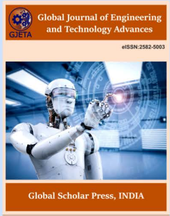
Global Journal of Engineering and Technology Advances

eISSN: 2582-5003

Cross Ref DOI: 10.30574/gjeta

Journal homepage: https://gjeta.com/



(RESEARCH ARTICLE)

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Tribological properties of copper (II) oxide nanoparticle-enriched sandbox bio-lubricant

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Global Journal of Engineering and Technology Advances, 2021, 08(03), 008–019

Publication history: Received on 02 October 2020; revised on 12 November 2020; accepted on 25 November 2020

Article DOI: https://doi.org/10.30574/gjeta.2021.8.3.0121

Abstract

The demand for bio-lubricant is constantly on the increase due to rapid depletion of world fossil fuel reserves, technological advancement, concern for environmental pollution and its potential to address the energy and environmental problems. This research evaluated the tribological properties of copper (II) oxide nanoparticle-enriched bio-lubricant developed from sandbox (*Hura crepitans*) seed oil. Copper (II) oxide nanoparticle was added to improve the tribological properties of the bio-lubricant. The experimental parameters were set up according to central composite design of response surface methodology to minimize the numbers of experiments. The bio-lubricants containing varying proportions of copper (II) oxide nanoparticle additive were prepared and their tribological properties were evaluated using ball-on-disc tribometer. The values of parameters used to assess the tribological behaviours were: load (2 N, 5 N, 8 N), speed (150 rpm, 200 rpm, 250 rpm) and nanoparticle concentration (0 wt%, 0.75 wt%, 1.50 wt%). The effects of these parameters on wear rate, friction coefficient and flash temperature parameter were evaluated. The lowest value of coefficient of friction (0.048) was obtained at a speed of 250 rpm and concentration of 0.75 % with load of 5 N. The lowest value of wear rate (0.012) was obtained with load of 5 N, speed of 200 rpm and concentration of 0.75 %. The highest value of flash temperature parameter (0.035) was obtained with load of 5 N, speed of 250 rpm and concentration of 0.75 %. The obtained values complied with the standard specified by American Standards and Testing Materials (ASTM) and compare favourably with conventional lubricant SAE 40 and other bio-lubricants. The results of the analysis were used for optimizing the parameters to obtain the best lubricant effects. The optimal combination of parameters for minimum coefficient of friction and wear rate as well as maximum flash temperature were found to be: 5.0909 N load, 217.6768 rpm speed and 1.1061 wt% concentration. The overall results revealed that enrichment of sandbox oil with copper (II) oxide nanoparticle improved the tribological behavior of the oil.

Keywords: Sandbox oil; Copper (II) oxide nanoparticle; Tribology; Bio-lubricant; Friction; Wear

1. Introduction

There is growing international concerns about environmental pollution associated with the use and disposal of mineral oil based lubricants. Talkit *et al.* (2015) reported the mineral oil lubricants produced from crude oil sources as non-degradable and non-renewable; causing environmental pollution like water pollution, soil pollution and air pollution, pose threatening conditions for danger and damage to well-being and human health, welfare of plants and animals, and the environment as a whole. The concern about the pollution created as a result of using mineral oil based lubricants as well as the depletion stock of petroleum has inspired research on alternative lubricants with focus on the formulation and development of biodegradable, renewable and eco-friendly lubricants.

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Recently, special emphasis has been on how to protect the environment against pollution resulting from the use of mineral oil lubricants. Bio-lubricants are becoming important alternative to mineral oil based lubricants due to improvement in awareness of environmental pollution. Mohammed *et al.* (2018) reported that researchers have recommended bio-based lubricants for their unique characteristics with a view to maintaining domestic energy security and addressing environmental concerns. Bio-lubricants, according to Strivastava and Sahai (2013), biodegrade rapidly and are non-harmful for human beings and environment. They possess better properties like renewability, environmentally friendly, biodegradability, non-toxicity, excellent lubricity, high fire point and flash point, high viscosity index, low volatility and low vapour emissions. Vegetable oils have already been used as lubricant for a long time. Vegetable oils are nowadays considered as viable bio-resource and promising candidates for the development of bio-based lubricants because they have some basic properties which make them suitable to be used for lubricant. Various studies have been carried out using different vegetable oils as raw materials for lubricants production. Naik and Galhe (2016) reported the presence of monounsaturated fatty acids in vegetable oils, such as oleic and palmitic, makes it to be considered as the best feedstock for lubricants.

Many researchers have worked on vegetable oils such as palm oil, soybean oil, castor oil, jatropha oil, neems oil and sunflower oil to make lubricant. Adewuyi *et al.* (2014) reported that more than 95% of world bio-lubricant is produced from edible oils such as rapeseed oil, soybean oil, canola oil, palm oil and palm kernel oil. But considering the daily rise in human population, this has brought high price of base oil and serious competition between food and lubricants arising from the extensive use of these edible oils for lubricants. It is also believed that the large-scale production of bio-lubricant from edible oil may bring global imbalance to food supply and demand market. In order to overcome this devastating phenomenon, researches have been made to produce bio-lubricant using non-edible oils which are very economical comparable to edible oils and potentially offer greatest opportunities in the longer term for effective lubricant production. The increasing need to search for more non-edible oils from vegetable sources to complement others has led to the employing sandbox seed oil as alternative raw material for lubricant production. Sandbox oil is highly neglected non-edible oil available at little or no cost of purchase. Giwa *et al.* (2016) reported that it has no specific use and no commercial value presently, as the seeds are discarded as waste. Sandbox oil as a promising alternative oil, is environmentally friendly, renewable, cheap, and easily manageable.

Nano-based bio-lubricant has been described by Koshy *et al.* (2015) as a lubricant obtained through the addition of nanoparticle to the vegetable base oil. Many research works have investigated the effect of nanoparticle additives on the properties of lubricants. Generally, majority of the study agree that nanoparticles improved the tribological properties of lubricants by deposition at the contact area thereby creating a protective layer. Nanoparticle additives, as reported by Singh *et al.* (2018), can easily mix with lubricating oil due to its mini-scale size to enhance the lubricating properties of the oil. They can significantly improve the wear-reduction capability, friction-reducing property and pure oil load carrying capacity according to Su *et al.* (2018). They provide large surface area to improve interaction with the surface material of friction pairs forming a protective film due to their high surface area to volume ratio, thereby reducing wear and friction as reported by Lopez *et al.* (2015). Nanoparticles of various chemical compositions have shown great tribological potential. Ali *et al.* (2016) reported the activeness of nanoparticle enhanced lubricant under boundary lubrication in forming a tribo-film at the asperity contact locations to improve the tribological properties. According to Le and Lin (2017), with its miniscule size, nanoparticle can easily mix with the lubricant oil to enhance its lubricating properties. Afzal *et al.* (2017) observed that the presence of nanoparticles in the lubricant affects its wear, friction, thermal, chemical and physical properties in many ways. Koshy *et al.* (2015) reported that the nanoparticles in lubricant prevent corrosion, modify the oil viscosity as well as reduce friction.

Copper oxide (CuO) nanoparticle has been observed by Abere (2017) to have potentials as anti-wear additive in a chemically modified rapeseed oil tested in high frequency reciprocating rig (HFRR) under boundary lubrication conditions. Ghaednia *et al.* (2015) reported copper oxide nanoparticle to be insusceptible to oxidation which makes them to be effective high temperature additive. Thottackkad *et al.* (2012) evaluated the tribological characteristics of lubricant from coconut oil using copper oxide nanoparticle as additive. Their results show that optimum concentration of nanoparticle in the lubricant reduces the pin surface roughness to a low value after sliding. As the quantity of nanoparticle increases beyond the optimum level, wear rate as well as coefficient of friction increased. Koshy *et al.* (2015) studied the tribological and thermo-physical properties of bio-based copper (II) oxide nano-lubricant which contains surfactant at elevated temperatures and observed that improvement in the properties like viscosity and fire point is more obvious for nano-lubricant with surfactant-modified copper (II) oxide nanoparticle than the corresponding nano-lubricant with unmodified copper (II) oxide nanoparticles. Su *et al.* (2018) examined the influence of carbon nanotube on the tribological behaviour of vegetable-based lubricant and discovered that short and thin multi-walled carbon nanotube can improve the tribological characteristics of vegetable-based lubricant more effectively than long and thick carbon nanotube. This study investigated and evaluated tribological properties of copper (II) oxide nanoparticle –

enriched sandbox bio-lubricant and predicted the optimum formulations for the production and utilization of the bio-lubricant in order to obtain the best lubricant effects.

2. Experimental setup

2.1. Materials and equipment

The materials and equipment used in this research included: sandbox (*Hura crepitans*) oil, methanol, potassium hydroxide, trimethylolpropane (TMP), oleic acid (OA), copper (II) oxide (CuO) nanoparticle and Anton Paar ball-on-disc tribometer (model TRN).

2.2. Methods

2.2.1. Formulation of nanoparticle-enriched bio-lubricant

The nanoparticle-enriched bio-lubricant was formulated in accordance with the method described by Bilal *et al.* (2013), using sandbox oil as base oil. The formulation involves a double transesterification process; methyl ester synthesis and bio-lubricant synthesis. During the first transesterification, an intermediate product, methyl ester of the oil was produced by mixing the oil sample with methanol using potassium hydroxide as catalyst. The ratio of weight of the oil – methanol was 3:1 and potassium hydroxide of 0.5 wt% of the oil was used. Bio-lubricant synthesis was achieved during the second transesterification by adding trimethylolpropane (TMP) to the sandbox methyl ester to produce the desired product, a polyol ester (bio-lubricant). The weight ratio of oil - trimethylolpropane (TMP) was 3.5:1 as reported by Zulkifli *et al.* (2013). The reaction was conducted at a temperature of 120^{o}C for two hours thirty minutes (2½ H) as prescribed by Jatti and Singh (2015). After the reaction has completed, vacuum filtration was used for separation of the catalyst from the product mixture. Thereafter un-reacted methyl esters were removed from the final product by vacuum distillation. Copper (II) oxide (CuO) nanoparticle was added in different concentrations (minimum 0.75 wt%; maximum 1.50 wt%) to the bio-lubricant to improve its tribological properties. Oleic acid (1 wt%) was also added as surfactant to prevent nanoparticle agglomeration during dispersion process and improve the dispersion stability of the nanoparticle. The bio-lubricant was produced using different concentration of nanoparticle additives.

2.2.2. Design of Experiment (DOE)

Minitab Release 16.00 was employed to conduct research on relevant parameters and to determine their values in order to optimize the production and utilization of the nanoparticle-enriched bio-lubricant. Response surface methodology was used to study the effects of nanoparticle additive concentration and subsequently in the optimization of the process. The method assists in optimizing the effective parameters with a least number of experiments and in analyzing the interaction between the parameters. A three-level, three factors (3^3) designs was used for the tribological evaluation of the bio-lubricant. The levels were coded as -1, 0 and +1. The three factors used known as independent variables were: load (2.0 N, 5.0 N and 8.0 N), speed (150 rpm, 200 rpm and 250 rpm) and concentration: copper (II) oxide (CuO) nanoparticle with varying weight concentrations (0, 0.75 wt% and 1.50 wt%). These factors as well as their levels as mentioned are specified in Table 1, while Table 2 shows the design values obtained from Minitab. These values were based on a number of recent studies on nanoparticles-enriched bio-lubricants by Le and Liu (2017), Gulzar (2017) and Abere (2017).

Table 1 Factors and levels for experiment

Factors	**Symbol**	**Level**		
		(-1)	**(0)**	**(+1)**
Load (N)	A	2.0	5.0	8.0
Speed (rpm)	B	150	200	250
Concentration (wt%)	C	0	0.75	1.50

Table 2 Design values obtained from Minitab

Run No.	Factors to be examined		
	Load (N)	**Speed (rpm)**	**Conc. (wt %)**
1	2.0	150	0
2	8.0	150	0
3	2.0	250	0
4	8.0	250	0
5	2.0	150	1.50
6	8.0	150	1.50
7	2.0	250	1.50
8	8.0	250	1.50
9	2.0	200	0.75
10	8.0	200	0.75
11	5.0	150	0.75
12	5.0	250	0.75
13	5.0	200	0
14	5.0	200	1.50
15	5.0	200	0.75
16	5.0	200	0.75
17	5.0	200	0.75
18	5.0	200	0.75
19	5.0	200	0.75
20	5.0	200	0.75

2.3. Tribological evaluation of the bio-lubricant

The tribological behaviour of sandbox lubricant with and without copper (II) oxide nanoparticle additive was evaluated in accordance with Abere (2017) using an Anton Paar ball-on-disc tribometer (Figure 1).

Figure 1 Anton Paar ball-on-disc tribometer

The standard test method of ASTM G99-05 (2010) was adopted to evaluate the tribological performance of the bio-lubricant. An Anton Paar ball-on-disc tribometer model TRN used consists of a lever-arm device to hold the ball, a driven spindle and chuck for holding the revolving disc, and attachments that allow the ball to move forcefully against the revolving disc with a controlled load. The tribometer also had a data acquiring system and user interface software which records and controls the experiment. The balls of 6 mm diameter made of stainless steel were fitted into the upper stationary ball holder on the tribometer. The aluminium alloy discs of 70 mm diameter and 6.35 mm thick were machined. Tests were conducted on the bio-lubricant using the loads of 2 N, 5 N and 8 N, speeds of 150 rpm, 200 rpm and 250 rpm, and concentration of nanoparticle as varying parameters. The sliding distance was 50 m and all the tests were done for duration range of 5 – 12 minutes. Balls and disc were cleaned with acetone. Load was applied on the ball by dead weight through pulley-string arrangement. Lubricant was applied between the disc and the ball to satisfy boundary lubrication conditions. The ball specimen was inserted carefully in the holder and it was perpendicularly adjusted to the disc surface when in contact to maintain the necessary contact conditions. Appropriate load was added to the system lever to the selected force pressing the ball against the disc. The speed and revolution counter were adjusted to desired values and the electric motor was turned on. The test was stopped after achieving the desired numbers of revolutions. The specimen was removed and cleaned. The procedure was repeated for all the tests to obtain wear rate and friction coefficient. The values of friction coefficient and wear rate obtained were recorded.

2.3.1. Coefficient of friction evaluation

The coefficient of friction, according to Battez and Rodriguez (2016), is a dimensionless number that describes the ratio between the friction force between two bodies and the normal force opposing them together. Friction evolution in the ball-on-disc tribo-tests gives the impact of nanoparticles on the bio-lubricants and the lubrication process. The coefficient of friction was measured using data acquisition system connected to the tribometer and recorded.

2.3.2. Wear rate evaluation

The aluminium alloy disc is softer than the ball sample (stainless steel). In agreement with Archard wear law, it is assumed that the harder ball sample wear is insignificant compared to that of the softer disc sample. The sliding wear volume loss was more significant on the disc surface than on the ball surface. Koshy *et al.* (2015) described wear rate of a tribological contact as the lost volume from the wearing surface per sliding distance. It is the wear volume (mm^3) per sliding distance (m). The wear rate was measured using data acquisition system connected to the tribometer and recorded.

2.3.3. Flash temperature parameter evaluation

The flash temperature parameter was calculated for all experimental conditions. The flash temperature parameter is a number used for the expression of the critical flash temperature at which a lubricant will function well under given conditions. Mohammed *et al.* (2018) described flash temperature parameter as the critical temperature below which the lubricant can create a film and withstand without breakdown; the higher the value of FTP, the better the performance of the lubricant.

According to Jabal *et al.* (2014), the FTP is usually calculated using:

$$FTP = \frac{W}{d^{1.4}} \tag{1}$$

Where, $W = load\ (kg)$

$d = mean\ wear\ track\ diameter\ (\mu m)$

2.3.4. Surface analysis

Surface analysis of the disc specimens was carried out in accordance with Gulzar (2017) to understand the lubrication mechanism of the lubricant. Scanning electron microscopy which is a versatile tool for investigation of surfaces at a broad range of magnifications and high resolution was used for the analysis. SEM, according to Gulzar (2017), provides qualitative information about topography, morphology and general appearance of tested surfaces. In using SEM, focused electron beam was scanned through the sample surface and related image was taken.

3. Results and discussion

3.1. Tribological performance of sandbox oil and formulated bio-lubricant

The tribological performances of sandbox oil and formulated copper (II) oxide nanoparticle-enriched bio-lubricant as well as that of conventional lubricant SAE 40 were determined. The observed tribological properties which include coefficient of friction, wear rate and flash temperature parameter are shown in Tables 3, 4 and 5. The estimated regression coefficients and analysis of variance (ANOVA) for coefficient of friction, wear rate and flash temperature of the bio-lubricant are presented in Tables 6 while Figure 1 shows optical micrograph of wear track on the discs.

Table 3 Tribological properties of the nanoparticle-enriched bio-lubricant

Run No.	Factors			Coefficient of friction	Wear rate (mm^3/Nm)
	Load (N)	Speed (rpm)	Conc. (wt %)		
1	2.0	150	0	0.074	0.012
2	8.0	150	0	0.088	0.029
3	2.0	250	0	0.106	0.013
4	8.0	250	0	0.072	0.035
5	2.0	150	1.50	0.070	0.010
6	8.0	150	1.50	0.079	0.026
7	2.0	250	1.50	0.079	0.013
8	8.0	250	1.50	0.068	0.033
9	2.0	200	0.75	0.075	0.012
10	8.0	200	0.75	0.060	0.031
11	5.0	150	0.75	0.058	0.022
12	5.0	250	0.75	0.048	0.014
13	5.0	200	0	0.077	0.019
14	5.0	200	1.50	0.069	0.015
15	5.0	200	0.75	0.052	0.012
16	5.0	200	0.75	0.052	0.012
17	5.0	200	0.75	0.052	0.012
18	5.0	200	0.75	0.052	0.012
19	5.0	200	0.75	0.052	0.012
20	5.0	200	0.75	0.052	0.012

Table 4 Flash temperature parameter of the nanoparticle-enriched bio-lubricant

Run No.	Factors			Wear scar diameter (μm)	Flash temp parameter (FTP)
	Load (N)	Speed (rpm)	Conc. (wt %)		
1	2.0	150	0	8.61	0.010
2	8.0	150	0	13.22	0.022
3	2.0	250	0	5.07	0.021
4	8.0	250	0	10.84	0.029
5	2.0	150	1.50	7.48	0.012
6	8.0	150	1.50	11.61	0.026
7	2.0	250	1.50	7.23	0.013
8	8.0	250	1.50	10.12	0.032
9	2.0	200	0.75	6.27	0.007
10	8.0	200	0.75	11.20	0.028
11	5.0	150	0.75	8.68	0.025
12	5.0	250	0.75	9.55	0.022
13	5.0	200	0	7.92	0.028
14	5.0	200	1.50	8.52	0.025
15	5.0	200	0.75	9.22	0.023
16	5.0	200	0.75	9.22	0.023
17	5.0	200	0.75	9.22	0.023
18	5.0	200	0.75	9.22	0.023
19	5.0	200	0.75	9.22	0.023
20	5.0	200	0.75	9.22	0.023

Table 5 Tribological properties of the conventional lubricant SAE 40

Run no.	Load (N)	Speed (rpm)	COF	Wear rate (mm^3/Nm)	FTP
1	2.0	150	0.065	0.010	0.013
2	5.0	150	0.047	0.019	0.022
3	8.0	150	0.039	0.022	0.027
4	5.0	200	0.042	0.015	0.034
5	2.0	250	0.060	0.011	0.024
6	5.0	250	0.038	0.023	0.028
7	8.0	250	0.030	0.025	0.036

3.1.1. Friction behaviour of the bio-lubricants

Coefficient of friction of the bio-lubricant reduced significantly (26% to 33% decrease) compared to coefficient of friction values of the crude sandbox oil as shown in Table 3. The results of coefficient of friction of the bio-lubricant under the loads of 2 N, 5 N and 8 N showed that nanoparticle additive in sandbox oil at a various concentration have better anti-friction properties than pure sandbox oil. There was increase in coefficient of friction with an increase in speed for 2 N load, but decreased for 5 N load and 8 N load. These results agreed with the works reported by Ali *et al.* (2016), Jabal *et al.* (2014) and Patil *et al.* (2014), where friction coefficients reduced over time in nanoparticles-enriched oils compared to the oils without nanoparticle.

Copper (II) oxide nanoparticle added to the sandbox oil reduced coefficient of friction of the sandbox oil at 150, 200 and 250 rpm speed under the loads of 2 N, 5 N and 8 N. The coefficient of friction varied with the increase in load. For 5 N load at 200 rpm speed, friction coefficient reduced from 0.077 to 0.052 after adding 0.75 wt% copper (II) oxide nanoparticle. As the concentration of copper (II) oxide nanoparticle in the bio-lubricant increases (from 0.75 wt% to 1.50 wt%), the coefficient of friction increased from 0.052 to 0.069. This shows that there is an optimum concentration level at which the friction coefficient is a minimum for a given load and speed as reported by Thottackkad *et al.* (2014). The lowest coefficient of friction was 0.048 at 0.75 wt% copper (II) oxide nanoparticle concentration, 200 rpm speed and 2 N load while the highest value of 0.079 was found at 1.50 wt% concentration at 150 rpm with 8 N load and 250 rpm speed with 2 N load as shown in Table 3. These values compete favourably with the values of conventional lubricant SAE 40 which ranged between 0.030 and 0.065 as shown in Table 5 and are within the acceptable range (0.01 – 0.14) of coefficient of friction of a lubricant to be used for wide range of automotive applications as reported by Habibullahi *et al.* (2014). Abere (2017), Bahari (2017) and Farhanah and Syahrullail (2015) also reported mean coefficient of friction of 0.0995, 0.093 and 0.099 respectively for SAE 40. For other bio-lubricants, mean coefficient of friction of 0.0833 was reported for rapeseed oil and 0.0893 for rapeseed oil enriched with nanoparticles under the load of 5 N by Abere (2017), 0.153 for palm stearin and 0.036 for palm stearin with 5% ZDDP by Farhanah and Syahrullail (2015).

Generally, at lower concentration levels, when the concentration of nanoparticle is increased, the nanoparticle in the lubricant develop a third body rolling effect between the sliding surfaces, causing a reduction in friction. This effect according to Thottackkad *et al.* (2014) becomes much more prominent at higher loads where the nanoparticles agglomerate and provide more supporting effort to the contacting area. However, as the concentration is further increased, interaction between the solid particles themselves becomes dominant, which increases friction. In another development, in accordance with the mechanical entrapment theory as reported by Zulkifli *et al.* (2013), nanoparticle present in the lubricant get deposited at the contacting surfaces, forming a layer that is subsequently removed during further sliding, the net effect being a reduction in friction. The effects of nanoparticle on friction coefficient are more pronounced at higher loads and lower nanoparticle concentration. According to Ghaednia *et al.* (2015), an increase in load would force more lubricant out of the contact region thereby reduces the lubricant film thickness between surfaces. This would escalate the possibility of direct contact or interaction between surfaces and therefore, increases probability of nanoparticles engagement in the contact. The variation in friction behaviour by changing nanoparticles concentration can be attributed to the dispersion stability. Stable dispersion helped in providing the continuous resistance against friction. Nanoparticle concentration plays a significant role in the development of stable dispersion which in turn results in improved friction behaviour (Gulzar, 2017).

The main reasons for the decrease in friction coefficient for the nanoparticle-enriched bio-lubricant according to Ali *et al.* (2016b) is the ability of the nanoparticle to change pure sliding friction to rolling friction due to reduced interfacial interaction for frictional surfaces and deposition of tribo-film on worn surfaces as well as secondary effect of surface enhancement resulting by the surface polishing effect by nanoparticle. The fatty acids effectiveness in lubricant is another reason. According to Bahari (2017), the strong polarity of fatty acids that exist in vegetable oils also contributes to the formation of mono-molecular layer through the attraction of carboxyl group (COOH) to the metallic surfaces.

3.1.2. Wear behaviour of the bio-lubricants

The wear behaviour of the bio-lubricants was analysed based on wear track width on the disc. The nanoparticle additives improved the wear rate of the bio-lubricant at all load, but as the load and nanoparticle concentration increased from 0.75 wt% to 1.50 wt%, wear rate increases. The wear rate of the bio-lubricant ranged from 0.010 – 0.033 mm^3/Nm which is approximately 6% to 37% reduction compare to the wear rate of crude sandbox oil. This is similar to that of conventional lubricant SAE 40 which ranged from 0.010 – 0.025 mm^3/Nm as shown in Table 5. The reduction in wear rate by the nanoparticle additive according to Abere (2017) may be due to the influence of the chemical nature of the bio-lubricant on film formation. The wear scar diameter (WSD) ranged between 0.00627 and 0.01161 mm as presented in Table 4. In comparison to the conventional lubricant, Abere (2017) and Imran *et al.* (2013) reported mean wear scar diameter (WSD) of 0.050 mm and 0.030 mm respectively for SAE 40. For other bio-lubricant, mean wear scar diameter of 0.067 mm was reported for rapeseed oil and 0.076 mm for rapeseed oil enriched with nanoparticle under the load of 5 N by Abere (2017).

Addition of copper (II) oxide nanoparticle additive to the base oil affected the wear scar on the disc. A relatively smooth surface is produced after testing as shown in Figure 1. The less severe wear on the disc according to Tao *et al.* (2019) is likely due to the ability of the bio-lubricant to form a protective tribo-film. The anti-wear benefits of the nanoparticle additives degrade gradually with increasing concentration. The wear rate of the nanoparticle-enriched bio-lubricant increased when the concentration was beyond the optimum concentration. This according to Battez and Rodriguez (2016) is the granule abrasions that take place due to excess nanoparticle in the contact area. The effects of nanoparticle on wear rate are more influential at higher loads and lower nanoparticle concentration. It was observed that wear rate is increasing with increase in speed and load. However this rate of increase as a function of load is more prominent than with that of speed. As the load and speed gets higher, wear, according to Mohan *et al.* (2014) becomes more than the deposition of nanoparticle formed due to tribo-film formation.

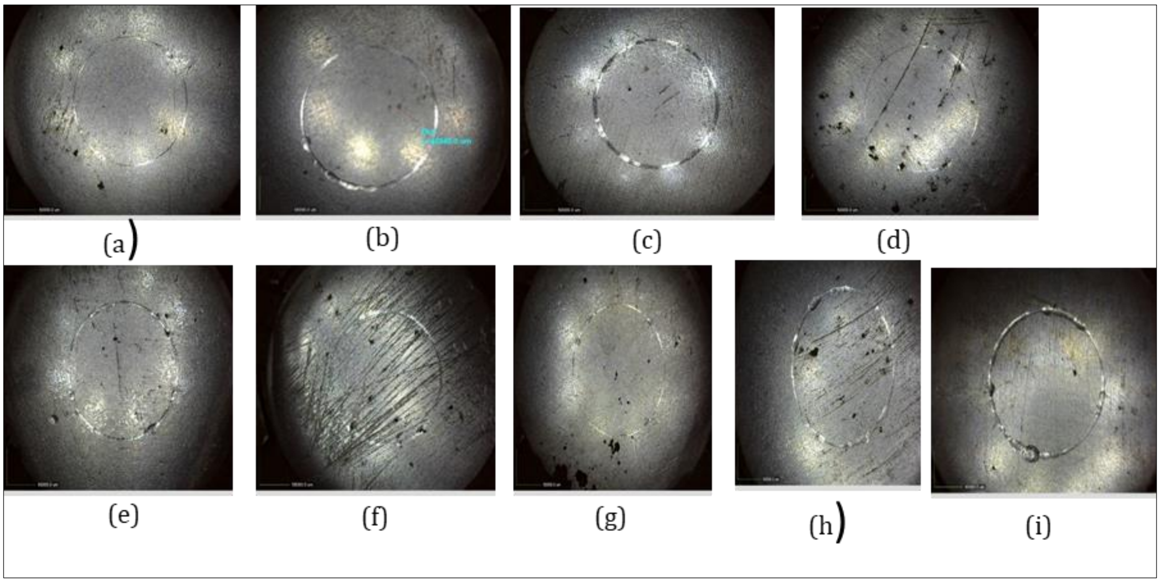

Figure 2 Typical optical micrographs of wear track on the discs

(a). Sandbox oil (2 N) (b). Sandbox oil (5 N) (c). Sandbox oil (8 N)
(d). 0.75 wt% CuO (2 N) (e). 0.75 wt% CuO (5 N) (f). 0.75 wt% CuO (8 N)
(g). 1.50 wt% CuO (2 N) (h). 1.50 wt% CuO (5 N) (i). 1.50 wt% CuO (8 N)

In Figure 2, it was observed that the wear tracks were almost invisible; it is believed that the nanoparticle successfully created a protective layer. This shows an improved wear protection. This, according to Gulzar (2017) may be attributed to the stable and uniform dispersion of nanoparticle in the bio-lubricant as well as the presence of fatty acid that could help the lubricant layer to maintain its thickness and stick well on the surface to reduce metal-metal contact. This anti-wear mechanism may also be attributed to deposition of nanoparticle between interactive surfaces, relatively smaller size of nanoparticle which made them able to penetrate the contact zones more easily and formation of protective layer.

From the analysis of friction and wear, it is clear that nanoparticle-enriched bio-lubricant shows better tribological properties than that of the base oil lubricant itself when applied at the interface of the ball and the disc. These results agreed with the previous researches reported by Zulkifli *et al*. (2013), Ghaednia *et al.* (2015), Battez and Rodriguez (2016), in which nanoparticle additives in base oil possess outstanding anti-friction and anti-wear properties.

3.1.3. Flash temperature parameter (FTP)

The flash temperature parameter, which is the critical temperature below which the lubricant can create a film and withstand without breakdown was calculated for all experimental conditions. The value of FTP was calculated using Equation 1, which shows that FTP value depends on applied load and wear track diameter. Flash temperature parameter is the first point at which oil will start to evaporate. According to Syahrullail *et al.* (2013), the bonding between the lubricants molecules were broke at this point and surface starts to be starve of lubricant. The value of FTP obtained means that the lubricant could maintain the lubricant layer for a longer period of time; the higher the value of FTP, the better the performance of the bio- lubricant.

Generally, the flash temperature parameter increased when the load is increased from 2 N to 8 N, this is due to increase in frictional force with increased load. Similar trends were reported for Jatropha bio-lubricant by Habibullah *et al.* (2014), flash temperature parameter increased as the load increases from 50 N to 150 N. The bio-lubricant had highest flash temperature value of 0.032 at 250 rpm speed, 8 N load and 1.50 wt% nanoparticle concentration as shown in Table 4, which means greater lubricant stability.

In comparison to the conventional lubricant SAE 40, the flash temperature parameter ranged between 0.013 and 0.036 as shown in Table 5. The obtained results agreed with the reported research works of Jabal *et al.* (2014) and Syahrullail *et al.* (2013) in which flash temperature parameter increased as load increases. Masjuki *et al.* (2011) reported flash temperature parameter of 0.086 for conventional lubricant SAE 40 using 5 N load. Habibullah *et al.* (2014) reported flash temperature parameter of 0.0055 and 0.007 for Jatropha oil using 15 kg and 40 kg loads respectively. It is therefore observed that the additions of nanoparticle reduced the probability for lubricant film to breakdown and raised the lubricity overall performance compare to base oil.

3.2. Surface analysis

Surface analysis of the disc specimens was carried out to understand the lubrication mechanism of the lubricant. Scanning electron microscopy was used for analyzing the surface characterization of the tested disc specimens (aluminium alloy) in order to understand the piston ring - cylinder conjunction lubrication mechanism. Figure 3 shows SEM micrographs of the disc specimens tested with copper (II) oxide nanoparticle-enriched bio-lubricant.

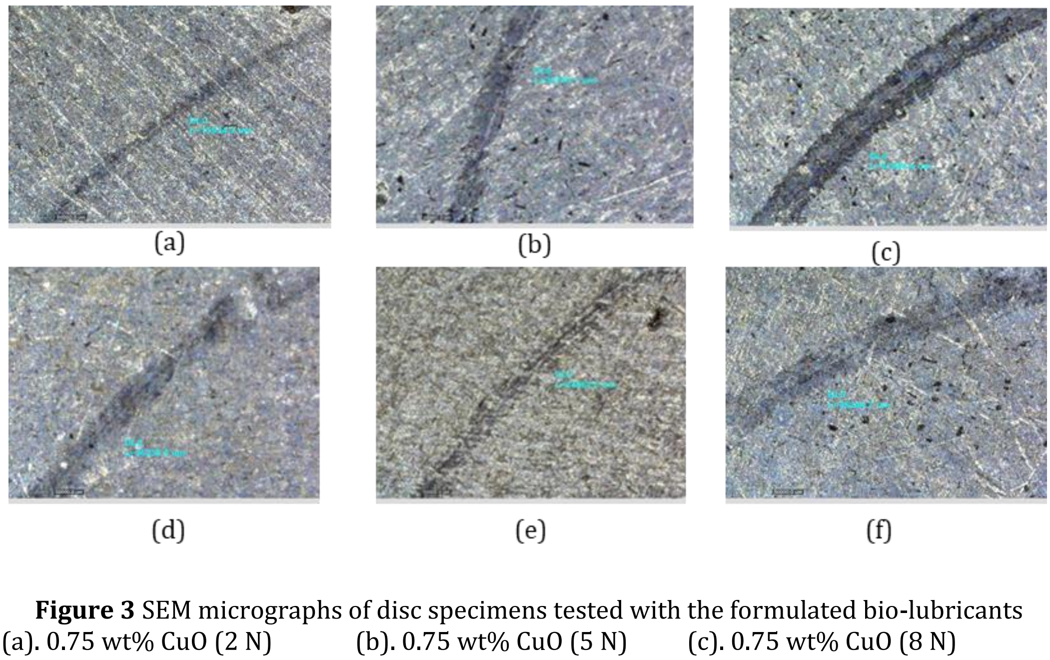

Figure 3 SEM micrographs of disc specimens tested with the formulated bio-lubricants
(a). 0.75 wt% CuO (2 N) (b). 0.75 wt% CuO (5 N) (c). 0.75 wt% CuO (8 N)
(d). 1.50 wt% CuO (2 N) (e). 1.50 wt% CuO (5 N) (f). 1.50 wt% CuO (8 N)

It can be observed from the micrographs that increasing nanoparticle concentration assisted in the surface mending by the nanoparticle deposition on the disc surface. At low concentration of the nanoparticle i.e. 0.75 wt%, higher tendency of wear tracks were observed. Increase in the nanoparticle concentration i.e. 1.50 wt% resulted in improved surface mending effect by filling the tracks. Such an effect of surface enhancement improves the wear protection ability. This improved behaviour of nanoparticle can be related to the tiny size of the nanoparticle as well as uniform and stable dispersion which made it possible to penetrate the rubbing surfaces and filled up the wear scar. This mending mechanism by the material filling using nanoparticle has been reported by many researchers including Gulzar (2017), Bahari (2017) and Abere (2017).

3.3. Optimization of tribological behavior of the bio-lubricant

The response optimizer of the RSM was used to investigate the optimum process parameters for the bio-lubricant production. The target and upper values of responses of bio-lubricant are shown in Table 6. The target parameters were chosen to be the minimum value of the coefficient of friction and wear rate as well as maximum value of flash temperature parameter.

Table 6 Response optimizer for the bio-lubricant

Parameters	Goal	Lower	Target	Upper
COF	Minimum	0.04800	0.04800	0.10600
Wear rate	Minimum	0.00952	0.00952	0.03495
FTP	Maximum	0.00690	0.03460	0.03460

The optimization results are as presented in Figure 4. The predicted operating conditions needed for the input factors to achieve minimum coefficient of friction and wear rate as well as maximum flash temperature parameter were: a load of 5.0909 N, speed of 217.6768 rpm and nanoparticle concentration of 1.1061 wt%. The minimum values obtained for the coefficient of friction and wear rate were 0.0547 and 0.0140 (mm^3/Nm) respectively, while the maximum value of flash temperature parameter was 0.0288.

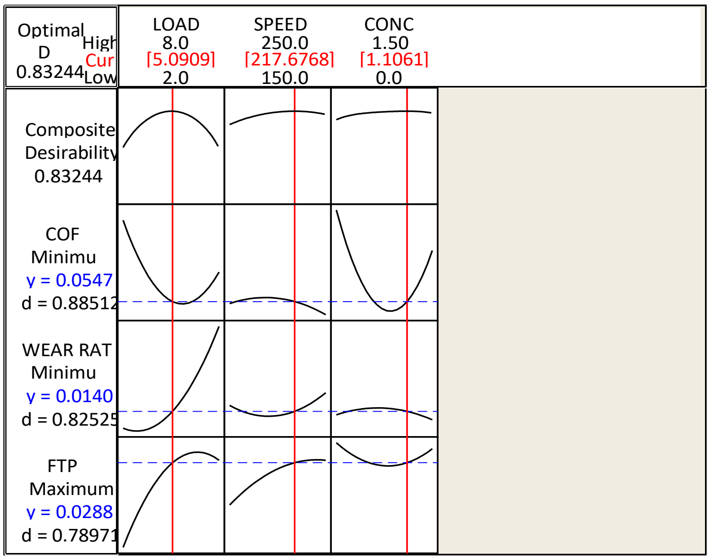

Figure 4 Optimization results for the bio-lubricant

4. Conclusion

This work studied the tribological behaviour of copper (II) oxide nanoparticle-enriched bio-lubricant produced from sandbox (*Hura crepitans*) oil. Sandbox oil is indeed a potential vegetable oil that can be used as alternative lubricant in automotive engine. Besides the advantages of vegetable oil like renewability, bio-degrability and non-toxic, it has shown positive response to both friction and wear resistance when treated with copper (II) oxide nanoparticle additives. The tribological properties of the lubricant were enhanced with the addition of the nanoparticle and its tribological performance depends on the tribological performance of its base oil as well as the concentration of the added nanoparticle. The tribological behaviour of the bio-lubricant showed that the additive improved the flash temperature parameter, reduced the friction and wear rate. The reduction could be due to deposition of nanoparticle between the rubbing surfaces and on the worn surfaces, which in turn decreased the shearing resistance. The value of wear rate, coefficient of friction and flash temperature parameter is the function of speed, load and concentration of the nanoparticle in the bio-lubricant. In comparison to the conventional lubricant SAE 40, the bio-lubricant showed the potential of providing adequate lubrication performance in the engine. The nanoparticle-enriched bio-lubricant had a better anti-wear and friction reducing properties showing promising application as lubricating oils. The overall tribological performance of the bio-lubricant indicates that its potential in practical applications like automotive engines, industrial machines, and others, is possible. The overall analysis also suggests that the sandbox oil has the potential in becoming alternative source of lubricant. In addition, it can be concluded from the results that copper (II) oxide nanoparticle can be used as additive to improve the tribological properties of sandbox oil as bio-lubricant.

Compliance with ethical standards

Acknowledgments

The authors gratefully acknowledge the Federal University Wukari and Ahmadu Bello University Zaria for providing laboratory facilities and personnel required for this research work.

Disclosure of conflict of interest

The authors declare no conflict of interest.

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