Sperm Oil Replacements: Synthetic Wax Esters from Selectively Hydrogenated Soybean and Linseed Oils

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ABSTRACT AND SUMMARY

Synthetic wax esters with properties similar to those of sperm whale oil have been prepared entirely from soybean and linseed oils. The synthesis required: (a) selective hydrogenation of the oils with copper-on-silica gel catalyst, (b) hydrogenolysis of fatty acids to fatty alcohols with copper-cadmium-chromium catalyst, and (c) esterification of hydrogenolysis products to yield predominantly long chain fatty esters which contained unsaturation in both the alcohol and acid moieties. Similarity of physical and chemical properties indicate that these wax esters are possible replacements for sperm oil. After sulfurization, the wax esters also have potential as extreme pressure lubricant additives.

INTRODUCTION

In 1970, the United States placed the sperm whale on the endangered species list and, in 1971, banned the import of its products. For this reason and because of shortages and increased prices of petrochemicals, we have investigated the preparation of sperm whale oil replacements from renewable agricultural sources.

Previously (1,2) we described esters of commercial partially hydrogenated soybean acids and of selectively hydrogenated soybean (SHSBA) and linseed acids (SHLSA) as possible replacements for sperm whale oil in lubricants. The saturated alcohols used in the preparation of these esters were derived mostly from petrochemicals.

This paper reports the preparation of wax esters entirely from soybean and linseed oils. The synthesis required the selective hydrogenation of the oils with copper-on-silica gel catalyst (3) at 170 C and 600 psi pressure followed by hydrogenolysis of the fatty acids, derived from the selectively hydrogenated oils (2), at 300 C and 2500-3000 psi pressure with a copper-cadmium-chromium catalyst. Conversion of fatty acids to fatty alcohols was 35-99+% with less than 5% increase in saturates and with 0-53% of diene reduced to monoene. The main side product of the hydrogenolysis was fatty alcohol-acid esters. Esterification of reaction product with a calculated amount of the corresponding fatty alcohols or fatty acids produced all wax esters. Kinematic viscosity data and smoke, flash, and fire points indicate that the wax esters from SHSBA (WESHSBA) and wax esters from SHLSA (WESHLSA) would be possible replacements for sperm oil. After sulfurization, these wax esters have potential as extreme pressure (EP) additives. EP additives prevent destructive metal-to-metal contact in lubrication at either high pressure or temperature or both. WESHSBA and WESHLSA were sulfurized, tested, and evaluated as EP and antiwear additives.

EXPERIMENTAL PROCEDURES

Materials and Methods

Refined and bleached soybean and linseed oils came from commercial sources. Fatty acid compositions of oils as determined by gas liquid chromatography (GLC) of methyl esters were soybean: 10.7%, C16:0; 3.6%, C18:0; 25.2%, C18:1; 53.2%, C18:2; and 7.5%, C18:3; and linseed: 6.7%, C16:0; 3.7%, C18:0; 23%, C18:1; 15.6%, C18:2; and 51%, C18:3. A 15% copper-on-silica gel catalyst was prepared with copper nitrate trihydrate and heat activated as outlined by Koritala (3).

SHSBA and SHLSA were obtained from saponification of the parent selectively hydrogenated oils.

Other Reagents

Girdler (Chemetron Corp., Louisville, KY) T-1057 catalyst (ca. 40.8% CuO, 19.7% CdO, and 14.3% Cr_2O_3) was used for hydrogenolysis. Topaz S105 paraffin oil (Topaz S105) is produced by Atlantic Richfield Co., Philadelphia, PA.

Engine crankcase base oil (AA), automatic transmission base fluid (BB), RGO-100 gear lubricant (CC), 100/100 viscosity solvent-extracted neutral oil (DD), and commercial sperm oil replacements were provided by Southwest Research Institute (SWRI), San Antonio, TX.

Hydrogenation

Soybean and linseed oils were selectively hydrogenated with the copper-on-silica gel catalyst at 170 C and 600 psi pressure as described in a previous publication (2). Selectively hydrogenated soybean oil (SHSBO) contained 10.2%, C16:0; 4.2%, C18:0; 76.2%, C18:1, 9.4%, C18:2; 0.0%, conjugatable C18:3; and 37.3% isolated *trans* double bonds. Selectively hydrogenated linseed oil (SHLSO) contained 6.2%, C16:0; 4.6%, C18:0; 37.6%, C18:1; 48.0%, C18:2; 3.6%, C18:3; 5.0%, conjugatable C18:2; 1.5% conjugatable C18:3; and 44.4% isolated *trans* double bonds.

Hydrogenolysis

For a typical hydrogenolysis, a 1000 ml stainless-steel Magne-Dash autoclave was charged with 600 ml of SHSBA and 30 g of Girdler T-1057 catalyst per 100 ml acids. After the vessel was purged with nitrogen and pressurized with hydrogen to 3000 psi at room temperature, the charge was heated with stirring to 300 C. Hydrogen pressure was then maintained at 3000 psi for 5 hr. At this stage, hydrogen uptake was nil over a period of 0.25 hr. After cooling the autoclave to 80 C, the batch was filtered with filter aid and the product was analyzed. When necessary, trace amounts of metals (from catalyst) were removed by vacuum bleaching with 1% activated clay (4). Analysis of product (experiment 1, Table I) showed 97.9% fatty alcohols, 0.2% free fatty acids, and 1.9% wax esters. This standard procedure (SP) was followed in all hydrogenolyses.

Esterification

A portion of the above hydrogenolysis product (472.3 g, 1.74 mole alcohols) and 446 g (1.67 mole) precursory SHSBA were refluxed in the presence of 2 g of a catalyst consisting of three parts by weight of calcium acetate and one part of barium acetate with 500 ml of xylene. Theoretical amounts of water of esterification were removed by a Dean-Stark trap. The reaction mixture was washed with water, dried, and stripped of solvent. Infrared (IR) analysis

TABLE I

Composition of Wax Esters and Products from the Hydrogenolysis of Selective Hydrogenated Soybean and Linseed Acids with Cd-Cu-Cr Catalyst

Acids and experiment no.	Catalyst (g/100 ml acids)	Hours at 300 C	Free acids as oleic (%)	Conversion acids to alcohols (%)	GLC analysis C16:0 Alcohols (%)	C16:0 Acids (%)	C18:0 Alcohols (%)	C18:0 Acids (%)	C18:1 Alcohols (%)	C18:1 Acids (%)	C18:2 Alcohols (%)	C18:2 Acids (%)	*trans* Isolated (%)	Iodine value	N_D30
SHSBA[a]			100			10.2		4.2		76.2	—	9.4	37.3	86.2	
SHSBA[b] 1	5	5.0	0.23	97.9	10.6	15.0	4.9	10.5	74.3	74.5	10.2	—	37.5	91.4	1.4572
WESHSBA[c]			0.58		10.6	10.3	5.6	4.9	74.2	75.6	9.6	9.2	40.2	92.3	1.4612
SHSBA[b] 2	5	4.5	1.3	95.6	10.3	8.9	4.7	10.6	75.0	75.0	10.0	5.5	37.5	90.2	1.4559
SHLSA[d]	—	—	100	—		6.2		4.6		37.6	—	48.0	44.4	125.7	
SHLSA[e] 3	1	4.5	39.6	35.0	6.5	6.2	8.7	5.3	63.2	62.2	21.5	27.3	50.4	104.5	1.4623
WESHLSA[f]			1.3	—	6.9	6.7	7.3	5.1	58.6	60.6	27.1	27.6	51.5	109.9	1.4666
SHLSA[d] 4	1	6.5	24.0	38.2	9.1	5.1	5.0	5.2	62.9	64.7	23.0	25.0	53.5	104.1	1.4665
SHLSA[d] 5	10	6.75	0.0	99+	7.2	—	6.1	—	54.9	—	31.8	—	54.3	113.0	1.4593
SHLSA[b] 6	5	4.5	7.2	48.8	9.9	7.4	6.2	4.2	52.7	59.7	31.2	28.7	44.5	108.0	1.4573

[a]SHSBA: Selectively hydrogenated soybean acids.
[b]Conducted at 3000 psi pressure.
[c]WESHSBA: Wax esters from selectively hydrogenated soybean acids.
[d]SHLSA: Selectively hydrogenated linseed acids containing 3.6% triene.
[e]Conducted at 2500 psi pressure.
[f]WESHLSA: Wax esters from selectively hydrogenated linseed acids.

of wax esters showed no hydroxyl present. Wax esters had acid value of 1.2.

Analytical Methods

For acid-alcohol analysis (Table I), wax esters and hydrogenolysis products were saponified with ethanolic potassium hydroxide and isolated, as detailed by Miwa (5). The reaction mixture was diluted with water and the fatty alcohol fraction was extracted with etheyl ether. The acid salts were acidified with dilute HCl, and the free fatty acids were extracted with ethyl ether. Isolation of fatty acids from fatty alcohols was essentially complete as determined by GLC and IR analysis. IR spectra of neat hydrogenolysis products, isolated fatty acids, methyl esters of isolated fatty acids, isolated fatty alcohols, and acetate esters of isolated fatty alcohols were compared for absorbance at 3310 cm^{-1} (OH stretching); 1055 cm^{-1} (C-O stretching for alcohol); 1735 cm^{-1} (C-O stretching); and 1170 cm^{-1} (C-O ester stretching). Conversion of acids to alcohols was calculated from weight percent of isolated acids and alcohols. For GLC analysis, acid fractions were converted to ethyl esters with 5% HCl in anhydrous ethanol and alcohol fractions were acetylated with acetic anhydride-pyridine reagent. Esters were analyzed on a 12ft-1/8 in. OD stainless-steel column packed with 10% EGSS-X on Gas Chrom. P, 100-120 mesh (organosilicon polyester packing, Applied Science Laboratories, Inc., State College, PA) in a Model 1625 C Varian gas chromatograph or in a Model 5750 Hewlett-Packard gas chromatograph. The column was held at 170 C with a helium flow of 35 ml/min. IR spectra were recorded with a Beckman IR 8 spectrophotometer, both for thin films of 100% material and for carbon disulfide solutions of compounds. Ultraviolet (UV) spectra were obtained on a Beckman DB spectrophotometer. Iodine value (IV) was calculated from GLC analysis or determined by official AOCS method Cd 1-25 (6). Acid value (AV) and percentage-free fatty acids were determined by official AOCS methods Da 14-42 and Da 4a-42 (6), respectively.

Nonconjugatable diene and triene were determined by the difference between total diene and triene by GLC analyses and conjugatable diene and triene by UV analysis. Percentage of isolated *trans* double bonds was determined by AOCS Official Method Cd 14-61 (6).

Viscosities of wax esters were taken in Cannon-Fenske-Ostwald viscometers. The viscosity indices were obtained from viscosities at 100 and 210 F by ASTM Method D 2270 (7). The kinematic viscosity was converted to saybolt universal viscosity (SUS) according to ASTM Method D 2161 (7).

Smoke, flash, and fire points were measured by the Cleveland open flash cup, ASTM D 92-33 (7) and AOCS Official Method Cc 9a-48 (6).

Evaluation of Sulfurized Sperm Oil Candidates

As we reported previously (2), sperm oil candidates were sulfurized and evaluated under a research contract with SWRI. Although SWRI had overall responsibility for sulfurizing and evaluating our sperm oil candidates, part of the work was performed at the Eastern Regional Research Center, such as the four-ball EP test ASTM Method 2596 (7), four-ball wear test ASTM Method D 2266-67 (7), and freezing and pour points ASTM Method D 97-57 (7).

EP tests were made on a Precision Scientific four-ball EP tester (1440 rpm) in which loads were successively increased first in 20- and then 10-kg increments until an immediate seizure occurred, representing the weld point. Scar diameters were determined with a Precision four-ball wear tester. Samples were run for 1 hr at 600 rpm at 120 C and under a 50-kg load with and without additive. After the balls were cleaned with naphtha and hexane, scar diameters were measured under a microscope assembly #73607, with

TABLE II

Physical Properties of Wax Esters of Selectively Hydrogenated Soybean and Linseed Acids

Wax esters[a]	IV	Viscosity[b] SUS (F)		Viscosity index	Points (F)		
		100	210		Smoke	Flash	Fire
WESHSBA	112.6	135.7	47.8	211	320	536	698
WESHLSA	91.4	92.4	42.0	207	311	482	770
Sperm oil[c]	82.0	109.0	44.8	223	275-325	490	655-675

[a]Wax esters: WESHSBA, wax esters from selectively hydrogenated soybean acids; WESHLSA, wax esters from selectively hydrogenated linseed acids.

[b]SUS = Saybolt Universal viscosity.

[c]Winterized at 45 F.

measuring grid (Precision Scientific).

The following tests were made by SWRI: Sulfur analysis, base oil solubility test, copper strip corrosion test ASTM Method D 130 (7), API gravity at 60 F ASTM Method D 287 (7), lead corrosion test FTM 5321 (8), foam test ASTM D 892 (7), emulsion test ASTM D 1401, *N*-pentane and benzene insolubles ASTM Method D 893-52 T (7), viscosity and viscosity index ASTM Method 2270 (7), and thermal stability test FTM 2504-1 (8).

RESULTS AND DISCUSSION

Previously described, selectively hydrogenated soybean and linseed acids containing increased amounts of monoene and nonconjugatable diene have unusual thermal and oxidative stability and yet retained sufficient reactivity to permit sulfurization (2). These acids were reduced to the corresponding alcohols in 35-99% yield with less than 5% increase in saturates and with 0-53% reduction of diene to monoene. Analysis of hydrogenolysis products, SHSBA, SHLSA, WESHSBA, and WESHLSA are given in Table I. The main side product of the hydrogenolysis was fatty alcohol-acid esters. GLC analysis of products showed the formation of trace amounts of hydrocarbons. However, head gases were not collected and analyzed for hydrocarbons. Because the selectivity of copper-cadmium catalyst in the reduction of unsaturated acids to unsaturated alcohols has been reported extensively (9-13), a comprehensive study of the reaction conditions was not made. The aim of this work was to prepare samples of wax esters entirely from soybean and linseed acids for comparison with sperm whale oil. Results of this work indicated that optimum conditions for hydrogenolysis was 5 g Girdler T-1057 catalyst per 100 ml of fatty acids, hydrogen pressure of 3000 psi, and a temperature of 300 C for 5 hr.

WESHSBA was obtained by the esterification of a calculated amount of SHSBA with reduction products from experiment 1. WESHLSA was obtained by the esterification of a calculated amount of alcohols from experiment 5 with reduction products from experiment 4. Also shown in Table I are the hydrogenolysis products from experiments no. 2, 3, and 6. These products were not used in evaluation work. Reduction products from experiment 6 contained 7.2% free fatty acids, 4.0% free fatty alcohols, and 88.8% wax esters. When this mixture was refluxed with xylene, and water of esterification was azeotropically removed, a product containing 97% wax esters and 3% Fatty acids was obtained.

The unusual chemical composition and physical properties of sperm oil make it useful in such diverse applications as cutting oils, fine cosmetics, leather-softening agents, and spinning lubricants in the textile industry. One large commercial source of oleyl alcohol in this country has been from the saponification of sperm whale oil. Oleyl alcohol has many established uses and many more potential uses could be realized if it were more readily available on a large scale (13, 14).

TABLE III

Physical Properties of Sulfurized Wax Esters[a]

Property	WESHSBA	WESHLSA	SSO
Sulfur, %	10.5	10.8	11
Pour point, F	82	73	64
Freezing point, F	77	68	59
Flash point, F	468	460	464
Fire point, F	514	525	536
Saponification number	97.1	98.4	166.8
Neutral number	3.25	5.46	3.05
Viscosity at 210 F, SUS[b]	243	537	331

[a]Sulfurized wax esters: WESHSBA, wax esters from selectively hydrogenated soybean acids; WESHLSA, wax esters from selectively hydrogenated linseed acids; SSO, sulfurized sperm oil.

[b]SUS = Saybolt Universal viscosity.

In particular, oleyl alcohol is an intermediate for biodegradable detergents and in different types of surface active and detergent applications for household or industrial use. Although unsaturated fatty alcohols described in this work have geometrical and positional isomers, they should be considered as a source of monounsaturated alcohols. Physical properties of WESHSBA and WESHLSA (not winterized) were studied and compared with sperm oil winterized at 45 F (Table II). WESHSBA have smoke, flash, and fire points higher than those of sperm oil. WESHLSA have smoke, flash, and fire points comparable to those of sperm oil. WESHSBA and WESHLSA also have high viscosity indices (uniform viscosity over a broad temperature range).

In the lubrication of certain gear elements in automotive vehicles and various industrial machines, high pressure can cause a film of lubricant to rupture with subsequent damage to the machinery. For this reason, EP lubricants are fortified with additives to augment lubricity at either high pressures or temperatures, or both. EP lubricants should have good lubricity, good cooling properties, high film strength, good load-bearing ability, and miscibility with the usual types of base oils. Sulfurized sperm oil (SSO) satisfies these requirements and has been used extensively in EP additives. For this reason, WESHSBA and WESHLSA was sulfurized for evaluation as EP and antiwear additives.

The pour and freezing points of the sulfurized wax esters were higher than those of SSO (Table III); however, these values are comparable with those of several commercial sulfurized sperm oil replacements and may be improved by winterization.

The sulfurized wax esters were evaluated in base oils used in EP automotive and industrial applications. Performance of SSO replacements were compared (Table IV)

TABLE IV

Performance Comparison of Sulfurized Additives in Base Oils

Base oil[a]	Sulfurized additive[b]	Extreme pressure	Wear	Copper[c] corrosion	Lead corrosion (mg/in^2)	Kinematic viscosity[d] Cs at F		Viscosity index	API[e] gravity (degree API 60 F)	Emulsion test (ml)			Foam test[f] (ml)		
		Weld point (kg)	Average wear scar (mm)			100	210			Oil	H_2O	Emul	I	II	III
AA	None	140	0.635	1B	–	–	–	–	–	–	–	–	–	–	–
	10% WESHSBA	240	0.575	3A	0.2	132.42	13.57	119	28.0	1	4	75	0-0	10-0	0.0
	10% WESHLSA	260	0.550	3A	38.6	153.18	15.20	119	27.8	1	8	70	0-0	20-0	0-0
	10% SSO	300	0.583	1A/B	22.5	131.19	13.57	101	27.8	1	0	79	0-0	40-0	0-0
	10% Com Sub A	280	0.480	3A	30.3	134.19	13.61	111	27.6	1	0	79	45-0	30-0	0-0
	10% Com Sub B	240	0.575	1A	3.5	127.97	13.20	113	27.7	75	0	79	10-0	20-0	20-0
BB	None	120	0.625	1B	–	–	–	–	–	–	–	–	–	–	–
	10% WESHSBA	240	0.675	2B	5.8	538.62	35.30	118	25.3	21	25	34	0-0	0-0	0-0
	10% WESHLSA	260	0.590	3B	25.3	643.44	41.14	112	25.1	15	24	41	0-0	0-0	0-0
	10% SSO	280	0.628	1A	12.7	537.80	36.02	113	25.7	9	24	47	0-0	0-0	0-0
	10% Com Sub A	320	0.653	3A	27.4	543.52	36.09	121	25.9	5	22	53	0-0	0-0	0-0
	10% Com Sub B	240	0.591	1B	4.7	549.00	35.37	103	25.7	40	37	3	0-0	10-0	0-0
CC	None	130	0.603	–	–	–	–	–	–	–	–	–	–	–	–
	10% WESHSBA	240	0.400	3A	36.1	250.30	21.57	112	26.1	26	19	35	0-0	10-0	0-0
	10% WESHLSA	260	0.660	3B	28.6	272.59	22.77	110	26.1	9	9	62	20-0	0-0	0-0
	10% SSO	260	0.642	3A	19.0	240.77	20.94	110	26.2	8	11	61	510-20	150-0	180-0
	10% Com Sub A	280	0.675	3B	31.4	244.79	20.90	113	25.9	6	20	54	420-0	160-0	120-0
	10% Com Sub B	270	0.613	3A	4.6	239.76	20.75	118	26.2	6	15	60	530-0	80-0	110-0
DD	None	110	1.020	–	–	–	–	–	–	–	–	–	–	–	–
	10% WESHSBA	280	0.665	3B	20.0	27.64	5.04	117	31.0	36	20	24	25-0	40-0	0-0
	10% WESHLSA	280	0.670	3A	21.2	32.02	5.73	135	31.1	13	7	60	100-0	20-0	95-0
	10% SSO	300	0.697	1B	12.9	27.78	5.05	122	31.4	12	2	66	250-0	20-0	80-0
	10% Com Sub A	360	0.713	1B	12.6	27.72	5.26	140	31.3	33	13	34	220-0	20-0	100-0
	10% Com Sub B	270	0.620	1B	16.8	27.68	5.09	125	31.1	5	0	75	280-0	30-0	100-0
Topaz S105	None	120	0.794	–	–	–	–	–	–	–	–	–	–	–	–
	5% WESHSBA	280	0.642												
	10% WESHSBA	280	0.675												
	5% WESHSLA	260	0.668												
	10% WESHSLA	280	0.553												
	5% SSO	230	0.558												
	10% SSO	300	0.623												
	5% Com Sub A	220	0.606												
	10% Com Sub A	320	0.500												
	5% Com Sub B	230	0.596												
	10% Com Sub B	280	0.670												

[a]Base oils: AA, Southwest Research Institute's (SWRI) crankcase base oil; BB, SWRI transmission base fluid; CC, SWRI (RGO-100) gear lubricant; DD, Mayco's (100/100 vis) solvent extracted neutral oil; Topaz S105, Topaz S105 paraffin oil (similar to 102 paraffin oil).

[b]Sulfurized additives: WESHSBA, wax esters from selectively hydrogenated soybean acids; WESHLSA, wax esters from selectively hydrogenated linseed acids; Com Sub A, commercial substitute A; Com Sub B, commercial substitute B.

[c]Copper strip corrosion test ASTM Method D 130-65: 1, slight tarnish; 2, medium tarnish; 3, dark tarnish; 4, corrosion.

[d]Cs, Centistokes.

[e]API = American Petroleum Institute.

[f]Foam test ASTM Method D 892, sequence of bubbling 5 min and settling 10 min: I, at 75 F; II, at 200 F; III, at 75 F after collapsing the foam.

in base oils AA, BB, CC, DD, and Topaz S 105. SSO and two commercial SSO substitutes (Com Sub A, Com Sub B) are included for further comparison. Most commercial sulfurized replacements are sold as "packages" containing a number of additives, such as viscosity improver, metal deactivator, antioxidants, and EP agents. The sulfurized wax esters from hydrogenated fatty acids contained no additives nor were they winterized before sulfurization. SSO replacements were added to each base oil at 10% by weight concentration levels. The blended oils were stored for 24 hr at 35 F, 24 hr at room temperature, 24 hr at 35 F, and then 1 mo at room temperature. All sulfurized materials had good solubilities in all the base oils.

Both wear and EP test data were obtained with sulfurized WESHSBA and WESHLSA as 10% by weight concentration in all base oils. Wear and EP tests for SSO, Com Sub A, and Com Sub B on the basis of 10% by weight concentration in all the base oils are given for comparison. Although data suggest that Topaz S 105 was a reasonable choice for screening candidate materials, replacements performed differently in each of the base oils; therefore, the choice of a given additive will depend on its intended application. All sulfurized products showed both EP characteristics and antiwear properties. At 5% concentration in Topaz S105, sulfurized additives WESHSBA and WESHLSA exhibited EP properties better than those of SSO or of Com Sub A and B. SSO, Com Sub A and B showed better antiwear properties than the wax esters.

Although the sulfurized wax esters appeared to be effective EP agents, they gave a copper corrosion test of 2B to 3B. EP additives should have a copper corrosion rating of 2C or better. The higher than desired corrosion ratings and antiwear values may have been caused either by a too high sulfurization level or by sulfur-contained impurities, or both. Copper corrosion tests run with wax esters as 10% blend in base oils AA, BB, CC, and DD showed near acceptable values as did SSO in base oil CC, and Com Sub A in base oils AA, BB, and CC. Perhaps with proper adjustment of either sulfur concentration or addition of metal-deactivators, or both, the additives would possibly improve greatly in antiwear, and anti-copper corrosion properties.

Sulfurized WESHLSA had higher than expected lead corrosion in base oils AA, BB, and CC; comparable to that of Com Sub A. In base oils AA and BB, sulfurized WESHSBA exhibited lead corrosion tendencies much less than did SSO or Com Sub A and B.

Values of kinematic viscosity data, viscosity indices, and API gravities of all materials tested as 10% blends in the four base oils are within most industrial and military specification for lubricants containing EP additives.

In summarizing emulsion test data of additives as 10% blends in the four base oils, all additives form stable emulsions with the four base oils and are suitable in this regard for marine engine lubrication and cutting oils. Candidate additives in base oils BB and CC exhibit excellent de-emulsification properties and should find application in forced-feed circulating lubrication systems, provided that the other physical and chemical properties required of such systems are also met.

Foam test data of additives as 10% blends in the four base oils demonstrate that all candidate additives met test requirements. Sulfurized WESHSBA and WESHLSA showed less foaming tendencies than did SSO or Com Sub A and B in base oils AA, CC, and DD. In base oil BB, sulfurized WESHSBA and WESHLSA, SSO and Com Sub A showed no foam tendencies.

Thermal stability test FTM 2504-1 was made with 10% by weight additives concentration in base oil CC. The sulfurized WESHSBA blend failed the test because the increase in viscosity was higher than permissible (100%). The sulfurized WESHLSA blend polymerized after 40 hr of test. Although this material is apparently not suitable for engine gear application, it did seem to perform reasonably well otherwise and may find application as an industrial lubricant requiring lower than 300 F operating temperature or thermal stability.

Since most lubricants are formulated with a number of additives, each having certain performance characteristics, sperm oil replacement candidates could not be expected to meet all lubricant specifications. However, the sulfurized candidate additives have good EP properties and are superior to SSO and commercial additives in foaming tendencies. Thermal stability and copper and lead corrosion tendencies exhibited by the wax esters are less than desired, but can be improved by including either an appropriate metal deactivator or antioxidant, or both.

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