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The Impact of Curcumin, Gingerol, Piperine, and Proanthocyanidin on the Oxidative Stability of Sunflower and Soybean Oils for Developing Bio-Lubricants

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Abstract

Vegetable oils can serve as a fundamental raw material for formulating lubricants due to their exceptional lubricating properties, which are indicated by viscosity indexes greater than 100. Vegetable oils, due to their unsaturated fatty acids with one or more double bonds, have two significant drawbacks: low oxidation stability and poor performance in low temperatures. The oxidative stability of sunflower and soybean oils was evaluated and correlated with the unsaturation degree calculated based on fatty acid profiles. Different percentages of piperine, curcumin, gingerol, and proanthocyanidin (0.5, 1, 2, and 3 wt.%) have been tested as potential bio-additives for sunflower and soybean oils. All four bio-additives have been observed to enhance oxidation resistance, with gingerol being the most effective, followed by curcumin, piperine, and proanthocyanidin. Bio-additives' effectiveness increases when applied to bio-oils with lower degrees of unsaturation, such as soybean oil. Adding gingerol significantly enhances the induction period, increasing it by about 10 times for soybean oil and 6 times for sunflower oil. This suggests that gingerol can effectively prolong the induction period of both oils.

Keywords: bio-additives; oxidation stability; bio-lubricants; induction period

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1. Introduction

The lubricant market is currently categorized into two primary segments: those utilizing mineral base oils and those composed of synthetic base oils. Importantly, synthetic base oils are also derived from hydrocarbon sources, which contributes to the fact that approximately 93% of all lubricants on the market are produced from hydrocarbon raw materials. With the global oil crisis and the pressing need for decarbonization, lubricant manufacturers are under increasing pressure to seek out innovative, abundant, but also biodegradable, non-toxic, and environmentally sustainable alternatives. This urgent need emphasizes the considerable promise of bio-based lubricants as a viable and responsible alternative to traditional options. Today, the bio-lubricant market is small, accounting for just 1% of the total lubricants market, but continuously expanding [1,2].

However, this transition to bio-based lubricants is not simply a matter of preference; it involves addressing significant challenges associated with using raw materials like vegetable oils. These challenges include ensuring bio-based options' stability, performance, and cost-effectiveness. As we strive to adopt more sustainable practices, overcoming these hurdles will be crucial in unlocking the full potential of bio-based lubricants and paving the way for a greener future in lubrication technology [3,4].

The formulation of a liquid lubricant is carefully designed, with approximately 75% comprising base oil, which can be sourced from mineral, synthetic, or vegetable origins. The remaining 25% consists of a variety of beneficial additives, including antioxidants, pour point depressants, anti-wear (AW) and extreme pressure (EP) agents, viscosity index (VI) improvers, anticorrosive agents, antirust compounds, detergents, and anti-foaming agents. By incorporating these specific additives in tailored proportions according to the lubricant's intended application, we can significantly enhance desirable properties while minimizing undesirable characteristics. This thoughtful composition ensures optimal performance and reliability in various environments, making the lubricant highly effective for its specific use [5–8].

A bio-lubricant is a sustainable option due to its inherent biodegradability, non-toxicity, and environmentally friendly nature. To formulate a successful bio-lubricant, it is crucial to ensure that all components, including the base oil and additives, are biodegradable, non-toxic, and environmentally sustainable. Furthermore, ensuring compatibility between the bio-additives and the base oil's chemical composition will enhance the lubricant's overall performance and effectiveness, contributing to a greener alternative in the industry [9,10].

Vegetable oils, edible or non-edible, present an excellent opportunity as base oils. They offer numerous advantages, including widespread availability, renewability, high lubricity, and a high viscosity index, contributing to their superior lubricating properties. Additionally, they meet important criteria for biodegradability and environmental safety [11].

Compared to mineral and synthetic base oils, vegetable oils have the highest viscosity indexes, so by default, they have the best lubricating properties. Vegetable oils can generate tribofilms upon contact with moving surfaces, significantly reducing friction and wear. These tribofilms are typically self-generated and can form even without harmful additives. The triacylglycerol structure inherent to vegetable oils is crucial in this process. Additionally, under elevated temperatures and pressures, vegetable oils may undergo reactions that form amorphous carbon (a-C) tribofilms on steel surfaces. These tribofilms prevent direct metal-to-metal contact, further mitigating friction and wear. Vegetable oils demonstrate varying tribological properties. Generally, oils with a low degree of unsaturation are known to form thicker boundary films, while those with a higher concentration of polyunsaturated fatty acids may produce comparatively thinner films [12].

However, some inherent challenges associated with vegetable oils must be overcome to enhance their performance to comparable levels of fossil fuel-based or synthetic lubricants. Key challenges include insufficient oxidation stability because of a high level of unsaturation, which can undermine their effectiveness. Additionally, poor performance at low temperatures may occur, and hydrolysis can happen in the presence of moisture, leading to increased corrosion. Moreover, the traditional additives developed for mineral oils in the past 80 years often fail when applied to vegetable oils.

These deficiencies can be mitigated through the implementation of specific chemical processes, the exploration of new feedstocks, or the advancement of more effective additives. Relevant chemical modification techniques—such as esterification, hydrogenation, the acetylation of double bonds, and epoxidation—represent a promising strategy for producing valuable commercial products from biodegradable raw materials [6,13].

Vegetable oils present an impressive diversity, encompassing both edible and non-edible varieties. While their chemical profiles share similarities, containing fatty acids ranging from C6 to C20, the specific compositions can vary significantly. Some oils are particularly high in oleic and linoleic acids, while others may contain greater palmitic, myristic, and other fatty acids.

Understanding the composition of fatty acids in vegetable oils is crucial for explaining their oxidation stability. Vegetable oils typically contain saturated fatty acids, which

have no double bonds (such as lauric, myristic, palmitic, and stearic acids), as well as monounsaturated fatty acids with one double bond (like oleic acid) and polyunsaturated fatty acids with two or more double bonds (such as linoleic and linolenic acids). By analyzing the fatty acid composition, it is possible to calculate the degree of unsaturation and correlate it with the oxidation stability of vegetable oils [6,14,15].

The second major component of lubricating oil consists of additives, which can comprise approximately 25% of its weight.

Depending on the specific application of the lubricant, the additives incorporated into the base oil exhibit significant diversity. These additives may encompass viscosity modifiers, pour point depressants, anti-wear agents, extreme pressure additives, antioxidants, anticorrosion agents, rust inhibitors, anti-foaming agents, demulsifiers–emulsifiers, and detergents–dispersants.

The use of mineral-based lubricants has driven the introduction of antioxidant additives. These additives serve various functions, including acting as scavengers and peroxide inhibitors. Common antioxidant additives include phenolic compounds, aromatic amines, and sulfur- and phosphorus-containing compounds [16–18].

In recent applications, nano-additives, including various metal oxides (such as ZnO and TiO_2), boron nitride, metal-based nanoparticles, MoS_2, carbon dots, and an array of carbon nanomaterials (notably carbon nanotubes, fullerenes, and nanowalls) have emerged as viable alternatives. These nano-additives have been shown to significantly enhance the performance, efficiency, and durability of bio-lubricants, rendering them suitable for a wide range of industrial applications [19,20].

However, when considering the formulation of a bio-lubricant from vegetable oils, it is important to evaluate these additives' use carefully. Due to the unique chemical composition of vegetable oils, these additives may not only fail to enhance oxidation stability but could potentially degrade it. Additionally, to maintain the integrity of the bio-lubricant, it is essential to avoid incorporating these additives as they would compromise its bio-status. By focusing on alternative approaches to enhance the stability of vegetable oils, it is possible to create effective and environmentally friendly lubricants.

Consequently, studies on specific additives for formulating bio-lubricants are currently lacking. However, some studies have explored plant-derived additives that are low in toxicity, biodegradable, and environmentally friendly to enhance anti-wear and anticorrosion properties [21–28].

Therefore, our investigation aims to identify and develop suitable antioxidants designed for vegetable oils. This approach will enhance their performance and reliability, producing more effective, eco-friendly lubricant solutions.

To ensure that vegetable oil maintains its bio-lubricant status, combining it with appropriate bio-antioxidant additives is essential. These additives belong to several promising classes of bio-antioxidants, each offering unique advantages:

- Carotenoids: This group includes valuable subclasses such as carotenes and xanthophylls, which are known for their strong antioxidant properties that can significantly improve oil stability.
- Phenolics: This category encompasses a range of subclasses, including phenolic acids, stilbenes, flavonoids, lignans, and tannins. These compounds effectively combat oxidative stress and enhance the oil's overall quality.
- Alkaloids: This group includes indoles, isoquinolines, tropanes, and pyrrolidines. Alkaloids contribute powerful antioxidant effects, playing a crucial role in oil preservation.
- Glucosinolates: This group features subclasses like indole, aliphatic, and aromatic glucosinolates, which are recognized for their strong antioxidant capabilities and are

crucial in oil preservation. These bio-antioxidants enhance the quality and stability of vegetable oils.

By thoughtfully integrating these diverse bio-antioxidants, it is possible to improve the performance and longevity of vegetable oil as a bio-lubricant, ensuring it meets the demands of various applications while supporting sustainable practices [29–32].

In our investigations, we assessed the antioxidant potential of specific active components derived from a range of plants, including the bioactive compounds in black pepper (especially piperine), the curcuminoids in turmeric, the gingerols in ginger, and the proanthocyanidins found in black grape seeds.

These bio-additives were selected primarily based on their derivation from food-grade materials, relative availability or ease of purchase, and because they enable the maintenance of the bio-lubricant status of vegetable oils.

We specifically evaluated the efficacy of these extracts against oxidative degradation in sunflower oil (SFO) and soybean oil (SBO). The atypical fatty acid profiles in soybean oil can result from various factors, including genetic modifications, environmental conditions during soybean cultivation, and processing techniques. These factors may decrease the levels of essential fatty acids, particularly linoleic acid and alpha-linolenic acid. However, we strategically selected both vegetable oils due to their differing degrees of unsaturation and fatty acid compositions.

2. Materials and Methods

2.1. Lubricants

Two edible vegetable oils, namely sunflower oil (SFO) and soybean oil (SBO), obtained after the first pressing, without synthetic antioxidants, were purchased for our investigations from a Romanian manufacturer specializing in the production of vegetable oils. The physical and chemical properties of the vegetable oils are presented in Table 1. Ethanol (>97%) was used for Soxhlet extraction, and hexane (95%) was purchased from Sigma-Aldrich Chemical Co. (St. Louis, MO, USA).

Table 1. Physical and chemical properties of vegetable oils.

Properties	SFO	SBO	Methods
Density (20 °C, kg/m^3)	920	926	ASTM D-1298-12b [33]
Kinematic viscosity (40 °C, cSt)	32.33	40.99	ASTM D-445-23 [34]
Kinematic viscosity (100 °C, cSt)	7.65	8.92	ASTM D-445-23 [34]
Viscosity index	219	206	ASTM D-2270-24 [35]
Flash point (°C)	285	238	ASTM D-92 [36]
Pour point (°C)	−21	−17	ASTM D-97-17 [37]
Copper corrosion (at 100 °C)	1a	1a	ASTM D-130 [38]
Acid value (mg KOH/g)	0.12	1.7	ASTM D-974-22 [39]
Oxidation stability by RBOT, min	60	220	ASTM D-2272-22 [40]
Wear scar diameter according to 4-ball tester, μm (75 °C, 60 min, 1200 RPM, 147N)	578	453	ASTM D-4172-94 [41]

2.2. Analysis of Fatty Acid Composition of Vegetable Oils

To determine the fatty acid composition of the vegetable oils, the samples were transformed into fatty acid methyl esters. The analyses of the reaction products were performed using gas chromatography on a gas chromatograph system (GC-Varian CP-3800) with a flame ionization detector (FID). The samples were analyzed without dilution before processing. The parameters of the analysis method are as follows: type of chromatographic column: Agilent BD-ASTM D6584, 123-BD11 (L = 15 m, D = 320 μm, d = 0.1 μm)—which

was used to determine monoglycerides, diglycerides, triglycerides, glycerol, and FAME according to the standardized ASTM D6584 method [42]; temperature program: 50 °C for 1 min, then increase by 15 °C/min to 180 °C, increase by 7 °C/min to 230 °C, and increase by 30 °C/min to 380 °C, where it was maintained for 8 min; carrier gas: He with a constant flow rate of 3 mL/min; injector temperature: 300 °C; detector temperature: 400 °C; and injected sample volume: 1 µL. The fatty acid composition of the vegetable oils is presented in Table 2, and the chromatograms of the vegetable oils are presented in the Supplementary Material.

Table 2. Fatty acid composition of vegetable oils.

Fatty Acid	Formula	SFO, wt.%	SBO, wt.%
Lauric	C12:0	0.0	14.5
Myristic	C14:0	0.0	41.0
Palmitic	C16:0	5.8	3.5
Stearic	C18:0	3.8	1.5
Oleic	C18:1	28.2	14.5
Linoleic	C18:2	62.0	5.7
Linolenic	C18:3	0.1	18.9
Others	-	0.1	0.4

2.3. Extraction of Bio-Additive Compounds

Soxhlet extraction is widely acknowledged as the standard and reference method for solid–liquid bioactive compounds from plants. This technique extracted active compounds with potential antioxidant properties from black pepper, turmeric, ginger, and black grape seeds. An amount of 12 g of organic material was carefully placed in a filter paper cartridge, creating a setup designed for efficient extraction. Next, 250 cm^3 of ethanol was introduced into the Soxhlet extractor. The heating apparatus was activated, initiating a controlled extraction process that lasted for 120 min, allowing the ethanol to circulate and extract the active compounds from the organic material. The remaining liquid extract was carefully transferred from the flask to an evaporation vessel after extraction. This vessel was then placed in a temperature-controlled oven set at 120 °C, where it was left to evaporate for 24 h. This step ensured that the solvent was thoroughly removed, leaving behind concentrated extracts that held the essence of the original organic material. The dry extract, measured to the precise quantity needed for the additive, was dissolved in 10 cm^3 of hexane using a sonication bath. This process occurred at a controlled temperature of 55 °C and was maintained for 30 min to ensure thorough dissolution. Once completely dissolved, the hexane solution was carefully mixed with 10 mL of vegetable oil. It was then placed back into the sonication bath to facilitate the complete removal of hexane, ensuring that no residual solvent remained in the mixture. After confirming that the sample was free of hexane, it was introduced into the designated apparatus for conducting oxidation stability investigations, allowing for an accurate assessment of its stability in the presence of oxidizing agents.

2.4. Determination of Oxidative Stability Using RapidOxy 100 Apparatus

Oxidation stability measurements were conducted according to ASTM D7545 using the RapidOxy 100 apparatus from Anton Paar GmbH (Graz, Austria). A sample of 4 g of vegetable oil was placed in a gold-plated aluminum chamber connected to a heating block. A stream of oxygen at a flow rate of 20 L/h was passed through the sample. The measurements were performed at a temperature of 110 °C, with an initial filling pressure of 700 kPa and a measurement pressure of 1800 kPa. During the analysis, oxygen was consumed in the test chamber as oxidation compounds were formed, and the test was

complete when the pressure in the chamber decreased by 10%. The induction period (IP), also referred to as the induction time, is the interval that elapses from the initiation of the oxidation process until it reaches completion. During this critical phase, chemical reactions begin to occur but are not yet fully established. This period can vary significantly depending on factors such as the nature of the substances involved, temperature, and unsaturation degree. Understanding the induction time is essential for effectively managing oxidation processes in various applications, including food preservation, materials science, and environmental chemistry.

The oxidative stability of each tested oil was determined by conducting two parallel repetitions, with the arithmetic mean of the results being reported as the final value.

2.5. FTIR Investigations

The samples were analyzed before and after oxidation using Attenuated Total Reflectance Fourier Transform Infrared (ATR-FTIR) spectroscopy. The samples subjected to FTIR analysis included vegetable oils without additives and those modified with 2 wt.% bio-additives. FTIR measurements were performed on all samples prior to and following oxidation investigations.

The FT-IR spectra of the synthesized samples were recorded with a Shimadzu IRTracer-100 spectrophotometer (Kyoto, Japan) over a scanning range of 400–4000 cm^{-1}.

3. Results and Discussions

3.1. Characterization of Vegetable Oils

The characterization analysis demonstrated that soybean oil and sunflower oil possess distinct lubricating properties attributed to their elevated viscosity indexes, which are 206 for soybean oil and 219 for sunflower oil, respectively. The selected oils were found to have moderate acidity and are classified as corrosion class 1a, indicating that they are not corrosive.

The flash points of soybean and sunflower oils are notably high at 238 °C and 285 °C, respectively. This characteristic renders these oils suitable for applications that necessitate elevated temperatures. Furthermore, their high viscosity index indicates that the viscosity of these oils exhibits minimal variation with temperature changes, reinforcing their stability in various conditions. The anti-wear characteristics are adequate and equivalent to those obtained with mineral base oils; the wear scar diameter is 0.58 mm for sunflower oil and 0.45 mm for soybean oil.

The results confirmed that vegetable oils have well-known drawbacks; they lack oxidation stability and perform poorly at low temperatures. Additionally, these two characteristics represent the primary limitations associated with using vegetable oils as base oils in bio-lubricant formulations. To enhance their performance, it is essential to either incorporate specific additives or use chemical modification techniques.

3.2. The Unsaturation Degree (UD)

The bio-additives' antioxidant potential was assessed using sunflower and soybean oils, both of which have different levels of unsaturation. The unsaturation degree (UD) was calculated with Equation (1) [43].

$$UD = (\text{monounsaturated Cn:1, wt.\%}) + 2\ (\text{polyunsaturated Cn:2,3, wt.\%}) \quad (1)$$

The data in Table 2 provides the basis for calculating the degrees of unsaturation for the vegetable oils under investigation using Equation (1).

The calculated UD for sunflower oil is 153, and the UD for soybean oil is 65.

The oxidative stability test determines the induction time of vegetable oils. When oxidation compounds form very quickly, the vegetable oil degrades quickly, resulting in a short induction period and low oxidation stability. One of the main factors influencing oxidation stability is the percentage of polyunsaturated fatty acids. The degree of unsaturation is closely correlated with oxidation stability. Vegetable oils with a high degree of unsaturation are less stable against oxidation. The induction time in Table 1 is consistent with the calculated unsaturation degrees for fresh vegetable oils. The higher degree of unsaturation of sunflower oil compared to soybean oil results in a shorter induction time. The degree of unsaturation in fatty acids is inversely related to their oxidative stability; as unsaturation increases, oxidative stability decreases. Unsaturated fatty acids, particularly those characterized by multiple double bonds (polyunsaturated fatty acids), exhibit a heightened susceptibility to oxidation. This vulnerability arises from reactive double bonds, which oxygen radicals can readily attack.

3.3. Bio-Additive Compounds

In our investigations, the active compound curcumin was extracted through Soxhlet extraction using ethanol as a solvent. Curcumin, a polyphenolic compound derived from turmeric (Curcuma longa), exists in two distinct forms: keto and enol. Both the keto and enol tautomers possess significant antioxidant properties, enabling them to neutralize free radicals and reduce oxidative stress. This duality in structure may also contribute to the diverse biological activities of curcumin, making it a valuable subject of research as an antioxidant. Curcumin ($C_{21}H_{20}O_6$), as shown in Figure 1a, also named [1,7-bis (4-hydroxy-3-methoxyphenyl)-1,6-heptadiene-3,5-dione], is solid at room temperature.

The extract of black pepper, known as piperine ($C_{17}H_{19}NO_3$), is a natural polyphenolic compound and has the chemical structure (2E, 4E)-5-(benzo[d][1,3] dioxol-5-yl)-1-(piperidin-1-yl)penta-2,4-dien-1-one, as shown in Figure 1b. There are four isomeric forms of piperine found in black pepper: the trans–trans isomer (piperine), the cis–cis isomer (chavicin), the cis–trans isomer (isopiperine), and the trans–cis isomer (isochaviocin).

The active substance extracted from ginger is gingerol ($C_{17}H_{26}O_4$). Gingerol can remain in two forms, either [6]-gingerol or [10]-gingerol, which depend on the position of the methoxy group on the phenolic ring, which can significantly influence their biochemical properties. Its chemical structure can be represented as (1-40-hydroxy-30-methoxyphenyl-5-hydroxy-3-decanone), as illustrated in Figure 1c.

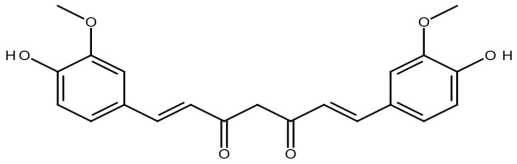
These variations may impact gingerol's antioxidant activity and anti-inflammatory properties.

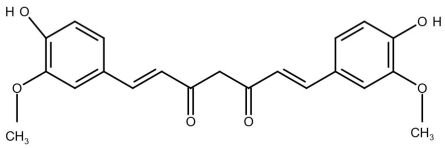
Proanthocyanidins, the active substance extracted from the seeds of black grapes, also called condensed tannins, are oligomers and polymers of monomeric flavonoids. The chemical formula of proanthocyanidin is $C_{31}H_{28}O_{12}$, and the chemical structure is presented in Figure 1d. Proanthocyanidins are known for their antioxidant properties.

3.4. Oxidative Stability Investigations

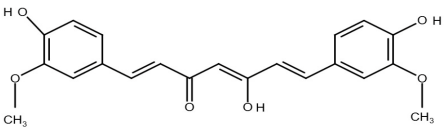
Oxidation studies evaluated the induction period of non-additivated vegetable oil samples and additive oil samples containing varying percentages of bio-additives (0.5, 1, 2, and 3 wt.%). The results of these investigations are presented in Figure 2.

A short induction period indicates that the sample under analysis degrades rapidly regarding oxidation. The induction period is directly linked to a given compound's unsaturation degree. Specifically, as the degree of unsaturation increases, meaning more double bonds are present in the molecular structure, the induction period tends to decrease. This correlation suggests that more highly unsaturated compounds react more readily and may require less time to initiate chemical reactions than their saturated counterparts.

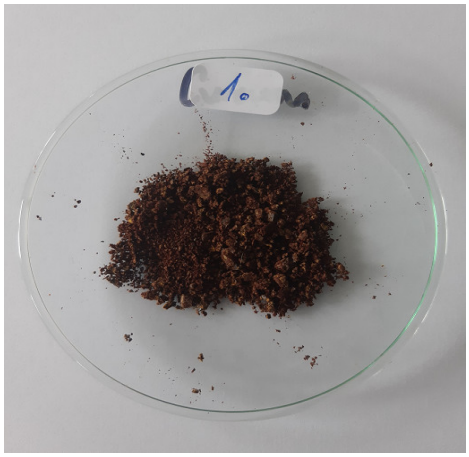




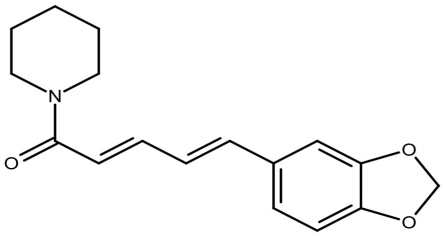
Curcumin keto form

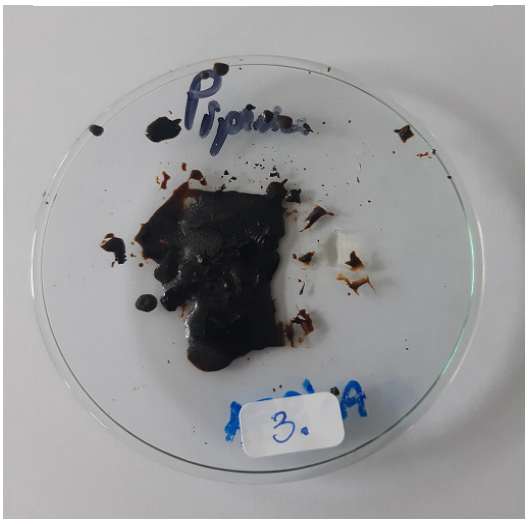


Curcumin enol form



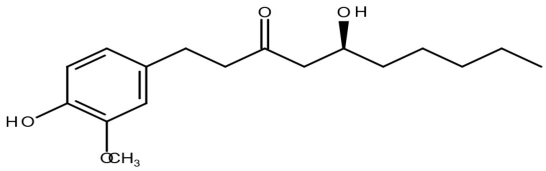
(**a**) Curcumin

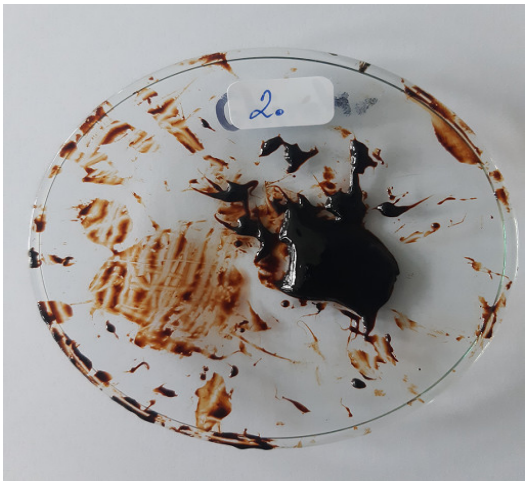




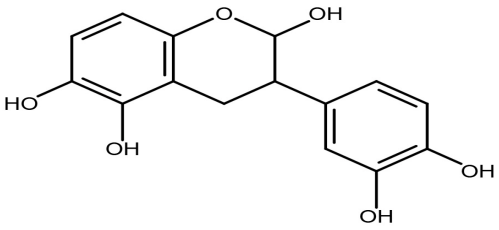
(**b**) Piperine

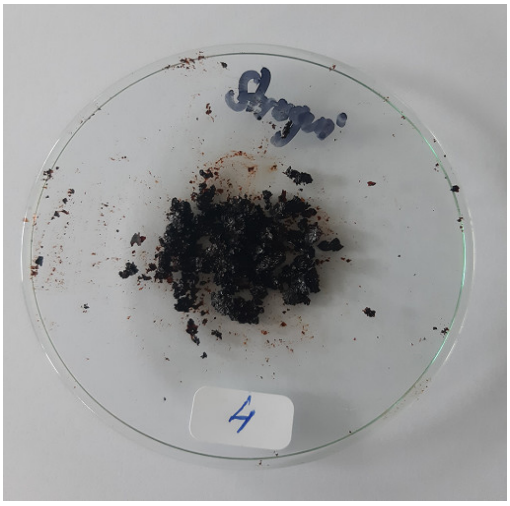
Figure 1. *Cont.*





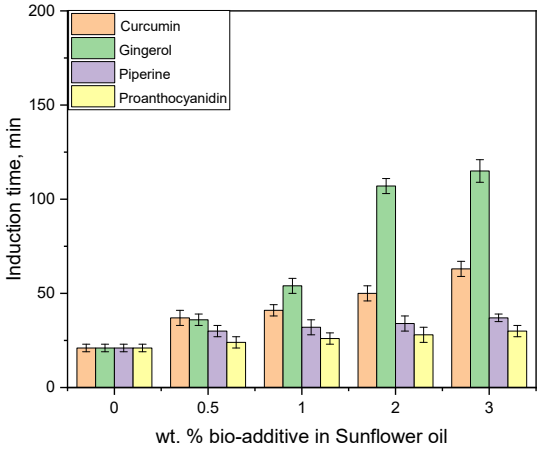
(**c**) Gingerol



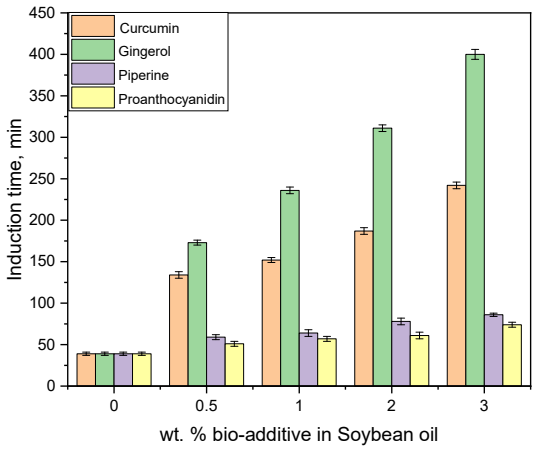


(**d**) Proanthocyanidin

Figure 1. Chemical structures of bio-additives. (**a**) Curcumin with its tautomers keto and enol. (**b**) Piperine. (**c**) Gingerol. (**d**) Proanthocyanidin.



(**a**)



(**b**)

Figure 2. Influence of bio-additive concentration on induction period (**a**) for sunflower oil and (**b**) for soybean oil.

The differences in induction periods between soybean oil and sunflower oil highlight the significance of their chemical compositions in determining oil stability. Non-additivated

soybean oil, with a lower degree of unsaturation, demonstrates a more extended induction period, contributing to its robustness against oxidative deterioration. On the other hand, sunflower oil, known for its higher degree of unsaturation, has a shorter induction period, indicating that it is more vulnerable to oxidation.

Interesting findings were obtained by adding different percentages of bio-additives. It has been shown that these natural additives enhance oil performance, consistently providing benefits regardless of the oil's degree of unsaturation. As the amount of bio-additive increases, the induction period—indicating the oil's resistance to oxidation—also lengthens. This suggests that the oil becomes increasingly resistant to oxygen exposure, improving stability and extending shelf life. Such findings highlight the promising role of bio-additives as effective stabilizers for vegetable oils, paving the way for further exploration and application in the industry.

Our investigation has shown that oils with a lower degree of unsaturation are more prone to improvement through antioxidant additives. Specifically, gingerol increases the induction time by 5.3 times for soybean oil and 2.9 times for sunflower oil. Similarly, curcumin enhances the induction time, increasing it by 3 times for soybean oil and 1.6 times for sunflower oil. The experimental results indicate that gingerol, the active ingredient extracted from ginger, possesses the most advantageous antioxidant qualities, followed by curcumin, piperine, and proanthocyanidin.

3.5. FTIR Results

The FTIR spectra of the bio-additives, vegetable oils without and with 2 wt.% bio-additives in the mid-infrared region of 4000–500 cm^{-1}, are presented in Figures 3–8.

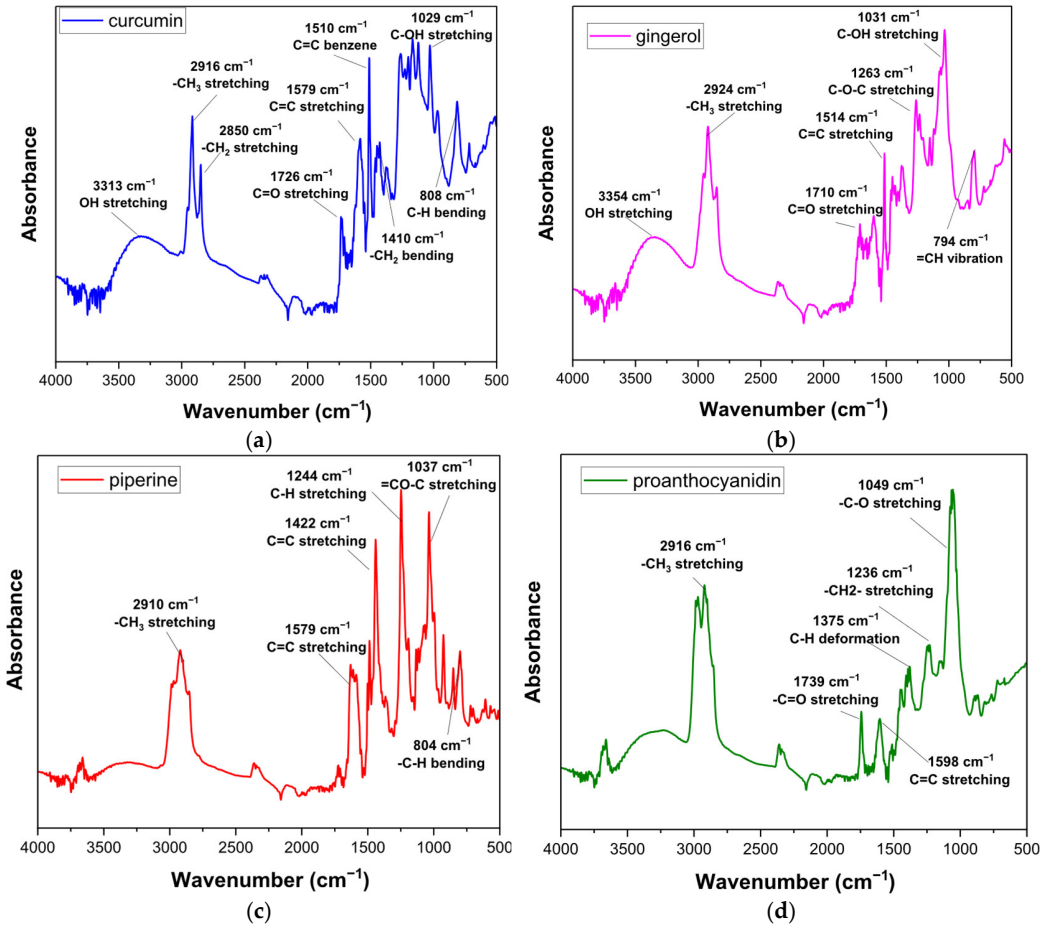


Figure 3. FTIR images for curcumin (**a**), gingerol (**b**), piperine (**c**), and proanthocyanidin (**d**).

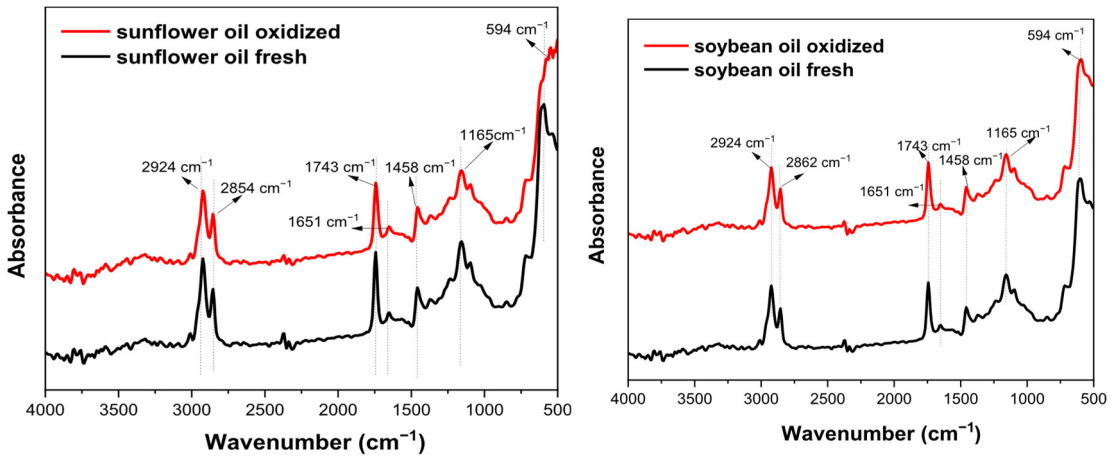


Figure 4. FTIR images of non-additivated vegetable oils, (**a**) sunflower oil and (**b**) soybean oil, before and after oxidation.

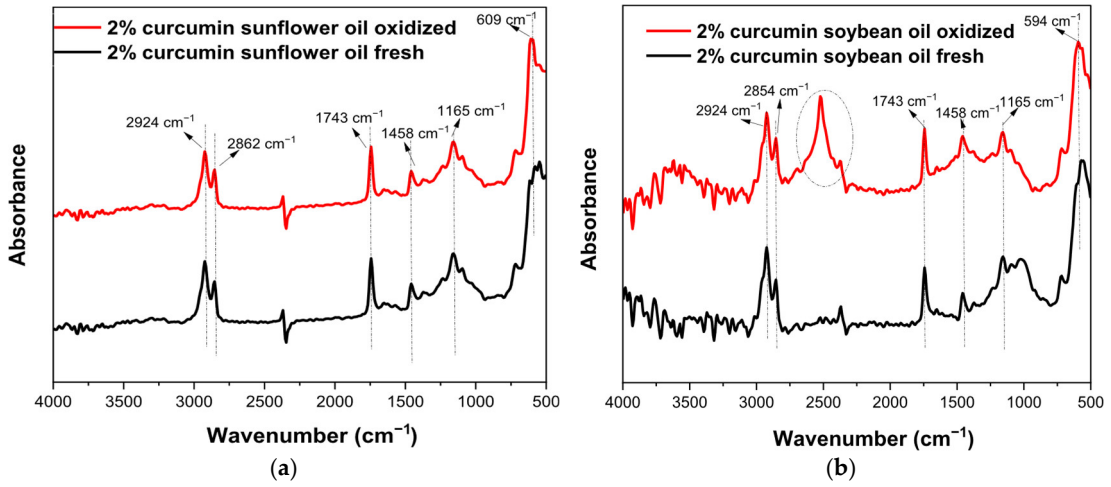


Figure 5. FTIR images of vegetable oils additivated with 2 wt.% curcumin (**a**) sunflower oil and (**b**) soybean oil before and after oxidation.

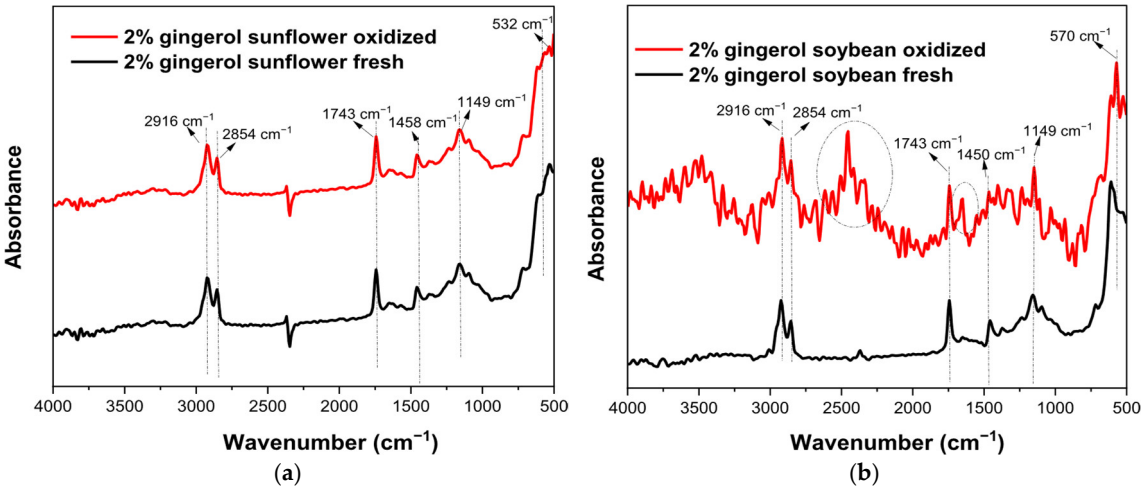


Figure 6. FTIR images for vegetable oils additivated with 2 wt.% gingerol (**a**) sunflower oil and (**b**) soybean oil before and after oxidation.

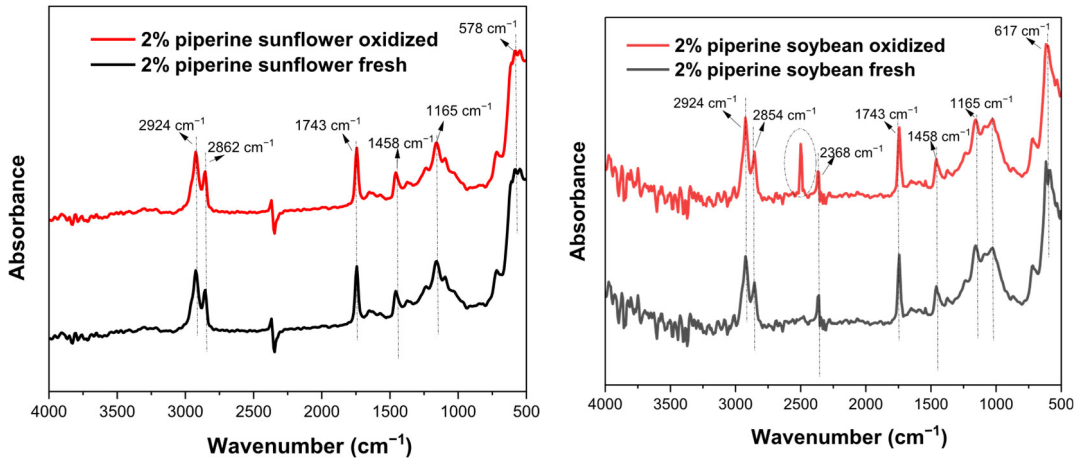


Figure 7. FTIR images of vegetable oils additivated with 2 wt.% piperine (**a**) sunflower oil and (**b**) soybean oil before and after oxidation.

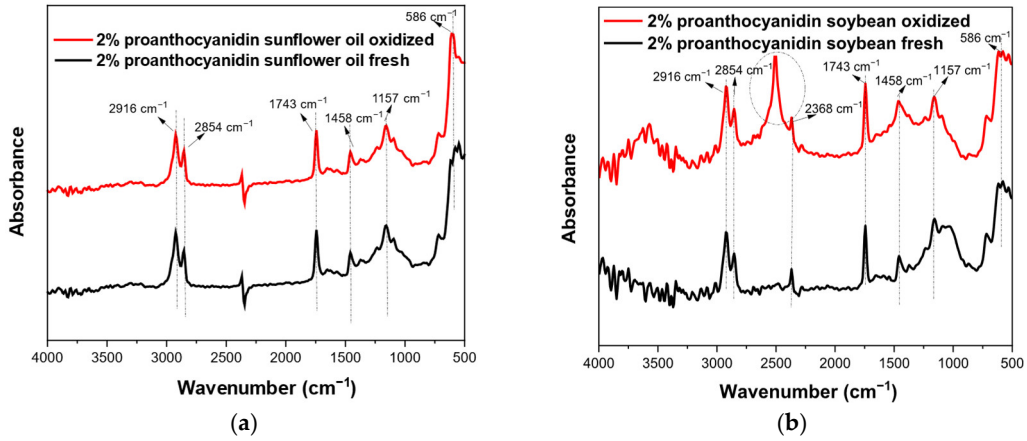


Figure 8. FTIR images of vegetable oils additivated with 2 wt.% proanthocyanidin (**a**) sunflower oil and (**b**) soybean oil before and after oxidation.

The FTIR spectra of curcumin extract usually display distinct absorption bands related to the molecule's functional groups. These bands are essential for recognizing and defining curcumin. Typically seen peaks consist of O-H (approximately 3500 cm^{-1}), C=O (roughly 1650 cm^{-1}), and aromatic C=C (about 1600 cm^{-1}) vibrations. A strong absorption band is seen near 3313 cm^{-1}, signaling the existence of phenolic hydroxyl groups (OH). A prominent absorption band near 1579 cm^{-1} is associated with the carbonyl (C=O) group. Bands related to C-H stretching (approximately 2850 cm^{-1}), C-C stretching in the aromatic ring (roughly 1510 cm^{-1}), and C-O stretching (about 1029 cm^{-1}) are commonly detected.

The FTIR spectra of gingerol help identify functional groups and analyze the composition of ginger extracts and powders. The spectra typically display peaks that indicate the presence of hydroxyl groups (O-H), carbonyl groups (C=O), and various distinctive bonds related to the chemical constituents of ginger. These peaks can help differentiate ginger from other materials and assess how processing affects its chemical composition. In the FTIR spectra, a broad peak at 3354 cm^{-1} indicates O-H stretching vibrations commonly associated with alcohols and phenols present in ginger. A peak at 1710 cm^{-1} corresponds to the C=O stretching vibration, usually linked to the ketone group found in gingerols. Additionally, the peak at 1031 cm^{-1} represents C-OH stretching vibrations observed in other oxygen-containing compounds, while the 1263 cm^{-1} peak signifies O-H bending vibrations.

In the FTIR spectrum of piperine, aromatic C-H stretching was observed in the range of 2910 cm^{-1}, and the peaks at 1579 and 1422 cm^{-1} are attributed to the stretching of -C=C- in the aromatic ring. For the methylene dioxy group, asymmetric and symmetric -CH_2- and aliphatic C-H stretching peaks were detected at 2910, 1422, and 1244 cm^{-1}, respectively. The peak at 1037 cm^{-1} is related to the symmetrical stretching of =CO-C-. The stretching frequency of -CO delocalization was observed at 1579 cm^{-1}; this stretching is crucial for piperine, which confirms its structure. The out-of-plane C-H bending of 1,2,4-trisubstituted phenyl (featuring two neighboring hydrogen atoms) can be detected at 804 cm^{-1}.

In the FTIR spectrum of proanthocyanidin, due to an additional hydroxyl group in the B-ring, a difference is observable in the 1598 cm^{-1} region, typically associated with the skeletal stretching modes of the aromatic ring. The absorption patterns, especially between 1300 and 1000 cm^{-1}, reveal significant variations alongside the shift from an entirely procyanidin polymer to primarily prodelphinidin. The wide band near 1375 cm^{-1} and the absorptions at 1236 and 1049 cm^{-1} intensify, which are associated with C-H deformation, -CH_2-, and -C-O- stretching and are presented in the FTIR spectra of proanthocyanidin.

To thoroughly elucidate the mechanism of the oxidation process, FTIR spectra were obtained for both fresh samples and aged samples, specifically those that underwent oxidation at a temperature of 110 °C in the presence of oxygen. This analysis is crucial for understanding the fundamental dynamics of the oxidation process.

The functional groups associated with the FTIR absorption bands are outlined in Table 3. Our analysis indicated that the spectra of the oil samples revealed notable similarities in their absorbance bands, which aligns with findings from previous studies [21,44].

Table 3. Functional groups in vegetable oils related to FTIR spectra.

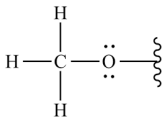
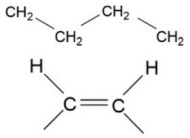
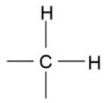
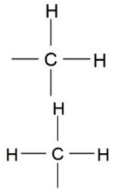
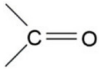
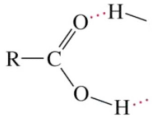
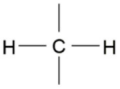
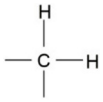
Wavenumbers (cm^{-1})	Functional Group	Mode of Vibration	Possible Structural Units	Absorption Intensity
594	-C-H- (methoxy group)	Bending		Strong
723	-$(CH_2)_n$- -HC=CH	Bending (rocking) and out-plane vibration		Medium–weak
1165	-C-H (CH_2)	Bending		Strong
1458	-C-H (CH_2) -C-H (CH_3)	Bending (scissor) and/or deformation		Medium
1743	-C=O (ester group)	Stretching		Very strong
2350–2500	O-H (carboxylic vibration)	Stretching		Very strong
2854	-C-H (CH_2)	Stretching (symmetrical)		Very strong

Table 3. *Cont.*

Wavenumbers (cm^{-1})	Functional Group	Mode of Vibration	Possible Structural Units	Absorption Intensity
2924	-C-H (CH_2)	Stretching (asymmetrical)		Very strong

In FTIR spectra, several peaks were noticed. The peak at 594 cm^{-1} may be related to the bending vibrations of C-H or O-H groups, while the peaks at 1149–1165 cm^{-1} can be attributed to C-O stretching (ester group). A broad absorption band around 2350–2500 cm^{-1} may indicate a carboxylic acid group, specifically due to the O-H stretching vibration.

The oxidation process of additive oils enhances the intensities related to the vibrational patterns of aliphatic chains, particularly the CH_2 and CH_3 groups. In the FTIR spectra, we observe key absorption frequencies in the fingerprint region (1800–650 cm^{-1}) that indicate the presence of long-chain aliphatic hydrocarbons. For instance, absorptions near 720 cm^{-1} are linked to these hydrocarbons, while the region between 1750–1500 cm^{-1} reveals significant contributions from -C=O absorption around 1750 cm^{-1} and C=C absorption near 1500 cm^{-1}. The O-H group absorbs broadly between 2500 and 3000 cm^{-1} because of strong hydrogen bonding. Furthermore, the 2920–2850 cm^{-1} range reflects the stretching vibrations of the CH_2 aliphatic chain, and the frequencies around 3000 cm^{-1} are associated with the stretching vibrations of the double bond, characteristic of unsaturated fatty acids. This analysis provides valuable insights into the structural changes occurring during the oxidation of vegetable oils.

The presence of an additive during oxidation, indicated by the peak at 2499 cm^{-1} (refer to the FTIR spectra of oxidized proanthocyanidin in Figure 8a,b), implies that the additive participates in the oxidation, possibly oxidized to form CO_2. A peak at 2461 cm^{-1} (refer to the FTIR spectra in Figure 6b for gingerol–soybean oxidation) in an FTIR spectrum is commonly linked to carbon dioxide (CO_2), particularly its asymmetric stretching vibration. This peak is frequently seen when examining materials exposed to or containing carbon dioxide, during oxidation reactions, or in the presence of carbonates. In additive oxidation, the detection of CO_2 at 2461 cm^{-1} implies that oxidation processes are occurring, possibly linked to the decomposition of organic compounds or the interaction of additives with oxygen.

3.6. Oxidation Mechanism

During usage, the characteristics of lubricants can change due to contamination from external substances such as water, fuels, and mechanical particles. Additionally, products resulting from thermal decomposition or oxidation reactions can significantly affect these properties. In most cases, oxidation leads to a loss of stability in the lubricant, while thermal decomposition is typically a secondary effect. Since lubricants are often exposed to air, the rate of oxidation reactions is generally much higher than that of thermal decomposition within the temperature ranges typically encountered during lubrication processes. This is particularly important when using bio-lubricants as they are derived from vegetable-based oils with poor oxidation stability [45–47].

The free radical chain mechanism is responsible for the susceptibility of unsaturated fatty acids in vegetable oils to oxidation [48,49]. The degree of unsaturation, indicated by the number of double bonds in these fatty acids, influences the speed of the oxidation reaction. Auto-oxidation is a natural process that occurs when vegetable oils react with oxygen in the air, changing their chemical properties. This reaction forms various oxygen compounds, including alcohols, aldehydes, ketones, lactones, esters, and acids. The oxi-

dation mechanism involves three main steps: initiation, propagation, and recombination or termination (Figure 9), and the oxidation mechanism in the presence of bio-additives is presented in Figure 10.

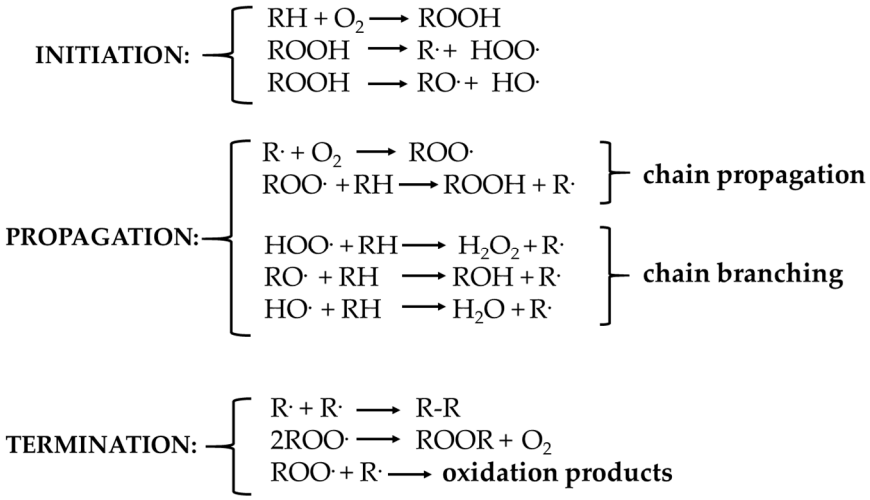


Figure 9. Oxidation mechanism of vegetable oils.

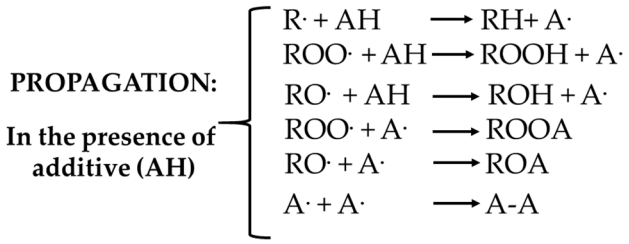


Figure 10. The antioxidant mechanism in the presence of bio-additives (AH).

In the initiation step, free radicals are generated by removing hydrogen atoms from fatty acids or acylglycerols. This process is frequently accelerated by exposure to heat, light, or the presence of metal catalysts. During the propagation step, lipid alkyl radicals engage in a reaction with atmospheric oxygen, resulting in the formation of lipid peroxy radicals. These peroxy radicals subsequently react with additional lipids, which leads to the production of hydroperoxides. The chain reaction is halted in the termination step when radicals combine to form non-radical species.

Oxidation products may encompass primary oxidation products such as alkyl hydroperoxides (ROOH) or dialkyl peroxides (ROOR′) that decompose into secondary compounds, including alcohols (ROH), aldehydes, ketones, and other compounds that have been identified through Fourier Transform Infrared (FTIR) investigations. Oxidation leads to the formation of undesirable compounds, the depletion of essential fatty acids, and the generation of toxic substances. This decomposition can result in undesirable off-flavors and a deterioration in the quality of vegetable oils.

The presence of methoxy, hydroxyl, and carbonyl groups in curcumin, which exhibits a polyphenolic structure, provides antioxidant and radical scavenging properties. The antioxidant activity of curcumin is mainly attributable to its distinct chemical structure, particularly phenolic hydroxyl groups and β-diketone groups. These structural features enable curcumin to function effectively as a free radical scavenger and a donor of hydrogen atoms. Gingerol exhibits antioxidant properties through its capacity to act as a free radical scavenger and an electron donor. This ability is mainly attributed to its phenolic structure, which facilitates the donation of electrons, thereby neutralizing free radicals and mitigating

the potential for oxidative damage. Piperine has antioxidant properties due to its ability to scavenge free radicals and reactive oxygen species. Proanthocyanidins demonstrate antioxidant activity due to their distinctive structure and capability to interact with free radicals and various reactive molecules. This structural property facilitates the efficient donation of hydrogen atoms, thereby neutralizing free radicals and preventing further oxidative damage. Gingerol reveals superior antioxidation performance compared to other bio-additives, likely due to its enhanced ability to neutralize free radicals more effectively than alternative additives [26,50–56].

4. Conclusions

Although the lubricant market is primarily dominated by products derived from mineral oils, vegetable oils present a compelling ecological alternative. These oils are independent of finite mineral resources and boast environmentally friendly properties that make them essential for numerous applications. For instance, in the food industry, bio-lubricants based on vegetable oils are valued for their safety and biodegradability. Additionally, they serve as effective lubricants for engine equipment utilized in forests and protected areas, where environmental integrity is vital. Their versatility and sustainable nature highlight their importance in a world increasingly focused on environmentally responsible choices.

The most important shortcoming of these vegetable oils, poor oxidation stability, could be overcome by adding appropriate antioxidant additives. Identifying suitable antioxidant additives lays the groundwork for formulating biodegradable lubricating oils, and their application can also be extended to the development of bio-greases.

This study presents an easy method for obtaining bio-components with antioxidant potential. The role of a few bio-additives (curcumin, gingerol, piperine, and proanthocyanidin) in the complex oxidation process of vegetable oils was examined in this paper.

Our studies have shown that when selecting vegetable oils for the formulation of bio-lubricants, it is important that they have a lower degree of unsaturation since they are more susceptible to additivation.

Various concentrations of piperine, curcumin, gingerol, and proanthocyanidin (0.5%, 1%, 2%, and 3% by weight) have been evaluated as potential bio-additives for sunflower and soybean oils. All four bio-additives enhanced oxidation resistance, with gingerol demonstrating the highest efficacy, followed by curcumin, piperine, and proanthocyanidin. The incorporation of gingerol significantly extends the induction period, increasing it by approximately tenfold for soybean oil and sixfold for sunflower oil. These findings suggest that gingerol is an effective agent for prolonging the induction period of both oil types.

Gingerol is likely to demonstrate superior oxidation stability in vegetable oils when compared to curcumin, piperine, and proanthocyanidin. This advantage can be attributed to its unique chemical structure and its efficacy as a primary antioxidant. Specifically, gingerol's long alkyl side chain and phenolic hydroxyl group facilitate its ability to scavenge free radicals and chelate metal ions, both of which are critical mechanisms in inhibiting lipid oxidation. Although curcumin, piperine, and proanthocyanidin also exhibit antioxidant properties, their chemical structures may be less effective in the context of vegetable oil oxidation. For instance, the planar structure and limited solubility of curcumin in oils can impede its effectiveness relative to gingerol. Furthermore, piperine possesses a generally weaker antioxidant activity compared to gingerol and may not perform optimally in lipid-rich environments. The large size and complex structure of proanthocyanidin could also limit its capacity to penetrate and protect the oil matrix as effectively as gingerol.

Further research is essential to assess the influence of incorporating these bio-additives on the anti-wear and extreme pressure properties of bio-lubricants. It is also critical to

investigate the interactions between these bio-additives and other compounds utilized to enhance the various characteristics of vegetable-based oils, thereby facilitating the advancement of bio-lubricant technology.

Supplementary Materials: The following supporting information can be downloaded at: https://www.mdpi.com/article/10.3390/lubricants13070302/s1, Figure S1: Gas chromatogram of fatty acid methyl esters from sunflower oil, Figure S2: Gas chromatogram of fatty acid methyl esters from soybean oil.

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