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RESEARCH

Experimental Study on Tribological, Rheological and Bio-degradability Characteristics of Canola Oil with TiO_2 Nanoparticles as Bio-nanolubricants

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A B S T R A C T

In response to growing environmental concerns about the adverse effects of conventional lubricants, there has been a surge in the demand for eco-friendly alternatives. Biodegradable lubricants, crafted from renewable sources, represent a sustainable solution to address these environmental apprehensions. This research describes the tribological, rheological properties and biodegradability of rapeseed oil with 0.25 to 1.25 weight percent TiO_2 nanoparticles as additives. The lubricant samples were prepared using a magnetic stirrer and an ultrasonic device. Friction and wear properties such as coefficient of friction and wear scar diameter were determined using a four-ball tester. In comparison with SAE20W40, samples 1 (0.25 weight percentage of TiO_2) and sample 2 (0.5 weight percentage of TiO_2) shown remarkably improved tribological performance, with 73.32% and 66.76% decreases in coefficient of friction (COF), respectively. Shear stress, shear rate and dynamic viscosity are analyzed using a rheometer. The acquired results are juxtaposed with both pure canola oil and various mineral oils for comparison. The nano-lubricant synthesized in this study emerges as a viable alternative to SAE10, DXT3 (steering fluid), and SAE20W50 grade oils, demonstrating lower friction and a reduced wear scar diameter. Biodegradability tests were performed on all generated samples using the Biochemical Oxygen Demand (BOD) apparatus, and all samples exhibited a BOD to Chemical Oxygen Demand (COD) ratio better than 0.5, indicating biocompatibility. The findings of the research investigation indicate that the combination of canola oil with TiO_2 blends holds significant promise as an alternative to conventional synthetic lubricant oils.

1. INTRODUCTION

Lubricants, crucial by-products of crude mineral oils, are essential for the optimal operation of equipment, machinery, and tools in diverse industries. Widely used in mechanical systems like engines and gears, they minimize friction, wear, and heat, enhancing equipment lifespan. In addition to friction reduction, they provide protective layers against corrosion and rust, contributing to equipment longevity. The widespread use of conventional lubricants, often toxic to soil and aquatic life, poses environmental challenges. Global oil crises in 1973 and 1979 have fuelled the search for alternatives to crude oil. Despite a limited market, biodegradable lubricants find applications in marine oils, outboard motor lubricants, agriculture, food sector lubricants, and subterranean storage oil [1]. The environmental impact of mineral oil-based lubricants contributing to global warming emphasizes the need for bio lubricants. Derived from vegetable oil, these lubricants offer superior biodegradability, low-temperature stability, and an elevated viscosity index, presenting a viable, less toxic, and environmentally friendly alternative [2]. TiO_2 nanoparticles, renowned for high hardness and thermal stability, play a pivotal role in enhancing lubricant lifespan [3].

The tribological properties of nano-lubricants are influenced by critical factors like nanoparticle size, concentration, and shape, with surface modification offering efficient stabilization [4]. Vegetable oils exhibit a high viscosity index and can be employed in Extreme Pressure (EHL) applications due to their exceptional pressure-viscosity coefficient. Coconut oil boasts the highest solidification temperature [5]. Sunflower oil, enriched with SiO_2 and TiO_2, demonstrates shear-thinning and shear-thickening behaviors, respectively, along with reduced wear volume loss and coefficient of friction [6]. The study highlights a significant rise in the coefficient of friction and wear due to the addition of nanoparticles to vegetable oil lubricants, with the noteworthy observation that an increased load on the lubricant sample containing nanoparticles correlates with a reduction in the wear scar diameter [7]. Research on nano-lubricants with ZnO, CuO, and ZrO_2 nanoparticles demonstrates enhanced performance in reducing the coefficient of friction and wear, especially with CuO nano-lubricant exhibiting superior tribo-film formation and lower wear, influenced by temperature variations [8,9]. The analysis suggests that incorporating CuO + FeO_3 nanoparticles into lubricating oil enhances its physical and tribological properties [10]. Sunflower oil, outperforming soybean oil categorized under SAE 75W, proves highly effective as a helical gear lubricant, demonstrating efficacy in higher temperatures despite suboptimal low-temperature qualities and a broad viscosity range [11]. Sunflower seed oil, with its biodegradability, wear prevention capability, and lubricating properties, emerges as a potential substitute for conventional biolubricants [12].

Friction, a significant contributor to energy losses in machines, is addressed through lubrication, commonly known as tribo-film, which introduces a friction-reducing film between moving surfaces [13]. Recent advancements in nanotechnology have spurred interest in nanoparticle additives, known for their quantum-size effect, small-size effect, and surface/interface effects, making them ideal candidates [14]. Vegetable oils with nanoparticles outperform mineral oils in terms of viscosity index, flashpoint, evaporation loss, and lubricity, ultimately leading to superior energy efficiency [15]. Lubricant oils containing nanoparticles significantly reduce friction coefficients and enhance load-bearing capacity, with proposed lubrication processes, such as the ball bearing effect, protective coating, mending effect, and polishing effect, as depicted in Fig. 1.

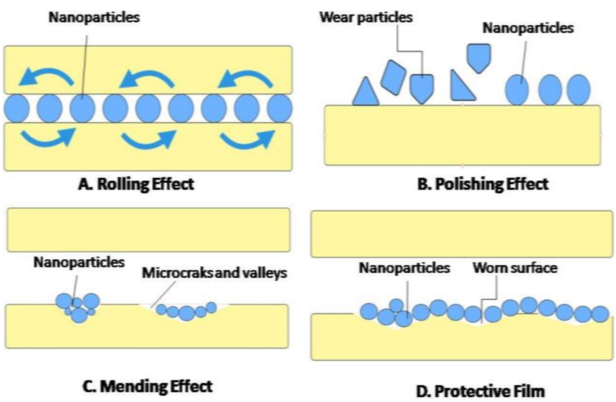

Fig. 1. Nanoparticle effect at the lubricant surface.

Suspended nanoparticles not only act as ball bearings but also form a protective film and restore lost mass, contributing to a more sustainable future [16-18]. Biolubricants possess the natural capacity to undergo gradual biodegradation, transforming into harmless components upon environmental release. This intrinsic quality not only aids in mitigating climate change but also plays a crucial role in promoting a more sustainable future [19]. Vegetable oils, whether used independently or in conjunction with synthetic lubricants, demonstrate versatile applications, with their performance being notably influenced by the addition of nanoparticles, resulting in lower friction coefficients and cutting temperatures [20]. The addition of nanoparticles, such as ZnO, CuO, ZrO_2, and TiO_2, to lubricants results in an increase in wear rate, friction coefficient, and viscosity. Notably, the incorporation of TiO_2 nanoparticles stands out due to their ability to prolong the life of lubricants, attributed to their high hardness, exceptional temperature stability, and chemical inertness [21].

This innovative study initiates an investigation into the unparalleled impact of TiO_2 concentration on the tribological, rheological, and biodegradability features of canola oil. Significantly, this research highlights the importance of canola oil's substantial oleic acid content (63%), a predominant monounsaturated fatty acid critical for its role as a base oil in developing biodegradable greases and lubricants. The oil's heightened stability and oxidability, surpassing that of other vegetable oils, further underscore its appropriateness for lubricant formulation [22]. Canola oil, rich in α-linolenic acid (ω-3) and linoleic acid (ω-6), stands out as an ideal option for applications needing specific pour point and properties at low and medium temperatures, supported by its widespread consumption and ready availability at a reasonable price worldwide [23]. The study's novel approach aims to comprehensively assess the unique blend of canola oil with varying TiO_2 concentrations, focusing on their impact on viscosity, shear stress, coefficient of friction, wear scar diameter, and biodegradability. This research holds promise for advancing lubricant performance and environmental sustainability, offering the potential to enhance efficiency and ecological friendliness.

2. EXPERIMENTION

2.1 Test materials

In the current study, TiO_2 nanoparticles without any surface modifications from Shilpa Enterprises (India) were dispersed in commercially available canola oil (Borges Agricultural & Industrial Edible oils, Spain) in different concentrations to formulate the nano-lubricants. The material properties of the lubricant and selected nanoparticles are shown in Table 1.

Table 1. Material properties.

Material	Properties
Canola oil	Density (40^0C): 0.92 g/cm^3 Viscosity (20^0C): 78.2 mPa.s Specific heat: 1.94 J/g Flash Point: 326^0C
Titanium dioxide nanopartices	Chemical formula: TiO_2 Purity: 99.9% Average Particle size: 30–50 nm Morphology: Spherical Surface area: 150 m^2/g Bulk density: 0.35 g/cm^3 Melting point: 1843^0C

2.2 Nano-Lubricants Preparation

The canola oil and TiO_2 nanoparticle blend is prepared by using magnetic stirrer using 500 ml beaker, stirred for about 1 hour at 1200 rpm and at room temperature in different compositions to formulate the nano-lubricants. Prepared samples were further sonicated, followed by ultrasonication for 30 minutes with a 120-Watt ultrasonicator with a frequency of 20 kHz to get a uniform dispersion and good suspension stability [24-26]. Four samples were prepared with various weight proportions of canola oil and TiO_2 nanoparticle as mentioned in Table 2. Both magnetic stirred and ultrasonicated components were stored for ten days for visual evaluation. After 10 days, it has been observed that there is no settling or layer formation in a magnetic and ultrasonicator stirred oil blend.

Table 2. Canola oil + TiO_2 compositions.

Sl no.	Weight percentage	
	Canola oil	TiO_2
1	99.75	0.25
2	99.50	0.5
3	99	1
4	98.75	1.25

2.3 Tribological characterization

Tribological tests were carriedout in accordance with ASTM D 4172 on Canola oil comprising TiO_2 nanoparticles using a four ball tribometer with friction torque ranging from 0- 16 Nm and load ranging between 100-10,000 N [27-30]. The 12.7 mm in diameter, mirror-finished, 64–66 HRC chromium steel balls from SKF were used for testing. A 40 kg weight was applied at 1200 rpm during the tests. A temperature of 25˚C was used to investigate the wear-preventive behavior of canola oil with TiO_2 samples. The coefficient of friction and wear scar diameter were determined by employing an optical microscope alongside the four-ball testing method. Upon the conclusion of the one-hour experiment, the wear characteristics of the steel ball bearings were assessed and analyzed using an optical microscope with high resolution and magnification set at 50x. The wear scar diameters were assessed using micrographs, followed by the utilization of the ASTM D 4172 standard to compute the average value of these diameters [30].

2.4 Rheological study

The rheological test employs the Anton-Paar MCR-302 Rheometer, which enables rheological measurements across a torque range spanning from 0.5 nN·m to 230 nN·m [32]. In the study, a parallel plate with a diameter of 25 mm and a gap of 1 mm is employed, with the shear rate ranging from 0.1 to 1000 s^{-1}. The test duration maintains a constant temperature of 25°C throughout.

2.5 Biodegradability test

The BOD number of biolubricants quantifies the amount of oxygen required by microorganisms to degrade these substances. As the BOD number increases, the demand for oxygen also rises, serving as a measure of the oxygen quantity necessary for microorganisms to degrade a biolubricant sample over a period of time. It usually takes 5-6 days to complete the BOD test [33].

Test samples for BOD were made by combining 1 ml of the 0.01 dilution sample, 10 ml of $K_2Cr_2O_7$, 19 ml of distilled water, and 30 ml of concentrated H_2SO_4. This mixture is referred to as the blank solution, and it is titrated until the clear yellow solution turns wine red using ferroin indicator, following which it is kept in the digestion unit for two hours. The COD and the volume of sample required to achieve BOD were calculated as a result of the titration process. A 20 ml portion of the produced samples were mixed with pond water containing microorganisms in separate incubation bottles, which were then sealed to prevent exposure to light. To the prepared incubated bottles, more ingredients were added: 1 ml of $MnSO_4$, concentrated H_2SO_4, and alkaline iodide azide. Starch serves as an indicator during the titration of these samples with sodium thiosulfate, continuing until a colorless solution is obtained [34]. The final stage involves obtaining BOD after the samples were incubated for five days at 27^0C .

3. RESULTS AND DISCUSSIONS

3.1 Friction characteristics

Coefficient of friction is calculated using the formula

$$\mu = F/N$$

where N is the applied normal force, μ is the coefficient of friction, and F is the friction force as determined by the tribotester's integrated force sensor. The average coefficient of friction for all the four samples were found to be approximately 0.0512. It was noted that SAE20W40 demonstrated a significantly higher coefficient of friction. One of the main causes of the increased COF is that the oil's viscosity significantly decreases at higher temperatures and speeds, increasing the relative shearing between the lubricating film and the top rotating ball. This finding was also confirmed by the study conducted by Dinesh et al. [34]. From the Fig. 2 it is evident that the sample 4 exhibited an enhanced high value COF as compared to SAE10W30. This might be attributed to the higher concentration of TiO_2 nanoparticles, which could enhance the breakdown of the lubricant film, resulting in a higher coefficient of friction. The COF value observed in this study surpasses that of lubricants containing copper oxide and zirconium dioxide nanoparticles as reported by Wang et al. [35]. It demonstrated the lowest coefficient of friction under typical operating conditions [36]. Sample 1 blend has an exceptionally low COF when compared to the other oil samples under investigation. This may be attributed to the low concentration of TiO_2 nanoparticles.

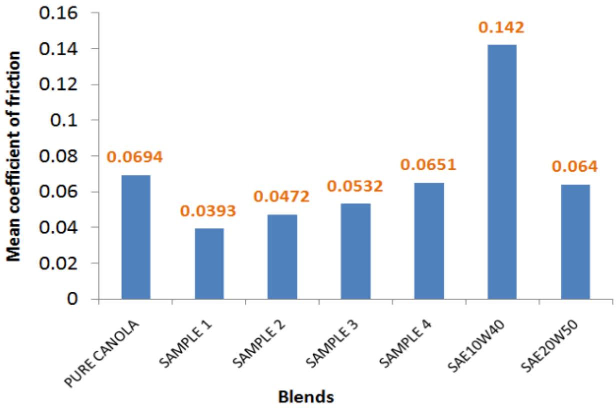

Fig. 2. COF variation with various blends.

A reduction of approximately 72.32% in the coefficient of friction was noted for sample 1, and a decrease of 66.76% was observed for sample 2 compared to SAE20W40. Likewise, there was a reduction of about 38.59% for sample 1 and 26.25% for sample 2 relative to SAE20W50. Under standard conditions, the blend of samples 1 and 2 exhibits better film thickness holding capabilities, which was confirmed by the attainment of a minimized COF. It has been also observed that the sample 4 with highest percentage of TiO_2 nanoparticles (1.25 wt %) exhibited a 1.71% increment of COF in comparison to SAE20W50. This could be due to the presence of more TiO_2 nanoparticles. It can also be concluded that the viscosity of the blends increases with the addition of a certain weight percentage of TiO_2.

3.2 Wear characteristics

It is critical to investigate the surface characteristics of steel balls in order to determine the wear that occurred during the investigation. In addition to the asperity level connections, TiO_2 nanoparticles also carry some of the load when the lubricant layer between interacting surfaces thins and the lubrication regime shifts.

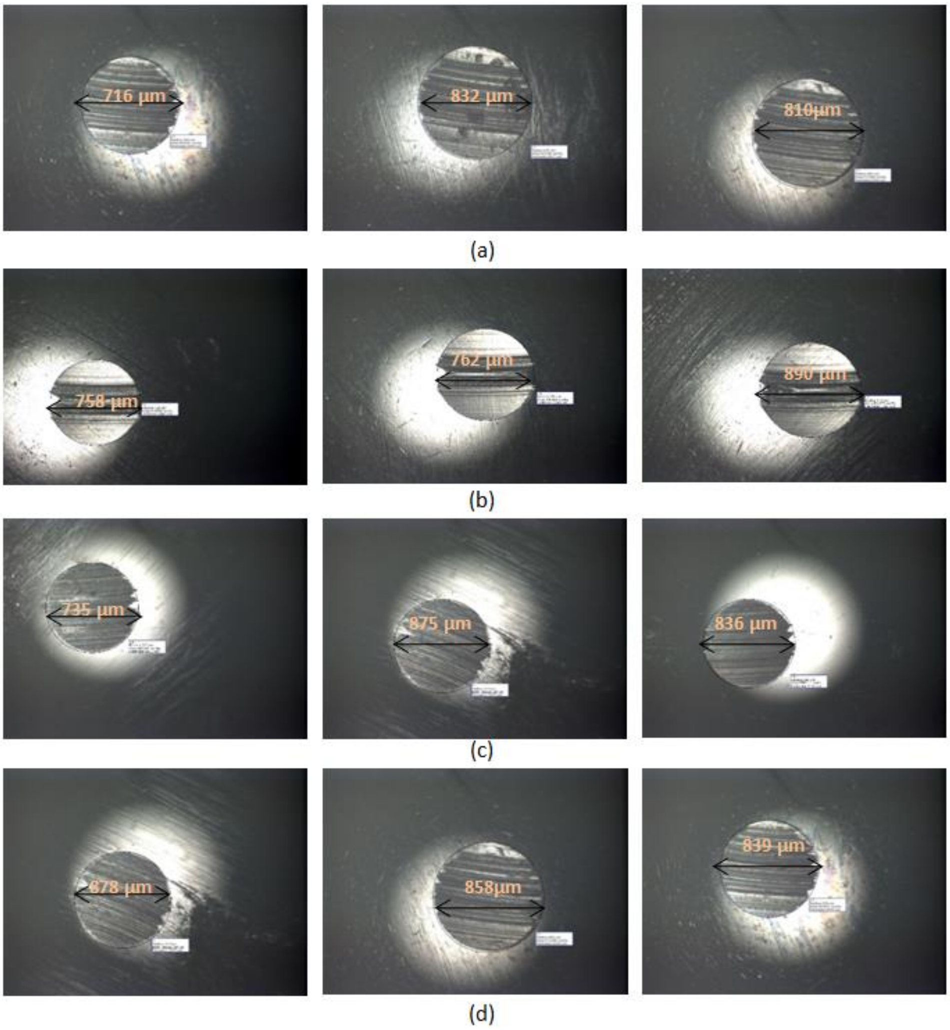

Fig. 3. (a) Wear scars for 3 balls of the Sample 1, (b) Wear scars for 3 balls of the Sample 2, (c) Wear scars for 3 balls of the Sample 3, (d) Wear scars for 3 balls of the Sample 4.

During the interaction between surfaces, a tribofilm could develop due to chemical reactions occurring between the lubricant particles and the surface material [37]. Lower metal-to-metal contact is a result of the tribofilm that has been developed between the surfaces. Consequently, wear is minimized due to the prevention of adhesion. It's widely acknowledged that temperature plays a role in tribofilm formation. From Table 3 it is evident that average wear scar diameter of sample 4 shows maximum value of 858 µm, and lowest is observed for the sample 1 with a value of 786 µm.

Table 3. Wear scar diameter of all the samples.

Wear scar diameter (µm)	Sample 1	Sample 2	Sample 3	Sample 4
Ball 1	716	758	735	878
Ball 2	832	762	875	858
Ball 3	810	890	836	839
Average diameter	786	803	815	858

The observed variation in the average diameter primarily arises from the potential absence of tribofilm formation when testing nanoparticles at ambient temperature. From Table 3 it is also evident that average wear scar diameter of sample 4 shows maximum value of 858 µm, and lowest is observed for the sample 1 with a value of 786 µm. This may be due the percentage variation in TiO_2 from sample 1 to sample 4. The ball-bearing lubricated with all the samplesdisplayed a worn surface that exhibited nearly identical wear patterns with parallel grooves under experimental settings with a 40 kg normal load. While some of the grooves were shallow, others were fairly deep, these variations are shown in Fig. 3.

3.3 Rheological analysis

The rheological behavior of canola oil is affected by the concentration of TiO_2 nanoparticles. The viscosity reduces as the TiO_2 concentration increases. Fig. 4 illustrates the effect of TiO_2 nanoparticles on canola oil shear stress at different shear rates. The graph clearly shows that for all four samples, the shear stress increases considerably as the shear rate increases. The slope between shear stress and shear rate represents the viscosity. However, Sample 3 has the steepest slope when compared to the other samples, indicating that it has higher viscosity [38].

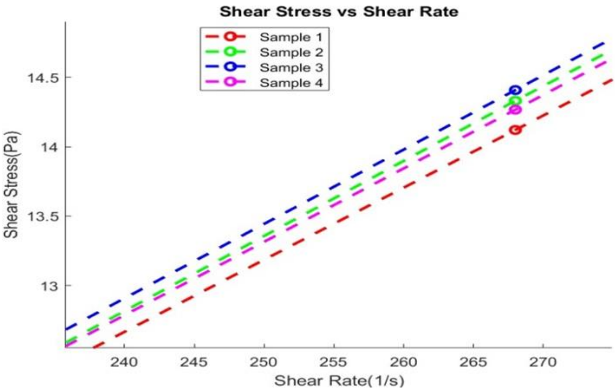

Fig. 4. Rheological comparison between shear stress vs. shear rate.

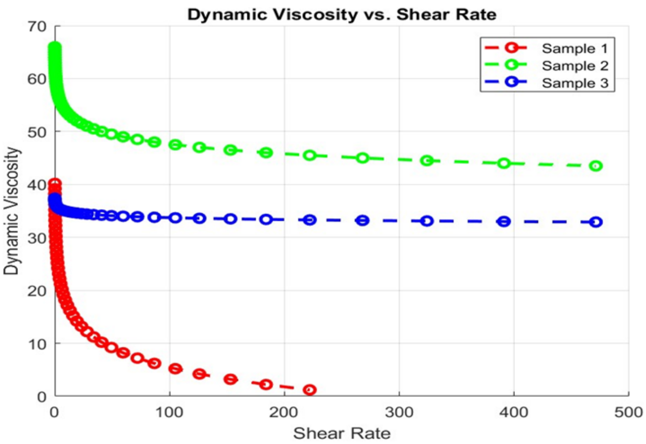

Fig. 5. Impact of viscosity versus shear rate on TiO_2 dispersion in different canola oil weight fractions.

The pseudo-plastic (non-Newtonian) behavior of the developed biolubricant samples is evident from the shear stress vs. shear rate plot. The plot of dynamic viscosity and shear rate is shown in Fig. 5. The graph shows that dynamic viscosity reduces and becomes constant as shear rate increases until it becomes insignificant. The viscosity of sample 1 was modest at first, then dropped dramatically to zero before the shear rate reached 300 s^{-1}. This may be due to the less concentration of TiO_2 nanoparticles. At high shear rates, the drop in viscosity becomes less significant. As a result, the sample has low shear stability since it releases the base oil quickly, shortening the biolubricants lifespan.

3.4 Biodegradability

Oxygen consumption by microorganisms during the breakdown of organic materials in a sample is measured by BOD, or biochemical oxygen demand. Sample 1 yielded a BOD value of 1200 mg/L, which is much higher than the 570 mg/L of the blank (distilled water). The identical sample's COD value is 1920 mg/L, and its BOD/COD ratio is 0.625, which is higher than 0.5. This suggests that the biodegradability of the developed biolubricant sample turns out to be excellent. It is generally considered biodegradable if the BOD/COD ratio exceeds 0.5. Additionally, it has been observed that the BOD/COD ratio is greater than 0.5 for every sample as shown in Table 4.

Table 4. BOD and COD values of all samples.

SAMPLES	BOD mg/L	COD	BOD/COD
1	1200	1920	0.625
2	1180	1954	0.604
3	1140	1966	0.58
4	1105	1980	0.558

An observed decrease in the biodegradability of the lubricants correlates with the increasing concentration of nanoparticles in canola oil.Hence, it is evident that the dispersion of TiO_2 nanoparticles into the vegetable oils does not have any environmental impact.

4. CONCLUSIONS

In conclusion, this study aimed to provide a comprehensive understanding of the tribological, rheological, and biodegradability properties of canola oil incorporating TiO_2 nanoparticles. The following key findings were observed:

- The incorporation of TiO_2 nanoparticles enhances the reactivity of the lubricant, resulting in improved dispersion stability and extended shelf life of the oil samples. This is attributed to the efficient mixing process involving magnetic stirring and ultrasonication.
- The COF results indicate consistent performance across the tested blends. Samples 1 and 2 exhibited lower COF values compared to pure canola oil and other synthetic oils, suggesting their potential for reducing friction.
- Samples with lower concentrations of TiO_2 nanoparticles demonstrated superior quality worn surfaces, indicating their suitability for medium loads and moderate pressure conditions.
- In terms of rheological properties, all samples exhibited superior shear stress and dynamic viscosity performance compared to other synthetic oil grades. Sample 3, containing 1% TiO_2, showed the highest viscosity value among all prepared samples.
- The ratio of BOD to COD exceeded 0.5 for every sample, indicating their potential for effective wastewater treatment and reducing the environmental impact of lubricant disposal.

Overall, the findings suggest that the incorporation of TiO_2 nanoparticles into canola oil has the potential to enhance its tribological performance while maintaining favorable rheological and biodegradability characteristics, thus offering a promising avenue for sustainable lubricant development.

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