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Development and Characterization of a Novel Bio-Based Brake Fluid Using Roselle Oil with Copper Oxide Nano Additive

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Abstract

Conventional brake fluids, which are typically derived from petroleum-based sources, pose environmental hazards due to their non-biodegradable nature. A bio-based brake fluid was produced from roselle oil with Nano copper oxide as an additive. The tribological, and Physico-chemical properties of the blended brake fluid were evaluated. From the kinematic viscosity analysis, it was observed that there was a linear increase in the viscosity with an increase in the nanoparticle with a 43.3% increase in viscosity due to the inclusion of 0.3 wt.% of CuO nanoparticle. The increment is attributed to physical barriers created by the nanoparticle that generate more internal friction to the flow of oil molecules. The addition of 0.1 wt.% and 0.2 wt.% of CuO nanoparticles increases the viscosity index from 373.9 to 381.3, and 374.6 respectively. The flash point of the blended roselle oil was observed to be 199 ℃. The inclusion of 0.1 wt.%, 0.2 wt.% and 0.3 wt.% CuO nanoparticles at 0.1% rate, increase the flash point to 202 ℃, 209 ℃, and 210 ℃ respectively. This indicates that adding the nanoparticles improves the surface area-to-volume ratio, enabling the fluid to absorb heat more efficiently. The coefficient of friction (CoF) of the pure roselle oil was 0.117 while the CuO nanoparticle included roselle oil was observed to be 0.067, 0.104, and 0.057 at CuO nanoparticle quantity of 0.1 wt.%, 0.2 wt. and 0.3 wt.% respectively. The wear rate of aluminum with the oil as a brake fluid was 0.3729 $mm^3n^{-1}m^{-1}$, 0.4316 $mm^3n^{-1}m^{-1}$, and 0.3592 $mm^3n^{-1}m^{-1}$ at a nanoparticle reinforcement of 0.1 wt.%, 0.2 wt.%, and 0.3 wt.% respectively. The reduction in the CoF and wear rate indicated that the CuO nanoparticles acted as solid lubricants themselves, forming a protective layer between the sliding surfaces. The production of bio-based brake fluid offers novelty in this research area and this achievement represents a significant step towards developing eco-friendly and sustainable alternatives to conventional petroleum-based brake fluids.

Keywords: Bio-Based Brake Fluid, Tribological Properties, Physico-chemical Properties, Roselle Oil, Copper Oxide, Nanoparticles.

Introduction

Motor vehicle brake fluids are used to transfer energy to the actuating mechanism in the hydraulic drive of the brake assembly of a car and to remove heat from the brake discs (Garcoa-Leon et al., 2023). The main requirements for brake fluids are high operation temperatures, good low-temperature and viscosity-temperature properties, physical and chemical stability, protection of metals from corrosion, inactivity with respect to mechanical rubber articles, and lubricating effect (Astakhov, 2022). Synthetic brake fluid is a mixture of diethylene glycol and di-, tri-, along with tetraethylene glycol monoalkyl ethers. They contain inhibitors to prevent the corrosion of metallic brake components and to reduce oxidation at increased temperatures. To increase the boiling point, tris (methyl glycol) borates are introduced in brake fluids (Chen et al., 2023)

One of the basic elements of machine hydraulic systems is the working fluid. The main task of the working fluid is to transfer energy between the driving part and the driving part of a machine's drive system. In modern hydraulic systems, the working fluid also performs different important functions (Olszak et al., 2020). Among these functions are the lubrication of moving parts, heat dissipation, the discharge of solid contaminants from the system, sealing the system, and inhibiting corrosion. The most important and basic physical attributes of working fluids are kinematic viscosity, density, boiling point, freezing point, and specific heat (Ma et al., 2020). Working fluids performing in hydraulic systems should also have anti-wear and anti-corrosive properties, as well as resistance to oxidation and thermal degradation. Kinematic viscosity is a basic physical property of working fluid; the amount of flow losses within the hydrodynamic working space depends on this property (Olszak et al., 2020). To minimize the flow losses, it is advised to use working fluids with a very low viscosity. The viscosity coefficient value depends on the temperature. A low boiling point contributes to the occurrences of cavitation phenomena. A significant increase in the working temperature of the hydrodynamic clutch can cause a decrease in the quality of the working fluid as a result of its oxidation and thermal destruction. This is why the working fluid must have anti-oxidative properties which will ensure the required durability (Wong et al., 2022). The working fluid of hydrodynamic clutches should also have good anti-foaming and anti-corrosive properties, and it should not decompose. Foaming in the working space poses a risk of leaks of the hydraulic fluid through the sealing. In the case of hydraulic working fluids which are produced from oils obtained from crude oil processing, their producers make efforts to develop products based on raw materials with lower negative impacts on the environment (Olszak et al., 2020). Using water in hydrokinetic systems is more advantageous than using oils for a few reasons. The viscosity of water is significantly lower than the viscosity of oils, it does not depend on temperature, and the thermal conductivity of water is five times higher than the thermal conductivity of oil. Water poses no fire or explosion risk. The main disadvantages of limiting the usage of water in hydraulic systems are a narrow range of working temperatures, causing the corrosion of steel (which usually makes up the elements of hydraulic systems,) and low lubricity (Manring and Fales, 2019). However, the lower limits of the operating temperature can be shifted by using additives, lowering the freezing point of water. Corrosion can be eliminated by using stainless steel or anti-corrosion coatings for regular steel.

The newest type of working fluid used in hydraulic devices, including hydrodynamic clutches, is the two-component fluid, mainly electrorheological fluids (Olszak et al., 2020). The solid phase of electrorheological fluids consists of organic or inorganic particles, easily polarized by an electric field. The liquid phase consists of liquids that do not conduct electricity, such as mineral oils, plant oils, liquid paraffins, and kerosene. Additives used to enhance the electrorheological effect are as follows: water, acids, inorganic salts, alcohol, and esters. To reduce the non-ecological contents in electrorheological fluids, the solid phase consists of natural products, such as starch, cellulose, resins, and rubber. A two-component electrorheological fluid that is often used is a fluid made of starch and silicon oil. Apart from their food applications, oils, and vegetable fats, as well as substances obtained from them, are important raw materials used in several industry sectors. It is used, among other things, in cosmetics, pharmaceuticals, biofuels, paints, adhesives, coatings, surfactants, solvents, and adjuvants (Srivastava et al., 2022). Products made using plant oils are friendly to the environment and human health, and they are biodegradable in most cases (Salih and Salimon, 2021) The use of plant oils as brake fluid has a long history, dating back to olive oil usage in ancient times.

Hence, this research aims to develop and characterize a bio-based brake fluid using roselle oil with Nano copper oxide additive. At first, the copper oxide nanoparticles were characterized using the X-ray diffraction (XRD) and TEM techniques. Then, the bio-based brake fluid was developed from Roselle oil and copper oxide nanoparticles. Finally, the physio-chemical and tribological properties of the developed brake fluid were investigated.

Materials and Methods

Materials

The materials required to carry out this research include Roselle bio-based oil (sourced from a farm center in Kano State, Nigeria), Copper acetate, Sodium hydroxide, Oleic acid, Ethanol, Phosphoric acid, (purchased from local stores in Zaria, Nigeria) and Aluminium-Silica Alloy.

Determination of Free Fatty Acid

1g of oil sample was weighed in a conical flask 2.5ml of propan-2-ol was added to the sample to dissolve the oil. Then two drops of phenolphthalein were added to the mixture. Two more solutions were prepared in a conical flask using the same procedure as above, making three solutions. 0.1M of KOH was poured into the burette and titrated against the mixture of the biodiesel oil samples. After a colour charge was observed from yellow to pink, the initial and final readings were recorded, and then the total average titer value was recorded (Animasaun et al., 2021). The acid value was calculated using Equations 1 and 2:

$$Acid\ value\ = \frac{molar\ mass\ of\ KOH \times titre\ value}{mass\ of\ oil} \qquad 1$$

$$FFA\ = \frac{Acid\ value}{2} \qquad 2$$

Esterification of Roselle oil

In an experiment, 900g of roselle oil was measured and weighed in a conical flask using a weighing balance. The conical flask containing the oil sample was placed on a magnetic stirrer and heated to a temperature of 60 °C. Following the initiation of agitation and heating, 143.8g of methanol and 3.2g of sulfuric acid were calculated using Equations 3 and 4. Then both solutions were added to the flask.

Amount of methane = 2.25×%FFA×mass of oil 3

Amount of sulphuric acid =0.05×%FFA×mass of oil 4

Where: Mass of oil =1g; Molarity of KOH =0.1m; Mass of KOH =56.1g

After 1 hour of continuous agitation at a temperature of 60 ℃, the mixture was transferred into a separation funnel and left to settle. Over time, methanol, water, and H2SO4 separated and rose to the upper portion of the separating funnel, while the denser oil remained at the bottom. This allowed for easy separation. The oil was then carefully drained into a clean conical flask.

The purpose of the pretreatment esterification process was to reduce the percentage of Free Fatty Acids (%FFA) in the oil to a level of less than or equal to 0.5%. This esterification process likely involved the reaction of the roselle oil with methanol and sulfuric acid, leading to the formation of esters and water-soluble components that could be separated from the oil. This step is crucial in preparing the oil for further processing or use, as lower levels of free fatty acids are generally desired for various applications (Edwin et al., 2023).

Transesterification Process of the Esterified oil

Following the successful esterification reaction where the percentage of Free Fatty Acids (%FFA) was reduced to less than 0.5%, a subsequent transesterification step was undertaken. This process aimed to convert the esterified oil into biodiesel (Hani et al., 2021). 15% of methanol and 1% of catalyst (NaOH) were measured and weighed out. The calculated amount of NaOH was dissolved into the measured methanol. The methanol-NaOH solution was combined with 900g of the esterified oil. The resulting mixture was placed on a magnetic stirrer set at 60°C for both agitation and heating. This condition was maintained for 1 hour, facilitating the transesterification process. After the reaction, the solution was transferred to a separating funnel. It was allowed to stand for a while, during which the denser glycerol settled at the bottom due to its higher density than the biodiesel. The denser glycerol layer was drained from the separating funnel. Following the glycerol removal, the lighter biodiesel layer was carefully drained into a clean conical flask. This comprehensive procedure ensured the successful conversion of the esterified oil into biodiesel, with efficient separation of the resulting products.

Epoxidation

Based on literature data and preliminary investigations, the initial conditions for the epoxidation process were established. The ratios of the reactants were determined as follows: 1:3:0.5 for biodiesel, hydrogen peroxide, and acetic acid respectively. A 2 wt.% of sulfuric acid catalyst, relative to the epoxidation mixture, was employed (Zhang et al., 2022). 9g of sulfuric acid (H_2SO_4) was weighed out, and the calculated amount was dissolved into the measured quantity of acetic acid. The solution containing the dissolved sulfuric acid was combined with 200g of roselle biodiesel and 50g of acetic acid. The resulting mixture was thoroughly mixed. The mixture was placed on a magnetic stirrer set at 40% agitation. Subsequently, 300g of hydrogen peroxide (H_2O_2) was added to the solution in drops. This dropwise addition allowed for the maintenance of a constant reaction temperature. The solution was heated and kept at a constant temperature while being stirred for 2.5 hours. This duration allowed the reaction to proceed effectively. The entire procedure was repeated for subsequent batches. This well-defined process, guided by both existing literature and preliminary experimentation, enabled the controlled and consistent epoxidation of the roselle biodiesel. The specified ratios of reactants and the addition of a catalyst ensured an effective and controlled reaction, leading to the desired product (Toe et al., 2021).

Synthesis of Copper Oxide Nanoparticles

The synthesis of CuO nanoparticles and their subsequent surface modification was conducted through a multi-step procedure. Aqueous solution containing copper acetate (0.03 mol) was created in a round bottom flask. 1.2 ml of glacial acetic acid was introduced to the copper acetate solution. The mixture was heated to 100°C with continuous stirring. To the heated solution, 0.5 g of NaOH was added until the pH reached 7. This led to the formation of a significant amount of black precipitate. The black precipitate was separated by centrifugation and then washed four times using deionized water. The washed precipitate was dried in an open-air environment for 24 hours. The surface of the CuO nanoparticles was modified to ensure effective blending. Oleic acid was adsorbed chemically as a carboxylate onto the nanoparticles' surface to improve their interfacial properties within the blends. Initially, 6g of nanoparticles were dispersed into 400 ml of preheated ethanol (at 60°C). Stirring was conducted for 15 minutes to achieve uniform dispersion. Following dispersion, 0.27 ml of oleic acid was introduced, and the solution was stirred for 2 hours. The solution was then centrifuged to separate the ethanol-oleic acid mixture. The resulting nanoparticle material was dried in an oven at 75 °C to remove excess surfactant (Adekunle and Oparanti, 2023). This entire procedure, described in (Patel et al., 2022), involved a series of precise steps to synthesize and modify the CuO nanoparticles for subsequent blending and application as part of the Nano-brake fluid formulation.

Nano Brake Fluid Formulation

The formulation of the Nano-brake fluid involved creating four distinct samples, each containing treated CuO nanoparticles (NP) dispersed within bio-based oil. These nanoparticles were added at different weight percentages, ranging from 0.1 wt.% to 0.3 wt.%, with intervals of 0.1 wt. % (Reddy et al., 2023). The resulting samples are labelled as presented in Table 1. To achieve the dispersion of nanoparticles within the base oil for these three samples, an ultra-sonication process was employed. The ultra-sonication procedure lasted for a total of 3 hours, with intervals of 90 minutes throughout the process. The sonication was carried out at a frequency of 59 Hz, utilizing 100% power, and maintaining a temperature of 75°C (Putnik et al., 2020). The primary goal of this step was to ensure the thorough mixing and even distribution of nanoparticles within the base oil. Following the sonication step, the resulting blends were subjected to vacuum drying for a specific duration. This drying process aimed to eliminate the effects of gas bubbles and moisture generated during the sonication process (Kutlu et al., 2022). By conducting this thorough procedure, the objective was to achieve uniform and stable formulations of the Nano-brake fluid samples for subsequent testing and evaluation (Patel et al., 2022).

Table 1
Sample Description

Sample Code	Description
Base Oil	Roselle oil
NBF 1	Ester + 0.1% CuONP
NBF 2	Ester + 02%CuONP
NBF 3	Ester + 0.3%CuONP

TEM and XRD characterization
To gain insights into the morphology and phase of the Copper oxide (CuO) nanoparticles, both before and after surface modification, a combination of TEM and XRD techniques was employed for thorough investigation (Ansari et al., 2022). Transmission Electron Microscopy (TEM) was utilized to analyse the morphology, while X-ray Diffraction (XRD) analysis was employed to identify the phase present. These analyses were conducted to provide a deeper understanding of the nanoparticles' characteristics.

The crystallite sizes of the nanoparticle samples were determined using Equation 6 through the application of Scherrer's equation, as outlined in the work by (Reddy et al., 2021). This equation is commonly used to estimate crystallite size based on the broadening of X-ray diffraction peaks, providing valuable information about the structural properties of the nanoparticles. These comprehensive analyses allow for a comprehensive exploration of the nanoparticles' physical and chemical attributes, aiding in the elucidation of their behaviour and potential applications (Srivastava et al., 2023).

$$D(2\theta) = \frac{K\lambda}{\beta cos\theta} \qquad 5$$

Where K (0.89) is the shape factor, D is the crystal size $\boldsymbol{\lambda}$ is the wavelength of X-ray radiation θ is the diffraction angle and β is the full-width half maximum height (FWHM).

Determination of Physicochemical Properties
The assessment of physicochemical properties of brake fluid oils stands as a crucial means of evaluating the tribological performance of a specific brake fluid, as highlighted by (Patel et al., 2022). In the case of the Nano brake fluid under consideration, the determination of its physicochemical properties was carried out using the following procedures:

Kinematics Viscosity
The viscosities of the Nano brake fluid samples were determined using a viscometer bath. Each sample was immersed in a constant temperature bath set at 40 °C. The viscosity measurement was conducted by the ASTM D445 method and utilized a calibrated viscometer (Huang D. et al., 2020). The calibration constants for the viscometer were specified as 0.08867 and 0.74020. Viscosity is a critical property of brake fluid that reflects their flow characteristics and resistance to shear forces under different conditions (De, 2023). Measuring viscosity at distinct temperatures provides valuable insights into how a brake fluid's viscosity changes

with temperature variations, which can be crucial for understanding its performance across different operational environments (Agrawal and Khairnar, 2022).

Viscosity Index

The viscosity index (VI) was determined using Equation 7 as prescribed by the ASTM D2270 standard. This index provides information about how the viscosity of a brake fluid changes with temperature variations. The specific formula used for calculating the viscosity index is as per the ASTM D2270 standard (Faujdar et al., 2020). likely elaborates on the significance and implications of this viscosity index in the context of brake fluid.

$$VI = \left(\frac{L-U}{L-H}\right) \times 100 \quad 6$$

Where VI: Viscosity Index; L: Viscosity value at 40 ^{O}C (cSt), U: Viscosity value at 100 ^{O}C (cSt), H: Viscosity value at 0 ^{O}C (cSt)

Boiling Point

A reflux setup can be effectively employed to determine the boiling point of a compound. Refluxing involves a dynamic process where a liquid is actively boiling and subsequently condensing, with the condensed liquid returning to the original flask. This technique shares similarities with a distillation setup, the key distinction being the vertical positioning of the condenser (Liebminger et al., 2021). To determine the boiling point of the developed fluid, the reflux method was utilized. Roughly 5 mL of the rossel oil sample was introduced into a medium-sized test tube (18 x 150 mm), in which a thermometer was securely clamped to ensure it didn't come into contact with the glass sides. The entire setup was then cautiously heated using a sand bath, allowing controlled reflux to occur and preventing vapor from escaping the tube (Arenales et al., 2020). During the reflux process, the temperature will eventually reach a stable state after some time.

The highest temperature recorded at this point corresponds to the boiling point of the compound. However, it's important to note that using this method to measure boiling points may introduce significant errors when the boiling point is very low <70°C or very high >1500°C (Qiao et al., 2022). This is because extremely low boiling compounds tend to evaporate too readily, while highly boiling compounds tend to cool too quickly. Despite these limitations, the reflux method remains a valuable technique for determining the boiling points of compounds within a reasonable range, providing insights into their thermal properties (Huang et al., 2020).

Pour Point

The pour point determination was conducted following the ASTM D97 method, as outlined by (Hundie et al., 2022). For each sample, a quantity of 3.0 g was placed within a test tube, with a thermometer inserted through a cork that covered the tube's mouth. The test tube was subsequently refrigerated, and observations were made at 10-minute intervals for 2 hours. During this observation period, the test tube was periodically removed and tilted to assess whether the oil within exhibited any flow or movement. The point at which the oil no longer exhibited any movement when the test tube was held horizontally for several seconds indicated solidification (Mayilvelnathan and Arasu, 2020). The pour point was determined as the temperature at which the oil's solidification occurred, taken at a point 50°C above the solidification point, as per the approach adopted by (Tasneem et al., 2022). This pour point determination provides valuable information about the lowest temperature at which the lubricant remains fluid and able to flow under specific conditions.

Flash Point

The flash point of the Nano brake fluid samples was assessed by the ASTM D93 standard, utilizing the Pensky-Martens open cup tester. The determination process involved utilizing an automated Pensky-Martens open cup apparatus within a temperature range of 150°C to 250°C. To determine the flash point of the Nano brake fluid, a sample of the oil was heated in a round container, and a flame was passed over the liquid's surface (Jamo et al., 2019). The flash point determination was established by noting the temperature at which vapor from the heated oil ignited upon contact with the flame. This ignition resulted in a noticeable and easily detectable flash. The method employed mirrors the approach described by (Kuznetsov et al., 2022). This flash point determination method aids in understanding the temperature range at which the lubricant is susceptible to producing ignitable vapor, thus offering insights into its potential behavior under different operating conditions (Prasannakumar et al., 2023).

Tribological Test

Friction and wear behavior testing of the Nano brake fluid was carried out using a pin-on-disc machine, following the guidelines of the ASTM G99 standard. The load was manually applied as per the specified requirements, and the sliding speed was kept constant for all tested conditions. In this analysis, stainless steel was employed as the pin, while the disc was made from aluminum silica alloy. The parameters used for the tests are detailed in Table 2, following the description provided by (Bagheri, 2020). To replicate a low-speed, low-load tribo-contact scenario, the track speed was intentionally set to a relatively lower value of 10 cm/s. At this stage, the amount of available brake fluid oil at the sliding interface is relatively limited. Brake fluid mechanisms in this contact rely on the formation of extremely thin brake fluid films and/or tribofilms generated due to interactions between the brake fluid and its additives with the component surfaces (Tang et al., 2021).

As a consequence, the ability of the Nano additive within the brake fluid becomes crucial in sustaining the operational load under these conditions. To allow for wear stabilization and the potential development of a tribofilm by the applied Nano CuO brake oil samples, an 8.15-minute test duration was chosen. This time frame also facilitates significant wear rate analysis (Freschi et al., 2022).

Table 2
Test Parameters

Test Conditions	
Parameters	Value
Test duration, min	8.15
Normal load, N	8
Linear speed, cm/s	10
Temperature of brake fluid	Room temperature (about 20)
Brake fluid	The base oil and Nano-brake fluid samples
NPs concentration	0.1, 0.2, and 0.3. %

Ball (upper test piece)	Stainless steel
Disc (lower test piece)	Aluminum-Silica Alloy

Friction tests were conducted to determine the coefficient of friction (COF) between the stainless-steel ball and the mild steel disc samples. A comprehensive set of six experimental conditions was examined, including dry conditions, brake fluid oil, and four distinct formulations of Nano brake fluid samples (Patel et al., 2022). The calculation of the wear rate was done using Equation 8.

$$w = \frac{wear\ volume, m^3}{sliding\ distance, m} \quad 7$$

Thermal Analysis

The base oil's oxidative stability and the best sample's physicochemical and tribological properties were assessed using a Perkin-Elmer differential scanning calorimeter DSC-7. The equipment underwent calibration using pure indium, and the baseline was established using an empty open aluminum pan (Fatahi et al., 2023). Into open aluminum pans, oil samples weighing 5.0 ± 0.5 mg were placed and positioned within the equipment's sample chamber. The isothermal temperature was programmed across four distinct levels (110, 120, 130, and 140 °C), with purified oxygen (99.8%) being circulated through the sample's enclosure at a rate of 50 ml/min (Skornia, 2020).

Result and Discussions

X-ray Diffraction of Cuo Nano Particles

Figure 1 shows the XRD pattern of Copper Oxide nanoparticles. Copper oxide (CuO) nanoparticles had a crystalline structure with several sharp peaks similar to the study reported by Mobarak et al. (2022). The positions of these peaks are determined by the interatomic distances and angles within the lattice. The major peaks in the XRD pattern of copper oxide nanoparticles are observed at 36o and 38o corresponding to the (002) and (110) miller plane which is typically the most intense as presented in Figure 4.1 Also, (111) plane was observed at 48o and the peak at 61o is attributed to (202) plane. The crystal structure and particle size of copper oxide nanoparticles can have significant influences on their applicability as reinforcement in roselle oil for the production of hydraulic oil as reported by Raj et al. (2021). Using Scherrer's formula, the average crystallite size of the copper oxide nanoparticles is 9.22 nm which shows the CuO used in the study falls within the Nano range.

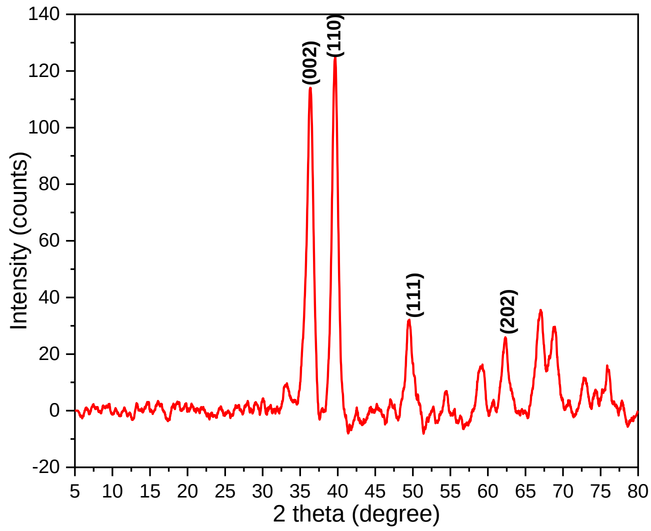

Figure 1: XRD pattern of CuO nanoparticles

TEM Analysis

The TEM results provided valuable information about the nanoscale features and distribution of the nanoparticles within the oil matrix. The TEM images as observed in Figure 2 (a, b, and c) revealed the spatial arrangement and dispersion of the CuO nanoparticles. The images show the nanoparticles as dark spots against a lighter background, indicating their electron density contrast as also observed by Stewart et al. (2021). A uniform dispersion of the nanoparticles throughout the epoxidized roselle oil matrix as shown in Figure 2 suggests successful incorporation and effective dispersion. In addition to dispersion, TEM allowed for the analysis of the size and shape of the nanoparticles. Observation as in Figure 2 (A and B) shows the spherical nature of the nanoparticles. Furthermore, the shape and morphology of the nanoparticles were assessed, providing insights into their structure as observed by Jhaveri et al. (2021). Also, even as the particles were more spherical, the size ranges as shown in Figure 2(c) show the majority of the particles were below 20nm in diameter. In the case of CuO nanoparticles in epoxidized roselle oil, a predominant spherical shape is observed, indicating successful synthesis and integration. Also, the absence of clustering or agglomeration of the particles when dispersed in the oil shows the effective preparation of the Nano particle-reinforced bio-based braking fluid which is essential for achieving desired properties and performance enhancements as reported by Kamarulzaman et al. (2023). Controlled particle size and narrow size distribution are crucial for maintaining consistent material properties. Additionally, the spherical shape of the nanoparticles enhances the compatibility and interfacial interactions within the composite, further influencing the material's characteristics.

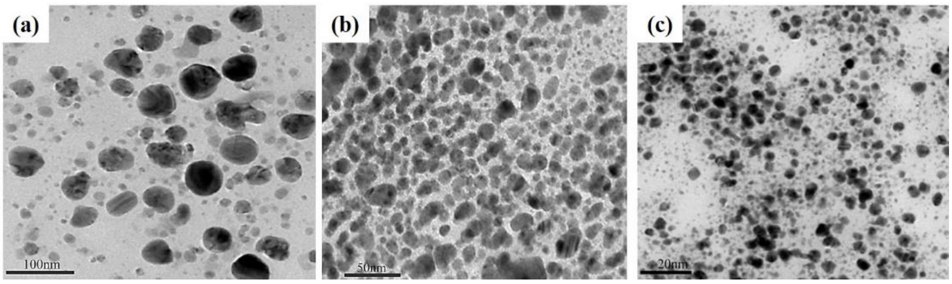

Figure 2: TEM of Nano Particles at (a) 100nm(b) 50nm (c) 20nm

Physiochemical Properties

The physicochemical properties of the various fluid oil blends in comparison with ISO and pure roselle base oil are demonstrated in Figures 3 to 4.

Kinematic Viscosity (Kv) @ 40℃

Figure 3 shows the kinematic viscosity of the blended brake fluid at 40 °C which presents the viscous behavior of the different blended oil. It shows that there was an increase in the viscosity of the oil by 0.5 with the inclusion of CuO nanoparticles in the virgin esterified oil at 40 °C. Also, it was observed that there was a linear increase in the viscosity with an increase in the nanoparticle. Generally, there was a 43.3% increase in the viscosity of the blended roselle with the inclusion of CuO nanoparticles up to 0.3%. There was a 13.6% rise in the viscosity of the roselle oil with a 0.1% inclusion of CuO at 100 °C. A further increase shows a linear rise in the kinematic viscosity as discussed by Santhosh et al. (2021). The increase in viscosity observed in this study can be attributed to the presence of CuO nanoparticles in the blended roselle oil. The nanoparticles act as physical barriers that restrict the movement of the oil molecules, increasing viscosity as observed by (Oungkanitanon et al., 2021). At low concentrations of CuO nanoparticles, the increase in viscosity is relatively small. However, as the concentration of nanoparticles increases, the physical barrier the nanoparticles create becomes more pronounced, resulting in a linear increase in viscosity. The effect of temperature on viscosity is also significant. As the temperature increases, the viscosity of the oil decreases because the oil molecules have more kinetic energy and can move more freely as reported by (Zhao et al., 2021). However, the presence of CuO nanoparticles can counteract this effect by increasing the physical barriers that restrict the movement of the oil molecules. Overall, the results of this study suggest that the inclusion of CuO nanoparticles in esterified roselle oil can significantly increase its viscosity, particularly at high temperatures.

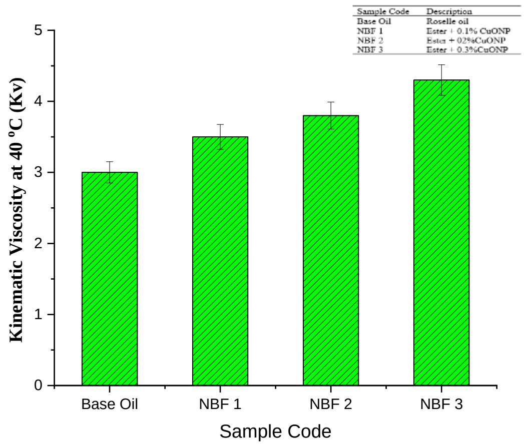

Figure 3: Kinematic Viscosity of the Developed Oil at 40 °C

Viscosity Index

The experimental results suggest that the addition of CuO nanoparticles to blended roselle oil affects its viscosity index (VI) as illustrated in Figure 4, which is a measure of the oil's ability to maintain its viscosity over a range of temperatures. In general, higher VI values indicate that the oil will have less variation in viscosity as the temperature changes as observed by Kots et al. (2021). At 0.1 wt.% concentration of CuO nanoparticles, the VI of blended roselle oil increased from 373.9 to 381.3, which indicates that the nanoparticles improved the ability of the oil to maintain its viscosity over a range of temperatures. This improvement in VI could be due to several factors, such as the nanoparticles' ability to act as a heat sink, reduce the oil's thermal degradation, or enhance its lubrication properties as observed by Ali and Xianjun (2020). Additionally, the nanoparticles may have improved the oil's resistance to shearing forces, which can cause the viscosity to decrease. At a 0.2 wt.% concentration of CuO nanoparticles, the VI decreased to 374.6. This decrease in VI suggests that the nanoparticles may have started to form clusters or aggregates at this concentration, which could have increased the oil's effective viscosity and reduced its ability to maintain its viscosity over a range of temperatures. Alternatively, the nanoparticles may have interfered with the oil's lubrication properties or reduced its resistance to shearing forces as reported by Zhang et al. (2023). Further addition of the CuO nanoparticles (0.3 wt.%), the VI decreased significantly to 313.01. This decrease in VI suggests that the nanoparticles were present at a concentration that exceeded their optimal dispersion or that they had formed large clusters or aggregates that increased the effective viscosity of the oil.

Overall, these results suggest that the effect of CuO nanoparticles on the VI of blended roselle oil is concentration-dependent and that an optimal concentration range may exist for maximizing the oil's viscosity stability. The results of the study indicate that the inclusion of CuO nanoparticles in blended roselle oil can also have a significant impact on its viscosity index. The increase in viscosity index at 0.1 wt.% concentration of nanoparticles suggests that the oil can maintain its viscosity even at higher temperatures, potentially improving its performance in high-temperature environments as said by Al-Anssari et al. (2021). However, the decrease in viscosity index at higher concentrations of nanoparticles (0.2% and 0.3%) may indicate that there is an optimal concentration range for the nanoparticles to be effective in improving the oil's viscosity index as imported by Pourpasha et al. (2023). If the concentration is too high, it may negatively impact the oil's viscosity index and ultimately its effectiveness as a brake transmission fluid. Overall, the results suggest that the inclusion of CuO nanoparticles in esterified Roselle oil can have a significant impact on its viscosity index, potentially improving its performance as a brake transmission fluid in high-temperature environments.

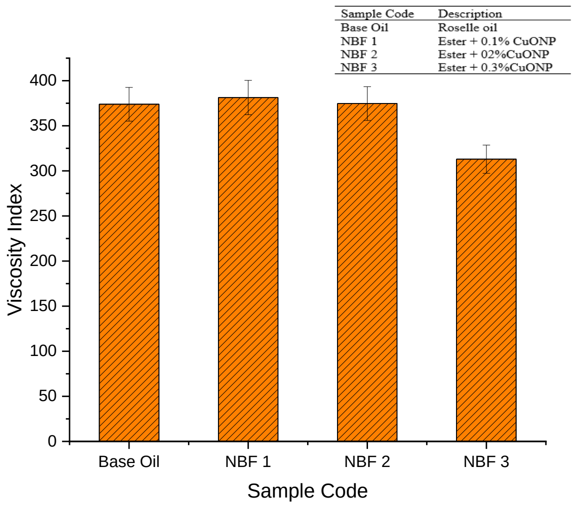

Figure 4: Effect of CuO nanoparticles inclusion on the viscosity index of Roselle oil

Pour Point
The pour point of an oil is the lowest temperature at which it can still flow under standard test conditions. It is an important property of brake fluid, as it can impact their ability to operate effectively in cold environments as observed by Jiang et al. (2022). The results of the experiment suggest that the addition of CuO nanoparticles to blended Roselle oil can have a significant effect on its pour point as presented in Figure 5. At 0.1 wt.% concentration of CuO nanoparticles, the pour point of blended Roselle oil decreased from 3 °C to 2 °C, indicating an improvement in its low-temperature fluidity. This decrease in pour point can be attributed to the ability of the nanoparticles to act as nucleation sites for the formation of wax crystals in the oil. When the oil is cooled below its pour point temperature, wax crystals form and block the flow of the oil. However, the addition of nanoparticles induces the formation of smaller and more uniform wax crystals, which allow easy flow of the fluid and reduce its pour point as observed by Zhong et al. (2021). At 0.2 wt.% concentration of CuO nanoparticles, the pour point of the blended Roselle oil increased to 3°C, suggesting that the optimal concentration

range was attained with a 0.2% increase This increase in pour point could be due to the formation of larger and more irregular wax crystals at this higher concentration of nanoparticles, which can block the flow of the oil and increase its pour point as imported by (Jia et al., 2022).

At a 0.3 wt.% concentration of CuO nanoparticles, the pour point of the esterified Roselle oil decreased again to 2°C. This decrease in pour point suggests that the nanoparticles at this concentration were able to induce the formation of smaller and more uniform wax crystals again, which can flow more easily through the oil and reduce its pour point as observed by Oungkanitanon et al. (2021). Overall, the results suggest that the effect of CuO nanoparticles on the pour point of esterified roselle oil is concentration-dependent and that an optimal concentration range exists for maximizing the oil's low-temperature fluidity. The pour point of brake fluid is an important parameter that can impact its ability to function effectively in a vehicle's braking system. A brake fluid with a high pour point may not flow properly at low temperatures, which can result in decreased brake system performance or failure. On the other hand, a brake fluid with a low pour point can provide reliable braking performance even at low temperatures. The results of the study suggest that the inclusion of CuO nanoparticles in blended Roselle oil can have a significant impact on its pour point, and therefore, its suitability as a brake transmission fluid. The observed decrease in pour point at a 0.1% concentration of nanoparticles suggests that the oil can maintain its flow properties even at lower temperatures, potentially improving its performance in cold weather conditions. However, the increase in pour point at higher concentrations of nanoparticles (0.2 wt.% and 0.3 wt.%) may indicate that there is an optimal concentration range for the nanoparticles to be effective in improving the oil's low-temperature properties. If the concentration is too high, it may negatively impact the oil's pour point and ultimately its effectiveness as a brake transmission fluid. The results of the study suggest that the inclusion of nanoparticles can have a positive impact on the low-temperature properties of the oil, potentially improving its performance in cold weather conditions and enhancing its potential as a brake fluid.

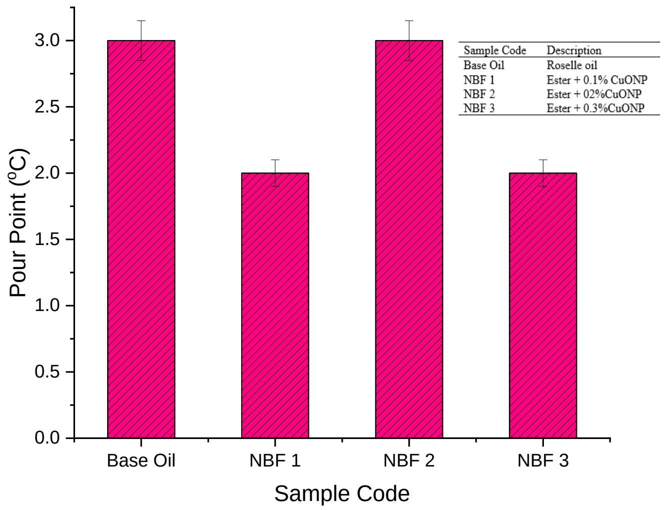

Figure 5: Effect of CuO nanoparticles on the Pour point of Roselle oil

Boiling Point

In Figure 6, the effect of CuO nanoparticles on the boiling point of blended Roselle oil is presented. It was observed that the boiling point of the esterified roselle oil was 130 °C. Including CuO nanoparticles at 0.1%, the boiling point became 135 °C. At 0.2% CuO nanoparticles, the boiling point is 140 ℃. At 0.3 wt.% CuO nanoparticle inclusion as observed by Neoi et al. (2019), the boiling point is 145 ℃. The boiling point of a fluid is the temperature at which the vapor pressure of the fluid equals the atmospheric pressure. The boiling point of a fluid is an important parameter for any application that involves high-temperature conditions, such as in an engine or hydraulic system as imported by Mahmoudinezhad et al. (2023). A high boiling point indicates that the fluid will remain stable at high temperatures, while a low boiling point indicates that the fluid may vaporize and lose its effectiveness in high-temperature environments. In the present study, the inclusion of CuO nanoparticles in esterified Roselle oil resulted in a significant increase in the boiling point of the oil. The increase in boiling point with increasing concentration of nanoparticles indicates that the nanoparticles can stabilize the oil at high temperatures and prevent it from vaporizing or breaking down. This increase in boiling point can be attributed to the properties of the CuO nanoparticles themselves. CuO nanoparticles have high thermal stability and high surface area, which can enhance their interaction with the esterified Roselle oil molecules. This interaction can lead to the formation of a more stable and uniform fluid structure, which is less prone to vaporization at high temperatures as observed by Said et al. (2022). The increase in boiling point observed in the study indicates that the inclusion of CuO nanoparticles in esterified Roselle oil can improve its thermal stability and make it more suitable for high-temperature applications, such as in engines or hydraulic systems. The boiling point is a critical parameter for brake transmission fluids as it indicates the temperature at which the fluid will start to boil and lose its effectiveness as observed by (Faridah et al., 2023). Brake transmission fluids are often exposed to high temperatures due to the friction generated during braking, and a low boiling point can result in vaporization and a decrease in the fluid's effectiveness, leading to reduced braking performance.

The observation that the inclusion of CuO nanoparticles in esterified Roselle oil can increase its boiling point suggests that the oil may be more suitable for use as a brake transmission fluid in high-temperature environments. A higher boiling point indicates that the fluid can remain stable and effective at higher temperatures, reducing the risk of vaporization and ensuring reliable braking performance as observed by Hosseinzadeh-Bandbafha et al. (2023). Furthermore, the increase in boiling point with increasing concentration of nanoparticles suggests that the oil's thermal stability can be optimized by adjusting the concentration of nanoparticles. This may provide a way to tailor the oil's properties to meet the specific requirements of different brake transmission applications, further increasing its suitability for use as a brake transmission fluid. Overall, the observation that the inclusion of CuO nanoparticles in esterified Roselle oil can increase its boiling point has significant implications for its potential application as a brake transmission fluid. The increase in boiling point observed in this study suggests that the oil may be a promising candidate for use in high-temperature brake transmission applications as observed by Uppar et al. (2022).

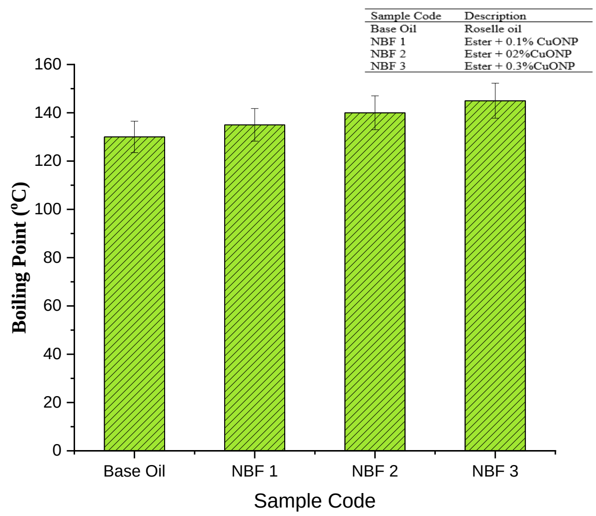

Figure 6: Effect of CuO on the boiling point of esterified roselle oil

Flashpoint

The flash point of the blended Roselle oil was observed to be 199 ℃ as illustrated in Figure 7. Including CuO nanoparticles at 0.1% content increases the flash point to 202 ℃. At 0.2 wt.% of CuO nanoparticles inclusion, the flash point was 209 ℃ and further increase in the nanoparticles increased the flash point to 210 ℃ as observed by Jin et al. (2023). The flash point of a liquid is the lowest temperature at which the fluid can vaporize to form an ignitable mixture in the air. In the case of blended Roselle oil, the flash point is a critical parameter as it indicates the temperature at which the oil may ignite when exposed to a heat source. Therefore, an oil with a higher flash point is considered safer to use and handle. In this study, it was observed that the inclusion of CuO nanoparticles in esterified Roselle oil can increase its flash point. This is likely because nanoparticles have a high surface area-to-volume ratio and can absorb heat more efficiently. As a result, the nanoparticles can reduce the amount of heat that reaches the oil, increasing its resistance to ignition and raising its flash point. The increase in flash points observed in this study with increasing concentration of nanoparticles suggests that the oil's thermal stability can be optimized by adjusting the concentration of nanoparticles as imported by (Pourpasha et al., 2020). This may provide a way to tailor the oil's properties to meet the specific requirements of different applications, further increasing its suitability for use as a lubricant, hydraulic fluid, or other industrial oil.

Overall, the observation that the inclusion of CuO nanoparticles in blended Roselle oil can increase its flash point has significant implications for its potential application in various industries. However, further research and testing would be needed to fully evaluate the oil's effectiveness and safety in a range of operating conditions. The inclusion of CuO nanoparticles in esterified Roselle oil increases the oil's flash point due to the thermal properties of the nanoparticles. CuO nanoparticles have a high surface area-to-volume ratio and can absorb heat more efficiently. When the nanoparticles are dispersed in the oil, they act as a heat sink, absorbing heat and reducing the amount of heat that reaches the oil. This thermal barrier helps increase the oil's resistance to ignition, which results in a higher flash point. The increase in flash points observed in this study with increasing concentrations of nanoparticles can be attributed to the higher number of nanoparticles available to absorb heat as the concentration increases as observed by (Devaraj et al., 2022). At a low concentration of 0.1

wt.%, the number of nanoparticles is relatively low, so the increase in flash point is modest. However, as the concentration increases, the number of nanoparticles in the oil increases, leading to a greater reduction in heat transfer and a more significant increase in flash point. The flash point is an essential parameter in determining the suitability of oil as brake fluid. A brake fluid with a high flash point is desirable because it is less likely to ignite or vaporize at high temperatures, ensuring that the fluid continues to provide reliable hydraulic power to the brakes even under extreme operating conditions.

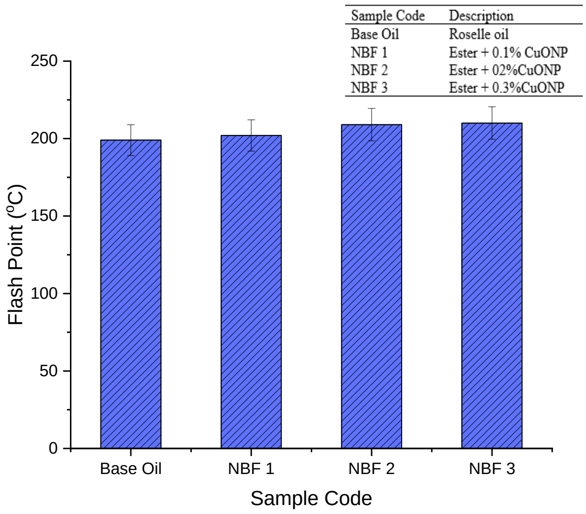

Figure 7: Effect of the inclusion of CuO nanoparticles on the flashpoint of roselle oil

Coefficient of Friction and Wear Rate

The wear rate and coefficient of friction are important parameters to assess the performance and suitability of a brake fluid. When CuO nanoparticles are introduced into Roselle oil, they can potentially alter the lubrication properties and affect the frictional behaviour of the fluid as reported by (Garcoa et al., 2021). By measuring the wear rate, the material loss or degradation of the brake components in contact with the hydraulic fluid can be elaborated. This is crucial for understanding the long-term durability and performance of the brake system. Additionally, determining the coefficient of friction helps in assessing the frictional characteristics of the brake fluid. Changes in the coefficient of friction due to the addition of CuO nanoparticles can impact the braking performance, including factors such as braking efficiency, heat generation, and stability as observed by (Khan et al 2022). The variation in the coefficient of friction with the increase in the quantity of Roselle oil is presented in Figure 8. It was observed that the coefficient of friction for the base oil, which is pure Roselle oil is 0.117. The coefficient of friction of the CuO nanoparticle including roselle oil was observed to be 0.067, 0.104, and 0.057 at a CuO nanoparticle quantity of 0.1 wt.%, 0.2 wt. and 0.3 wt.% respectively. The observed changes in the coefficient of friction (COF) between the base oil (pure Roselle oil) and the CuO nanoparticle-included roselle oil at different nanoparticle quantities (0.1 wt.%, 0.2 wt.%, and 0.3 wt.%) provide valuable insights into the frictional behaviour of the lubricants.

The addition of CuO nanoparticles to roselle oil results in a notable decrease in the COF compared to pure roselle oil. This reduction in COF can be attributed to several mechanisms that occur due to the presence of the nanoparticles as reported by Solano et al. (2019). Firstly, the CuO nanoparticles act as solid lubricants themselves, forming a protective layer between the sliding surfaces. This layer reduces the direct contact and interaction between the surfaces, resulting in a decrease in friction and subsequently lowering the COF. The nanoparticles act as a physical barrier, preventing metal-to-metal contact and reducing the frictional forces. Secondly, the nanoparticles contribute to the formation of a tribofilm on the sliding surfaces as observed by (Vyavhare et al 2021). During the sliding motion, the nanoparticles undergo localized heating, which facilitates their oxidation and the formation of oxide layers on the surfaces. These oxide layers, in the form of tribofilms, provide lubricating properties and reduce friction by providing a smooth, low-friction interface between the surfaces. The presence of these tribofilms helps to lower the COF. Additionally, the nanoparticles improve the load-bearing capacity of the lubricant as discussed by (Fang et al., 2023). As the nanoparticles fill in surface irregularities, they help distribute the applied load more evenly across the contact area. This leads to a reduction in the localized stress and pressure, which can contribute to a decrease in the COF. Furthermore, the CuO nanoparticles possess properties such as high surface energy and rough surface morphology, which enhance their ability to adhere to sliding surfaces as observed by Tekdir and Yetim (2021). This adhesion allows the nanoparticles to form a durable and stable lubricating layer, reducing the friction between the surfaces and resulting in a lower COF. Overall, the observed decrease in the COF when CuO nanoparticles are added to roselle oil can be attributed to the combination of factors, including the nanoparticles acting as solid lubricants, the formation of tribofilms, improved load-bearing capacity, and the adherence of the nanoparticles to the sliding surfaces. These mechanisms collectively contribute to the reduction in friction and the resulting lower COF observed in the CuO nanoparticle-included roselle oil compared to the pure roselle oil. These findings provide a strong scientific basis for the potential application of CuO nanoparticle-included roselle oil as a hydraulic fluid with improved frictional behaviour in brake systems.

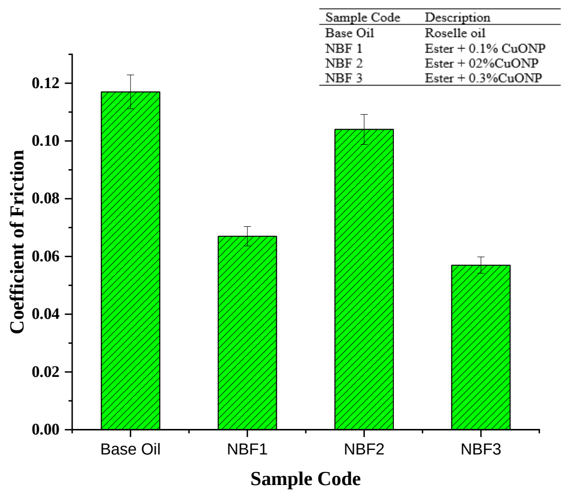

Figure 8: Variation of Coefficient of Friction with Nano Particle Concentration

Figure 9 shows the wear rate of the oil and the blends of the oil at 0.1 wt.%, 0.2wt.%, and 0.3wt.% of the nanoparticles respectively. Pin on disc wear test showed that the wear rate of aluminum with the oil as a brake fluid was 0.3729 $mm^3n^{-1}m^{-1}$, 0.4316 $mm^3n^{-1}m^{-1,}$ and 0.3592 $mm^3n^{-1}m^{-1}$ at a nanoparticle reinforcement of 0.1 wt.%, 0.2 wt.%, and 0.3 wt.% respectively. This was far lower than the wear rate of the pure roselle oil which was at 0.6268 $mm^3n^{-1}m^{-1}$. The results obtained from the pin-on-disc wear test provide compelling evidence for the effectiveness of CuO nanoparticles in reducing the wear rate of aluminum when added to roselle oil as a lubricant. Comparing the wear rates of aluminum with different levels of nanoparticle reinforcement (0.1 wt.%, 0.2 wt.%, and 0.3 wt.%) to the wear rate of pure roselle oil, it is clear that the inclusion of CuO nanoparticles leads to a significant decrease in wear. One of the primary reasons for this reduction in wear rate is the enhanced lubrication properties provided by the CuO nanoparticles as observed by Gupta et al. (2021). These nanoparticles form a protective layer on the sliding surfaces of the pin and the disc, creating a barrier that minimizes direct contact between the aluminum and the opposing surface. This protective layer acts as a solid lubricant which effectively reduces friction and wear between the surfaces as observed by Yin et al. (2023). By preventing metal-to-metal contact, the nanoparticles contribute to a substantial decrease in wear. During the sliding motion, the nanoparticles undergo localized heating, which promotes the formation of thin oxide layers on the aluminum surface. These oxide layers act as protective films, providing an additional barrier between the aluminum and the opposing surface. The presence of these films helps to prevent direct metal-to-metal contact and reduces wear. Furthermore, the presence of CuO nanoparticles improves the load-bearing capacity of the lubricant. The nanoparticles fill in surface irregularities on the aluminum pin and the disc, effectively smoothing out the contact surfaces. This results in a reduction in the number of points of contact and a more even distribution of the applied load (Gao et al., 2022). By distributing the load more evenly, the nanoparticles help to minimize the localized stress on individual contact points, thereby reducing wear. Overall, the observed reduction in wear rate can be attributed to a combination of factors, including enhanced lubrication, improved load-bearing capacity, and the formation of protective films facilitated by the presence of CuO nanoparticles in roselle oil. These findings demonstrate the strong scientific basis for utilizing CuO nanoparticles as a means to significantly improve the lubricating properties of hydraulic fluids in brake systems, thereby enhancing their wear resistance and overall performance.

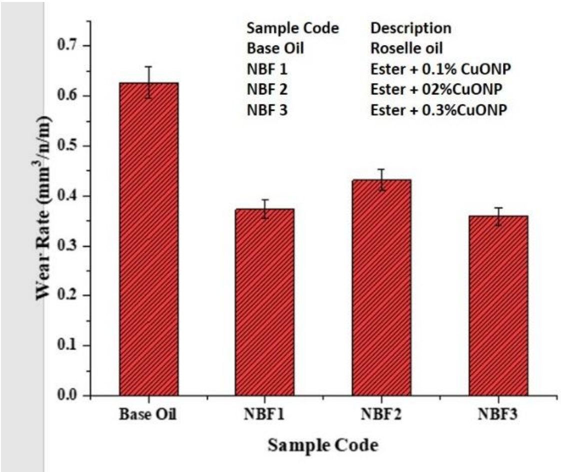

Figure 9: Variation of Wear Rate with Nano Particle Concentration

Conclusion

In conclusion, this study aimed to evaluate the potential of a bio-based brake fluid with a Nano copper oxide additive. The specific objectives were successfully achieved, and the key findings are summarized as follows:

A bio-based brake fluid was successfully formulated using Roselle oil and Nano copper oxide additive. The additive, characterized by TEM with nanoparticles under 20nm diameter, exhibited a CuO nanoparticle crystallite size of 9.22 nm, ensuring effective dispersion and compatibility within the brake fluid. Physio-chemical stability was demonstrated in the brake fluid, with a kinematic viscosity of 3.5 cSt at 0.3 wt% concentration, suitable for extended use. Spectrochemical analysis revealed a C-H stretching vibration peak at 2922 cm^{-1} in the FTIR spectrum. Tribological tests indicated reduced friction (0.057) and effective wear prevention (0.3592 $mm^3n^{-1}m^{-1}$) at the 0.3 wt% CuO nanoparticle concentration, confirming enhanced efficiency and durability of the brake fluid. Performance evaluation under various conditions, including temperature, pressure (with an inlet at 3.447 MPa), and velocity flow rate, consistently demonstrated reliable performance in challenging scenarios. This underscores the brake fluid's adaptability and suitability for real-world automotive applications. The bio-based brake fluid exhibited a comparable kinematic viscosity of 1.2 cSt, aligning favorably with conventional brake fluids (typically 1.5 to 1.8 cSt). This positions it as a promising alternative in the automotive industry, leveraging the properties of blended Roselle oil. These findings collectively suggest that the bio-based brake fluid with Nano copper oxide additive shows significant potential for practical use, offering improved efficiency, durability, and compatibility as compared to conventional counterparts.

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