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Oxidation and low temperature stability of vegetable oil-based lubricants

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Abstract

Vegetable oils are promising candidates as base fluid for eco-friendly lubricants because of their excellent lubricity, biodegradability, viscosity–temperature characteristics and low volatility. Their use, however, is restricted due to low thermo-oxidative stability and poor cold flow behavior. This paper presents a systematic approach to improve the oxidation behavior and low temperature fluidity of vegetable oil derivatives. Among the various possible avenues available, the combination of chemical additives, diluent (polyalphaolefin), and high-oleic vegetable oils offer the best option for achieving the ultimate goal. Vegetable oil-based lubricants formulated using the above approach exhibit superior oxidative stability, and improved low temperature properties such as pour points compared to commercially available industrial oils such as bio-based hydraulic fluids. The above vegetable oil-based formulations compare at par with petroleum-based lubricants for use in high-temperature applications and often outperform the competition in some of its properties.

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1. Introduction

The industrial and automotive lubricants have a significant lubricant market of 2.4 billion gallons per year (Lubricants Industry Sourcebook, Lubes 'n' Greases, 2004–2005). Vegetable oils currently provide only about 0.1% of this lubricant market. Due to strict environmental regulations and a concern for depletion of world fossil fuel reserves, there is an increasing demand for environmentally friendly lubricants (Rhee, 1996; Rudnick, 2002; Hwang and Erhan, 2002; Adhvaryu and Erhan, 2002). An opportunity to accelerate the development of this market exists in the 2002 Farm Bill (Farm Security and Rural Investment Act, published January 11, Federal Register). Section 9002 includes language directing all Federal Government Agencies to give preference to "bio-based" products, unless it is unreasonable to do so, based on price, availability or performance. Lubricant is one category specified in this guideline.

Most current lubricants contain petroleum base stocks, which are toxic to environment and difficult to dispose of after use. Environmental concern continues to increase of pollution from excessive lubricant use and disposal, especially total loss lubricants. Over 60% of the lubricants used in the U.S. are lost to the environment. Vegetable oils have a capability to contribute towards the goal of energy independence and security since they are a renewable resource. Vegetable oils with high oleic content are considered to be potential candidates as sub-

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stitutes for conventional mineral oil-based lubricating oils and synthetic esters (Randles and Wright, 1992; Asadauskas et al., 1996).

Vegetable oils as lubricants are environmentally preferred to petroleum-based oil because they are biodegradable and non-toxic (Randles and Wright, 1992; Battersby et al., 1998). Other advantages include very low volatility due to the high molecular weight of the triglyceride molecule and excellent temperature–viscosity properties. Their polar ester groups are able to adhere to metal surfaces, and therefore, possess good lubricity. 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. These active sites are highly susceptible to radical attack and subsequently the molecules undergo oxidative degradation and form polar oxy compounds. This phenomena eventually results 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). Excessive water contamination must be prevented since it can result in the formation of emulsions.

Low temperature studies have 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). Not all vegetable oils possess equivalent chemical and performance properties. Mixing of renewable oils will be required to serve the major market of lubricants. There is limited data available on blending of vegetable oils.

This study presents a systematic approach to improve the oxidation and cold flow behavior of vegetable oil derivatives using a synergistic combination of additives in high-oleic vegetable oil blended with synthetic fluid. The oxidation properties are studied using pressure differential scanning calorimetry (PDSC) and rotary bomb oxidation test (RBOT), while pour point measurements are used to study low temperature properties.

2. Experimental procedure

2.1. Materials studied

Five vegetable oils are used in this study: an alkali refined soybean oil (SB) obtained from ADM Packaged Oils, Decatur, IL, a high-linoleic soybean oil (HLSB) and a mid-oleic soybean oil (MOSB) obtained from the United Soybean Board; a high-oleic soybean oil (HOSB) obtained from Optimum Quality Grains, LLC, West Des Moines, IA; a high-oleic safflower oil (HOSF) and a high-oleic sunflower oil (HOSN) from Spectrum Ingredients, Petaluma, CA. A polyalphaolefin (PAO) from BP, Naperville, IL was used as a low temperature blending stock in this study. All these oils were used as received without any further purification.

A commercially available biodegradable hydraulic fluid (COM) was also used for comparison in the study.

Two additives used in the study, zinc diamyl dithiocarbamate (ZDDC) and antimony dialkyldithiocarbamate (ADDC), were obtained from R.T. Vanderbilt, Norwalk, CT.

2.2. Pressure differential scanning calorimetry (PDSC) method

The PDSC experiments use a computerized DSC 2910 thermal analyzer from TA Instruments (New Castle, DE). Typically a 2 μl sample is placed in a hermetically sealed aluminum pan containing a pinhole lid for interaction of the sample and the reactant gas (dry air). The 2 μl sample results in a film thickness of less than 1 mm. This ensures proper oil–air interaction and eliminates gas diffusion limitation. The module is temperature calibrated using indium metal (m.p. 156.6 °C) and a 10 °C/min heating rate. Dry air (Gateway Airgas, St. Louis, MO) is used to pressurize the module at a constant pressure of 1379 kPa (200 psi). A scanning rate of 10 °C/min was used in the temperature ramping experiments. The onset temperature (OT) of oxidation is calculated from the exotherm in each case.

The oxidation stability of vegetable oil-based lubricants was also studied in the PDSC using the oxidation induction time (OIT) obtained in isothermal experiments at 200 °C in constant pressure mode at 200 psi with air flow rate of 34 ± 3 ml/min.

2.3. Rotary bomb oxidation test (RBOT)

Rotary bomb oxidation tests (RBOT) of all the oil formulations are carried out in presence of copper catalyst at 150 °C in dry conditions as per ASTM Test Method D-2272. In the RBOT test, the vessel is sealed, charged to 90 psi pressure with oxygen, and rotated axially in a constant temperature oil bath set at 150 °C. The pressure in the bomb is continuously recorded. The RBOT time is the time at which the pressure of the bomb has dropped by 25.4 psi.

2.4. Long-term oven oxidation stability test

In this test, the vegetable oils, additized vegetable oils, and their PAO blends are placed into glass sample vials and placed in oven at 85 °C for 28 days. The weight loss/gain were determined and total acid number (TAN) at 0 and 28 days were obtained.

2.5. Pour point method

Pour points are measured by following the ASTM D97 method (ASTM method, 2000). A 50 ml sample is placed in a test jar, stoppered. The stopper contains thermometer measuring from +20 to −80 °C that is immersed into the sample. These test jars are placed in a digital pour point bath (Petrolab, NY). Temperature is measured in 3 °C increments until the sample stops pouring. The pour point is defined as the temperature in °C at which 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.

3. Results and discussion

3.1. Oxidation stability of vegetable base oils

The vegetable base oils used in this study were selected to provide a wide variation in oleic acid percentage (Table 1). The oleic acid varied from 24.2% in SB to 89.1% in HOSN. The unsaturation number (UN) shown in Table 1 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. The oxidation stability of various neat vegetable oils was determined as their onset temperatures (OT) using PDSC and is shown in Table 2. The OT is the temperature at which a rapid increase in the rate of oxidation is observed and obtained by extrapolating the tangent drawn on the steepest slope of reaction exotherm to the baseline (Fig. 1). A high OT would suggest a high oxidation stability of the oil. The oxidation stability of neat base oils increased as the degree of unsaturation decreased, which concurs the previous research (Lal and Carrick, 1994; Honary, 1996; Asadauskas et al., 1996). The correlation coefficient value (R^2) of 0.993 was obtained for correlation between unsaturation number and PDSC onset temperatures of different soybean oils with varying oleic acid content. The correlation between OT and different fatty acid percentages did not yield very high R^2 values. The maximum value (0.93) of R^2 was obtained between OT and oleic acid percentage. This shows that oxidation stability of soybean oil is well predictable using its fatty acid composition rather than individual fatty acid percentage. Among the neat high-oleic vegetable oils, HOSF has poorest oxidation stability compared to HOSB and

Table 1
Fatty acid composition of various soybean oils by GC (AACC, 58-18, approved methods of the American Association of Cereal Chemists, 10th ed., vol. II, 2000)

Vegetable oils	Palmitic C16:0%	Stearic C18:0%	Oleic C18:1%	Linoleic C18:2%	Linolenic C18:3%	UN
SB	11.14	4.77	24.20	53.60	6.29	1.50
HLSB	10.61	5.63	27.49	56.27	–	1.40
MOSB	9.13	4.33	60.71	24.18	1.65	1.14
HOSB	7.08	3.30	86.78	1.08	1.76	0.94
HOSF	4.82	2.03	80.27	12.87	–	1.06
HOSN	3.27	2.86	89.11	4.76	–	0.99

Table 2
PDSC onset temperatures (OT) of vegetable oils and their PAO blends with and without additive mixture

Vegetable oils	Onset temperature (°C)			
	Neat oils	20% PAO8 blend	Neat oils + Additive	20% PAO8 blend + Additive
PAO8	188.0			
SB	172.9	173.9	215.0	226.6
HLSB	179.2	184.6	235.8	241.8
MOSB	190.3	191.5	243.1	250.4
HOSB	197.7	199.7	254.4	256.5
HOSF	177.4	187.9	257.1	260.4
HOSN	193.2	198.5	260	264
COM			244.2	

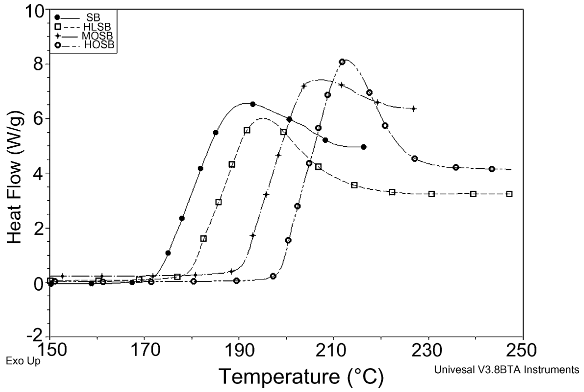

Fig. 1. Typical PDSC exotherm of various soybean oils (SB, HLSB, MOSB, and HOSB) using temperature ramping method (at 10 °C/min in presence of air at 200 psi pressure).

HOSN, which can be explained on the basis of higher linoleic acid content present in HOSF. PAO is an oxidatively more stable base fluid with an onset temperature of 188 °C compared to 173 °C of SB (Table 2). The PAO is used as a diluent in various vegetable oils in 20–40% range. There is some increase in oxidation stability as seen from increased OT (Table 2) and improvement in low temperature stability when adding up to 20% PAO. Higher amounts of PAO did not show further improvement. Also higher amounts of PAO8 will decrease the biodegradability of these oils, as PAO8 itself has 30% biodegradability by OECD 301B method compared to >70% in case of vegetable oils (Lawate, 2002). The choice and concentration of diluent in vegetable oils is thus critical for environmentally friendly lubricants since it dictates the biodegradability and ecotoxicity of finished lubricants. Therefore, all these vegetable oils were tested for their oxidation stability and low temperature stability using 20% PAO. To offset the poor oxidation stability of the vegetable oils in these PAO–vegetable oil blends, it is necessary to optimize the antioxidants and pour depressants used in the blends.

3.2. Effect of antioxidant–antiwear additive mixture

ZDDC is selected because of its outstanding performance as antioxidant in vegetable oils as reported by Becker and Knorr (1996). In a study on the optimization of additives by Sharma et al. (2006), it was established that a mixture of additives is required to substantially improve the oxidation stability of soybean oil. The combination of 2% ZDDC and 2% ADDC provided a synergistic effect and it was concluded that in soybean oil, the combination of ZDDC as the antioxidant and ADDC as the antiwear additive provide the best results. As a result, these additives were selected for use in the vegetable oils in this study.

ZDDC has been reported to function both as radical scavenger and hydroperoxide decomposer (Becker and Knorr, 1996). It converts the hydroperoxides formed during the oxidation process to non-radical products, thus preventing chain propagation. The mechanism of oxidation inhibition by ZDDC involves complex interactions between alkylperoxy radicals, organic hydroperoxides and intermediate decomposition products of initial compounds. The mechanism of oxidation inhibition appears to be very effective. The possible reason for the synergism between antioxidant ZDDC and antiwear ADDC is the multifunctional behavior of ADDC. Although it is an antiwear and extreme pressure additive, it also exhibits antioxidant properties in the RBOT and PDSC. In evaluations of ADDC with soybean oil a time of 30 min compared to 14 min of soybean oil and onset temperature of 185 °C compared to 172 °C were obtained.

The effectiveness of this additive combination was tested in various vegetable oils and their 20% PAO blends. The PDSC temperature ramping method was used to study the effect of the additives on base oils oxidation stability. This method has an advantage that it can be used for oxidation stability of neat base oils as well as additized oils, while the PDSC isothermal method is good only for additized oils. The PDSC onset temperatures of neat vegetable oils, their PAO blends, and additized oils are shown in Table 2. The ZDDC and ADDC additive combination increased the oxidation stability of all the vegetable oils by 50 °C. The differences between different high-oleic oils are also narrowed on use of PAO and the additive combination. Also the difference between vegetable oil and their PAO blends are more visible in additized samples. The additized high-oleic oils have higher OT compared to the commercially available biodegradable hydraulic fluid (COM).

A modified PDSC isothermal method was also used to assess the oxidative stability of formulated products. In this method, the oil is heated in an 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 differences in oxidation stability of additized oils (Sharma and Stipanovic, 2003). The OIT of additized vegetable oils and additized PAO blends are shown in Fig. 2. There is a significant difference in OIT of SB and HLSB. The OIT for MOSB doubled to 26 min from 13.4 min for HLSB, which is due to two-fold increase in oleic acid content of MOSB compared to HLSB. High polyunsaturation (linoleic + linolenic acid content) in triacylglycerol molecule of vegetable oils decreases the oxidation induction time, while high sat-

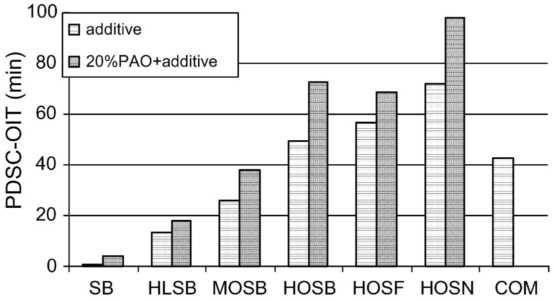

Fig. 2. Oxidation induction time (OIT) of vegetable oils and their PAO blend based lubricant formulations containing additive mixture using PDSC isothermal experiment.

uration and monounsaturation (palmitic + stearic + oleic acid content) increases the OIT of vegetable oil based lubricants. Among the additized high-oleic oils, HOSN has higher OIT compared to HOSB and HOSF. This is attributed to the high oleic fatty acid content and the lowest polyunsaturation content compared to the other two. All three exhibit a higher OIT than the COM. The addition of 20% PAO8 to additized vegetable oils improved the effectiveness of the additives by increasing the OIT through 35%.

The oxidation stability of the vegetable oil based lubricants was also evaluated using the RBOT method. The fluids were evaluated with and without additives. The additives concentrations were similar in all tests. The RBOT time usually coincides with an induction-type period. A rapid pressure drop due to an increase in the rate of consumption of oxygen occurs after a period of time. This signals the depletion of the antioxidant and rapid oxidation of the lubricant. With this test, in the absence of added antioxidants, soybean oil demonstrates RBOT time of only 14 min and an order of magnitude increase with the PAO and additives.

The RBOT results of vegetable oil and vegetable oil–PAO blend based lubricants along with the COM for comparison are found in Fig. 3. With the exception of SB, all of the oils achieved oxidation stability comparable to the COM. The RBOT time increased more from SB (109 min) to HLSB (233 min), than from HLSB to MOSB (284 min) although oleic acid content increased more in the latter case than former. This is attributed to the negligible amount of linolenic acid present in HLSB compared to SB. The importance of reducing the polyunsaturation in the vegetable oils is also shown in the high-oleic oils. HOSN has a much higher (40%) RBOT time compared to HOSB and HOSF. This cannot be solely explained on the basis of higher oleic acid content of HOSN compared to HOSB and HOSF. It also shows that the type of polyunsaturation (linoleic versus linolenic) contributing to the UN is also a factor. A similar trend was obtained in vegetable oil–PAO blend based lubricants. Addition of 20% PAO8 in vegetable oils increased the oxidation stability by as much as 35%. This PAO response on various vegetable oils using RBOT is similar to that obtained in PDSC isothermal experiments. This data suggests that an increase in oxidation stability of normal soybean oil (SB) can be achieved by modification of the composition of the fatty acids and use of suitable additive combinations.

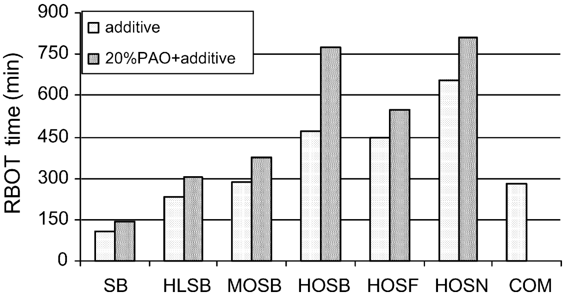

Fig. 3. RBOT times of vegetable oils and their PAO blend based lubricant formulations containing additive mixture.

The oven oxidation test performed on neat vegetable oils, additized vegetable oils and their blends is somewhat similar to Baader oxidation test (Lawate, 2002), in which the test is conducted at 95 °C for 72 h. The poor oxidation stability of vegetable oils prevent their use in high temperature applications like engine oils. Therefore, the test temperature at 85 °C is selected, which is suitable for other applications such as drip, hydraulic, elevator, compressor fluids for agricultural and heavy equipments. In this study, the oil samples were heated at 85 °C in oven for 28 days. The weight loss/gain and total acid number increase after 28 days were measured for all these vegetable oils and their formulations. In case of neat oils, there is some weight gain, which is due to oxidation of neat oils in the absence of antioxidants resulting in formation of some oxopolymerization products (Adhvaryu et al., 2000; Erhan and Adhvaryu, 2002). The weight gain is less in high-oleic oils, while it is slightly higher in SB and HLSB, due to the presence of more polyunsaturation in these oils (Table 3). In the present conditions, the weight loss in additized oils and their PAO blends is <0.5%, which is lower than COM sample (1%). Therefore, in vegetable oil formulations, volatility is not a problem, which is due to higher molecular weight associated with triacylglycerol structure of vegetable oils. The volatility relates to the potential loss of oil and to the environmental impact of the volatile components of the fluid. Both European and U.S. OEM and industry organizations include volatility in their specifications (Rudnick, 2002).

Table 3
Weight loss or gain of neat vegetable oils, additized oils and their PAO blends in oven oxidation stability test at 85 °C for 28 days

Vegetable oils	% Weight loss (+) or gain (−)		
	Neat oils	Neat oils + additive	20% PAO8 blend + additive
SB	−1.56	0.3	0.4
HLSB	−1.7	0.5	0.4
MOSB	–	0.4	0.5
HOSF	−1.1	0.5	0.5
HOSN	−1.0	0.4	0.4
COM		1.0	

The TAN values of the vegetable oils are in the range of 0.18–0.37 mg KOH/g. The TAN increased to 2.0–3.0 mg KOH/g after 28 days in the absence of antioxidants. There was no change in TAN of additized vegetable oils and their PAO blends. Under the test conditions of the oven oxidation test, the additive combination used in this study does an excellent job by preventing oxidation of vegetable oils and their PAO blends.

3.3. Low temperature improvement using PAO and PPD

The low temperature flow property of vegetable oils is extremely poor and this limits their use at low operating temperatures 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 (Erhan and Adhvaryu, 2002). SB, HLSB, and MOSB have a pour point of −9 °C, whereas, high-oleic oils have pour-points in the range of −18 to −24 °C (Fig. 4). The excellent thermal and oxidative stability of high-oleic oils due to removal of polyunsaturation (low unsaturation numbers) also results in improved cold flow property due to reduction in saturated fatty acids. However, the high oleic oils are still limited in their use in low temperature applications.

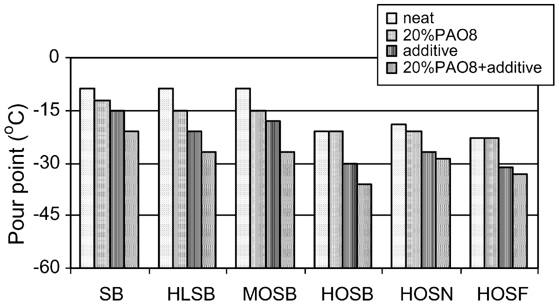

Fig. 4. Pour points of vegetable oils and their PAO blends with and without pour point depressant (additive).

In order to improve cold storage stability, polyalphaolefin (PAO) having pour point of −57 °C was added as a diluent with various soybean oils. Twenty, 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 SB, HLSB, and MOSB improved their pour point slightly by −3 to −6 °C, but no difference was observed in high-oleic vegetable oils. Further addition of PAO to 40% made no significant improvement in the pour point, therefore, only 20% PAO-blends were used in final formulations. The use of higher amounts of PAO also results in decreased biodegradability of final formulations. Further improvement in the low temperature fluidity 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. The purpose of PPD is to hinder crystallization of triglyceride molecules at low temperature by further disrupting the stacking mechanism. An optimum PPD concentration of 1% in final formulations of various soybean oils and soybean oil–PAO blends significantly improved the cold flow behavior (Fig. 4). The response of PPD is more visible in HLSB, MOSB, and high-oleic oils, where pour point decreased by −12, −9, and −9 °C, respectively, while in SB, pour point decreased by −6 °C. The PPD response is slightly strong in PAO blends, where in pour point decreased by −9 to −15 °C in vegetable oil–PAO blends. The presence of more oleic acid and less saturated fatty acids (palmitic acid and stearic acid) in triacylglycerols of high-oleic vegetable oils 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 HOSB, PPD, and PAO diluent had a pour point of −36 °C.

4. Conclusions

The study describes a systematic approach to improve the oxidation stability and low temperature flow property of soybean oil. It is shown that thermo-oxidative stability and cold flow property can be best improved using a

Table 4
Commercial biodegradable hydraulic fluid (COM) compared with formulated lubricant using HOSN–20% PAO8, antioxidants and pour point depressant

	ASTM test method	HOSN–20% PAO blend + additives	COM
General performance			
Viscosity at 40 °C	D445	42.33	53.45
Viscosity at 100 °C	D445	8.93	–
Pour point (°C)	D97	−36	−52
Oxidation performance			
RBOT, min to fail	D2272	768	278
PDSC OIT (min)	As described	98.1	42.6
PDSC OT (°C)	As described	264.7	244.1
Oven test, % weight loss		0.4	1.0
Friction and wear			
Four-ball wear; scar diameter (mm)	30 min, RT, 1200 rpm, 40 kg (D4172)	0.316	0.304

combination of proper blending of chemical additives, diluent (PAO) and high-oleic vegetable oils.

Comparisons were made with a commercially available biodegradable hydraulic fluid. The oxidation behavior of the COM and the fluids in this study are compared in Tables 2 and 3, and Figs. 2 and 3. HLSB–PAO blend, HLSB and MOSB show essentially the same onset temperature (Table 1) as COM, while MOSB–PAO blend and all high-oleic oils and their PAO blends are oxidatively more stable than COM (244 °C) as shown by PDSC ramping experiment.

Similarly in PDSC isothermal experiments, all additized high-oleic oils and their PAO blends have higher OIT compared to COM (Fig. 2). RBOT results show that HLSB–PAO blend, mid-oleic, high oleic and their PAO blends have excellent oxidation stability compared to COM (Fig. 3).

The other properties of COM fluid are compared with the best lubricant formulated in this study using HOSN–PAO8 blend and are listed in Table 4. The additive combination used in this study improves the oxidation stability of the SB (109 min) and SB–PAO blend (145 min) formulations. The oxidation stability exceeds that of another commercially available soy based lubricants (83 min).

Cold flow properties have also been improved using a combination of PPD and PAO diluent. Among vegetable oils, SB is not very useful due to their poor oxidation stability. The formulations with mid-oleic soybean oil and high-oleic oils shows excellent thermo-oxidative stability and low temperature flow property when blended with PAO and additive combinations, and is acceptable for most industrial applications. It is possible that with better bean initiative of United Soybean Board, MOSB will be widely available with price much less than high-oleic oils. High-oleic oils cost about twice as much as normal vegetable oils but only about half compared to synthetic fluids such as PAO8, trimethylol propane (TMP) trioleate. It is expected that as high-oleic oils become more readily available, the price difference between high-oleic oils and normal soybean oil will also decrease.

Acknowledgements

Names are necessary to report factually on available data; however, the USDA neither guarantees nor warrants the standard of the product, and the use of the name by USDA implies no approval of the product to the exclusion of others that may also be suitable.

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