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Biolubricants from Waste Cooking Oil and Natural Antioxidants: A Review of Extraction Technologies, Conversion Techniques, and Performance Enhancement

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Abstract

Effective management of agricultural and industrial by-products is essential for promoting circular economic practices and enhancing environmental sustainability. Agri-food wastes and waste cooking oil (WCO) represent two abundant residual streams with significant potential for sustainable biolubricant production. Valorizing biomass and WCO aligns with Sustainable Development Goal (SDG) 7, as it improves energy efficiency through enhanced lubricant performance and reduced energy loss. Furthermore, this sustainable approach contributes to SDG 12 and SDG 13 by minimizing waste production and accumulation, thereby mitigating negative environmental impacts and climate change. This critical review addresses existing gaps in the production of biolubricants from WCO and the incorporation of natural antioxidants as versatile enhancers or additives. It examines and compares various techniques for the extraction, chemical and physical modification, and characterization of WCO-derived biolubricants. Specific methods, including esterification, transesterification, and antioxidant incorporation, are evaluated for their effectiveness in converting WCO into biolubricants. The review also discusses the influence of residual bioactive compounds on oxidative stability and lubricating properties. While vegetable oils demonstrate superior friction-reducing capabilities compared to petroleum-based lubricants, their triglyceride structure often results in poor oxidative stability, limiting their practical applications. Modification strategies and antioxidant inclusion are proposed to enhance this stability. A comprehensive analysis of the physicochemical properties and tribological performance of biolubricants, both pre- and post-processing, is presented. This systematic evaluation of extraction and upgrading methodologies aims to facilitate the development and industrial adoption of sustainable biolubricants.

Keywords: Biolubricant; antioxidant; tribology; extraction methods; chemical modification, waste cooking oil; sustainability

Abbreviation

ABTS	2,2’-azino-bis-3-ethylbenzothiazoline-6-sulphonic acid
BDE	Bond dissociation energy
BHA	Butylated hydroxyanisole
BHT	Butylated hydroxytoluene
COF	Coefficient of friction
COM	Cost of manufacturing
CUPRAC	Cupric ions (Cu^{2+}) reducing antioxidant power assay
DD H_2O	Double distilled water
DESE	Deep eutectic solvent extraction
DMDP	N,N-Dimethyl-p-phenylenediamine
DPPH	2,2-Diphenyl-1-picrylhydrazyl
DW	Dry weight
EAE	Enzyme assisted extraction
EDGE	Ethylene glycol di-ester
EFB	Empty fruit bunch
EG	Ethylene Glycol
FAME	Fatty acid methyl ester
FRAP	Ferric ion reducing antioxidant power assay
GAE	Gallic acid equivalents
HAT	Hydrogen atom transfer
HBA	Hydrogen bond acceptors

HBD	Hydrogen bond donors
MAE	Microwave assisted extraction
NPV	Net present value
OIT	Oxidation induction time
OOT	Oxidative onset temperature
ORAC	Oxygen radical absorbance capacity
PBT	Pay back time
PEFE	Pulsed electric field extraction
PKS	Palm kernel shell
PLE	Pressurized liquid extraction
RBD	Refined-bleached-deodorized
ROI	Return of investment
RPF	Relative protection factor
RPVOT	Rotating pressure vessel oxidation test
SET	Single electron transfer
SFE	Supercritical fluid extraction
TAC	Total anthocyanin content
TAG	Triacylglycerol
TBHQ	Tert-butylhydroquinone
TCC	Total carotenoid content
TE	Total ellagitannin
TFC	Total flavonoid content
TMP	Trimethylolpropane
TOSCA	Total radical scavenging capacity assay
TOT	Thermal onset temperature
TPC	Total phenolic content
TRAP	Total radical trapping antioxidant parameter
UAE	Ultrasonic-assisted extraction
UEAE	Ultrasonic enzyme assisted extraction
WCO	Waste cooking oil
WCOME	Waste cooking oil methyl ester
WSD	Wear scar diameter
ZDDP	Zinc dialkyl dithiophosphate

1. Introduction

The mineral oil market was valued at approximately USD 4.11 billion in 2022 and is projected to grow at a compound annual growth rate (CAGR) of 3.71%, reaching an estimated USD 5.70 billion by 2031 [1]. This growth is largely driven by the increasing demand for lubricants, which is expected to rise in response to the alarming growth of the global population. As the population expands, the demand for natural resources and energy consumption will inevitably increase. The U.S. Energy Information Administration (2023) reported global total primary energy consumption of 637.8 quadrillion British thermal units (Btu) in 2022 [2]. Projections indicate that this figure could rise to approximately 854.7 quadrillion Btu by 2050, reflecting a 34% increase from 2022 levels.

This surge in energy consumption is anticipated to result from increased machinery use and transportation needs associated with population growth and human activity. Notably, around 23% of total energy consumption is lost due to friction and wear in the moving parts of engines [3]. Lubricants play a critical role in reducing this loss by minimizing friction and enhancing the performance of mechanical systems. Advanced lubricants, for instance, can reduce energy consumption by up to 40% [4]. Industrial lubricants typically comprise base oils (70–99% of the formulation) and various additives [3]. The conventional lubricants used are mineral oil, which can be further categorized into 3 classifications which are paraffinic, naphthenic, and aromatic [5]. Paraffinic mineral oils have long alkyl side chains, which results in high viscosity index (VI) and high pour point. On the other hand, naphthenic mineral oils have comparatively shorter side chains and, consequently, a low to medium VI. Low viscosity mineral oils are used as spindle oil for textile machinery and high viscosity mineral oils are used as cylinder oils in steam engines. Different oil grades for different applications (such as automobile, gear, turbine, hydraulics, and aviation) can be produced through mineral oil blending [6]. The properties of these mineral oils are enhanced with additives for commercial use so that better lubricity performance can be achieved depending on different applications. In general, mineral-oil-based lubricants are used together with conventional additives such as tert-butylhydroquinone (TBHQ), butylated hydroxytoluene (BHT), and butylated hydroxyanisole (BHA) [7]. These synthetic additives are highly toxic, carcinogenic, and non-biodegradable materials. Therefore, the usage of conventional mineral oil-based lubricants is highly detrimental to the environment, given the difficulties in proper lubricant disposal management. It is reported that approximately 1 million litres of drinking water are polluted by 1 kg of mineral oil-based lubricant due to improper disposal [3].

As an alternative to the mineral oil-based lubricant, biolubricants offer comparable or sometimes better lubricating performance with less environmental burden. Biolubricants are derived from vegetable oils, waste cooking oil, or animal fats, making them biodegradable and non-toxic compared to petroleum-based lubricants. These biolubricants possess desirable qualities that are favorable in applications such as metal cutting, heavy machinery, aircraft, and hydraulics due to their superior lubricity, flash point, and VI [8, 9]. The global biolubricants market, valued at USD 2.13 billion in 2021, is expected to grow at a CAGR of 4.1%, driven by rising environmental awareness and diminishing crude oil reserves [10]. This shift is expected to positively impact environmental concerns. A systematic study of biolubricant lifecycle, particularly focusing on motor oil for automobiles, is underway to understand their environmental effects during processing, usage, and disposal. The growth of the automotive sector and the increased availability of high-performance, cost-competitive green base oils are anticipated to propel the bio-lubricants market. Increased interest in environmental issues is expected to drive demand for bio-based lubricants. The United States and Europe are leading this trend, promoting the use of bio-based materials due to their lower environmental impact. Rising oil prices and environmental regulations on conventional lubricants are also fueling market expansion. Legislation favoring bio-lubricants, along with increased government spending on research and development and product innovation, is expected to boost the industry in the coming years. The market growth is further supported by tax incentives, unpredictable crude oil prices, and socio-economic factors such as urbanization, population growth, rising disposable incomes, and improved living standards.

Figure 1 below lists the advantages, disadvantages, modification methods and end uses of biolubricants.

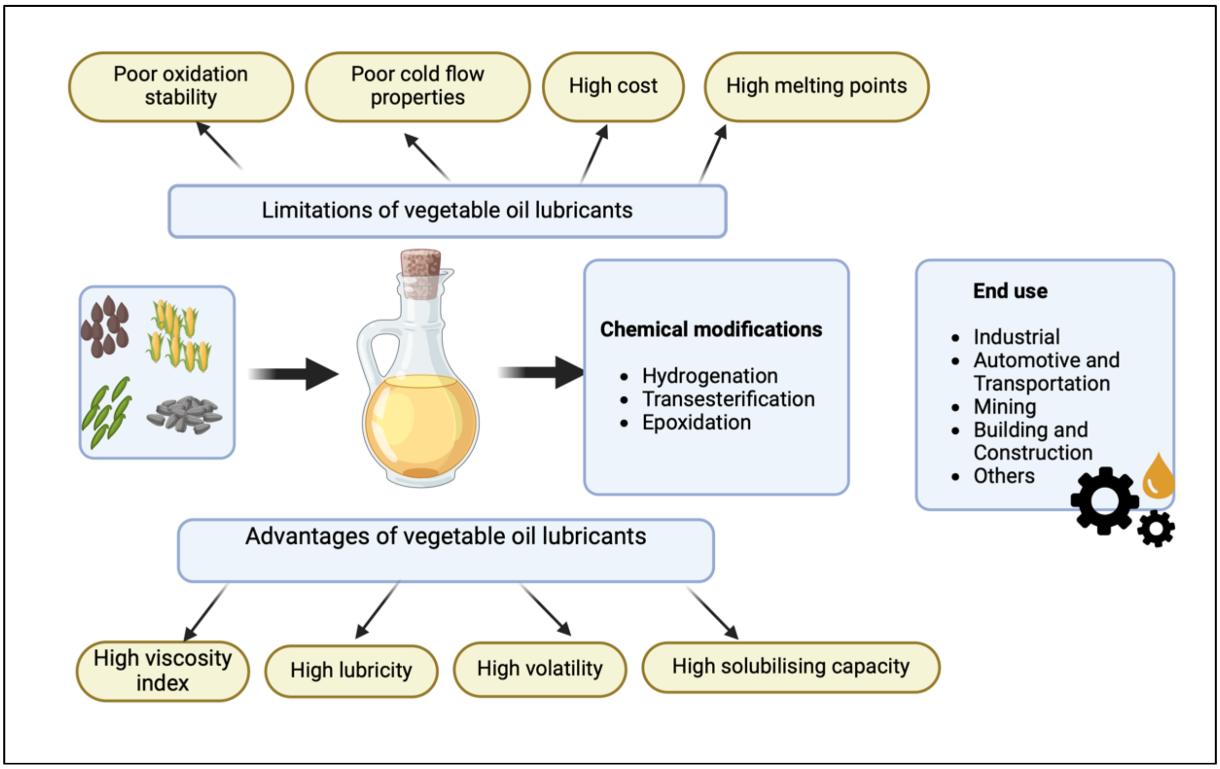


Figure 1. Advantages, limitations and applications of biolubricants

Most recent overviews in 2024 have primarily focused on the synthesis, properties, and applications of WCO biodiesel [11]. However, critical parameters for biolubricants, although similar to those for biodiesel, are often underreported. For instance, Awogbemi and Kallon [12] reviewed WCO valorization for non-fuel applications such as biolubricants, bioasphalt, bioplasticizers, and 3D printing resins, but their discussion on improving the oxidative and tribological performance of WCO biolubricants remains limited. While reviews of lubricant additives often emphasize nano-enhancers like metal- and carbon-based nanomaterials [13, 14], they tend to overlook natural antioxidants. Metal nanoparticles are typically analyzed for their impact on lubricity and lubricating mechanisms, yet the role of antioxidants in enhancing oxidative stability and their potential effects on biolubricant lubricity receive scant attention.

Despite their common use as antioxidative additives, the potential of natural antioxidants to improve the tribological performance of biolubricants remains underexplored. This review addresses these gaps by examining antioxidant extraction methods and classifications, summarizing key physicochemical properties of vegetable- and WCO-based biolubricants, and evaluating how antioxidants enhance oxidative and tribological performance.

2. Background of Oxidation and Antioxidants

2.1. Oxidation of Biolubricants

Oxidation is an undesirable process in carbon-based lubricants (C20–C70) that causes degradation and reduces machinery performance. Vegetable oil-based lubricants or bio-lubricants before any chemical modifications consist of mainly triacylglycerol (TAG), which is around 98%. These TAGs are prone to oxidation degradation as they consist of different polar bonds, which are active sites for oxidation initiated by oxygen, humidity, temperature, and light [15]. Oxidation of vegetable oils happens more readily due to their higher degree of unsaturation. There are three oxidation stages: initiation, propagation, and termination. An auto-oxidation reaction is a process in which the antioxidant capacity of the lubricant is effectively consumed in the oxidative chain reaction. Auto-oxidation initiation usually occurs at the allylic carbon (the carbon atom close to the double bond), where the dissociation energy of the C–H is at its weakest. The hydrogen at the C–H bond would directly react with the reactive oxygen species such as the hydroxyl radical ($OH^{\bullet}$), hydrogen peroxide (H_2O_2), or the superoxide anion radical ($O_2^{\bullet-}$). As the C–H bond readily dissociates to bond with the reactive oxygen species, the TAG is left with a vacant orbital ($R^{\bullet}$), whereby it will undergo resonance molecular rearrangement and the bonding with triplet oxygen ($^3O_2$) to form new radicals ($R–O–O^{\bullet}$). The alkyl peroxyl radical will then react with the hydrogen atom from other TAGs or FA to form hydroperoxides (R–O–OH) and a new radical ($R^{\bullet}$), hence the propagation stage. Lastly, the chemical reaction will proceed until there are no more radicals in the oil or when the FA is diminished. The reaction will be terminated once these conditions are met. The auto-oxidation process is shown through Equation **Error! Reference source not found.** to (6) [15-17]. The role of antioxidants in the oxidation cycle is represented in

Figure 2 below.

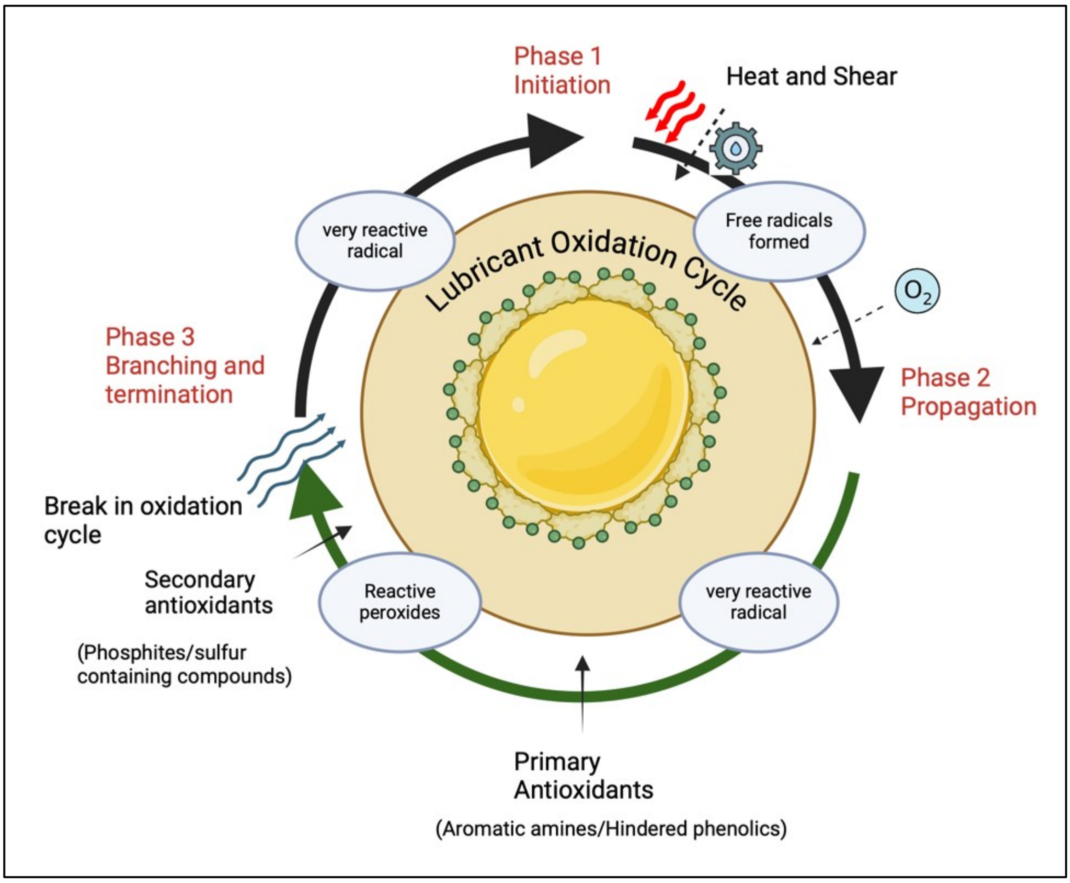


Figure 2. The role of antioxidants on the lubricant oxidation cycle.

On the other hand, the photo-energized oxygen (singlet oxygen – $^1O_2$) would directly react with the C=C double bond to form hydroperoxide without the formation of radicals [18]. The reaction is known as the Alder-ene reaction. The hydroperoxides formed from auto-oxidation and photo-oxidation are detrimental to the lubricating performance as they can decompose into smaller molecules (such as aldehydes, alcohol, short-chain hydrocarbons, and epoxides), acidic/corrosive products, and sludge sediment [15]. The formation of higher molecular weight by-products, especially polymers, during the oxidation stage, can also be attributed to the loss of tribological performance due to the increase in oil viscosity, which can lead to increased friction and reduced efficiency. The oxidation process is represented schematically in

Figure 3 below.

Initiation Stage

$$RH + OH^{\bullet} \rightarrow H_2O + R^{\bullet} \tag{1}$$

Propagation Stage

$$R^{\bullet} + O_2 \rightarrow ROO^{\bullet} \tag{2}$$

$$ROO^{\bullet} + RH \rightarrow ROOH + R^{\bullet} \tag{3}$$

Termination Stage

$$R^{\bullet} + R^{\bullet} \rightarrow RR \tag{4}$$

$$R^{\bullet} + ROO^{\bullet} \rightarrow ROOR \tag{5}$$

$$ROO^{\bullet} + ROO^{\bullet} \rightarrow ROOR + O_2 \tag{6}$$

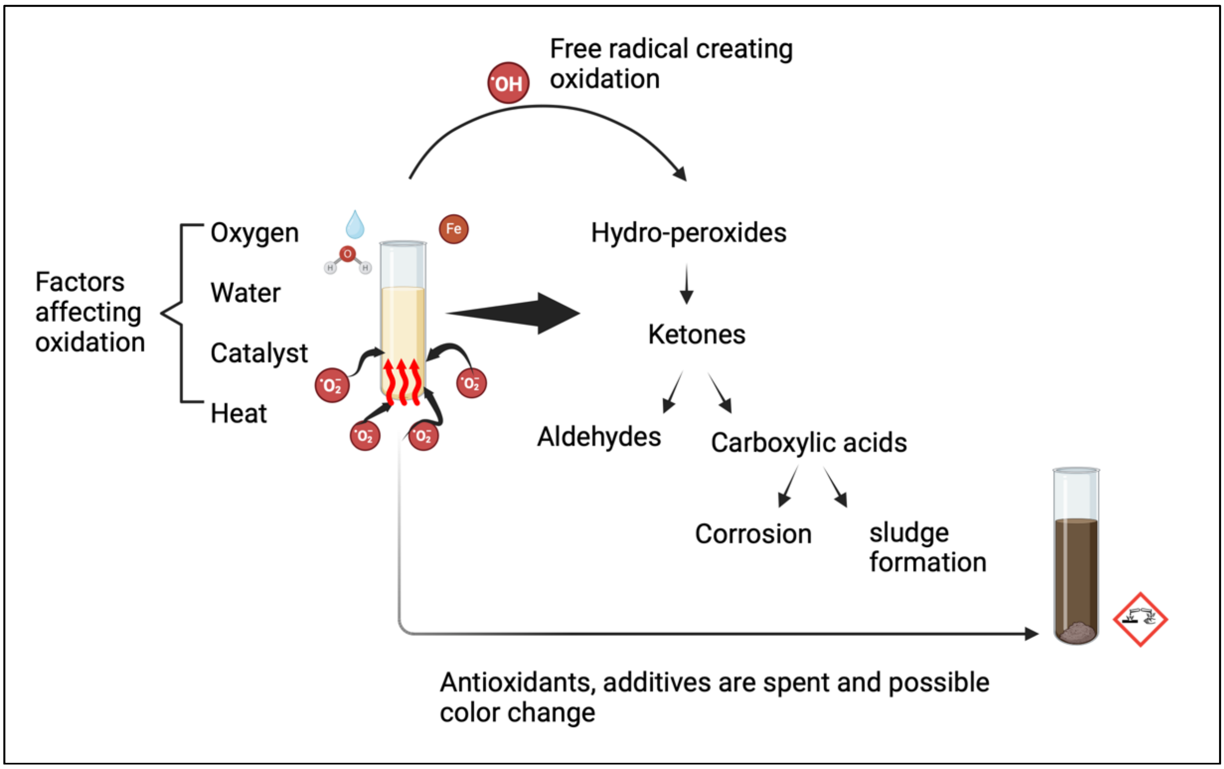


Figure 3. Oxidation process in lubricants.

2.2. Role of Antioxidants and Antioxidant Activity Analysis

Oxidation of lubricants is accelerated in harsh environments such as high temperature, pressure, metal content, presence of moisture and friction [15]. Antioxidants are compounds that, when mixed with base oils, enhance their properties and neutralize the free radicals produced during operation, hence preventing oxidation and improving the useful life of the lubricant.

Figure 4 shows the antioxidant classification based on source, solubility and mechanism of action.

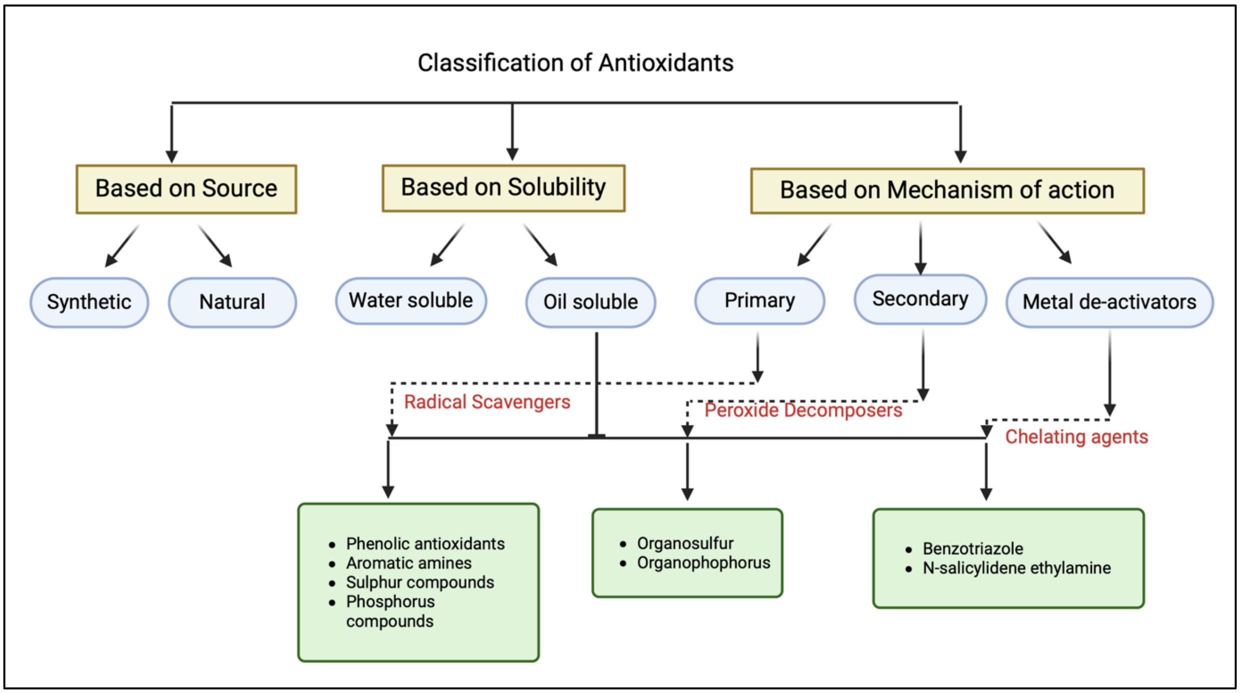


Figure 4. Classification of antioxidants.

Antioxidants are broadly classified as primary antioxidants (radical scavengers) and secondary antioxidants (peroxide decomposers) [15, 19]. The primary antioxidant functions as a radical remover in which the antioxidant actively scavenges the radicals to form non-reactive molecules (through the donation of hydrogen atoms) to inhibit the propagation of oxidation. On the other hand, the secondary antioxidants react with the hydroperoxides to convert them into non-radical products such as alcohol. The two types of antioxidants are often used together as an additive in the production of lubricants due to their synergistic effects, which have been found in many studies [17]. Metal deactivators act as surface film-forming compounds or chelating agents, which reduces the catalytic effect of metal ions on oxidation, thus forming stable complexes and preventing corrosion. Monomeric and dimeric phenolic compounds are primary antioxidants. It is found that the dimeric phenolic compound is more potent than the monomeric phenolic compounds in terms of antioxidation ability due to their double functionality of bonding with hydrogen atoms separately and quenching radicals, whereas the monomeric phenolic compound mostly only reacts via hydrogen bonding, hence reducing their effectiveness [20].

The role of phenol-based antioxidants in lubricating oils is to donate hydrogen cation from the phenolic hydroxyl group to the $R–O^{\bullet}$ and $R–O–O^{\bullet}$ radicals, which terminate the propagation of oxidation in lubrication oils [21]. The dehydrogenation of phenolic compounds is related to the bond dissociation energy (BDE) of the O–H bond. The rapid increase in $R–O–O^{\bullet}$ radicals during oil oxidation/autooxidation facilitates the dehydrogenation at the weak BDE site (due to its instability) and the capturing of hydrogen atoms as the main reaction in the lubricating oil. The strength of which phenolic compounds exude better antioxidative performance depends on the position of electron-donating substituents in the phenol structure. In the para position, the electron density of the oxygen atom in the phenol structure increases the dipole moment of the O and H atoms while decreasing the BDE. For phenols with ortho-substituent structures, it effectively prevents the autoxidation of the phenols due to its large steric hindrance, thereby providing sufficient H atoms to stabilize the alkyl peroxyl radical [22]. In addition, the scavenging activity of the phenolic antioxidants can be enhanced by having electro-donating groups on the ortho position, which increases the rate of reaction with the R–O–O$^{\bullet}$ radicals while having the ability to stabilize the phenoxyl radical through hyperconjugative and inductive effects [23].

Antioxidant performance is commonly assessed using radical scavenging assays, which operate through two primary mechanisms: hydrogen atom transfer (HAT) and single electron transfer (SET) [24]. The HAT method evaluates the hydrogen donating ability of bioactive compounds for the quenching of free radicals. Some of the HAT methods include oxygen radical absorbance capacity (ORAC), total radical trapping antioxidant parameter (TRAP), total radical scavenging capacity assay (TOSCA), chemiluminescent assay, β-Carotene bleaching assays, and inhibition of induced LDL oxidation [25]. These HAT methods are rapid reactions and are pH and solvent independent. On the contrary, the SET method evaluates the ability of electron donation of bioactive compounds to reduce radicals, metals, and carbonyl groups. Unlike HAT methods, SET methods are pH dependent. Examples of SET methods are total phenolics assay by Folin–Ciocalteu reagent assay, Trolox equivalence antioxidant capacity assay (TEAC), Ferric ion reducing antioxidant power assay (FRAP), 2,2-Diphenyl-1-picrylhydrazyl (DPPH) radical scavenging assay, 2,2-Azinobis 3-ethylbenzthiazoline-6-sulfonic acid (ABTS) radical scavenging assay, N,N-Dimethyl-p-phenylenediamine (DMPD) radical scavenging assay, and cupric ions (Cu^{2+}) reducing antioxidant power assay (CUPRAC) [24]. Some of the assays (such as the DPPH scavenging assay, ABTS scavenging assay, and FRAP assay) are widely used in antioxidant performance evaluation due to their simplicity, sensitivity, and consistency.

In the DPPH scavenging assay, the antioxidant performance is evaluated by the difference in the absorbance of the stable free radical of 2,2-Diphenyl-1-picrylhydrazy. The DPPH$^{\bullet}$ radical has an absorbance of 517 nm, which is characterized by its deep purple color. As the antioxidants in present in the DPPH solution, the color intensity of the DPPH solution will be reduced due to the radical scavenging activity of the antioxidants in which a hydrogen atom or an electron is donated to the antioxidant to quench and stabilize the $DPPH^{\bullet}$ radical [26]. The antioxidant performance is then evaluated through the degree of discoloration or the reduction of absorbance. The ABTS scavenging assay is a spectrophotometric analysis method that is used to measure antioxidant activity. The ABTS scavenging assay is applicable for both hydrophilic and lipophilic compounds. There are two ways of evaluating the antioxidant activity. (1) The bioactive compound will first be mixed with the 2,2-Azinobis 3-ethylbenzthiazoline-6-sulfonic acid. Then, the ABTS will be oxidized to form $ABTS^{\bullet+}$ cation radical. The bioactive compound added in the beginning will then cause the $ABTS^{\bullet+}$ formation and cause the increase in the inhibition absorbance percentage [27]. (2) The ABTS will first be oxidized to form its radical. Then, the bioactive compound will be added to react with the $ABTS^{\bullet+}$, which will cause discoloration. The antioxidant activity is evaluated through the inhibition of $ABTS^{\bullet+}$ absorbance, in which the absorbance maxima are 414, 734, and 815 nm [24]. The FRAP assay assessed the antioxidant activity through the degree of ferric ion (Fe^{3+}) complex reduction. The colorless 2,4,6-tripyridyl-s-triazine complex $[Fe^{3+}\text{-}(TPTZ)_2]^{3+}$ will turn into a deep blue color when it is reduced to $[Fe^{2+}\text{-}(TPTZ)_2]^{2+}$. Then, the antioxidant activity of the bioactive compound is tested by comparing the absorbance at 593 nm with that of a positive control (either an antioxidant standard solution or a ferrous ion standard solution) [28]. In summary, all three of the methods use the colorimetric test whereby the performance of antioxidants is measured with its absorbance at 517 nm for the DPPH assay, at 414, 734, and 815 nm for the ABTS assay, and 593 nm for the FRAP assay.

2.3. Antioxidants from Crops, Agricultural Waste and Biomass

Besides synthetic antioxidants, antioxidants can be extracted from multiple natural sources such as first-generation crops, second-generation crops, and agricultural wastes and biomass. The first-generation crops are from foods (green tea, bell pepper, berries, apples, pomegranate), herbs (rosemary, oregano, ginger, mint), and spices (clove, turmeric, cumin, nutmeg) [29-31]. While first-generation crops produce a considerable amount of antioxidants, it is not recommended for the commercialization of industrial antioxidants as it directly competes with food security. As for second-generation crops, antioxidants can be found in various plants that are not usually for direct consumption. Natural antioxidants are mainly classified as phenolic compounds, vitamins and carotinides [32-34]. Antioxidants can be extracted from crops such as *Pseuderanthemum palatiferum* (Nees) Radlk. leaves, *Rubia truppeliana* (and other Rubia species), *Pistacia lentiscus* L., and *Trapa quadrispinosa* Roxb. [35-38]. However, these crops are used as folk medicines and traditional treatments. These second-generation crops are found to be strongly beneficial to the field of pharmacology. They can be exploited for cancer treatment and disease treatment [39-41]. Hence, the use of first- and second-generation sources as antioxidant extraction feedstock for industrial application may not be feasible in terms of global health and economics as the sources may be more beneficial when used in the medical field or food science.

On the contrary, agricultural waste and biomass are the preferred feedstock for the industrial use of antioxidants. With the increase in global population, modern agricultural methods focus on the profitability of crop production by over-exploiting natural resources to achieve global supply and demand [42]. Modern agriculture produces a substantial amount of agricultural waste, such as fruit and crop residues, peels, stalks, leaves, and shells. Moreover, it is reported that the agri-food industries generate large amounts of by-products

(20 – 60 wt. % of fresh produce) from fruit and vegetable processing [43]. The increase in agri-food waste generation may lead to detrimental and irreversible environmental effects given the process of landfilling and composting of these wastes generates nitrous oxide and methane gas which are forecasted to be 20 times the amount of carbon dioxide generation [44]. Therefore, the valorization of agri-food by-products is highly preferred, and it may help with waste generation and sustainable production. Table 1 shows the types of agri-food waste, generation and antioxidant compounds. The highest waste generation is maize residue, followed by sugarcane bagasse, palm oil biomass, potato by-products, and banana peels. The most common antioxidant compounds found in these agri-food wastes are chlorogenic acid, gallic acid, quercitrin, protocatechuic acid, catechin, ferulic acid, and p-coumaric acid. There are mainly primary antioxidants.

Table 1. Generation of different agricultural wastes and biomass from different countries and their antioxidant compounds.

Country	**Agri-food Waste/ Biomass**	**Generation (metric ton/year)**	**Antioxidant Compounds**	**Reference**
Malaysia	Palm oil biomass (PKS, EFB, fronds, trunks)	75,610,000	• Pyrogallol (P) • Gallic acid (P) • 4-Hydroxybenzoic acid (P) • Ferulic acid (P)	[45, 46]
	Rice husk	770,000	• Momilactone B (S) • *p*-coumaric acid (P) • Ferulic acid (P) • Protocatechuic acid (P) • Gallic acid (P)	[47, 48]
	Coconut waste (husk and shell)	351,450	• Syringic acid (P) • Homovanillic acid (P) • *p*-Coumaric acid (P) • Catechin (P) • Epigallocatechin (P) • Epicatechin (P) • trans-cinnamic acid (S)	[49-51]
	Durian husk	311,399	• Quercetin (P) • Catechin (P) • Centaureidin-3-glucoside (P) • Procyanidin B (P) • Rutin (P)	[49, 52, 53]
China	Maize (corn) residue	451,724,992	• Ferulic acid (P)	[54-58]

			• *p*-Coumaric acid (P) • *p*-Hydroxybenzoic acid (P) • Chlorogenic acid (P) • Flavan-3-ol (P) • Proanthocyanins • cis,cis-9,12-octadecadienoic acid (P) • n-hexadecanoic acid (P)	
	Potato by-products	33,470,989*	• Chlorogenic acid (P) • Gallic acid (P) • Protocatechuic acid (P) • Caffeic acid (P)	[54, 59, 60]
	Soy pulp/ okara	2,800,000	• Phytic acid (S) • Phenolic compounds (P) • Polypeptide (S)	[61-63]
India	Pomegranate peel	1,607,980*	• Catechin (P) • Chlorogenic acid (P) • p-hydroxybenzoic acid (P) • Epicatechin (P) • Protocatechinic acid (P) • Epicatechin (P) • Coumaric acid (P) • Gallic acid (P)	[64-66]
	Groundnut waste (shell and skin)	3,980,536*	• Proline (S) • *p*-coumaric acid (P) • *t*-ferulic acid (P) • Protocatechuic acid (P) • Vanillic acid (P) • 4-hydroxybenzoic acid (P)	[67-70]
	Banana peel	13,811,200*	• 9,19-Cyclolanostan-3-ol, acetate, (3β) (S) • Quercetin (P) • Quercetin-3-rhamnoside (P) • *p*-hydroxybenzoic acid (P) • Catechin (P) • Chlorogenic acid (P) • Protocatechuic acid (P) • Gallic acid (P)	[54, 71-73]
USA	Tomato pomace	2,549,938*	• Lycopene (S) • Chlorogenic acid (P) • Gallic acid (P) • Caffeic acid (P) • Ferulic acid (P) • Quercetrin (P)	[54, 74-76]
	Apple pomace	1,218,066*	• Epicatechin (P) • Hyperin (P) • Chlorogenic acid (P) • Cynaroside (P) • Quercitrin (P)	[54, 77]
Africa	Cassava peel	6,620,000	• Gallic acid (P) • Catechin (P)	[78, 79]
	Cacao by-products	2,900,000	• Epicatechin (P) • Catechin (P) • Procyanidin (P)	[80-82]

	Citrus waste (orange, lemon, tangerine, grapefruit)	1,725,000	• Eriocitrin (P) • Hesperidin (P) • Protocatechuic acid (P) • Chlorogenic acid (P) • Isoquercetin (P) • Quercetrin (P)	[83-85]
Spain	Olive byproducts (leaves and pomace)	1,871,533*	• Oleuropein (P) • Ligustroside (P) • Caffeic (P) • Luteolin (P) • Rutin (P) • Chlorogenic (P) • Quercetin (P)	[54, 86-88]
Brazil	Sugarcane bagasse	197,044,453*	• Gallic acid (P) • *p*-Coumaric acid (P) • Ferulic acid (P) • Resveratrol (P) • Quercetin (P)	[54, 89, 90]

Remark:
*Average waste generation based on literature.
P – primary antioxidant   S - secondary antioxidant

3. Methods of Antioxidant Extraction

There are two classifications of antioxidant extraction methods, namely, conventional methods and green advanced technologies. The conventional method involves the combination of heat and agitation in the solvent extraction. Its extraction efficiency is dependent on the types of solvent. The conventional method is usually carried out over a prolonged period, which was proven to be a limitation given the excessive time and energy consumption [43]. In addition, the drawback of the conventional method extends to large quantities of solvent use, low extraction yield of targeted bioactive compounds, and usage of environmentally polluting solvents [43, 44]. To facilitate antioxidant extraction efficiency, many other advanced green technologies have emerged over the years to tackle the shortcomings of the conventional method. Examples of green advanced techniques are ultrasonic-assisted extraction (UAE), microwave-assisted extraction (MAE), pressurized liquid extraction (PLE), supercritical fluid extraction (SFE), deep eutectic solvent extraction (DESE), pulsed electric field extraction

(PEFE), and enzyme assisted extraction (EAE). The various extraction methods are schematically summarized in

Figure 5.

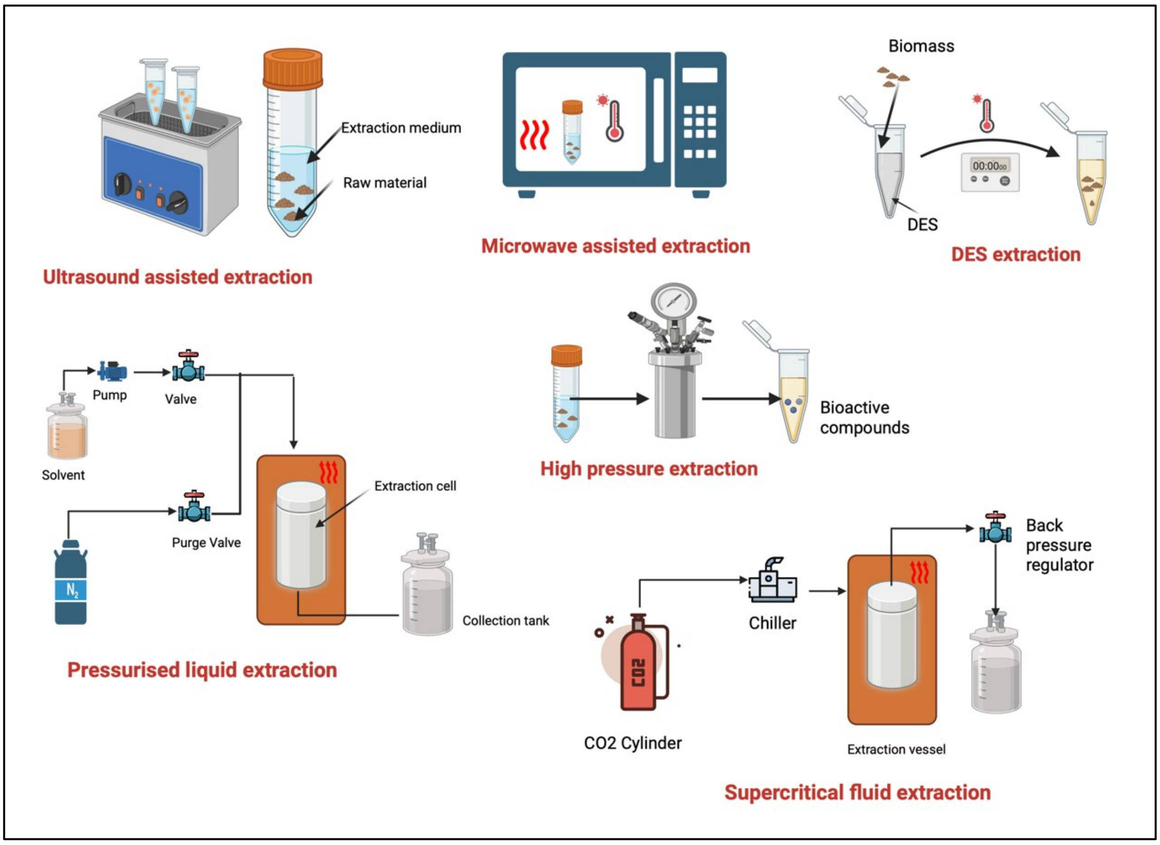


Figure 5. Schematic representation of various extraction methods.

Table 2 outlines the extraction methods employed to isolate antioxidants and phenolic compounds from various biomass sources. The data indicate that the efficiency and yield of antioxidants vary significantly across different extraction techniques, influenced by a range of critical factors. Key general parameters affecting recovery and selectivity include the solid-to-liquid ratio, solvent concentration, extraction temperature, and duration. Additionally, method-specific factors such as ultrasonic intensity, amplitude, irradiation power, rotational speed, pressure, field strength, and frequency play a crucial role in determining the effectiveness of the extraction process. Referring to Table 2, the typical operating frequency and extraction time for the UAE is 20 kHz to 40 kHz and >15 mins. MAE typically operates at an irradiation power of 600 W for < 40 mins. PLE generally operates at an elevated pressure and temperature of > 100 bar and > 150°C. Similar to PLE, SFE operates at an elevated pressure of around 200 bar. However, the operating temperature is milder (60°C to 80°C) compared to PLE. Many factors may affect the performance and efficiencies of each operational parameter. Therefore, the following subsections discuss the respective performance of different methods concerning the varying factors and further compare their advantages and disadvantages.

Table 2. Extraction conditions of different extraction method and their respective antioxidant content and activity.

Agricultural Waste	Extraction condition and Yield	Antioxidant Content	Antioxidant Activity	References
Conventional solvent extraction				
Sugar beet (*Beta vulgaris* L.) leaves	EtOH (50%), Ambient Temp., Rotation speed = 150 rpm, Extraction time = 24 hr, S:L Ratio = 1:10 (w/v) Yield: 77.98%	TPC: 0.93 g GAE/100 g DW	DPPH: 77.98 µM TE/g FRAP: 95.14 µM Fe^{2+}/g ABTS: 58.92 µM TE/g	[91]
Walnut (*Juglans regia*) shells & husks*	*For shell:* Acetone (75%), 50℃, Extraction time = 60 min, S/L Ratio = 1/20 g/mL *For green husk:* EtOH (100%), 50℃, Extraction time = 30 min, S/L Ratio = 1/20 g/mL	TPC: 10.54 mg GAE/g shell TPC: 1.29 mg GAE/g green husk	DPPH: 28.74 ± 2.16 mg TE/g shell DPPH: 36.70 mg TE/g green husk	[92]
Surian (*Toona sinensis)* leaves	EtOH (96%), S:L Ratio = 1:10 (w/v) Yield: 11.396%	TPC: 351.63 mg GAE/g TFC: 324.61 mg QE/g	DPPH: 3.38 ppm	[93]
Saffron (*Crocus sativus*) petals	EtOH:water (3:2), 66℃, Extraction time = 104 min	TPC: 1134 ± 3 mg GAE/100 g, TFC: 85 ± 2 mg QE/100 TAC: 71 ± 2 mg/100 g	DPPH: 61 ± 1% FRAP: 3.2 ± 0.2 mM	[94]
Ultrasonic-assisted extraction (UAE)				
Sugar beet (*Beta vulgaris* L.) leaves	EtOH (50%), 70℃, Frequency = 40 kHz, Extraction time = 30 min, S:L Ratio = 1:10 (w/v), Ultrasonic intensity: 60 W/L (watt/litre) Yield: 70.76%	TPC: 1.61 g GAE/100 g DW	DPPH: 70.76 µM TE/g FRAP: 287.50 µM Fe^{2+}/g ABTS: 150.70 µM TE/g	[91]
Pomegranate (*Punica granatum* L.) peel	EtOH (70%), 50℃, Frequency = 20 kHz, Extraction time = 15.12 min, Amplitude: 30% Yield: 42.45%	TPC: 354.67 mg GAE/g TE: 348.0 mg TAE/g	DPPH: 94.78%	[95]
Sage (*Salvia officinalis* L.) by-products	EtOH (60%), 75.4℃, 80 min, Frequency = 40 kHz, Ultrasonic intensity: 42.54 W/L (watt/litre)	TPC: 9.881 ± 0.157 g GAE/100 g DW TFC: 6.997 ± 0.341 g CE/100 g DW	DPPH: 9.02 – 21.44 µg/mL FRAP: 124.1 – 221.0 mg AAE/g DW	[96]
Pomegranate (*Punica granatum* L.) peel	H_2O, S:L Ratio 1:50 (w/w), Frequency = 20 kHz, Ultrasonic intensity = 59.2 W/cm^2, Extraction time = 60 min, 25 ± 2℃, Pulse Duration 5 s, Pulse Interval: 5 s	Total phenolic yield: 14.5 – 14.8%	DPPH: 5.5 – 5.8 g/g	[97]

	Yield: 14.5 – 14.8%			
Microwave assisted extraction (MAE)				
Sugar beet (*Beta vulgaris* L.) leaves	EtOH (70%), Extraction time = 10 min, Irradiation power = 600 W, S:L Ratio = 1:10 (w/v) Yield: 31.53%	TPC: 1.7171 g GAE/100 g DW	DPPH: 76.53 µM TE/g FRAP: 290.97 µM TE/g ABTS: 168.74 µM TE/g	[91]
Aloe vera skin by-products	EtOH (80%), 80°C, Extraction time = 36.6 min Yield: 17.3 ± 0.1%	TPC: 116.4 ± 4.5 mg GAE/g AVE	DPPH: 69.0 ± 1.9 mg TE/g AVE FRAP: 131.9 ± 6.5 mg TE/g AVE	[98]
Sage (*Salvia officinalis* L.) by-products	EtOH (46.2%), 75.4°C, 18.7 min, Irradiation power = 600 W, L/S Ratio = 40 mL/g	TPC: 10.379 ± 0.193 g GAE/100 g DW TFC: 7.544 ± 0.098 g CE/100 g DW	DPPH: 10.40 – 17.24 µg/mL FRAP: 68.19 – 92.57 mg AAE/g DW	[96]
Pressurised liquid extraction (PLE)				
Granadilla (*Passiflora ligularis* Juss) peel	EtOH (50%), 150°C, Pressure = 100 bar, Extraction time = 30 min Yield: 1.99 ± 0.11%	TPC: 2.01 ± 0.07 mg GAE/g biomass TCC: 0.54 ± 0.03 mg/g biomass	ABTS: 3.6 ± 0.1 mg Trolox/g biomass	[99]
Sugar beet (*Beta vulgaris* L.) leaves	EtOH (50%), 150°C, Pressure = 103.4 bar, Extraction time = 10 min, S:L ratio = 1:20 (w/v), Purge time: 90 s Yield: 32.89%	TPC: 1.32 g GAE/100 g DW	DPPH: 49.82 µM TE/g FRAP: 292.78 µM Fe^{2+}/g ABTS: 127.86 µM TE/g	[91]
Wild thyme (Thymus serpyllum L.) dust	EtOH (30%), 170°C, Pressure = 103.4 bar, Extraction time = 30 min, S:L Ratio = 1:20 (w/v), N_2 Purge Time: 90 s Yield: 32.14%	TPC: 6.7464 ± 0.1860 g GAE/100 g DW	DPPH: 0.3173 ± 0.0035 mM TE/g FRAP: 0.8587 ± 0.0032 mM Fe^{2+}/g ABTS: 0.6943 ± 0.0204 mM TE/g	[100]
Supercritical fluid extraction (SFE)				
Alpeorujo (semi-solid residue from the olive oil)	60°C, Pressure = 200 bar Yield: 7.7 – 11.6%	TPC: 1147 – 1748 mg GAE/kg TTC: 232 – 454 ppm	ABTS: 1.74 ± 0.05 mM TEAC	[101]
Aurantiochytrium sp. biomass*	80°C, 200 bar, Flow rate = 6 g/min Yield: 2.1%	TPC: 2.09 ± 0.27 mg GAE/g extract	TEAC: 1.4 ± 0.4 mg TEAC/ g extract	[102]
Saffron (*Crocus sativus*) petals	62°C, Extraction time = 47 min, Pressure = 164 bar	TPC: 1423 ± 7 mg GAE/100 g, TFC: 180 ± 4 mg QE/100 TAC: 103.4 ± 2 mg/100 g	DPPH: 14.5 ± 1% FRAP: 3.9 ± 0.5 mM	[94]
Deep eutectic solvent extraction (DESE)				
C. *vulgaris* biomass	ChCl:1,2 butanediol (1:4)/H_2O 70/30 (w/w), 60°C, Extraction time = 6.34 hr, S:L Ratio = 1:40	TPC: 12.749 mg GAE/g DW TCC: 3.705 mg /g DW	DPPH: 0.119 g DW/mL solvent	[103]

Red grape pomace	GL-CA-Pro (2:1:1)/H_2O 80/20 (w/w), 50°C, Extraction time = 180 min, Rotational speed = 500 rpm, S/L Ratio: 10 mL/g	TPC: 28.33 mg GAE/g DW	DPPH: 178.92 µmol DPPH/g DW FRAP: 146.93 µmol AAE/g DW	[104]
Olive leaves	GL-CA-Pro (2:1:1)/H_2O 80/20 (w/w), 50°C, Extraction time = 180 min, Rotational speed = 500 rpm, S/L Ratio: 10 mL/g	TPC: 28.61 mg GAE/g DW	DPPH: 234.94 µmol DPPH/g DW FRAP: 152.39 µmol AAE/g DW	[104]
Tomato pomace extract	ChCl:LA (1:2)/H_2O 90/10, 65°C, Extraction time = 60 min, Ultrasonic Power = 100 W, Frequency = 30 kHz	TPC: 51.75 ± 1.15 mg GAE/g, TFC: 17.31 ± 1.15 mg QE/g	DPPH: 43.35 % ABTS: 31.71 %	[76]
Pulsed electric field extraction (PEFE)				
Grape stalk	H_2O, 100 kJ/kg, Field strength = 3 kV/cm, S:L Ratio 1:10 (w/v), Frequency: 2 Hz, Pulse Duration: 100 ms	TPC: 11.84 mg GAE/g DW	TEAC: 106.35 µmol TE/g DW ORAC: 357.75 µmol TE/g DW	[105]
Grape marc	H_2O, 100 kJ/kg, Field strength = 3 kV/cm, S:L Ratio 1:10 (w/v), Frequency: 2 Hz, Pulse Duration: 100 ms	TPC: 11.45 mg GAE/g DW	TEAC: 89.69 µmol TE/g DW ORAC: 71.87 µmol TE/g DW	[105]
Exhausted grape marc	H_2O, 100 kJ/kg, Field strength = 3 kV/cm, S:L Ratio 1:10 (w/v), Frequency: 2 Hz, Pulse Duration: 100 ms	TPC: 21.03 mg GAE/g DW	TEAC: 40.54 µmol TE/g DW ORAC: 366.47 µmol TE/g DW	[105]
Pomelo peel	EtOH (40%) Field strength = 4 kV/cm, 40°C, 30 pulses, S/L Ratio = 1 g/90 mL Yield: 16.19 mg/mL	ND	DPPH: 38.58%	[106]
Moringa oleifera freeze-dried leaves	DD H_2O, Field strength = 7 kV/cm, Ambient Temp., Extraction time = 40 min, Pulse Duration: 20 ms, Pulse Interval: 100 µs	TPC: 40.24 mg GAE/g DW	DPPH: 98.31% FRAP: 108.22 µmol AAE/g DW	[107]
Enzyme assisted extraction (EAE)				
Banana (*Musa acuminate* cv. sagor) peel	Viscozyme® L, Concentration: 1.0%, 55°C, Extraction time = 9 hr, pH 4.0, S:L Ratio = 1:25	TPC: 25.37 mg GAE/g DM	TFC: 13.99 mg QE/g DW DPPH: 81.59% ABTS: 88.25%	[108]

Mango (*Mangifera indica* L.) peels	Alcalase, Concentration: 3.3%, 63°C, Extraction time = 110 min, pH 5.5, Ultrasonication power: 90 W Yield: 27.74 ± 0.94%	TPC: 33.56 ± 1.04 mg GAE/g fresh weight	DPPH: 11.38 µg/mL extract TEAC: 215.42 ± 1.21 mM TE/g extract	[109]
Blackcurrant (*Ribes nigrum* L.) press cake*	Pectinase + A. *oryzae*, 40°C, Rotational speed = 140 rpm, Extraction time = 4 hr, S:L Ratio = 1:4	TPC: 898 mg/100 g	DPPH: 1080 mg AAE/100 g CUPRAC: 3155 mg AAE/100 g	[110]

*Optimum results were taken based on the highest antioxidant activity.

3.1. Conventional Solvent Extraction

Conventional solvent extraction involves the usage of solvent to draw out a desired substance (a solute) from another substance. Solvent extraction can be divided into two categories: homogeneous extraction and maceration extraction [111]. Homogeneous extraction involves the extraction of a dissolved substance in an existing solvent with a second solvent in which the second solvent has the affinity of said dissolved substance but is not miscible with the existing solvent. On the contrary, maceration extraction is a solid-liquid extraction method. The desired substance is extracted through the immersion of the solid in a solvent. As the solid is immersed in the solvent, the desired substance will diffuse from the solid body into the solvent phase. In general, the driving force for solvent extraction is the difference in chemical equilibrium caused by the different solute concentrations between the two phases. For ideal solvent extraction, the solvent should possess high selectivity/affinity for the desired solute, high extraction capacity, and be chemically stable [112]. The most widely used solvent for antioxidant extraction is ethanol due to its cheap, non-toxic, and reusable nature, as it can be produced from a renewable source (e.g., sugarcane)[113].

The yield of antioxidant extraction and its antioxidant activity are governed by the solvent concentration, temperature, and time. Maulana et al. [93] examined the effectiveness of maceration of the *Toona sinensis* leaves with varying aqueous ethanol concentrations (70%, 80%, 90%, and 96%) in antioxidant extraction. The highest yield of total phenolic content and total flavonoid content were obtained at 90% (444.68 mg GAE/g) and 96% (324.61 mg GAE/g) aqueous ethanol, respectively. Depending on the desired product, the solvent concentration can vary for optimal extraction. The use of different solvent concentrations can fractionate the phenolics or flavonoid compounds based on their polarity. Mokrani and

Madani [114] investigated the factors influencing the extraction of TPC, TFC, and antioxidant activity from peach (*Prunus persica L.*) fruit. Their study highlighted the crucial impact of temperature on the extraction process. The findings indicate that increasing the temperature leads to a reduction in TPC while enhancing TFC. Specifically, the highest TPC of 363 mg GAE/100 g was obtained at 25°C. However, as the temperature was raised from 37.5°C to 70°C, TPC declined from 313 to 280 mg GAE/100 g, suggesting that phenolic compounds are highly sensitive to heat, undergoing oxidation and decomposition at elevated temperatures. In contrast, TFC increased from 38 to 81 mg QE/100 g as the temperature was elevated from 25°C to 60°C. This is due to the weakened phenol-polysaccharide and phenol-protein interaction from the denatured cell wall, causing more polyphenols to migrate into the solvent phase. The study also reported a decrease in antioxidation activity of 9% when the extraction temperature increases from 25℃ to 37.5℃, possibly due to the decreased TPC. Mokrani and Madani [114] manipulated the extraction time from 30 to 450 min. The TPC yield increased with time. However, the extraction time beyond 180 min does not improve the TPC yield. Moreover, the prolonged extraction time led to a decrease in TPC yield and a decrease in DPPH scavenging activity due to phenolic oxidation.

3.2. Ultrasonic-Assisted Extraction

UAE is a green extraction method that introduces ultrasonic waves with a frequency of around 20 kHz to 10 MHz during the extraction process. The ultrasonic waves induce a simultaneous formation, development, and collapse of bubbles, which induce cavitation and disrupt the cell wall, causing the deeper penetration of solvent into the cellular material and increasing the mass transfer rate of antioxidants into the solvent [95, 113]. Aside from the type of solvent and solvent concentration, the process is governed by temperature, time, frequency, sample-to-solvent ratio, and wave amplitude. In a study of phenolic compound extraction from pomegranate (Punica granatum) waste under 20 kHz sonication frequency, Foujdar et al. [95] found that the increase in sonication time of more than 15 minutes decreased the TPC extraction due to structural deformation of bioactive compounds and creation of free hydroxyl radicals that subsequently decrease the phenolic compounds and antioxidant activity. Furthermore, the increase in wave amplitude from 20% to 30% facilitated the TPC extraction process due to the increased mass transfer efficiency. Nonetheless, amplitude beyond 30% showed antagonistic effects in terms of TPC yield and antioxidant activity. In fact, at 30% amplitude, the antioxidation efficiency is 94.96%. However, at 40% amplitude, the antioxidation efficiency decreased from around 54.26% to 72.81%, which was at least a 22% decrease in performance. Hence, the prolonged ultrasonication and excess wave amplitude tend to cause structural deformation of phenolic compounds and create free hydroxyl radicals that subsequently decrease the phenolic compounds and antioxidant activity [115]. Quiroz-Reyes and Aguilar-Méndez [116] studied the antioxidant extraction of different ultrasonication modes and reported that the pulsed ultrasonication increased the TPC extraction yield. However, it is also noted that the other modes did not significantly impact the TPC extraction yield and its HDDP scavenging activity [97].

3.3. Microwave Assisted Extraction

MAE has emerged as a widely used method for extracting soluble products, particularly in the last decade, due to its recognition as a green technology. Operating at frequencies ranging from 300 MHz to 300 GHz, MAE is versatile and applicable to a broad range of materials [117]. The operating principle involves the conversion of electromagnetic energy to heat energy through the dipole rotation mechanism and ionic conduction. Alupului et al.

[118] outlined three key phases in the MAE extraction process: (1) elevated temperature and pressure disrupt the sample matrix, releasing solutes from their active sites; (2) the solvent penetrates the matrix; and (3) the solutes diffuse into the solvent, completing the extraction. The efficiency of MAE depends on several factors, including the sample-to-solvent ratio, solvent concentration, irradiation time, and irradiation power. Dang et al. [119] studied the effects of solvent concentration, irradiation time, and irradiation power on the extraction of phenolic compounds from the alga *Sargassum vestitum*. In their study, the effects of the independent factors in the performance indicators such as TPC yield, ABTS total antioxidant capacity, DPPH free radical scavenging capacity, and FRAP were found to be in the order of solvent concentration, followed by either irradiation time or irradiation power. The increase in solvent concentration increased the TPC yield and antioxidation activities as opposed to lower solvent concentration due to the medium polarity of phenolic compounds in the algae.

Bhuyan et al. [120] also reported similar results whereby the effects of irradiation time and irradiation power of the MAE were not significant to the TPC extraction yield, proanthocyanidins yield, and antioxidation activities of *Eucalyptus robusta* leaf. An increase in the sample-to-solvent ratio significantly decreased the TPC extraction yield and increased the proanthocyanidins yield and the ABTS total antioxidant capacity. The decrease in TPC extraction yield is due to the increased suspension density. The increased inhibition effect from the dense suspension of solute from the cell leads to the eventual decrease in effective solvation. Notably, the increase in irradiation time increased the TFC yield and the iron chelating capacity (analyzed with CUPRAC assay). Despite the above findings, the irradiation time and irradiation power of MAE should not be disregarded as it is found that the increase of 400 W to 500 W in irradiation power increased the TPC yield from *Myrtus communis* L. leaves by around 13% (from 134.63 to 152.25 mg GAE/g DW) [121]. However, the increase of irradiation power beyond 600 W reduced the TPC yield (lowest yield of 129.95 mg GAE/g DW at 900 W) due to the thermal decomposition of phytochemicals. It is also reported that an irradiation time beyond 90 seconds reduced the TPC yield. Therefore, researchers need to consider the solvent affinity (types and concentrations of solvent, sample-to-solvent ratio) and the optimal thermal settings (irradiation time and irradiation power) for the targeted phenolics during the extraction.

3.4. Pressurized Liquid Extraction

Similar to solid-liquid extraction, the PLE involves the usage of solvent to extract phenolic compounds at an elevated temperature. The PLE is operated with an additional factor of pressure. The operating pressure is elevated so that the system can operate beyond the normal boiling point of the solvent [117]. At a higher operating temperature, the phenolic compound extraction is facilitated due to the reduced solvent viscosity, improved penetration into the plant matrix, and increased solute diffusion [100]. Aside from the operating pressure, the factors affecting the performance of PLE are like those of the conventional solid-liquid extraction, such as pressure, temperature, type of solvent, solvent concentration, extraction time, sample particle size, and more. Toledo-Merma et al. [122] studied the recovery of phenolic compounds from pomegranate (*Punica granatum* L.) by-products. They reported an increase in phenolic compound yield from 27.41% to 44.99% with an increase of pressure from 20 bar to 80 bar. The increase is due to the polarity change, favouring extraction from the pomegranate by-products. However, the further increase in the pressure to 100 bar decreased the yield as the surface contact of the solvent and the sample matrix was hindered. However, some studies showed that the influence of pressure on PLE is of insignificance. In the case of TPC and TFC extraction from mung bean (*Vigna* *radiata* L.) seed coat, Supasatyankul et al. [123] found that the increase in pressure does not affect the TPC yield and the ABTS scavenging activity. Instead, the increased pressure showed a significant effect on the TFC yield. Aside from pressure, temperature and extraction time also play an important role in PLE. Mrkonjić et al. [100] reported an increase in TPC with the increase in reaction temperature and extraction time. In PLE, temperature should be increased in conjunction with pressure. Insufficient temperature will decrease the extraction yield of bioactive compounds as the increased pressure decreases the viscosity of the solvent, thereby reducing the mass transfer, diffusivity, and extraction efficacy [123]. However, an upper limit should be set as the increase of both factors may accelerate the rate of bioactive compound degradation. Similar to other studies, the antioxidant activity is linearly correlated to the yield of the extracted product [100]. Therefore, various variables mentioned above should be considered for the optimization of the PLE performance for different raw materials.

3.5. Supercritical Fluid Extraction

SFE is a process in which the extraction of a product is carried out in a solvent pressure beyond its critical point. In a supercritical state, the solvent or supercritical fluid has a liquid-like density and solvation power in addition to having gas-like properties of viscosity, diffusion, and surface tension. One of the widely used supercritical fluids is carbon dioxide (CO_2), as its critical temperature is around 31°C, which is close to room temperature. However, the polarity of CO_2 is inherently unfavourable for polar component extraction; hence, SFE is usually carried out in conjunction with a polar solvent, such as a water-ethanol mixture, for antioxidant extraction [112]. The main factors affecting the SFE efficiency are temperature, pressure, and extraction time. During the antioxidants extraction from the *Crocus sativus* petals, Ahmadian-Kouchaksaraie and Niazmand [94] found that the increase in process temperature increased the extraction yield of TPC, TFC, and total anthocyanin content (TAC) until the respective maximum temperature of 63°C, 70°C, and 51°C. The increase in temperature improved the mass transfer rate due to the decreased viscosity coefficient, increased thermal desorption, enhanced diffusion coefficient, and improved phenolic solubility. The further increase in temperature reduced the yield of each respective compound due to thermal decomposition. The increase in pressure was also found to increase the extraction yield to a certain extent, as the increased pressure caused a rupturing effect on the plant material, thereby enhancing the interaction between the solvent and sample matrix. The increased pressure increases the fluid density and modifies the solute solubility, which may have either synergistic or antagonistic effects on the extraction yield, depending on the desired compounds.

On the other hand, it is also reported that the difference in the flow rate of supercritical fluid affects the extraction yield. In the propolis extraction by Fachri et al. [124], the increase in CO_2 flow rate increased the extraction yield as it reduced the mass transfer resistance due to increased solvent saturation. However, once the equilibrium is reached, a further increase in fluid flow rate is deemed undesirable as solvent saturation no longer dictates the extraction efficiency. Instead, the rate of diffusion is the determining factor; therefore, the high fluid flow rate may cause incomplete diffusion, causing the solvent to leave the system unsaturated.

3.6. Deep Eutectic Solvent Extraction

Deep eutectic solvent emerges as a class of green solvents that are similar to ionic liquids (ILs). Deep eutectic solvent got its name and garnered increasing interest after Abbott et al. noted its abnormally deep melting point depression in a eutectic mixture of hydrogen bond donors (HBD) and hydrogen bond acceptors (HBA) [125]. Unlike ionic liquids, the deep eutectic solvent possesses general characteristics of ILs (such as tune-able polarity, high thermal stability, low vapor pressure, and low volatility) with the upper hand in terms of cost-effectiveness, biodegradability, solvent recovery, non-toxicity, and easier synthesis [126]. DES is a designer solvent due to its flexibility in changing its composition based on different extraction requirements. Naturally, its antioxidant extraction efficiency is affected by the composition of HBD and HBA and its respective ratio. In the extraction of polyphenolic antioxidants from orange peels, Ozturk et al. [127] evaluated the performance of different HBD (glycerol (Gly) and ethylene glycol (EG)) in the choline chloride-based ([Ch]Cl) DES by manipulating their respective ratio. The extraction efficiency is reported to be higher when glycerol is used in the mixture of DES. At a mixture ratio of 1:2 ([CH]Cl:Gly), the highest recorded TPC is (2.9 mg GAE/g OP), which is higher than pure ethanol (1.5 mg GAE/g OP), pure methanol (1.9 mg GAE/g OP), pure acetone (2.0 mg GAE/g OP), and aqueous ethanol with 30% water (2.31 mg GAE/g OP) [127, 128].

Aside from the composition of the DES itself, the polarity of the DES can be manipulated through dilution (addition of water). Ozturk et al. reported that the [CH]Cl:Gly (1:2) with 10% water increased the extraction yield by 9.31% [127]. The further increase in water content decreased the extraction yield due to the limitation of hydrogen bond formation between the phenolic compounds and the DES. A similar finding was also reported by Pal and Jadeja [129] for the extraction of antioxidants from onion (*Allium cepa* L.) peel. Like conventional solvent extraction, the extraction efficiency of DES is affected by the process temperature, time, and sample-to-solvent ratio. In their study, Ozturk et al. [127] also reported an increase in polyphenol yield with the rise in temperature from 30℃ to 60℃, the increase of extraction time from 100 min to 120 min, and the increase of the sample-to-solvent ratio of 1:5 to 1:20. Despite the general increase in performance due to the decreased sample-to-solvent ratio, the ratio is 1:10 is considered optimal as higher solvent usage may not be economically viable. Furthermore, DESE can be coupled with other extraction methods, such as the UAE and MAE, for further performance enhancement [36, 129]. It is reported that the DESE assisted with microwave irradiation performed better than the conventional heating and stirring method and Soxhlet method in terms of TPC yield and total processing time [129].

3.7. Pulsed Electric Field Extraction

PEFE is a non-thermal extraction method that uses electric pulses to facilitate cell disruption in the intercellular vacuole for better extraction of bioactive compounds. The main principle of PEFE is to pass the electric potential through the cell membrane to separate the membrane molecules based on its dipole nature [117]. Electroporation is achieved under the induced transmembrane voltage from the pulse electric field. Once the transmembrane potential exceeds the critical threshold, pores will form on the weak areas in the membrane which would increase the permeability of the plant material [130, 131]. Several factors affect PEFE efficiency, namely extraction chamber geometry, electric field strength, pulse duration, pulse interval, and pulse shape. The PEFE can be carried out as a pretreatment of raw materials [131, 132], or it can also operate as a standalone extraction method [107, 130]. Pappas et al. [130] studied the optimization of PEF on the extraction of polyphenols from fresh olive leaves. They reported that the rectangular extraction chamber for PEF is superior to the cylindrical extraction chamber due to its uniformity of the electric pulses generated. The authors manipulated the electric field strength from 0.7 kV cm^{-1} to 1.0 kV cm^{-1} and found that the highest total polyphenol content is obtained at 0.85 kV cm^{-1} field strength, which is a 38.1% improvement as compared to the control sample. As the extraction time increased from 10 minutes to 15 minutes, the polyphenol yields significantly increased by about 48.77%. The further increase in extraction time to 30 minutes decreased the polyphenol yield, possibly due to the oxidation of some phenolic compounds. It is also reported that a short pulse duration and pulse interval of 2 µs and 100 µs are over other configurations as it produced the highest polyphenol yield. However, it is noteworthy that the configuration of different pulse durations and pulse intervals influenced the selectivity of various components in the extracted polyphenol compound despite different extraction yields, as reported in their study. In another study, Bozinou et al. [107] studied the antioxidant yield extracted from *Moringa oleifera* leaves and its antioxidant activity. They reported that the increase in pulse duration decreased the TPC yield while the decrease in pulse interval increased the TPC yield. The result is similar to that of the results reported by Pappas et al. in which the increase in the number/frequency of pulses increased the TPC yield. This phenomenon suggested that the permeability of the cell membrane increases with the number of pulses [106]. Also, it is reported that the DPPH of radical scavenging activity and the ferric-reducing antioxidant power showed a linear relationship with the TPC yield, as expected [107].

3.8. Enzyme Assisted Extraction

EAE is a novel extraction method that is catalyzed by enzymes under an optimal process condition. The working principle of EAE involves the hydrolysis of plant cells, which disrupts cell walls and facilitates the release of intracellular components such as polyphenols [133]. During the EAE, the active sites of the enzyme will bind to the cell wall. The enzymes would change their shapes to match their active sites with the substrate, followed by the bond breakage and rupturing of the cell matrix [134]. The selectivity and specificity of the product yield by the enzyme-assisted extraction are dependent on the specific characteristics and properties of the enzyme. For example, the use of pectinolytic enzymes has better polyphenol extraction efficiency [135]. Naturally, the yield of antioxidants through EAE is strongly affected by the types of enzymes used. Besides the main contributing factor, extraction efficiency is governed by the reaction temperature, pH of the system, enzyme concentration, operation time, and particle size of the substrate [134]. In the extraction of the phenolic compound from olive pomace, Macedo et al. [136] studied the extraction efficiencies of different enzyme formulations. Various cellulase, pectinase, and tannase formulations were tested to determine their effectiveness in extracting total phenolic content (TPC). The results showed that the combination of pectinase and cellulase (P + C) yielded the highest TPC extraction, followed by pure tannase, the combination of all three enzymes (C + P + T), pure pectinase, and pure cellulase. The lower extraction efficiency observed with pure cellulase may be attributed to its less effective hydrolytic activity, while the higher efficiency of the other formulations is likely due to the significant pectic and tannic content in olive pomace. Additionally, TPC increased from 38 to 81 mg QE/100 g when the temperature was raised from 25°C to 60°C.

In a study conducted by Islam et al. [108], several factors were examined on the effects of phenolic compound extraction from banana peel. It was found that the increase in enzyme concentration increased the extraction efficiency of TPC, except for pectinase, which had a negative correlation. They reported a slight increase in TPC and TFC with the increase in temperature from 35°C to 60°C. The slight increase in extraction yield is attributed to higher enzymatic activities that break down the cellular structure of the banana peel. Moreover, the TPC and TFC yield were found to increase with the extraction time. The positive correlation was similar to the one reported by Granato et al. [110]. However, a prolonged extraction time of >9 hours decreased the extraction yield of TPC and TFC due to the degradation and disintegration of bioactive compounds [108]. Moreover, the TPC and TFC extraction yield increased as the sample-to-liquid ratio decreased from 1:5 to 1:25. The increase is attributed to the enhanced solubility and diffusion of the bioactive compound when in contact with more solvent. Nevertheless, the increase in sample-to-liquid ratio was deemed excessive as the solute in the cell matrix and the unsaturated solution had a solubility limit lower than the polyphenols [137]. Lastly, Islam et al. also reported a positive correlation between the antioxidant activity (examined with DPPH and ABTS assay) and the increase of factors discussed above [108]. All in all, researchers should consider various factors when selecting the enzymes and process conditions so that they are best suited for antioxidant extraction from different sources, as the performance may differ from one to another.

3.9. Comparison of Different Extraction Methods

There are various antioxidant extraction methods available for selection. Different extraction methods are studied and compared by researchers across the years to determine the optimal performance of the extraction from different sources. Comparing the efficiency of conventional solvent extraction with UAE, Karadbhajne et al. [138] found that the UAE yielded the highest TPC. Despite a longer extraction time (48 hours) with constant stirring compared to the UAE (5 – 20 minutes of sonication), the conventional method showed a comparatively lower metal chelating activity and DPPH scavenging activity than the UAE. Lin et al. [139] compared various conventional methods with the green advance techniques when extracting flavonoids from India *Moringa oleifer*a L. leaves. With the optimized UAE parameters, it is found that the UAE performed the best in terms of TFC yield and antioxidant activities, followed by MAE, stirring-assisted extraction, and Soxhlet-assisted extraction. The author reported that an increase in operation temperature above 30°C resulted in a drop in TFC and antioxidant performance due to degradation. It is reported that the Soxhlet extraction had the lowest performance as it was performed at a high temperature (90°C). The MAE, on the other hand, had a slightly lower performance as the operating principle of MAE involves heating through electromagnetic waves, which resulted in lower TFC yield. Another study by Cheng et al. [140] reported a higher TPC yield and antioxidant activity of extracts from jackfruit (*Artocarpus heterophyllus* Lam.) pulp when MAE was carried out. In this case, the MAE performed better than the UAE due to the facilitation of antioxidant diffusion due to higher heat energy applied during the extraction process. Variations in findings among researchers are likely due to differences in the phenolic composition and stability of the materials studied [141].

In the antioxidant extraction from olive (*Olea europaea* L.) leaves, Dobrinčić et al. [141] reported the highest yield and antioxidant activity achieved by UAE and MAE, followed by PLE and conventional extraction. The performance of UAE and MAE showed no significant difference. However, the lower performance from PLE is due to the increased pressure, causing the solvent viscosity to increase and hindering its mass transfer ability (as discussed in Section 3.4.). Generally, the conventional solvent extraction method is reported to have lower extraction efficiency compared to other green extraction techniques. However, there are some cases whereby the conventional solvent extraction yielded a comparable result in antioxidant extraction and performance efficiency. Casquete et al. [142] made the performance comparison between the conventional agitation extraction, UAE, and SFE in the extraction of red grape stalk (wine-making by-product). It is reported that the DPPH and ABTS activity is marginally better than that of UAE under the same operating conditions (4AE and 4UE, which both produced the highest TPC yield) despite having slightly lower overall yield and TPC yield. In their study, SFE performed significantly lower in antioxidant yield and activity. However, SFE was reported to have better efficiency in the extraction of bioactive compounds with high antihypertensive activities. Notably, the extraction of antioxidants using the conventional method may be sufficient depending on different feedstock. A similar result was reported by Zhang et al. [143] in the extraction of different walnut by-products (walnut pellicle, *Diaphragma juglandis* fructus, *Juglans regia* flower) in which the bioactive compound yield and antioxidant activity from the condensation reflux method was the highest. Nevertheless, the authors reported that the addition of cellulase for EAE resulted in the best performance for both walnut pellicle and *Diaphragma juglandis* fructus. The better performance compared with conventional solvents used in UAE can be attributed to the enzyme that could readily break down the lignocellulosic content.

Besides the standalone extraction techniques, many researchers studied more advanced methods by combining different extraction technologies. For example, Sharif et al. [109] combined ultrasonication with EAE (UEAE) during the extraction of Mango (*Mangifera indica* L.) peels. It is reported that the UEAE performed better than the EAE. The UEAE facilitated the extraction efficiency as the added sonication increased the contact surface area of the sample and solvent, which allowed a higher release of bound phenolics due to the degradation of the cell matrix through enzymatic reactions. On top of that, the utilization of ultrasonication may potentially cut short the operation time when compared to the standalone EAE (110 minutes as opposed to 4 and 9 hours), as shown in Table 2. Studies and comparison of performance for MAE and PEF+MAE techniques were carried out on coffee pulps by Macías-Garbett et al., and it was found that the combination of PEF and MAE increased 14.58 – 23.59% TPC yield and 5.76 – 8.48% DPPH activity when compared to MAE alone [144]. Aside from that, researchers may study the utilization of deep eutectic solvents more as they are a recently emerged green extraction technique. The utilization of deep eutectic solvent may be the way forward for advancing antioxidant extraction, given its flexibility, selectivity, and benign environmental effects, as discussed in Section 3.6. In the extraction of bioactive compounds from *Moringa oleifera* L. leaves, Wu et al. [145] reported a huge improvement in TPC and TFC yield and its antioxidant activities (DPPH, ABTS, OH^-, and FRAP) when the antioxidants extraction was performed with DES + UAE. As opposed to the usage of conventional solvent reported in their study, in which TPC and TFC yield are usually one higher than the other due to the difference in the polarity of bioactive compounds, the use of UAE resulted in an equally high extraction efficiency of TPC and TFC yield. This can be ascribed to the tunable solubility of DES, which is a promising factor for the multi-targeted extraction of bioactive compounds. Besides, DES can also couple with MAE for a better antioxidant extraction yield as compared to the conventional stirring-heating extraction and Soxhlet extraction technique [129]. With the help of MAE, not only the extraction yield can be enhanced, but the time requirement compared to the stirring-heating extraction and Soxhlet extraction techniques were significantly reduced by 9 times and 56 times, respectively; hence the improved productivity and extraction yield.

Overall, the extraction and efficacy of antioxidants can vary based on the type of raw material used as feedstock. Therefore, conventional extraction methods may still be considered viable due to their established reliability, simplicity in operation, fewer operating variables, and the lack of need for costly additional equipment. Table 3 outlines the general advantages and disadvantages of various extraction methods. Based on the yield, total phenolic content, and antioxidant activity, MAE appears to be the most efficient method overall. It provides a good balance of high yield, high total phenolic content, and high antioxidant activity. In terms of the cost-effectiveness of the extraction methods, Oke et al. [146] studied the techno-economics of MAE of phytochemicals from garlic powder. It is reported that the cost of manufacturing (COM) declined as the manufacturing volume was upscaled from 5 L (1480 US$/kg) to 500 L (56 US$/kg). The garlic extract production at the highest scale has a net present value (NPV) of US$ 38,133, return of investment (ROI) of 34% and payback time (PBT) of 2.94 years. The process is economically feasible due to the high and positive NPV and a PBT of 2 to 3 years (typical for small-scale plants).

UAE is also very efficient in terms of phytochemical extraction. In a techno-economic analysis carried out by Ochoa et al. [147], the extraction of anthocyanins from purple yam was reported to be economically feasible in a 500 L extraction scale as compared to the conventional Soxhlet extraction and low-pressure solvent extraction. The UAE extraction has the lowest COM of US$ 124.08 per kg and positive NPV as opposed to the conventional Soxhlet extraction and low-pressure solvent extraction, which have negative NPV even at the highest selling price of US$ 250 per kg. The conventional extraction method is not economically feasible due to its low productivity, longer extraction time, and higher solvent consumption [147]. Through life cycle assessment (LCA), it was reported that the environmental impact of antioxidant extraction from chicory grounds could be reduced by 25% with the help of the UAE instead of conventional solvent extraction. The improvement in environmental friendliness is attributed to the decreased extraction time and energy consumption of UAE, making the conventional extraction method undesirable [148]. When comparing MAE and UAE, Bouchez et al. [149] conducted LCA to extract polyphenols from sugar beet seeds. It was found that MAE offers a higher polyphenol concentration and antioxidant activity (33% and 23% increment, respectively) with a short processing time (by a factor of around 6). Conversely, UAE slightly compromises the TPC yield and antioxidant activity but provides a greener operation in terms of energy consumption (3.6-fold lower), occupational health (35% lower ionizing radiation), and climate change (12% lower). Therefore, the choice between MAE and UAE might depend on specific requirements, such as the importance of antioxidant yield and activity versus operational cost, occupational health, and environmental impact.

In terms of cost-effectiveness, UAE and DESE generally strike a good balance between equipment costs, operational costs, and scalability. UAE offers faster extraction times and reduces solvent use, making it suitable for medium to large scales. DESE uses inexpensive solvents and moderate equipment costs, making it versatile and cost-effective for various applications. However, it is noteworthy that the use of DESE, despite having a higher TPC yield compared to conventional solvents (e.g. 20% ethanol and water), does not outweigh the negative environmental effects it brings. The contributing factors of DES environmental damages stem from the DES preparation and absorption stage due to resins and dimethyl hexanediol use. To make DES more preferable and viable in an upscaled production, the TPC extraction efficiency should be greatly improved, in which less resin is needed per mL of extract during absorption. Furthermore, the DES recovery and the resin reuse cycle should also be extended to minimize the environmental impact [150].

PLE and SFE offer high efficiency but come with higher equipment and operational costs, making them suitable for high-value products. This is evident as the capital expenditure and the operating expenses for a PLE plant for acai (*Euterpe oleracea*) by-product-derived antioxidant with a capacity of 2 × 500 L is estimated to be around 15.2 million US$ and 6.5 million US$/year [151]. However, at a COM of 16 US$/kg and a selling price as high as 50 US$/kg, the production is deemed economically feasible due to sustainable ROI (63.87%) and short PBT (1.6 years). In an LCA of beet waste antioxidant extraction, it was reported that PLE and SFE have the lowest environmental impact compared to Soxhlet extraction, maceration, and UAE, hence the most promising technology for upscaling [152]. Although both technologies are similar in terms of yield and environmental impact, the selection can be justified according to different feedstock requirements as different feed characteristics may be more favourable towards one or the other method. For example, the production of antioxidants from beet leaves using SFE (1964.65 kg/year) is higher than PLE (1397.96 kg/year), and vice versa for antioxidant production from beet stalk [152]. PEFE and EAE are effective for specific applications but require significant investment in equipment and reagents. The LCA for *Moringa oleifera* leaves polyphenol extraction showed that the environmental impact for PEF is the lowest, followed by MAE, maceration, and UAE [153]. The better performance of PEF and MAE is attributed to the reduced electricity consumption. Similarly, Jara-Quijada et al. [154] validated the use of PEF as a sustainable technology as the LCA presented the lowest emissions (0.006 kg $CO_{2\ eq}$) compared to MAE, UAE, and maceration. Nonetheless, the PEF should be further evaluated in terms of its scalability, economic feasibility, and environmental impact.

Table 3. Advantages and disadvantages of different extraction methods.

Extraction Method	Advantages	Disadvantages	Reference
Conventional solvent extraction	• Simple method • Well-known technique	• Prolonged reaction time • Require further separation steps	[113, 155]
UAE	• High mass transfer rate • High solvent penetration rate • Cost effective • High recovery	• Requires specific equipment • May degrade carotenoids and other unstable compounds • Expensive upscaling • High energy consumption	[95, 155]
MAE	• Short processing time • High recovery	• Degradation of thermo-labile compounds • High equipment cost	[119, 156]
PLE	• Lesser solvent required • Lesser downstream process	• Degradation of thermo-labile compounds	[100, 113]

		• May degrade chemical structure and functional activities • Expensive upscaling	
SFE	• Higher extraction efficiency of chemical constituents	• Requires expensive equipment • The extraction fluid is predominantly non-polar (CO_2)	[112, 124]
DESE	• High solvent recovery • Higher extraction yield of TPC compared to conventional solvent • Tunable selectivity	• Requires further separation/ filtration steps • High viscosity compared to conventional solvent	[127, 156, 157]
PEFE	• Able to prevent decomposition of thermos-labile compounds • Less power demanding	• Dependent on the medium's conductivity • Intricate design and difficult optimization for full potential	[107, 156]
EAE	• Less power demanding • Higher extraction yield • Environmentally friendly	• Long extraction time • High cost	[112, 134]

4. Biolubricant Feedstock

As the demand for environmental sustainability and green technology increases, the usage of mineral oil-based lubricants becomes a concern in various industries involving processing and manufacturing. Conventional mineral oil-based lubricants are synthesized from crude oil. These lubricants have various additives such as acrylates, zinc dialkyl dithiophosphate (ZDDP), and molybdenum disulfide (MoS_2) are added to them to improve their physical and chemical properties or for performance enhancement (anti-wear, extreme pressure, and anti-corrosion application) [158-160]. In addition to the toxicity and low biodegradability of the mineral-based lubricants, the sulfur and heavy metal-containing additives may facilitate the production of acidic substances during high temperatures and extended working conditions [161]. This will lead to acidic sludge formation, which will cause corrosion to machinery, clogging of machine filters, and a decrease in machinery life [161, 162]. Besides, the improper disposal of mineral oil-based lubricants may inhibit the growth of plant species due to heavy metal content and decrease oxygen content in the water bodies due to the formation of low-density organic films [163].

With the realization of adverse environmental effects brought by conventional lubricants and the help of governmental bodies encouraging the use of greener substitutes through subsidies, the lubricant market and its research have been increasing over the years. This is reflected in the increase in publications on biolubricant research, as shown in Figure 6. The increase in the research interest also signifies the potential of lubricants in replacing conventional lubricants in the industries, as many had found vegetable-based lubricants to have superior physicochemical properties than mineral-based lubricants in terms of flash point, viscosity, VI, and volatility [164, 165]. The downsides of using vegetable-based lubricants are their high pour point and poor thermal-oxidative stability, which are attributed to their natural triglyceride backbone structures and the presence of unsaturated fatty acids [3, 164]. Those mentioned above can negatively affect the cold flow properties and the oxidative stability of the biolubricant due to the formation of macro crystalline structures at lower temperatures and the degradation of lubricant quality caused by exposure light, humidity, heat, and impurities, respectively [166]. To make vegetable-based lubricant a competitive lubricant in the market, chemical modifications or the addition of additives can be carried out. Several chemical modifications can be carried out to improve the properties of the biolubricant, namely esterification/transesterification, epoxidation, selective hydrogenation, estolide, and hydrolysis [167]. Table 4 shows the advantages and disadvantages of different chemical modifications.

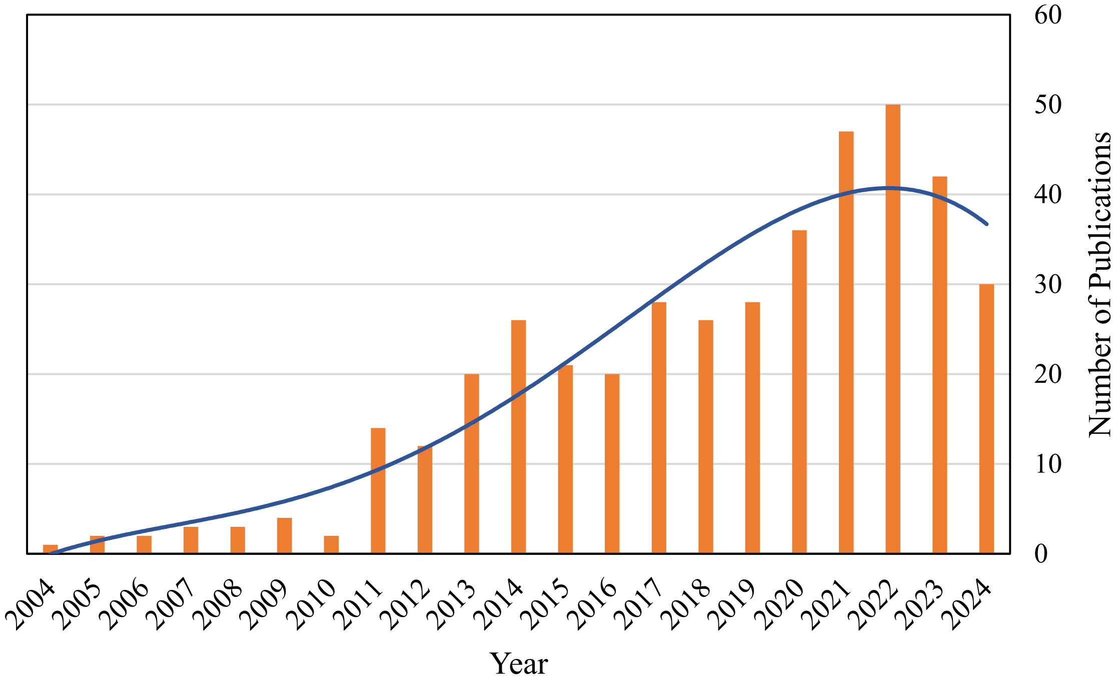


Figure 6. Bibliometric trends for lubricant research. Search algorithms on Scopus: (TITLE-ABS-KEY (bio-lubricant OR biolubricant AND vegetable oil OR crop oil) AND PUBYEAR > 2003 AND PUBYEAR < 2025).

Table 4. Various chemical modifications for biolubricant production of vegetable/crop oil and their respective advantages and disadvantages.

Chemical Modification	**Synthesis Method**	**Advantages**	**Disadvantages**	**Reference**
Esterification / transesterification	**Acid catalyzed.** Conversion of vegetable oil to fatty acid alkyl ester by reaction with various alcohols in the presence of acid catalyst.	• High yield • Enhanced oxidative stability • Improved temperature performance • Flexible feedstock selection (mostly used for feed stock with high FFA content) • Improved lubricity	• Slower reaction • High cost due to expensive acid-resistant equipment required	[168-170]
	Base catalyzed. Conversion of vegetable oil to fatty acid alkyl ester by reaction with various alcohols in the presence of alkali catalyst.	• Enhanced oxidative stability • Improved low-temperature performance • Increased VI	• Stringent molar ratio of alcohol to oil ratio	[169, 171]
Epoxidation	Introduction of an epoxide functional group (cyclic ether) through the reaction of unsaturated fatty acid with oxygen atoms (such as using H_2O_2).	• Increased lubricity • Increased oxidative stability	• Decreased VI	[172]
Selective hydrogenation	Introduction of hydrogen to the vegetable oil to react with the C=C bond and transform the unsaturated fatty acid into a single fatty acid.	• Reduce the degree of unsaturation • Increased oxidative stability	• High temperature reaction (250 – 300 °C) • Decrease in cold flow properties	[173]
Estolide formation	Carbocation reaction in which a carboxylic acid group of a fatty acid is linked to the site of another unsaturated vegetable oil (triacylglycerol)/fatty acid to form oligomeric esters.	• Decreased pour point and improved cold flow properties • Increased VI • Increased resistance to hydrolytic degradation • Improved oxidative stability	• Decreased pour point and improved cold flow properties • Increased VI • Improved oxidative stability	[173, 174]
Enzymatic Hydrolysis	A two-step process which (1) introduces water and a suitable enzyme/lipase to obtain free fatty acids, and (2) carries out esterification to obtain ester and glycerol.	• High activity in mild conditions • High selectivity	• High cost to maintain consistent enzymatic activity • Difficult enzyme recovery	[175]

4.1. Edible and Non-edible Vegetable Oil-based Biolubricant

There are 2 classifications of vegetable oil, which are edible and non-edible. Non-edible oil usually contains a certain amount of toxic components that are not safe for human consumption. However, they have a wide range of applications, such as oil-illuminant, surface coating, biodiesel, and cosmetics [176]. Non-edible crop oils are generally preferred over edible crop oils in biolubricant production due to their environmental adaptability, food security, and overall sustainability, as they will not create an issue in the land competition with edible oils and in the demand for edible oil in the consumer market and the industrial market. As compared to mineral oil-based lubricants, vegetable/crop oil-based lubricants have a superior lubrication performance in high-temperature applications due to their high oxidative degradation temperature. For example, coconut oil, sesame oil, and sunflower oil have an oxidative degradation onset temperature of 241℃, 246℃, and 257℃, respectively [177]. Vegetable oils also have a considerably high flash point as compared to mineral oil-based lubricants (Table 5). Generally, edible vegetable oil has a higher flash point, which is averaged at 268℃, while the average flash point of non-edible oil is around 229℃. Despite the high flash point, vegetable/crop oils are not suitable to be used directly without any chemical modifications due to their high degree of unsaturation (C=C double bond), which is very susceptible to oxidative/hydrolytic degradation due to their reactive nature to atmosphere oxygen, breaking down the ester molecules into acid and olefins [178].

Table 5 shows the pour point of each vegetable oil. The pour point temperature is directly correlated with the fatty acid composition. Vegetable oils with high saturated fatty acid content result in a high pour point. As the temperature decreases, the saturated fatty acids tend to bundle together rapidly due to their high degree of linearity [179]. This facilitates the formation of closely packed crystalline structures, which reduce the flowability of the vegetable oils. This phenomenon is apparent in coconut oil, which has the highest level of saturation and pour point of 96% and 23°C, respectively. The high pour point decreases the versatility of vegetable oils in extremely low-temperature applications due to their poor flow characteristics, as they will not be pumpable for the equipment in operation [180]. On the other hand, vegetable oils with higher levels of unsaturation will have a lower pour point. They are more resistant to change at lower temperatures due to the restricted rotation around the C=C bonds, making the molecular structure more rigid and slightly bent at an angle and causing the molecules not to be readily stacked [181].

Table 5. Fatty acid composition, pour point, and flash point of different edible vegetable oils.

Vegetable Oils	**Composition of Fatty Acids (%)**			**Pour Point (°C)**	**Flash Point (°C)**	**References**
	Saturated	**Mono-unsaturated**	**Poly-unsaturated**			
Edible Oil						
Soybean	16.9	23.6	59.5	-16	300	[182, 183]
Palm (RBD)	54.7	37.1	8.1	9	250 – 260	[182, 184]
Rapeseed/ Canola	10.1	55.6	29.4	-13	288	[185, 186]
Groundnut	22.5	50.1	27.4	3	>280	[182, 187]
Olive	17.2	73.7	9.1	-10	227	[182, 188]
Sunflower	11.9	35.0	53.1	-8.7	315	[182, 189]
Grapeseed	17.6	26.75	55.65	-4	250.7	[190]
Corn	15.9	27.5	56.7	-26	280	[182, 191]
Coconut	96.0	3.3	0.7	23	188	[177, 182, 186]
Sesame	19.1	40.6	40.3	-6	245	[182, 192, 193]
Rice bran	22.02	41.38	36.47	-8	316	[194-196]
Non-edible Oil						
Moringa	22.6	75.8	1.0	4	268.5	[185, 197]
Jatropha	34.5	15.0	49.8	-11	224	[185, 186]
Cottonseed	29.7	18.5	50.8	1	252	[185, 198]
Castor	2.5	89.7	7.8	-2	250	[183, 199]
Karanja	26.1	52.6	18.6	3	252	[195, 200, 201]
Mahua	48.5	37.5	14.3	2 – 3	212	[202, 203]
Neem	37.0	44.5	18.5	7.0	151	[204, 205]
Linseed	11.01	18.51	70.46	-15	220	[206, 207]

Besides the pour point and flash point, many other properties are important in indicating the performance of lubricating oils, namely, viscosity, VI, acid value, peroxide value, iodine value, thermal onset temperature (TOT), oxidative onset temperature (OOT), and oxidation induction time (OIT). Table 5 presents the viscosity index (VI) for different vegetable oils, highlighting their significantly higher VI compared to mineral oils. This superior VI indicates enhanced performance in high-temperature applications, as vegetable oils exhibit a less pronounced viscosity reduction with rising temperatures [208]. Furthermore, higher viscosity at elevated temperatures improves lubricating film formation and film thickness at frictional interfaces, minimizing performance losses [209]. The oxidative stability of vegetable oils is often assessed using the iodine value, which reflects the amount of double bonds present. Higher iodine values correspond to increased double bond content, leading to poorer oxidative stability due to the lower dissociation energy of allylic carbons. This relationship is evident when comparing the fatty acid composition of vegetable oils in Table 5 with their respective iodine value in Table 6. Generally, vegetable oils have a higher unsaturated fatty acid content, which resulted in a high iodine value of >55.2 g I_2/100 g, except coconut oil, which has a low iodine value of 9.37 g I_2/100 g. Acid values and peroxide values evaluate the concentration of acidic compounds and the concentration of peroxides in the vegetable oil, respectively.

Biolubricants with high acid and peroxide values are highly undesirable as the former would result in an acidic environment that promotes rusting. At the same time, the latter would facilitate the oil oxidation process, which would further deteriorate the quality and performance of the biolubricant by producing more acidic by-products and sludge [162]. Furthermore, the thermo-oxidative stability of vegetable oil can be gauged through TOT, OOT, and OIT. The TOT of vegetable oils is measured under an increasing temperature and in an inert environment, such as under a constant flow of N_2 gas, where the only factor of degradation is temperature. Conversely, the OOT was performed under a constant flow of oxidizing gas of either O_2 or air. The higher the TOT and OOT, the higher the thermal and oxidative stability. Table 6 shows that TOT is generally higher than the OOT as the oxidizing agents are not introduced during the testing. When the oxidizing gas is introduced, the onset temperature decreases significantly. For example, the onset temperature of soybean oil and sunflower oil dropped from 371℃ to 173.10℃ and from 342℃ to 165.81℃, which is around 53% and 52%, respectively. This asserted the claim that vegetable oils have thermal stability and poor oxidative stability. Based on the data presented in Table 6, most OOT tests were conducted using a constant flow of pure O_2 gas. However, the author recommends that OOT testing be performed using a constant airflow to replicate real-world conditions better. In cases where air was used, such as with Moringa oil and Jatropha oil, the percentage decrease in onset temperature was slightly lower (approximately 45% and 48%, respectively). This reduction may be attributed to the lower oxygen concentration in the air compared to pure O_2. While the differences observed in other studies may not be substantial, using airflow in OOT testing is likely to provide a more accurate assessment of the oxidative stability of biolubricants under actual environmental conditions.

OIT is also another parameter that assesses the oxidative stability of vegetable oil whereby the time interval between the beginning of the O_2/airflow and the initiation of the oxidation reaction is determined. However, the method requires an adequate selection of temperature range, or else it would result in an inaccurate representation of the oxidative stability [210]. In general, vegetable oils have low oxidative stability; therefore, chemical modifications should be applied to improve the overall properties. The tribological performance of vegetable oils is evaluated with coefficient of friction (COF) and wear scar diameter (WSD). While the data collected for each of the vegetable oils are vastly different, Sharma et al. (2009) found that the WSD of vegetable oils are in the descending order of cottonseed oil > moringa oil > sunflower oil > canola oil > jatropha oil. Jatropha oil had the lowest WSD possible due to its higher free fatty acid (FFA) content. It was also speculated that the COF is related to their fatty acid composition, in which a higher saturated fatty acid content and a lower unsaturated fatty acid content would lead to higher COF and vice versa [185].

Table 6. Physicochemical properties, oxidative performance and tribological performance of various vegetable oils.

Vegetable oil	**Viscosity (cSt)**		**Viscosity Index**	**Acid value (mg KOH g^{-1})**	**Peroxide value (meq. kg^{-1})**	**Iodine value (g I_2 / 100 g)**	**Onset Temp. (°C)**	**Oxidative Induction Time (min)**	**COF**	**WSD (µm)**	**Reference**
	40 °C	**100 °C**									
***Edible* oil**											
Soybean oil	32.93	8.08	233.608	0.16	9.76	120 – 143	371 [1] 173.10 [2]	20 (140°C)	0.4059	ND	[211-215]
Crude Palm oil	ND	ND	ND	ND	7.30 mol/kg (un-bleached)	77 (un-bleached)	347.57 [1]	ND	0.133	ND	[216-218]
RBD Palm Olein	35.78	9.22	255	4.60 (ASTM D664)	1.09 mol/kg (Bleached palm oil)	55.2 (Bleached palm oil)	179 [2]	14 (RPVOT), 82.36 (140°C)	ND	ND	[214, 218-220]
Rapeseed/ Canola	34.9	8.1	219	0.17 – 0.33	1.3	99	164 [2] 339 [1]	60.28 – 67.05, 3.4 h [3], 37.41 (140°C)	0.076	149	[185, 214, 221]
Groundnut/ Peanut	44.9	11.26	255.274	3.402 – 3.446	8.5	82 – 106	378 [1]	12.33 (140°C)	0.0975	824	[213, 214, 222, 223]
Olive	ND	ND	ND	1.571	8.5	86.4	178.65 – 192.28 [2] 377 [1]	13.94 (140°C)	0.0778		[212, 214, 224]
Sunflower	36.8	8.5	218	1.941	2.5	130	165.81 [2] 342 [1]	1.1 h [3], 19.98 (140°C)	0.069	151	[185, 212, 214, 224]
Grapeseed	26.92	ND	ND	0.30 – 0.39	10.6	141	ND	7.53 (140°C)	ND	ND	[190, 214]
Corn	30.8	6.57	176	0.22 – 0.25	3.93	129	175.79 [2] 372 [1]	21.45 (140°C)	0.4458	ND	[212-214, 225]
Coconut	27.6	5.9	166	ND	1.72	9.37	241 [2] 257 [1]	44.86 (140°C)	ND	ND	[177, 214]
Sesame	25.78	ND	ND	ND	1.13	109	246 [2] 370 [1]	69.55 (140°C)	0.3184	ND	[177, 193, 213, 214]
Rice bran	41.13	5.19	ND	1.13	12.5	97.08	ND	10.11 (RPVOT)	ND	591	[194, 195]
Non-edible oil											
Moringa	27.1	7.0	239	ND	1.65	70	347 [1] 191 [2]	15.3 h [3]	0.092	156	[185]
Jatropha	34.6	8.0	213	ND	4.26	107	322 [1] 169 [2]	2.6 h [3]	0.096	146	[185]

Cottonseed	34.3	8.0	216	ND	1.12	111	343 [1] 159 [2]	1.9 h [3]	0.097	202	[185]
Karanja	43.39	6.93	117.176	0.4 – 12	6.8	86.5	ND	13.40 (RPVOT)	ND	585	[195, 200]
Neem	54.37	10.39	134	32.583	ND	81.28	ND	ND	0.1167	1,486	[204, 205, 226]
Linseed	ND	ND	262.5	0.8	0.95	177	177 [1] 186 [2]	ND	ND	ND	[206, 227, 228]

Remarks:
ND – no data
[1] TOT
[2] OOT
[3] Oxidative stability index (OSI), Rancimat test method

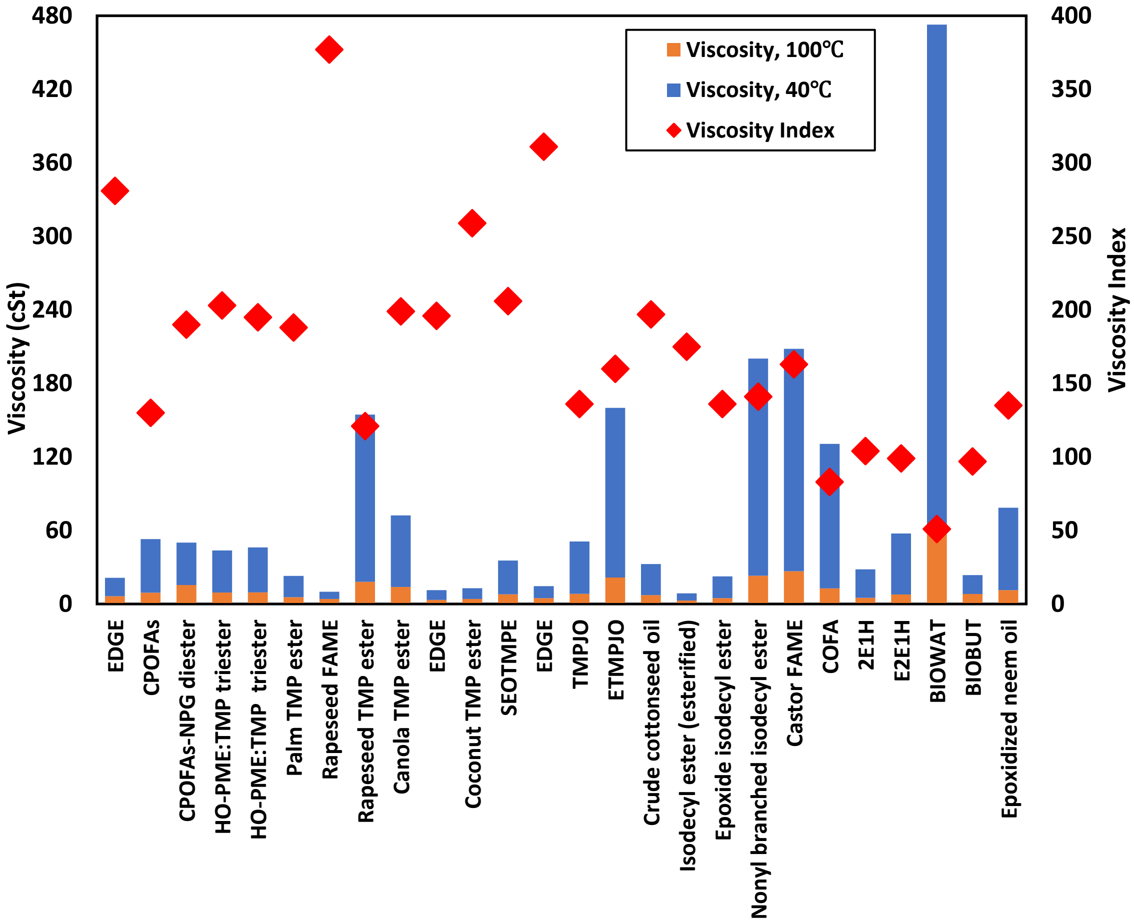
Esterification/transesterification is more widely adopted as the chemical modification of vegetable oils to enhance their biolubricant potential. Through transesterification, triglycerides in vegetable oil are converted into diglycerides, monoglycerides, and glycerol. At optimum conditions, fatty acid methyl esters (FAME) are produced. The FAME obtained from the first transesterification is normally used as a feedstock to react with polyhydric alcohols such as trimethylolpropane (TMP), EG, neopentyl glycol, and pentaerythritol in a second transesterification reaction [229]. For example, Attia et al. [230] synthesized ethylene glycol di-esters (EDGE) through a two-stage transesterification of soybean oil. The soybean biolubricant was found to have a pour point of -6°C and a thermal stability of up to 200°C. The VI of the soybean EDGE is 281, which is around a 20.3% increase from the crude soybean oil. The soybean EDGE behaves as a Newtonian fluid, which allows it to be adopted as a hydraulic lubrication oil.

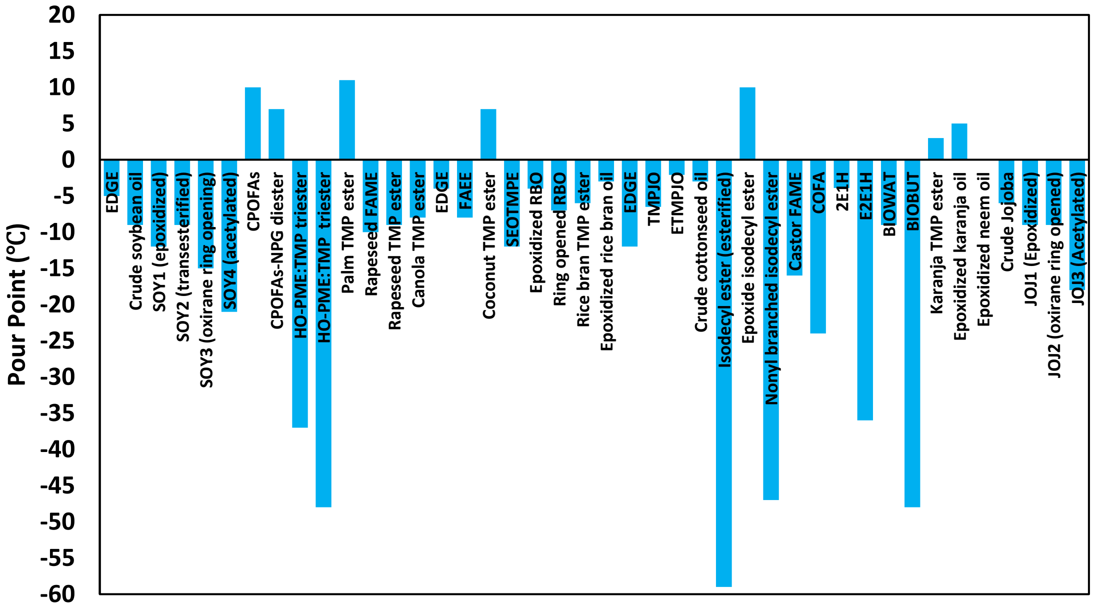
In addition to the transesterification reaction, various chemical modifications and the properties of the resulting biolubricants are summarized in Table 7. The comparison of properties such as viscosity, VI, pour point, and flash point for different vegetable-based biolubricants is shown in Figure 7. Thampi et al. [231] studied the tribological performance of epoxidized and ring-opened rice bran oil. The epoxidation process was optimized to produce an intermediate lubricant (epoxidized rice bran oil) with a reduced VI of 58.33 g I_2/100 g, which is about 40% lower than that of unmodified rice bran oil (Table 6), indicating a decrease in unsaturation. The reaction with H_2O_2 removes the double bond in the triglycerides, forming an epoxide ring. The tribological performance improved as the coefficient of friction (COF) and wear scar diameter (WSD) of epoxidized rice bran oil were lower than those of pure rice bran oil. This improvement is attributed to cross-linking of the epoxide ring on the metal surface, reducing friction. However, the epoxidation process increased the oil's pour point, likely due to the reduction in double bonds, making the molecular structure more linear. When the epoxidized rice bran oil underwent oxirane ring opening, it showed significant improvement in cold flow properties, with a pour point of -21°C. The attachment of aliphatic alcohol to the oxirane ring increased steric hindrance, lowering the pour point [231].

Similarly, Abdel-Hameed et al. [180] modified soybean oil through a sequence of epoxidation, transesterification, oxirane ring opening, and acetylation. The oxirane ring opening improved the flash point and cold flow properties of both soybean and jojoba oils. Acetylation of the oxirane ring opened oils further enhanced these properties, with pour points of -21°C for soybean oil and -18°C for jojoba oil. Additionally, acetylated soybean oil exhibited thermal stability up to 350°C. While the VI of acetylated soybean oil slightly decreased compared to the unmodified oil, it still met the ISO VG 100 grade. This modification also reduced the acid value (AV) and resulted in superior properties compared to commercial lubricants, making it suitable for a wide range of applications in the automotive, industrial, and metalworking sectors.

The selection and initial modification of vegetable oils are crucial for enhancing their physicochemical properties as biolubricants. He et al. [209] demonstrated this by using cottonseed oil, which contains over 70% unsaturated fatty acids, including more than 45% linoleic acid (18:2) [232], to convert raw oil into a biolubricant. In their study, cottonseed oil underwent a series of chemical modifications: hydrolysis, separation, esterification, epoxidation, and oxirane ring opening. Hydrolysis removed the triglyceride backbone, producing fatty acids, which were then separated and esterified with isodecyl alcohol. The ester underwent epoxidation followed by ring opening, but instead of using a short-chain alcohol, a medium-chain nonanoic acid was employed. This modification resulted in a biolubricant with a superior pour point of -47°C, attributed to the branched structure enabled by multiple reaction sites on the linoleic acid double bonds. The medium chain nonanoic acid also increased steric hindrance, preventing molecular stacking. In addition to its low pour point, the biolubricant exhibited excellent oxidative and thermal stability (307.21°C and 310.11°C, respectively) and superior tribological performance, with 1.5 times lower WSD and 100% oil film coverage. The high VI, low pour point, and excellent thermo-oxidative stability make this biolubricant suitable for industries requiring performance at both high and low temperatures [209].

Through chemical modifications, the tribological performance of the biolubricant can also be improved. Dodos et al. [192] synthesized sesame oil trimethylolpropane ester (SEOTMPE) as a biolubricant and the cold flow properties of sesame oil were improved as the pour point decreased from -6°C to -12°C. The tribological performance of SEOTMPE was evaluated against a conventional mineral oil-based lubricant. The reference mineral oil has a WSD of 328 μm and COF of 0.147. The SEOTMPE is found to have a 33.23% WSD reduction and 59.18% COF reduction. Similar results were reported by Menkiti et al. [233] regarding the improved cold flow properties.


Viscosity, 100℃
Viscosity, 40℃
Viscosity Index
Viscosity (cSt)
Viscosity Index
480
420
360
300
240
180
120
60
0
400
350
300
250
200
150
100
50
0
EDGE
CPOFAs
CPOFAs-NPG diester
HO-PME:TMP triester
HO-PME:TMP triester
Palm TMP ester
Rapeseed FAME
Rapeseed TMP ester
Canola TMP ester
EDGE
Coconut TMP ester
SEOTMPE
EDGE
TMPJO
ETMPJO
Crude cottonseed oil
Isodecyl ester (esterified)
Epoxide isodecyl ester
Nonyl branched isodecyl ester
Castor FAME
COFA
2E1H
E2E1H
BIOWAT
BIOBUT
Epoxidized neem oil


Pour Point (℃)
20
15
10
5
0
-5
-10
-15
-20
-25
-30
-35
-40
-45
-50
-55
-60
EDGE
Crude soybean oil
SOY1 (epoxidized)
SOY2 (transesterified)
SOY3 (oxirane ring opening)
SOY4 (acetylated)
CPOFAs
CPOFAs-NPG diester
HO-PME:TMP triester
HO-PME:TMP triester
Palm TMP ester
Rapeseed FAME
Rapeseed TMP ester
Canola TMP ester
EDGE
FAEE
Coconut TMP ester
SEOTMPE
Epoxidized RBO
Ring opened RBO
Rice bran TMP ester
Epoxidized rice bran oil
EDGE
TMPJO
ETMPJO
Crude cottonseed oil
Isodecyl ester (esterified)
Epoxide isodecyl ester
Nonyl branched isodecyl ester
Castor FAME
COFA
2E1H
E2E1H
BIOWAT
BIOBUT
Karanja TMP ester
Epoxidized karanja oil
Epoxidized neem oil
Crude Jojoba
JOJ1 (Epoxidized)
JOJ2 (oxirane ring opened)
JOJ3 (Acetylated)

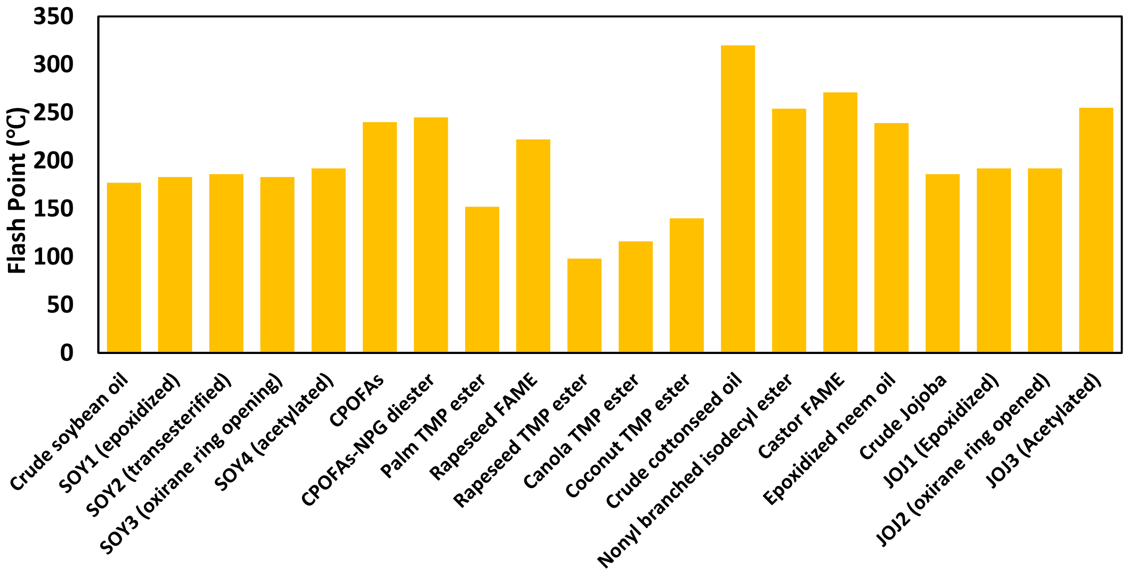


Figure 7. (a) Viscosity at 40°C and 100°C, viscosity index, (b) pour point, and (c) flash point of vegetable-based biolubricants.

Table 8 shows the tribological performance of different chemically modified vegetable oils. Not many have reported the tribological aspect of the chemically modified biolubricant, as more are focused on their physicochemical properties, such as viscosity, VI, pour point, and flash point. However, it is recommended that the tribological analysis be carried out so that it may serve as a baseline for future references.

Table 7. Physical properties and thermo-oxidative performance of various modified vegetable-based biolubricants.

Vegetable Oil	Chemical Modification	Biolubricant	Viscosity (cSt)		Viscosity Index	Pour Point (℃)	Flash Point (℃)	Onset Temperature (℃)	Oxidation Induction Time (min)	References
			@ 40℃	@ 100℃						
Edible Oil										
Soybean oil	Two-stage transesterification	EDGE	21.30	6.31	281	-5	ND	200 [1]	ND	[230]
Soybean oil	Epoxidation + transesterification + oxirane ring opening (hydroxylation) + acetylation	Crude	ND	ND	230	-9	177	ND	ND	[180]
		SOY_1 (epoxidized)	ND	ND	147	-12	183	ND	ND	
		SOY_2 (transesterified)	ND	ND	153	-9	186	ND	ND	
		SOY_3 (oxirane ring opening)	ND	ND	183	-15	183	ND	ND	
		SOY_4 (acetylated)	ND	ND	194	-21	192	350 [1]	ND	
Palm oil	Hydrolysis	CPOFAs	52.9	9.2	130	10	240	ND	ND	[234]
	Hydrolysis + esterification	CPOFAs-NPG diester	50.1	15.5	190	7	245	ND	ND	
	Enzymatic esterification	HO-PME:TMP triester	43.7	9.3	203	-37	ND	ND	41 (RPVOT)	[235]
	Enzymatic esterification	HO-PME:TMP triester	46.2	9.5	195	-48	ND	ND	ND	[236]
	Two-stage transesterification	Palm TMP ester	22.95	5.54	188	11	152	362 [1]	ND	[237]
Rapeseed/ canola oil	Two-stage transesterification	Rapeseed FAME biolubricant (AV 0.39 mg KOH/g)	10.04	4.09	377	-10	222	ND	0.94 h [3]	[238]
	Two-stage transesterification	Rapeseed TMP ester	154.50	18.03	121	-9	98	401 [1]	ND	[237]
Olive oil	Two-stage transesterification	Canola TMP ester	72.29	13.85	199	-8	116	384 [1]	ND	[237]
Sunflower oil	Transesterification	EDGE	11.24	3.35	196	-4	ND	200 [1]	ND	[230]
Grapeseed oil	Transesterification	FAEE	4.32	ND	ND	-8	ND	ND	2.01 h [3]	[239]
Coconut oil	Two-stage transesterification	Coconut TMP ester	12.87	4.12	259	7	140	345 [1]	ND	[237]
Sesame oil	Transesterification	SEOTMPE	35.43	7.93	206	-12	ND	ND	378 (@110℃)	[192, 233]

Rice bran oil	Epoxidation + ring opening	Epoxidized RBO	89.28	ND	ND	-4	ND	ND	ND	[231]
		Ring opened RBO	174.8		ND	-7	ND	ND	ND	
	Two-stage transesterification	Rice bran TMP ester	32.25	4.55	ND	-6	ND	ND	14.21 (RPVOT)	[195]
	Epoxidation	Epoxidized rice bran oil	93.51	14.94	ND	-3	ND	ND	22.47 (RPVOT)	
Non-edible oil										
Jatropha oil	Transesterification	EDGE	14.60	4.73	311	-12	ND	200 [1]	ND	[230]
	Esterification	TMPJO	51	8.3	136	-6.5	ND	ND	ND	[240]
	Esterification + epoxidation	ETMPJO	160	21.6	160	-2.1	ND	ND	ND	
Cottonseed oil	Hydrolysis + esterification + epoxidation + ring opening & branching	Crude (AV 5.88 mg/g)	32.55	7.25	197	-3	320	370.01 [1] 303.11 [2]	13	[209]
		Isodecyl ester (esterified) (AV 1.02 mg/g)	8.74	2.75	175	-59	ND	ND	ND	
		Epoxide isodecyl ester (AV 10.64 mg/g)	22.56	4.77	136	10	ND	ND	ND	
		Nonyl branched isodecyl ester (ring opening & branching) (AV 0.65 mg/g)	200.3	23	141	-47	254	310.11 [1] 307.21 [2]	21	
Castor oil	Two-stage transesterification	Castor FAME biolubricant (AV 0.45 mg KOH/g)	208.25	26.74	163	-16	271	ND	-	[238]
		COFA	130.7	12.76	83	-24	ND	ND	0.31 h [3]	[241]
	Esterification + Epoxidation + Oxirane ring opening	2E1H	28.25	5.03	104	-3.9	ND	ND	3.60 h [3]	
		E2E1H	57.49	7.79	99	-36	ND	ND	4.30 h [3]	
		BIOWAT	472.78	62.25	51	-9	ND	251.5 – 297.1 [1]	3.27 h [3]	
		BIOBUT	23.53	8.12	97	-48	ND	233 – 265.7 [1]	4.22 h [3]	
Karanja oil	Two-stage transesterification	Karanja TMP ester	33.33	4.76	ND	3	ND	ND	19.32 (RPVOT)	[195]
	Epoxidation	Epoxidized karanja oil	78.79	10.17	ND	5	ND	ND	21.17 (RPVOT)	

Neem oil	Epoxidation	Epoxidized neem oil	78.51	11.30	135	ND	239	ND	ND	[205]
Jojoba		Crude Jojoba	ND	ND	205	-6	186	ND	ND	[180]
	Epoxidation + Oxirane ring opening + Acetylation	JOJ$_1$ (Epoxidized)	ND	ND	109	-9	192	ND	ND	
		JOJ$_2$ (Oxirane ring opening)	ND	ND	155	-9	192	ND	ND	
		JOJ$_3$ (Acetylated)	ND	ND	167	-18	255	200	ND	

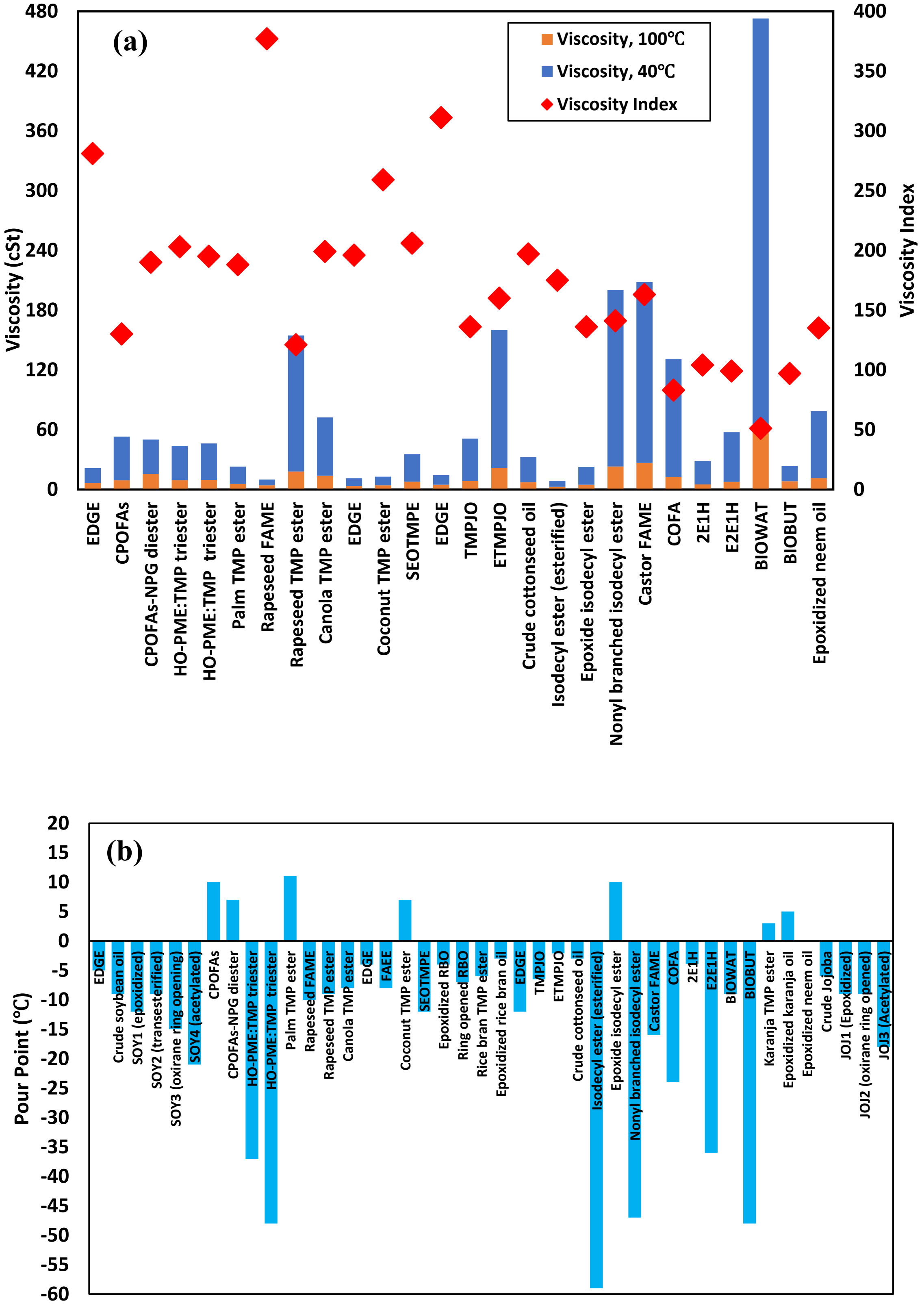
Remarks:

ND – no data

[1] TOT

[2] OOT

[3] Rancimat test method


(a)
Viscosity (cSt)
0
60
120
180
240
300
360
420
480
Viscosity Index
0
50
100
150
200
250
300
350
400
Viscosity, 100℃
Viscosity, 40℃
Viscosity Index
EDGE
CPOFAs
CPOFAs-NPG diester
HO-PME:TMP triester
HO-PME:TMP triester
Palm TMP ester
Rapeseed FAME
Rapeseed TMP ester
Canola TMP ester
EDGE
Coconut TMP ester
SEOTMPE
EDGE
TMPJO
ETMPJO
Crude cottonseed oil
Isodecyl ester (esterified)
Epoxide isodecyl ester
Nonyl branched isodecyl ester
Castor FAME
COFA
2E1H
E2E1H
BIOWAT
BIOBUT
Epoxidized neem oil
(b)
Pour Point (℃)
20
15
10
5
0
-5
-10
-15
-20
-25
-30
-35
-40
-45
-50
-55
-60
EDGE
Crude soybean oil
SOY1 (epoxidized)
SOY2 (transesterified)
SOY3 (oxirane ring opening)
SOY4 (acetylated)
CPOFAs
CPOFAs-NPG diester
HO-PME:TMP triester
HO-PME:TMP triester
Palm TMP ester
Rapeseed FAME
Rapeseed TMP ester
Canola TMP ester
EDGE
FAEE
Coconut TMP ester
SEOTMPE
Epoxidized RBO
Ring opened RBO
Rice bran TMP ester
Epoxidized rice bran oil
EDGE
TMPJO
ETMPJO
Crude cottonseed oil
Isodecyl ester (esterified)
Epoxide isodecyl ester
Nonyl branched isodecyl ester
Castor FAME
COFA
2E1H
E2E1H
BIOWAT
BIOBUT
Karanja TMP ester
Epoxidized karanja oil
Epoxidized neem oil
Crude Jojoba
JOJ1 (Epoxidized)
JOJ2 (oxirane ring opened)
JOJ3 (Acetylated)

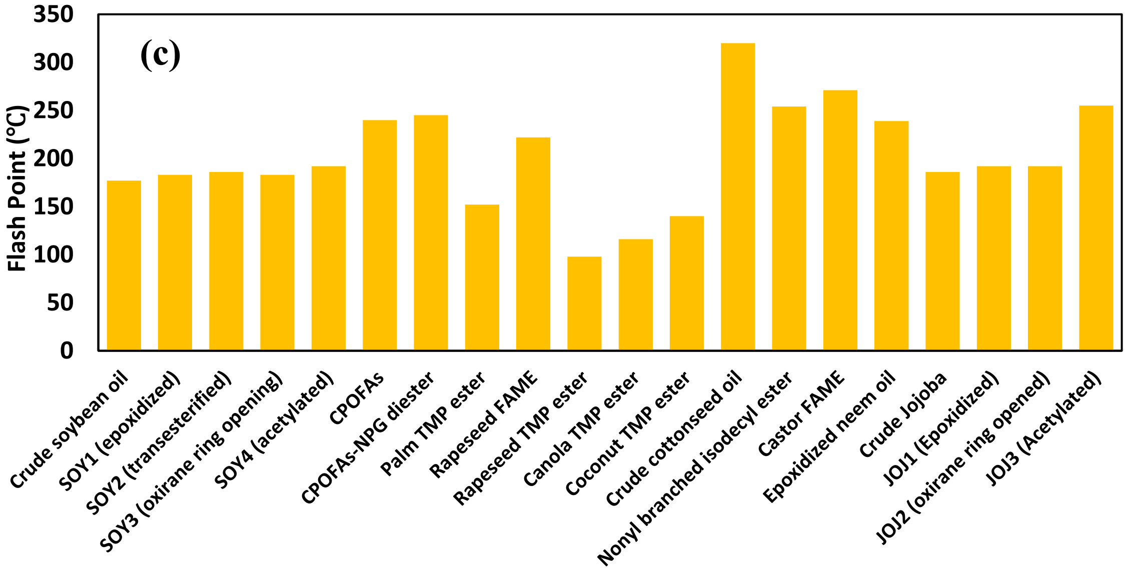


Figure 7. (a) Viscosity at 40°C and 100°C, viscosity index, (b) pour point, and (c) flash point of vegetable-based biolubricants.

Table 8. WSD and COF of various vegetable oils after chemical modifications

Vegetable Oil	Chemical Modification	Biolubricant	Wear Scar Diameter (µm)	Coefficient of Friction	References
Palm	Hydrolysis	CPOFAs	ND	0.2 (40°C) 0.24 (100°C)	[234]
	Hydrolysis + esterification	CPOFAs-NPGDE	ND	0.19 (40°C) 0.20 (100°C)	
Grapeseed	Transesterification	FAEE	163.47	ND	[239]
Sesame	Transesterification	SEOTMPE	219	0.060	[192]
Rice bran oil	Epoxidation + ring opening	Epoxidized RBO	547	0.047	[231]
		Ring opened RBO	503	0.053	
	Two-stage transesterification	Rice bran TMP ester	598	ND	[195]
	Epoxidation	Epoxidized rice bran oil	578	ND	
Karanja oil	Two-stage transesterification	Karanja TMP ester	593	ND	[195]
	Epoxidation	Epoxidized karanja oil	565	ND	

Remarks:
ND – no data

4.2. Waste Cooking oil Biolubricant

The utilization of non-edible oil as a biolubricant feedstock may seem to be plausible considering that it is low-prized and will not compete with the consumer market of edible oil. However, for non-edible oil to be a sustainable source of biolubricant feedstock, a large land mass is required for both edible and non-edible oil crops so that the demand for edible oils can be met without causing issues such as food scarcity [167]. Besides, some downsides come with the effective exploitation of non-edible oil, which are the scattered oil harvest location (which are tied to certain regions), the unreliable availability of oil-producing fruits/seeds for continual biolubricant production, and the lack of post-harvest technology for further processing [176]. Alternatively, WCO can be used as a biolubricant feedstock. Recent studies reported that the global generation of waste cooking oil is 20 – 30% of the total vegetable oil production, which amounts to around 63 million tonnes/year [242]. These WCOs may cause adverse effects on the environment if not disposed of correctly. There have been many studies on the valorization of WCO, including hydrogen gas production, electricity generation, and biofuel for internal combustion engines [243]. This paper focuses on the utilization of WCO as a biolubricant feedstock, as the high content of free fatty acid (FFA) in WCO can be exploited through further chemical modification, such as transesterification and hydrolysis. Not only does it abide by the circular economy principle, but the successful valorization of WCO can also reduce adverse environmental impacts.

In research conducted by Hoong et al. [244], palm-based waste cooking oil was valorized to form biolubricant through estolide formation. The lauric acid-capped estolide formed from the palm-based WCO was further reacted to produce 2-ethylhexyl Ester of Lauric Acidcapped Estolide (2EHLaE) and Dibutylamide of Lauric Acid-capped Estolide (DBALaE) esterification and amidation, respectively. The VI, pour point, OOT, and WSD are 190, -12°C, 199.8°C, and 0.58 mm for 2EHLaE, and 169, -12°C, 200.3°C, and 0.56 for DBALaE, respectively. The physicochemical properties of 2EHLaE were found to be comparable to that of HLP68 (a commercial mineral-oil-based hydraulic oil); in fact, the properties are slightly better than that of HLP68. 2EHLaE can be classified under ISO VG 68 lubricant, which is suitable for hydraulic fluid, gear oil, and compressor oil. DBALaE can be classified under ISO VG 150 due to higher kinematic viscosity caused by branched dibutyl amine moiety and increased intermolecular chain entanglement. DBALaE can be utilized in applications that need better friction and wear prevention.

In a two-step transesterification, Nogales-Delgado et al. [245] produced a WCO biolubricant using pentaerythritol with a viscosity of 62.89 cSt and 11.24 cSt at 40°C and 60°C. It has a high VI of 173 and a high flash point of 242 – 249, which is suitable for high-temperature applications due to its resistance to viscosity change at an elevated temperature. The WCO biolubricant is reported to have an oxidative stability of 2.63 h, which can be further improved up to 8 h by adding commercial antioxidants (e.g. propyl gallate and TBHQ). Similarly, Joshi et al. [246] synthesized 3 different biolubricants from WCO through a two-step transesterification process with 3 different alcohols 1- Heptanol, 2-ethyl-1-hexanol, and neopentyl glycol (NPG). Among the 3 biolubricants, the transesterification of WCO with NPG yielded the best physicochemical properties. It has exceptional viscosity (44.9 cSt at 40°C and 20.55 cSt at 100°C) and VI (457.442). Its viscosity at 40°C is within that of mineral oil. At 100°C, the viscosity of WCOME – NPG is at least 86% higher than that of mineral oil. This shows its superior resistance to change due to elevated temperature. As discussed earlier, biolubricants with higher viscosity would result in a better tribe-film formation. This phenomenon can be observed through the tribological test. During the tribological test, heat is generated due to friction. From the study, the WCOME – NPG showed the lowest COF and WSD due to its higher viscosity and VI, which is attributable to its superior tribo-film formation. On top of that, the structure of NPG allows the bonding of 2 WCOME molecules during transesterification. WCOME

– NPG is advantageous over the 2 other biolubricants due to its additional ester group, which readily allows the absorption of the polar ester onto the friction surface for a better tribo-film formation. The ester groups essentially strengthen the tribo-film by binding the molecules strongly and providing strong resistance to shear forces. Furthermore, Hussein et al. [247] produced dioleoyl ethylene glycol ester as a biolubricant by undergoing transesterification and distillation. The biolubricant displays great viscosity (74.74 cSt at 40°C, and 35.79 cSt at 100°C), viscosity index (466), pour point (-13°C), and flash point (235). The biolubricant fulfils the ISO VG68 to a great extent, especially in terms of VI and pour point, making it comparatively better in both elevated-temperature and low-temperature applications.

In a novel method proposed by Nurulita et al. [248], a blend of WCO and *Callophyllum inophyllum* oil (UCOCI) underwent esterification and two-stage transesterification using an infrared-irradiation-assisted heating method. The addition of EG in the transesterification of UCOCI methyl ester resulted in a significant increase of viscosity at 40°C (83.46 cSt) as opposed to the viscosity of UCOCI methyl ester itself (5.717 cSt). The transesterification also yielded a biolubricant with good VI of 216.32 and exceptional oxidative stability of 1642.67 min, which is similar to that of SAE 15W-40 lubricant (1703.2 min). Further tribology tests for the UCOCI biolubricant suggested highly efficient anti-friction performance due to its effective tribo-film coating (due to high viscosity), the presence of triglyceride (that enhances tribo-film strength), and the presence of high oleic acid content (that reduces friction due to COOH group polar coating on metal surface). The COF and WSD of the biolubricant are 0.0811 and 0.451 mm, which is around 45.97% and 17.86% lower than that of SAE 15W-40 lubricant. Notably, the tribological enhancement may be attributed to the selection and blending of the *Callophyllum inophyllum* oil before the chemical modification, as it has high viscosity and oleic acid content.

Aside from transesterification, the combination of epoxidation and oxirane ring opening poses greater potential in the valorization of WCO into biolubricants. Zhang et al. [249] transformed WCO into a highly versatile biolubricant by carrying out the chemical modifications in the sequence of hydrolysis > urea complexation separation > esterification (with 2-ethyl hexanol) > epoxidation > oxirane ring opening (with octanoic acid). In their study, unsaturated fatty acids are concentrated through the separation process and are utilized in further chemical modification. The esterification of the unsaturated fatty acids with ethylhexanol decreased the pour point to -47.33°C due to the increased steric hindrance attributed to the increased chain length, decreased linearity, and ethyl branching. Despite its good cold flow characteristics, this intermediate product is not suitable as a lubricant due to high double bonds and low oxidative stability. Similar to the explanation in Section 4.1. , the epoxidation process would decrease the pour point of the intermediate product due to the complete removal of double bonds, which in turn increased the linearity of the molecules, allowing them to stack and accumulate readily from the increased intermolecular forces between the ethylene oxide rings and form microcrystals at low temperature. Through the oxirane ring opening reaction, each of the ethylene oxide ring sites has the potential to bond with 2 octanoic acids, forming a dioctylated biolubricant that is highly branched. The branched structures prevent molecular packing at extremely low temperatures. Not only the octylated branched biolubricant has a superior pour point of -61°C, it has considerable VI (149) and substantial flash point, TOT, OOT, WSD, COF, and film coverage rates of 303.67°C, 326.35°C, 312.06°C, 203 μm, 0.09, 90%. The biolubricant produced is better than most mineral oils in terms of lubricity as the COF of the biolubricant is 40% lower than that of mineral oil. The biolubricant's high viscosity of 67.51 cSt at 40°C provides a thicker tribo-film coverage of 90% instead of 17% from the mineral oil, making it a very promising replacement for mineral oil. Table 9 highlights biolubricants with high viscosity indices, along with notable pour and flash points [250].

Figure 8 compares key properties such as viscosity, viscosity index (VI), pour point, and flash point among various vegetable-based biolubricants. High-performance lubricants, defined by a VI >120, surpass conventional mineral oils in thermal stability [251]. However, many such biolubricants exhibit low viscosity, rendering them unsuitable for practical applications. Insufficient viscosity hinders adequate oil film formation, especially under high load or pressure, leading to lubricant expulsion from the friction surface and subsequent friction loss [252].

The WCO can also be epoxidized first and then trans-esterified into biolubricant. Li and Wang [251] trans-esterified the epoxidized WCO with iso-octanol, iso-tridecanol, and iso-octadecanol. The biolubricants synthesized are denoted as EWO, EWC, and EWT, respectively. In their research, the biolubricants are evaluated for their oxidative stability under the OIT. The OIT of the biolubricants is increased to 35 – 79.2 min from the initial 3.5 min for the neat WCO. The huge improvement in the OIT is attributed to removing the double bond from the epoxidation reaction. It was reported that EWO displayed the highest OIT, followed by EWC and EWT. This is because the longer alkyl chains are transesterified with the epoxidized WCO, making the biolubricant more susceptible to oxidation due to a more accessible reaction site than short chains.

One notable study stood out for its innovative approach using physical treatment modification, specifically by employing molecular distillation. Fernández-Silva et al. [253] fractionated WCO into two fractions – light and heavy fractions. The study showed an improvement in viscosity and VI for the heavy fractions when compared to the neat WCO due to the absence of FFA. On the other hand, the light fractions were found to have higher OOT, which was higher than that of the neat WCOs and their respective heavy fractions. In the mixed friction regime, the light and heavy fractions of the WCOs were found to have reduced WSD of 28 – 43.9% for light fractions and 11.7 – 44.3% for heavy fractions. Both the fractions had reduced COF compared to the neat WCO, with the heavy fractions having a comparatively lower COF than light fractions. This can be attributed to its higher viscosity, which enhances the tribo-film coverage on the friction surface. However, the light fractions showed a better lubrication performance in the boundary friction regime than the heavy fractions despite having a lower viscosity. This is due to the presence of entirely FFA and polar compounds in the light fractions, which facilitated the strong tribo-film adhesion to the fraction surface through chemical bonding with the polar molecules and lateral attraction between hydrocarbon chains.

Figure 9 compares the tribological performance of conventional mineral oil with that of WCO-based biolubricant. The WSD and COF of mineral oil are around 328 – 343 μm and 0.15 [192, 249]. It is observed that most WCO-based lubricants show significant improvement regardless of chemical or physical treatment. One of the most promising chemical modifications to date is the combination of esterification + epoxidation + oxirane ring opening, which was discussed above. The researchers synthesised a highly versatile biolubricant with exceptional tribological performance. As shown in
Figure 9, it has the lowest WSD of 203 μm and a low COF of 0.09. The oxirane ring opened biolubricant has the potential to replace mineral oil as it is superior not only in terms of tribological performance but also in terms of physicochemical properties such as viscosity, VI, pour point, and flash point. Therefore, researchers may conduct further modifications

rather than stopping at the transesterification stage to produce a sustainable biolubricant with a wide range of industrial applications (for example, high temperature, extremely cold temperature, and high pressure). The physicochemical properties, oil grades, and industrial applications of biolubricants and mineral oil-based lubricants are compared in Table 10. Biolubricants derived from vegetable oil and WCO through chemical modifications can be classified as hydraulic oils, with grades ranging from ISO VG 10 to ISO VG 460. These biolubricants are suitable for various industrial applications, including use as circulating oil, gear oil, reciprocating oil, spindle oil, air compressor oil, and turbine system oil. While biolubricants may exhibit higher pour points than their equivalent oil grades, the values reported are based on formulations without additives. In contrast, the properties of mineral oil-based lubricants are enhanced with additives such as VI improvers, pour point depressants, anti-wear agents, extreme pressure additives, and friction modifiers [6].

Despite the absence of additives, the physicochemical properties of chemically modified biolubricants are comparable to commercial-grade lubricants and can be further optimized with additive incorporation. Beyond physicochemical properties, biolubricants demonstrate comparable or superior tribological performance. For example, Choudhury et al. [254] reported that Thevetia peruviana oil-based biolubricant exhibited a lower COF of 0.0469 and a comparable WSD of 0.462 mm, compared to the COF of 0.0722 and WSD of 0.395 mm for ISO VG 46 oil. However, gaps in research remain, particularly in the areas of TOT, OOT, and OIT. These factors are critical for assessing the thermal and oxidative stability of biolubricants, which determine their suitability for industrial applications, especially under conditions of high temperature and exposure to ambient air. Addressing these research gaps will be essential for advancing the adoption of biolubricants in industrial settings.

Table 9. Physical properties and thermo-oxidative performance of waste cooking oil-based biolubricants.

Chemical Modification	**Biolubricant**	**Viscosity (cSt)**		**Viscosity Index**	**Pour Point (°C)**	**Flash Point (°C)**	**Onset Temperature (°C)**	**Oxidation Induction Time (min)**	**References**
		@ 40°C	**@ 100°C**						
Transesterification	WCO methyl ester	52.9	8.74	ND	6	278	ND	ND	[255]
Transesterification	WCO FAOE	10.47	2.75	103	ND	ND	ND	ND	[256]
Transesterification	WCO TFATE	15.5	4.24	196	-9	198	ND	ND	[257]
Two-stage Transesterification	WCO EGDE	24.3	5.06	147	-8	ND	200 [1]	ND	[230]
Two-stage Transesterification	WCOBL	62.89	11.24	173	ND	242 – 249	ND	2.63 h [3]	[245]
Two-stage Transesterification	WCOME – EG	10.35	3.12	183.6	-9	ND	ND	ND	[258]
Two-stage Transesterification	WCOME	ND	ND	ND	9	125	ND	ND	[246]
	WCOME – 1 - heptanol	24.9	4.96	126.605	0	210	ND	ND	
	WCOME – 2 -ethyl 1-hexanol	34.91	6.09	121.59	-1	216	ND	ND	
	WCOME – NPG	44.9	20.55	457.442	-1	238	ND	ND	
Two-stage Transesterification	Dioleoyl ethylene glycol ester	74.74	35.79	466	-13	235	294 [1]	ND	[247]
Two-stage Transesterification	WCO TMPE	14.25	ND	106.94	-6	149	ND	ND	[259]
Two-stage transesterification	UCO	47.444	10.654	223	-3	ND	ND	ND	[250]
	UCO-FAME	5.0352	1.7740	ND	0	190	ND	ND	
	UCO-BL	7.2844	2.5390	215	-15	180	ND	ND	
Esterification + Two-stage Transesterification	UCOCI biolubricant	83.46	13.2	216.32	ND	243.3	ND	1642.67	[248]
Epoxidation + Transesterification	EWO	15.9	3.4	157	-17	ND	194.5 [2]	79.2	[251]
	EWC	24.7	5.1	143	-20	ND	180.3 [2]	50.4	

	EWT	43.4	7.4	135	-24	ND	163.9 [2]	35.0	
Transesterification + Epoxidation	WCOME	4.71	ND	ND	-3	ND	175 [1]	ND	[260]
	WCOME epoxide	10.42	3.29	ND	10	ND	187 [1]	ND	
Base Catalyzed Transesterification + Epoxidation	WCO	49.84	ND	ND	-7.8	ND	ND	ND	[261]
	EWCO	278.9	32.55	164.94	-6.2	ND	ND	ND	
	EWCOME	12.15	2.57	151.97	2.4	ND	ND	ND	
Estolide formation	2EHLaE	63.1	11.8	190	-12	ND	199.8 [2]	ND	[244]
	DBALaE	136.2	20.3	169	-12	ND	200.3 [2]	ND	
Enzymatic hydrolysis + esterification + epoxidation + oxirane ring opening	WCO	34.33	7.36	188	2.67	177.33	ND	12	[249]
	2-ethylhexyl ester	8.43	2.83	216	-47.33	213.33	ND	ND	
	Epoxy 2-ethylhexyl ester	21.76	4.71	140	11.33	232.00	ND	ND	
	Octylated branched biolubricant	67.51	10.74	149	-61	303.67	326.35 [1] 312.06 [2]	21	
Fractionation	FF	40.63	7.87	169	ND	ND	370.4 [1] 165.6 [2]	ND	[253]
	FF-Light	28.16	5.94	163	ND	ND	371.4 [1] 170.7 [2]	ND	
	FF-Heavy	42.12	8.90	199	ND	ND	348.0 [1] 164.3 [2]	ND	
	FR	44.29	8.70	179	ND	ND	345.7 [1] 159.4 [2]	ND	
	FR-Light	30.29	6.16	158	ND	ND	378.6 [1] 161.2 [2]	ND	
	FR-Heavy	46.36	9.40	192	ND	ND	346.2 [1] 159.5 [2]	ND	
	WM	40.66	8.90	208	ND	ND	363.8 [1] 153.0 [2]	ND	
	WM-Light	33.61	6.93	173	ND	ND	386.3 [1] 172.0 [2]	ND	
	WM-Heavy	39.52	9.57	239	ND	ND	376.5 [1]	ND	

157.5 [2]

Remarks:
ND – no data
[1] TOT
[2] OOT
[3] Rancimat test method

Table 10. Comparison of physicochemical properties, oil grade, and industrial application between mineral-based lubricants and biolubricants.

Oil Type	**Viscosity (cSt)**		**VI**	**Flash Point (°C)**	**Pour Point (°C)**	**Specific gravity/ density at 15°C**	**Oil Grade**	**Application**	**Reference**
	40 °C	**100 °C**							
Conventional Mineral Oil									
Automobile	28	4.9	106	204	-28	0.878	SAE 10W	Automobiles. trucks and marine engines	[6]
	48	7.0	103	218	-24	0.884	SAE 20W		
	93	10.8	100	218	-20	0.890	SAE 30		
	134	13.7	97	238	-16	0.895	SAE 40		
	204	17.8	94	250	-10	0.901	SAE 50		
	65	10.4	147	210	-36	0.875	SAE 5W-30		
	62	10.3	155	208	-36	0.880	SAE 10W-30		
	138	15.3	114	246	-21	0.897	SAE 20W-40	Railroad diesels	
	108	15.0	145	218	-27	0.885	SAE 15W-40	Diesels	
Gear	144	14.0	93	192	-22	0.900	80W-9Q	Automotive & industrial gear units	
	416	27.5	91	210	-14	0.907	85W-140		
Turbine	31	5.4	107	206	-10	0.863	Light	Steam turbines. electric motors, industrial systems	
	64	8.7	105	220	-6	0.876	Medium		
	79	9.9	103	230	-6	0.879	Heavy		
Hydraulic fluids	30	5.3	99	206	-24	0.868	Light	Machine tools, hydraulic systems	
	43	6.5	98	210	-23	0.871	Medium		
	64	8.4	97	216	-22	0.875	Heavy		
	14	5.1	370	96	-62	0.859	Extra Low Temp.	Aircraft hydraulic systems	
	>28.8	>4.1	>90	204	-8	0.857* (20°C)	ISO VG 32	Hydraulic fluid, air compressor oil	[247, 262]
	>41.4	>4.1	>90	220	-6	0.861* (20°C)	ISO VG 46		
	>61.4	>4.1	>198	226	-6	0.865* (20°C)	ISO VG 68		
	>90	>4.1	>216	246	-6	0.869* (20°C)	ISO VG 100	Circulating oil, gear oil, reciprocating oil	
Aviation	98	11.2	100	218	-23	0.876	Grade 65	Reciprocating aircraft engines	[6]
	139	14.7	105	232	-23	0.887	Grade 80		
	216	19.6	100	244	-18	0.898	Grade 100		

	304	23.2	95	244	-18	0.893	Grade 120		
Biolubricants									
Di-oleate-palm olein	417.5	45.5	166	245	-5	ND	ISO VG 460	Gear oil	[263]
Oleate-ether-palm olein	113.7	21.5	217	210	1	ND	ISO VG 100	Circulating oil, gear oil, reciprocating oil	[262, 263]
Palm kernel oil methyl ester – EG	42.10	4.22	232	183	-6	ND	ISO VG 46 ISO VG 220	Light gear oil, hydraulic oil, air compressor oil	[262, 264]
Oxirane ring opened oleic acid	30.13 – 32.90	5.9 – 6.046	132 – 145	ND	3 – -3	0.906 – 0.908* (20°C)	ISO VG 32	Hydraulic/turbine fluid, air compressor oil	[262, 265]
CPOFAs-NPG diester	50.1	15.5	190	245	7	0.878* (25°C)	ISO VG 46	Hydraulic oil, compressor and turbine oil system	[234]
WCO-based TFATE	15.5	4.24	194	198	-9	0.895*	ISO VG 10	Spindle oil	[257]
WCO TMP based ester	35.084	8.651	239.41	244	-12.6	0.9535*	ISO VG 32	Hydraulic fluid, air compressor oil	[266]
Epoxidized WCO	278.9	ND	164.94	ND	-6.2	0.802*	ISO VG 220	Gear oil	[261]
Epoxidized WCO methyl ester	12.15	ND	151.97	ND	2.4	0.773*	ISO VG 10	Spindle oil	
WCO-based dioleoyl ethylene glycol ester	74.74	35.79	466	235	-13	0.895* (20°C)	ISO VG 68	Hydraulic fluid, air compressor oil	[247, 262]

Remarks:
ND – no data
*Density value

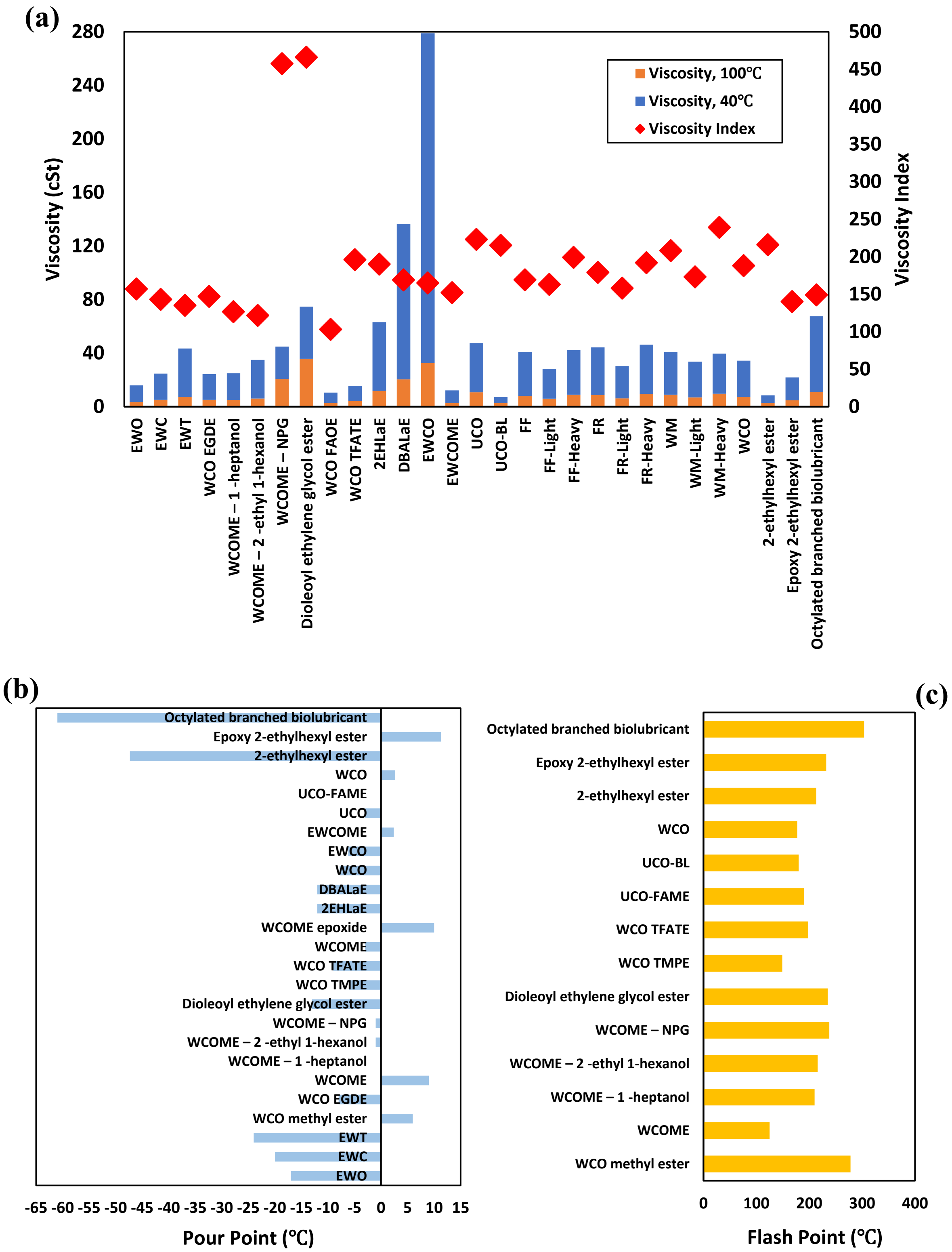


Figure 8. (a) Viscosity at 40°C and 100°C, viscosity index, (b) pour point, and (c) flash point of WCO-based biolubricants.

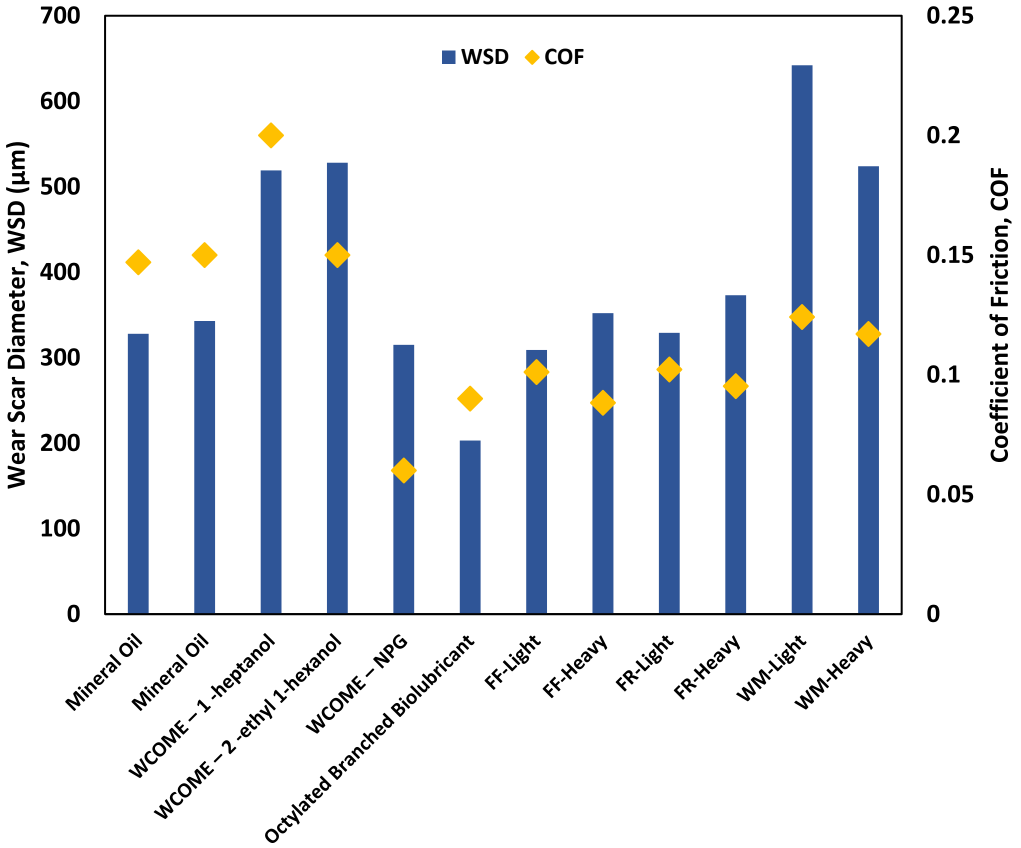


Figure 9. Comparison of tribological performance between conventional mineral oil and WCO-based biolubricants [192, 246, 249, 253].

5. Properties enhancement of Antioxidants on Biolubricants

It is generally known that one of the limitations of biolubricants being a competitive industrial lubricant is its poor oxidative stability. While their cold flow characteristics and viscosity-temperature performance can be further enhanced through chemical and physical modifications, as discussed above, their poor oxidative stability should be enhanced by other means as it is difficult for chemical and physical modifications to improve their oxidative stability. Chemical and physical modifications may be one of the factors that contribute to the low oxidative stability given their hash chemical and high temperature (e.g., fractionation) exposure. A study by Nie et al. [267] showed the deterioration of oxidative stability of different crop oils at different stages of chemical modification. For example, the OSI of *Zea mays* L. oil decreased from 7.2 h to 3.7 h and then to 2.7 h after the first and second transesterification reactions. The conversion of crop oil to TMP esters involves long contact time with a strong base at elevated temperatures during the two-step transesterification. This leads to the deterioration of natural antioxidants initially present in the oil. Furthermore, their study showed that the refining/purification of the TMP esters with silica and Celite absorbents also removes the majority of antioxidants, as indicated by the loss of around 62.5% of OSI after the soybean TMP esters were refined with the silica and Celite column. Therefore, it is important to reintroduce antioxidants into biolubricants so that they are more viable for adaptation in the industry.

Figure 10 shows the graphical summary of the discussion of the potential antioxidant enhancement on biolubricants below. One of the studies carried out by Sediawan [111] proposed a new chemical modification pathway for biodiesel in which antioxidant extraction of the base oil is carried out before further chemical reactions, such as transesterification. After chemical modification, the extracted antioxidants are reintroduced into the biodiesel. This method can also be applied to biolubricant production as the intermediate product (biodiesel) can undergo subsequent chemical reactions such as transesterification and epoxidation for biolubricant conversion. The enhancement of antioxidants on FAMEs and biodiesels has been widely studied; however, the enhancement of antioxidants on biolubricants is not commonly reported in literature despite the similarity in the chemical composition and structures [268]. Considering the similarity in synthesis method and its molecular structure, biolubricants should expect a similar enhancement as that of biodiesel. De Sousa et al. [269] examined the antioxidative effectiveness of curcumin and β-carotene against the testing standard of TBHQ in soybean biodiesel. Curcumin was found to have the best performance compared to β-carotene as the DPPH radical reduction of curcumin (90%) was similar to TBHQ (93%). β-carotene, on the other hand, exhibited less than 15% DPPH radical reduction despite having conjugated double bonds that facilitate the capture of free radicals. The oxidative stability of the biodiesel with different concentrations of antioxidants is evaluated using Rancimat. The oxidative stability of the soybean biodiesel corresponded to the scavenging efficacy of curcumin and β-carotene, with curcumin exhibiting a maximum induction time of 9.11 h at 1500 ppm while β-carotene demonstrated counteractive behavior regardless of dosage. The curcumin-doped soybean biodiesel was examined for its oxidative stability after 30 days, and its induction time decreased as expected due to the presence of high unsaturated fatty acid content. The decrease in oxidative stability in biolubricants due to storage or prolonged lubrication activity can be expected to be less prominent, especially epoxidized due to the removal of double bonds.

Besides curcumin (from turmeric, *Curcuma longa* L.), many other phenolic extracts were studied for their antioxidation performance. Jeyakumar and Narayanasamy [270] compared the antioxidant activity of turmeric, cinnamon, black pepper, red bell pepper, and watermelon seed extract in WCO biodiesel. The turmeric extract showed the strongest scavenging activity of 92%, followed by red bell pepper (89%), black pepper (88%), cinnamon (85%), and watermelon seed extract (80%). When the extracts were added to the WCO biodiesel, the oxidative stability enhancement of the WCO biodiesel corresponded to the respective scavenging activity, with turmeric extract being the highest and watermelon seed extract being the lowest. Despite curcumin showing consistently superior antioxidation performance, some other phenolic compounds, such as one found in passion fruit seed extracts, hold the potential to exceed the antioxidation performance of curcumin. Jain et al. [271] found that the relative protection factor (RPF) of passion fruit seed extract exceeds that of curcumin. The performance of passion fruit seed extract in WCO biodiesel was evaluated through different dosages. At 200 ppm, the extract's performance was similar to that of BHA and BHT; at 400 ppm, the extract outperformed BHA; the performance of the subsequent concentration increments to 1000 rpm was slightly lower than BHA and BHT but it is expected to have a better performance after the crude extract is further refined. Aside from that, several other extracts were also found to have comparable antioxidant performance as the BHA. Tsouko et al. [46] compared different anti-oxidation performances of palm oil processing wastes, namely palm kernel cake, palm pressed fibre, palm kernel shell, and empty fruit bunch. Adding the palm kernel cake extract to sunflower oil increases the oxidation induction time of the sunflower oil by around 60%, similar to that of BHA. Considering that many of the antioxidant extracts are unrefined and consist of other compounds that may act as pro-oxidants, extracts from different sources have the potential to surpass the performance of and replace commercial antioxidants. Not only should the antioxidant performance of the extract be examined with radical scavenging assays, but it should also be analyzed with its addition into lubricants as any synergistic and anti-synergistic effects from the functional group interactions can be identified.

Moreover, the addition of antioxidants to biolubricants not only enhances oxidative stability but can also improve tribological performance, a less commonly recognized benefit. Sneha et al. [272] investigated the tribological properties of rice bran oil modified with halloysite nanoclay and turmeric oil. While nanoparticles are well-known for reducing friction and wear, their findings revealed that turmeric oil exhibited comparable tribological benefits when used individually. Furthermore, combining both additives in rice bran oil resulted in significant improvements, with COF and WSD reductions of 16.11% and 13.40%, respectively, compared to pure rice bran oil. Similarly, Kumar et al. [273] examined the tribological performance of ginger, pepper, and garlic essential oil in coconut oil. They were found to reduce the COF and WSD while aiding in several enhancements such as pour point, OOT, and oxidative stability.

The ginger essential oil reduced the COF of pure coconut oil from 0.0902 to 0.0446. The garlic essential oil was found to be the best as it had the lowest WSD of 530 µm from the initial 608 µm while having a considerably low COF of 0.0575. From both studies, the antioxidants can ameliorate the tribological performance when added to biolubricants as they can reduce the formation of triglyceride hydroperoxides, which may lead to friction and wear. Besides that, the involvement of the phenolic OH groups in the antioxidant extracts can adhere to the friction surface, thereby reducing the metal-to-metal contact [272]. Although antioxidants are not traditionally associated with tribological improvement, their friction-reducing capabilities warrant consideration for biolubricant applications.

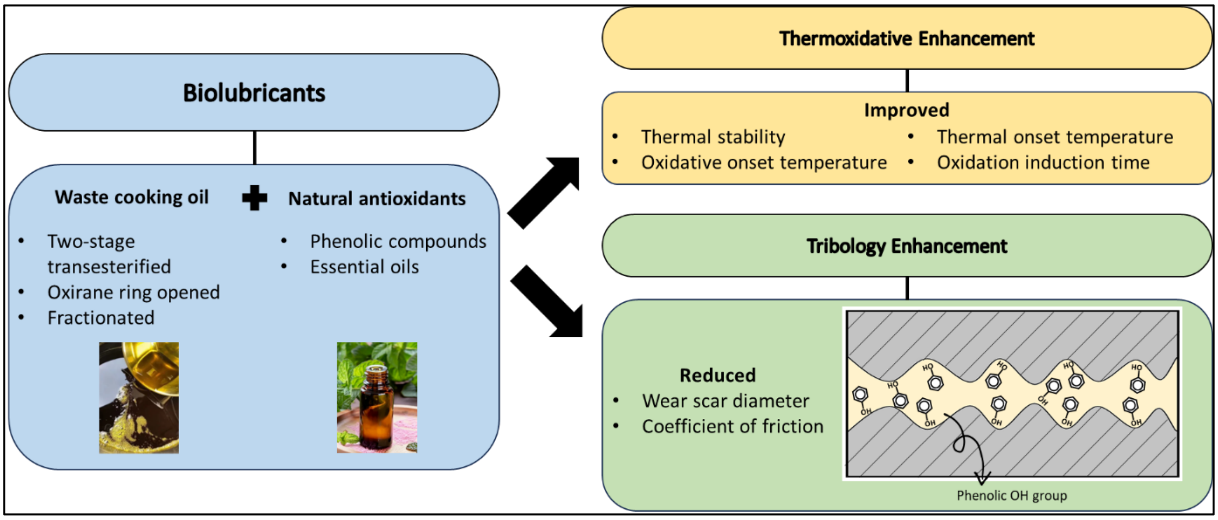


Figure 10. Graphical summary of the enhancement of antioxidants on biolubricants.

6. Outlook and Conclusion

The utilization of waste products as raw materials/ base stocks in both antioxidant extraction and biolubricant is highly impactful for the environment to the United Nations' Sustainable Development Goal 12 (SDG 12) – Responsible Consumption and Production and SDG 13 – Climate Action. The use of WCO and PKE in the synthesis of biolubricant promotes a circular economy approach whereby waste products are recycled and valorized into value-added products, and agri-food industrial wastes are subsequently reduced. The valorization of biomass into value-added products is guided by the principles of the 9R framework (Figure 11), focusing on repurposing, recycling, and recovering. Waste valorization not only follows the 9R framework but also transcends linear economy into the circular economy that imitates the life/biological cycle of nature [274]. The purpose of circular economy implementation is to counteract environmental problems such as resource depletion, waste accumulation, and global warming. The waste valorization through innovative green technologies reduces the need for raw materials in product manufacturing. It transformed the abundant wastes into high-value products that are desirable from an economic and socio-technical standpoint due to sustainable resource recovery and profit conversion [275]. The circular economic approach may improve environmental health and sustainability due to reduced landfilling, global warming, and GHG emissions from the fermentation of bio-waste fields [276]. In the current antioxidant extraction research, many studies have thoroughly explored the potential of technologies in antioxidant extraction. New green solvents such as DES are constantly developed to replace conventional solvents, and they warrant prioritized research as they are highly customizable in terms of the affinity of the target antioxidants. The flexibility and selectivity of the DES may further enhance the efficiency of antioxidant extraction. Future research should also focus not only on green solvents but also on the combination of advanced techniques, such as the utilization of novel DES in UAE or MAE, to reach even higher extraction yields and productivity gains. Despite the current zero-waste approach focusing on valorizing agri-food wastes for high-value products, the wastes generated (e.g. waste solvent, solid residues from raw materials) from the process itself are not generally assessed and discussed. Therefore, future research should also evaluate the wastes generated in terms of their reusability and repurposing to minimize waste.

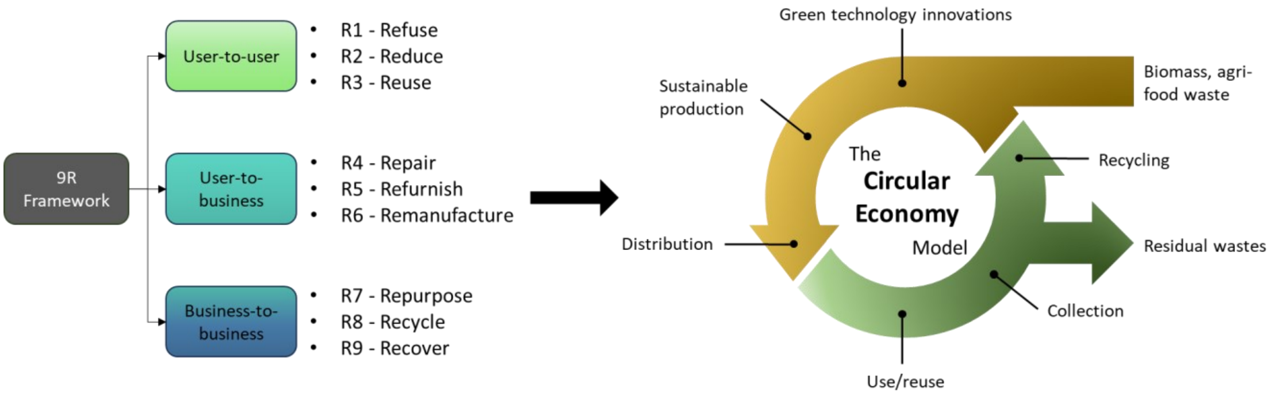


Figure 11. The 9R framework and the circular economy for waste valorization.

Biolubricant production has seen advancements through various chemical modifications, particularly the two-stage transesterification reaction. A standout innovation is the infrared heating transesterification method combined with *Callophyllum inophyllum*-blended oil, which produces biolubricants with promising oxidative stability and tribological performance. Blending WCO with high oleic acid oils prior to chemical modification enhances tribofilm formation, reducing wear and friction on metal surfaces due to the beneficial presence of carboxylic (COOH) groups. In addition to transesterification, the combined epoxidation and oxirane ring-opening process is emerging as a highly effective method for synthesizing biolubricants with excellent pour points, flash points, and lubricity. While physical modifications, such as fractionation, are underexplored, studies show that fractionation significantly improves the physicochemical and tribological properties of biolubricants. Future research should focus on optimizing WCO-blended oil modifications, prioritizing the epoxidation–oxirane ring-opening method and fractionation techniques. Furthermore, evaluating oxidative stability metrics (e.g., TOT, OOT, and OIT) and tribological performance is crucial. The synergistic effects of incorporating natural antioxidants on oxidative and tribological properties should not be overlooked. Finally, biolubricant research must extend beyond developing new antioxidants and synthesis methods to include life cycle assessments (LCA) and techno-economic analyses. These evaluations will help determine the environmental and economic impacts of emerging technologies, including comparisons between chemical and physical modification routes for WCO-based biolubricants.

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Conflict of interest

The authors declare no conflict of interest

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