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Evaluation of Tribological Properties of Transesterified Cottonseed Oil by Adding Silicon Dioxide (SiO_2) as an Additive[1]

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Abstract
The usage of vegetable oils has increased in many applications as they possess desirable lubrication properties. However, the use is limited due to poor tribological properties. Many researchers have attempted to explore the performance of various vegetable oils and the effect of adding anti-wear additives on tribological properties. In the present work, bio lubricant is obtained from pure cottonseed oil (CSO). Silicon Dioxide, a useful catalyst, is used as an anti-wear additive. Oleic Acid surface-modified Silicon Dioxide (OA-SiO2) is added at 0.25%, 0.5%, 0.75%, and 1% weight concentrations in transesterified cottonseed oil (TCSO). After the addition of OA-SiO2 nanoparticles, the improvement in the lubrication properties has been seen. A four-ball tester is used to measure the coefficient of friction (COF) and wear scar diameter (WSD) of CSO, TCSO, and additive-added TCSO as per ASTM standard D 4172. Friction and wear tests reveal that the TCSO with OA-SiO2 nanoparticles shows better tribological properties. It is observed that WSD and COF of TCSO are reduced by 11.85 % and 24.88 % respectively by adding 0.75 weight % of OA-SiO2 nanoparticles. Present work shows that TCSO, on adding OA-SiO2 additives, can be a strong alternative for mineral oil.

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Keywords

Vegetable oil, cottonseed oil, transesterification, Silicon dioxide, WSD, COF.

1 Introduction

Mineral oil-based lubricants provide a significant environmental risk due to disposal issues. Furthermore, the human species faces other concerns in this area, such as the decrease of fossil fuel resources and climate change. These challenges prompted researchers to consider environmentally friendly alternatives to mineral oil-based lubricants. The use of edible or non-edible vegetable oils is the solution to the mentioned problem because they have many required features including high viscosity index and flash and fire points, nontoxic in nature, good lubricity, renewability, eco-friendliness, and so on. Jayadas (2008) described that, because of weak oxidation stability, a narrow viscosity range, and poor tribological properties, their use is restricted. Various vegetable oils, such as maize oil, palm oil, soybean oil, sunflower oil, coconut oil, rapeseed oil, rice bran oil, rubber seed oil, and others, have been proposed and explored as lubricant basis stocks [Jayadas and Nair, 2006; Wu, 2000; Rani, 2015; Castro, 2006; Aravind, 2015]. India has a lot of potential for the production of edible and non-edible vegetable oilseed. These oilseeds have the potential to be a great source of vegetable oil which can be used for producing lubricants. If the thermal and lubrication properties of vegetable oil-based lubricants are improved, they can be used to substitute mineral oil-based lubricants. To increase the tribological properties of vegetable oil-based lubricants, various types of metal nanoparticles and metal oxides were used. To improve the lubrication properties, vegetable oils have to be chemically modified by transesterification or epoxidation along with the addition of different anti-wear and extreme pressure additives. The friction and wear characteristics are greatly influenced by additives that are additionally included in the base oil. These additives in base oil tend to improve the characteristics of oils. Liu *et. al.* (2004) found that, due to their small size nanoparticles have a mending effect by filling surface roughness points and developing a thin tribo film that, bears the load and also isolates the rubbing surfaces during the frictional process. Peng *et. al.* (2010) describe in their study that nanoparticles can act as insertions between friction surfaces, converting pure sliding friction to mixed sliding and rolling friction. Lee *et. al.* (2009) investigate that, lowering the roughness of the rubbing surfaces, the abrasiveness of hard nanoparticles may have a polishing effect. Dispersing the nanoparticles in the liquid base oil is one of the most difficult aspects of developing a stable nano lubricant formulation. Surfactant ultrasonic dispersion is also effective in stable nano lubricant formulation. Organic and inorganic nanoparticles have been employed as lubricant additives by researchers which show improvement in the lubricant properties [Deorsola, 2012; Padgurskas, 2013; Zhang, 2011; Park, 2011; Jiao, 2011]. Mahipal *et. al.* (2014) worked on Zinc-dialkyl-dithiophosphate (ZDDP) and it shows good tribological performance when added to Karanja oil as compared to 20W40 engine oil. Surywanshi and Pattiwar (2018) reported that the viscosity of the lubricant is enhanced due to the addition of TiO_2 nanoparticles and frictional coefficient and wear scar diameters of balls in the lubricants are reduced in the range of 6-26 and 2-7 percent, respectively. Hongmei Xie *et. al.* (2016) adopted MoS_2 and SiO_2 as additives and showed better results in improving the tribological properties of mineral lubricating oil, EOT5. SiO_2 nanoparticles are also inexpensive and readily available. Peng *et. al.* (2010) examined the tribological properties of liquid paraffin with oleic acid surface-modified SiO_2 nanoparticles, and it was found that optimum concentrations of SiO_2 nanoparticles showed better performance in tribological properties than pure paraffin oil. It was also found that the performance of SiO_2 nanoparticles in tribological properties was dependent on nanoparticle size with 58 nm diameters showing a higher reduction in friction and increase in load-carrying capacity and anti-wear property than pure paraffin oil. Silicon dioxide (SiO_2) is suitable as a catalyst and semi-conductive material and is widely used in different fields.

2 Material and Sample Preparation

2.1 Selection of base oil and additive

Many researchers have worked on different vegetable oils. It has been proved that there is potential in vegetable oil-based lubricants. Edible and non-edible vegetable oils possess some better physio-chemical properties.

From the literature survey, it is observed that different vegetable oils have the potential to be a great source of vegetable oil-based lubricants. Cotton seed oil is one of them. Cotton seed is non-edible oil and can be used for the production of bio-lubricants. Cotton seed oil is widely available in India, primarily in Gujarat and Maharashtra. It is also less inexpensive as compared to other oils. The work reported in this paper attempts to check the probability of replacing mineral oil-based lubricants with cotton seed oil-based lubricants. Silicon dioxide nanoparticles are inexpensive and readily available. Silicon dioxide (SiO_2) is suitable as a catalyst and semi-conductive material and is widely used in different fields.

In this research work, Oleic acid surface-modified Silicon dioxide (OA-SiO_2) nanoparticles with different concentrations were used as an additive. The surface of SiO_2 particles was modified using the method explained in the various papers [Ma, 1999; Chen, 1999]. The physical and chemical properties of nano silicon dioxide are shown in Table I.

Table I. Various properties of SiO_2 nanoparticles

Sr. No.	Parameter	Values
1	Size	40 nm
2	Size Range	30-50 nm
3	Molecular Weight	60.08 g/mol
4	Color	White
5	Density	2.4 g/cm^3
6	Melting Point	1713 °C
7	Boiling Point	2950 °C
8	Morphology	Spherical
9	Nature	Amorphous
10	Purity	99.9 %

2.2 Transesterification

Cotton seed oil (CSO) is taken as base oil and its physicochemical properties were tested in the CHEMTECH Laboratories, Pune. CSO cannot be desirable to use in the pure form because of their poor tribological properties. Its properties can be enhanced by chemical modification and using suitable additives. Cotton seed oil is converted to fatty acid methyl esters (FAME) using a methanol transesterification process. In the first stage of the transesterification process, 25 ml methanol, and as a catalyst 5 gm sodium methoxide, was added in 125 ml cotton seed oil. The mixture was heated for 30 min. The biodiesel (methyl ester) obtained was with glycerin. The obtained biodiesel (methyl ester) was treated with 7.5 gm silica gel for 30 min to remove soap. Two layers are obtained with which upper layer of biodiesel (methyl ester). The sample was filtered with filter paper to obtain biodiesel (methyl ester). The rich biodiesel was further washed with deionized water to remove unreacted oil and dried overnight in an oven at 105^0C to remove the moisture completely. The biodiesel (methyl ester) obtained by the first stage of transesterification was tested for the Fatty Acid Methyl Ester (FAME) test. The other properties like density, kinematic viscosity, flash point, and gross calorific value were tested for confirmation of the process of transesterification. In the second stage of the process, 25 gm of Trimethylolpropane (TMP) was added stepwise into a small amount (40 ml) of the obtained biodiesel with the aid of heating at 75^0C and stirring to melt the crystalline solid. The mixture is heated at 125^0C for 20 min and the remaining amount of biodiesel (60 ml) is added. The mixture was kept for reaction; the reaction duration was 3 hours. Ethyl acetate (10 ml) was added and the mixture was heated for the next 1 hour. After the reaction was completed, the reaction mixture was cooled to atmospheric temperature. The mixture was filtered to remove the catalyst and solid materials followed by fractional distillation.

2.3 Preparation of Oleic Acid surface-modified SiO_2 nanoparticles

The SiO_2 nanoparticles used as anti-wear nanoparticle additives are purchased from Nano Shell, India. The oleic acid is used as a surfactant that modulates the available surface energy of the particles so that the surface tension decreases, allowing more particles to escape the aggregation process. Continuous stirring was used to combine oleic acid and n-hexane, and then a predetermined amount of SiO_2 nanoparticles; accurately weighed using a precision digital electronic balance was added. Then the mixture was heated at 60^oc for 4 hours, under vigorous stirring. After that, the solution was filtered with filter paper, and the precipitate was washed with a solvent mixture of alcohol and de-ionized water. For drying, the precipitate was kept in a vacuum desiccator, for 48 hours. The oleic acid surface-modified SiO_2 (OA-SiO_2) nanoparticles were produced as a white powder. Field emission scanning electron microscope (FESEM) and energy dispersive X-ray spectrometers (EDS) were used to inspect the surface morphologies of OA-SiO_2 nanoparticles.

2.4 Sample Preparation

The test samples were prepared by adding surface-modified OA-Silicon Dioxide (OA-SiO_2) each of size 30-50 nm, as an anti-wear additive at 0.25%, 0.5%, 0.75%, and 1% weight concentrations in transesterified cottonseed oil (TCSO). A sample of 20 ml is prepared. A precision digital electronic balance is used to measure the weight of the OA-SiO_2 nanoparticles. Ultrasonic Probe Sonicator and Magnetic Stirrer are used for uniform dispersion and good suspension stability. The additives are thoroughly mixed in the TCSO after 30 minutes of ultra-sonication, followed by one hour of magnetic stirring at room temperature. The light transmittance effect is considered to study the dispersibility of OA-SiO_2 nanoparticles in the TCSO. The TCSO containing OA-SiO_2 showed better light transmission than the CSO and TCSO without OA-SiO_2 nanoparticles. Even though after a few months, there were no sediments observed.

The abbreviations of different samples used for experimentation are as given in Table II.

Table II: Abbreviations of Samples

Sr. No.	Testing Samples
A1	CSO
A2	TCSO
A3	TCSO +0.25 % by wt of OA+SiO_2
A4	TCSO +0.50 % by wt of OA+SiO_2
A5	TCSO +0.75 % by wt of OA+SiO_2
A6	TCSO +1.00 % by wt of OA+SiO_2

3 Experimentation

A calibrated Ducom TR 30 L four-ball tester is used to examine the tribological parameters of CSO, TCSO, and additive-added TCSO. The wear scar diameter (WSD) and coefficient of friction (COF) are the measurable parameters considered here for the test. By comparing the wear scar diameter of all samples, the amount of wear is evaluated. The hardness of the balls used in the four-ball tester is 64-66 HRC and the diameter is 12.7 mm. The balls are made of chrome steel alloy (AISI standard steel no. E-52100). The balls are cleaned with acetone before the test. The wear tests are carried out by ASTM D 4172 standard test method for anti-wear characteristics of lubricating oil where the load is 392 ± 2 N, speed is 1200 rev/min, test duration is 60 min, and a test temperature of 75± 2°c. All the experiments were carried out three times, and then the average values were recorded. The WSD of bottom balls is measured and examined using the data acquisition system. The balls are smoothly cleaned with acetone before analyzing worn surfaces. To remove the traces of cleaning agents from the scar area, the balls were dried carefully.

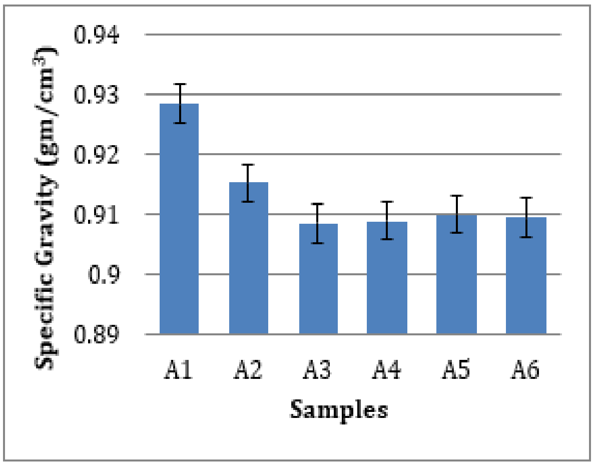
4 Results and Discussion

4.1 Effect of Transesterification

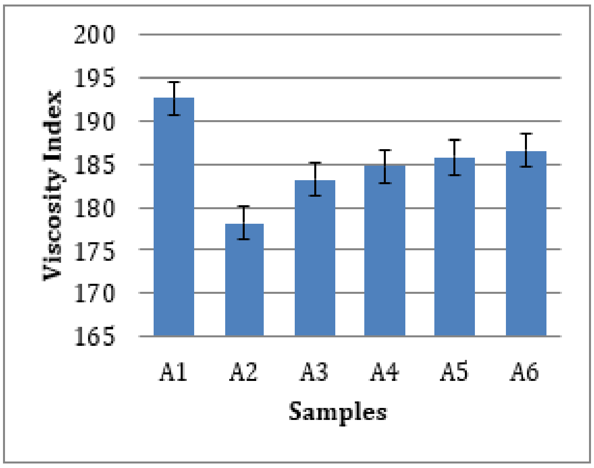
Pure cotton seed oil (CSO) and transesterified cottonseed oil (TCSO) were analyzed for their lubrication properties. The lubrication properties of CSO, TCSO, and of four test samples were tested as per ASTM standards and listed in Table III.

Table III: Lubrication properties of oil Samples

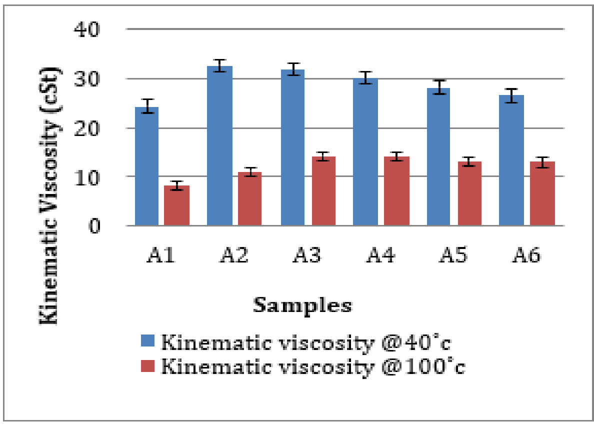
Oil → Properties ↓	Cottonseed Oil	TCSO	TCSO +0.25 % OA-SiO_2	TCSO +0.50% OA-SiO_2	TCSO +0.75 % OA-SiO_2	TCSO +1.00% OA-SiO_2	Test Method
Specific gravity	0.9285	0.9153	0.9085	0.9090	0.9100	0.9095	ASTM D 4052 2016
Kinematic viscosity @40˚c	24.33	32.66	33.83	35.12	37.19	39.55	ASTM D 7042 2016
Kinematic viscosity @100˚c	8.27	11.11	12.16	14.03	16.21	18.96	ASTM D 7042 2016
Viscosity index	192.67	178.17	183.20	184.80	185.78	186.59	ASTM D 2270 2016
Pour point ˚c	-6	-9	-22	-24	-25	-26	ASTM D 97 2017 b



(a)



(b)



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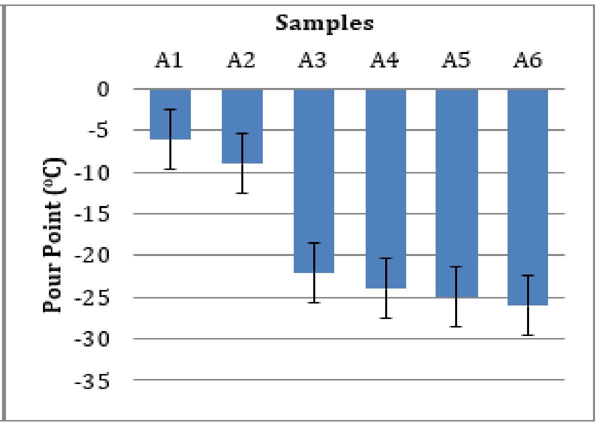


(d)

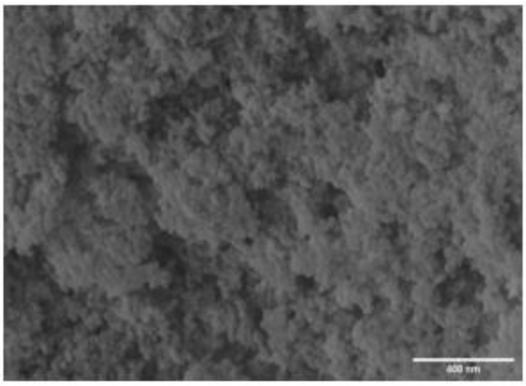
Figure 1: Change in the Lubrication properties, (a) Specific Gravity, (b) Kinematic Viscosity, (c) Viscosity Index and (d) Pour point.

From the above Table III and Figure 1, it has been observed that because of the transesterification process, viscosity, one of the most important properties of the oil, is improved at 40˚C and 100˚C. This improvement in viscosity is about 34 %. Change is not observed in the specific gravity, whereas the pour point is improved from -6 ˚c to -9 ˚c. A small reduction of 7.52 % is observed in the viscosity index. The viscosity of TCSO improves as the length of the hydrocarbon chain of the carboxylic acid or alcohol in ester bio-lubricants increases. The viscosity index is altered as hydroxyl groups are present in the fatty acid or as polyols added via esterification due to enhanced hydrogen bond interactions. After the addition of OA-SiO_2 the improvement in the lubrication properties can be seen. The viscosity improves at 40ºC as well as at 100ºC as the concentration of additive increases. This improvement is in the range of 3.8 % to 21 %. The viscosity index shows improvement slightly in the range of 2.8 % to 4.7 %.

4.2 Morphology of SiO_2 nanoparticles

Figure 2 shows, the FESEM micrograph and the qualitative analysis of SiO_2 nanoparticle composition using EDS. The micrograph of SiO_2 nanoparticles shows their spherical shape and uniform distribution of their sizes. EDS shows that the nanoparticles comprise Si and O elements.

By coating SiO_2 nanoparticles with Oleic Acid, the aggregation of these nanoparticles in TCSO is reduced.



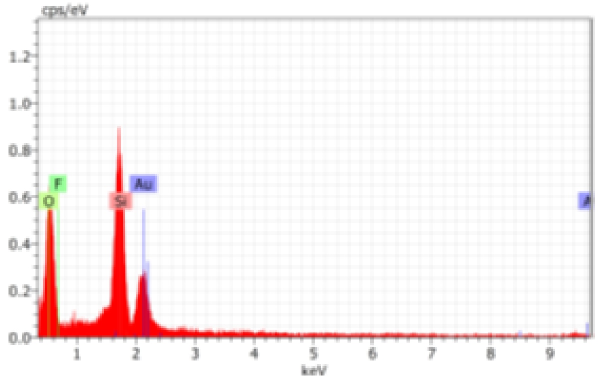


Figure 2: FESEM image and EDS spectrum of SiO_2 nanoparticles

4.3 Worn Surface Analysis

The form and nature of wear that occurred on the bottom balls tested in a four-ball tester with varied compositions of oil are studied with FESEM. A data acquisition system is used to measure the WSD of the ball specimens. The mean wear scar diameter is 866, 515, 592, 574, 505, and 525 micrometers for CSO, TCSO, and four samples after adding OA-SiO_2 with 0.25, 0.5, 0.75 and 1 weight %.

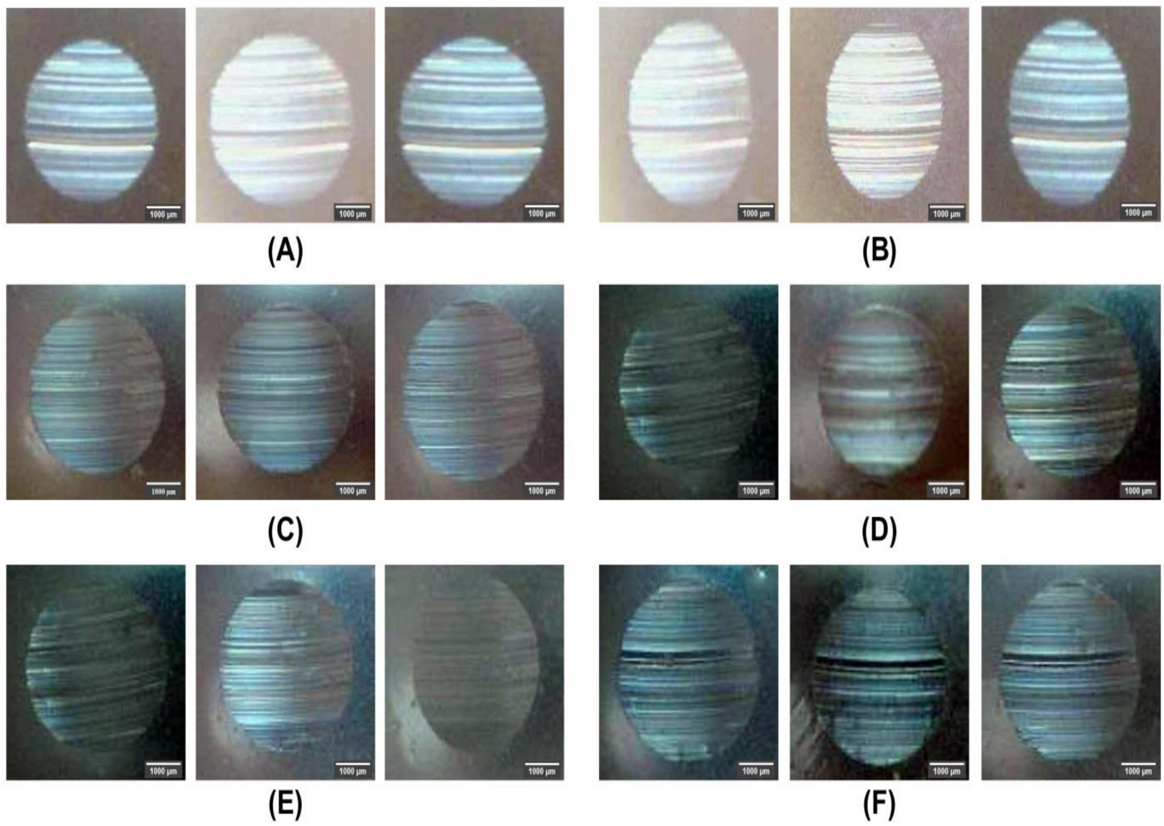


Figure 3: Wear Scar images of the worn surface of the balls lubricated with (A) CSO, (B) TCSO, (C) TCSO+0.25 % OA- SiO_2 (D) TCSO+0.50 % OA- SiO_2 (E) TCSO+0.75 % OA- SiO_2 and (F) TCSO+1.00 % OA- SiO_2

Figures 3 (a), (b), (c), and (d) describe the WSD measured by the data acquisition system for all samples. Grooves can be seen in all samples when scar pictures are examined, showing the presence of abrasive wear. Few pitting and spalling are also detected in the worn region because of metal-to-metal contact. The WSD, furrows, and spalling have been reduced to a minor amount with the addition of 0.75 weight % OA-SiO_2 nanoparticles in TCSO, which could be because of the mending effect and protective film coating by nanoparticles on the worn region. It is observed that the coefficient of friction is 0.0872, 0.0868, 0.0812, 0.0832, 0.0663, and 0.0758 for all the test samples.

The COF and WSD of CSO, TCSO, and TCSO combined with various weight % of OA-SiO_2 nanoparticles are shown in Figure 4 (a and b).

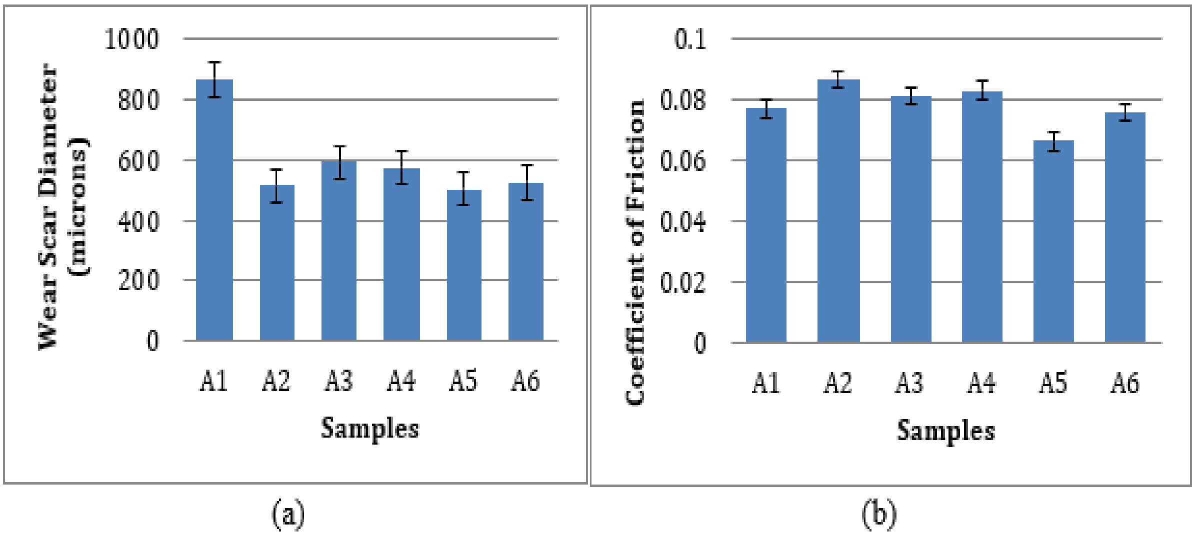


Figure 4: Change in (a) Wear Scar Diameter and (b) Coefficient of friction of samples

COF and WSD are both reduced by adding OA-SiO_2 nanoparticles into TCSO. WSD of TCSO is reduced by 11.85 % when 0.75 weight % of OA-SiO_2 nanoparticle is added. The COF is lowered by 24.88 percent when 0.75 weight percent of OA-SiO_2 nanoparticles are added. This improvement in tribological properties could be because of the suitable size of SiO_2 nanoparticles, which help to reduce friction and wear through mending, rolling, and creating a surface protective film. Higher weight % (> 0.75 weight %) of OA-SiO_2 nanoparticles, results in increased friction and wear. Beyond 0.75 weight % concentration, the nanoparticles of OA-SiO_2 may clump together and form large-size particles, which may not be able to fill the dents and troughs closely, which results in increased friction and wear. Hence in the present study, it is identified that 0.75 weight % concentration of nanoparticles in TCSO is optimum for minimum wear, and this combination reports better tribological properties than that of pure TCSO. However, as compared to commercial lubricants, the value of WSD obtained is slightly higher. This study shows that there is a scope for the use of cottonseed oil as a lubricant by improving its tribological properties by adding suitable additives.

5 Conclusion

The enhancement in the tribological properties of cottonseed oil has been studied after process of transesterification and using oleic acid surface-modified silicon dioxide nanoparticles (OA-SiO_2). From this study, the following conclusions have been drawn.

- It is observed that because of the transesterification process, the viscosity of the oil is improved by 34 % whereas, a small reduction of 7.52 % is observed in the viscosity index.
- After the addition of OA-SiO_2 nanoparticles, the improvement in the lubrication properties can be seen. The viscosity improves at 40ºC as well as at 100ºC as the concentration of additive increases. This improvement is in the range of 3.8 % to 21 %. The viscosity index shows improvement slightly in the range of 2.8 % to 4.7 %.
- The stability of OA-SiO_2 nanoparticles in the transesterified cottonseed oil is significantly observed due to excellent dispersivity.
- The wear tests carried out on a four-ball tester show that the transesterified cottonseed oil lubricant with OA-SiO_2 nanoparticles exhibits superior tribological properties.
- It is identified that minimum WSD and COF are obtained for the 0.75 weight % concentration and 30-50 nm size of nanoparticles in TCSO. By addition of 0.75 weight % of OA-SiO_2 nanoparticles, the WSD is reduced by 11.85 %, and COF is reduced by 24.88 %.

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