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RESEARCH

Tribological Characteristics of Cooking Oil Waste-Based Lubricants with TiO_2 Nano Powder Additives on Aluminum 6061

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A B S T R A C T

Waste cooking oil (WCO) was valorized into an eco-friendly biolubricant and further enhanced with titanium dioxide (TiO_2) nanopowder (0.15 wt%) for aluminum alloy 6061 (AA6061) sliding contacts. WCO was converted to fatty acid methyl ester (FAME) and subsequently transesterified with ethylene glycol to produce the base biolubricant, while TiO_2 was dispersed by stirring–sonication. Physicochemical (density and Fourier transform infrared spectroscopy, FTIR), rheological (viscosity–temperature and Vogel–Tammann–Fulcher, VTF, modeling), thermal (thermogravimetric analysis/differential thermal analysis, TG/DTA), and tribological (pin-on-disc; SKD-11 pin against AA6061 disc) characterizations were conducted. The biolubricant showed near-Newtonian behavior and viscosity decreased with increasing temperature and with TiO_2 addition. TG/DTA showed improved early-stage thermal resistance with TiO_2 (20–400 °C mass loss decreased from 23.98% to 16.73%), while the DTA exotherm shifted from 490.46 °C to 284.59 °C. Tribologically, the average coefficient of friction (n=5) decreased from 0.471 (dry) to 0.231 (biolubricant) and 0.131 (biolubricant+TiO_2). Wear was markedly reduced: compared with the biolubricant without TiO_2, adding TiO_2 decreased disc mass loss from 0.0046 g to 0.0020 g and reduced specific wear rate from 0.000341 to 0.000148 $mm^3/N{\cdot}m$ (≈56.6% reduction). Relative to dry contact, the TiO_2-enhanced biolubricant reduced mass loss and specific wear rate by ≈92.8%. Overall, the TiO_2-enhanced WCO-based biolubricant is promising for aluminum-related metalworking and other Al–steel sliding interfaces under moderate operating conditions.

1. INTRODUCTION

Global petroleum reserves are steadily diminishing, while the world's population continues to grow alongside an ever-increasing number of motor vehicles. Consequently, the demand for petroleum-derived fuels keeps rising. Greater reliance on motorized transport powered by fossil fuels further accelerates the depletion of these finite resources. Given the non-renewable nature of fossil fuels, this trajectory exacerbates the risk of a major energy crisis [1]. Shrinking energy supplies also drive up oil prices, creating significant economic challenges, particularly for developing countries [2].

In this context, introducing renewable and clean energy resources has become one of the foremost global challenges. This imperative is reinforced by heightened public awareness as well as environmental and health authorities' concern over the toxic externalities associated with the combustion of fossil fuels such as coal and petroleum-based diesel [3]. Accordingly, the development and deployment of environmentally benign, biodegradable alternatives to fossil fuels or other nature-based, eco-friendly resources have attracted sustained research attention over the past decade [4].

Vegetable-oil prices have risen significantly in recent years owing to growing use across the food, fuel, and lubricant sectors [5]. Vegetable-oil-based products are considered environmentally friendly alternatives because of their advantageous attributes, including high lubricant polarity, high viscosity index, high flash point, very low volatility, and renewability and, crucially, their biodegradability. It has been estimated that vegetable-oil-based lubricants could replace roughly 90% of lubricants used worldwide [6].

Waste cooking oil (WCO) refers to fats and oils that have been used for cooking or frying in commercial or industrial food processing, including restaurant operations [7]. Because WCO is essentially a waste stream, its price differs from that of virgin vegetable oils. When appropriately modified and formulated with suitable additives, WCO can serve as an excellent substitute for mineral-based lubricants. Numerous chemical pathways employing either conventional reagents or biocatalysts are available to convert WCO into biolubricants [6].

A principal challenge for WCO-derived lubricants is maintaining thermal stability and durability at elevated temperatures. To address this, several studies have explored TiO_2 nanoparticles as additives. The incorporation of TiO_2 has been shown to improve tribological performance, reduce friction and wear, and increase thermal stability over a broad temperature range [8]. Such additives also refine the physical and mechanical properties of the lubricant and can improve surface quality and service life of materials [9]. Tribological investigations further indicate that WCO-based lubricants can lower the coefficient of friction and enhance wear resistance on metallic substrates such as aluminum, which is critical in manufacturing contexts [10]. The addition of TiO_2 can further augment these benefits, reducing friction and extending tool life in high-load and high-temperature applications [11-13]. However, despite these reported advantages, the evidence remains fragmented across different base oils, test configurations, and reporting practices, which makes it difficult to identify clear research gaps and to generalize the role of TiO_2 in WCO-based biolubricants for specific engineering substrates.

Despite the growing literature on vegetable-oil and WCO-derived lubricants with nanoparticle additives, key gaps remain for WCO-based formulations. Many studies treat WCO mainly as a blending component in commercial oils and focus on application-specific contacts (e.g., piston skirt–liner), rather than evaluating formulated WCO biolubricants under widely used sliding configurations such as pin-on-disc [14]. In addition, performance is often reported using four-ball tests or mineral-oil blending approaches, while tribological evidence on AA6061 under pin-on-disc sliding is less consistent [15]. Similarly, TiO_2 nano-biolubricant studies commonly employ non-WCO base oils, leaving limited data for the specific combination of WCO-based biolubricant + TiO_2 under sliding contact [16]. Finally, because WCO undergoes thermo-oxidative and hydrolytic changes during frying that alter composition and generate polar / oxidation products [17], further work is needed to connect feedstock pretreatment / quality control with lubrication mechanisms and tribological outcomes [18].

Accordingly, this study investigates the tribological characteristics of a waste cooking oil (WCO)-based biolubricant with and without a TiO_2 nanopowder additive on AA 6061 using pin-on-disc testing. The work provides quantitative friction and wear evaluation (including repeatability and specific wear rate) supported by worn-surface morphology analysis and is complemented by physicochemical characterization to strengthen the mechanistic interpretation of lubricant performance. Ultimately, the findings are intended to support the development of eco-friendly lubricants for aluminum-related manufacturing applications by reducing friction-induced surface damage.

2. METHODS

2.1 Waste cooking oil preparation

The waste cooking oil was obtained from several local fried chicken vendors. The collected waste oil is purified by mixing it with bleaching earth, using a ratio of 10:1 for waste oil and bleaching earth [19]. Bleaching earth and waste cooking oil are mixed at a temperature of 80°C, then left to stand for 24 hours [20]. The resulting sediment is separated and filtered to remove residues.

2.2 Preparation of CaO catalyst

Calcium oxide (CaO) catalyst derived from scallop shells was prepared through a wet ball-milling route using a planetary ball mill [21], [22]. Scallop shell–derived $CaCO_3$ micropowder was used as the precursor, and deionized water served as the milling medium at a water-to-powder mass ratio of 3:1 [23]. Wet milling was carried out in a closed zirconia milling jar under ambient atmosphere (air). A mixed-ball configuration was used, consisting of four ball sizes with three balls for each size [24], and zirconia balls were used as the milling media. The sample-to-ball mass ratio was fixed at 1:20 based on the total ball mass [24,25]. Milling was performed at 500 rpm for 40 h, with the rotation direction alternated between clockwise and counterclockwise every 15 min [23]. The wet-milling step was intended for particle size reduction and homogenization of the $CaCO_3$ precursor.

After milling, the suspension was filtered using filter paper, and the recovered precipitate was oven-dried at 110 °C [26,27]. The dried material was then ground [26] and subsequently calcined at 900 °C for 2 h to convert the $CaCO_3$ precursor into CaO [28]. After calcination, the CaO catalyst was stored in a vacuum desiccator to minimize moisture uptake and carbonation prior to use.

2.3 FAME production based on waste cooking oil

The FAME used in this work was prepared following a previous study [29]. Waste cooking oil was mixed with the shell-waste-derived CaO catalyst at a loading of 1 wt% relative to the oil. The mixture was stirred continuously at 60 °C and 1400 rpm for 90 min [30]. Upon reaching homogeneity, methanol was introduced at a molar ratio of 8:1 (methanol:oil) [30], and the transesterification reaction was allowed to proceed. At the end of the reaction, the catalyst was separated by centrifugation at 3000 rpm for 5 min [31].

2.4 Production of biolubricant based on waste cooking oil

The biolubricant based on waste cooking oil was prepared via transesterification. An illustrative schematic diagram of the biolubricant manufacturing process up to the addition of TiO_2 additive is presented in Figure 1.

The biolubricant based on waste cooking oil was prepared via transesterification of the previously produced FAME with ethylene glycol at a molar ratio of 6:1, using CaO catalyst at 1.2 wt% relative to the FAME mass. The transesterification was carried out at 130 °C for 120 min with an agitation speed of 1000 rpm. Upon completion, the catalyst was separated by allowing the reaction mixture to settle for 24 h, followed by a two-stage filtration using filter paper.

The resulting biolubricant was then doped with TiO_2 nanoparticles at 0.15 wt%. The TiO_2 concentration of 0.15 wt% used in this study was selected as a guided reference concentration based on previous literature and the authors' prior experimental work [32-34]. The TiO_2 nanopowder used in this study was a commercial product (Sigma-Aldrich). The dispersion was mixed on a magnetic stirrer for 20 min at 1250 rpm, and subsequently sonicated using an ultrasonic homogenizer for 30 min.

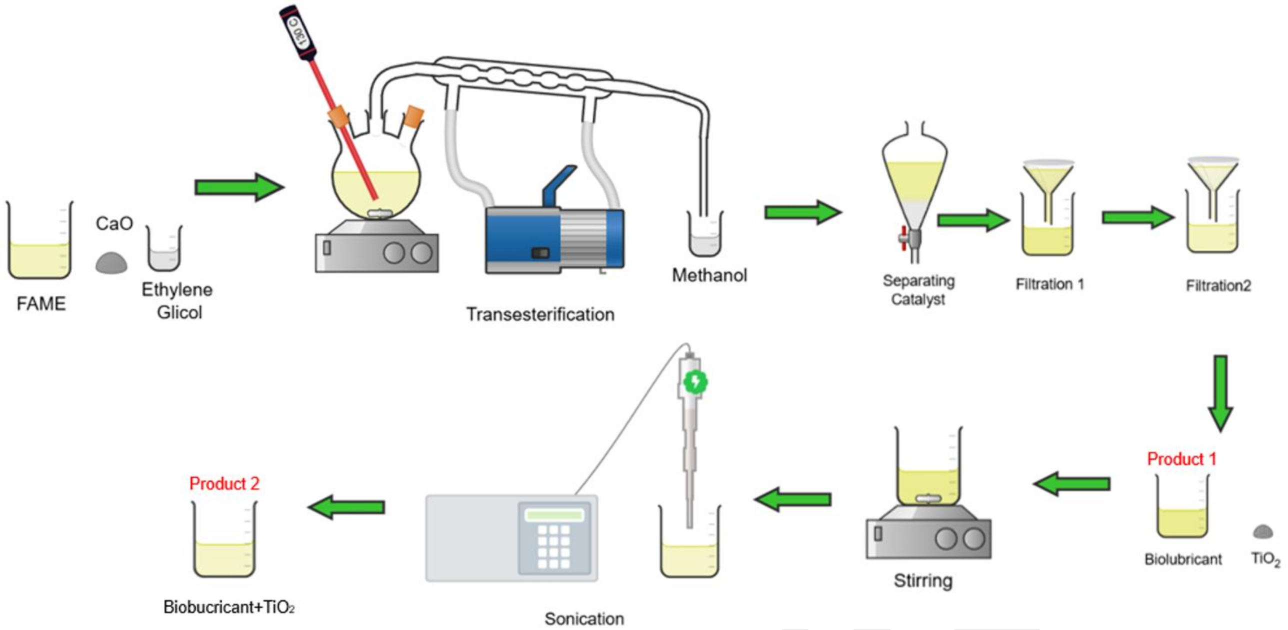


Fig. 1. Production of biolubricant scheme.

2.5 Additive nanoparticle characterization

The morphology of the nanoparticles was analyzed using SEM (FEI Inspect-S50). FTIR (Shimadzu IR Prestige 21) was used to identify functional groups, and XRD (PANalytical X'Pert PRO) was used to determine the crystal structure.

2.6 Physicochemical testing of waste cooking oil-based biolubricants

The physicochemical characterization comprised FTIR, density, viscosity, and TG/DTA measurements. FTIR analysis was performed to identify the functional groups of the waste-cooking-oil-based biolubricant using a Shimadzu IRPrestige-21 spectrometer. Viscosity was measured to determine the rheological behavior of the biolubricant with a NDJ-8S viscometer [35]. Density was determined to quantify the specific mass of the biolubricant and was measured at the standard test temperature of 40 °C [36,37]. Thermogravimetric/differential thermal analysis (TG/DTA) was conducted to evaluate the thermal decomposition behavior of the waste-cooking-oil-based lubricant using a Linseis STA PT 1600 instrument.

2.7 Rheology test of waste cooking oil-based biolubricants

The shear stress was obtained by multiplying the shear rate value by the dynamic viscosity, where the equation of the shear rate is shown in Equation 1 [22,38]:

$$\gamma = \frac{2\,\omega\, R_c 2\, R_b 2}{x^2\,(R_c 2\, R_b 2)} \quad (1)$$

where γ is shear rate (/s), ω is angular speed of the shaft (rad/sec), R_c is vessel radius (cm), R_b is spindle shaft radius (cm), x is shear rate radius (cm).

2.8 Tribology test of waste cooking oil-based biolubricants

Performance testing was carried out using a pin-on-disc tribometer to evaluate the ability of the biolubricant to reduce wear on both the pin and the disc [12,39]. The tests employed an SKD-11 steel pin against an AA 6061 aluminum disc. The wear test followed ASTM G99, with the operating parameters summarized in Table 1. An illustrative schematic of the pin-on-disc setup is provided in Figure 2.

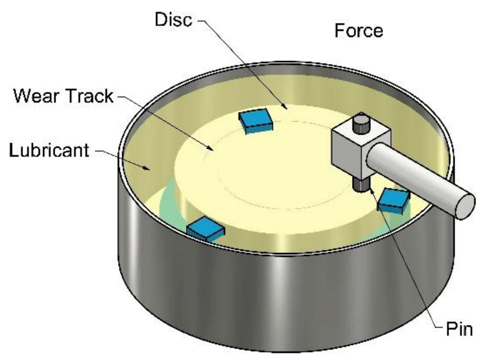


Fig. 2. Pin on disc test scheme.

Table 1. Parameters of pin on disc testing.

Parameters	Value
Force (N)	10
Sliding Speed (m/s)	0.1
Sliding Distance (m)	5000
Track Diameter (mm)	35
Temperature (°C)	23
Pin Diameter (mm)	6
Disc Diameter (mm)	60

Disc wear was quantified using a gravimetric approach by measuring the specimen mass before and after testing (mass loss). The wear volume was obtained by converting mass loss to volume using the material density (equation 2), and the specific wear was calculated using equation 3, which is defined by the volume difference of the material after wear (V), applied normal load (F), and the sliding distance (l) [40], [41]. In addition, the wear morphology of the worn disc surface was examined using scanning electron microscopy (SEM) to support the interpretation of the dominant wear mechanisms.

$$V = \frac{m}{\rho} \quad (2)$$

$$W_s = \frac{\Delta V}{F \times l} \quad (3)$$

3. RESULTS AND DISCUSSION

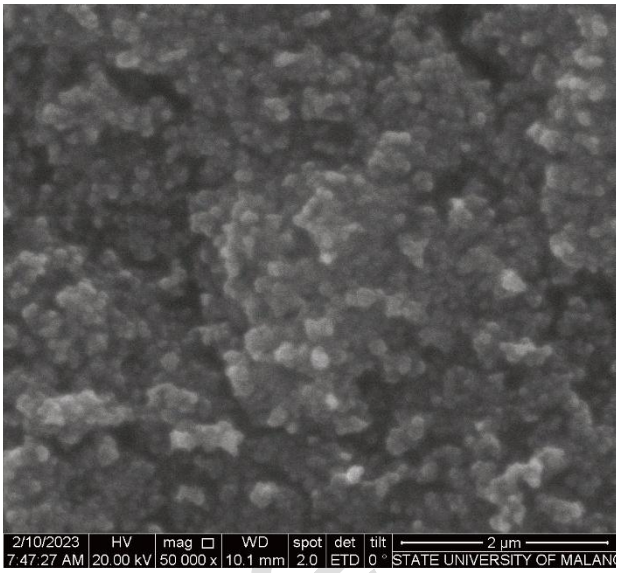
3.1 Characterization of TiO_2 additive nanoparticles

Based on the SEM results shown in Figures 3a and 3b, the TiO_2 nanoparticles exhibit a roughly spherical morphology and a tendency to agglomerate [42]. The spherical shape can promote a rolling effect in the nano-cutting fluid [43].

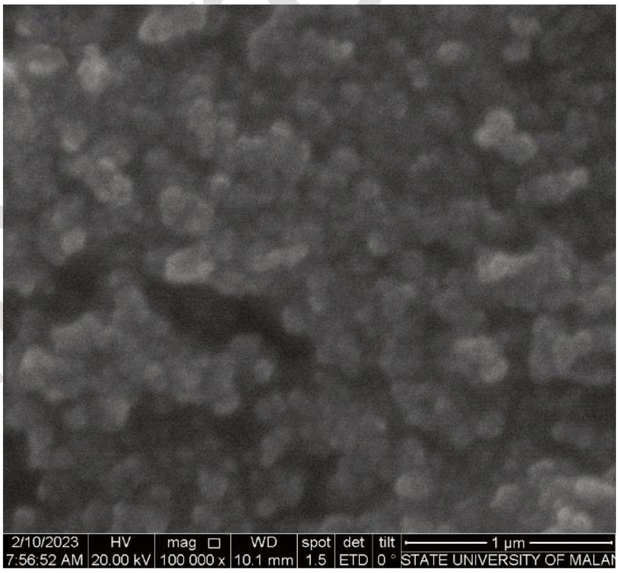
XRD analysis of the TiO_2 additive nanoparticles was conducted to identify the crystallographic phase(s) and estimate the crystallite size. The crystallite size was calculated using the Scherrer equation, as given in Equation 4 [44].

$$d = \frac{K\,\lambda}{\beta \cos\theta} \quad (4)$$

where d is the crystallite diameter, $K = 0.9$, $\lambda = 1.5406$ Å, and β is the full width at half maximum (FWHM).



(a)



(b)

Fig. 3. Morphology of TiO_2 additive nanoparticles at magnifications of (a) 50kx and (b) 100k.

Based on Figure 4, the XRD pattern indicates that the employed TiO_2 nanoparticles exhibit their most intense diffraction peaks at 25.352° and 48.0901°. According to Ali et al. [43], the absence of a diffraction peak near 27.4° characteristic of the rutile (110) reflection supports assignment of the sample to the anatase phase. The crystallite size, calculated using the Scherrer equation, is 20.2806 nm.

FTIR analysis was conducted to identify the functional groups of the TiO_2 nanoparticle additive. The FTIR spectra obtained in this study are presented in Figure 5. From the principal absorption features observed within 3390.86–

702.09 cm^{-1}, the functional-group assignments for the TiO_2 nanoparticles can be determined as follows: the band at 3390.86 cm^{-1} is attributed to O–H stretching; the band at 1614.42 cm^{-1} corresponds to Ti–OH bending; the feature at 2308.29 cm^{-1} is associated with C=O; the band at 1122.57 cm^{-1} is assigned to Ti–O–Ti stretching; and the band at 702.09 cm^{-1} is assigned to O–Ti–O vibrations [45].

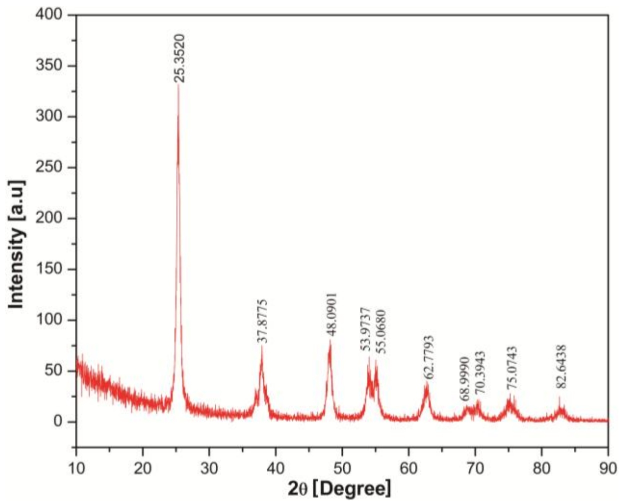


Fig. 4. XRD Results of Nanoparticle TiO_2.

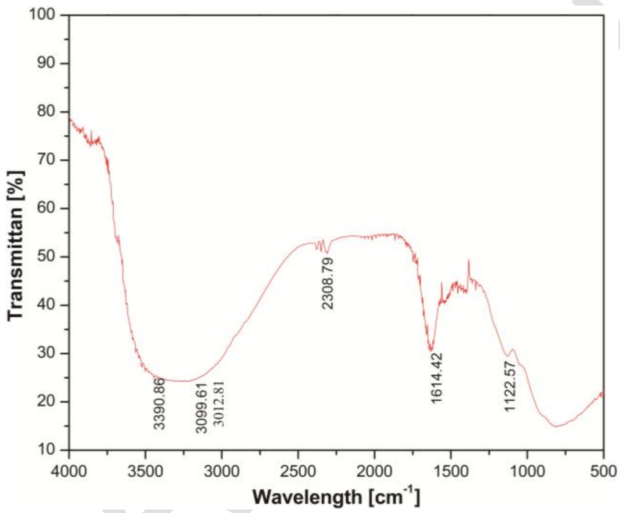


Fig. 5. FTIR Results of Nanoparticle TiO_2.

3.2 Physicochemical properties of waste cooking oil-based biolubricant samples

Based on Figure 6, for the neat biolubricant a slight red-shift of the C=O band to lower wavenumbers and/or peak broadening can be interpreted as evidence of weak interactions (coordination or hydrogen bonding) between the ester carbonyl and Lewis/Brønsted sites on the TiO_2 surface. Recent FTIR studies on nanoparticles commonly regard such subtle spectral shifts as indicators of interfacial interactions rather than the formation of new functional groups [46]. Accordingly, the spectrum suggests that TiO_2 is successfully dispersed without altering the primary ester framework, while introducing inorganic active sites that may modulate interfacial behavior in subsequent tribological applications [47].

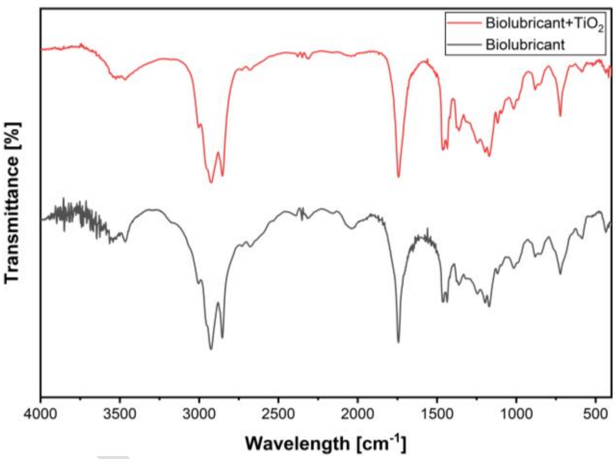


Fig. 6. FTIR Result of Waste Cooking Oil-Based Biolubricant Samples.

For the biolubricant + TiO_2 spectrum, the principal bands of the biolubricant remain observable, indicating that the triglyceride backbone is not degraded by nanoparticle addition. Nevertheless, small shifts and changes in intensity are apparent for several polar bands particularly around ν(C=O) ≈ 1740–1725 cm^{-1} and ν(O–H) ≈ 3400 cm^{-1} signaling interactions between the lubricant's polar groups (carbonyl/hydroxyl) and the TiO_2 surface (e.g., via hydrogen bonding or weak coordination). Such shifting patterns are widely reported in metal-oxide nano-additive systems for lubricants/biolubricants, wherein the presence of nanoparticles modifies the local environment of functional groups without fundamentally altering the ester framework [46,48].

Based on Table 2, the density increases after the addition of TiO_2 nanoparticles. This trend is consistent with the principle that a high–mass-density solid additive can raise the bulk density of a composite liquid when it is well dispersed. Recent studies also report that density changes in nanolubricants (lubricants containing nanoparticles) are generally minor; however, small increases may occur depending on nanoparticle concentration and dispersion quality [49].

Table 2. Density of waste cooking oil-based biolubricant samples.

Samples	Density [kg/L]
Biolubricant	908.535 ± 0.362
Biolubricant + TiO_2	912.404 ± 0.06

The measured density of the biolubricant (≈ 0.908 g mL or 908 kg m^{-3}) is relatively higher than that of conventional mineral or synthetic lubricants, which typically lie in the range of 0.85–0.90 g mL^{-1} (850–900 kg m^{-3}) at room temperature [48]. This discrepancy can be attributed to the molecular characteristics of ester products formed via transesterification, which tend to have more dense structures [50]. As also noted in the study by Trzepieciński [51], Bio-based lubricants often exhibit higher densities; nevertheless, this does not diminish their ability to form lubricating films, particularly because the polar ester functionalities facilitate adsorption onto metal surfaces.

Based on Figure 7, the addition of TiO_2 to the biolubricant generally lowers the viscosity, indicating enhanced fluid mobility and reduced internal friction. This reduction can be attributed to the disruption of cohesive van der Waals interactions among long-chain hydrocarbon/ester molecules, allowing TiO_2 nanoparticles to interact with the oil microstructure and weaken intermolecular friction more effectively [52,53]. On the other hand, viscosity also decreases as temperature increases. This trend is typical of lubricating fluids, wherein elevated temperature weakens intermolecular interactions, rendering the fluid less viscous [54]. At intermediate temperatures (70–90 °C), the curves show small fluctuations, with viscosity tending to stabilize or exhibit a slight increase before declining again at 100 °C. This phenomenon may be associated with unsaturated ester groups in the biolubricant's molecular structure, which can lead to non-monotonic rheological responses under specific conditions [55]. To strengthen the analysis quantitatively, the viscosity–temperature relationship was fitted using the Vogel–Tammann–Fulcher (VTF) model. The VTF fitting parameters and goodness-of-fit are summarized in Table 3. Based on VTF fitting the fitted curves confirm the dominant viscosity-decreasing trend with temperature and provide a quantitative basis to compare formulations. Although some VTF parameters exhibit relatively large standard errors, the model is used here primarily for comparative description of temperature sensitivity rather than strict mechanistic inference. Nevertheless, the overarching trend indicates that the biolubricant maintains workable viscosity across the investigated range, supporting its applicability under realistic operating conditions.

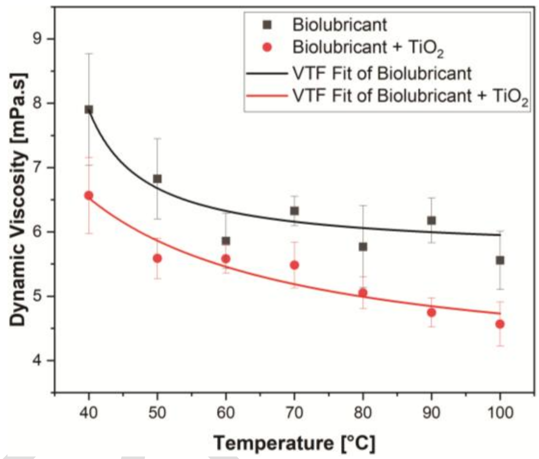


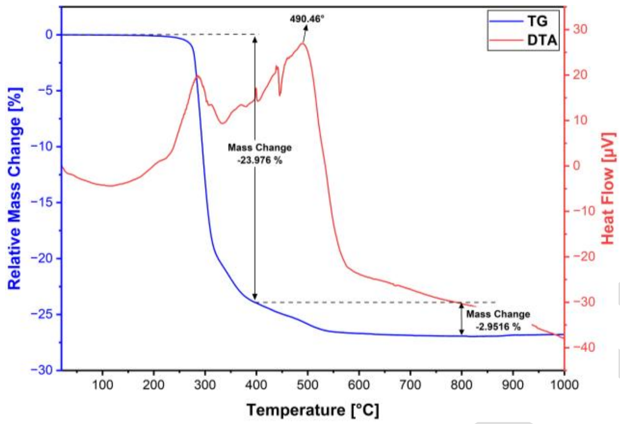
Fig. 7. Viscosity of Waste Cooking Oil-based Biolubricant Samples.

Table 3. Vogel–Tammann–Fulcher (VTF) fitting parameters for viscosity–temperature data.

	Samples	
	Biolubricant	**Biolubricant + TiO_2**
A (±SE)	0.7549 ± 0.0399	0.5817 ± 0.0983
B (±SE)	1.3701 ± 2.0243	9.3264 ± 12.4186
X_0 (±SE)	30.3940 ± 12.5821	0.0000 ± 39.9985
R^2	0.6352	0.8762
Adj. R^2	0.4527	0.8143

Based on Figure 8, the TG/DTA analysis was conducted in air atmosphere at a heating rate of 10 °C/min from room temperature to 1000 °C, representing an accelerated oxidative ramp. The addition of TiO_2 to the biolubricant reduces the early-stage mass loss (20–400 °C) from 23.98% to 16.73%, indicating that the volatile fraction and the initial triglyceride/ester degradation are more effectively suppressed. This behavior suggests improved early thermal stability for vegetable-oil-based lubricants and is consistent with the use of TGA to map lipid conversion or oxidation stages, as well as reports that formulation modifications or additives can mitigate volatilization near the oxidative onset in air [56,57].

In contrast, within 400–800 °C the biolubricant+TiO_2 sample exhibits a slightly higher mass loss (2.96% → 3.25%), accompanied by a pronounced shift of the exothermic DTA peak from 490.46 °C (without additive) to 284.59 °C (with TiO_2). Under an oxidative ramp in air, this exotherm shift is interpreted qualitatively as an earlier onset of oxidation or combustion of organic residues, which can be facilitated by the thermocatalytic activity of TiO_2 surface sites or defects (e.g., activated surface oxygen and vacancy-related reactive species), rather than implying a quantified reduction in activation energy [58-60]. This behavior should not be attributed to TiO_2 photocatalysis because photocatalytic activity requires light irradiation to generate electron–hole pairs, whereas the TG/DTA experiment does not involve UV or visible illumination [61].



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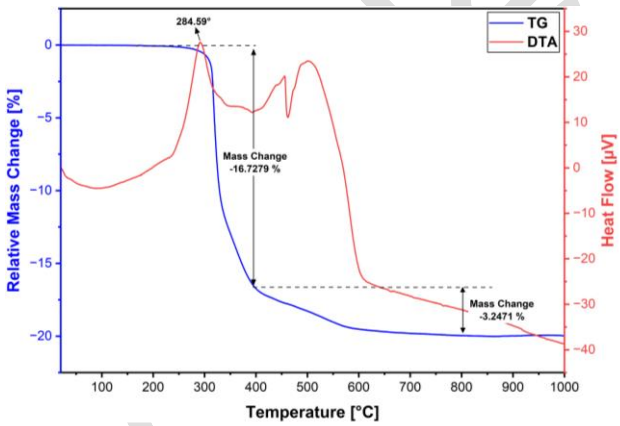


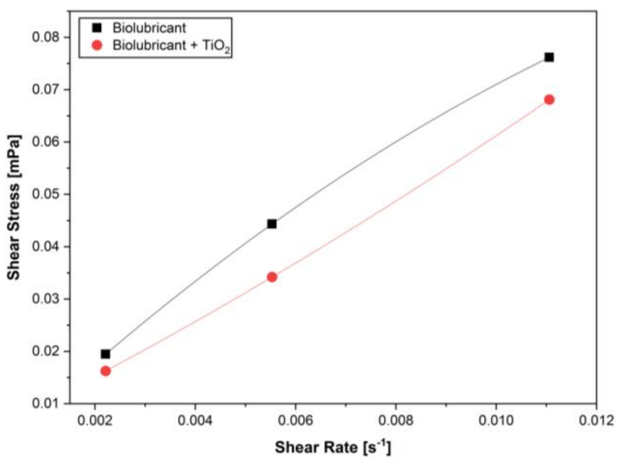
(b)

Fig. 8. TG and DTA curves for (a) Biolubricant and (b) Biolubricant+TiO_2.

From a practical lubrication standpoint, the early-stage stabilization is generally the most relevant benefit because bulk lubricant operating temperatures in many conventional systems are typically far below extreme high-temperature oxidation regions (often limited to around ~200 °C for hydrocarbon-based oils), whereas the later-stage TiO_2-assisted oxidation tendency should be regarded as a caution/limitation under severe overheating and oxygen-rich exposure rather than a universal benefit [62]. At the same time, tribological contacts may experience transient localized "flash" temperatures substantially higher than the bulk temperature at asperity junctions, which can influence surface chemistry; thus the high-temperature oxidative tendency is discussed as a potential risk under extreme conditions rather than typical service behavior [63]. Importantly, the practical relevance of using TiO_2 as a lubricant additive remains supported by tribological studies that report reduced friction and wear when TiO_2 nanoparticles are incorporated into lubricating oils and bio-based oils [8,16].

3.3 Rheology properties of waste cooking oil-based biolubricant samples

Based on Figure 9, the difference in shear-stress values between the neat biolubricant and the TiO_2-doped biolubricant indicates a modification of rheological properties due to nanoparticle addition. In general, vegetable-oil-based lubricants exhibit a pronounced decrease in viscosity with increasing temperature [64]. With TiO_2 present, however, this decline is somewhat more controlled, suggesting that the nanoparticles help retard thermally induced viscosity degradation. As reported in the study by Uniyal et. al. [65], metal-oxide nanoparticles such as TiO_2 can interact with polar groups in the oil matrix to form an interfacial layer that reinforces the fluid structure, thereby stabilizing the fluid's temperature response. This effect also contributes to the stability of the lubricating film under high-temperature operating conditions.



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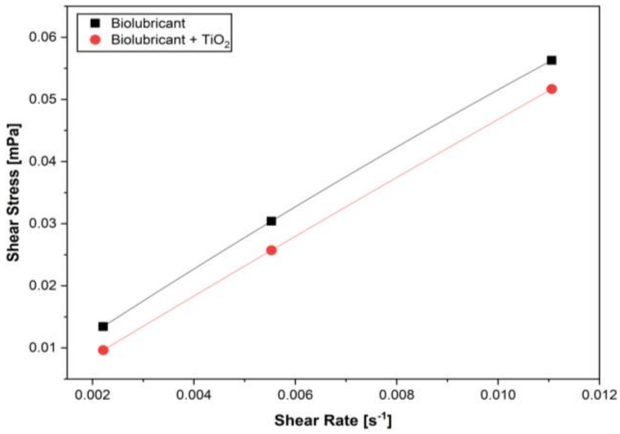


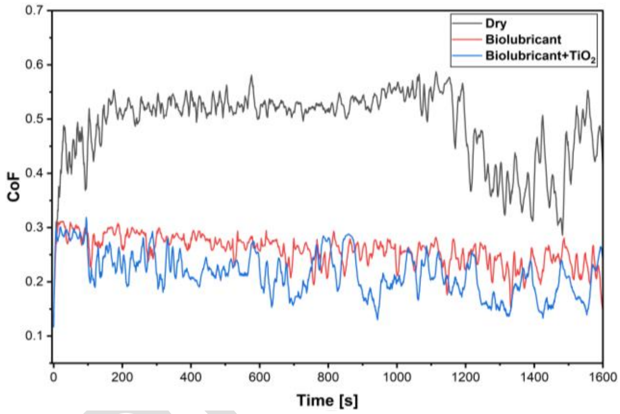
(b)

Fig. 9. Rheological properties of waste cooking oil-based biolubricant samples at (a) 40°C and (b) 100°C.

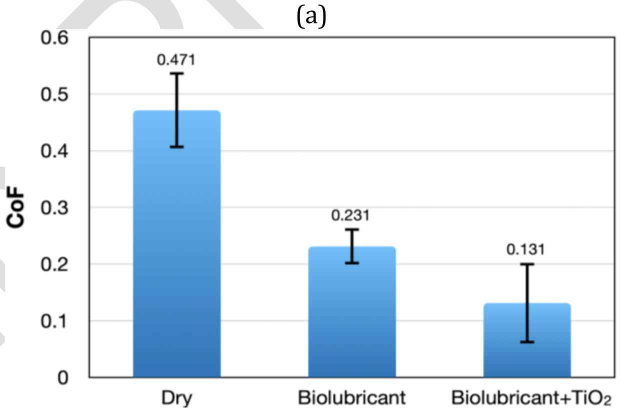
In addition, a mild shear-thinning phenomenon can be observed particularly at high shear rates and low temperatures where the shear stress of the TiO_2-containing biolubricant is slightly lower than that of the neat fluid. This observation aligns with previous study by Mousavi and Heris [66], which notes that introducing nanoparticles into lubricant systems can reduce internal shear resistance and facilitate flow without substantially altering the fluid's near-Newtonian behavior. Furthermore, the review in Duan et. al. [67] emphasizes the dual role of nanomaterials: enhancing thermal stability while improving tribological performance (e.g., lowering friction coefficient and wear), which is consistent with the tendency toward reduced shear stress in the TiO_2-modified system.

3.4 Tribology properties of waste cooking oil-based biolubricant samples

The coefficient of friction during the transition period and the average CoF results from the pin-on-disc tests are presented in Figure 10. Tests were conducted on an AA 6061 aluminum disc using a pin-on-disc tribometer under three lubrication conditions: dry, biolubricant, and biolubricant + TiO_2. The average CoF values show a clear trend: the dry condition exhibits an average CoF of 0.471; applying the biolubricant reduces the CoF to 0.231; and with biolubricant + TiO_2, the average CoF further decreases to 0.131. The use of lubricant both without and with TiO_2 markedly lowers friction due to the formation of a fluid film/tribofilm that separates asperities and enhances heat dissipation; this observation aligns with film-forming and heat-transfer mechanisms reported for nano-enhanced biolubricants [68]. In general, metal-oxide nanoparticles (including TiO_2) can reduce friction via tribofilm formation, rolling/bearing effects, and a filler effect that smooths micro-scratches; the effectiveness of these mechanisms strongly depends on nanoparticle concentration and dispersion.



(a)



(b)

Fig. 10. (a) Coefficient of friction during the transitional period of the test and (b) Avarage of CoF (n=5).

Figure 11 shows that the tribological mass-loss and specific wear rate data reveal pronounced differences in wear among dry contact, biolubricant, and biolubricant with TiO_2 nanoparticle additive. Under dry contact, severe material removal occurs, with a wear mass of 0.028 g. This high wear rate arises from direct asperity–asperity interactions that simultaneously trigger adhesive and abrasive wear mechanisms. The resulting high friction, excessive heat generation, and plastic deformation at the contact interface are hallmark features of unlubricated tribosystems [69]. The use of a biolubricant fundamentally alters interfacial interactions and drastically suppresses the wear rate. This improvement is attributed to the ability of biolubricant molecules to form a thin yet robust lubricating film between the mating surfaces. The film acts as a physical separator, preventing direct metal-to-metal contact and shifting the lubrication regime from solid contact to boundary or mixed lubrication, thereby effectively protecting the surfaces from severe damage [70].

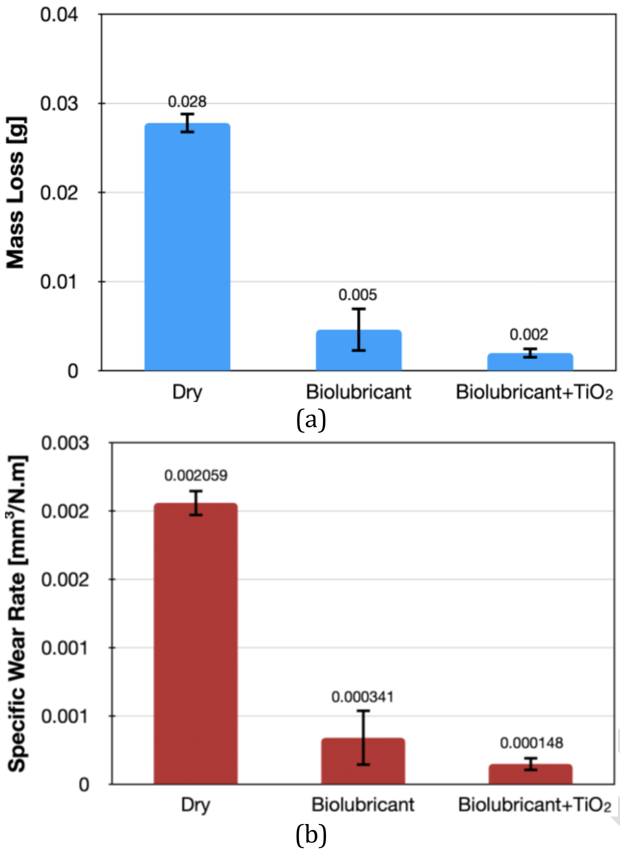


Fig. 11. (a) Mass loss and (b) Specific wear rate of aluminum 6061 disc after pin-on-disc test (n=5).

Incorporating TiO_2 nanoparticles into the biolubricant further enhances anti-wear performance, indicating that the nanoparticles actively reinforce the lubricating film. The improvement can be explained by several mechanisms, including the nano-bearing effect where particles roll between the surfaces and the in situ formation of a protective tribofilm on the worn tracks. This tribofilm refines surface topography and supports contact loads, thereby providing superior protection against wear [43].

Figure 12a presents the disc morphology after the pin-on-disc test under dry conditions. The image reveals damage in the form of tearing/material fragmentation and cavitation-like pits (circled). This pattern is consistent with an adhesive-wear mechanism accompanied by plastic deformation at asperities, ultimately leading to delamination under repeated cyclic loading. The presence of trapped tribo-debris within surface crevices can further accelerate acute abrasion in adjacent regions. These observations accord with general accounts of wear mechanisms in metal–metal contacts operating in boundary/semi-boundary regimes, where the absence of a protective film promotes direct metal-to-metal contact and the formation of pits due to adhesive wear and fatigue [71].

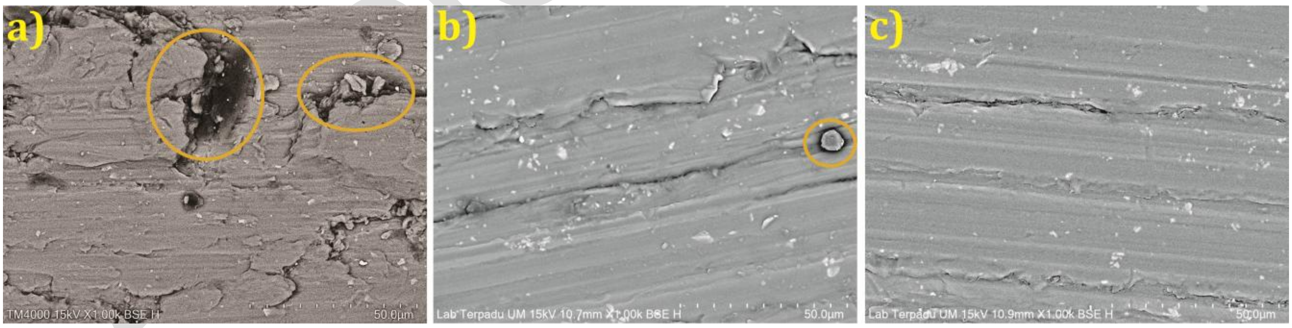


Fig. 12. Surface Morphology of Aluminum 6061 Disc after Pin-on-Disc Test with sample media (a) dry, (b) biolubricant, and (c) biolubricant+TiO_2.

Figure 12b shows the disc morphology lubricated with WCO, exhibiting more regular wear tracks and relatively smaller pits compared with the dry condition. This indicates that WCO forms a lubricating film (hydrodynamic/semi-boundary) that mitigates the severity of metal–metal contact, thereby lowering the coefficient of friction and shifting the dominant mechanism from severe adhesive wear to predominantly mild abrasive/adhesive wear. The literature reports that vegetable oils and WCO contain polar groups (e.g., ester, acid) capable of adsorbing onto metal surfaces to form a molecular film that reduces direct asperity contact and wear; however, the oxidative stability of WCO and the need for modification/antioxidants are often cited as performance limitations relative to synthetic base oils [15].

Figure 12c presents the disc morphology after testing with the biolubricant + TiO_2, which yields the smoothest surface among the three conditions, indicating a marked improvement in tribological performance when TiO_2 nanoparticles are added to the bio-base oil. The relevant mechanisms, supported by contemporary studies, include: (1) tribofilm formation, wherein particles react/interact at the contact to produce a protective layer; (2) a mending effect, whereby particles adhere to/fill asperities, effectively "patching" worn regions; (3) a rolling/ball-bearing effect, in which (quasi-)spherical nanoscale particles act as micro-bearings that partially convert sliding to rolling and reduce friction; and (4) enhanced load-carrying capacity of the nano-suspension, which decreases inter-asperity stresses. All of these mechanisms have been observed in studies of TiO_2 and other metal-oxide nanoparticles as lubricant additives [8,72,73].

4. CONCLUSION

This study demonstrated that waste cooking oil (WCO) can be valorized into a biolubricant and further improved by a low TiO_2 nanopowder addition (0.15 wt%) for AA6061 sliding contacts. The TiO_2 additive was identified as predominantly anatase with nanoscale crystallite size, and physicochemical results indicate that the base ester structure of the biolubricant is retained after TiO_2 incorporation while showing interfacial interactions consistent with a nano-additive system. Thermally, TG/DTA in air confirmed a reduced early-stage mass loss with TiO_2 (20–400 °C: 23.98% → 16.73%), indicating improved resistance to early volatilization/initial degradation; however, the shifted DTA exotherm to a lower temperature suggests that TiO_2 may promote oxidation pathways under severe oxidative/high-temperature exposure, thus this benefit is most relevant for moderate operating regimes.

Tribologically, pin-on-disc tests (SKD-11 vs AA6061) showed that lubrication markedly reduced friction compared to dry contact, and TiO_2 addition provided the lowest friction, with the average CoF decreasing from 0.471 (dry) to 0.231 (biolubricant) and 0.131 (biolubricant+TiO_2). Wear evaluation using mass-loss-derived wear volume and specific wear rate, supported by SEM observations, confirmed that the biolubricant suppresses severe adhesive/abrasive damage, while TiO_2 further improves surface protection, consistent with fluid-film/tribofilm formation and nano-bearing/mending effects. Overall, the TiO_2-enhanced WCO biolubricant is a promising sustainable lubricant for aluminum-related manufacturing tribosystems (e.g., Al–steel sliding interfaces) where friction and wear reduction are needed under moderate service conditions, while extended oxidation/stability verification under service-like aging remains recommended for future work.

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