

Thermo-Oxidative Stabilities of Biolubricant Blends Derived from Butter, Palm Oil, Shea Butter, and Tallow as Potential Alternatives to Fossil Oils

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Abstract

The growing interest in biobased lubricants presents a sustainable alternative to petroleum-derived base oils, with significant potential to reduce environmental pollution. Vegetable oils, known for their rapid biodegradability, offer promising prospects as base fluids in the formulation of environmentally friendly lubricants. However, their practical application is often hindered by inherent limitations such as poor oxidative and low-temperature stability. This study evaluates the thermo-oxidative performance of six biolubricant blends derived from Shea butter, palm oil, butter, and tallow oils. Using thermogravimetric analysis (TGA), the thermal and oxidative stability of each blend was assessed. Among the tested samples, the Shea butter–butter oil blend (SBBOL) exhibited the highest thermal and oxidative stability, with decomposition temperatures of approximately 560 °C and 450 °C at 90% weight loss, respectively. The tallow–Shea butter blend (TSBOL) demonstrated a thermal stability of 520 °C, while the butter–tallow blend showed enhanced

oxidative resistance at 410 °C. Other blends, including palm oil–tallow (POTOL) and butter–tallow (BTOL), exhibited thermal stability values of 510 °C and 410 °C, respectively, with corresponding oxidative stabilities of 367 °C and 410 °C. The findings confirm that physical blending of biolubricants enhances their thermo-oxidative properties. The role of oxygen in accelerating degradation at elevated temperatures further underscores the need for optimized formulations to extend lubricant life and performance. This study contributes to the advancement of eco-friendly lubrication technologies through improved biolubricant formulation strategies.

Keywords: Biolubricants; Thermo-Oxidative Stability; Vegetable Oil Blends; *Thermogravimetric Analysis*; Sustainable Lubrication

INTRODUCTION

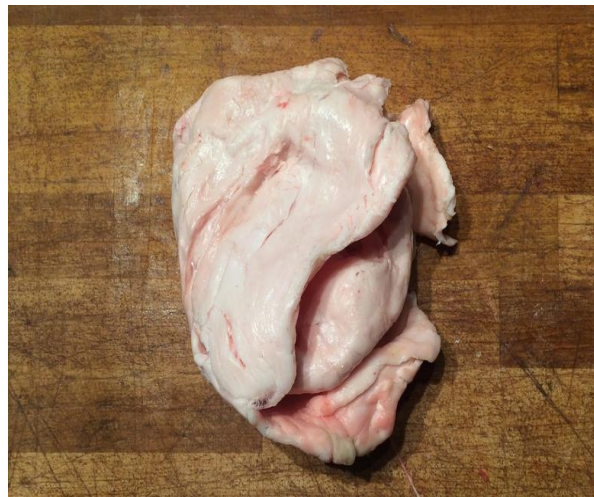
The global demand for lubricants keeps increasing alongside the concern about the environmental hazards associated with non-sustainable petrochemicals. This has called for the need for biolubricants with less hazards to the ecosystem and biosphere. Lubricants are used primarily to reduce friction between surfaces and increase the lifespan of machinery (Karmakar et al., 2017). Typical lubricants consist of mineral or synthetic oils that contain mainly paraffinic, naphthenic and aromatics (to a lesser extent) hydrocarbons (Zzeyani et al., 2017). As occurs during the extraction and refining of petroleum-derived products, spilling during transportation and improper disposal of lubricants of mineral origins may lead to soil contamination and groundwater pollution (Suganthi et al., 2018; Sabarinth et al., 2019). Therefore, economic and ecological factors require renewable lubricant base oils with higher biodegradability and lower toxicity to satisfy growing performance standards and increasingly stringent environmental regulations.

Every year, about 5– 10 million tons of petroleum products enter into the environment from spills, industrial and municipal waste, urban runoff, refinery processes, and condensation from marine engine exhaust (Garwilo, 2005 and Horner, 2004). In certain applications, strict specifications on various environmental matters, such as biodegradability, toxicity, occupational health and safety, and emissions, have become mandatory. At present over 125 million metric tons of vegetable oils are produced worldwide. These vegetable oils offer excellent lubricity, biodegradability, favorable viscosity temperature characteristics, high flash points, and compatibility with mineral oil

and additive molecules. The restriction in using vegetable oils for formulating lubricants are their insufficient thermal and oxidative stability (Becker and Knoor, 1996), low temperature fluidity, and hydrolytic instability [6]. Some of these restrictions can be overcome by using high oleic varieties of vegetable oils in combination with available additives (antioxidants, pour point depressants) and diluents or functional fluids (Assadukas et al., 1996)



Shea butter (Source:Wikipedia, 2024)





Animal fat or tallow (source: google.com, 2024)

MATERIALS AND METHODS

Sample Collection and Preparation

Collection and preparation of oils from source materials

Palm kernels was collected from the Head of Department, Chemistry, Modibbo Adama University, Yola, Adamawa State; Shea butter nut were obtained from Lapai Local Government Area of Niger state; Milk for Butter was locally sourced from Damare Village, Girei Government Area of Adamawa State and Animal fat was purchased from the Abattoir, Yola, Adamawa State, Nigeria.

Extraction of Bio-lubricants

Shea Butter Biolubricant

In the experiment, 30 g of 0.45 mm particle size of the prepared shea nut particles was placed in a wrapped cloth in place of the thimble and placed into the received portion of the Soxhlet extractor. 150 cm³ of methanol was transferred into the flask and placed on an electro thermal heating mantle. The Soxhlet extractor was connected with a condenser having rubber tubing for inlet and outlet water flow for cooling. The miscella, a mixture of oil and solvent was subjected to evaporation to recover the solvent and oil (Prasanna and Rahul, 2018).

Palm kernel oil extraction.

The freshly collected seeds were washed and dried at room temperature in order to remove dirt and other contaminants. The dried kernels were shredded to smaller sizes and

heated to 80 °C so as to elevate the moisture content and also to allow for penetration of solvents. The 100 g of the crushed sample was placed in a Soxhlet with diethyl ether as (about 300 cm³) as solvent (Bong, *et al.*, 2018).

Synthesis of Biolubricant from Animal fat (Tallow)

The beef tallow collected from a local butcher was subject to several rendering processes to separate fat from protein-rich tissues. For boiling water (BW) fat extraction, the fat tissues were boiled with water (enough to cover the fat material) in a pressure cooker for 40 min (1.5 atm; 112 °C). After the residues were removed, the liquid is cooled at room temperature and the fat layer will be removed with a drainer. The cooking time was optimized to extract the maximum fat. Acetone fat extraction, at reflux temperature, was accomplished in a glass 500 cm³ capacity round flask equipped with a reflux column heated by a nest-shaped heating mantle. The fat tissues were then boiled with acetone (1.28 w_{fat}/w_{acetone} ratio) for about 4 hours. After removing the solid material, the liquid was now cooled at room temperature and the fat layer removed with a drainer. All the extraction methods were replicated 3 times to evaluate their reproducibility (Soares Dias, *et al.*, 2013).

Synthesis of Biolubricant from Buttermilk

About 4 litres of raw cream (40 % fat) was batch pasteurized at 68.3 °C for 30 minutes each and was then cooled, stored in separate containers and aged overnight at 7 °C. A part of the pasteurized cream was manually churned on a butter churn machine without addition of color. Break point will be reached about 55 to 60 minutes and the buttermilk will be drained and stored in containers (<https://www.journalofdairyscience.org/>)

Blending of Biolubricants

The experimental design will involve treatments of six (6) samples; namely Animal fat (Tallow), Shea Butter, Buttermilk and palm oil. Others include the (50:50) blends obtained from these four oils. The six (6) blends mixed in a container in order to obtain a homogeneous whole Osemeahon (2024) were studied as well as those obtained from the addition of lubricant additives separately in order to ascertain the effects of additives on the biolubricants. The blends and samples containing additives were code-named for easy identification and classification Maitera (2024). Palm oil and buttermilk BPOL, palm oil and shea butter SBPOL, Tallow and Shea butter Oil TSBOL, Butter and Tallow BTOL, palm oil and Tallow oil POTOL and shea butter and butter oil, SBBOL respectively. The

essence of the blend is to determine if there will be a significant change in lubricating properties (Ozioko, 2017).

Determination of Thermal Stability

A Thermal Gravimetric Analyzer (TGA) Model TG400 PerkinElmer (Shimadzu, Japan) was used to monitor the weight changes of the samples, while the derivative of a TG curve (DTG) evaluated the rate of weight loss during heating versus temperature. Samples of 10.0 mg were placed in an aluminum crucible and exposed to inert atmosphere (40 mL/min). The temperature range for this TGA analysis extended from 30 °C to 700 °C at a constant heating rate of 10 °C/min (Maria et al., 2022).

Determination of Oxidation Stability of Biolubricant Oils

A Thermal Gravimetric Analyzer (TGA) Model TG400 PerkinElmer (Shimadzu, Japan). 1.5 mg of sample was placed into an aluminum pan with a pinhole cover to allow interaction between ester product and oxygen gas which acts as reaction gas. Samples of 10.0 mg were placed in an aluminum crucible and exposed to inert atmosphere in the presence of Nitrogen gas (40 mL/min). The temperature range for this TGA analysis extended from 30 °C to 700 °C at a constant heating rate of 10 °C/min (Maria et al., 2022).

RESULTS AND DISCUSSION

Oxidative Stability

Thermo-oxidative stability is one of the chemical properties that characterize a biolubricant. It predicts the biolubricants' service life, especially in higher temperature conditions. Oxidative stability is determined by the oxidation stability index (OSI) which is expressed as onset temperature, the temperature at which a sample loss at least 5 % mass, also is the temperature at which the occurrence of the rate of oxidation of the biolubricant samples namely: She butter, Butter from milk, tallow and palm oils occur due to exposure to air carrier gas typically nitrogen.

Effects of blending on the Thermal Stabilities of the biolubricant.

The biolubricants blends where tested under same conditions in order to ascertain the effects of applied temperatures and relatives mass losses at 10 %,20 %,50 %, 75 % and 90 % from their different onset temperatures for their thermal stabilities. The blends are: Butter and palm oil, BPOL, shea butter and palm oil, SBPOL, Tallow and shea butter oil,

TSBOL, butter and Tallow, BTOL, palm oil and Tallow, POTOL; and shea butter and butter, SBBOL.

The onset temperatures which is the temperature of 5 % mass loss for butter and palm oil biolubricant blend, BPOL was recorded at 183.8 °C. Subsequent mass losses of 10 %, 20 %, 50 %, 75 % and 90 % were experienced at temperatures 254.1 °C, 286.6 °C, 327.1 °C, 394.4 and 510.1 °C respectively as seen in figure 4.16 below. From the values obtained it can be seen also that a significant thermal event was recorded from onset temperature of 183.8 °C and the temperature at 10 % mass loss, 254.1 °C. Another observable thermal event is seen between the temperature at 50 % mass loss, 327.1 °C and the temperature at 90 % loss, 510.1 °C. The temperature at 5 % mass loss for shea butter palm oil biolubricant blend, SBPOL was recorded at 185.7 °C whereas those 10 % and 20 % were 270.8 °C and 295.3 respectively while in the temperatures 310.4 °C and 420.3 °C, the corresponding observable mass losses were recorded at 50 % and 90 % respectively. These values for shea butter and biolubricant blend, SBPOL revealed a steady increase in the temperature as the oil lost its mass. It can be seen however, that the highest thermal event for the oil was recorded between 369.4 °C and 420 °C indicating where the highest thermal event occurred. Likewise, another important thermal event was seen between the mass losses of 20 % and 50 % that is from 295.3 °C to about 310.4 °C. These values obtained for butter palm oil and shea butter palm oil biolubricant blend are similar to the work of (Braga et al 2024) in whose analysis of babassu oil and recorded temperature ranges of 330 and above. Similarly, Maria et al (2023) reported at onset temperature of 219 °C in their castor oil thermal analysis.

However, Tallow and shea butter biolubricant oil blend exhibited an elevated temperature of 520 °C at 75 % mass loss beyond which there was no any observable thermal event. Other thermal events which occurred were recorded between the onset, 199.0 °C and 10 % mass loss at 290.4 °C.

As seen in figure 4.16, there was an elevated thermal event of 410.3 °C at 90 % mass loss for Palm oil and Tallow blend, TOPOL indicating about 100 OC raise in the temperature from the 410. 8 °C at which the sample lost about 75 % of its weight. Like TSBOL, POTOL had lost 10 % mass at about 290 °C and recorded another elevated temperature of 350.1 °C at 50 % weight loss.

Butter and Tallow oil blend on the other hand lost 5 % of its weight at 163 °C and showed a tie highest thermal stability at 400.6 °C at 90 % weight loss. However, there was no significant between 50 % and 75 % weight lost at 375.4 °C and 380.1 °C respectively. Yet a significant event was recorded between onset and 10 % weight loss.

Shea butter and butter oil blend, SBBOL as seen in figure 4.17 below recorded the highest thermal stability of 560.2 °C although with the least onset temperature of 153.1 °C. The temperature of 410.6 °C at weight loss of 75 % indicated the stability of the biolubricant at higher temperatures. It can also be seen that SBBOL exhibited a thermal stability of about 267.1 before it lost at least, 10 % of its weight from the onset.

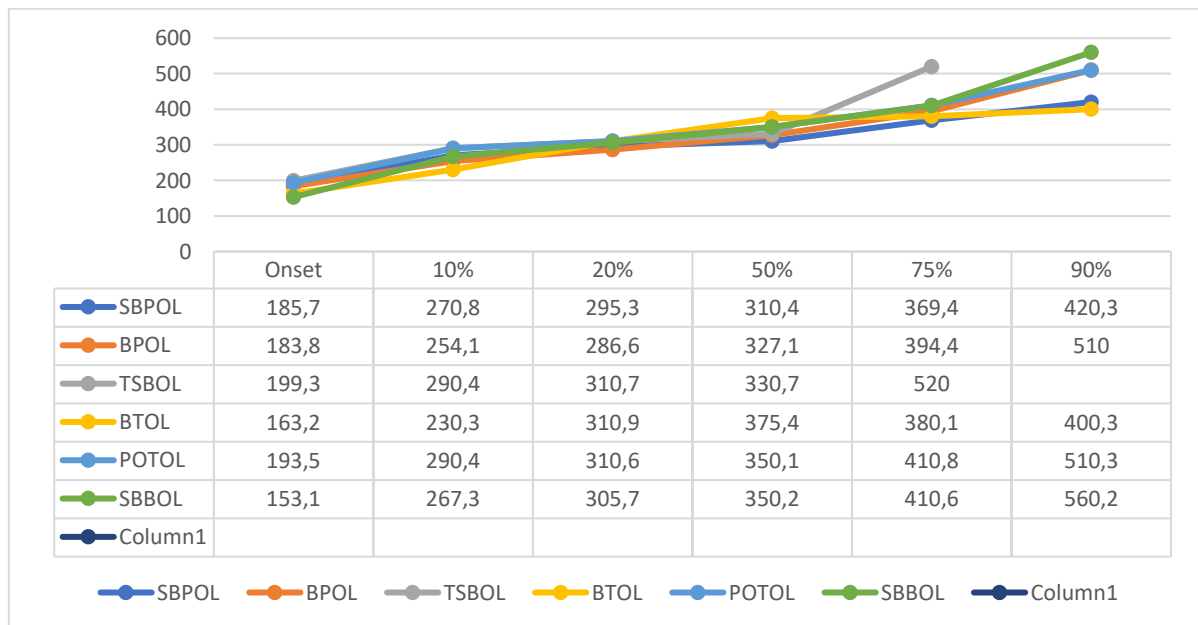


Figure 1: Thermal stabilities of the biolubricant blends

Effects of Oxidation Stabilities of the Biolubricant Blends

As seen in the figure 2 below, for the analysis of tallow and shea butter oil blend, TSBOL, an onset temperature of 185.4 °C was recorded and subsequent temperatures at mass losses 10 %, 20 %, 50 % and 90 % were recorded as 226.30 °C, 289.4 °C, 300.3 °C and 400.1 °C respectively. However, another important oxidation event occurred at 350.4 °C indicating a mass loss of about 75 % of the oil due to oxidation. Here, the largest thermal event occurred between 350.4 °C and 400.1 which is the temperature at 90 % mass loss of the blend. The value obtained however, for 50 % mass loss which was 300.1 °C is lower than the value of 312.53 °C obtained by Luna et al (2015). However, within the

value range of 174.8 °C at onset and 236.1 °C for same mass loss of 50 % as studied by Rawat and Harsha (2022).

Figure 5.17 also shows that about 148 °C was recorded as the onset temperature of the butter and tallow biolubricant blend, BTOL while 90 % mass loss occurred at 410.7 °C. At 20 % and 50 % mass losses however, the noticeable temperatures of 250.1 °C and 340.9 °C were observed. This oil experienced its significant oxidation events between these temperatures that is 148.2 °C and 190.50. Similarly, another important thermal event can be seen to have occurred between 250.1 °C and 340.9 °C indicating a 20 % mass loss due to oxidation. 75 % mass loss was also observed at 360.4 °C as seen in figure 2 below which showed another significant event.

The analysis of palm oil and tallow oil blend, POTOL a significant thermal event was recorded between the temperatures of 20 % and 50 % mass loss at 220.2 °C and 292.1 °C respectively which indicates the difference of about 88 °C. A similar although lower event occurred between the onset temperature, 159.1 °C and the temperature at 10 % mass loss, about 195.7 °C. According to Syahir et al., (2027) higher degree of branches, more hydroxyl groups, blending ratios, additives and saturation may be responsible for the improved thermal stabilities of biolubricants. Similarly, however, the blend of shea butter and milk butter had an onset temperature of 200.30 °C, 226.50 °C at 10 % mass loss. The significant oxidative events occurred between 20 % and 50 % mass loss with temperatures of 220.2 °C and 292.1 °C respectively. Another is that which is observable between mass loss of 50 % and 75 % at 229.1 °C and 310.2 °C respectively. However, at 90 % mass loss, the observed temperature was about 378.0 °C beyond which there was no any noticeable mass loss.

Kalam et al. (2017) evaluated the thermogravimetric analysis for olive oil and SAE15W40. The decomposition temperature of olive oil and SAE15W40 are 395.15 °C and 249.20 °C. As a result, as compared to lubricating oil, olive oil demonstrated greater temperature stability. The increased thermal stability of the biodegradable oil is primarily due to the high amount of unsaturated fatty acids present.

The oxidation stabilities of the oil blends show the effects of the blends of these oils as they are exposed to a various temperature over time. Figure 2 revealed that shea butter blend with palm oil, SBPOL exhibited longer oxidative stability of about 400 °C at 90 % mass loss at its highest oxidative event. It is event that between the onset of sample

SBPOL, 168.1 °C and 10 % mass loss, a significant oxidative event was also noticed at a temperature of 230.8 °C and another 263.4 °C at 20 % mass loss. When butter and palm oil were blended however, BPOL an onset of 170.7 °C was recorded with the highest oxidative event occurring at 370 °C indicating a 90 % mass above which there was no any noticeable event which showed that it's the least stable oil at oxidative activity. Between 268.1 °C and 320.3 °C was another oxidative event showing 50 % and 75 % mass losses respectively. Similar event was noticed at 257.3 °C between 10 % and 20 % mass loss oil the oil.

Butter and tallow oil blends, BTOL showed greater oxidation stability than BPOL. In that, BTOL showed a steady oxidation resistant to about 400 °C at 90 % mass loss the two important events one occurred at 340.9 °C between 20 % and 50 % mass loss and 410.7 °C between 75 % and 90 5 mass loss. Tallow blend with shea butteroil, TSBOL, palm oil and tallow, POTOL exhibited similar trends of mass losses of 90 5 at 300.3 °C and 262.1 °C at 50 % mass loss. However, POTOL showed similar oxidative stability as BPOL of 310.2 °C and 320.3 °C respectively Similarly, as seen in Figure 2 below it is evident that Shea butter and butter oil, SBBOL however had the highest oxidation stability of about 450 °C at 90 % mass loss similar to that of butter and palm oil, BTOL as seen in the 4.17 below. Their varying oxidative stabilities of the lube blends indicate an approximation on the time it will take for the biolubricant to show a tremendous decline in their activities at same temperature due to exposure with oxygen.

The modified chicken fat MCF with onset temperature OT:189.5 OC displayed improved oxidative stability compared to the RCF (OT:153.9 OC as indicated by the increased OT value. The degree of unsaturation is the main factor that affects the oxidative stability of fatty acids and higher degree of unsaturation corresponds to lower oxidative stability. Higher OT corresponds to better oxidative stability which means the lipid is resistant to oxidation and it can be useful for a longer period.

Thus, the higher oxidative stability of MCF compared to the Regular Chicken fat , RCF and High oleic sunflower oil HOSuO can be attributed to the reduction in the number of unsaturated bonds. This also confirms that the double bonds of the unsaturated fatty acid reacted with the alkyl halide to form isopropyl branches according to a study carried out by Hailemichael et al., (2022). The effect of the modified chicken fat concentration on kinematic viscosity of the HOSuO blends at 40, 75 and 100 °C. Blends of

tallow methyl esters and diesel was used on four stroke engine by Nautiyal et al (2017) and also reduction in exhaust emissions of carbon oxides and hydrocarbon were recorded except a slight increase in nitrogen oxides. Similar result was obtained by Shahir et al. (2022)

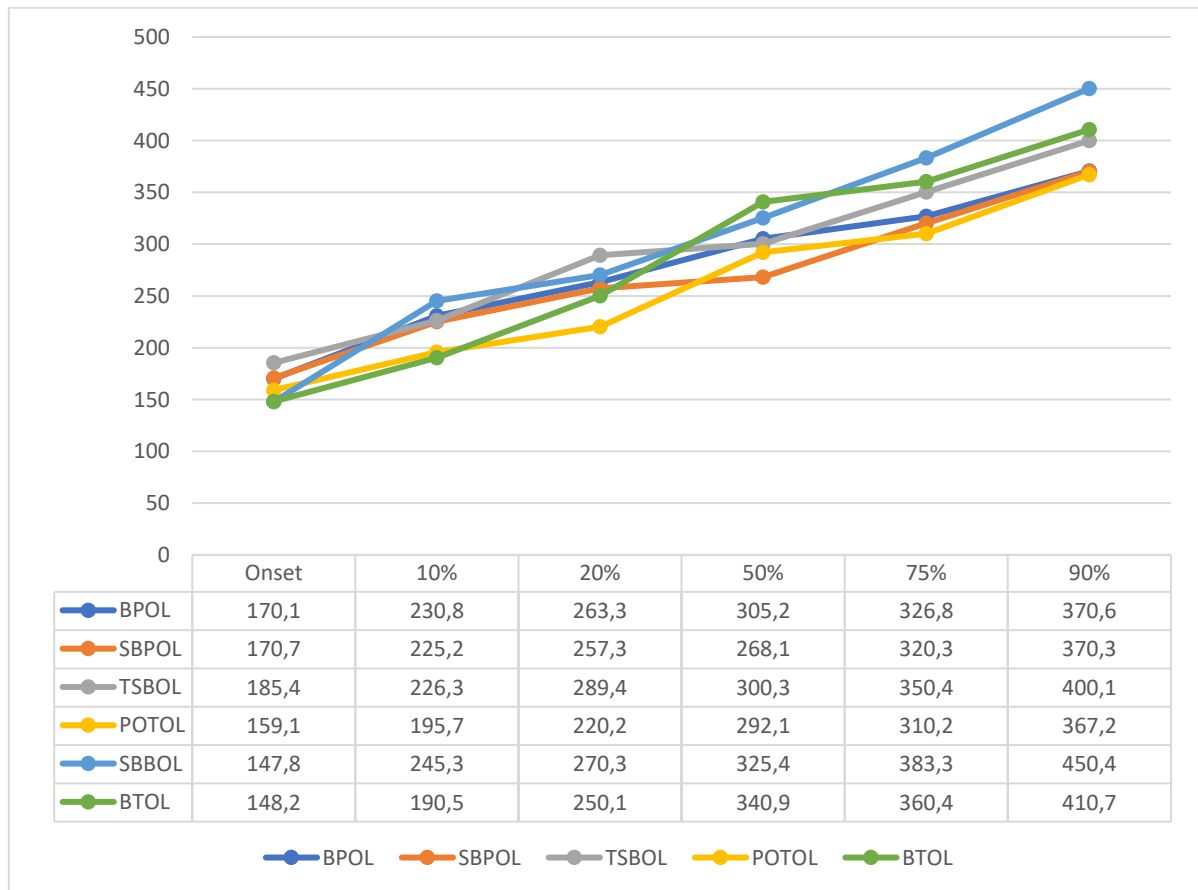


Figure 2: Plot of Oxidation stabilities of the biolubricants Blends

CONCLUSION

The thermos-oxidative investigation of the biolubricant oil blends of Tallow, Butter, Palm oil and butter revealed a significant raise both thermal and Oxidation stabilities of these oils at different weight losses. However, the highest Thermoxidative performance was recorded for shea butter and butter, SBBOL and Palm Oil and Tallow blends, POTOL at 90 % weight loss. On the other hand however, due to oxidation as a result of a nitrogen as a carrier gas, palm oil and tallow oil and butter and palm oils blends, suffered a drastic oxidation stability over a time. It is evident that blending of biolubricants

of plant and animal origins have thermoxidative potentials of increased stabilities in the presence and absence of oxygen.

Conflict of Interest

The authors affirm that there are no conflicts of interest associated with this publication.

Authors' Declaration

The authors confirm that the research presented in this article is entirely original. They accept full responsibility for any claims or issues arising from the content herein.

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