

**Final Report: 18 – 422 - 19**

**Project Title: Multi-Viscosity Blends of High Oleic Soybean Oil and Esters for Industrial Applications**

**Project Period: July 10,'18 – January 31,'20.**

**Investigators:**

Racha Seemamahannop, Department of Chemistry, Missouri S & T (University of Missouri),  
326 Schrenk Hall, Rolla, MO 65409. Phone (573) 341-6688.

e-mail: [racha@mst.edu](mailto:racha@mst.edu)

Shubhen Kapila, Department of Chemistry, Missouri S & T (University of Missouri),  
329 Schrenk Hall, Rolla, MO 65409. Phone (573) 341-4986.

e-mail: [kapilas@mst.edu](mailto:kapilas@mst.edu)

**Summary:**

The project was aimed at developing Soyleic<sup>®</sup> based lubricant oils. Soyleic<sup>®</sup> is a high oleic soybean oil developed through USDA/University Missouri soybean breeding program. Soyleic oil with high oleic acid content and very low linolenic content exhibits high resistance to oxidation yielding long term stability. Thus, this oil is a very good candidate for applications where oils are subjected to oxidative environment such as the lubricating oils and applications where long term (many years) stability of oil is desired. We had investigated application of Soyleic as dielectric oils for electrical transformers. Results obtained clearly showed that Soyleic oil is clearly superior to generic soybean oil in terms of oxidation stability and possesses high fire point relative to the traditional dielectric oils i.e. petroleum derived mineral oils.

The same attributes of the Soyleic oil are of value in very high applications such as lubricating and hydraulic oils. It has been estimated that volume of oils used in these applications in the United States exceeds 8.40 million tons, the worldwide demand is estimated to reach USD 166.59 billion by 2021, registering a CAGR of 2.4% between 2016 and 2021.

The critical factors for lubricating oils are high viscosity index (VI) and oxidation stability, vegetable oils including generic soybean oil are prone to gelation and oxidation due to high unsaturated high linoleic and linolenic content. To suppress gelation and oxidation oils are fortified with high amounts of additive (up to 5% of the total). High additive content leads to deterioration of compatibility with metal surfaces and require frequent monitoring of oils, lowering their potential for long term use. Soyleic was found to be significantly more resistant to gelation and oxidation and proved to be a very attractive candidate for lubricating and hydraulic oil applications. *Friction and wear test results showed that Soyleic oil provided highest protection against abrasion among three test oils; mineral oil, generic soybean oil and the Soyleic oil. Thus, Soyleic is the most suitable natural oil for these applications and can fulfill the large demand for carbon neutral, “naturally stable” oil for lubricating and other industrial applications. Since, Soyleic was developed in Missouri, Missouri soybean growers have an excellent opportunity to take a part of the 166.59 billion lubricating oil market.*

## Objectives

The overall project goal is to demonstrate suitability of Soyleic oil and its derivatives for lubricating and hydraulic oil applications, superior long-term stability, properties and environmental compatibility in lubricating oil and hydraulic oil application through standard tests.

The specific research efforts were directed at:

- Extraction of and refinement of Soyleic oil from seeds.
- Characterization of Soyleic oil.
- Synthesis of Soyleic oil monoesters.
- Determination of pertinent physical properties of generic soybean oil, Soyleic oil and monoesters.
- Formulation of generic soybean oil, Soyleic oil and monoesters with suitable additives.
- Evaluation of oxidative and aging stability of unformulated generic soybean oil, Soyleic oil and formulated oils.
- Evaluation of compatibility of oils with mechanical systems

## Experimental Approach:

### I. Preparation of seeds and oil extraction.

Both types of soybeans were roasted in an oven at 76°C (170°F) for 30 minutes. Beans were allowed to cool down to ambient temperature and dehulled through gentle cracking in a grinder. Dehulled beans were ground to ~20 mesh and extracted in 100 g batches in Soxhlet extractors with hexanes for 12 hours.

### II. Fatty acid composition of generic soybean oil and Soyleic oil.

Fatty acid composition of oils (triglycerides) obtained from the generic and high oleic soybeans was determined with gas chromatograph equipped with flame ionization detector (GC-FID) and will be confirmed with a gas chromatograph – mass spectrometer (GC-MS). Triglycerides were transformed to methyl esters through base catalyzed transesterification with methanol. Methyl esters were separated with a capillary column and quantified with FID. The GC-FID system was calibrated with certified fatty acid methyl esters (FAMES) obtained from commercial vendors.

Identity of fatty acid methyl esters was assigned by matching retention times of FAME standards peaks with those in the SO and Soyleic methyl esters. Percent composition of the two fatty acids in the two oils was calculated from FID signal. The average percent fatty acid composition of the generic soybean oil (SO) and the Soyleic are given in Table -I.

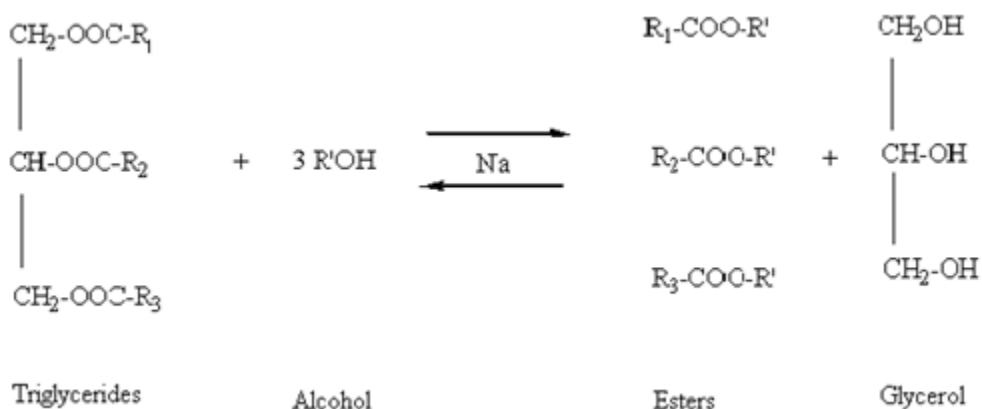
Table –I  
Percent fatty acid composition of Generic soybean oil and Soyleic oil

Fatty acid	Generic soybean oil	Soyleic oil
Palmitate [C <sub>16:0</sub> ]	9.5	5.4
Stearate [C <sub>18:0</sub> ]	5.1	3.2
Oleic [C <sub>18:1</sub> ]	25.8	79.5
Linoleic [C <sub>18:2</sub> ]	52.5	9.5
Linolenic [C <sub>18:3</sub> ]	7.1	2.4

Percent fatty acid composition oil extracted from the most recent batch of Soyleic were in good agreement with the fatty acid composition of oil extracted from the previous batches of Soyleic.

### III. Synthesis of Generic Soybean oil and Soyleic oil esters

Generic soybean oil and Soyleic oil were made to react with selected alcohols methanol, ethanol, iso-propanol and t-butanol. Transesterification was catalyzed with sodium alcoholate obtained by reacting sodium metal with the selected alcohol.



Transesterification of generic soybean oil / Soyleic triglycerides.

Excess alcohol was removed with rotary evaporation. Esters were further purified with Fuller's earth. Yield of purified esters was determined with gel permeation chromatography (GPC). Ester yields varied from 90 – 98%. Highest yields were for methyl esters and lowest for the t-butyl esters.

### IV. Physical properties of esters

Relevant physical properties of esters are being determined. Properties include viscosity, density, pour point, cloud point and boiling range. Properties are being determined with standard methods Viscosity in accordance with ASTM D 445 (1). The kinematic viscosity of each ester was determined with Cannon-Fenske calibrated viscometers. To cover the range of viscosities, different Cannon-Fenske viscometers were used including 75 E365, 75 E 367, 75 E368, 100 W 578, 150 Q 223, 150 Q224, 200 544P, 200 545P. Each Viscometer has a specific viscometer constant at a given temperature, viscosities were determined at different temperatures. Photograph of the viscometer setup is shown in Figure 1.



Figure 1: Photograph of viscometer setup used in the lab.

### Kinematic Viscosities of generic soybean oil esters

The kinematic viscosities of generic soybean oil esters determined at different temperatures are given in Table – II. Viscosities increased with the increase in the alcohol chain at ambient temperature (25°C), viscosities ranged from 7.1 for methyl esters to 23.5 for t-butyl ester.

Table – II  
Kinematic Viscosities of generic soybean oil esters (centistokes)

Temperature °C	Methyl Ester SOME	Ethyl Ester SOEE	Isopropyl Ester SOPE	T-Butyl Ester SOBE
50	4.2	4.4	4.8	13.6
45	4.5	4.8	5.3	14.4
40	5.0	5.4	5.9	15.9
35	5.5	6.0	6.6	17.78
30	6.2	6.7	7.5	20.5
25	7.1	7.7	8.5	23.5
20	7.9	8.8	9.7	26.7
15	9.0	10.1	11.3	31.4
10	10.2	11.7	13.7	36.9
5	12.7	14.3	16.3	42.6
0	15.5	17.4	19.2	52.7
-5				67.6

As expected, viscosities decreased with an increase in temperature, increase shows an exponential relationship with temperature, Figure 2.

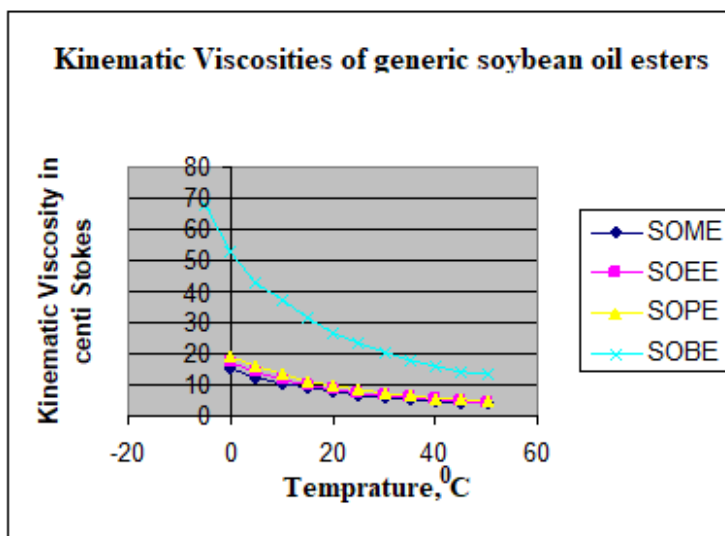


Figure 2: Kinematic Viscosities of generic soybeans oil esters at different temperatures.

### Kinematic Viscosities of Soyleic oil esters

The viscosities of Soyleic oil esters determined at different temperatures are given in Table – III.

Table III  
Kinematic Viscosity of Soyleic (high oleic - SOY) Esters (centistokes)

Temperature	Methyl Ester SOYME	Ethyl Ester SOYEE	Isopropyl Ester SOYPE	T-Butyl Ester SOYBE
50	5.1	5.0	5.2	12.5
45	5.6	5.5	5.8	15.6
40	6.2	6.1	6.2	18.9
35	6.8	6.7	7.1	20.8
30	7.7	7.6	8.0	24.9
25	8.5	8.4	9.0	30.1
20	9.7	9.4	10.6	34.3
15	10.4	10.2	12.4	41.3
10	13.0	12.8	14.5	54.3
5	14.9	14.6	17.1	67.5
0	18.9	18.1	20.8	86.3
-5	22.1	20.7	25.1	106.6
-10	28.3	27.2	30.2	129.5
-13			35.4	

Viscosities of Soyleic esters were generally higher than those of the generic soybean oil. However, unlike generic soybean oil esters, viscosities of Soyleic ethyl ester were found to be lower than Soyleic methyl esters. Change in viscosities with change in temperature are shown graphically in Figure 3.

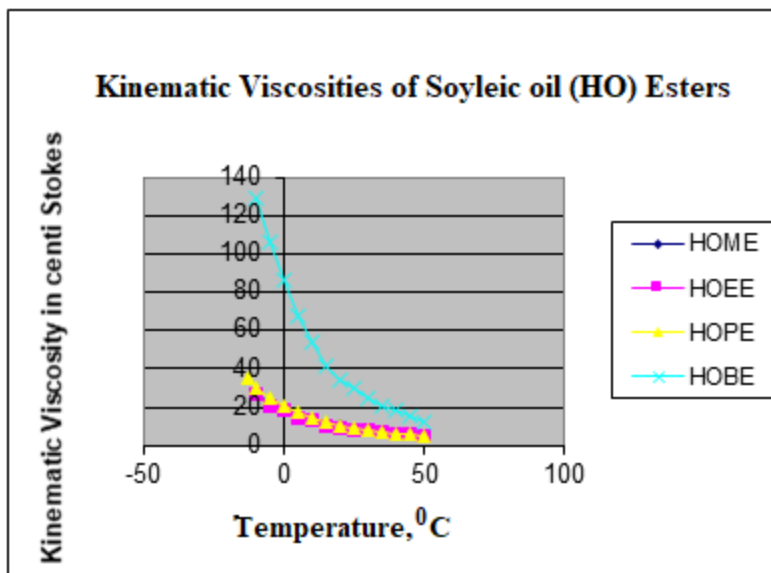


Figure 3: Kinematic Viscosities of Soyleic oil esters at different temperatures.

**Pour Point.** Pour Point is the lowest temperature at which movement of the test specimen is observed under the conditions of the test. The pour point of a crude oil is the index of the lowest temperature of handleability for certain applications. Low Pour Point of the Biogenic oils is desired to avoid localized heat buildup in cold start operations

ASTM D 5853-95 and ASTM D-97 are the standard methods used for the determination of pour points of the biogenic esters(2,3). Also, Pour Point analyzers are available in the market which directly gives the pour point of oil.

#### Pour point determination

The pour point of oil is the index of the lowest temperature at which the oil can be handled for certain applications like biodiesel, dielectric fluids etc. Low Pour Point of the Biogenic oils is desired to avoid localized heat buildup in cold start operations. A known volume of esters (8mL) of the sample was poured into 10mL glass tubes. Tubes were put in a rack and rack was and all the samples are placed in freezer. The temperature of the freezer was adjusted with a decrement of 1°C every 8 hrs. The samples were observed and the lowest temperature at which movement of the test specimen is observed has been noted. A photograph of freezer with ester samples used for cloud point determinations is shown in Figure 4.

Results showed that the isopropyl esters and t-butyl esters of high oleic soybean oil had the lowest pour point compared to the methyl and ethyl esters of all the samples. Pour points for different esters of generic soybean oil and the Soyleic oil are given in Table – IV.

Table – IV  
Pour Points of the generic soybean and Soyleic oils esters

Oil	Methyl Ester	Ethyl ester	Isopropyl ester	t-Butyl ester
Generic soybean oil	-4	-7	-10	-16
Soyleic oil	-12	-14	-18	-21



Figure 4: Photograph freezer with ester samples in 10mL vials used for pour point and cloud point determinations.

**Cloud Point.** In Petroleum Products and Biodiesel fuels, the temperature of a liquid specimen when the smallest observable cluster of wax crystals first appears upon cooling under prescribed conditions is called the Cloud Point. **The Cloud Point of a petroleum product is an index of the lowest temperature of their utility for certain applications.** Cloud Point analyzers available in the market directly give the cloud point of oils.

#### Cloud point determination

Cloud points of generic soybean oil and Soyleic oil were determined with a standard method ASTM D 2500-02(4). Cloud Point of oils serves as an index of the lowest applicable temperature for the use of oil for a given application such the dielectric fluids and lubrication. About 8ml of ester samples were taken in a 10ml glass tube and placed in a freezer capable of reach reaching a low temperature of  $-50^{\circ}\text{C}$ . Temperature of the freezer was decreased incrementally  $1^{\circ}\text{C}$  every 8 hrs. The samples were observed for the presence of smallest observable wax crystal cluster of wax crystals and the temperature was recorded.

Results obtained for different generic soybean oil esters and the Soyleic esters are given in Table – V.

Table - V  
Cloud Points of Generic Soybean Oil and Soyleic Oil Esters

Oil	Methyl Ester	Ethyl Ester	Isopropyl Ester	T-Butyl Ester
Generic soybean Oil	-1	-3	-5	-8
Soyleic oil	-6	-7	-13	-14

Results showed that esters obtained with branched chained alcohols i.e. isopropyl esters and t-butyl esters of both the generic soybean oil and the Soyleic oil had lower cloud points than esters obtained with short straight chain alcohols i.e. the methyl and the ethyl alcohols. Furthermore, cloud point for high oleic soybean oil esters had markedly lower cloud point than the generic soybean oil esters.

### **Flash point**

The temperature at which a liquid will yield enough flammable vapors to ignite. The flashpoint of a petroleum product indicates the fire hazard in handling and storing it. The flashpoint of the oil must be above the operating temperature of the engine in which it is to be used.

**Fire point.** The fire point is the temperature at which a sustained flame is obtained (longer than four seconds). The fire point is always higher than the flash point.

**Boiling point and boiling range.** The boiling point is the temperature at which the vapor pressure of a liquid is one atmosphere. ASTM D 1120-94 is the Standard test method for boiling point of biogenic oil esters (5).

Boiling points of liquid with low boiling points can be determined through distilling the liquid under atmospheric pressure, however, high molecular weight liquids such as the Soybean oils tend to decompose, oxidize or undergo molecular rearrangement at temperatures below their atmospheric boiling points. These problems are circumvented by distilling the oils at lower pressures. It is well established that a liquid boil at a temperature when its vapor pressure is equal to external pressure, thus it is possible to lower the boiling point of the liquid by distilling oils at pressures lower less 1 atmosphere (Vacuum Distillation). Vacuum distillation was carried out in accordance with standard method ASTM D 1160-18(6). This test method covers the determination, at reduced pressures, of the range of boiling points for petroleum products that can be partially or completely vaporized at a maximum liquid temperature of 400°C.

A distillation setup was assembled with a vacuum adapter which was in turn connected to a vacuum pump, Figure 5.



Figure 5: Vacuum Distillation setup used for determining boiling ranges of soybean oil and high oleic soybean oil esters.



One hundred mL of the ester was placed in the flask and heated with the heating mantle with thermostat controller. Water was circulated through the condenser. The vacuum pump was turned on and vacuum was monitored and recorded with a vacuum gauge. The boiling flask and connectors were insulated with Pyrex wool, temperature of vapors was monitored with thermometer placed at the inlet end of the condenser. The temperatures were recorded for every 10 mL batch of the collected oil. The actual boiling ranges were calculated using a correction expression.

The correction factor permits calculation of temperature needed for distillation at one atmosphere pressure. A general expression for the correction factor ( $\Delta t$ ) is:

$$\Delta t = 0.00012 (760 - p) (t_b + 273)$$

Where,  $\Delta t$  is the correction in degrees Celsius,  $t_b$  is the observed boiling point and  $p$  is the measurement pressure in mm of Hg.

The corrected boiling point ( $T_b$ ) is:

$$T_b = t_b + \Delta t$$

### Boiling point range

Boiling point range obtained for generic soybean oil and high oleic soybean oil esters are given in Table – VI.

Table – VI.  
Boiling point ranges of generic soybean and Soyleic esters

Oil	Methyl Ester	Ethyl Ester	Isopropyl Ester
Soybean Oil	336-353	338-352	348-366
Soyleic oil	334-351	342-366	349-368

Fractions of soybean oil and Soyleic oil esters collected at different temperature are given in Table – VII and VIII. respectively.

Table – VII  
Fraction of generic soybean oil esters collected at different temperatures

Percent ester	Methyl Ester	Ethyl Ester	Isopropyl Ester
10	336	338	348
20	337	343	348
30	337	346	351
40	341	348	352
50	341	348	352
60	346	351	356
70	347	352	357
80	351	352	358
90	353	353	364
95	353	352	366

Table – VIII  
Fraction of Soyleic oil esters collected at different temperatures

Percentage of the oil collected	Methyl Ester	Ethyl Ester	Isopropyl Ester
10	334	342	349
20	334	343	349
30	336	343	351
40	339	346	352
50	339	351	353
60	344	358	356
70	346	358	357
80	349	359	361
90	351	363	367
95	351	366	368

Results show that boiling characteristics of generic soybean oil and the Soyleic oil are nearly the same.

#### Densities of generic soybean oil and Soyleic oil esters

The densities of generic soybean oil and Soyleic oil esters were determined at different temperatures. The mass of a set volume of each ester was determined at a set temperature. The samples were placed 25 mL conical flasks and kept in a temperature-controlled bath for 30 minutes. Volumes of samples in the flasks were adjusted when required by adding or removing small volumes of oil with micro syringe, once constant volume had been maintained for 30 minutes flasks were weighed with a tared semi-micro balance. The density at the particular temperature was determined expressed as:

$$\text{Density} = \text{mass/volume (kg/m}^3\text{)}$$

The density of all the esters has been calculated at temperatures ranging - 15°C to 50°C in increments of 5°C.

Densities of different esters of generic soybean oil esters are given in Table –IX.

Table – IX  
Densities of Generic Soybean Oil Esters

Temperature °C	Methyl ester	Ethyl ester	Isopropyl ester	t-Butyl ester
50	0.8585	0.8552	0.8476	0.8665
45	0.8616	0.8581	0.8514	0.8693
40	0.8648	0.8612	0.8546	0.8726
35	0.8686	0.8643	0.8579	0.8761
30	0.8717	0.8675	0.8606	0.8796
25	0.8749	0.8713	0.8637	0.8838
20	0.8789	0.8751	0.8675	0.8881
15	0.8832	0.8789	0.8719	0.8924
10	0.8873	0.8851	0.8763	0.8966

5	0.8917	0.8893	0.8819	0.9009
0	0.8959	0.8922	0.8866	0.9052
-5	0.8983	0.8956	0.8884	0.9099
-10	0.9018	0.8996	0.8924	0.9127
-15	0.9053	0.9036	0.8964	0.9167

Densities of Soyleic oil esters are given in Table – X.

Table – X.  
Densities of Soyleic Oil Esters

Temperature °C	Methyl ester	Ethyl ester	Isopropyl ester	t-Butyl ester
50	0.8597	0.8606	0.8432	0.8561
45	0.8612	0.8623	0.8452	0.8576
40	0.8641	0.8646	0.8501	0.8593
35	0.8657	0.8664	0.8522	0.8617
30	0.868	0.869	0.8541	0.8639
25	0.8728	0.8702	0.8573	0.8728
20	0.8776	0.8746	0.8595	0.8776
15	0.8821	0.8766	0.8635	0.8821
10	0.8897	0.8794	0.8676	0.8897
5	0.8906	0.8822	0.8711	0.8906
0	0.8969	0.8843	0.8741	0.8969
-5	0.893	0.8890	0.8781	0.8932
-10	0.9015	0.8964	0.8815	0.9015
-15	0.9065	0.8978	0.8849	0.9085

Densities of esters obtained from generic soybean oil and Soyleic oil were very similar and showed similar trends, densities increased with decrease in temperature and decreased with an increase in temperature linearly, with correlation co-eff of ~0.99. Thus, their density behavior with respect to temperature can be readily predicted.

### Accelerated aging

Unformulated and formulated oils were subjected to accelerated aging under test conditions described in an International Standard for Natural Ester Transformer oils. Which is based on evaluation under accelerated aging conditions similar to those described in Method C of IEC 61125:2018(7). Aliquots of the natural esters' samples were maintained at set temperature in the presence of a solid copper catalyst.

Kinematic viscosities of unaged oils and Results obtained for kinematic viscosity (at 40°C) for oils obtained after aging are shown in Table -XI.

Table – XI  
Kinematic viscosity of formulated oils before and after accelerated aging in sealed ampoules under an air atmosphere

Aging period [hrs.]	Soyleic	Soybean generic
0	33.5±0.5	33.3±0.5
12	34.0±0.5	33.5±0.5
48	34.0±0.5	34.0±0.5
72	35.0±0.5	34.5±0.5
96	35.0±0.5	36.0±0.5

Results showed that there was little change in Kinematic viscosities of oils even after 96 hours.

Unformulated and formulated oil samples have been aged under two other atmospheric conditions:

- i. Under inert conditions (in sealed ampoules with Ar atmosphere).
- ii. In glass tubes with continuous air flow.

#### Accelerated aging

Unformulated and formulated oils were subjected to accelerated aging under test conditions described in the third progress report. These conditions were specified in ASTM standard for lubricating oils. Aliquots of the generic soybean oil and Soyleic oil esters were maintained at 120°C for time period ranging up to 96 hours in sealed borosilicate glass ampoules. Esters were also aged with a continuous air stream of 2.5 mL min.<sup>-1</sup> in test tubes.

Esters were removed from the ampoules or test tubes and kinematic viscosities of “aged” esters were determined at 40°C with standard viscometer tubes. Kinematic viscosity results for unformulated generic soya oil esters in sealed ampoules with air atmosphere oils are shown in Table -XII

Table – XII  
Kinematic Viscosities of Unformulated Generic Soybean Oil Esters after Aging 120 °C sealed ampoules under air atmosphere

Aging Period, hrs.	Generic Soybean oil Esters			
	Methyl ester	Ethyl ester	Iso-propyl ester	t-Butyl ester
Initial	4.96	5.36	5.86	15.89
12	5.01	5.41	5.85	15.92
48	5.01	5.43	5.88	15.95
72	5.15	5.55	5.95	16.04
96	5.45	5.75	6.02	16.1

Results showed that there was small change in Kinematic viscosities of esters in sealed ampoules even at 96 hours, increase was highest in the case of methyl and t-butyl esters (~10 percent) and lowest in the case of the iso-propyl esters (~2 percent).

Results for kinematic viscosity for unformulated Soyleic oil esters in sealed ampoules with air atmosphere are shown in Table -XIII.

Table – XIII  
Kinematic Viscosities of Unformulated Soyleic Oil Esters after Aging  
120 °C sealed ampoules under air atmosphere

Aging Period, hrs.	Soyleic Oil Esters			
	Methyl ester	Ethyl ester	Iso-propyl ester	t-Butyl ester
Initial	6.09	6.19	6.24	18.88
12	5.98	6.2	6.25	18.95
48	6.10	6.22	6.26	19.1
72	6.10	6.25	6.27	19.15
96	6.14	6.28	6.30	19.22

Results for Soyleic oil esters showed that under sealed conditions Kinematic viscosities of these esters did not change, increase for most esters was less than 2 percent. Soyleic oil esters are measurably more stable than the generic soybean oil esters under sealed environments.

Kinematic viscosity results for unformulated generic soybean oil oil esters “aged” with continuous air stream at 120 °C are shown in Table -XIV.

Table - XIV  
Kinematic Viscosities of Unformulated Generic Soybean Oil Esters after Aging  
120 °C with continuous air at 2.5mL min.<sup>-1</sup>

Aging Period	Generic Soybean oil Esters			
	Methyl ester	Ethyl ester	Iso-propyl ester	t-Butyl ester
Initial	4.96	5.36	5.86	15.89
12	5.23	5.63	6.33	16.68
48	6.54	7.07	7.73	20.08
72	7.21	7.81	8.21	22.56
96	9.32	10.13	10.43	28.65

All esters showed a marked increase in kinematic viscosities, viscosities increased with “aging” period. Viscosities of all generic soybean oil esters increased to double the initial values to 96 hours aging.

Kinematic viscosity results for unformulated Soyleic oil esters “aged” with continuous air stream at 120 °C are shown in Table -XV.

Table – XV  
Kinematic Viscosities of Unformulated Soyleic Oil Esters after Aging  
120 °C with continuous air at 2.5mL min.<sup>-1</sup>

Aging Period, hrs.	Soyleic Ester			
	Methyl ester	Ethyl ester	Iso-propyl ester	t-Butyl ester
Initial	6.09	6.19	6.24	18.88
12	6.15	6.68	6.55	20.07
48	7.00	7.42	7.38	22.86
72	7.61	7.72	7.80	23.62
96	8.22	8.23	8.42	24.95

Kinematic viscosity measurements for Soyleic oil esters showed that increase in viscosities was markedly less than the increase observed in the case of generic soybean oil esters. Increase was approximately 33 percent vs nearly 100 percent in the case of generic soybean oil esters.

Kinematic viscosities for esters formulated with antioxidant (BHT) and metal passivator (Irgamet 30) and aged under continuous air streams are shown in Tables XVI and XVII for generic soybean oil esters and Soyleic oil esters respectively.

Table – XVI  
Kinematic Viscosities of Formulated Generic Soybean Oil Esters after Aging  
120 °C with continuous air at 2.5mL min.<sup>-1</sup>

Aging Period	Generic Soybean oil Esters			
	Methyl ester	Ethyl ester	Iso-propyl ester	t-Butyl ester
Initial	4.96	5.36	5.86	15.89
12	5.23	5.63	6.15	16.36
48	5.35	5.73	7.22	16.62
72	6.62	6.94	7.45	20.65
96	7.91	8.12	9.12	25.15

Increase in kinematic viscosities of generic soybean oil esters formulated with antioxidant (BHT) and metal passivator averaged around 55 percent, compared to 100 percent for the unformulated generic soybean oil esters. Significantly lower increase in kinematic viscosities was observed in the case of formulated Soyleic oil esters. Increase in viscosities was only about 10 percent, Table – XVII. Results clearly show that Soyleic oil esters are markedly more stable than the generic soybean oil esters.

Table - XVII  
Kinematic Viscosities of Formulated Soyleic Oil Esters after Aging  
120 °C with continuous air at 2.5mL min.<sup>-1</sup>

Aging Period	Soyleic Ester			
	Methyl ester	Ethyl ester	Iso-propyl ester	t-Butyl ester
Initial	6.09	6.19	6.24	18.88
12	6.15	6.37	6.36	19.45
48	6.55	6.65	6.68	20.33
72	6.62	6.68	6.80	20.55
96	6.71	6.93	7.10	21.52

### Measurements of Viscosity index

Viscosity indexes of generic soybean oil, Soyleic and mineral were determined through kinematic viscosity measurements at 40°C and 100°C in accordance with a standard method ASTM-D 2270 -2016, Standard Practice for Calculating Viscosity Index from Kinematic Viscosity. Results for kinematic viscosities and viscosity index are given in Table – XVIII.

Table – XVIII  
Relative Kinematic Viscosities and Viscosity Indexes of Unformulated Generic Soybean Oil,  
Soyleic oil and White Mineral oil

Oil	Viscosity (cSt)		Viscosity index
	40°C	100°C	
Generic soybean oil	33.8	7.3	245
Soyleic oil	35.4	7.6	251
Mineral oil	20.0	4.05	105

Results showed that Soyleic oil has higher viscosity index than the generic soybean oil and significantly higher than the mineral oil, thus should be a better at higher temperatures under limited oxidation conditions.

### Friction and wear Test

Relative lubricating properties of Soyleic oil, generic soybean oil and mineral oils were assessed with a ball-on-disc tribometer at ambient temperature ( $\sim 23 \pm 2^\circ\text{C}$ ). The stainless-steel disc and ball were cleaned with acetone to remove residual oils prior to each measurement. Measured amount of Soyleic oil, generic soybean oil and the mineral oil was placed on initial contact zone with a dropper at the start of each test. Experiments were carried out with applied load varying from 2 to 4 N (0.448 – 0.896 lbf). The rotational speed of the disc was maintained at 100 rpm, disc was rotated for time period varying between 1 - 400s. After each run wear scar diameter on the ball was measured with an optical microscope. Each run was carried out in duplicate.

The average abrasion rate ( $A_n$ ) was determined as the volume (V) of steel from ball during defined distance traveled by the ball (n d, where n is the number of cycles and d is the distance travelled by the ball in one cycle ( $d=2\pi r$ );  $[V/n d]$ ). The average rate for Soyleic oil, generic soybean oil and mineral oil under applied load on 2 N are shown in Figures 6. Similar results were obtained at applied load of 4N.

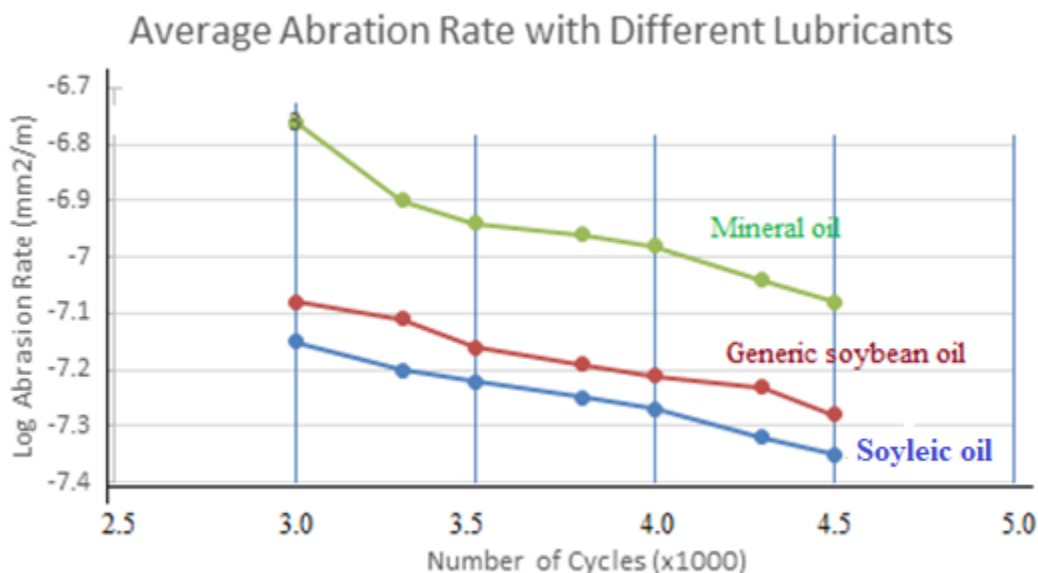


Figure 6: Average abrasion rate obtained with Mineral oil, Generis soybean oil and Soyleic oil.

The average value of the abrasion rate for Soyleic oil was significantly lower than the mineral oil. The abrasion rate was statistically lower than observed with the generic soybean oil, indicating that higher oleic content of the Soyleic oil provides better lubricity i.e. lower friction between the moving surfaces.

### Presentations and Publications

A manuscript on comparative lubricating properties of generic soybean oil and Soyleic oil and esters is being prepared and shall be submitted to Journal of Synthetic Lubrication with a month.

### References

1. ASTM D445 -19a Standard Test Method for Kinematic Viscosity of Transparent and Opaque Liquids.
2. ASTM D5853 -17 Standard Test Method for Pour Point of Oils.
3. ASTM D-97 – 17b Standard Test Method for Pour Point of Petroleum Products.
4. ASTM D2500 – 17a Standard Test Method for Cloud Point of Petroleum Products and Liquid Fuels.
5. ASTM D 1120 – 17 Standard Test Method for Boiling Point of Engine Coolants.
6. ASTM D1160 – 18 Standard Test Method for Distillation of Petroleum Products at Reduced Pressure.
7. IEC 61125:2018 Insulating liquids – Test methods for oxidation stability Test method for evaluating the oxidation stability of insulating liquids in the delivered state