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Assessing Antioxidant and Pour Point Depressant Capacity of Turmeric Rhizome Extract in Biolubricants

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Abstract: Natural polyphenols found in plants are secondary metabolites and act as natural antioxidants. Phenols prevent lipid oxidation by donating their hydrogen to free radicals generated between reactions of oxygen with unsaturated fatty acids. This work aims to examine turmeric extract for its capacity to act as an antioxidant and pour point depressant additive in biolubricants. The study involved extracting turmeric rhizome and analyzing the extract using the gas chromatography-mass spectrometry (GC-MS) and Fourier-transform infrared spectroscopy (FTIR) techniques to identify phenolic compounds and the nature of bonds in terms of abundance peak areas. The yield of concentrated turmeric rhizome extract by weight was 3.7%. The FTIR analysis revealed O-H band at 3336 cm^{-1}, C-H asymmetric and symmetric stretching at 2940 and 2834 cm^{-1}, C=C cyclic ring at 1680–1515 cm^{-1}. The phenols detected by the GC-MS technique are phenol, 2 -methoxy-3-(2-propenyl) occupying 36.3% area at 16.5 min retention time and Phenol, 2-methoxy-4-(2-propenyl)-, acetate having 3.8% area at 3.8 min retention time. The results show promising capacity of turmeric rhizome extract to act as antioxidant and pour point depressant additive in biolubricants.

Keywords: turmeric rhizome; phenol; oxidation; pour point; biolubricants; free radical

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1. Introduction

Lubricants possess anti-friction characteristics and enable the proper functioning of machinery without undesirable failures. For improved properties, lubricants are usually mixtures of base oil and additives in specific proportions; mostly 90% base oil and less than 10% additives. The majority of base oils in lubricants are derived from fossil sources; coupled with the problems of pollution, depletion and climate change, there is a need to find alternative sources of base oil. Plant oils are among the alternatives for mineral-based oils [1]. However, plant oils in their natural environment are stable, and are not easily oxidized before any extraction or processing is carried out. During the process of

extraction and processing for application as biolubricants, they lose their oxidative stability and become a major disadvantage. Therefore, additives are required to improve these properties [2]. Oxidation is the reaction of plant oils with oxygen, and the chain continues through the activity of free radical mechanisms, as represented by Equation (1) [3]. The oxidation of biolubricants produces compounds that affect their viscosity, and this occurs mostly during storage.

$$\begin{array}{l} Initiation: RH \rightarrow R^{\cdot} \\ Propagation: R^{\cdot} + O_2 \rightarrow ROO^{\cdot} \\ ROO^{\cdot} + RH \rightarrow ROOH + R^{\cdot} \\ Termination: R^{\cdot} + R^{\cdot} \rightarrow R - R \\ ROO^{\cdot} + R^{\cdot} \rightarrow ROOR \\ ROO^{\cdot} + ROO^{\cdot} \rightarrow Non - radical\ products \end{array} \quad (1)$$

The most widely used synthetic antioxidants for lubricant application are butylated hydroxy anisole (BHA), butylated hydroxytoluene (BHT), and tert-butylhydroquinone (TBHQ), but they promote carcinogenesis [4]. Due to safety concerns, there is a trend to replace synthetic antioxidants with natural ones. The most used natural antioxidants are tocopherols, polyphenols and phenols [5,6]. These naturally occurring polyphenols are found in fruits, vegetables and cereals, and their basic structure is the phenolic ring [7,8]. Their antioxidant mechanism is based on their ability to act as free radical scavengers and release hydrogen ions that quench the reactive tendencies of oxidation propagation radicals [9]. This is represented by Equation (2), where AH is the antioxidant molecule [3].

$$\begin{array}{l} ROO^{\cdot} + AH \rightarrow ROOH + A^{\cdot} \\ R^{\cdot} + AH \rightarrow RH + A^{\cdot} \\ RO^{\cdot} + AH \rightarrow ROH + A^{\cdot} \\ ROO^{\cdot} + A^{\cdot} \rightarrow ROOA \\ RO^{\cdot} + A^{\cdot} \rightarrow ROA \\ A^{\cdot} + A^{\cdot} \rightarrow A - A \end{array} \quad (2)$$

The effect of *Urtica dioica* aerial part extract on the stability of soybean oil was studied [10]. The results showed that mixing 200–800 ppm of the extract with soybean oil decreased the oil oxidation. The oxidative status and dietary energy of lipids were analyzed [11]. The results showed a limited correlation between individual measures of oxidation. A review of the application and stability of natural antioxidants in edible oils was investigated [12]. It was concluded that natural antioxidants can be used as substitutes for synthetic ones because of their low cost and good thermal stability. Polymeric biomaterials as additives were reviewed [13]. It was found that the additives improved the viscosity index, pour point and anti-friction properties when compared to commercial petroleum-based additives. There were changes in polyphenolic content and antioxidant activity of grape seed extract and grape pomace after thermal treatments [14]. It was observed that furnace thermal treatment did not affect the total extractable polyphenol content or the antioxidant activity of the grape seed extract and the grape pomace. Antioxidant properties of different turmeric species in Bangladesh were assessed [15]. It was observed that the aqueous extract had lower antioxidant properties compared to the ethanolic extract, and turmeric is a source of natural antioxidants. Biomass-based carbon materials as additives in lubricants were studied [16]. The results showed that when SN 900 was used, biochar had improved kinematic viscosities on the base oil more than hybrid biochar. A review of plant leaf extracts as green inhibitors for corrosion of carbon steel was conducted [17]. The study showed that extracts of plant leaves gave significant inhibition efficiencies of 84 to 98%. Garlic oil was assessed as an additive in rubber seed oil [18]. It was observed that friction values decreased and the viscosity index improved when compared to SAE20W40. The antioxidant effect of essential oils or rosemary, clove and cinnamon on hazel and poppy oils was investigated by Özcan and Arslan [19]. Delgado et al. [20] studied the application of

rosemary extracts in vegetable oil-based lubricants. The extract of Olive leaves was investigated for their antioxidant effect on sunflower oil by Rafiee et al., [21]. Xia et al. [22] studied the leaf-surface wax of desert plants as an additive in lubricant. The production of lubricant additives from red Alga dixoniella grisea was investigated by Gavalás-Olea et al. [23].

In this study, the investigation of renewable, non-carcinogenic turmeric rhizome extract as an antioxidant and pour point depressant additive in a biolubricant blend is carried out. Turmeric was chosen because it contains naturally occurring phenols and it is readily available in Nigeria, which is the fourth largest producer [24]. Turmeric has found diverse applications in the culinary [25,26], confectionery [27], cosmetic, and pharmaceutical industries [28,29].

Although there are some exceptions where edible products can be used in industrial applications, such as the extraction of glycerin from oils and fats or the use of curcuma pigment for wood paints [30], there are limitations associated with the industrial use of edible agricultural products. These limitations mainly arise from the availability of edible raw materials at reasonable prices and food safety concerns, which restrict the widespread adoption of edible products in industrial applications. However, as various industries aim to incorporate more sustainable and environmentally friendly components to align with the objectives of the United Nations [31], the utilization of *Curcuma longa* by-products and residues or non-edible varieties should be investigated for its potential as a sustainable solution for industrial applications such as lubricants. In this context, the current study aims to explore the capacity of turmeric rhizome extract to act as an antioxidant and pour point depressant additive for bio-based lubricants.

2. Materials and Methods

2.1. Raw Material and Processing

For the experimental process, rhizomes of *Curcuma longa* (Turmeric) were obtained from Nigeria, specifically from Kafanchan, Kaduna State. The Turmeric samples obtained were cleaned and washed to remove any impurities that could affect the study results. Subsequently, they were ground using a manual grater. All Turmeric samples were maintained at a temperature of 22 °C for a period of 72 h to prevent moisture absorption. Following the drying process, 100 g of Turmeric samples were weighed and chemically treated in a closed 250 mL acetone container, allowing for a 24-h reaction period. Subsequently, the solvent-solute separation process was carried out using filter paper, collecting 165 mL of solvent. The solvent extracted from the separation process was concentrated into a semi-solid dark brown mass by evaporating acetone at atmospheric pressure for two hours at 56 °C. The percentage of yield of the extracts was determined according to Equation (3) [22]. The obtained mass of concentrated extract was 3.7 g from the initial sample of 100 g. Different blends with turmeric extract additive were investigated including transesterified oil (TO), Shea oil-based biolubricant, SN 500 mineral oil, blended (BSAE 40) and standard (SSAE 40).

$$Yield = \frac{Weight\ of\ sample\ extract}{Initial\ weight\ of\ sample} \times 100 = 3.7\% \quad (3)$$

2.2. Determination of Functional Groups and Compounds

2.2.1. Fourier-Transform Infrared (FT-IR) Spectroscopy

To determine functional groups, an Agilent Technologies FT-IR spectrometer was utilized. The infrared spectrum of each sample was characterized using the potassium bromide disc method. The scanning range employed was from 650 to 4000 cm^{-1}. This facilitated the identification of functional groups in the crude Turmeric extract.

2.2.2. Gas Chromatography-Mass Spectrometry (GC-MS)

Agilent Technologies 7890B GC and 5977A MSD machines were employed for gas chromatography and mass spectrometry. A fused silica column packed with Elite-5MS (5% biphenyl and 95% dimethylpolysiloxane, 30 mm × 0.25 mm internal diameter × 250 µm

film thickness) was used. Components were separated using helium as the carrier gas at a constant flow rate of 1 mL/min. The injector temperature was set at 250 °C during chromatographic execution. A 1 µL extract was injected into the instrument at a temperature of 60 °C for two minutes. Subsequently, the temperature was increased to 300 °C at a rate of 10 °C/min, with a soak time of six minutes at 300 °C. Mass detector conditions included a transfer line temperature of 240 °C, an ion source temperature of 240 °C with electron impact ionization mode at 70 eV, a scan time of 0.2 s, and a scan interval of 0.1 s. Fragment masses ranged from 40 to 600 Da. Finally, the spectra of the components were compared with the database of known spectra stored in the NIST GC-MS library.

2.3. Blending of Biolubricant with Antioxidant

The weighed turmeric rhizome extract was dissolved in methanol; then, these were added to the biolubricant (synthesized from Shea butter) according to a design experiment and stirred for 10 min. The biolubricant range was between (85, 90 and 95 mL), the turmeric rhizome extract was between (15, 10 and 5 mL) and the reaction temperatures ranged between (50, 55, and 60 °C).

2.4. Determination of Peroxide Value (PV) and Pour Point

The biolubricant blended with turmeric additive is characterized by determining the antioxidant capacity and pour point. The peroxide value is a parameter that indicates the oxygen content in the form of hydroperoxides in a compound. This value measures the oxidation present in an oil. This test was performed according to a procedure outlined by [32]. An oil sample of 2.0 g was transferred into a 250 cm^3 flask, and 1 g of powdered potassium iodide (KI) and a solvent mixture (2:1 of glacial acetic and trichloromethane) were then added. For complete dissolution, the solution was placed in a water bath for five minutes. An amount of 20 cm^3 of potassium iodide 50% was added and then titrated with 0.1 M $Na_2S_2O_3$; a starch solution was used as an indicator. Using the same conditions, a blank experiment was also conducted. The peroxide value was given by Equation (4), with R and B as the titer values of the oil and blank samples, respectively, and W as the weight of oil.

$$Peroxide\ value = \frac{(R \times B) \times Molarity\ of\ Na_2S_2O_3}{W} \quad (4)$$

The pour point was determined according to ASTM 97 standard. The test jar was filled with the blended biolubricant and placed in a cooling media (water bath). The temperature was measured in 3 °C decrements until the oil stopped pouring.

2.5. Statistical Analysis

Results were expressed as mean ± standard deviation of at least three repeated tests. Analysis of Variance (ANOVA) statistical significance was determined using Microsoft Excel statistical software 2021. The p-values < 0.05 were considered to be significant.

3. Results and Discussion

3.1. Fourier-Transform Infrared Spectroscopy (FTIR) of Crude Turmeric Extract

Figure 1 shows the FTIR spectra of *Curcuma longa*. The valleys in the spectrum of the figure provide information about the composition and molecular structure of the sample being analyzed.

O-H band occurs at (3336 cm^{-1}), C-H asymmetrical and symmetrical stretch at (2940 and 2834 cm^{-1}), C=C cyclic ring at (1680–1515 cm^{-1}), C-H methyl rock at 1342 cm^{-1}, C-O ester at (1278 and 1126 cm^{-1}). Nasibi et al. [16] confirmed characteristic bands at 3408, 2928, 1607 and 1314 cm^{-1} that are related to the -OH, -CH, C=C and C-O groups, respectively. A measurement of 1021 cm^{-1} was assigned to the vibration of C-O-H distortion and 3400 to 3600 cm^{-1} to O-H stretching [33]. A wide band O-H group was seen between (3594.85–3262.40 cm^{-1}) [34].

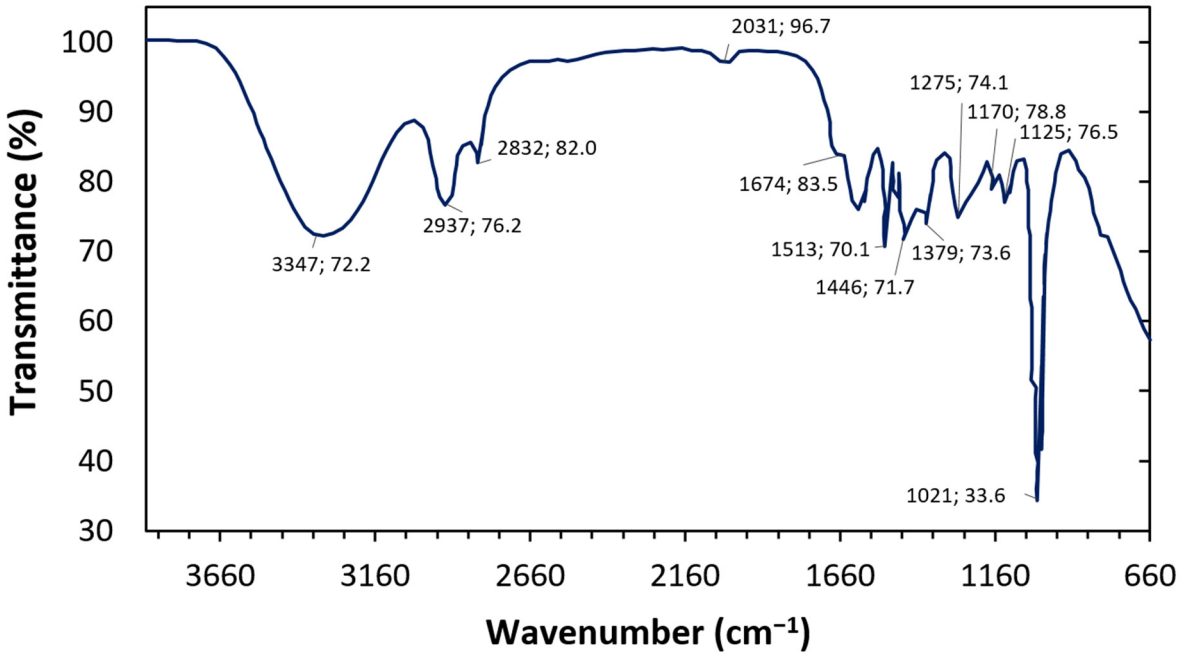

Figure 1. FTIR spectra of *Curcuma longa*.

Based on Table 1, it can be concluded that alcohols, alkanes, alkenes, esters and cyclic rings are present in turmeric rhizome extract.

Table 1. Functional groups in Turmeric rhizome extract.

Wavelength (cm^{-1})	Functional Group
3335	O-H medium stretch (alkanols)
2940, 2834	C-H medium stretch (alkanes)
1680, 1515	C=C cyclic ring stretch
1342	C-H medium stretch (terminal alkane bend)
1278, 1126	C-O medium stretch, (ester)
1020	=C-H strong stretch (alkene bending)

3.2. Gas Chromatography-Mass Spectrometry (GC-MS) Analysis

The extract from the turmeric rhizome was insoluble in water but soluble in methanol. Figure 2 presents the results of gas chromatography of the extract. Fourteen major components were identified, as listed in Table 2, accounting for 85.3% of the total area in *Curcuma longa*. The compounds from the extract were phenol, 2 -methoxy-3-(2-propenyl)- (36.3% mass spectra), making the maximum peak area followed by Turmerone (6.98% mass spectra), 2-Methyl-6-(4-methylenecyclohex-2-en-1-yl)hept-2-en-4-one (5.50% mass spectra), Trans-Isoeugenol (5.14% mass spectra), Cis-13-Octadecenoic acid, methyl ester (4.85% mass spectra), Ar-tumerone (4.68% mass spectra), (Z,Z)-.alpha.-Farnesene (4.42% mass spectra), Phenol, 2-methoxy-4-(2-propenyl)-, acetate (3.84 mass spectra), Hexadecanoic acid, methyl ester (3.06% mass spectra), Methyl stearate (2.42% mass spectra), Ethyl Oleate (2.28% mass spectra), Hexadecanoic acid, ethyl ester (1.66% mass spectra), 9-Octadecenoic acid (Z)-, methyl ester (1.62% mass spectra), Octadecanoic acid, ethyl ester (1.47% mass spectra), (E)-Atlantone (1.09% mass spectra). The presence of Artumerone and curlone have also been confirmed. Based on Table 2, the GC-MS chromatogram identified phenols such as phenol, 2-methoxy-3-(2-propenyl) and phenol, 2-methoxy-4-(2-propenyl)-acetate. The GC-MS result is consistent with the FTIR result that shows the presence of alcohols and cyclic rings. These phenols have been identified in the turmeric rhizome extract. Therefore, phenols have been established to be present in turmeric rhizome extract, although in complex forms.

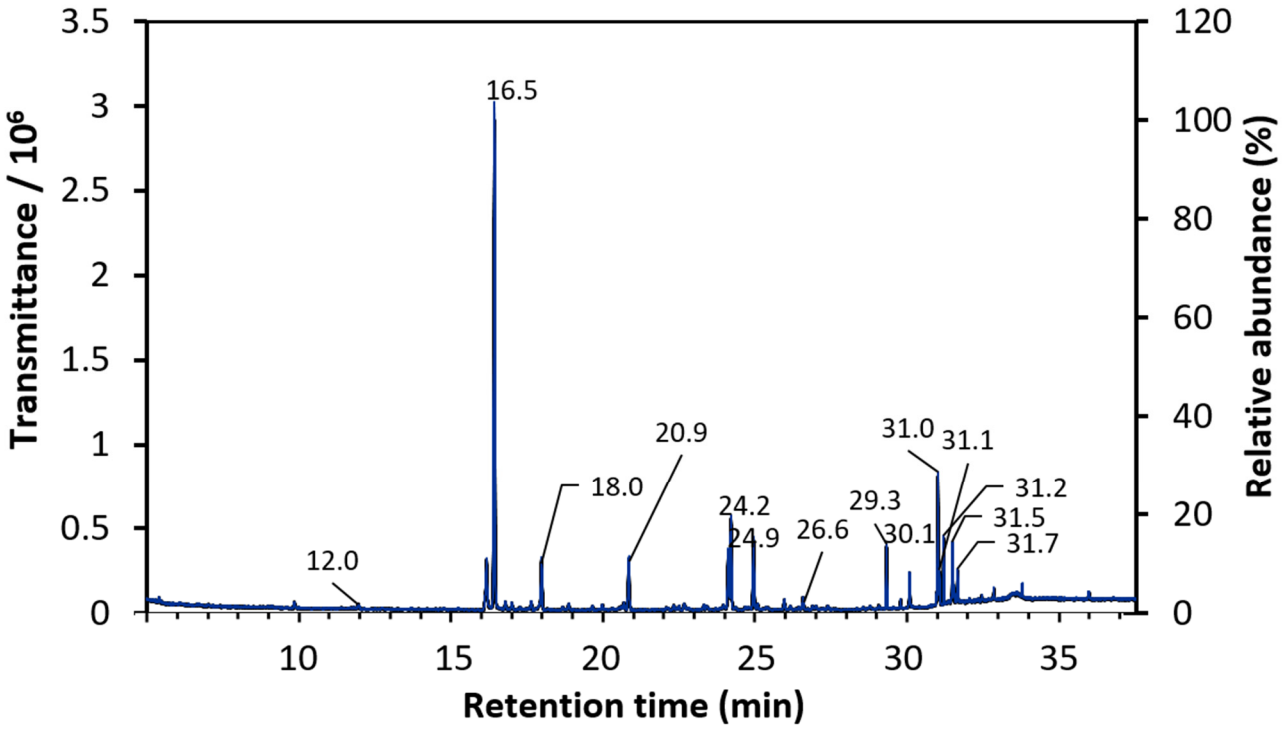

Figure 2. GC-MS analysis of Turmeric rhizome extract.

Table 2. Compounds present in Turmeric rhizome extract.

Peak	Retention Time	Area (%)	Library/ID
5	16.5	36.3	phenol, 2 -methoxy-3-(2-propenyl)
19	24.2	6.98	Turmerone (α-turmerone)
20	24.9	5.50	2-Methyl-6-(4-methylenecyclohex-2-en-1-yl) hept-2-en-4-one (β-turmerone or Curlone)
4	12.0	5.14	Trans-Isoeugenol
28	31.0	4.85	Cis-13-Octadecenoic acid, methyl ester
10	18.0	4.42	(Z,Z)-.alpha.-Farnesene
13	20.9	3.84	Phenol, 2-methoxy-4-(2-propenyl)-, acetate
24	29.3	3.06	Hexadecanoic acid, methyl ester
30	31.2	2.42	Methyl stearate
32	31.5	2.28	Ethyl Oleate
26	30.1	1.66	Hexadecanoic acid, ethyl ester
29	31.1	1.62	9-Octadecenoic acid (Z)-, methyl ester
31	31.7	1.45	Octadecanoic acid, ethyl ester
23	26.6	1.09	(E)-Atlantone

3.3. Peroxide Value of Biolubricant Blended with Turmeric Extract as Additive

The effect of three different contents of turmeric extract (5, 10 and 15 mL) in different base oil contents of 80, 85 and 95 mL were analyzed in terms of peroxide value (Figure 3). The minimum PV was observed at 10 mL of turmeric extract additive. The amount of decrease was 0.51, 0.25 and 0.285 meq/kg in the base oils (85, 90 and 95 mL), respectively, suggesting antioxidant activity. Then, as the extract concentration increases beyond 10 mL, the PV also increases except for the base oil concentration at 85 mL. The amount of increase was 0.33 and 1.49 meq/kg base oils (90 and 95 mL), respectively, suggesting pro-oxidant activity. The decrease in PV is attributed to the liberation of phenols from the cell matrix of the extract to participate in the scavenging process, whereas the increase in PV is due to the weakening of the scavenging power of the extract and hydrolysis. The decrease in peroxide

value observed in this work due to extract addition is consistent with reports by Rafiee et al. [21] and Ebrahimzadeh et al. [6]. However, Delgado et al. [20] reported the highest antioxidation at 1% (*w*/*w*) of extract, which is lower than the concentrations considered in this study. Another study by Mariod et al. [35] revealed general reduction in peroxide value irrespective of extract concentration.

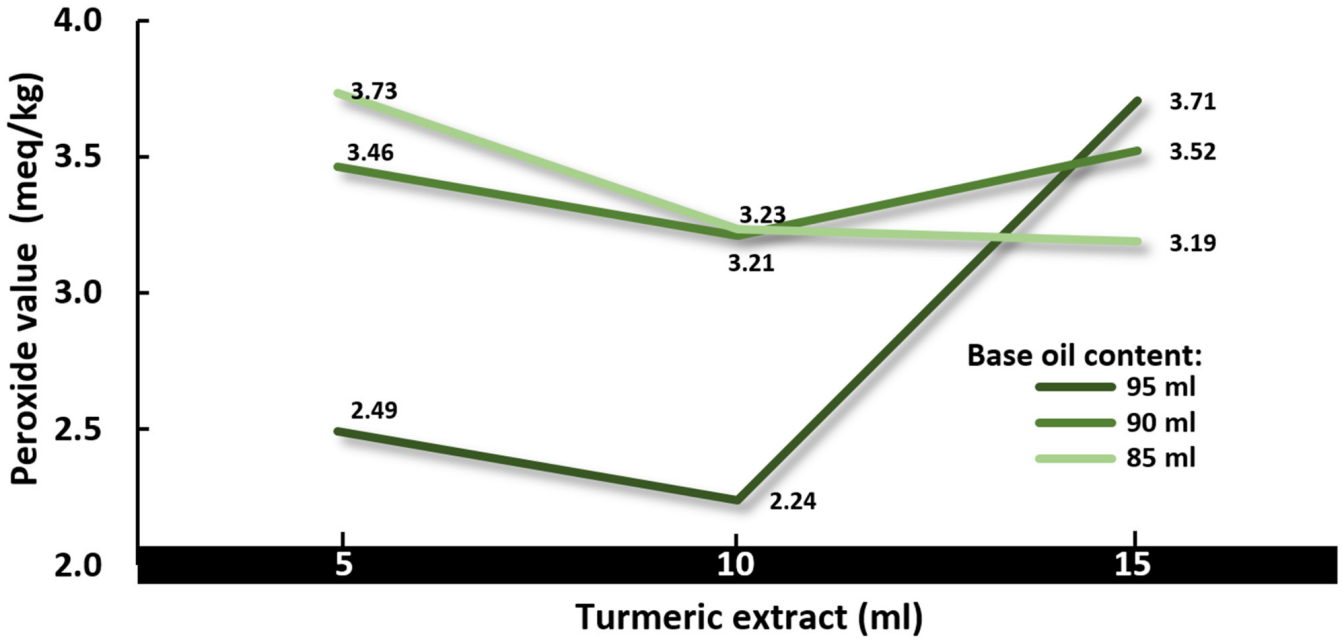

Figure 3. Peroxide values for different volumes of biolubricant blended with three volume contents of turmeric extract additive.

To analyze the relatively low variation of turmeric content in the peroxide value, the data shown in Figure 3 is now presented in Figure 4 in terms of additive content (*v*/*v* %). In addition, ANOVA is performed in Table 3 from data displayed in Figure 4. The comparison between the turmeric extract concentration in the base oil indicated that the antioxidant effect of the turmeric rhizome extract on the biolubricant was insignificant to the variation of turmeric concentration in the range between 5 to 20% turmeric concentration, as observed in the peroxide values. A similar trend was reported by Farag et al. [7], where the antioxidant effect of residual phenolic compounds was statistically insignificant on sunflower oil stability because the phenolic fractions were not considered free. However, in the analyzed range between 5% to 12.5% (5:95 and 1:8) of turmeric concentration in biolubricant, a *p*-value obtained lower than 0.05 confirms that turmeric extract reduced peroxide value in the biolubricant.

Table 3. One-way ANOVA of Turmeric concentration effect in biolubricant different ranges.

Turmeric Concentration (% *v*/*v*)	Source of Variation	Sum of Squares	Degree of Freedom	Mean of Squares	*F*	*p*-Value	*F* Critic
Between 5 to 20	Between Groups	0.5247	2	0.262	0.939	0.441	5.143
	Within Groups	1.676	6	0.279			
	Total	2.201	8				
Between 5 to 12.5	Between Groups	1.479	2	0.739	11.8	0.0379	9.552
	Within Groups	0.187	3	0.062			
	Total	1.677	5				

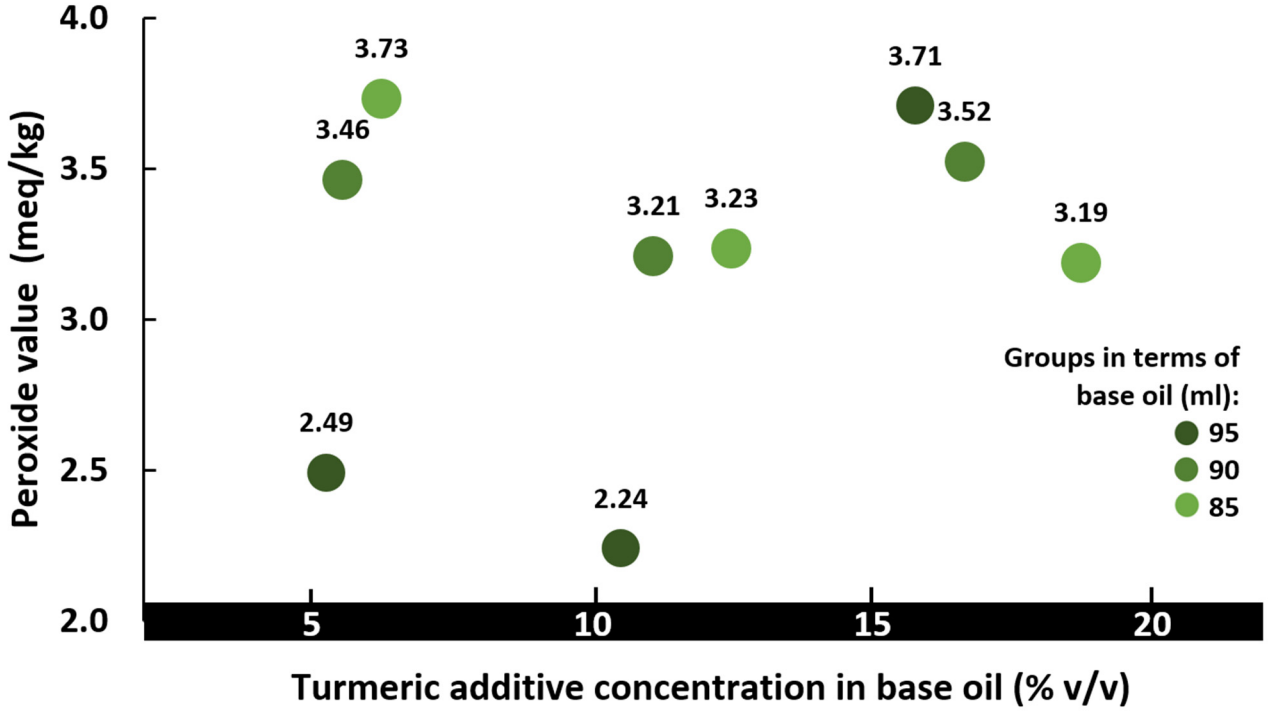

Figure 4. Peroxide values for different turmeric additive concentrations in biolubricant are shown for different groups of samples.

3.4. Pour Point Response of Different Blends with Turmeric Additive

Vegetable oils with higher unsaturated fat contents have lower pour points due to chains in the bent configuration, as observed by Syahir et al. [36]. At low temperatures, plant oils have a tendency to form macro crystalline structures through uniform stacking of the bent triglyceride backbone. Such macro-crystals restrict the easy flow of the system due to the loss of kinetic energy of the individual molecules during self-stacking. It can be assumed that the presence of a large branching group at the mid-point of a fatty acid chain creates a steric barrier around the individual molecule and inhibits crystallization, the result of which is lower pour, as shown in Figure 5.

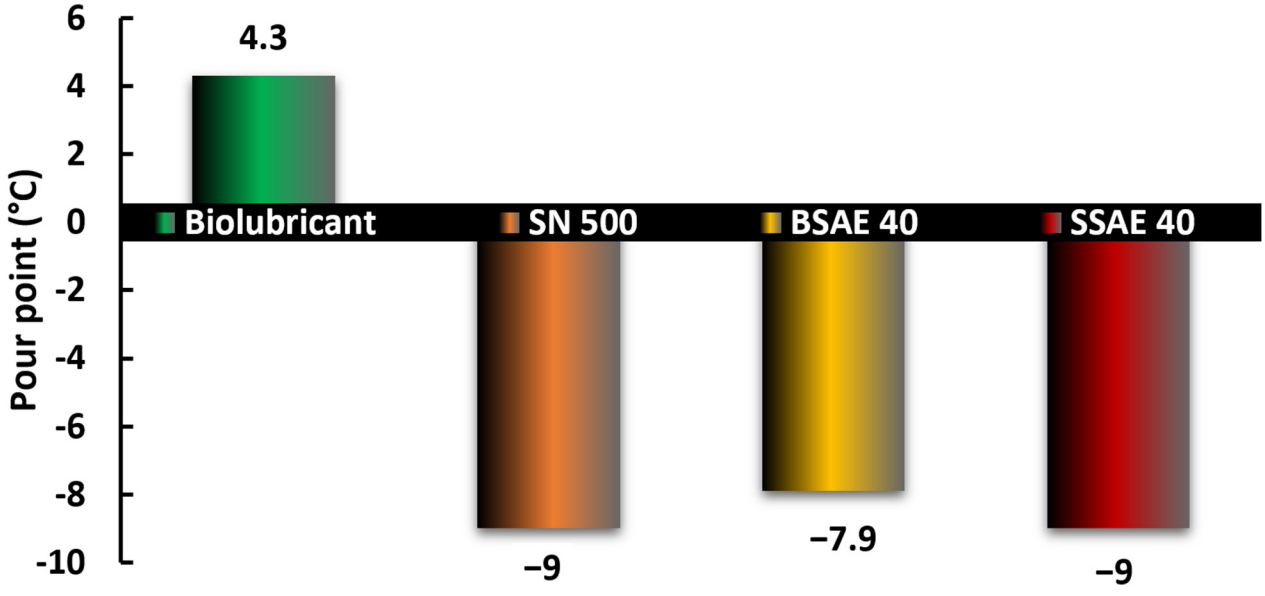

Figure 5. Pour point plots of different blends.

Blending the biolubricant and SN 500 mineral oil with turmeric extract additive to formulate an SAE 40 grade lubricant yielded a pour point of −7.9 °C. The pour point of the blended SAE 40 (BSAE 40) showed a significant improvement, as it is 12.2 °C lower than the biolubricant pour point and only 1.1 °C higher than SN 500 mineral oil, indicating successful blending of the oils. However, comparing the value with the standard pour point value for SAE 40 (SSAE 40), which is −9 °C, more is demanded from the turmeric extract additive to depress the pour point in order to meet industry standards effectively. Without this enhancement in the pour point, BSAE 40 may not be suitable for cold regions with temperatures below −7.9 °C. This makes the blended lubricant better suited for use in hot temperate regions rather than colder climates. This study is consistent with Aluyor and Audu [3], who reported a reduction in the pour point of soybean oil from −1 to −6 due to 2% additive addition

4. Conclusions

The capacity of phenols in turmeric rhizome extract for application as an antioxidant and pour point depressant in biolubricants was investigated. From the analysis of results, the following conclusions are drawn:

The FTIR technique identified the O-H band at (3336 cm^{-1}), C-H asymmetrical and symmetrical stretch at (2940 and 2834 cm^{-1}), C=C cyclic ring at (1680–1515 cm^{-1}) in the turmeric rhizome extract. This result showed the presence of alcohols, alkanes and cyclic ring functional groups in the turmeric rhizome extract.

- The GC-MS technique detected phenol, 2 -methoxy-3-(2-propenyl) and Phenol, 2-methoxy-4-(2-propenyl)-, acetate having 36.3% and 3.8% area, respectively. The phenols observed in this study are not in their pure state, but rather in combination with other atoms.
- The addition of the turmeric rhizome was observed to have a substantial impact on decreasing the pour point, and also decreasing the peroxide values for a proportion range between 5 to 12.5% turmeric additive content (5:95 to 1:8) with biobased shea oil.

Even if the evidence presented here shows the high potential of turmeric extract for antioxidant and pour point depressant additive for bio-based lubricants, using an edible source [25,26] and a medicinal stem [29,37,38] for any kind of industrial product is highly objectionable and cannot be considered sustainable in the true sense. Industries use vegetable products for biodegradable products, but all those are from non-edible sources. Encouraging the usage of edible items for industrial usage will create an imbalance in the economy. Therefore, there is a need to explore new sources of non-edible stems of similar family species for industrial application and mass production.

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