et al*., 2022) noted that biodiesel is a plant-based and essential fuel resource. Biodiesel is a Fatty Acid Methyl Ester (FAME) produced by various methods such as micro emulsification. The most widely used and recognized approach to producing biodiesel is the trans-esterification process, which consists of a catalytic reaction between renewable feedstock's such as vegetable oil or animal fats with methanol, producing glycerol as a by-product. (Anwar, 2021). It has been found that the addition of nanoparticles, either as a catalyst or as an additive, improves the trans-esterification process and some of the properties.
Recently, many researchers have turned their attention to fuel formulation techniques to achieve better performance and emissions properties. Newer fuel additives for biodiesel contain nanoparticles as an additive. Biodiesel has emerged as a new promising fuel to achieve maximum performance increase and exhaust emission reduction (Mohadesi *et al*., 2021).
Biodiesel, an alternative diesel fuel, is made from renewable biological sources such as vegetable oils and animal fats. Biodegradable and non-toxic, it has low emission profiles and is therefore environmentally friendly (Wang and Mujumdar, 2007).

Biodiesel has many advantages including the following: it is renewable, safe for use in all 8 conventional diesel engines, offers the same performance and engine life as petroleum based diesel, is non-flammable and non-toxic, and reduces tailpipe emissions, visible smoke and noxious fumes and odours. The use of biodiesel has increased significantly in recent years (Singh *et al.,* 2021).
The use of nanoparticles in neem biodiesel (Nano liquid) is said to improve the physical properties of neem biodiesel. Commercially available diesel oil is a combination of fossil diesel and several additives, added in multiple amounts to perform specific functions. Among other things, there are additives to reduce harmful emissions; improving fluid stability over a broader range of conditions; improve viscosity index by reducing the rate of viscosity change with temperature; Improving ignition by reducing lag time, flash point, etc.; and reduce wear with agents that adsorb onto metal surfaces and provide chemical contact rather than metal-to-metal contact under high load conditions.
Neem is a tree from the Maliaceae family that grows in the southern part of Asia and Africa. Its scientific name is Azadirachta indica. The evergreen tree is large, reaching a height of 12 to 18 meters with a girth of 1.8 to 2.4 meters. The seeds contain 40% oil, which has high potential for biodiesel production. It has a higher molecular weight, viscosity, density and flash point than diesel fuel. Neem oil is generally light to dark brown in colour, bitter, and has a strong odour said to combine the smell of peanut and garlic.(Islas *et al.*, 2020). The use of non-edible neem oil as a biodiesel via trans-esterification can help towards solving some of problems enumerated. It also expected that the addition of nanoparticles into the trans-esterified neem oil to enhance the viscosity of oil.
Therefore the aim of this research is to study the effects of nanoparticles on the viscosity of transesterified neem oil.

MATERIAL AND METHODS

Chemicals

The chemicals and materials used in carrying out the research are; Crude Neem oil, Sodium hydroxide (NaOH), methanol and ZnO.

Equipment

The equipment used in carrying out this study are; Hot plate magnetic stirrer with thermostatically controlled rotary [IKA CMAG HS10], beakers, conical flask, 24 cm filter paper, funnel, thermometer, measuring cylinder, Digital weight balance, Digital stop watch, Sampling bottles, Spatula, Fourier transform infrared spectroscopy (FTIR) machine SHIMADZU FTIR-8400S, X-ray fluoroscopy machine ARL QUANT'X EDXRF Analyser (S/N 9952120) and Scanning Electron Microscopy Machine [PHENOM PROXMVE016477830, Digital viscometer [RVDV-1].

Methodology

The Zinc oxide Nano-particles powder was purchased from sky spring Nanomaterial's, Inc., U.S.A, with Eproxy Group and its dispersible as mentioned by the company. Nano fluids are prepared by two step process. The volume concentration of 0.2%. 0.4%, 0.6%, 0.8% and 1.0% of powdered nanoparticles and purified neem oil was made respectively. To make the nanoparticles more stable and remain more dispersed, each sample was stirred for 3-4 hours using magnetic stirrer, then the sample was taken for analysis.

SEM of ZnO Nanoparticles

The surface morphology of the ZnO was observed using multipurpose Scanning Electron Microscope [SEM] PHENOM PROXMVE016477830, done at Umaru Musa Yar'adua University Katsina, operating at 15kV employing secondary signals. The SEM images presented; was observed at a magnification of times (×520).

XRF of ZnO Nanoparticles

The XRF of the ZnO was carried out at Umaru Musa Yar'adua University Katsina, in order to study the percentage concentration of oxide composed in the sample of nanoparticle used to carry out the research. X-ray Fluorescence (XRF) Spectroscopy involves measuring the intensity of X-rays emitted from a specimen as a function of energy or wavelength. The energies of large intensity lines are characteristics of atoms of the specimen. The intensities of observed lines for a given atom vary as the amount of that atom present in the specimen. Qualitative analysis Involves identifying atoms present in a specimen by associating observed characteristic lines with their atoms. Quantitative analysis involves determining the amount of each atom present in the specimen from the intensity of measured characteristic X-ray lines.

Trans-esterification of Neem Oil with Nanoparticles

60g of the neem oil has been measured in 200ml of conical flask and then heated and stirred to a temperature of 60-70°C 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 70°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 residue oil and the lower layer is purified oil.
Transesterification of neem oil using zinc oxide nanoparticle 60g of the neem oil was measured in 200ml of conical flask and then heated and stirred to a temperature of 60-70°C on a hot magnetic stirrer plate, 0.6g of NaOH and 0.2wt% zinc oxide was 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 70°C, then it was poured into the separating funnel through a glass funnel. The mixture was allowed to cool for about 40 minute. Afterwards, it was observed to separate into two liquid layers. The upper layer is the trans-esterified oil and the lower layer is residue. The same procedure has been applied to 0.4wt%, 0.6wt% 0.8w% and 1.0w% of Zinc oxide.

Infrared Spectral Analysis of Transesterified Neem Oil

The FTIR spectral analysis was done at Umaru Musa Yar'adua 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 disc 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 analogue to digital converter respectively. Finally the signal is transferred to a computer in which Fourier transform is carried out.

RESULTS AND DISCUSSION

SEM of Zinc Oxide

Figure 1 revealed the SEM of the ZnO nanoparticle. It indicates the presence of dispersed particle and cloud structure. This is similar to the result obtained by Kavitha *et al.,* (2019).

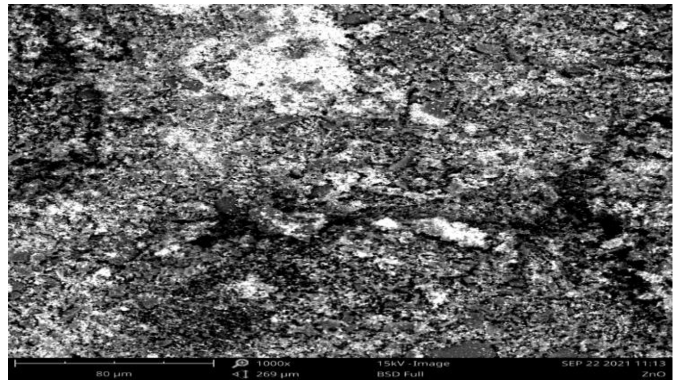

Figure 1: SEM of Zinc oxide at the Magnification1000X

Table 1: Result of X-Ray fluorescence

Oxides	ZnO	Al_2O_3	MgO	TiO_2	P_2O_5	LOI
Percentage (%)	95.44 %	1.25 %	0.7 %	0.52 %	0.45 %	1.64

Table 1 above shows the XRF of the ZnO nanoparticles. It could be seen from the table that ZnO (95.44%) has the highest percentage in the sample. This result shows that Zinc oxide nanoparticle used to carry out this work is of 95.44% purity.

Viscosity of Neem Oil

It can be seen from the Figure 2 below that the viscosity of the crude neem oil decreases with increase in temperature, the viscosity decreases from the value of 142mpa.s at the temperature of 10°C to a value of 73mpa.s at the temperature of 100°C. Also the viscosity of the purified neem oil decreases from 133mpa.s at the temperature of 10°C decrease to 58mpa.s at the temperature of 100°C. Furthermore, the viscosity of the transesterified neem oil decreases from 96mpa.s at the temperature of 10°C to 24mpa.s at the temperature of 100°C. It could be said that crude neem oil has higher values of the viscosity followed by purified oil while the transesterified oil has the least values of viscosity. This is as a result of the fact that as the amount of nanoparticles increases in the fluid, more nanoparticles are driven to the liquid surface and try to get closer to each other. As a result, a strong, cohesive force exerted between the molecules and resulting higher surface tension of nanofluids (Jamo *et al.*2019).

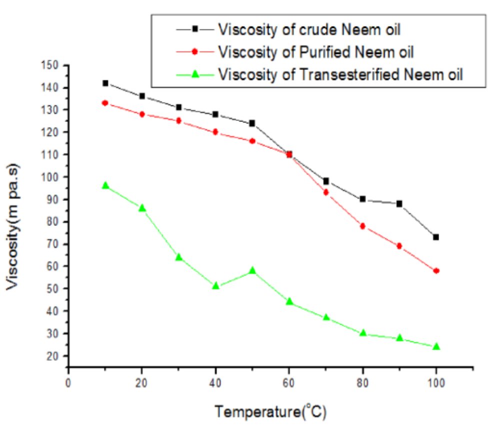

Figure 2: graph of viscosity against Temperature

Figure 3 depicts a graph of nanofluid versus temperature. The nanofluid decreases as the temperature rises. The nanofluid with 0.0% addition has a viscosity of 96mpa.s at the temperature of 10°C and a viscosity of 24mpa.s at the temperature of 100°C. When 0.2% of the nanoparticle is added, the values drop from 94mpa.s at the temperature of 10°C to 26% at the temperature100°C. Furthermore, when 0.4% nanoparticles are added to the nanofluid, the viscosity drops to 92mpa.s at the temperature 10°C and 32mpa.s at the temperature 100°C. When 0.6% of the nanoparticles are added at a temperature of 10°C, the viscosity drops to 28mpa.s at a temperature of 100°C. This is attributed to the reaction between the nanoparticles and the fatty acid. However, when 0.8% of the nanoparticles were added to the nanofluid, the viscosity values increased. This is as a result of the reduction in the fatty acid.

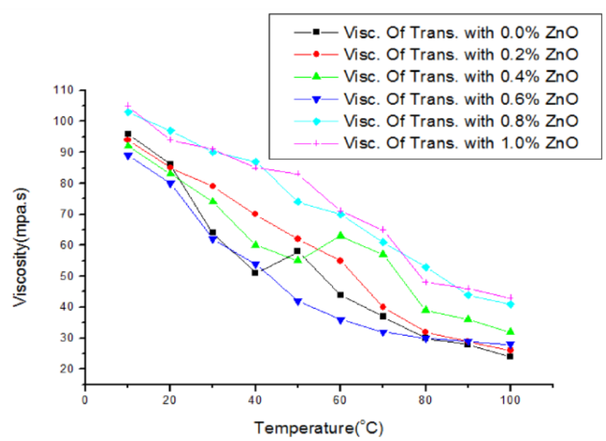

Figure 3: Graph of Viscosity of Nano fluid against Temperature

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FT-IR Spectra

C-H stretching is shown in Figure 4 by the band with a peak at 2854 cm^{-1}. The vibrations of C=O and C-O are what cause the bands with peaks at 1674 cm^{-1} and 1033 cm^{-1}. These are common bands used to describe esters. The shearing and shaking of methylene is what causes the peak bands at 1238 cm^{-1} and 840 cm^{-1}. The liquid's FTIR spectra are anticipated to alter after the addition of ZnO nanoparticles. Around 840 cm^{-1} and 848 cm^{-1}, a band that was detected at 0.6% is most likely an enclosed ester C-O band. According to the amount of light absorbed by specific molecules found in purified neem oil, Figure 4 shows the FTIR spectrum plotted for transmittance against the wave number ($cm^{(-1)}$). The ester is at peaks at 724.8811, 1123.7717, 1164.64986, 1100.77686, 1238.8188, 1380.88661, 1465.81290 and 1748.53848. These show that there has been an improvement over neem oil that has been refined.

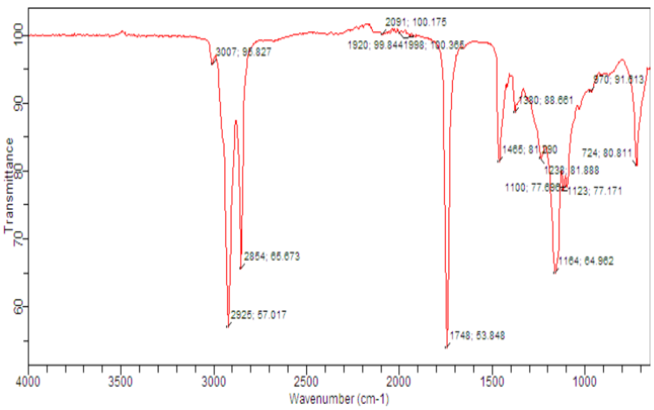

Figure 4. FT-IR Spectra of Transesterified Neem Oil

CONCLUSIONS

The chemical properties of ZnO nanoparticles were studied by XRF. The XRF result shows that ZnO accounts for 95% of the sample. The morphological properties of the ZnO nanoparticle were also studied by SEM. The result showed that ZnO is dispersed particle with cloudy structure. The FTIR result shows that ester was produced between 1674 cm^{-1} and 1033 cm^{-1} peak. The viscosity of the neem oil was analysed via viscosity for the crude, purified and transesterified neem oil. The result shows that the viscosity of the crude oil is high. It has also been found that the viscosity of the neem oil decreases after purification and subsequently after the transesterification. It was also found that the viscosity of the transesterified oil decreases as the concentration of the nanoparticles increases, with addition of 0.6% nanoparticles the optimal value of viscosity was achieved with a value of 96mpa.s. The viscosity of the transesterified oil is at its best when 0.6% of nanoparticles are added. It could be concluded that the addition of 0.6% ZnO nanoparticles has a positive influence on the viscosity of transesterified neem oil.

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