

Review

Influence of Molecular Structure on the Physicochemical and Tribological Properties of Biolubricants: A Review

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Abstract: The increase in the price of crude oil, the environmental impact, or the depletion of fossil resources has increased the need for bio-based alternatives. This has led to the search for renewable, biodegradable, and environmentally friendly raw materials to obtain lubricants that meet these characteristics. This review deals with the state of the art of biolubricants along with their most common raw materials and molecular structures, processes of chemical modification of bio-oils, as well as the relationship between their structural features and physicochemical/tribological properties. This review concludes that the production of fatty acid alkyl esters from vegetable oils is the most promising chemical route to produce a wide range of biolubricants through double transesterification reactions. It also highlights the need to explore this route for the production of microalgae-derived biolubricants due to its environmental benefits during cultivation and production processes.

Keywords: biolubricant; molecular structure; tribological properties; physicochemical properties

1. Introduction

Water and food shortages are just some of the direct consequences of global warming caused by the increase in CO₂ emissions in recent decades. According to Lindsey's report [1], global sea levels have risen 24 cm since 1880, triggered by the start of the second industrial revolution. The highest peak emissions in history were recorded last year with 40.5 tons, behind pre-COVID-19 levels of 40.9 tons, with the use of fossil sources being the main emitter of greenhouse gases, leading with 90.47%, of which 25% came from energy losses [2].

Maintaining machinery properly, conserving energy, and finding potential substitutes for petroleum derivatives are necessary to combat accelerating climate change. One of the sciences that can contribute to this purpose is tribology, which is responsible for optimizing lubrication in the interaction between moving surfaces, and which has led to large amounts of energy and money being saved in industry. Jost [3] estimated that GBP 515 million could be saved by improving tribological conditions in the UK industry. In addition, recent research into natural lubricant sources is helping to move society toward more sustainable industries.

To achieve these goals, it is necessary to review the minimum requirements for lubricants, the advantages and disadvantages of natural sources, and the existing techniques for improving these materials to produce potential alternatives to petroleum-based lubricants. The basic functions of lubricants can be summarized as follows: (a) to reduce energy losses; (b) to protect surfaces from wear due to friction; (c) to protect against corrosion; (d) to reduce oxidation effects; (e) to cool down surfaces; (f) to decrease heat losses due to contact between moving surfaces; or (g) to increase tightness and prevent the leakage of contaminants and sediments [4,5], in addition to certain requirements depending on the application.

Lubricants can be classified according to their physical state as solid, liquid, or semi-fluid (greases). The former is used when it is difficult to maintain contact with the fluid,



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while the latter is used in situations where liquid lubricants are not applicable. They can be further classified according to their source material as mineral-, synthetic-, animal-, or vegetable-based lubricants (Figure 1). All base fluids directly refined from crude oil are called mineral bases, those refined from natural sources are called vegetable bases, and those synthesized (natural or mineral) are called synthetic bases [6].

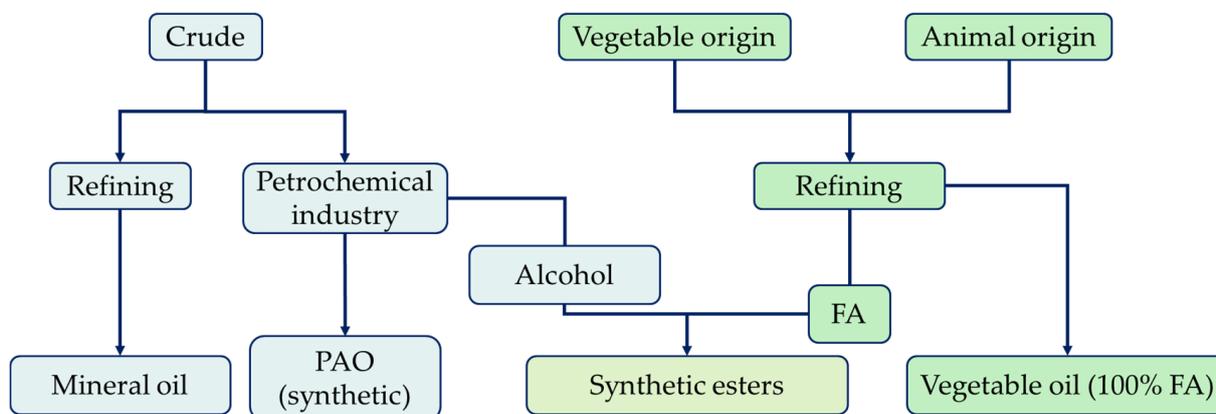


Figure 1. Origin and classification of base fluids.

Environmental concerns about the accelerated development of global warming have led to the promotion of the use of new biodegradable and more environmentally sustainable materials. As part of this movement, new competitive lubricants are being sought from organic materials such as vegetable oils and animal fats to create what are known as biodegradable lubricants or biolubricants. As lubricants, they must fulfill the basic lubricating properties listed above. Thus, biolubricants show important attractive advantages such as high biodegradability, low toxicity, sustainability (eco-friendly), increased worker safety, increased machine life, reduced labor costs, and reduced energy consumption as well as other tribological and physicochemical properties such as increased lubricity, higher viscosity index, higher boiling point, or lower volatility [7,8]. On the contrary, they have certain disadvantages, including poor oxidative, thermal, and hydrolytic stability (leading to shorter shelf life); low corrosion inhibition properties; and poor to bad pour point (PP) [7,9].

According to Verified Market Research [10], the biolubricants market was valued at USD 2.82 billion in 2018 and is expected to reach USD 3.63 billion by 2026, growing at a Compound Annual Growth Rate (CAGR) of 3.2% from 2019 to 2026. Asia and the Americas are also expected to have the highest growth rate in this sector between 2019 and 2024 [11]. These data show the growing industry interest in replacing petroleum-based lubricants with more environmentally friendly ones, and therefore the need to find solutions to concerns about production costs and other more functional aspects, including poor oxidation stability.

This review deals with the state of the art of biolubricants along their most common raw materials and molecular structures, processes of chemical modification of bio-oils, as well as the relationship between their structural features and physicochemical/tribological properties. In addition, due to the close relationship between physicochemical and tribological properties, from the physicochemical characterization of bio-oils, the necessity of using some additives to improve these properties can be considered. The aim of this work is to provide comprehensive information for the selection of bio-oils for the production of biolubricants.

2. Biolubricants

According to the UNE-EN 16807 standard [12], the term “bio” is considered synonymous with good for the environment. Its use in lubricants is linked to its environmental

properties; therefore, it is expected that all compounds called bio-lubricants will degrade in the environment. According to this standard, bio-lubricants and bio-based lubricants must fulfill a minimum requirement:

- Biological carbon (C14) content greater than or equal to 25%.
- Biodegradability of oils greater than or equal to 60% (50% for greases).
- Ecotoxicity: not classified as “dangerous for the environment”.

Proper Classification for Biolubricants from Feedstock

An important characteristic of biodegradable lubricants is the raw material used to produce them. Therefore, these materials could be classified into first, second, third, and fourth generations based on the feedstock, as shown in Figure 2 [13,14]. The first generation would include all lubricants derived from edible crops like sunflower, rapeseed, soybean, palm, palm kernel, coconut, olive, or castor [15]. These oils are characterized by a low oil production yield and encourage the deforestation and destruction of ecosystems for their cultivation [16]. In addition, the use of arable land competes with food sources, which increases the cost of the final product and is counterproductive in the current context of global food shortages. The second generation comes from non-edible materials such as *Jatropha*, tobacco, or cotton seeds [17]. These materials are more widely available than the previous ones, but they do still require arable lands for their growth and compete with edible crops for land as in the first case. Third-generation biolubricants derived from microalgae are emerging to solve this problem. Also, biolubricants derived from macroalgae, bacteria, and fungi can also be included in this category [16,18].

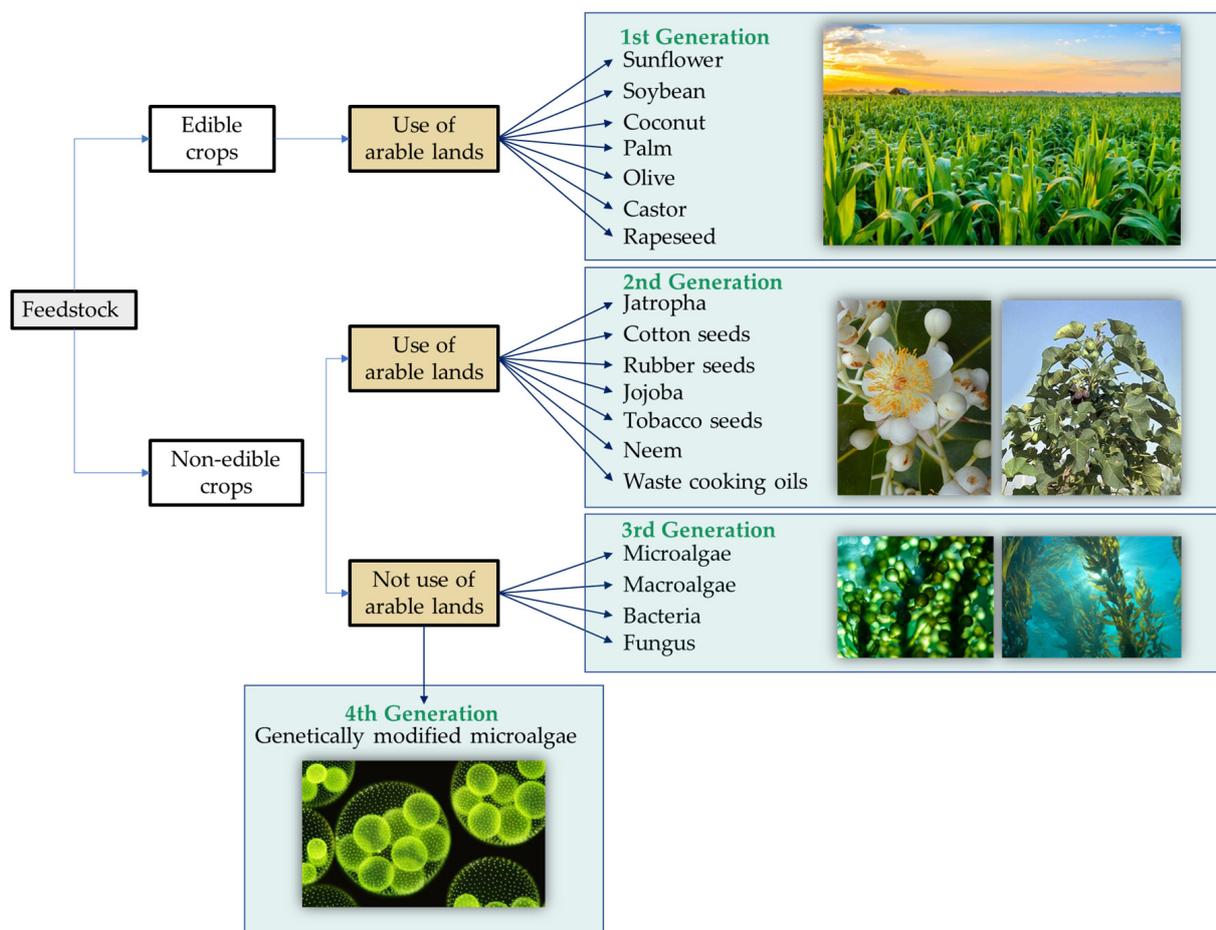


Figure 2. Biolubricant classification according to feedstock.

Microalgae are microorganisms capable of photosynthesis in freshwater, seawater, or wastewater and therefore do not require arable land for their cultivation. Among the requirements for the growth of these microorganisms, the presence of nutrients such as nitrogen, carbon, phosphorus, and potassium makes it possible to use them for wastewater treatment [19,20]. Compared to previous plant sources, they have additional economic and environmental advantages for their application as biolubricants: (a) high growth rate; (b) high biomass production; (c) high lipid content; (d) cultivable all-year-round; (e) higher CO₂ reduction during photosynthesis; and (f) effective removal of phosphates and nitrates in wastewater during cultivation [21,22].

Finally, a fourth generation of biomass derived from genetically modified microalgae is being considered. The possibility of manipulating microalgae through mutagenesis or genetic transformation will open the door to the production of suitable bio-oils for biolubricant formulation without the need to improve them using chemical techniques such as epoxidation or the use of additives [18,23,24].

3. Common Techniques for Biolubricant Production

Obtaining biomass from crops is the first step in the biolubricant production process. Depending on the type of crop, this can involve simple collection and purification processes, for example, using agricultural residues or waste cooking oil [25]. Raw materials such as macro- and microalgae require more elaborate harvesting processes, which can be physical, chemical, or biological (Figure 3). Centrifugation is the most widely used technique in the biomass-derived microalgae industry, usually complemented by drying systems such as lyophilization to efficiently remove moisture [13,22]. Figure 3 shows the bio-oil production chain including harvest, drying, pretreatment (if necessary), and extraction, and identifies the most used techniques at each stage.

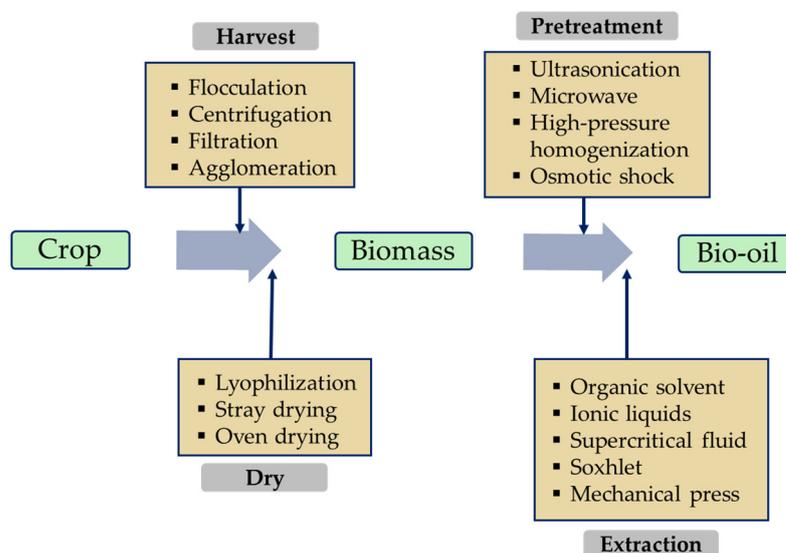


Figure 3. Various techniques to produce bio-oil from plants.

Once the biomass has been obtained, it is subjected to bio-oil production techniques. For materials with simple matrices, such as vegetable oils, more rudimentary methods are used, for example, mechanical or expeller pressing [26,27]. In the case of complex matrices like microalgae, pretreatment for cell disruption is required to facilitate bio-oil extraction. The use of high-pressure homogenization or ultra-high-pressure homogenization as a pretreatment resulted in high lipid extraction yields, reaching an increase of 30% in some cases [28–30]. The disadvantage of these techniques is the high operating cost due to the increased working pressures (supercritical conditions). As a solution, other procedures are being studied, including ultrasound, microwave, or osmotic shock [31–33].

In terms of extraction techniques, organic solvents are the most implemented, which can be alone or supported by complementary pretreatments [27]. If the biomass is semi-solid or solid, solvent extraction would be carried out using a Soxhlet device [34,35]. Ionic liquids with magnetic nanoparticles were also introduced as a potential alternative in 2021 by Egesa and Plucinski [36], where an extraction efficiency of 99% was achieved with a hexane-ionic liquid mixture.

3.1. Bio-Oils (Triglycerides) as Biolubricants

Vegetable oils are mainly composed of triglycerides: three fatty acids (FA) linked by a glycerol. Compared to the long-chain hydrocarbons of mineral oils, which have between 20 and 50 carbon atoms, FAs have shorter chains of between 4 and 26 carbon atoms [37]. They may also contain one or more double bonds and branches. Figure 4 shows the most common FA found in olive oil (mainly oleic acid).

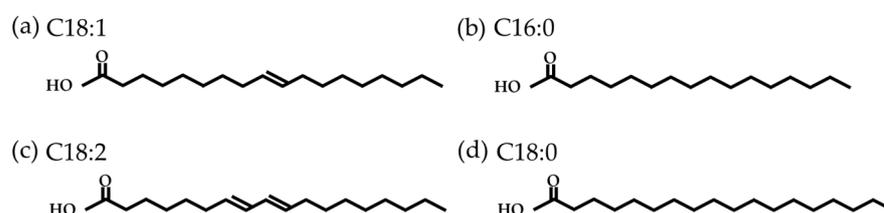


Figure 4. Common fatty acids found in olive oil: (a) oleic acid; (b) palmitic acid; (c) linoleic acid; (d) and stearic acid.

The use of these oils as lubricants has usually been studied as additives to improve a specific property, such as viscosity modifiers. Gallardo-Hernández [38] studied the use of *Jatropha* oil as an additive in a mineral oil (SAE40W oil) to evaluate its lubricating and thermal properties. The improvement in the lubricity in terms of friction and the deterioration in the anti-wear properties were observed, both related to the tribosystem created by the blend. A strong influence on the thermal properties was also observed at a content of less than 20%. Later, Contreras-Gallego [37] studied the variation in density and kinematic viscosity when the above bio-oil was added at 10% and 20% in four different mineral oils (SAE 5W-30, SAE 15W-40, SAE 25W-50, and SAE 40), as well as thermal conductivity and specific heat. An improvement in thermal properties associated with the increase in *Jatropha* oil was demonstrated. There was also a reduction in viscosity at higher additive contents, related to the substitution of long-chain hydrocarbons (mineral base oil) by shorter bio-oil ones. Recently, the feasibility of curcumin-extracted soybean waste cooking oil as a 10%, 20%, and 30% additive in N-150 mineral oil was verified by analyzing the tribological and physicochemical properties. In contrast to the previous case, a reduction in wear volume and coefficient of friction (COF) of up to 16% and 32%, respectively, was observed compared to N-150, due to a stronger tribofilm formed by the additive. The function of curcumin as a natural antioxidant to prevent the oxidation of soybean waste cooking oil was also confirmed. Finally, the increase in viscosity index with the molecular weight was confirmed and the decrease in PP from $-12\text{ }^{\circ}\text{C}$ (N-150) to $-30\text{ }^{\circ}\text{C}$ (10% bio-oil) was attributed to the increase in blend polarity [25].

3.2. Environmentally Friendly Modifications of Vegetable Oils

Despite the advantages offered by biolubricants based on bio-oils, it is inevitable to highlight the need for improvement to provide functional storage properties, in addition to modifying physicochemical and tribological properties depending on the final applications. This led to the search for techniques capable of modifying the chemical structure of triglycerides, producing so-called modified esters, as shown in Figure 5.

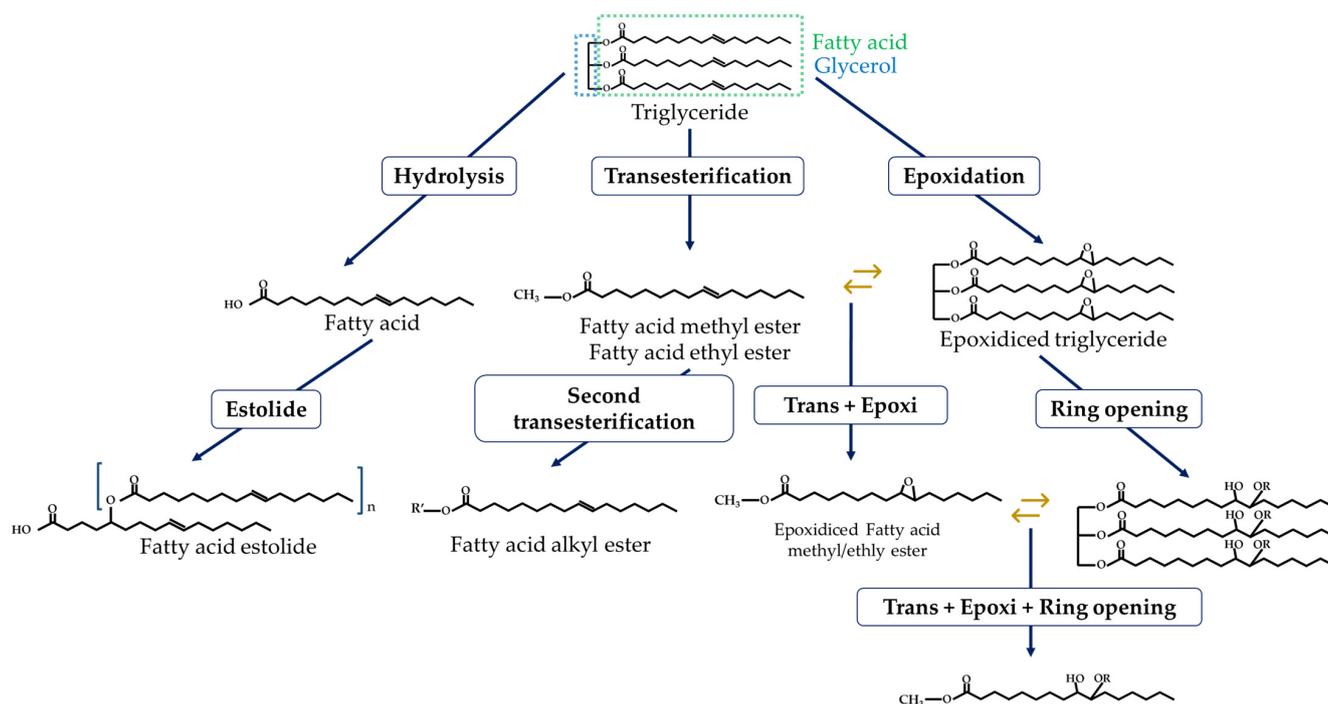


Figure 5. Eco-friendly modifications of oil sources (esters)—palmitoleic acid example.

3.2.1. Hydrolysis

As explained above, bio-oils have poor hydrolytic stability and tend to hydrolyze easily in the presence of water or steam. This leads to the breakdown of their triglycerides and the formation of the corresponding free fatty acids (FFA). This is a spontaneous secondary reaction, promoted by the increase in temperature, which must be prevented [39,40].

3.2.2. Transesterification

Transesterification reactions are the most widely used technique for ester modifications, especially in the biodiesel industry. As shown in Figure 6, it is based on the reaction between a triglyceride and an acyl acceptor (an alcohol) in the presence of a catalyst, and under temperature conditions, to produce fatty acid methyl ester (FAME) as the main product and glycerol as the secondary product. The catalyst function aims to assist the reaction and the deprotonation of the alcohol so that it can join the ester group. These can be acid, basic, or enzymatic catalysts, as well as homogeneous or heterogeneous (depending on the reaction phase) [4,13,41].

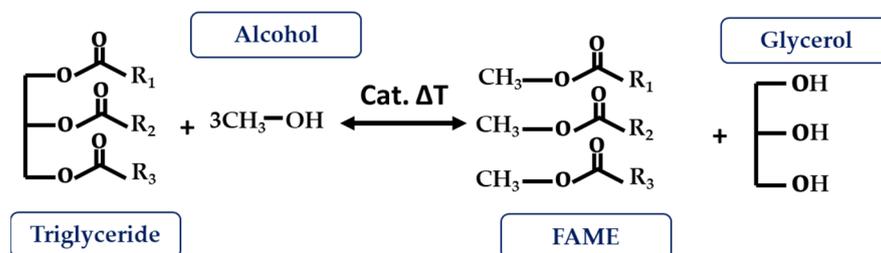


Figure 6. Transesterification reaction.

To avoid secondary reactions such as hydrolysis and thus soap formation at high FFA contents (>2–4 mg KOH/g), it is necessary to neutralize them through an esterification process using an acid catalyst, usually sulfuric acid [42]. It is also necessary to control the humidity of the bio-oil and remove any existing moisture, as this also promotes the saponification reaction.

In the last decade, a new modality called direct or in situ transesterification has attracted attention. It combines lipid extraction (bio-oil extraction) and the transesterification reaction into one process, using biomass as a reagent instead of bio-oil, which means energy and economic savings [43]. The water content is also no longer a critical parameter, as it has greater hydrolytic stability. However, it requires more severe temperature conditions and longer reaction times [13]. A notable application is the processing of microalgal biomass. A wide range of species of these organisms develop strong cell walls, which complicates the extraction process and thus requires an efficient prior cell disruption method. As mentioned above, the operating costs of lyophilization or high-pressure homogenization are high, even more so for complex matrices such as microalgae. The ability to skip the cell disruption and extraction steps is advantageous in terms of both operating cost and energy consumption. Table 1 shows the research that has used this technique for FAME (biodiesel) production in recent years, highlighting the presence of microalgae [44–49].

More recently, Encinar et al. [50–54] have worked on the modification of vegetable-oil-derived FA using double transesterification reactions. In the first step (Figure 7), the triglyceride is degraded to the corresponding FAME. The novelty of this process lies in the second step, in which another transesterification reaction is carried out on these FAMES, this time using a complex alcohol as a reagent, to obtain the so-called fatty acid alkyl [54] esters (FAAEs). According to the researchers, the advantage of this refinery model is the diversity of the main products, starting with the use of bio-oil; the production of biodiesel (FAME) and biolubricants (FAAE); the economic and energy savings with the recovery of materials such as glycerol, as well as the recycling of methanol produced during the second transesterification, which can be reintroduced into the process as a reagent in the first transesterification [55]. So far, only vegetable feedstocks such as safflower, cardoon, rapeseed, soybean, or *Jatropha* oil have been subjected to this technique.

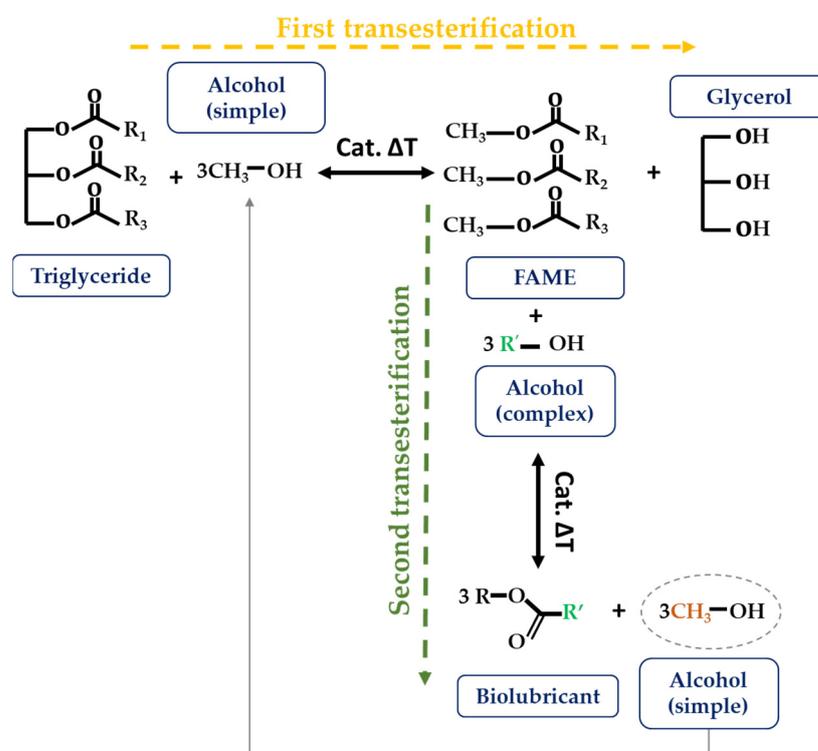


Figure 7. Double transesterification reaction.

3.2.3. Epoxidation/Ring Opening

A significant problem with bio-oils is their poor thermal and oxidative stability due to the effect of double bonds on C-H bond strength. For this reason, the authors recommend raw materials that consist as much as possible of saturated fatty acids (SFAs) because

they do not contain double bonds in their structure [20,56]. One way to neutralize these double bonds is through epoxidation reactions. These are based on the reaction of alkenes with peroxyacids to form a cyclic ether (epoxide) compound [57,58]. Epoxidized compounds have been shown to have superior frictional and anti-corrosive properties and better performance at low temperatures, providing a more economical, sustainable, and environmentally friendly alternative to mineral-based lubricants [58–60].

3.2.4. Estolide Synthesis

Another eco-friendly modification of FFA is the formation of estolides. These are synthetic compounds derived from the linkage of the respective triglycerides or their FFA, which use their hydroxyl groups to form the estolide bonds [59,61]. The advantages of these structures include better oxidative stability, improved PP, and higher flash point (FP). However, this modification does not resolve the hydrolytic stability problems of the compound [62].

Table 1. Environmentally friendly modifications for the production of and improvement in biolubricants. Microalgae species in bold text.

Feedstock	Technique	Molecular Structure	Ref.
High-oleic safflower oil	Double transesterification	FAAE	[52]
Cardoon oil	Double transesterification	FAAE	[50]
Cardoon oil	Double transesterification	FAAE	[53]
Rapeseed oil	Double transesterification	FAAE	[51]
Rapeseed and castor oils	Double transesterification	FAAE	[54]
Indian mustard seed oils	Double transesterification	FAAE	[63]
Rapeseed, seed and frying oils	Double transesterification	FAAE	[55]
Soybean oil	Double transesterification	FAAE	[64]
<i>Jatropha</i> oil	Double transesterification	FAAE	[65]
Coconut oil	Hydrogenation		[66]
Waste cooking oil	Epoxidation + Transesterification	Epoxidized FAME	[60]
Karanja seed oil	Simple transesterification	FAME	[67]
<i>Schlichera oleosa</i> oil	Simple Transesterification	FAME	[68]
Refine bleached palm kernel oil	Simple Transesterification	FAME	[69]
Soybean oil	Epoxidation	Epoxidized triglycerides	[58]
<i>Madhuca indica</i> oil	Epoxidation	Epoxidized triglycerides	[70]
<i>Michelia champaca</i> oil	Epoxidation	Epoxidized triglycerides	[71]
<i>Moringa olifera Lam</i> oil	Epoxidation	Epoxidized triglycerides	[26]
<i>Passiflora edulis</i> oil	Epoxidation	Epoxidized triglycerides	
Crude Palm oil	Hydrolyzation + Esterification	Modified esters	[72]
Crude <i>Jatropha</i> oil	Esterification + Transesterification		
	Esterification + Ultrasound—assisted transesterification	FAME	[73]
Waste ayurvedic oil	Ultrasonic irradiation assisted Transesterification	FAME	[74]
Pequi oil	Hydrolyzation + Esterification	FA	[75]
<i>Dunaliella salina</i>	In situ transesterification	FAME	[44]
<i>Chlorella vulgaris foamate</i>	In situ transesterification	FAME	[45]
<i>Chlorella pyrenoidosa</i>	In situ transesterification	FAME	[46]
<i>Chlorella vulgaris</i>	In situ transesterification	FAME	[47]
Rubber seeds	In situ transesterification	FAME	[48]
<i>Botryococcus braunii</i>	In situ transesterification		
<i>Coccomyxa subellipsoidea</i>	In situ transesterification	FAME	[49]

4. Influence of Structural Features on Biolubricant Performance

Among the structural characteristics that determine the physicochemical and tribological properties of a biolubricant, the following stand out: the presence of double bonds or unsaturation; the length of the chains present; or the molecular weight, polarity, and the presence of branches in the structures that make it up. Cecilia [56] and Chan [76] have previously reviewed the influence of these parameters on their performance as bio-lubricants. Table 2 shows in more detail how the different structures can modify some of the main properties of lubricants.

Table 2. Effect of some structural features on the physicochemical and tribological properties of biolubricants.

Property	Unsaturation (Double Bounds)	Chain Length (Molecular Weight)	Polarity	Branching Degree
Pour Point	↓	↑	↑	↓
Flash Point	-	↑	-	-
Viscosity	↓	↑	-	-
Viscosity Index	-	↑	↓	↑
Oxidation stability	↓	↓	↓	↑
Lubricity	↓	↑	↑	↓
Wear protection	↓	↑	↑	↓
Tribofilm thickness	-	↑	↑	-
Tribofilm adhesion strength	↓	-	↑	↓

4.1. Presence of Double Bonds

The molecular structure of these compounds can contain zero (saturated), one (monounsaturated), or multiple C=C double bonds (polyunsaturated). Each FA introduces the double bonds at a specific position in its structure, for example, at the 6, 9, and 12 carbon atom positions [77]. The presence of double bonds promotes oxygen attack on the carbons adjacent to these bonds, as shown in Figure 8. The areas most affected by this phenomenon are the carbons atoms located between two double bonds, so that the oxidative stability of the structure decreases as the areas of oxygen attack increase [57,78,79]. This oxidative stability refers to the ability of lubricants to retain their properties when exposed to environmental oxygen without suffering oxidative degradation [80]. It has also shown an increase in PP and a tendency to increase viscosity and decrease viscosity index (VI) by reducing the content of unsaturated FAs [57,78,81].

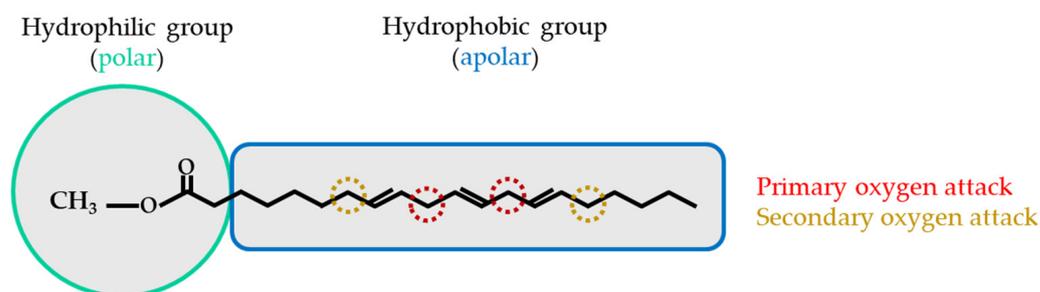


Figure 8. Polarity of the FAME structure and main oxygen attack areas.

4.2. Carbon Chain Length

Chain length and molecular weight are parameters that directly influence important physicochemical properties. Increasing the chain length leads to structures with higher molecular weights, resulting in a higher FP, which is relevant as the FP is an important factor in determining the required safe transport and storage temperatures [79]. In addition, kinematic viscosity increases with the molecular weight and the VI also increases as denser structures help to ensure that the viscosity does not fluctuate significantly with increasing temperature [82].

In terms of tribological properties, it has been found that COF and wear volume decrease with increasing chain length due to the better anti-wear performance of the film formed by the ester groups on the contact surfaces. This is achieved due to the stronger film formed by the biolubricant [25,83].

4.3. Polarity of the Structures

The structures of vegetable oils are linear, unbranched chains with polar end groups, as shown in Figure 8. The polarity of these esters increases the effectiveness of wear reduction by forming an adsorbed protective layer on the contacting metal surfaces, which reduces the surface energy and increases the strength of the formed tribological film due to their high polarity. These tribological films exhibit a low COF at both low and high temperatures [83–85]. This is why the degree of polarity is important when looking at the molecular structure of a lubricant: $\text{COOH} > \text{CHO} > \text{OH} > \text{COOCH}_3 > \text{CO} > \text{COC}$ (decreasing polarity degrees) [40]. Parameters such as VI or PP are also strongly influenced by the high polarity of these structures, resulting in higher VI values as the polarity increases, or in a drop in PP due to polar functionality [25,82].

4.4. Branching Degree

The chains that constitute these oils are characterized by the fact that they are formed by linear chains of 4 to 26 carbon atoms, with a natural lack of branches. For this reason, the shorter the chain length, the more the structure tends to clump together, while the longer the chain length, the more linear they are due to the absence of branches. As a result, these chains have higher PP values than branched structures with the same number of carbon atoms, due to the molecular structure, which tend to compact [79,86].

4.5. Selecting Vegetable Oils for Biolubricant Formulation

Based on the evaluation of the influence of the molecular structures of vegetable oils on their physicochemical and tribological properties, the flow chart shown in Figure 9 has been prepared as a guideline for the selection of vegetable oils for the biolubricant formulation. This scheme follows the one proposed by Farfán-Cabrera et al. [20], dedicated to the selection of microalgae species for the production of biolubricants. Critical input variables are considered to be the FFA content or acidity, the degree of unsaturation, and the chain length of the corresponding FAs, which have more influence on the properties of interest of biolubricants, such as hydrolytic, oxidative stability, lubricity, PP, and viscosity. If the balance between these properties does not meet the minimum requirements for a given application, the diagram shows the chemical conversions typically used as a function of the input variable to be improved. Other alternatives may be found in the use of additives or the selection of less demanding applications.

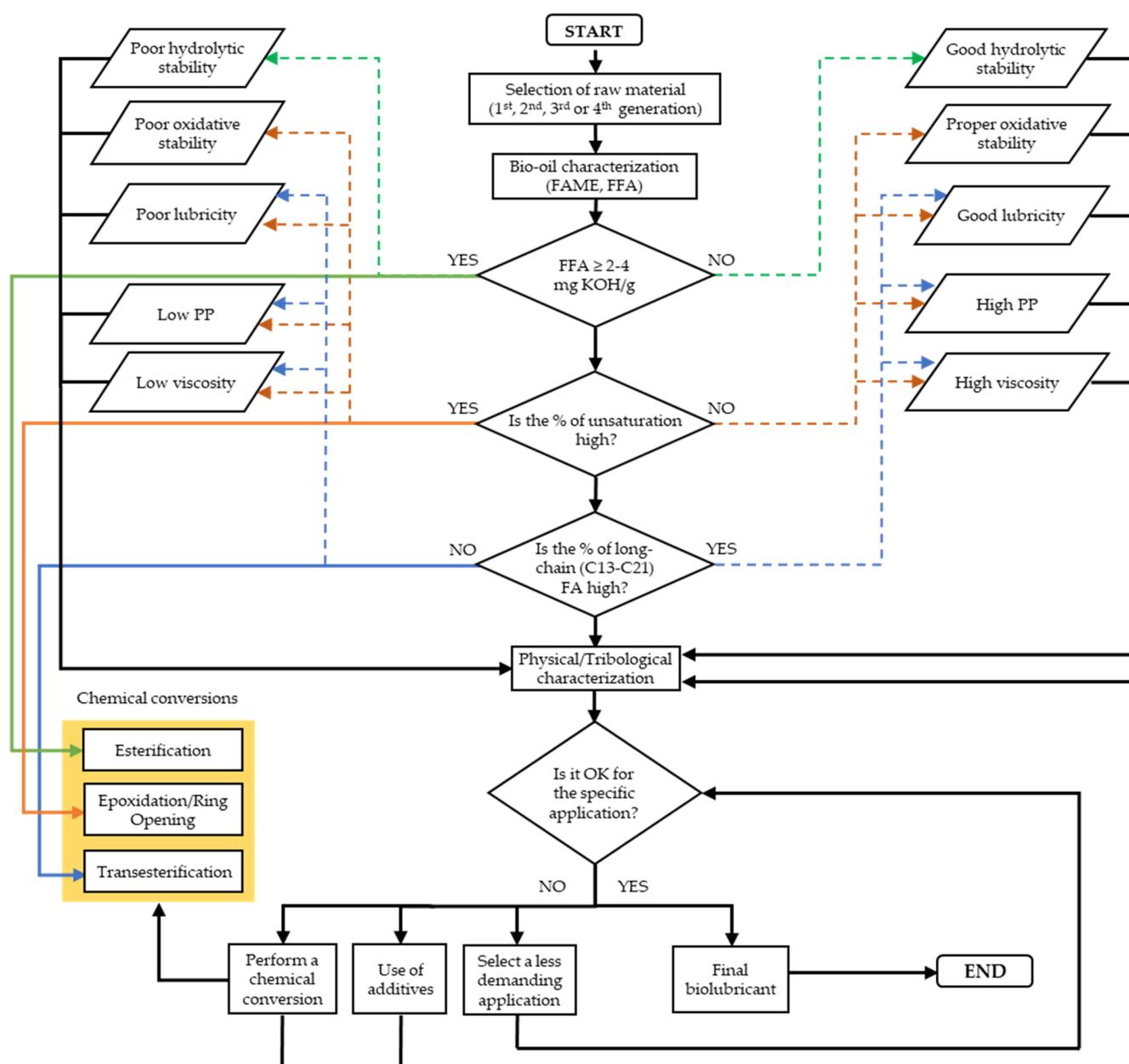


Figure 9. Flow chart for selecting vegetable oils for biolubricant formulation.

5. Tribological Characterization and Performance of Vegetable-Derived Structures

One of the objectives of using vegetable derivatives for the formulation of biolubricants is to reduce the use of elements such as chlorine, sulfur, or phosphorus, as in commercial lubricants formulated from mineral sources, and also to achieve competitive tribological properties. Chan et al. [76] reported the state of the art of tribological tests performed on biolubricants up to 2017. Now, a summary of the publications from 2018 to 2023 in which tribological tests were performed on 1st-, 2nd-, and 3rd-generation biolubricants is shown in Table 3. Table 3 shows the fluids tested, the tribological tests and test conditions used, and the COF and wear results obtained. For studies performed using different additive concentrations and loads, only the results obtained with the highest load and the most representative additive concentrations are included in Table 3.

Table 3. Tribological tests and conditions used for evaluating biolubricants from 2018 to 2022 (a 10^{-3} mm³; b mm³/m; c mm³/Nm; d mm; e 10^{-7} mm³/s; f g).

Test Fluid	Equipment	Test Conditions	COF	Wear	Ref.	
Lithium grease (MVI500/PAO6/DOS)	Ball-on-disc	10–50 Hz, 50 N (1.74 GPa), RT, 30 min	0.16	125×10^{-2} a	[87]	
Lithium grease (MVI500/PAO6/DOS) + 3% cho-ricinoleic			0.12	5.00×10^{-2} a		
Gallate oil ester	Ball-on-disc test	Steel Cooper Steel Cooper	25 Hz, 100 N (2.19 GPa), RT, 30 min	0.095 0.12 0.085 0.069	0.055 ^a 0.045 ^a 0.045 ^a 0.040 ^a	[80]
Phe-3Ci8						
<i>Auxenochlorella protothecoides</i> <i>Chlorella sorokiniana</i> <i>Aurantiochytrium limacinum</i> SR21 <i>Auranti-ochytrium</i> sp. T66 <i>Rhodospiridium toruloides</i> <i>Cryptococcus curvatus</i> PEG 200	Ball-on-disc test	50 Hz, 77 and 150 N (2 and 2.5 GPa), 25 °C, 60 min	0.065–0.07 0.07–0.08 0.1–0.14 0.09–0.13 0.09–0.08 0.08–0.1 0.12–0.13	0.107–0.175 ^a 0.099–0.148 ^a 0.124–0.184 ^a 0.110–0.169 ^a 0.139–0.227 ^a 0.128–0.246 ^a 1.140–7.120 ^a	[88]	
<i>Jatropha</i> oil (JO) Mineral engine oil (SAE 10W-30) Mineral engine oil + 20% JO	Ball-on-disc test	0.25 m/s, 50 N (1.2 GPa), 25 °C	0.06 0.095 0.08	6.00×10^{-4} b 1.50×10^{-4} b 1.50×10^{-4} b	[89]	
Canola oil (CaO) CaO + 0.05% CuO CaO + 0.08% CuO CaO + 0.1% CuO	Ball-on-disc test	800 rpm, 130 N (2.39 GPa), 25 °C, 15 min	0.125 0.08 0.07 0.05	15.5×10^{-6} c 13.0×10^{-6} c 9.10×10^{-6} c 6.00×10^{-6} c	[90]	
Codonosis pilosula wax (grease) Codonosis pilosula wax (base grease) + multilayer graphene	Ball-on-disc test	25 Hz, 100 N (2.25 GPa), 150 °C, 20 min	0.148 0.145	9×10^{-8} c 7×10^{-8} c	[91]	
Castor oil Castor oil + epoxide cellulose pulp (grease)	Ball-on-three-plates	10 rpm, 20 N, 25 °C, 10 min	0.11 0.08	0.47 ^d 0.37 ^d	[92]	
Castor oil (CO) CO + epoxide-functionalized alkali lignin dispersion grease (EAL-1) CO + epoxide-functionalized alkali lignin dispersion grease (EAL-2) CO + epoxide-functionalized alkali lignin dispersion grease (EAL-3) CO + epoxide-functionalized alkali lignin dispersion grease (EAL-4) CO + epoxide-functionalized alkali lignin dispersion grease (EAL-5)	Ball-on-three-plates	20 rpm, 20 N, RT, 30 min	0.084 0.09 0.07 0.09 0.08 0.22	0.508 ^d 0.524 ^d 0.416 ^d 0.366 ^d 0.450 ^d 0.440 ^d	[93]	
N-150 mineral oil Curcumin-extracted soybean waste cooking oil	Four-ball test	1200 rpm, 392 N, 75 °C, 60 min	0.117 0.08	0.685 ^d 0.573 ^d	[25]	
SAE40W + 5% JO SAE40W + 20% JO SAE40W + 50% JO	Four-ball test	395, 20 min	0.101 0.113 0.125	0.950 ^d 1.350 ^d 1.500 ^d	[38]	
SAE 15W40 Palm oil (PO) PO + 0.1% hBN PO + 0.5% hBN	Four-ball test	1200 rpm, 392 N, 75 °C, 60 min	0.115 0.08 0.079 0.11	2.000 ^e 1.850 ^e 1.180 ^e 1.100 ^e	[94]	

Table 3. Cont.

Test Fluid	Equipment	Test Conditions	COF	Wear	Ref.
Soybean oil FAAE Soybean oil FAAE + 5% ZnAl	Four-ball test	1200 rpm, 392 N, 60 min	0.11 0.055	n/a n/a	[64]
Castor oil + seed oil Castor oil + seed oil + 0.25% halloysite clay nanotube	Four-ball test	1200 rpm, 392 N, 75 °C, 60 min	0.0697	0.919 ^d	[86]
Castor oil + seed oil + 0.5% halloysite clay nanotube			0.0551	0.848 ^d	
Castor oil + seed oil + 0.75% halloysite clay nanotube			0.0527	0.845 ^d	
Castor oil + seed oil + 1% halloysite clay nanotube			0.0525	0.779 ^d	
Pequi oil Mineral oil	Four-ball test	4.95 × 10 ⁵ μm/s, 55 N, 75 °C, 60 min	0.0588 0.0849	0.371 ^d 0.195 ^d	[75]
Cucurbita pepo L. oil SAE 20W40	Four-ball test	1200 rpm, 392 N, 75 °C, 60 min	0.0506 0.0459	0.333 ^d 0.413 ^d	[95]
Refine bleached palm kernel FAME Engine oil	Four-ball test	1200 rpm, 60, 80, and 100 kg, 75 °C, 60 min	0.07 0.10	2.250 ^d 2.000 ^d	[69]
Sunflower oil Soybean oil	Four-ball test	1200 rpm, 392 N, 75 °C, 60 min	0.06 0.055	0.600 ^d 0.700 ^d	[96]
Karanja FAME	Four-ball test	1200 rpm, 15 and 40 N, 75 °C, 60 min	0.05–0.14	0.300–0.440 ^d	[97]
Rice bran and sunflower oil (RB + SFO) RB + SFO + 0.01% CuO RB + SFO + 0.04% CuO	Four-ball test	1200 rpm, 392 N, 75 °C, 60 min	0.332 0.3298 0.314	0.911 ^d 0.865 ^d 0.830 ^d	[98]
Coconut oil (CO) Mustard oil (MU) SAE20W40 CO + 10% MU CO + 50% MU	Four-ball test	600 rpm, 392 N (1 GPa), 75 °C, 60 min	0.09 0.12 0.103 0.092 0.099	0.587 ^d 0.478 ^d 0.496 ^d 0.585 ^d 0.489 ^d	[99]
Jojoba oil (JJO) Polymerized JJO 1 h (grease) Polymerized JJO 2 h (grease) Polymerized JJO 3 h (grease)	Four-ball test	800 rpm, 492 N, RT, 30 min	0.04 0.015 0.03 0.068	0.422 ^d 0.385 ^d 0.471 ^d 0.420 ^d	[100]
Lithium-based paraffin grease Lithium-based castor oil grease Lithium-based coconut oil grease	Four-ball test	1200 rpm, 392 N, 75 °C, 60 min	0.091 0.07 0.082	0.850 ^d 0.700 ^d 0.930 ^d	[101]
Pailm oil FAME DF-CN48 DF-CN48 + 50% FAME DF-CN51 DF-CN51 + 50% FAME	High-frequency reciprocating test	50 Hz, 200 g, 60 °C, 75 min	0.126 0.15 0.135 0.29 0.13	0.220 ^d 0.290 ^d 0.250 ^d 0.460 ^d 0.300 ^d	[102]
Styrax officinalis oil Styrax officinalis FAME	Pin-on-disc test	350 rpm, 35, 70, 105, and 140 N (1.14, 1.43, 1.64, and 1.81 GPa), 125 °C	0.0094 0.0076	0.880 ^d 0.840 ^d	[103]

Table 3. Cont.

Test Fluid	Equipment	Test Conditions	COF	Wear	Ref.					
Neem oil (NO) NO + 0.15 SiO ₂ NO + 0.9 SiO ₂	Pin-on-disc test	100 rpm, 40, 60, 80, and 100 N	0.075	59 ^d	[104]					
		(1.03, 1.17, 1.29, and 1.39 GPa), 150 °C	0.073	57 ^d						
			0.94	66 ^d						
Karanja oil Karanja oil + 1% TiO ₂ Rice bran oil Rice bran oil + 1% TiO ₂	Pin-on-disc test	20, 40, 60, and 80 N (1.28, 1.61, 1.84, and 2.03 GPa)	0.086 0.061 0.056 0.043	4.20 × 10 ^{-6 c} 2.89 × 10 ^{-6 c} 4.70 × 10 ^{-6 c} 3.40 × 10 ^{-6 c}	[105]					
Jatropha oil Jatropha oil + 0.2% SiO ₂ Jatropha oil + 1% SiO ₂		Pin-on-disc test	120 rpm, 50, 80, 120 N (1.28, 1.5, and 1.72 GPa)	0.085 0.08 0.045		0.890 ^f 0.850 ^f 0.760 ^f	[106]			
Waste ayurvedic oil			Pin-on-disc test	300 rpm, 80 N (2.36 GPa), RT, 60 min		0.040		0.800 ^d	[74]	
Schlichera oleosa oil Schlichera oleosa FAME			Pin-on-disc test	200 rpm, 40, 60, 80, and 100 N (1.03, 1.17, 1.29, and 1.39 GPa), 125 °C		0.0089 0.0065		0.780 ^d 0.720 ^d	[68]	
PO PO + 0.5% CuO PO + 0.5% TiO ₂ Brassica oil (BO) BO + 0.5% CuO BO + 0.5%v TiO ₂	Pin-on-disc test	1000 rpm, 40 N, RT, 20 min		0.045 0.034 0.039 0.047 0.040 0.043	3.60 × 10 ^{-8 c} 1.25 × 10 ^{-8 c} 2.00 × 10 ^{-8 c} 4.00 × 10 ^{-8 c} 1.60 × 10 ^{-8 c} 2.50 × 10 ^{-8 c}	[107]				
Michelia champaca oil (MCO) Epoxidized MCO E-MCO + 1.2% CeO ₂			Pin-on-disc test	100 rpm, 117 N (1.3 GPa), 75 °C, 60 min	0.084 0.065 0.055		0.1 ^f 0.85 ^f 0.7 ^f	[71]		
Vegetable oil (VO) VO + 0.6% GNPs VO + 0.6% hBN VO + 0.6% (GNPs + hBN)				Pin-on-disc test	550 rpm, 18 N (0.98 GPa), RT, 60 min		0.45 0.43 0.42 0.41		8.100 ^e 5.000 ^e 4.500 ^e 3.900 ^e	[108]
Putranjiva oil (PTO) PTO + 1.3 CuO					Pin-on-disc test		500 rpm, 150 N (1.85 GPa), 125 °C		0.66 0.88	
Crambe abyssinica oil Crambe abyssinica FAME			Pin-on-disc test				150 rpm, 30, 60, 90, and 120 N (0.93, 1.17, 1.34, and 1.48 GPa), 125 °C	0.009 0.007	0.780 ^d 0.600 ^d	
Coconut oil Olive oil Rapeseed oil Soybean oil Sunflower seed oil Linseed oil					Reciprocating cylinder-on-disc tests		50 mm/s, 5 N, RT, 10 min	0.079 0.071 0.077 0.083 0.075 0.088	n/a	

The tribological performance of vegetable oils depends on their molecular structure, as discussed in Table 2. Having shorter chains than hydrocarbons do would tend to improve lubricity and wear protection, but the presence of double bonds in the structure of vegetable oils has the opposite effect [75,89,95]. Therefore, the higher the degree of unsaturation of these oils, the lower the improvement in friction and wear. It has also been shown that the effect of increased unsaturation on tribological properties is more pronounced at higher

temperatures [112]. Yoshida et al. [111] also investigated the effect of double bonds on the COF of six vegetable oils with different levels of unsaturation in relation to the presence of mono-, di-, and tri-unsaturated FA. The oil with the highest content (%) of triunsaturation showed the highest COF results (linseed oil), followed by the oils with the highest content (%) of monounsaturation (rapeseed and olive oils).

Regarding the modified oils, Singh et al. [68,103,110] carried out comparative tests of three vegetable oils with their corresponding FAME by means of a pin-on-disc test, verifying in all of them the improvement in the anti-friction and anti-wear properties of the FAME compared to the unmodified oil. This could be due to the polarity of these compounds, thanks to the polar COOH groups present in FAME, which are adsorbed on metal surfaces, forming a more resistant film.

Another widely used alternative for improving pure oils is the use of nanoparticles as additives, particularly CuO, TiO₂, and SiO₂. For example, the use of CuO was tested in canola oil and a significant reduction in wear was achieved from 15.5×10^{-6} to 6×10^{-6} mm³/Nm with 1% of the additive [90]. According to Table 3, at the same maximum contact pressure, pure *Jatropha* oil has lower COF values (0.027) compared to other pure oils such as neem (0.055), karanja (0.08), or rice bran (0.05) [104–106]. Regarding nanoparticle additives, the study using karanja and rice bran combined with TiO₂ showed a higher COF reduction of about 28% at a nanoparticle concentration of 0.5% [105]. In the case of neem, SiO₂ nanoparticles were selected and showed a reduction in friction of 21% at a concentration of 0.33% [93]. In the case of *Jatropha* oil, it was necessary to increase the concentration of SiO₂ nanoparticles to 1% to achieve a friction reduction of 24% [106]. On the other hand, in the case of neem and *Jatropha* oils with SiO₂, the wear was relatively constant [104,106]. On the contrary, a significant reduction in wear was observed when TiO₂ was used as an additive in karanja and rice bran oils [105].

With regard to greases, there is also an increase in the study of obtaining bioalternatives, especially using a vegetable oil as a base oil mixed with an ecological thickener such as natural wax or modified cellulose derived from a vegetable source [92,113–116]. The Chemical Process and Product Technology Research Center (Pro2TecS) of Huelva (Spain) has studied the formulation of bio-greases from castor oil using thickeners such as epoxide-functionalized alkali lignin (EAL) or epoxidized cellulose pulp from *Eucalyptus globulus* as potential substitutes for traditional lithium and calcium greases [92,93]. EALs showed a significant reduction in wear for most of the grease formulations prepared by the authors, while a sharp increase in COF values was found for a high epoxy index (0.79 mol/kg) and a higher thickener content (10%) [93]. In the second case, the COF variation was more significant, with a reduction from 0.11 (castor oil) to 0.08 (grease) and an improvement in wear reduction of 21%, and with the cellulose pulps modified with aromatic epoxides being more efficient in protecting the metal surface [92]. Xie et al. [91] studied the improvement in the tribological properties of a vegetable wax (*Codonopsis pilosula*)-based grease by using a multilayer graphene as an additive, proving that this material is a good alternative for improving anti-wear properties. Another study by Abbas et al. [100] also compared the tribological performance of polymerized jojoba oil (grease) with the pure oil and found that the polymerized jojoba grade also improved the tribological properties after 1 h of polymerization reaction.

Figure 10 shows the distribution of the different raw materials used in previous papers on the tribological characterization of biolubricants between 2015 and 2023, indicating the raw material generations (1st, 2nd, 3rd, and 4th), the molecular structures (triglyceride, FA, FAME, FAAE), as well as the most used combinations in the formulation of biolubricants. According to Figure 10a, the use of 1st-generation biolubricants derived from vegetable sources (edible plants) dominates with 72% of the references, followed by the 2nd generation with 24% and, to a lesser extent, the 3rd generation with 4%. On the other hand, there are no articles with biolubricants derived from genetically modified (GM) microalgae (4th generation). However, articles with GM oils of the 1st generation have been reported, as in the case of high oleic soybean [23,117,118]. The large number of tribological

studies carried out on biolubricants formulated from pure oils (triglycerides) is shown in Figure 10b, as well as the use of esters modified using the transesterification technique, such as FAME (biodiesel) or FAAE, which have been shown to have better physicochemical and tribological properties [64,68,69,97,102,103]. Finally, Figure 10c highlights the need to use additives when the biolubricant is formulated from pure oil, with the most studied being CuO and TiO₂ nanoparticles. However, it can be observed how those formulated from FAME (better anti-friction and anti-wear properties) are desired as pure base oils or in blends with mineral base oils.

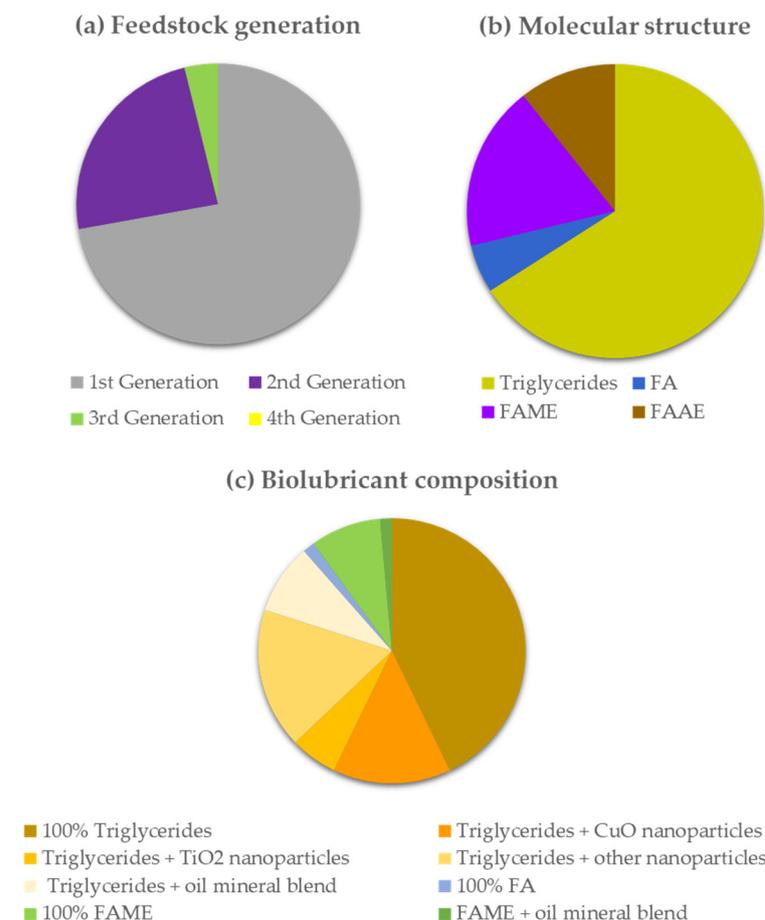


Figure 10. Summary of studies on biolubricant tribology from 2015 up to now.

6. Conclusions

The use of natural feedstocks instead of those derived from petroleum for lubricant production constitutes an alternative in the fight against climate change, but the consumption of edible crops or crops that require arable land is a concern that needs to be overcome. The use of third-generation feedstocks is emerging as an attractive solution. In particular, microalgae have the advantage of being cultured in a wide variety of systems, including wastewater, which offers the possibility of exploiting the growth cycle of these microorganisms in wastewater bioremediation.

Considering the above facts, some weaknesses of bio-oils such as poor oxidation stability due to the presence of double bonds or poor flow properties at low temperatures should be faced. In order to overcome these problems, it is necessary to select the raw material correctly, and then check its lipid profile and verify that its molecular structure is suitable for the properties that may be required by the future biolubricant. Many additives based on nanoparticles have also been tested to improve the tribological performance of biolubricants, with remarkable results at concentrations below 1%.

The double transesterification technique developed for the production of FAAE biolubricants appears to be a promising route for the synthesis of these materials, with the ability to manipulate the degree of branching and chain length almost indefinitely. However, this technique has only been applied to 1st- and 2nd-generation feedstocks with the associated limitations. It would be interesting to apply this technique to more attractive feedstocks such as 3rd-generation feedstocks. In addition, the need to remove the double bonds can be achieved by using the epoxidation technique. Finally, the combined use of 3rd-generation bio-oils, with their molecular structure modified through transesterification and epoxidation techniques, and nano-additives can be a good solution for formulating biolubricants for multiple applications.

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