Modification of Vegetable Oils for use as Industrial Lubricants

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ABSTRACT

There has been a lot of interest in using vegetable oils (particularly soybean oil) as renewable raw materials for new industrial products including lubricants. This emphasis on environmentally friendly lubricants is largely due to the rapid depletion of world fossil fuel reserves and increasing concern for environmental pollution from excessive mineral oil use. Vegetable oils are promising candidates as base fluid for eco-friendly lubricants, but their use is restricted due to low thermo-oxidative stability and poor cold flow behavior. This paper presents use of two different approaches, i.e. genetically modified oils and chemically modified oils in combination with additives to improve the oxidation and cold flow behavior of vegetable oils. The oxidation behavior was studied using Pressure Differential Scanning Calorimetry (PDSC) and Rotary Bomb Oxidation Test (RBOT), while the low temperature property was studied using pour-point measurements. The combination of high-oleic vegetable oils and chemical additives offer the best option for achieving the ultimate goal. The lubricants formulated using high-oleic vegetable oils exhibit superior oxidative stability, and improved low temperature properties compared to some of the commercially available bio-based industrial oils, and are at par with petroleum-based lubricants.

Key-words: Vegetable oil, high-oleic, PDSC, RBOT, chemically modified, additives

INTRODUCTION

There has been a constant demand for environmentally friendly lubricants. A significant lubricant market of some 9 million metric tons per year of industrial and automotive lubricants exists (Lubricants Industry Sourcebook, Lubes 'n' Greases, 2004-2005). Currently, vegetable oils provide only about 0.1% of the lubricant market. Farm Bill of 2002 provides an opportunity to accelerate the development of this market (Farm Security and Rural Investment Act, published January 11, Federal Register). Section 9002 includes language directing all Federal Government Agencies to give preference to "Biobased" products, unless it is unreasonable to do so, based on price, availability or performance. Lubricant is one category specified in the guidelines to be published. Also due to strict environmental regulations and a concern for depletion of world fossil fuel reserves, there has been a constant demand for environmentally friendly lubricants (Rhee, 1996). There is an increasing concern for environmental pollution from excessive petroleum based lubricants use and their disposal especially in loss lubrication, military applications, and in outdoor activities such as forestry, mining, railroads, dredging, fishing and agriculture hydraulic systems. Over 60 % of the lubricants used in the U.S. are lost to the environment. Vegetable oils with high oleic content are considered to be potential candidates to substitute conventional mineral oil-based lubricating oils and synthetic esters (Randles and Wright, 1992; Asadauskas et al., 1996). Vegetable oils are preferred over synthetic fluids because they are renewable resources and cheaper.

Vegetable oils as lubricants are preferred because they are biodegradable and non-toxic, unlike conventional mineral-based oils (Randles and Wright, 1992; Battersby et al., 1998). They have very low volatility due to the high molecular weight of the triglyceride molecule and have a narrow range of viscosity changes with temperature. Polar ester groups are able to adhere to metal surfaces, and therefore, possess good boundary lubrication properties. In addition, vegetable oils have high solubilizing power for polar contaminants and additive molecules.

On the other hand, vegetable oils have poor oxidative stability (Gapinski et al., 1994; Becker and Knorr, 1996) primarily due to the presence of bis allylic protons and are highly susceptible to radical attack and subsequently undergo oxidative degradation to form polar oxy compounds. This phenomena result in insoluble deposits and increases in oil acidity and viscosity. Vegetable oils also show poor corrosion protection (Ohkawa et al., 1995). The presence of ester functionality renders these oils susceptible to hydrolytic breakdown (Rhodes et al., 1995).

Therefore, contamination with water in the form of emulsion must be prevented at every stage. Low temperature study has also shown that most vegetable oils undergo cloudiness, precipitation, poor flow, and solidification at -10°C upon long-term exposure to cold temperature (Rhee et al., 1995; Kassfeldt and Goran, 1997) in sharp contrast to mineral oil-based fluids.

These physical and chemical properties can be improved either using genetically modified oils or chemically modified oil with suitable combination of additives. Oleic acids are more thermally stable than polyunsaturated fats, and therefore are highly desired component in vegetable oils for lubricant applications. Breeding successes helped to develop high oleic vegetable oils, which are now available for commercial uses. On the other hand, modification of the oil through chemical processing to improve oxidation stability and low temperature fluidity is also possible. These two options in combination with chemical additive offer the greatest opportunity for achieving the ultimate goal. This study presents use of these two approaches to improve the oxidation and cold flow behavior of vegetable oils for use in lubricant applications.

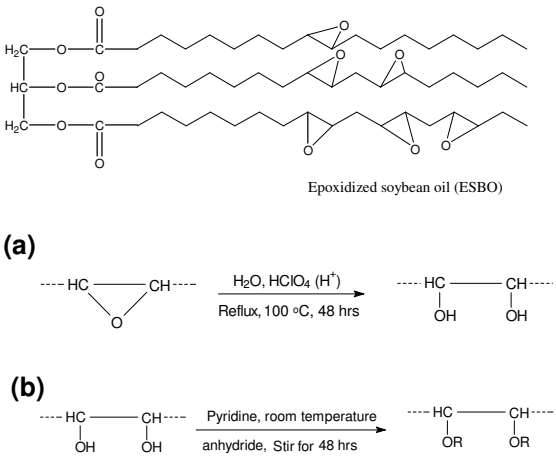
Experimental

MATERIALS

Soybean oil (SO) used in this study was alkali refined (ADM Packaged Oils, Decatur, IL) and used without any further purification. High-linoleic soybean oil (HLSO), mid-oleic soybean oil (MOSO) from United Soybean Board, high-oleic soybean oil (HOSO) from Optimum Quality Grains, LLC, West Des Moines, IA, and poly alphaolefin (PAO) from BP, Naperville, IL were used in this study. The epoxidized soybean oil (ESBO, 100% epoxidized, ~ 4.2 epoxy groups per triacylglycerol molecule) was obtained from Elf Atochem Inc. (Philadelphia, PA) and used without any further purification. All other chemicals including perchloric acid ($HClO_4$, 70%, ACS reagent), and acid anhydrides were obtained from Aldrich Chemical (Milwaukee, WI), and used without further purification.

SYNTHESIS

The chemically modified soybean oils (CMSBO) were prepared using two-step reaction as described in detail in our previous work (Sharma, 2006). The nucleophilic attack by water molecule on the oxirane ring of ESBO in presence of perchloric acid results in ring opened product DiOH-SBO, as shown in Scheme 1a. In the second step, the dihydroxy derivative is reacted with acetic, butyric, isobutyric and hexanoic anhydride to yield the diesters Ace-SBO, But-SBO, Isobut-SBO and Hex-SBO respectively (Scheme 1b). Both reactions were monitored using FTIR. Diester derivatives were characterized using FTIR, GPC, ^{1}H and ^{13}C NMR (Sharma, 2006). TGA Noack volatility of CMSBO samples were obtained using TGA (model Q 500, TA Instruments, New Castle, DE) as per ASTM D 6375-99a method.



Scheme 1 (a) Epoxy ring opening of ESBO with formation of dihydroxy derivative; (b) Reaction with anhydride to form diester derivatives Ace-SBO if R= $-COCH_3$; But-SBO if R= $-COCH_2CH_2CH_3$ (R1); Isobut-SBO if R= $-COCH(CH_3)_2$ (R2); Hex-SBO if R= $-COCH_2CH_2CH_2CH_2CH_3$ (R3).

OXIDATION METHODS

PDSC: The PDSC experiments were done using a DSC 2910 thermal analyzer from TA Instruments (New Castle, DE) attached to a computer. Typically 2 µL sample was taken in a hermetically sealed type aluminum pan with a pinhole lid, and dry air (Gateway Airgas, St. Louis, MO) was pressurized in the module at a constant pressure of 1379 kPa (200psi), and heated at a rate of 10 °C/min during the length of the temperature ramping experiment. The onset (OT) and signal maxima temperatures (SM) of oxidation were calculated from the exotherm in each case. The oxidation stability of various soybean oils was also studied by measuring their oxidation induction time (OIT) using isothermal experiment at 200°C in constant pressure mode at 200 psi with flow rate of 20mL/min.

RBOT: Rotary bomb oxidation tests (RBOT) of all the oil formulations were carried out as per ASTM D 2272 method in presence of copper catalyst at 150°C in dry conditions. In RBOT the vessel is charged with oxygen to 90 psi pressure, and rotated axially in a constant temperature oil bath set at 150°C. The pressure in the bomb was recorded with time and RBOT time is the time at which the maximum pressure of bomb has dropped by 25.4 psi. This RBOT time usually coincides with an induction-type period of rapid

pressure drop due to consumption of higher amounts of oxygen and corresponds to the time when the rate of oxidation of lubricant in the bomb is maximum and thus signals the depletion of antioxidant. With this test, in the absence of added antioxidants, soybean oil demonstrates RBOT time of 14 min.

POUR POINTS: Pour points were measured by following the ASTM D97 method. The pour point is defined as the temperature in ℃ when the sample still pours when the jar is tilted. Statistically the method has shown quite good consistency for determining low temperature flow property of fluids.

Results and Discussion

EFFECT OF CHEMICAL MODIFICATION ON PROPERTIES OF VEGETABLE OILS: The TGA profiles of CMSBO samples showed that these are thermally as stable as ESBO even at high temperatures. Noack volatilities were also determined as per ASTM D6375 method using TGA and shown in Table 1. Noack volatilities for all the samples are less than 3%, which are excellent compared to 10-30% in mineral oils and synthetic esters.

PDSC experiments measured the oxidation stability of CMSBO samples. This test is widely used in the lubricant industry and considered reliable for base oils as well as finished lubricants (Sharma and Stipanovic, 2003). The PDSC results, OT and SM, for CMSBO samples are shown in Table 1. The CMSBO samples showed higher oxidation stability than dihydroxy derivative and also SBO. This shows that oxidation stability has considerably improved due to the removal of unsaturation. The oxidation onset temperature decreased with the chain length of the branching group. Ace-SBO has the highest value (174°C) of OT, while Hex-SBO has the lowest (161°C). Longer side chains are more prone to oxidative cleavage than small compact ones. This observation is supported in a study on synthetic esters (Randles, 1999), wherein, they mentioned that short chain acids are more stable than long chain acids.

Table 1: TGA Noack volatility and PDSC data of dihydroxy and diester products at 10℃/min in air under 200 psi pressure.

Test fluids	TGA-Noack volatility, %	OT, ℃	SM, ℃
SBO	2.6	173	193
DiOH-SBO	3.6	163	213
Ace-SBO	2.7	174	213
But-SBO	2.5	169	208
Isobut-SBO	3	157	241
Hex-SBO	2.4	161	234

Onset temperature (OT), Signal maximum temperature (SM) values are average of 3 experiments with standard error of ± 1 ℃.

Antioxidant additives are used to further improve the oxidation stability of these CMSBO samples. Different antioxidants were tried with their varying concentration in SBO. The best additive combination was then used 4% (w/w) in CMSBO samples. The effect of these antioxidants was studied using PDSC temperature ramping and isothermal experiments (Sharma and Stipanovic, 2003). The results obtained are shown in Table 2. It was found that the antioxidant additive responded very well to these CMSBO samples and increased their OT by 80-100°C.

A modified PDSC method was also used to assess the oxidative stability of formulated products. In this method the oil is heated in air atmosphere at 200°C and the time at which oxidation starts was determined as oxidation induction time (OIT). This method is more sensitive to see the subtle differences in oxidation stability of oils (Sharma and Stipanovic, 2003). The OIT data also suggested that the antioxidant additive could prevent the start of oxidation for at least 50 min at 200°C. Both OT and OIT of these CMSBO samples are higher compared than SBO. Even mineral oil based formulations have an OIT of 20min at 200°C (Brian et al., 2004). This shows that chemical modification of SBO along with suitable antioxidant additive can improve their oxidation properties comparable to mineral oils. The effect of chain length on oxidation stability of inhibited CMSBO samples is similar to that for neat CMSBO samples, i.e. decreasing oxidation stability with increasing chain lengths of substituent on CMSBO.

Table 2: PDSC temperature ramp at 10℃/min in air under static 200 psi pressure, and isothermal experiment at 200℃ (SBO at 170℃) under 200 psi pressure with 20mL/min flow of air, for diester SBO derivatives with additives (4% w/w).

Test fluids	OT[a], ℃	OIT[b], min
SBO	192	25.7[c]
ace-SBO	256	75.3
But-SBO	247	51.0
Isobut-SBO	255	56.2
Hex-SBO	254	55.6

[a] Onset temperature.
[b] Oxidation induction time.
[c] Isothermal experiment at 170°C

The cold flow property of vegetable oils is extremely poor and this limits their use at low operating temperature especially as automotive and industrial fluids. Vegetable oils have a tendency to form macro crystalline structures at low temperature through uniform stacking of the 'bend' triglyceride backbone. Such macro crystals restrict the easy flow of the system due to loss of kinetic energy of individual molecules during self-stacking. Cold flow properties of these samples were determined using their pour points. The pour point of all these CMSBO samples is -3°C (ESBO is 0°C). Excellent thermal and oxidative stability of CMSBO samples as a result of removal of unsaturation, however, doesn't significantly improve cold flow properties. Therefore, these samples are good for use in formulating high temperature lubricants, but for use in low temperature applications, suitable pour point depressant (PPD) along with diluents (such as PAO, dibutyl adipate,) will be required. The purpose of PPD is to hinder crystallization of CMSBO molecules at low temperature by further disrupting the stacking mechanism. Use of high oleic sunflower oil as diluent and an additive pack containing an antioxidant, antiwear and PPD, could improve the pour point of CMSBO significantly (-18 °C). This fully formulated lubricant using CMSBO as base fluid has excellent oxidation stability (PDSC onset temperature of 243°C). Rotary bomb oxidation test (RBOT) is another rigorous measure of oxidation stability of finished lubricants. RBOT time of 135 min was obtained for CMSBO formulated lubricant, which is much higher than commercially available soy based lubricants (83 min).

EFFECT OF GENETIC MODIFICATION ON PROPERTIES OF VEGETABLE OILS: Earlier studies on vegetable oils as lubricants (Lal and Carrick, 1994; Honary, 1996; Asadauskas et al., 1996) indicated that increasing the monounsaturated fatty acid, i.e. oleic acid, and decreasing the polyunsaturated fatty acids tend to increase the oxidation stability of the base oil. Therefore the vegetable base oils tested in this study were selected to provide a wide variation in oleic acid percentage (Table 3). The oleic acid varied from 24.2 % in SO to 86.78% in HOSO. The unsaturation number (UN) shown in Table 3 was calculated to indicate the relative amounts of saturated, monounsaturated and polyunsaturated fatty acids in the vegetable oil. The greater the UN the greater is the degree of unsaturation in the vegetable oil.

Table 3: Fatty acid composition of various soybean oils by GC (AACC, 58-18, Approved methods of the American Association of Cereal Chemists, 10th edition, vol. II, 2000). **Palmitic $C_{16:0}$, Stearic $C_{18:0}$, Oleic $C_{18:1}$, Linoleic $C_{18:2}$, Linolenic $C_{18:3}$.**

Oils	$C_{16:0}$	$C_{18:0}$	$C_{18:1}$	$C_{18:2}$	$C_{18:3}$	UN
SO	11.1	4.8	24.2	53.6	6.3	1.5
HLSO	10.6	5.6	27.5	56.3		1.4
MOSO	9.1	4.3	60.7	24.2	1.7	1.1
HOSO	7.1	3.3	86.8	1.1	1.7	0.9

The oxidation stability of various neat soybean oils was determined as their onset temperatures using PDSC and is shown in Figure 1. The oxidation stability of neat base oils increased as the degree of unsaturation decreased, which concurs the previous research. The correlation coefficient value (R-square) of 0.993 was obtained for correlation between unsaturation number and onset temperatures of vegetable oils determined using PDSC. The correlation between UN and different fatty acid percentages didn't yield very high R-square values. The maximum value (0.93) of R-square was obtained between UN and oleic acid percentage. This shows that oxidation stability of soybean oil is well predictable using its fatty acid composition rather than on individual fatty acid percentage. PAO is oxidatively more stable base fluid with onset temperature of 188°C compared to 173°C of SO. Therefore PAO was blended in various soybean oils in 20-40% range. There is an increase in oxidation stability and improvement in cold flow property on adding 20% PAO, but higher amount of PAO doesn't help much. Therefore all these soybean oils were tested for their oxidation stability and cold flow property using 20% PAO.

Effect of antioxidant-antiwear additive mixture*:* From the synergism study of one antioxidant additive, zinc diamyl dithiocarbamate (ZDDC) with three antiwear additives in soybean oil (SO), it has been found that the combination of ZDDC and antimony dialkyldithiocarbamate (ADDC) as antiwear additive provides the best result. ZDDC was selected because of its outstanding performance as antioxidant in vegetable oils as reported earlier. ZDDC has been reported to functions both as radical scavenger and hydroperoxide decomposer that converts the hydroperoxides formed during the oxidation process to non-radical products, thus preventing chain propagation.

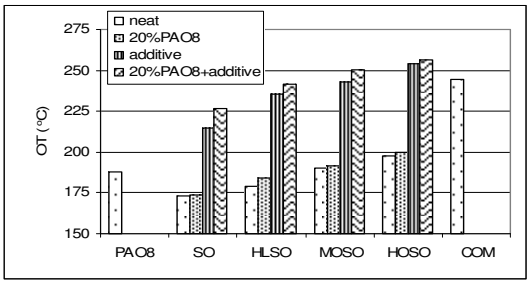


Figure 1. PDSC onset temperatures (OT) of various soybean oil (SO, HLSO, MOSO, and HOSO), and their PAO blends with and without additive mixture.

The effectiveness of this additive combination was tested in various soybean oils (SO, HLSO, MOSO, and HOSO) and their 20% PAO blends. The 20% PAO blends were used because of their better low temperature properties. The PDSC onset temperatures of inhibited soybean oils and their PAO blends are shown in Figure 1. The additive combination has increased the oxidation stability of all the soybean oils by 50°C. Also the difference between soybean oil and PAO blends are more visible in inhibited samples.

The Oxidation Induction times (OIT) of various inhibited soybean oils and PAO blends were obtained using isothermal PDSC experiment and are shown in Figure 2. There is a large difference in OIT of SO and HLSO and again when oleic acid content increased two times in MOSO compared to HLSO, the OIT also doubled to 26 min from 13.4 min. Another fold increase in oleic acid from MOSO to HOSO, increased the OIT further to 73 min. The difference in OIT of inhibited oil and PAO blend is maximum in HOSO, which shows that PAO responds better in HOSO than other oils to increase the oxidation stability.

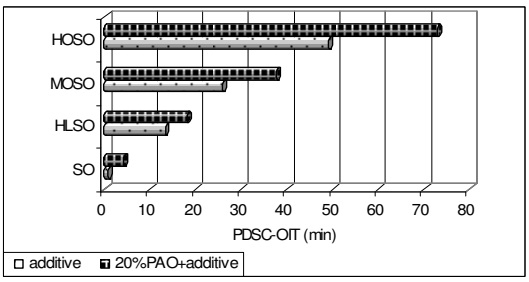


Figure 2. Oxidation induction time (OIT) of various soybean oil (SO, HLSO, MOSO, and HOSO) and their PAO blend based lubricant formulations containing additive mixture using PDSC isothermal experiment.

The RBOT results of soybean oil and soybean oil-PAO blend based lubricants are shown along with commercially available biodegradable hydraulic fluid (COM), as standard in Figure 3. RBOT time of 100 to 400 min (considered good) can be achieved with proper combination of additives. The oxidation stability increased more from SO (109 min) to HLSO (233 min), than from HLSO to MOSO (284 min) although oleic acid content increased more in the latter case than former. The reason for more increase in HLSO may be due to negligible amount of linolenic acid present in HLSO compared to SO. Further increase in oleic content from MOSO to HOSO resulted in RBOT time of 469 min. The similar trend was obtained in soybean oil-PAO blend based lubricants. The increase in oxidation stability on blending PAO was maximum in HOSO (304 min) followed by MOSO (91 min), HLSO (70 min) and SO (36 min). This PAO response on various soybean oils using RBOT is similar to that obtained in PDSC isothermal experiments. This data suggested that a moderate increase in oxidation stability of normal soybean oil (SO) can be achieved by either decreasing the linolenic content and increasing slightly oleic and linoleic acid (HLSO), or increasing oleic acid content two times (MOSO), and a higher increase in oxidation stability using HOSO along with suitable additive combination and PAO blending.

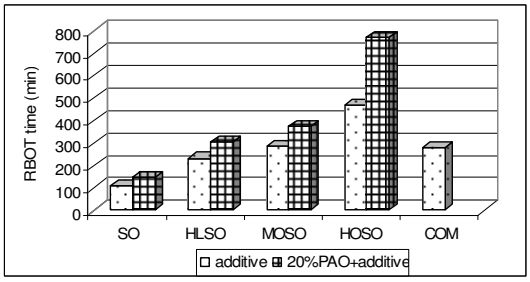


Figure 3. RBOT times of various soybean oil (SO, HLSO, MOSO, and HOSO) and their PAO blend based lubricant formulations containing additive mixture.

<u>Comparison with commercial lubricant</u>: The oxidation behavior of commercially available biodegradable hydraulic fluid (COM) is shown in Figures 1 and 3 along with soybean oil based lubricants formulated in this study. HLSO-PAO blend, HLSO and MOSO showed almost same onset temperature (Figure 1) as COM, while MOSO-PAO blend, HOSO, and HOSO-PAO blend are oxidatively more stable than COM (244°C) as shown by PDSC ramping experiment. RBOT results showed that HLSO-PAO blend (303 min), MOSO (284 min), MOSO-PAO blend (375 min), HOSO (469 min), and HOSO-PAO blend (773 min) have excellent oxidation stability compared to COM (277 min). The additive combination used in current study has given the SO (109 min) and SO-PAO blend (145 min) formulations also a higher

oxidation stability compared to commercially available soy based lubricants (83 min).

Low temperature improvement using PAO and PPD: SO, HLSO, and MOSO have a pour point of -9℃, whereas HOSO freezes at -21℃ (Figure 4). The excellent thermal and oxidative stability of HOSO due to removal of unsaturation (low unsaturation number), also results in improved cold flow property, but still limits its use in low temperature applications.

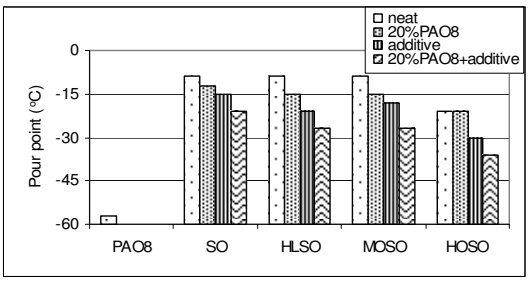


Figure 4. Pour points of various soybean oil (SO, HLSO, MOSO, and HOSO) and their PAO blends with and without pour point depressant (additive).

In order to improve cold storage stability, poly alpha olefin (PAO) having pour point of -57℃ was added as a diluent with various soybean oils. 20, 30 and 40% PAO blends were prepared through blending and pour point was determined in each case. Addition of 20% PAO to various soybean oils SO, HLSO, and MOSO improved their pour point slightly by -3 to -6℃, but no difference was observed in HOSO. Further addition of PAO to 40% made no significant improvement in the pour point, therefore, only 20% PAO-blends were tried for final formulations. Also use of higher PAO amounts (40% PAO blends) will decrease the biodegradability of final formulations. Further improvement in the cold flow property of soybean oil and soybean oil-PAO blends can be achieved by using suitable pour point depressant (PPD) additives. The PPD concentration was optimized by determining the pour points of several blends. An optimum PPD concentration of 1% in final formulations of various soybean oils and soybean oil-PAO blends significantly improved the cold flow behavior (Figure 4). The response of PPD is more visible in HLSO, MOSO, and HOSO, where pour point decreased by -12, -9, and -9℃ respectively, while in SO, pour point decreased by -6℃. The PPD response is slightly strong in PAO blends, where in pour point decreased by -15℃ in HOSO-PAO blend, -12℃ in HLSO- and MOSO-PAO blends, and -9℃ in SO-PAO blend. It can be assumed that the presence of more oleic acid in triglycerides of HOSO does not allow individual molecules to come close for easy stacking due to break in symmetry of molecules. This results in the formation of microcrystalline structures rather than macro structures. At lower temperatures, such microcrystalline structures can easily tumble and glide over one another resulting in better fluidity of the total matrix. The optimized formulation containing HOSO, PPD, and PAO diluent had a pour point of -36℃.

Conclusion

The approach of using chemically modified vegetable oils in combination with additives improved the thermo-oxidative stability and cold flow property of biobased lubricants to some extent. The other approach of using genetically modified soybean oils with higher oleic content along with combination of additives and PAO as diluent improved the properties to a much greater extent. The formulations with mid-oleic soybean oil also show excellent thermo-oxidative stability and low temperature flow property when blended with PAO and suitable additive combinations. The lubricants formulated using the later approach exhibit superior oxidative stability, and improved low temperature properties compared to some of the commercially available bio-based industrial oils for heavy duty applications and agriculture equipments.

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