# Comprehensive Analysis of Physicochemical, Rheological, Tribological and Thermal characteristics of Transesterified Grape seed oil, Neem oil and Waste cooking oil for environment friendly lubricant applications

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**Abstract -** *This study provides a thorough examination of transesterified grape seed oil (TGSO), neem oil (TNO), and waste cooking oil (TWCO) as potential biolubricants. The oils were chemically changed via a transesterification technique to produce their corresponding methyl esters. The physicochemical, rheological, and tribological properties of both modified and untreated oils were thoroughly examined. The physicochemical investigation found that transesterification generally improved oil characteristics. TWCO had lower acid value (0.53 mgKOH/g), peroxide value (1.87 meq/kg), tox value (4.27), and density (0.734 g/cm$^3$) than untreated waste cooking oil (WCO). TGSO and GSO had lower acid and peroxide levels, indicating higher oxidative stability. The rheological properties, particularly the viscosity-temperature relationship, were also examined in this study. WCO had considerably higher viscosity values over the temperature range, whereas transesterified oils had lower viscosities. Tribological studies were carried out to determine the coefficient of friction (COF) and wear scar diameter (WSD). WCO had the lowest COF (0.0372) and WSD (541.009 µm), indicating enhanced lubrication and wear resistance. However, transesterified oils had greater COF and WSD values than their untreated counterparts, indicating a possible trade-off between oxidative stability and tribological performance. Thermal performance and energy efficiency were assessed using the flash temperature parameter (FTP) and energy consumption (TE). GSO had the highest FTP value (71.78), showing greater thermal stability, whereas WCO had the highest TE value (0.0525), indicating increased energy consumption. The study concludes that transesterification can increase the physicochemical qualities of vegetable oils, but it may impair tribological performance. Waste cooking oil had appealing tribological capabilities due to its high viscosity and polar components, but its physicochemical properties were quite poor. The thermal conductivities of unmodified oils were higher than that of the chemically modified oils. WCO was found to have the highest thermal conductivity and the least thermal conductivity was observed for TGSO. Lubricant compositions should be optimised by balancing their physicochemical and tribological qualities using additive packages or blending procedures.*

***Key Words***: **Transesterification, Totox Value, Flash Temperature Parameter, Energy Consumption, Thermal Conductivity.**

## 1.INTRODUCTION

Lubricants have a significant role in reducing friction between surfaces in contact. Bio lubricants manufactured from vegetable oils such as soybean, sunflower, and rapeseed offer an environmentally benign alternative to petroleum-based lubricants. These bio lubricants have a higher viscosity than mineral oils and provide benefits such as biodegradability, renewable resources and improved anti-friction qualities. However, some constraints, such as low oxidation stability and heat stability, must be addressed. Several research investigations have looked into how these bio lubricants are made, their properties, and the challenges that they confront.

Grape seed oil (GSO), a byproduct of the wine industry, has numerous potential applications in healthcare and beyond. Packed with beneficial nutrients and compounds that act as antioxidants, grape seed oil is being explored as a source of nutraceuticals [1]. Researchers have also investigated its lubricating properties, particularly when combined with SiO2 and TiO2 nanoparticles. The findings revealed that this blend of grapeseed oil and nanoparticles exhibited exceptional tribological properties, making it a promising environmentally friendly lubricant [2]. Additionally, studies have explored alternative uses for grape seed oil that do not involve consumption. One such study demonstrated that grape seed oil can be transformed into eco-friendly materials. Two specific processes were examined: converting grape seed oil into biodiesel when mixed with methanol, which remains liquid in cold temperatures, and adding oxygen to grape seed oil through an epoxidation reaction. Both processes have the potential to create biodegradable lubricants. Grape seed oil holds promise in creating eco-friendly lubricants that can easily decompose [3]. Additionally, this oil has proven to be advantageous for the skin and is free of any scent or flavour, making it a popular choice for natural personal care products [4]. As a result, grape seed oil has demonstrated its versatility and potential in a range of applications, especially as a lubricant. Its adaptability when combined with nanoparticles and its ability to contribute to the production of sustainable materials further emphasise its benefits.

Extracted from the seeds of the Azadirachta indica tree, neem oil (NO) is renowned for its potential as a bio-lubricant. This is due to its composition of fatty acids, namely oleic and stearic acids [5]. A recent investigation has demonstrated that cutting fluids formulated with neem oil can effectively lessen the force required for machining. By utilising cutting fluids derived from vegetable oils, productivity can be enhanced, resulting in higher machining rates. Interestingly, the utilisation of plain water, without any lubricant, can actually improve machining performance. Vegetable oils possess inherent lubricating properties that can be harnessed to create cutting fluids that outperform traditional mineral oil-based counterparts. These cutting fluids have proven to be effective in reducing cutting forces during machining. Additionally, they have been found to enhance the surface quality of machined surfaces, resulting in superior surface finishes. It has also been observed that the implementation of bio cutting fluids based on neem oil can extend the lifespan of tools by diminishing the rate of tool wear [6]. Extensive studies have shown that the incorporation of stearic acid into neem oil significantly enhances wear resistance and diminishes the coefficient of friction, rendering it a superior alternative to conventional mineral oil options [7]. Research has found that neem oil has potential applications in the production of biodiesel and biolubricants, as well as in machining processes. The oil was extracted using a solvent extraction method with N-hexane as the solvent, and its chemical and physical properties such as density, pour point, acid value, and free fatty acid (FFA) level were analysed. The results indicate that neem oil shows promise in lubrication fields. The presence of fatty acids like oleic and stearic acids in neem seed oil makes it suitable as a lubricant for machining mild steel. Furthermore, the use of neem seed oil as a lubricant reduces friction between the tool and workpiece, as evidenced by the steady decrease in the coefficient of friction related to chip formation [8]. Additionally, neem oil-based biolubricants exhibit superior tribological, thermal, rheological properties, and biodegradability [9].

Waste cooking oil (WCO) is a by-product of the food industry that can be used to make biofuel. WCO consists of various compounds like triacylglycerols, glycerols, free fatty acids (FFAs), and polymerization compounds. It is dark, thick, and has a low smoking point [10]. In a detailed review by Manikandan et al., the health effects of using used cooking oil were discussed, along with the potential for generating income from WCO. The review found that blending WCO with diesel up to 20% reduces pollutants without requiring significant engine modifications. Additionally, the study suggests that rural farmers could benefit from a circular economy and create sustainable income [11]. Another study focused on chemically modifying WCO to produce bio-lubricants. The study showed that through chemical reactions, WCO's characteristics can be improved, making it suitable for use as a bio-lubricant in various industries [12]. Abdulbari et al.'s research explored the use of waste, specifically from palm oil industries, to create a new multi-purpose grease. The utilisation of base oil and guar gum as a thickening agent in a recently formulated grease has been thoroughly examined. This article extensively analyses the characteristics of the resulting grease [13]. Additionally, a comprehensive review of existing literature was conducted to address various concerns surrounding the application of newly developed lubricants derived from vegetable oils. The potential of utilising waste cooking oil as a lubricant for tribological purposes was also explored [14]. Furthermore, a separate study investigated the creation of composite materials for construction applications using waste cooking oil as a binding agent. The study examined the process parameters involved in incorporating solid materials with vegetable oil as a binder, as well as evaluated their strength and absorbency [15].

This study examines the transformation of grape seed oil, neem oil, and waste cooking oil into methyl esters through a transesterification reaction. The chemically modified oils are then subjected to physicochemical, rheological, tribological and thermal analyses, which are compared to the unmodified oils. The uniqueness of this research lies in its comprehensive investigation of three different vegetable oil sources, assessing their properties before and after transesterification. The study evaluates various properties of these oils and highlights the potential trade-offs between improved stability and optimum tribological performance. To achieve a balance for specific bio-lubricant applications in different industries, the study suggests the need for optimization through additive packages or blending strategies.

## 2. MATERIALS AND METHODS

### 2.1 Materials

Grape seed oil was purchased from BRM Chemicals, Delhi. Neem oil was procured from Krishna Ayurvedics, Thiruvananthapuarm, Kerala. Waste cooking oil was obtained from Toc H Institute Canteen, Kochi, Kerala. The corresponding neet oil was coconut oil and was replaced when used for frying for two consecutive times at a maximum temperature of 100°C. This oil was taken as waste cooking oil for this study. All chemicals used in this study such as Potassium hydroxide, Methanol and distilled water were purchased from Nice Chemicals Ltd, Kochi, Kerala.

## 2.2 Transesterification of GSO, NO and WCO

A known quantity of the oil was filtered using Whatman filter paper, taken in a beaker, heated to 80°C in a hot air oven with constant air flow for 30 minutes and was left to cool gradually to room temperature. This was done to remove any moisture content present in these oil samples. After cooling, known quantities of potassium hydroxide and methanol were added to it and were kept for stirring in a water bath at 60°C for one hour (Fig. 1). Following this the mixture was kept in a separating funnel overnight. Separation of the mixture takes place slowly and two layers are formed. The upper layer is the methyl ester of the oil and the lower layer is glycerol (Fig. 2). The methyl ester was separated out and was water washed five times using warm distilled water (60°C). This was done to remove any unreacted chemicals and impurities in the sample. Following this the sample was heated under vacuum to remove the remaining moisture and the final sample of the methyl ester was obtained. The final products were Transesterified Grape seed oil (TGSO), Transesterified Neem oil (TNO) and Transesterified Waste cooking oil (TWCO). The chemical reaction taking place is depicted by the chemical formula as shown below.

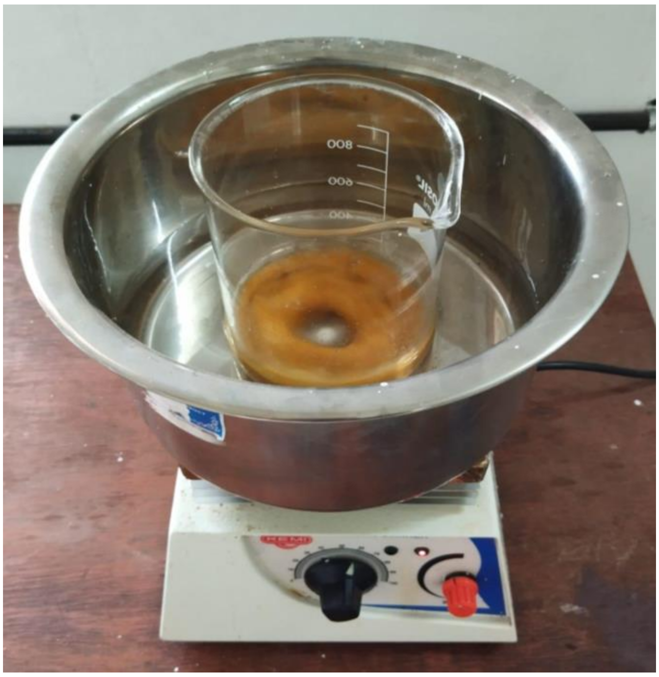


**Figure -1**: Transesterification reaction setup

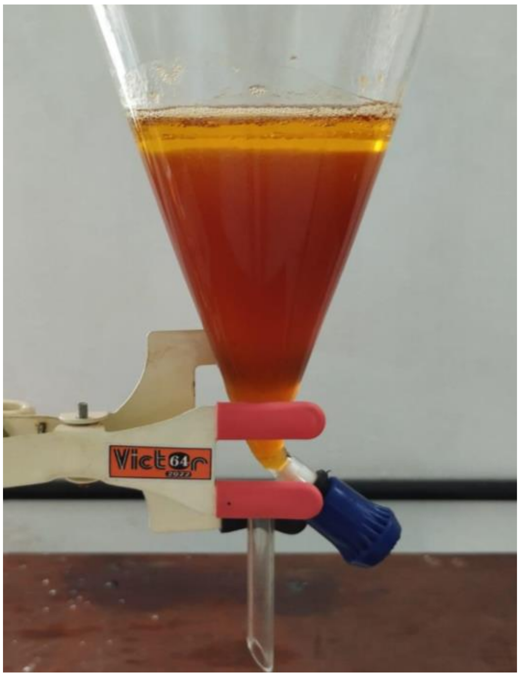


**Figure -2**: Separating funnel setup

## 2.3 Physicochemical properties of GSO, NO, WCO and TGSO, TNO, TWCO

Physical properties such as colour, density and pH were measured for both modified and unmodified samples. Colour of the oil samples were identified by visual inspection. Density was measured at room temperature as per ASTM D369. pH was measured using a standard pH paper. Chemical properties such as Acid value (AV), Peroxide value (PV) and Iodine value (PV) were determined as per IS 548 part 1 (1964) standards. Totox value (TV) was calculated using the equation

$$TV = AV + 2.PV \qquad \text{……….(1)}$$

## 2.4 Rheological properties of GSO, NO, WCO and TGSO, TNO, TWCO

Rheological properties such as kinematic viscosity for all the samples were evaluated using Canon Fenske viscometer as per ASTM D445 standards.

## 2.5 Tribological properties of GSO, NO, WCO and TGSO, TNO, TWCO

Tribological properties such as Coefficient of friction (COF) and Wear scar diameter (WSD) were determined using a four ball tribo tester as per ASTM D4172 standard. The test conditions were 392 N load, 1200 rpm spindle speed, 75°C temperature and 3600 seconds running time.

## 2.6 Flash temperature parameter and Energy consumption of GSO, NO, WCO and TGSO, TNO, TWCO

Flash temperature parameter (FTP) and Energy consumption (Total energy consumed (TE)) are two important parameters in evaluating the performance and energy efficiency of lubricants and fuels in combustion processes. FTP is a measure of the maximum temperature attained by a lubricant film during transient elastohydrodynamic lubrication conditions, such as those encountered in gear tooth contacts or rolling element bearings. A higher FTP value generally indicates better lubricant performance and resistance to thermal degradation [16]. FTP is calculated using the equation

$$FTP = W/(d^{1.4}) \qquad \text{……….(2)}$$

Where, d = mean wear scar diameter (mm) and W = applied load (kg).

TE represents the amount of energy required for a given process or operation. In the context of lubricants and fuels, it is often associated with the energy losses due to friction and other inefficiencies. Lower TE values are desirable as they indicate better energy efficiency and reduced energy consumption [17]. TE is calculated using the equation

$$TE = fgwr/1000 \qquad \text{……….(3)}$$

Where, TE = thermal energy generated (Joules), g = acceleration due to gravity (m/s2), f = coefficient of friction, w = load applied (kg) and r = distance between the centre of the contact surface on the lower balls and the axis of rotation (i.e.3.67 mm).

## 2.7 Thermal Conductivity of GSO, NO, WCO and TGSO, TNO, TWCO

The thermal conductivity of the oil samples (GSO, NO, WCO, TGSO, TNO, and TWCO) was determined using the liquid thermal conductivity apparatus according to ASTM D7896. The apparatus was set up and the water circulation was established to maintain a steady flow rate through the test section. The cooling water flow rate was measured and recorded. Using the recorded temperatures and the known dimensions of the test section (thickness of the liquid film and area of the test section), the thermal conductivity of the liquid sample was calculated using the formula

$$Q = K.A.\ (T1- T2)/\Delta x \qquad \text{………(4)}$$

Where Q is the rate of heat transfer, k is the thermal conductivity of the liquid, A is the area of the test section, T1 and T2 are the temperatures measured before and after the test section, respectively, and Δx is the thickness of the liquid film. The experiment was repeated for each oil sample (GSO, NO, WCO, TGSO, TNO, and TWCO) to obtain their respective thermal conductivity values.

## 3. RESULTS AND DISCUSSIONS

Table 1 shows the physicochemical properties of various oil samples. The colour of the oils (Fig. 3) indicates the presence of impurities, oxidation products, and other chemical components. For instance, WCO's dark brown colour results from exposure to high temperatures during cooking, which leads to the formation of oxidation products [18]. The pH values of all the oil samples range from 6 to 8, which is typical for vegetable oils. A slightly acidic or neutral pH is preferred for lubricant applications to prevent corrosion and compatibility issues [19]. The density values range from 0.612 g/cm$^3$ (TGSO) to 0.876 g/cm$^3$ (WCO). Higher densities generally suggest the presence of heavier molecular components or impurities [20]. The transesterification process decreases density by converting triglycerides (the denser part of the oil) into lighter fatty acid esters [21].

**Table -1**: Physicochemical properties of GSO, NO, WCO and TGSO, TNO, TWCO

| Oil Sample | Physical Properties | | | Chemical Properties | | | |
|---|---|---|---|---|---|---|---|
| | Colour | pH | Density (g/cm$^3$) | Acid Value (mgKOH/g) | Peroxide Value (meq/kg) | Totox Value | Iodine Value (gI/100g) |
| GSO | Light Brown | 6 | 0.701 | 1.13 | 7.71 | 16.55 | 109 |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| NO | Light Green | 7 | 0.736 | 7.83 | 2.17 | 12.17 | 103 |
| WCO | Dark Brown | 6 | 0.876 | 1.45 | 9.78 | 21.01 | 93 |
| TGSO | Light Orange | 7 | 0.612 | 0.67 | 1.39 | 3.45 | 67 |
| TNO | Light Yellow | 8 | 0.657 | 1.25 | 1.46 | 4.17 | 71 |
| TWCO | Light Brown | 8 | 0.734 | 0.53 | 1.87 | 4.27 | 64 |

The quantity of unbound fatty acids present in the oil is indicated by the acid value. Increased acid values, such as 7.83 mgKOH/g for NO, can result in elevated levels of corrosion and oxidation [22]. Through the transesterification process, the acid value is typically reduced by transforming unbound fatty acids into esters [23]. The peroxide value serves as a gauge for the extent of oxidation in the oil. Elevated peroxide values, like 9.78 meq/kg for WCO, indicate the existence of oxidation products that can have a negative impact on the performance of lubricants [24]. The peroxide value can be decreased through transesterification and the inclusion of antioxidants [25]. The totox value combines the acid value and peroxide value to offer a comprehensive assessment of the oil's oxidative deterioration. Higher totox values, such as 21.01 for WCO, signify greater oxidation and potential instability [26]. The iodine value measures the level of unsaturation in the oil. Higher iodine values, like 109 gI/100g for GSO, indicate a larger proportion of unsaturated fatty acids which can affect properties like oxidative stability and viscosity [27].

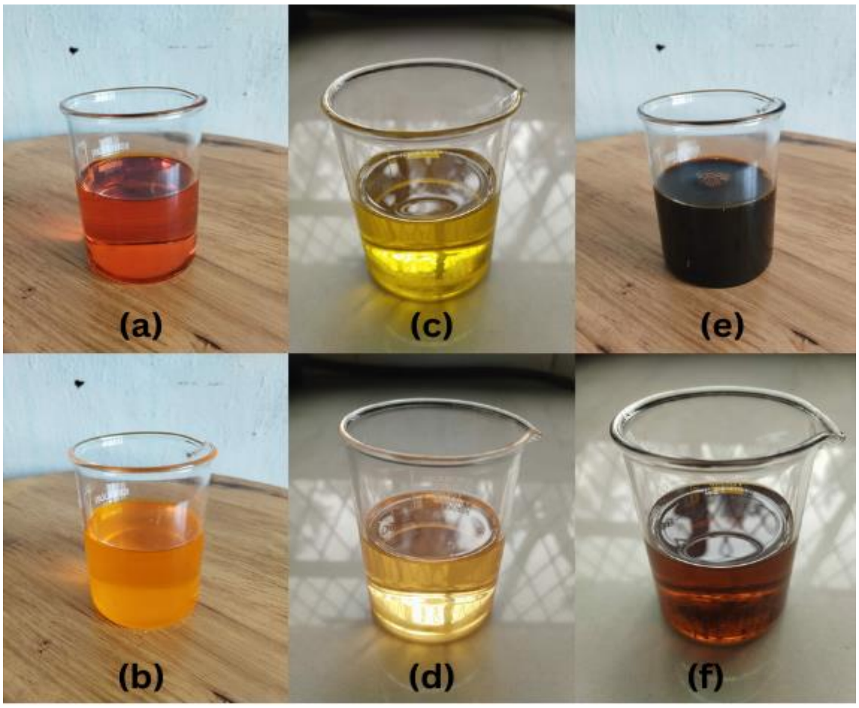


**Figure -3:** Various oil samples a) GSO b) TGSO c) NO d) TNO e) WCO f) TWCO

The transesterification process, which converts triglycerides into fatty acid esters, can improve the properties of the oils for lubricant applications by reducing the acid value, peroxide value and density [28]. The data suggests that Waste cooking oil (WCO) has inferior qualities when compared to the other oil samples. It has higher levels of acid value (1.45 mgKOH/g), peroxide value (9.78 meq/kg), totox value (21.01), and density (0.876 g/cm$^3$). These values indicate a greater extent of oxidation and degradation, likely due to exposure to high temperatures during cooking. The process of transesterification, which converts triglycerides into fatty acid esters, generally enhances the properties of oils. For instance, transesterified waste cooking oil (TWCO) exhibits lower acid value (0.53 mgKOH/g), peroxide value (1.87 meq/kg), totox value (4.27), and density (0.734 g/cm$^3$) compared to untreated WCO. Grape seed oil (GSO) and its transesterified form (TGSO) have lower acid values (1.13 and 0.67 mgKOH/g, respectively) and peroxide values (7.71 and 1.39 meq/kg, respectively) compared to the other oil samples. This indicates better stability against oxidation and lower levels of free fatty acids, making them potentially suitable for lubricant formulations. Neem oil (NO) has a relatively high acid value (7.83 mgKOH/g), which can cause increased corrosion and oxidation problems. However, its transesterified form (TNO) shows a significant improvement in this property, with a much lower acid value (1.25 mgKOH/g). The levels of unsaturation, which indicate the amount of double bonds, differ among the oil samples. GSO has the highest value (109 gI/100g), while TGSO has the lowest value (67 gI/100g). Higher iodine values can affect properties such as stability against oxidation and thickness, which can impact the performance of lubricants.

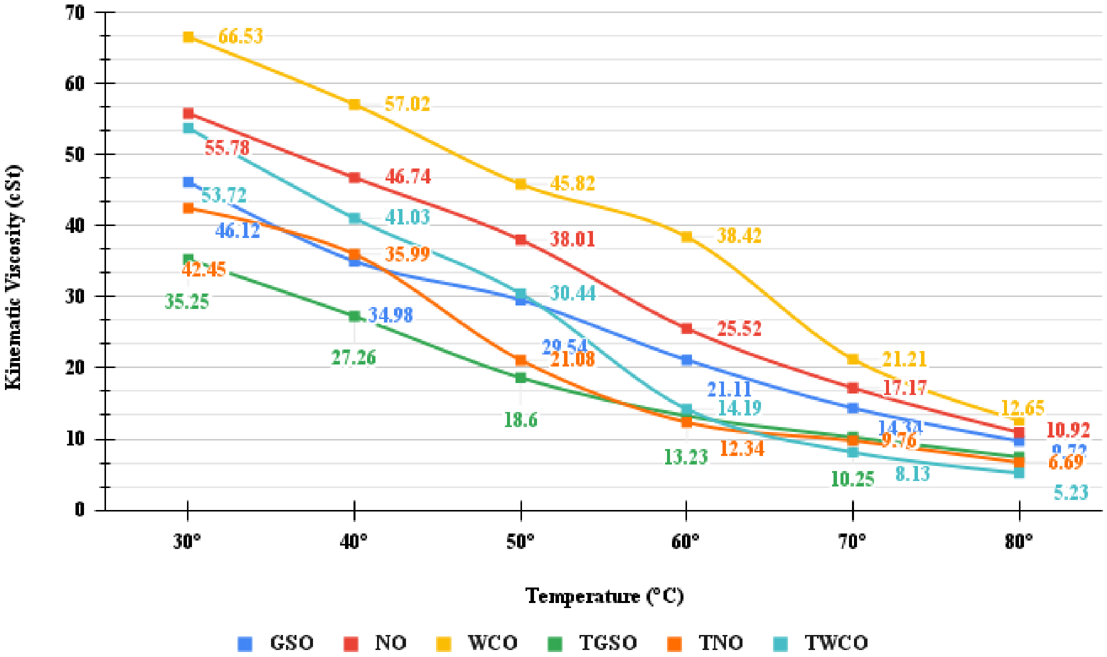


**Chart - 1**: Kinematic Viscosity vs Temperature of various oil samples

Chart 1 displays how the viscosity of different oil samples (GSO, NO, WCO, TGSO, TNO, and TWCO) changes as the temperature increases. This temperature-viscosity relationship is a common behaviour observed in lubricating oils. The viscosity of these oils is greatly influenced by temperature. As the temperature rises, the movement of molecules within the oil increases, causing a decrease in intermolecular forces and a subsequent decrease in viscosity. This trend is evident for all the oil samples shown on the graph. The viscosity-temperature behaviour of the oils is affected by their chemical composition, including the presence of triglycerides, fatty acid esters, and other components. For example, WCO has higher viscosity values throughout the temperature range due to its higher molecular weight and the formation of polar compounds during cooking. On the other hand, transesterified oils (TGSO, TNO, and TWCO) generally have lower viscosity values compared to their untreated counterparts (GSO, NO, and WCO) at the same temperatures. This is because the transesterification process converts triglycerides into lighter fatty acid esters, resulting in a decrease in viscosity. WCO demonstrates elevated levels of viscosity throughout various temperatures, making it a potential choice for applications that need lubricants with higher viscosity. Transesterified oils (TGSO, TNO, and TWCO) have lower viscosity compared to their untreated counterparts, which can be beneficial for applications that require lubricants with lower viscosity or improved performance in low temperatures.

**Table -2**: Tribological Properties of GSO, NO, WCO and TGSO, TNO, TWCO

| Oil Sample | Tribological Properties | |
|---|---|---|
| | Mean COF | Mean WSD (µm) |
| GSO | 0.0582 | 649.109 |
| NO | 0.0456 | 667.220 |

| | | |
|---|---|---|
| WCO | 0.0372 | 541.009 |
| TGSO | 0.0815 | 776.113 |
| TNO | 0.0778 | 782.663 |
| TWCO | 0.0672 | 765.194 |

Table 2 displays the tribological characteristics of the different oil samples, specifically the average coefficient of friction (COF) and average wear scar diameter (WSD). These properties are essential for assessing the lubricating performance of the oils and their potential for use in creating biodegradable lubricants. COF measures the level of friction between two surfaces that are sliding against each other. Lower COF values are preferred because they signify better lubrication and reduced friction, resulting in lower energy losses and wear. Waste cooking oil (WCO) has the lowest average COF of 0.0372, which can be attributed to its high viscosity and the formation of polar compounds during the cooking process, contributing to improved lubrication [29]. Transesterified oils (TGSO, TNO, TWCO) exhibit higher average COF values compared to their untreated counterparts (GSO, NO, WCO). This is due to the decrease in viscosity and removal of polar components during the transesterification process, which affect the lubricating ability [30]. Neem oil (NO) has a relatively low average COF of 0.0456, thanks to the presence of natural antioxidants and polar compounds that enhance lubrication [31]. It is ideal for the WSD to have smaller values, as this indicates lower wear and better protection for the surfaces that come into contact. In comparison to the other samples, GSO and NO have relatively high WSD values of 649.109 μm and 667.220 μm, suggesting lower wear resistance. On the other hand, WCO has the lowest WSD of 541.009 μm, which indicates better wear protection. This is attributed to its high viscosity and the presence of polar compounds that are formed during the cooking process [32]. The transesterified oils (TGSO, TNO, TWCO) generally have higher WSD values compared to their untreated counterparts. This suggests a potential decrease in wear resistance due to the removal of polar compounds during transesterification [33]. Among all the samples, WCO demonstrates the most promising performance in terms of tribological properties, with the lowest COF (Coefficient of Friction) and WSD values. This is mainly due to its high viscosity and the presence of polar compounds formed during cooking. However, it should be noted that WCO's physicochemical properties, such as its high acid value and peroxide value, may limit its direct application as a lubricant without further treatment or the addition of additives.

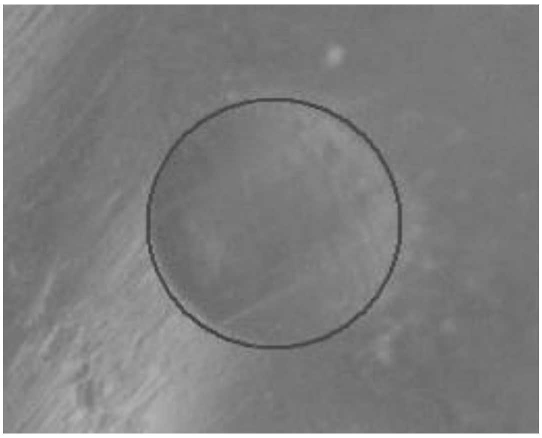
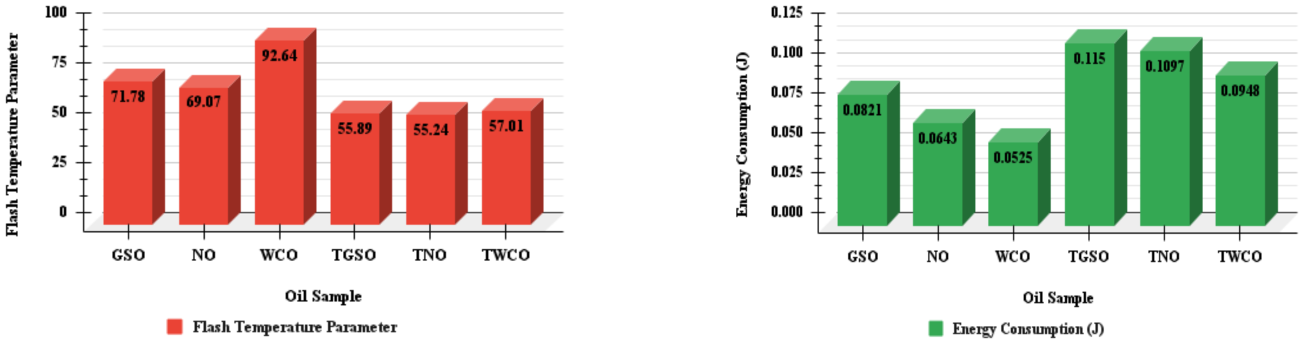


**Fig -4**: Optical image of wear scar for TWCO

Fig 4. shows the wear scar image of TWCO sample. The wear scar is circular in shape with a relatively smooth appearance within the scar region, suggesting uniform wear during the testing process. The outer periphery of the scar exhibits a distinct boundary, separating the worn area from the surrounding unworn surface. In order to enhance the quality of lubricants, it is crucial to find a harmonious combination of certain properties like oxidative stability and tribological performance. This can be achieved by using suitable additive packages or blending strategies, which help maintain the balance between physicochemical properties and tribological properties such as coefficient of friction (COF) and wear resistance.

**Chart -2**: FTP and TE values for various oil samples



(a) (b)

Chart 2 shows the flash temperature parameter (FTP) and energy consumption (TE) values for various oil samples. The data reveals that GSO has the highest FTP value of 71.78, implying superior thermal stability and performance in high-temperature situations. Conversely, the WCO sample has the highest TE value of 0.0525, indicating greater energy consumption compared to the other samples. Higher FTP values are generally preferable for enhanced thermal stability and lubricant performance, particularly in high-temperature conditions. Lower TE values signify improved energy efficiency and reduced energy consumption, which can result in cost savings and environmental benefits.

**Chart -3**: Thermal Conductivity of GSO, NO, WCO and TGSO, TNO, TWCO

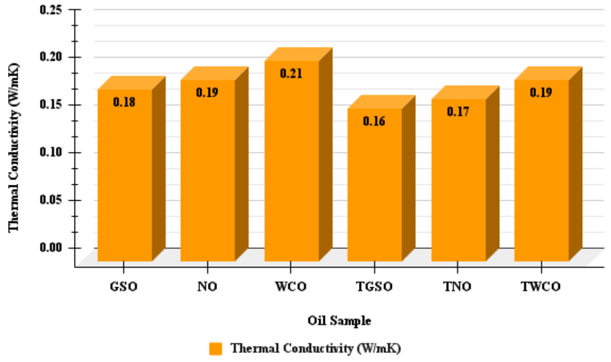


Chart 3 shows the thermal conductivity values of various oil samples. Due to the formation of polar compounds and increased molecular interactions during the cooking process, Waste cooking oil (WCO) exhibited the highest thermal conductivity (≈0.21 W/m.K). The transesterification process generally led to a slight decrease in thermal conductivity caused due to the changes in molecular structure and intermolecular forces after the conversion of triglycerides to fatty acid esters. The differences in thermal conductivity values among the samples can impact their performance in applications where heat transfer is crucial, such as lubricants or heat transfer fluids. Higher thermal conductivity can improve heat dissipation and thermal management, while lower values may result in poorer heat transfer characteristics.

## 4. CONCLUSIONS

- Transesterified oils (TGSO, TNO, TWCO) exhibited lower physicochemical properties like acid values, peroxide values, and densities compared to their untreated counterparts (GSO, NO, WCO). This reduction in physicochemical properties enhances the oxidative stability and reduces corrosion tendency of these transesterified oils.
- Transesterified oils (TGSO, TNO, TWCO) displayed lower viscosity values compared to their untreated counterparts making these oils potentially beneficial for low-temperature applications.

- Waste cooking oil (WCO) demonstrated the lowest coefficient of friction (COF) and wear scar diameter (WSD), indicating improved lubricity and wear resistance. However, its physicochemical properties, such as high acid value and peroxide value, may limit its direct application without further treatment or additive formulation. Transesterified oils generally exhibited higher COF and WSD values compared to their untreated counterparts, suggesting a potential decrease in tribological performance.

- Grape seed oil (GSO) had the highest FTP value, indicating better thermal stability and performance under high-temperature conditions. Waste cooking oil (WCO) had the highest TE value, suggesting higher energy consumption compared to other samples.

- Appropriate additive packages or blending strategies should be explored to achieve the desired combination of properties for specific applications.

- Further research and development are needed to address the limitations and trade-offs identified, and to explore the viability of these bio-based lubricants in real-world applications.

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