Final Report

Federal Agency & Organization: Department of Energy – Office of the Biomass Program

Project Title: National Agricultural-Based Lubricants (NABL) Center

Award Number: DE-FG36-08GO88038

Recipient Organization: University of Northern Iowa

Project Location: 360 Westfield Avenue, Tech 1 Level 3, Waterloo, IA 50701

Reporting Period: September 30, 2012 – September 30, 2013

Date of Report: December 2013

Written by: Lou Honary, Professor and Director, lou.honary@uni.edu, 319-939-1397

Executive Summary

This project, while defined as a one year project from September 30, 2012 – September 30, 2013, was a continuation of a number of tasks that were defined in previous years. Those tasks were performed and were finalized in this period. The UNI-NABL Center, which has been in operation in various forms since 1991, has closed its facilities since September 2013 and will be phasing out in June 2014. This report covers the individual tasks that were identified in the previous reports and provides closure to each task in its final stage.

The primary general objective of this project was to provide programmatic support to the UNI-NABL Center, offer technical support to the industry, and disseminate research result to the biobased industry. Additionally, several specific tasks were identified with the goal of providing useable technical information to the industry in order to promote the use of biobased lubricants and greases. Those were:

Task A: CONTINUOUS OIL RECIRCULATION SYSTEM (CORS) RESEARCH

Task B: BIOBASED GREASE MANUFACTURING ENERGY EFFICIENCY RESEARCH

> Investigate the potential to process biobased lubricating greases with microwave transmitters as the process heat source, and identify benefits and drawbacks inherent in this unique heating method for biobased products, including vegetable oils.

Task C: FUNDAMENTAL VEGETABLE OIL RESEARCH ACTIVITIES

> NABL will conduct research targeting new methods of improving the inherent characteristics and fundamental properties of vegetable oils.

Task D: CONTINUED PREPARATORY ACTIVITIES

> Qualify the NABL Center to serve the bio-lubricants industry as a respected testing facility.

Task E: CONTINUED RESEARCH ACTIVITIES

> NABL will pursue various research activities to be continued from previous scopes of work, which were not completed previously due to either budgetary constraints, or limited staff time and technical capabilities, in the following sub-tasks:
>
>> NABL will continue laboratory work conducting fundamental vegetable oil based research, studying vegetable oil and vegetable oil derivatives, identifying desirable properties of various seed oils for use in lubricants, and relevant chemical structures.
>>
>> New applications for the oils mentioned in E.2 will continue to be researched.

These tasks were further delineated and their progress were reported in quarterly reports that were filed regularly. This report provides sections corresponding to the five abovementioned task. Each Appendix includes the capstone reports including some raw data and laboratory reports.

The Primary Investigator (PI) served on the board of directors of the National Lubricating Grease Institute (NLGI) and served as a coordinator for the working groups as well as serving as chairman of a working group for biobased greases for both NLGI and European Lubricating Grease Institute (ELGI). At those positions the PI has had opportunities to increase awareness for biobased lubricants. Additionally, the research results were used in numerous presentations and publications; a book and two book chapters which were reported in earlier quarterly reports.

Project Title: National Agricultural-Based Lubricants (NABL) Center

Introduction

This project, while defined as a one year project from September 30, 2012 – September 30, 2013, was a continuation of a number of tasks that were defined in previous years. Those tasks were performed and were finalized in this period. The UNI-NABL Center, which has been in operation in various forms since 1991, has closed its facilities since September 2013 and will be phasing out in June 2014. This report covers the individual tasks that were identified in the previous reports and provides closure to each task in its final stage.

Task number: A: CONTINUOUS OIL RECIRCULATION SYSTEM (CORS) RESEARCH

A. Utilize engine testing and lubricant testing resources to develop an on-engine oil recycling system and associated components to allow the use of renewable lubricants in an engine crankcase environment. This will be accomplished through removal of vegetable based oils from the crankcase and after conditioning, supply appropriate amounts of this oil to the fuel system.

This task was completed. The concept of Continuous Oil Recycling System (CORS) was the base of this research. A patent was applied for and an article was published in the OEM Off-Highway magazine. The system uses transducers to monitor the property of the biobased engine oil in the engine. As the oil degrades some of its property changes are monitored. Before the oil is oxidized to an unacceptable level, some of the oil is drawn from the crankcase and after conditioning it is fed into the fuel systems to be burnt. A reservoir of fresh oil whereby the provides oil for replacing the oil in the crankcase. The feeding of the oil in the fuel takes place when the engine is warmed up to reduce changes of soot built up which could take place at colder engine temperatures.

Several engine tests had been performed and the result had been reported in previous reports. Basically, a simple engine oil was formulated and tested in a diesel engine under load using a dynamometer. Appendix A shows a report generated for this activity. A proposed field testing of the concept was not completed due a number of factors including a lack of time.

Since one of the main shortcomings of vegetable oils for use as engine oil is lack of oxidation stability, attempts were made to combine the efforts in this are with other tasks that involved collaboration with the industry. As chairman of a working group with the National Lubricating Grease Institute (NLGI) and the European Lubricating Grease Institute (ELGI), the primary investigator incorporated some of the testing of the vegetable oils in a round robin testing for oxidations stability (Appendix B).

Engine oil formulations were tested both to test performance and determine the degree of oxidation. (Appendix B)

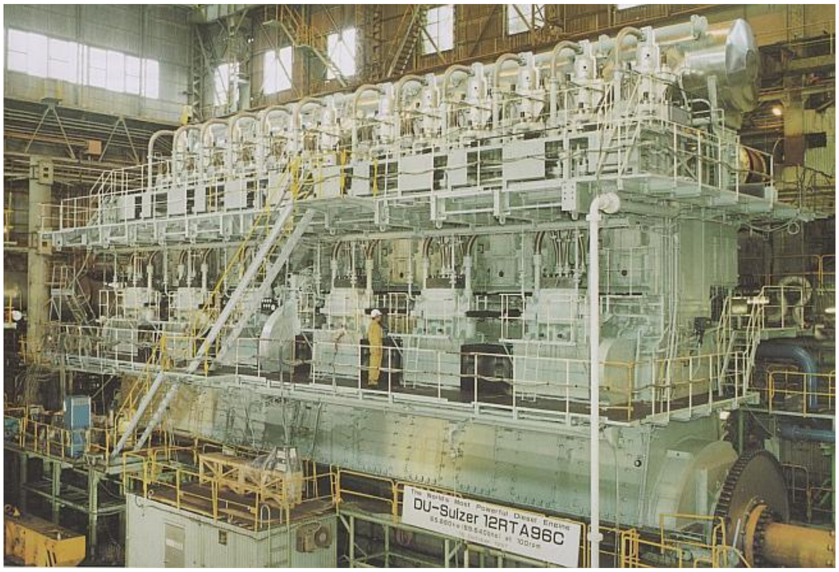
Actual Accomplishments: The main objectives of this project which included evaluation of base oils, laboratory and engine testing to determine feasibility were completed. The longer term engine tests and field testing could not be completed due to time delays for obtaining permission from local fire marshal for installation of a fuel tank and the decision to close the research center due to lack of overall funding for the center.

Explanation of Variance: As NABL did not purchased a High Frequency Reciprocating test Rig (HFRR), and no work has was completed toward tasks involving sample evaluations through HFRR testing. This was partially due to the fact the future of the research center was uncertain and acquisition of a piece of equipment for limited use was not justifiable.

First diesel engine test stand operational post-modifications – Completed
Second diesel engine test stand operational post-modifications – Completed
Fuel tank obtained for engine testing – Completed
Fuel tank was not installed and plumbed for engine testing

The following sections present the results of various activities associated with this task.

Supportive Docs
Continuous Oil Recycling System (CORS)
CORS Report



Lou Honary
Professor and Director
National Ag-Based Lubricants Center
University of Northern Iowa

Biobased Engine Oils Possible via CORS

For the last 20 years various small and large entities have been experimenting with biobased engine oils. Large entities abandoned the search after numerous trials and being faced with the ever changing specifications. Other smaller groups created ventures and drove a truck filled with biobased engine oil, licking the dipstick at various stops to make a point for 'environmental friendliness' of the product. Yet others in Europe experimented with the Continuous Oil Recycling concept in diesel passenger cars. While complex esters derived from vegetable oils can become economical and stable enough to handle the engine environment, many of the current vegetable oil based technologies have lacked the necessary stability and cold temperature flowability to perform in the engines the way mature and perfected petroleum engine oils do.

In 2006, after 15 years of research focused on Industrial Lubricants and greases, the University of Northern Iowa expanded the scope of the research at its National Ag-Based Lubricants Center to include automotive oils and biofuels. The Center's researchers then began to explore the concept of CORS which they had briefly explored 10 years earlier with an agricultural machinery OEM.

Continuous Oil Recycling System (CORS) was born from an after-market product that was added to a diesel engine to continuously steal a small amount of engine oil and feed it into the fuel line of a diesel engine. Add-on reservoir containing engine oil would then be used to refurbish the crank case with fresh engine oil. As a result, after so many refueling, the operator would purchase oil for the add-on oil tank. The main problem with this approach was that the burning of used engine oil resulted in a significant, negative impact on the already hard to achieve emission standards.

UNI-NABL researchers, along with their industrial collaborators, modified the CORS concept to use vegetable-oil based engine oils and created a sophisticated ‘black box’ called the **CORS Convertor**. The CORS Convertor not only communicates with the main processor of the engine, but it also has its own sensors and transducers to monitor the conditions of the engine oil and engine fuel before introducing the oil into the fuel line. The CORS Convertor is an add-on unit that conceptually resides between the crank case and the fuel injectors and ensures that the recycling of the oil as fuel is performed in a way that is beneficial to the engine.

CORS Convertor utilizes an array of sensors plugged into the crankcase to monitor the physiochemical conditions of the engine oil, which is made of a mixture of modified vegetable oils and minimal amount of performance enhancing additives. The CORS Convertor continuously draws oil samples from the crank case and in addition to evaluating the oil; it conditions it as needed, to be used as fuel. In effect , the CORS Convertor is like a human kidney that ensures what is fed into the fuel system is cleansed of harmful constituents and is only sent to the fuel system when the engine conditions indicate the best time to use this oil as fuel. For example, a cold engine would not receive vegetable based engine oil into its fuel system, and the amount of oil that is fed into the fuel system is varied based on the ability of the engine’s combustion process to provide the cleanest combustion possible.

Whereas the original idea of feeding used engine oil into the fuel system had a negative impact on the emissions, the CORS combined with the Convertor can actually improve exhaust emissions.

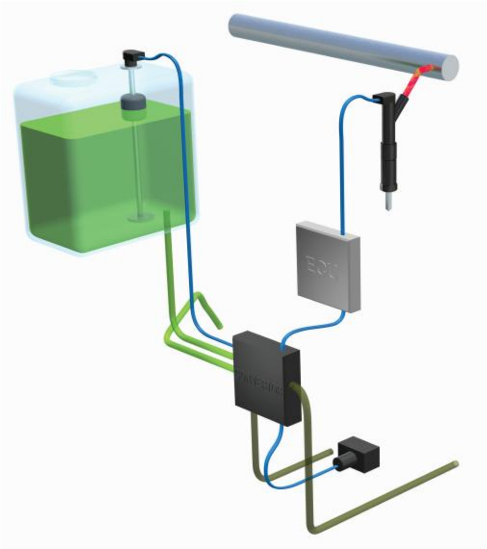


Figure 1: Conceptual Representation of the CORS Components Plus the Engine ECU and Injector

Stationary Diesel Engines for CORS

Initial target market for the CORS is the stationary diesel engines where addition of an oil tank next to the fuel tank does not present the logistic problems of dealing with mobile equipment. To test the concept, however, three John Deere diesel engines were prepared and instrumented for testing using an Eddy Current dynamometer to test the engine under various load conditions. Figure 2 shows the engine, dynamometer, and monitoring equipment associated with the data collection system.

Figure 2: 120 HP John Deere Diesel Engine

Figure 3: 300 HP Eddy Current Dynamotor

The engine and dynamometer senor and transducers were interfaced with a data monitoring system to allow mentoring of the engine performance.

Figure 4: Monitoring Equipment Associated with the Engine and Dynamometer

For testing the engine, the maximum load and horse power setting was selected in order to accelerate the degradation of the oil. The four cylinder engine was rated at 120 Hp and the test parameters were set to simulate the full load at about 120 HP with a range of +/- 5 Hp.

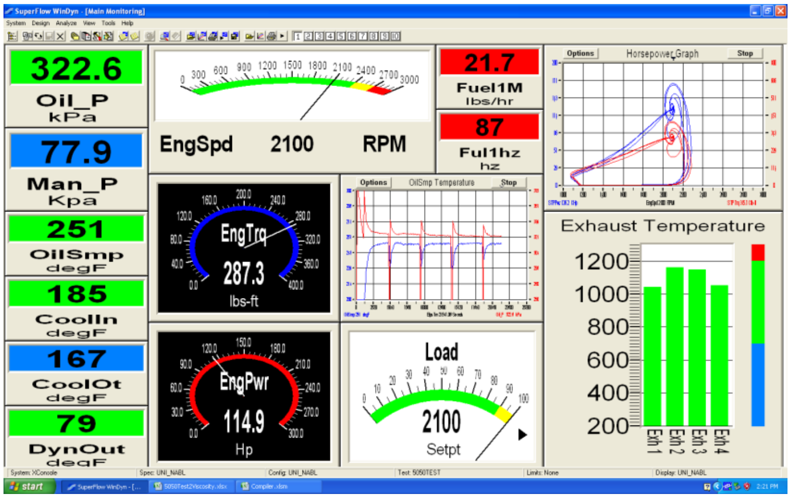


Figure 5: A screen Shot of the Parameters Set for the Engine Tests

It is assumed that data gathered using these diesel engines will be transferrable to the stationary engines used as back up or small scale electric generators for utility companies, hospitals, and any place using diesel engine for electric generation or for pumping.

When the engine starts, regular diesel engine fuel is used without any mixing with the engine oil. The CORS Convertor in the meantime, draws small quantities of the oil from the engine via a positive displacement pump. The physio/chemical properties of this oil are constantly monitored by an array of sensors placed in the crank case oil. The CORS Convertor receives the oil and depending on its conditions sends it through a number of conditioning steps based on a proprietary design that could include physical and chemical filtration, addition of chemical catalysts, heating or cooling, etc. Some of this oil will remain in the CORS Convertor in a small reservoir ready to be sent into the fuel system while some of it might return back to the engine crank case if not needed at that moment.

The CORS Convertor has three oil lines attached to it; one for bringing in the engine oil for analysis and conditioning, one from the fresh oil reservoir for preparation for injection into the crank case to replenish the used oil, and one to the crank case to inject of either fresh oil, excess re-conditioned oil, or a mixture of fresh and reconditioned oil back into the crank case (Figure 6). When the data from the engine Electronic Control Unit (ECU) and the engine sensors from CORS indicate that the conditions are right to feed the oil into the fuel, a small quantity, currently not exceeding 2% of the weight of the fuel being consumed, is sent into the inlet side of the fuel injector pump. This creates a B-2 biodiesel fuel and since the vegetable oils have shown to improve the lubricity of low sulfur fuels, the result is a tribiologically more effective fuel. The conditioned oil is void of most of the impurities often found in the used engine oils. Also, the oil is conditioned to proper temperature and at 2% level, is highly diluted in the fuel. This results in the minimal impact on the power output of the engine due to the naturally lower BTU/volume

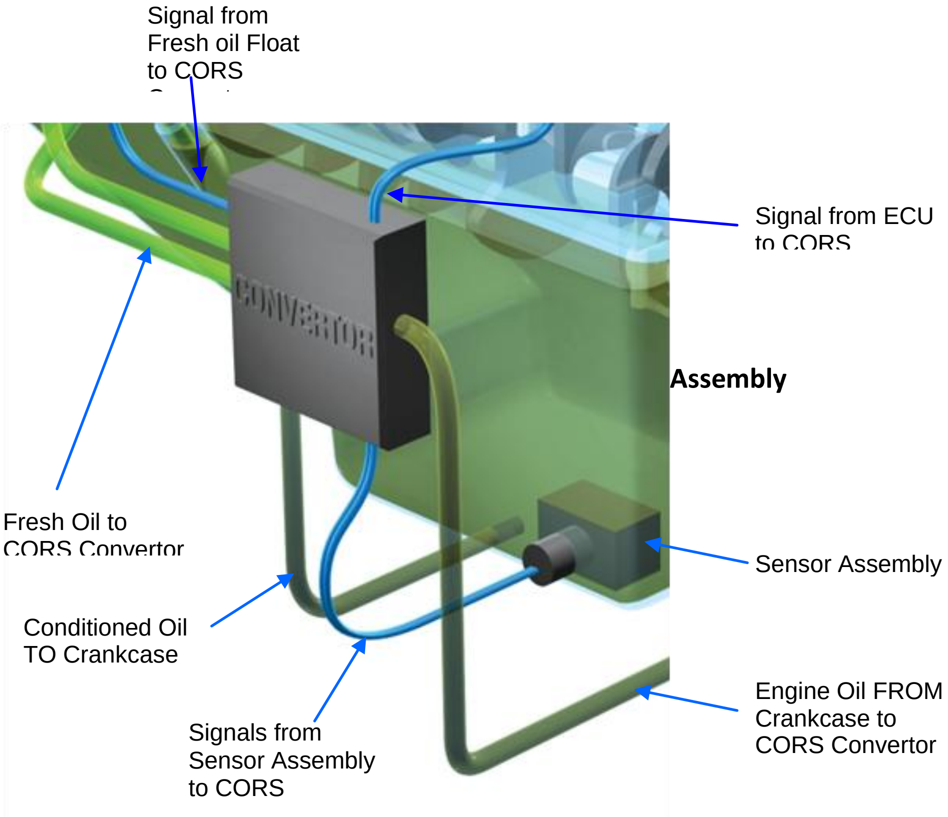


Figure 6: CORS Convertor and the Sensor Assembly

(list BTU/or Calorie content of High Oleic [soy] Bean Oil (HOBO) and #2 diesel for comparison) content of vegetable oils compared to say #2diesel fuel.

The system does require adding on the sensor assembly into the crank case and the CORS Convertor to the engine. The CORS Convertor requires a 12 V power supply which will be powered by the engine battery and has its own positive displacement pumps for drawing the used oil from the crankcase and injecting the conditioned oil or the fresh oil into the crankcase. The fresh vegetable engine oil reservoir can be placed anywhere close to the engine. For stationary diesel engines, the fresh vegetable based engine oil will sit in the proximity of the fuel tank with plumbing to the CORS Convertor.

Figure X sows an illustration of the complete system. The fresh oil reservoir is monitored by a float system controlled by the CORS Convertor and could shut the engine down in case the oil reservoir is depleted; the same way the fuel depletion could shut down the engine.

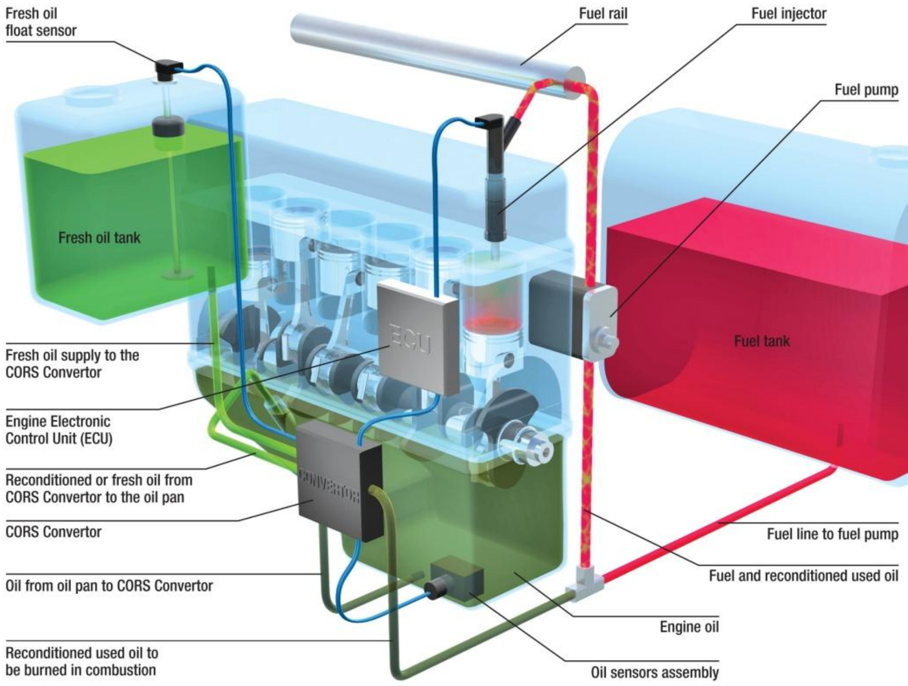


Figure 7: An illustration of the complete CORS/Engine system

Selected Test Results

A review of several diesel engines, commonly used for agricultural machinery, allowed the researchers to determine the time residency of the oil in the crankcase at different oil consumption rates. Table 1 shows the reservoir sizes and the percentage of the oil that has to be removed from the crank case in order to meet the desired residency time.

In order to test the concept, UNI-NABL researchers prepare a pure vegetable base oil with a viscosity of 107 Centistokes at 40 ˚C as a replacement for 10W-40 diesel engine oil that has a viscosity of 120 Centistokes at 40 ˚C. The reason for the lower viscosity is because the vegetable base oil has a much higher viscosity index of about 220; as compared to equivalent petroleum oil with a VI of about 100. This allows the use of thinner oil and less starting torque requirement, while maintaining the viscosity of the oil at the operating temperatures.

The viscosity of the oil was checked every hour, knowing that increase in viscosity is the sign of oxidation. After some initial shearing of the oil, the viscosity began to increase after about 10 hours of operation (Figure 8). During this test, the CORS was turned off and no oil was being removed from the crank case for burning as fuel. The untreated vegetable oil showed stable viscosity for about 10 hours at full load before the viscosity began to increase rapidly and the test was terminated after 14 hours. This viscosity increase was not unexpected as similar viscosity changes had been observed in high pressure-high temperature hydraulic pump tests. Since the engine was running at full load, it is anticipated that a lower level of engine load could increase this operational performance to several hours more than the 10 hours.

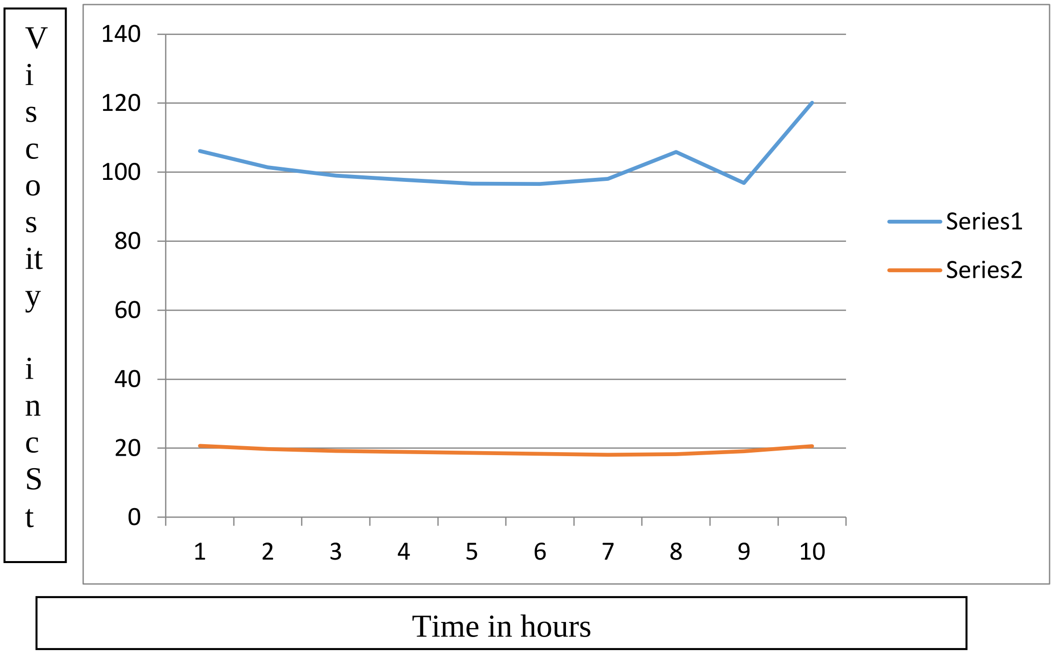


Figure 8: Untreated Vegetable Oil Performed about 10 hours before testing stopped due to increase in viscosity

The following figure shows the change of the viscosity due to shear when initially introduced into the engine, and the viscosity at 100 ˚C for engine oils with 50-50 vegetable oil/petroleum mix which ran for 100 hours without load at a high idle setting of 2000 rpm. For this test the CORS was on to remove oil from the crank case and replace it with fresh oil, but the reconditioned oil was not being fed into the fuel system to avoid introducing the fuel variable at this point.

This test was also repeated for 100 hours at full engine load with the same results. The oil in the engine remained stable in terms of viscosity and visual inspection of engine components did not indicate and abnormalities.

Next, the same mixture of and 75-25 vegetable oils/petroleum was tested with the engine at full load, and the viscosity of the oil remained flat for the entire 100 hours with CORS on. All other variables in the engine indicated that the mixture could be run in the engine indefinitely as long an oil volume equal to 2% of the fuel consumption is removed and replaced with fresh oil. Since similar tests have been performed on the fuel using 2% purified vegetable oil in the fuel system without any impact on the engine performance, the researchers were convinced that CORS is viable and will run a clean engine and a clean combustion indefinitely. The development activities are on-going for optimizing the size and components of the CORS Convertor, improving the accuracy of the sensors assembly, and optimizing the algorithm used on the CORS processor. The engine oil is undergoing further improvements to replace the25% petroleum portion of the blend with additives in the 75% vegetable oil portion. While the engine tests are continuing in the laboratory, field tests sites are being identified to for long term tests. Figure 9 presents the viscosities at 40C and 100C and the calculated Viscosity Index for each of ten samples taken during a 100-hour engine test. The viscosity was initially reduced due to the shearing of additives and then remained stable during the test.

Economically, when the oil is consumed at the rate of 2% of the fuel consumption, its residency in the engine would range from 10 to 50 hours depending on the size of the engine and crankcase. Since the residency of the oil is limited, there is reasonably little need for and as a result little cost for the performance enhancing additives. With the estimated cost of the CORS-based engine oil ranging from $4-$8/gallon tracking soybean oil prices at $3.00-$7.00/gallon, as long as diesel fuel prices are over $4.00/gallon, then the oil cost would be zero. This is because the oil would be used as lubricants first and then would be brunt with almost par value for the fuel. The inconvenience of having to resupply the fresh oil tank would be compensated with the elimination for the need to change the engine oil. Understandably, CORS is still in developmental stage, but it promises to deliver the first real biobased oil based engine lubricant technology based on vegetable oils. With combination of ingenuity in adapting some new hardware to overcome the shortcoming of vegetable oils, a vegetable oil based engine oil may be at hand.

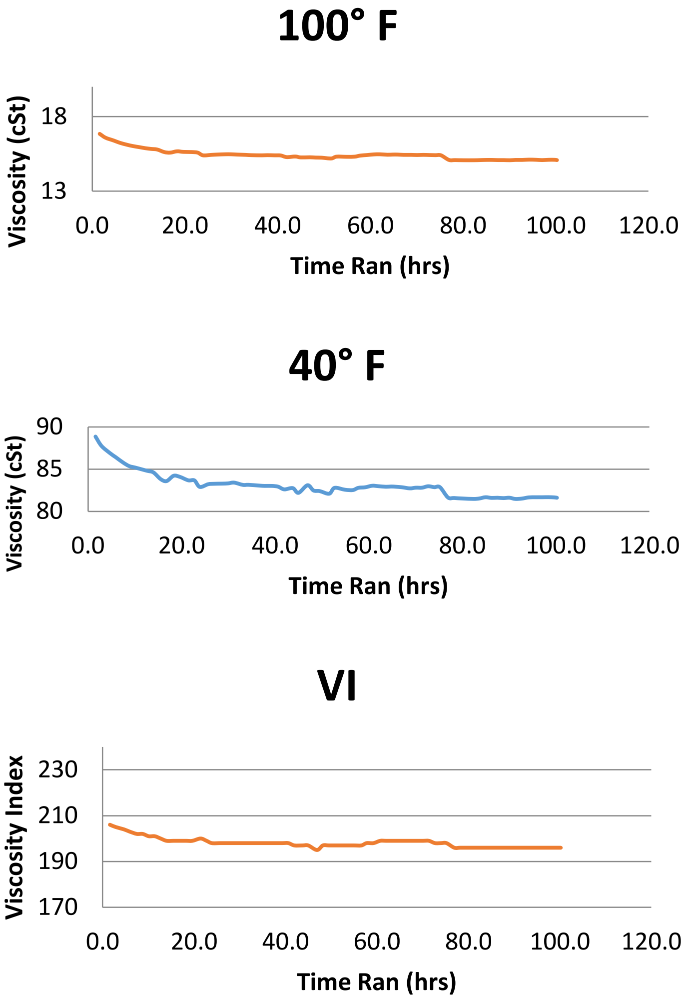


Figure 9: Viscosities at 40C and 100C and Viscosity Index of samples taken during the engine test

Figure 10 presents a sampling of the pictures of fuel injectors and samples taken of various engine tests performed.

Figure 11: A sampling of pictures of fuel injectors and samples taken of various engine tests

A number for vegetable oils were investigated for possible use in the CORS project. To that end, the soot tendencies of several vegetable oils were studied. These oils were later analyzed for their fatty acid profiles and performance as well as physiochemical properties. The following report the results.

Soot Tendency in Bio-Based Oils

The goal of this task was to determine the soot tendency of various vegetable base oils in order to compare and predict soot build up in engines by combusting specific oils at high temperatures.

Procedure

To determine soot tendency, Carbon Residue testing was performed on each oil per ASTM method D4530. In this test the sample is weighed into a vial and heated to 500°C under a nitrogen atmosphere for 15 minutes. The residue remaining in the vial is weighed and reported as a percent of the original sample as % Carbon Residue. Three vials were tested for each oil and the average is reported. After experimenting with various sample sizes it was determined 2.5g of sample using the large sample vials supplied the greatest amount of sample while avoiding boil over.

Results

Below are the results for the carbon residue testing listed alphabetically. To determine if there is a correlation with fatty acid profile and soot tendency the major fatty acid component of each oil is also listed.

Base Oils	Avg Carbon Residue (%)	Major Fatty Acid Component (%)
Apricot Kernel Oil	0.44	Oleic Acid (63.9)
Avacado Oil-Refined	0.52	Oleic Acid (64.5)
Castor Oil-Refined	0.25	Ricinoleic Acid(~85)
Coconut Oil-Refined	0.16	Lauric Acid (46.6)
Corn Oil	0.52	Linoleic Acid (56.0)
Cottonseed Carrier Oil	0.52	Linoleic Acid (51.5)
Flaxseed Oil	0.90	Linoleic Acid (~47)
Grapeseed Oil	0.48	Linoleic Acid (63.8)
Hempseed Oil-Unrefined	0.78	Linoleic Acid (54.4)
Jojoba Golden Carrier Oil	0.00	Gadoleic Acid (71.8)
Macadamia Nut Carrier Oil	0.28	Oleic Acid (60.4)
Palm Oil-Refined	0.22	Palmitic Acid (45.5)
Poppyseed Carrier Oil	0.46	Linoleic Acid (70.1)
Ricebran Carrier Oil	0.38	Oleic Acid (46.4)
Safflower Oil	0.32	Linoleic Acid (73-79)
Sesame Carrier Oil	0.44	Linoleic Acid (46.2)
Sunflower Oil-Organic	0.28	Oleic Acid (79.0)
Walnut Carrier Oil	0.62	Linoleic Acid (59.6)

Flaxseed Oil	0.90	Linoleic Acid (~47)
Hempseed Oil-Unrefined	0.78	Linoleic Acid (54.4)
Walnut Carrier Oil	0.62	Linoleic Acid (59.6)
Corn Oil	0.52	Linoleic Acid (56.0)
Cottonseed Carrier Oil	0.52	Linoleic Acid (51.5)
Avacado Oil-Refined	0.52	Oleic Acid (64.5)
Grapeseed Oil	0.48	Linoleic Acid (63.8)
Sesame Carrier Oil	0.44	Linoleic Acid (46.2)
Poppyseed Carrier Oil	0.46	Linoleic Acid (70.1)
Apricot Kernel Oil	0.44	Oleic Acid (63.9)
Ricebran Carrier Oil	0.38	Oleic Acid (46.4)
Safflower Oil	0.32	Linoleic Acid (73-79)
Macadamia Nut Carrier Oil	0.28	Oleic Acid (60.4)
Sunflower Oil-Organic	0.28	Oleic Acid (79.0)
Castor Oil-Refined	0.25	Ricinoleic Acid (~85)
Palm Oil-Refined	0.22	Palmitic Acid (45.5)
Coconut Oil-Refined	0.16	Lauric Acid (46.6)
Jojoba Golden Carrier Oil	0.00	Gadoleic Acid (71.8)

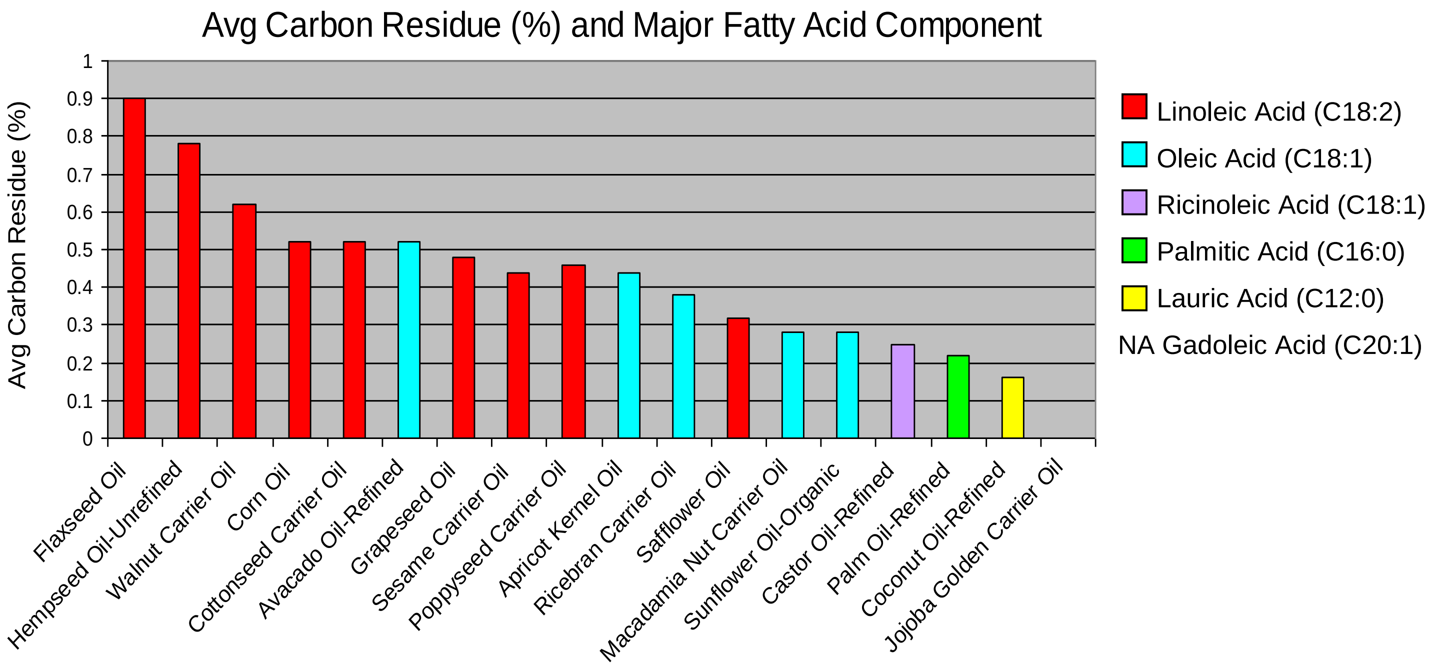


Figure 12: Average Carbon Residue and Major Fatty Acids Components

Conclusions on Carbon Residue Testing

Using the tables and graph above, a trend can be seen of % Carbon Residue when compared to the fatty acid composition of the oils. It can be seen on the graph that the oils with the highest carbon residue are composed mostly of linoleic acid, which is an 18 carbon fatty acid with 2 double bonds (C18:2). Oleic acid has 1 fewer double bond than oleic acid, and oils composed mostly of this fatty acid have slightly lower % carbon residue. It appears from the oils tested that as the number of double bonds and/or the fatty acid chain length decreases, the % carbon residue also decreases. The one exception is Jojoba oil, which is composed of a comparatively long fatty acid, Gadoleic acid, with a 20 carbon chain and one double bond. This oil showed no carbon residue.

Another observation was that of this small sample of oils, the refined and carrier oils typically had lower carbon residue values, though further testing would be needed to make a definitive conclusion.

While these are important observations, it must also be noted that the range of results covers only 1% Carbon residue, and this is overall a small amount of residue and a not an ideal range for data comparison. However these results can give some insight on the oil behavior in a combustion engine, as oils with a high average carbon residue value will most likely cause a large volume of soot to build up in the engine, making it inefficient to use in the industry. Further research would be warranted in this area.

Task B: BIOBASED GREASE MANUFACTURING ENERGY EFFICIENCY RESEARCH

> Investigate the potential to process biobased lubricating greases with microwave transmitters as the process heat source, and identify benefits and drawbacks inherent in this unique heating method for biobased products, including vegetable oils.

The use of microwaves for processing biobased products has been a revolutionary project that was supported under this project. A patent applied for and the technology has been licensed to a major mixing tank manufacturing company in Iowa. The following presents contents of two papers that were presented at both the NLGI and ELGI annual conferences.

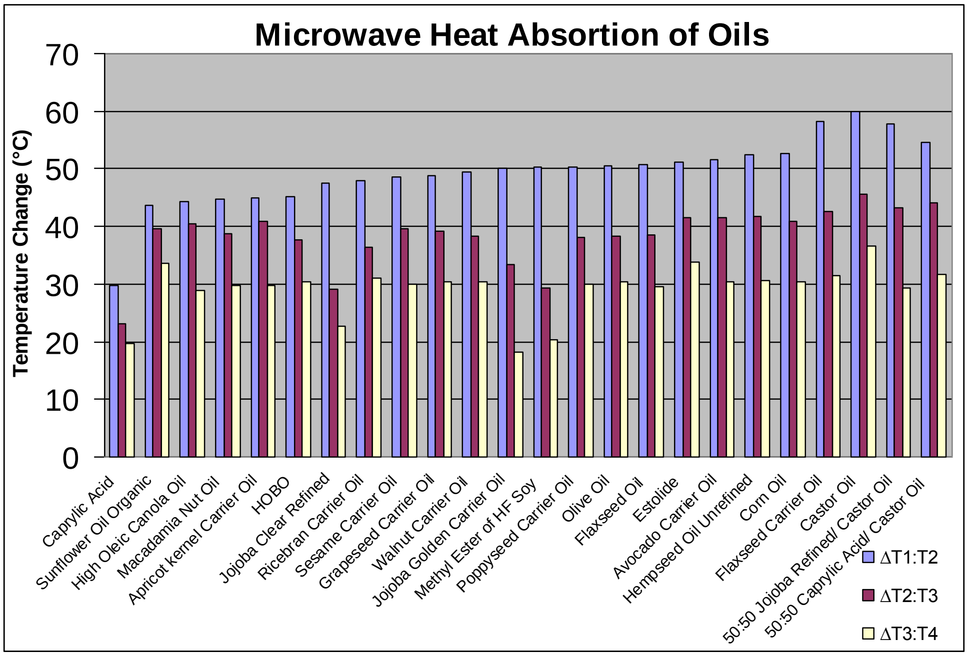
Lab Report for Microwave Power Heat Absorption For Vegetable Oils

Introduction

The task for this experiment was to determine the amount of heat absorption between many different vegetable oils and a methyl ester. All 21 samples were heated in the microwave three times for a minute each and the temperature was recorded before and after each heating. An Emerson 1100 watt microwave was used for this project.

Procedure/ Data

Eight ounces of each oil sample was needed for this work request, which converts over to 226.796 grams. Each sample of oil was placed in a 400 ml beaker for accurate heating measures within each oil in the microwave. The initial temperature was taken before any heating was done (T1), using a digital thermometer. Then the sample was placed into the microwave for 1 minute. The temperature was taken immediately after the one minute and recorded as T2. This was repeated two more times and recorded as T3 and T4. The changes in temperature were then recorded; ΔT1:T2, ΔT2:T3, ΔT3:T4, ΔT1:T4.



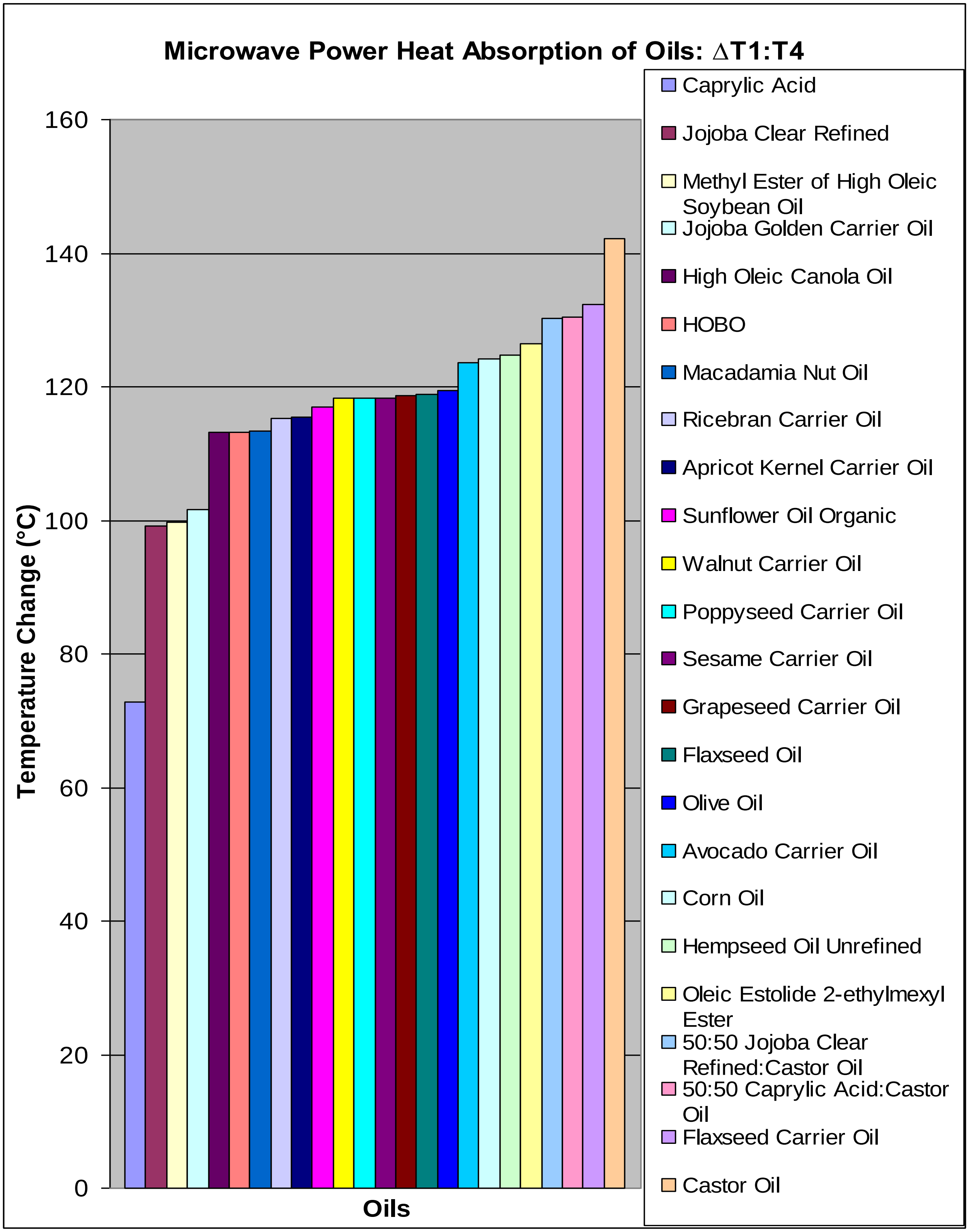


Figure 13: Heat absorption of various vegetable oils in microwave

Table 2: Heat absorption of various vegetable oils in microwave (all temperatures in C)

ID#	Sample	Mass	T1	T2	T3	T4	ΔT1:T2	ΔT2:T3	ΔT3:T4	ΔT1:T4
07-066	Caprylic Acid	226.8	21	50.8	74	93.8	29.8	23.2	19.8	72.8
09-025	Jojoba Clear Refined	226.7	20.9	68.4	97.5	120.1	47.5	29.1	22.6	99.2
09-106	Methyl Ester of HF Soy	226.8	20.7	70.9	100.2	120.5	50.2	29.3	20.3	99.8
09-026	Jojoba Golden Carrier Oil	226.7	20.9	71	104.5	122.6	50.1	33.5	18.1	101.7
09-096	High Oleic Canola Oil	226.8	21	65.3	105.8	134.1	44.3	40.5	28.8	113.1
10-088	HOBO	226.8	21.3	66.4	104	134.5	45.1	37.6	30.5	113.2
10-103	Macadamia Nut Oil	226.8	20.7	65.5	104.3	134.1	44.8	38.8	29.8	113.4
09-021	Ricebran Carrier Oil	226.8	21.1	69	105.4	136.4	47.9	36.4	31	115.3
09-023	Apricot kernel Carrier Oil	226.8	21.2	66.2	107	136.7	45	40.8	29.7	115.5
09-032	Sunflower Oil Organic	226.8	21	64.7	104.2	137.9	43.7	39.5	33.7	116.9
09-034	Walnut Carrier Oil	226.8	20.6	70.1	108.5	138.8	49.5	38.4	30.3	118.2
09-028	Poppyseed Carrier Oil	226.8	21	71.2	109.2	139.2	50.2	38	30	118.2
09-033	Sesame Carrier Oil	226.8	21	69.7	109.3	139.3	48.7	39.6	30	118.3
09-036	Grapeseed Carrier Oil	226.7	21.3	70.2	109.4	139.9	48.9	39.2	30.5	118.6
10-098	Flaxseed Oil	226.8	20.2	70.9	109.5	139	50.7	38.6	29.5	118.8
09-042	Olive Oil	226.7	20.9	71.4	109.8	140.3	50.5	38.4	30.5	119.4
09-024	Avocado Carrier Oil	226.8	21	72.6	114.1	144.6	51.6	41.5	30.5	123.6
09-041	Corn Oil	226.8	20.8	73.5	114.4	144.9	52.7	40.9	30.5	124.1
09-037	Hempseed Oil Unrefined	226.8	21.5	73.9	115.6	146.3	52.4	41.7	30.7	124.8
08-243	Estolide	226.8	20.7	71.8	113.3	147.2	51.1	41.5	33.9	126.5
09-039	Flaxseed Carrier Oil	226.8	21	79.2	121.9	153.4	58.2	42.7	31.5	132.4
09-020	Castor Oil	226.8	20.6	80.5	126.2	162.7	59.9	45.7	36.5	142.1
10-169 /09-020	Jojoba Clear Refined/ Castor Oil	113.4/ 113.4	20.3	78	121.2	150.5	57.7	43.2	29.4	130.2
07-066/ 09-020	Caprylic Acid/ Castor Oil	113.4/ 113.5	21.3	75.8	120	151.7	54.5	44.2	31.7	130.4

Conclusions for Heat Absorption of Vegetable Oils

As the data shows, castor oil had the highest heat absorption among the oils and jojoba clear refined had the smallest amount of heat absorption. However, the most of the oils did not vary too significantly among their Δ values. Among some of the higher heat absorbers were flaxseed carrier oil, hempseed oil, and corn oil. Other oils that absorbed the least amount of heat were methyl ester of HF soy and jojoba golden carrier oil.

When compared to the dielectric breakdowns that we had previously tested, the results are not conclusive when thinking in terms of polarity. Jojoba clear had the lowest change in temperature and also the lowest dielectric breakdown value. Flaxseed oil had the highest dielectric breakdown but it did not have the highest change in temperature, but it was among one of the higher changes in temperature. The others all show up in the middle with some low dielectric values and high ΔT's and vice versa.

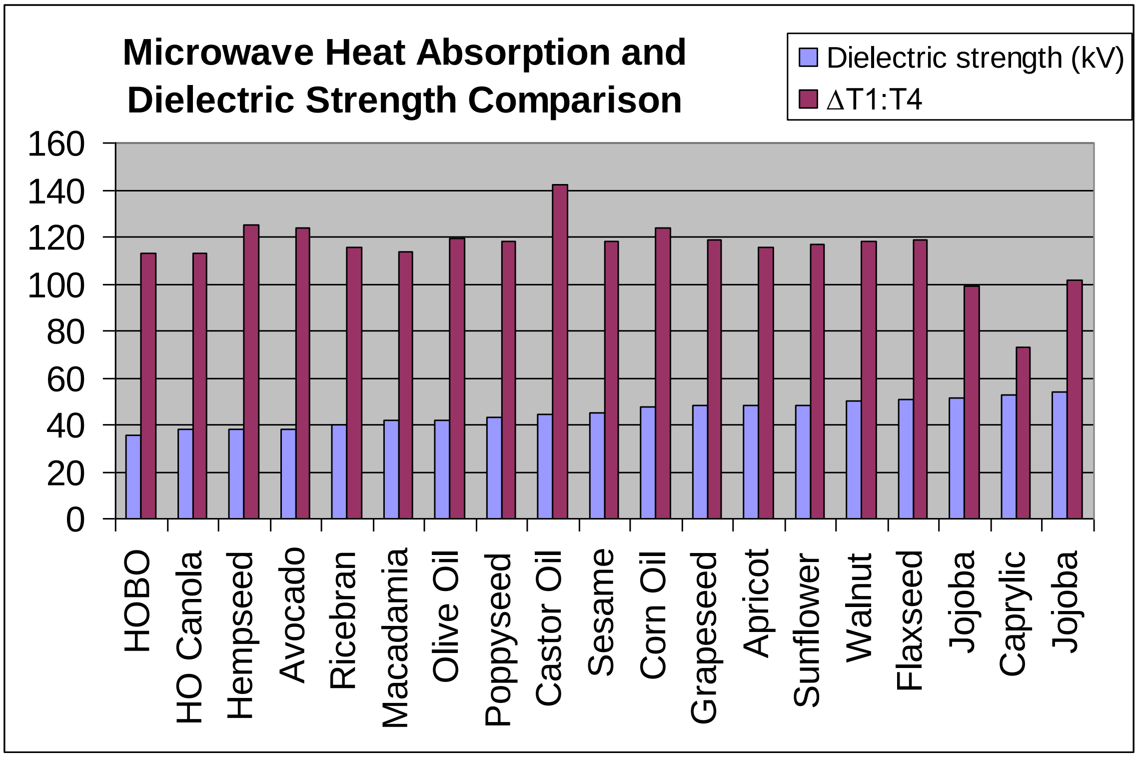


Figure 14: Comparison of microwave heat absorption and dielectric strength

:

When mixing caprylic acid with castor oil to see the effect on temperature change it was found that the heat absorption for castor oil only dropped ~ 12 degrees Celsius. Thus in turn it raised the caprylic acid absorption significantly from 72.8 degrees to 130.4 degrees. Castor oil was also mixed with the next lowest oil of heat absorption, jojoba clear refined oil. Jojoba clear had about 26 degrees more absorption than did caprylic acid, yet when mixed with castor oil the final heat absorption was 130.2, very close to that of the caprylic acid and castor mix. This suggests that castor oil is a very strong heat absorber.

Appendix

Data Table from Work Request #2 Report

Sample #	Sample Name	Dielectric strength (kV)
10-048	HOBO	35.4
09-096	High Oleic Canola Oil	38.0
09-037	Hempseed Oil	38.08
09-024	Avocado Oil	38.24
09-021	Ricebran Carrier Oil	39.8
09-038	Macadamia Nut Carrier Oil	41.78
09-042	Olive Oil	42.2
09-106	Methyl Ester of HF Soy	43.0
09-028	Poppyseed Carrier Oil	43.39
09-020	Castor Oil	44.42
09-033	Sesame Carrier Oil	45.08
09-041	Corn Oil	47.44
09-036	Grapeseed Carrier Oil	48.0
09-023	Apricot Kernel Oil	48.18
09-032	Sunflower Oil	48.48
09-034	Walnut Carrier Oil	50.13
09-039	Flaxseed Oil	50.68
09-025	Jojoba Refined	51.22
09-318	Caprylic Acid	52.9
09-026	Jojoba Golden Carrier Oil	54.2

Data Table from previous Work Report

Substance	Dielectric Breakdown Strength (Kv)
HOBO (08-204)	34.25
Estolide	23.9
Ethanol	1.4
Air	6.6
Jojoba oil (09-025), refined	25.5
Jojoba oil (09-026) crude	31.9
Castor oil (09-020)	43.6
Apricot kernel oil	48.1
Sunflower oil	48.5
Hempseed oil	38.1
Sesame Carrier oil	48.4
Macadamia nut carrier oil	41.8
Corn oil	47.4
Avocado oil	38.2
Flaxseed oil	50.7
Caprylic acid	52.9

Table: Heat absorption of Silicone oil by microwaves

Silicone Grease Project- Microwave Test									
	Mass	**T0**	**T1 (1 min)**	**T2 (2 min)**	**T3 (1 min)**	**ΔT0:T1**	**ΔT1:T2**	**ΔT2:T3**	**ΔT0:T1**
11-120 ESCO 200 CS Silicone Oil	113.8	20.5	37.7	62	**67.8**	17.2	24.3	5.8	**47.3**
11-121 ESCO 350 CS Silicone Oil	112.9	20.1	32.4	53.5	**62.2**	12.3	21.1	8.7	**42.1**
11-122 ESCO 500 CS Silicone Oil	114	20.4	33.1	57.2	**65.6**	14.7	24.1	8.4	**45.2**
11-123 ESCO 1000 CS Silicone Oil	113.1	20.3	35.7	58.1	**68**	15.4	22.4	9.9	**47.7**

An Update on Manufacturing Biobased Grease Using Microwaves

Lou A. Honary

National Ag-Based Lubricants Center,
University of Northern Iowa:
360 Westfield Avenue
Waterloo, Iowa, 50701, USA
www.uni.edu/nabl

Synopsis:

In recent years, there has been a growing interest in the production of biobased and biodegradable greases. The use of these newer raw materials has introduced a different level of complexity, and sometimes unpredictability to grease making. This paper presents an update report on the construction and operation of a production quantity, microwave based, and biobased grease manufacturing system. Changes and improvements that were made to the equipment and the process are explained, along with recommendations for future research.

Manufacturing Biobased Grease Using Microwaves: Use of microwaves in grease manufacturing process

Attempts to increase predictability and consistency of greases have been described by the authors in earlier papers. To increase predictability, manufacturers have attempted to use preformed dehydrated soaps, preformed “oily” soaps (high consistency base grease), micro-sizing of lithium hydroxide, creation of slurry to reduce reaction time, and introduction of polymer based thickeners. Additionally, interesting new manufacturing processes have been attempted to reduce variability of the final products. Those include introduction of pressurized vessels, like the “contactor” reactor, introduction of continuous (vs. batch) processes, and alternative heating techniques, which are described further in this report. This paper provides an overview of actual grease manufacturing using industrial microwaves. The initial processing unit has been modified to address the shortcomings observed in large volume production batches. Modifications made to the reactor and the differences in the outcome as compared with conventional jacketed grease reactors are discussed.

Description of the basic process

The process of making soap based greases includes neutralizing an [fatty] acid with a base at temperatures of about 130 °C (266 °F), which results in the formation of soap and water. Then the product is heated to nearly 200 °C (392 °F) to boil off the water. Then cooling oil is mixed with the soap to make grease. During the reaction process, especially if water is present in the product, foaming can occur, which would require some form of suppression scheme, such as using pressurized vessels, slow introduction of the base material, and/or the use of dehydrated base material, e.g. anhydrous lithium hydroxide.

When properly processed the result would be the highest yield from the same amount of thickener (combination of fatty acid + the chemically basic material) and the same quantity of oil. The presence of un-reacted lithium, failure to remove all the water, or overheating of the product will result in quality issues with undesired variability in the final product. As a result, careful monitoring of variables and the expertise of the operator become important factors. Sometimes grease manufacturers initially produce thicker grease than desired and then thin it back down by adding additional oil, to reach the desired consistency. Ideally, however, the goal should be to manufacture the grease in a way that a predictable product at the proper consistency (thickness) is achieved from the process.

Vegetable Oil Based Greases

The University of Northern Iowa’s National Ag-Based Lubricants Center (UNI-NABL) has been engaged in research and development of manufacturing processes and biobased greases made from vegetable oils. Several commercial grease products, including large volumes of rail curve grease made from soybean oils, owe their origin to this center. Since vegetable oils, in general, range in viscosity from 35-45 cSt at 40 °C, UNI-NABL processes have generally included the introduction of higher viscosity vegetable oils to increase the viscosity of the starting base oil.

Vegetable oils exhibit unique behavior when exposed to high temperatures. In the case of some vegetable oils, once the oil temperature exceeds 150 ^{o}C (302 ^{o}F), the oil begins to oxidize rapidly, and if steps are not taken to remedy this rapid oxidation, the product will begin to polymerize, resulting in irreversible change. In such cases, the product could partially or fully polymerize or change state from a soap into a polymer with little lubrication value. But, several methods exist for stabilizing soybean or other vegetable oils so they can be reacted with lithium and produce stable greases. A new patent pending process developed at the UNI-NABL Center has employed the use of microwave heating for the saponification reaction of vegetable oils with lithium.

It has been observed that when the vegetable oil is exposed to the surface of a hot plate, or to hot reactor walls in a jacketed reactor vessel, at temperatures of 350-450°C (662-842°F) the vegetable oil is immediately oxidized by a rapid chemical reaction at these high temperatures. Scraped-surface agitation continually replaces the oxidized layers with more oil on the wall surface. Energy wise, this method of heating is relatively inefficient because natural gas, fuel oil, or electricity must first be used to heat the heat-transfer oil with some degree of inefficiency. The hot external surfaces of the pipes, kettles, and other units lose a considerable amount of heat even when insulated. The heat loss becomes excessive when the ΔT between the heat transfer oil and the ambient temperature is large.

Vegetable oils, due to their polar nature, respond to microwave energy like water does, by vibrating, and can be effectively heated with surprisingly high efficiency. When polar molecules of vegetable oils are exposed to high energy microwaves, they absorb the microwave energy which results in a rapid heat rise. The molecules of mineral oils and non-polar liquids, when exposed to microwaves, do not vibrate. Instead they pick up speed and can rotate resulting in less friction at the molecular level and less heat rise.

To illustrate simply, one can visualize that the industrial microwaves at 915 MHz send waves of energy cycling 915 million times per second into the product, with each energetic sine wave changing from positive to negative to positive etc. The vegetable oil molecules, being dipolar with molecules having negative and positive poles, are forced to re-align themselves with the microwaves at this rate of 915 million times every second. This results in the oil molecules vibrating and impacting each other resulting in friction, which generates heat. The heat is then conducted throughout the product causing expansion and increasing impacts. Note most household microwaves operate at 2450 MHz as compared to industrial microwaves operating at 915 MHz.

There are means available for increasing the effective energy absorption of non polar materials like mineral oils, thus rendering them a better candidate for processing with microwave energy. But, the focus of this present paper is on the use of microwave energy for heating vegetable oils for grease processing and to: a) avoid degraded oxidation stability due to exposure to high temperatures at the point of contact with the walls of the heating vessels; b) reduce the time needed to heat the oil to the needed reaction temperatures; c) reduce the energy consumption by a more focused and instantaneous energy input; and finally d) reduce the level of fire hazard by eliminating the use of high temperature heat transfer oils. Examples of effective use of microwave energy for manufacturing of biobased grease are provided below.

Table 1 – Figure 1 shows results of exposure of 300 ml of mineral oil and vegetable oil to 90 seconds of microwave energy through a 1.75KW transmitter (2450 MHz); and then results of exposing mixtures of mineral oil and vegetable oil to the same level of 1.75kw of microwave energy.

90 Sec Microwave Exposure	Temperature	Temperature after	
Sample	at start (^{0}C)	90 Sec (^{0}C)	∆ Temperature
HOBO	22	109	87
Mineral Oil	23	39	16
5/95 HOBO/ Mineral Oil	23	44	21
10/90 HOBO/ Mineral Oil	24	51	27
25/75 HOBO/ Mineral Oil	24	60	36
50/50 HOBO/ Mineral Oil	23	75	52
75/25 HOBO/ Mineral Oil	23	96	73

*** 1.75KW microwave input**
*** * HOBO = High Oleic Bean Oil**

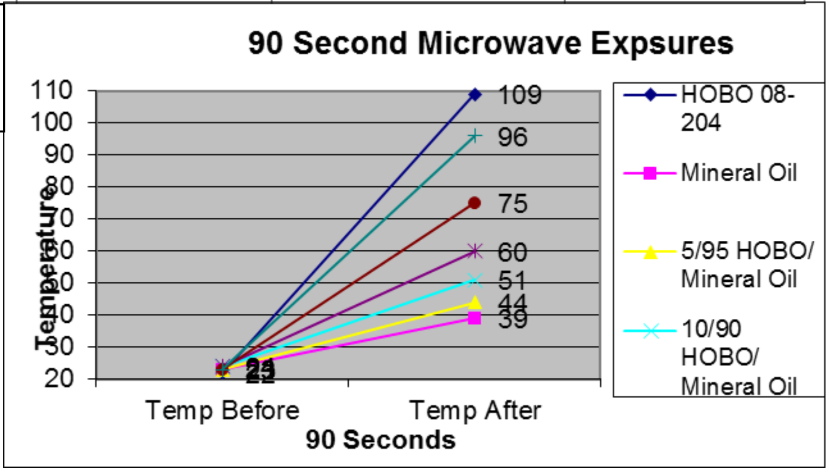


Table 1 - Figure 1: Absorption of Microwave Energy for Mixtures of Polar (vegetable oil) and Non-Polar (mineral oil) Materials

The use of a hot plate to simulate conduction and convection heating results is also demonstrated using a sample of 300 ml of vegetable oil on a hot plate heated to 300 °C (572°F) to record the time needed to raise the temperature to 160 °C. Similarly the same oil was exposed to microwave energy to reach to 160 °C (320 °F). The following chart (Figure 2) presents the results of this experiment, with noticeable differences in the time required.

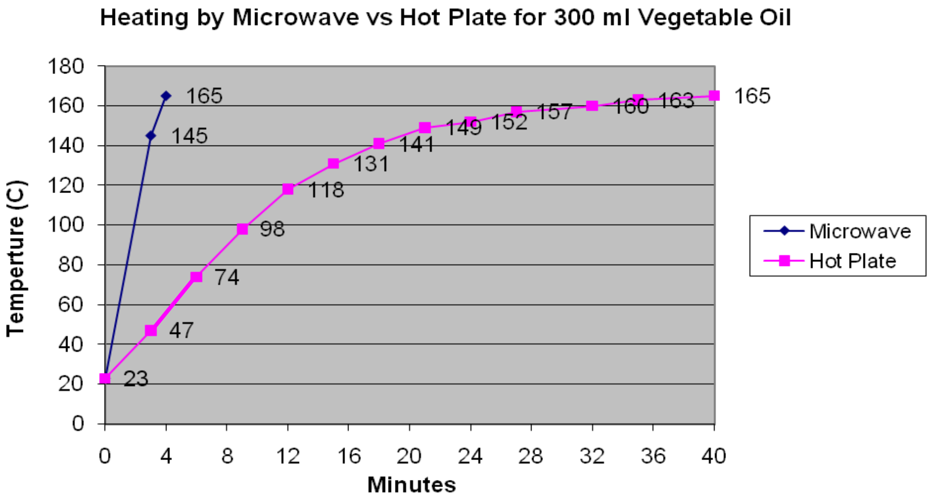


Figure 2: Time to Raise Temperature of Vegetable oil to 165 ^{0}C (329 °F) by Microwave VS by Hot Plate

Since the walls of the beaker are exposed to the ambient temperature, the heat loss on the hot plate method will increase and more accurate results would need an adiabatic environment. So, the faster heating in the microwave actually approaches adiabatic process when compared to the oil heated on the hot plate.

This faster heating as well as the uniformity of heating (no hotspots) is expected to cause less oxidation of the oil. To further investigate this, a sample of vegetable oils with known oil stability index (OSI), was heated to 165 °C (329 °F) on the hot plate for 6 hours, and a same size sample of the same oil was heated to 165 °C (329 °F) by microwave heating. This was then maintained at the same temperature by pulsing one minute of energy every 5 minutes for 6 hours. The two oils were then tested for oxidation stability index, using an Oxidation Stability Instrument. The results indicated that both oils oxidized due to extended exposure to heat, but the oil exposed to the heating on the hot plate had a change in oxidation Stability Index of 2.5 times that of the oil heated by microwave. In other words, the oil heated with microwave energy showed 2.5 times better oxidation stability. Future reports will show that these trials can be duplicated in larger quantities with higher levels of microwave energy.

Table 2: Impact of Heating Methods of Oxidation Degradation of Vegetable Oil

Vegetable Oil RBD-HOBO*	**Oxidative Stability Index**
OSI Before Heating	41.2
OSI After Heating – Hot Plate	9.12
OSI After Heating -- Microwave	23.28
*Refined-Bleached-Deodorized High Oleic Bean Oil	

Production Scale Microwave Grease Manufacturing

The following describes major components of a production scale microwave grease manufacturing system.

Microwave Source

The microwave source is a compact electrical unit that converts electrical energy in this case from a 480 Volt power source to microwave energy at 75 kW of power. The output microwave power is adjustable between 0 and 75 kW allowing for the exact amount of energy needed to be applied to the media being heated. The magnetron within the transmitter is considered consumable and would need to be replaced after some hours (several thousand) of operation depending on the severity of usage. In general these transmitters, like their smaller household versions, can provide years of operation without major maintenance requirements. The systems are networkable and troubleshooting can be accomplished remotely by the manufacturer of the system. Most components are easily replaceable by a qualified electrician making the transmitter a rather trouble free and clean heat source with a small footprint. Figure 2 shows a 75 kW transmitter with the inset showing the inside components.

Figure 2: Two 75 kW Microwave Transmitters

Grease Reactor

The grease reactor, in this case, was an atmospheric stainless steel tank that was adapted for manufacturing grease. Despite common misconceptions, provided that there is a sufficient mass of product to absorb the microwave energy, metal containers can be used for microwave heating. In order to facilitate circulation and mixing, the reactor was fitted with a pump that circulated the product from the bottom center of the tank into the vessel through four pipes welded to its sides. Figure 3, shows the reactor and some of its attachments.

Figure 3: An 800-Gallon Stainless Steel Grease Reactor

Energy Transfer Waveguides

The microwave energy can be effectively transmitted through special conducting ducts called waveguides. These are specially designed, usually aluminum, square ducts that allow microwaves to travel and go around the corners without any notable losses. The complexity of the design and construction of the waveguides are beyond the scope of this paper. But, this feature would allow the microwave transmitter to be placed close to, above, or away from the reactor. Figure 4 shows the waveguides as attached to the top of the reactor.

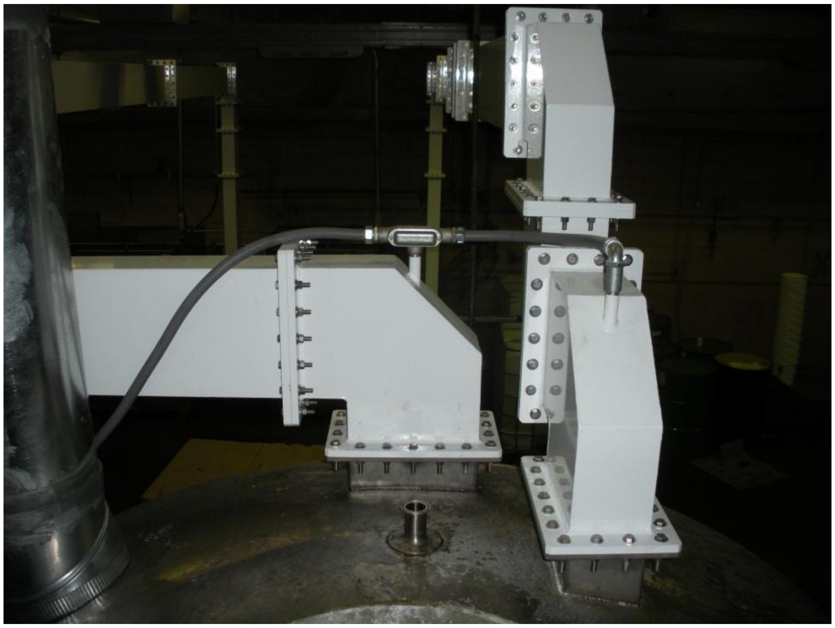


Figure 2: Two Waveguides from Two Transmitters Attached to Top of the Grease Reactor

Viewing the grease production process, or inspection of the product during the process can be performed through Viewing Ports on top of the reactor where, using a flash light, the operator could peak through to inspect the condition of the product while the microwaves are being applied. The concept incorporates a design that prevents microwave leakage, based on the size of the viewing ports. As is done with household microwaves, the screen that is placed behind the viewing glass on the microwave door is actually a metal plate whose thickness is dependent on the size of the holes of the screen. The larger the holes, the thicker the screen would need to be. Similarly there could be 2” or 4” inch pipes poking out of the reactor that would not allow microwaves to escape if their length is designed in consideration of calculated factors including the microwave wavelength. Figure 3 shows the design of viewing ports on top of the reactor and a corresponding viewing port on a household microwave. The viewing port on top of the reactor could also be used for the addition of additives and oils as needed.

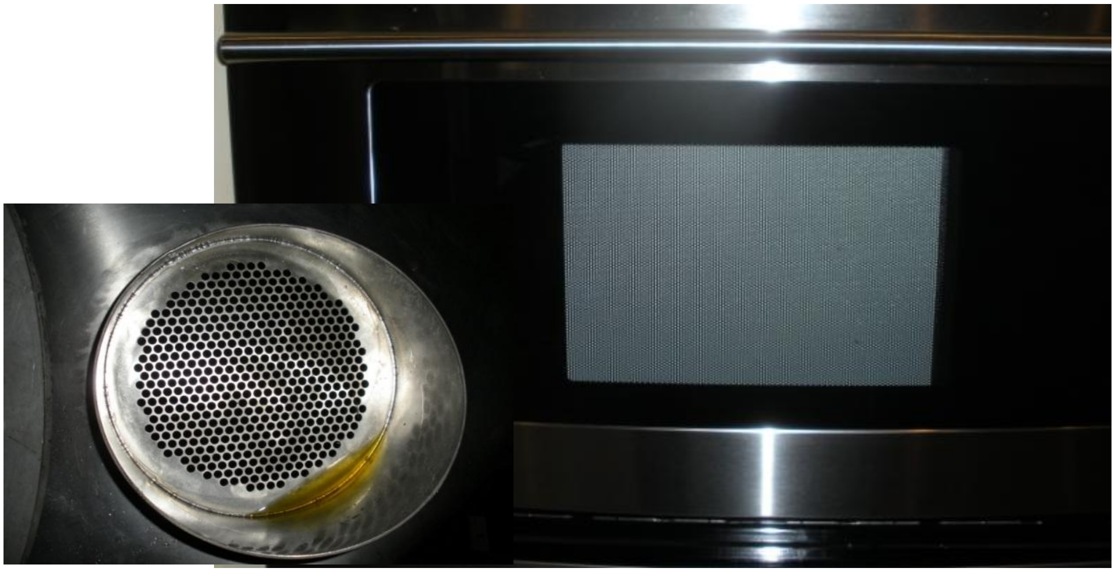


Figure 3: Viewing Ports on top of reactor (left) and a Household Microwave

Programming the Control System

A programmable logic controller (PLC) which can be interfaced with a number of sensors, can be used to control the microwaves power level. A temperature sensor placed at the bottom of the reactor was interfaced with the controller. Once the desired temperature and microwave power level is set on the touch screen controller the heating starts until the desired temperature is reached. At that time, the PLC would reduce the power level to near zero and would ramp up or down the power level as needed to maintain the set temperature. This assures a near perfect reaction temperature every time. Figure 4 shows the control screens when the temperature is rising to the set level, in this case 130 ^{o}C with the wattage for each transmitter set to 60 kW for a total of 120 kW input. When the set temperature is reached, the controller reduces the transmitters, in this case to 0 kW for one transmitter and 3 kW for the second one, thus, maintaining the temperature at 130 ^{o}C.

Figure 4A: The set temperature is 130 °C transmitters are at 60 kW each. Figure 4B: tank temperature is 129.5 °C and the controller has automatically adjusted the transmitters to 0 kW and 3 kW.

Process Time

In order to determine the efficiency of this method of heating, several experiments were conducted with neat vegetable oils. The reactor was charged with 400 gallons of soybean oil as the starting base oil, the temperature was carefully recorded during the process. The oil temperature was initially measured at 25.5 °C, and with the application of microwave energy at a microwave transmitter setting of 120 kW, it reached 110 °C.

While the single wall construction of the reactor, the lack of insulation, and changes in the ambient temperature could introduce some variability, the temperature rise was nevertheless recorded at about 2.7 °C per minute. It is estimated that with an insulated tank, this rise in temperature could increase to about 3 °C per minute. Other observations include, interestingly, the fact that if only one transmitter was used at only 60kW of microwave power input, the time required to reach the temperature would double with an almost direct relationship. The same reaction process was repeated for several batches with almost identical temperature rise in the same volume of the oil.

From the beginning of trial grease production, the operators observed a lighter soap color because there are no hot spots caused by the hot walls typically present in jacketed kettles; the microwave grease product is heated uniformly but not burnt. The reactions also seemed to be more complete as observed by the highest yields compared to previous batch data.

Improvements

To date, over 550,000 pounds of biobased greases have been manufactured using this first ever microwave based grease reaction process. Several modifications have been made to the system based on observed performance. First the transmitters that were sitting close to the reactor were moved back. This is not a trivial change as the concept illustrates the flexibility of using microwaves and the fact that the waves can travel as far as needed without significant losses. Figures 5A and 5B show the location of the transmitters before and after the move.

5A 5B

Figure 5A: transmitters within 5 feet of reactor; and Figure 5B: transmitters within 25 feet of the reactor.

Secondly, the viewing ports which were used also for ventilation and for addition of materials had to be revised to allow for better access. Viewing ports are effectively an opening into the reactor, capable of providing visibility inside the reactor while they simultaneously prevent the microwaves from escaping as described earlier. To provide a larger opening for the addition of solid materials into the reactor, a new opening was built on the manhole cover, consisting of eighteen 1 ½ in^2 (3.81 cm^2) ports. In order to ensure proper choking of microwaves, the height of this viewing port was determined to be 8 in (20.32 cm).

6A 6B

Figure 6A: Eighteen 1½ in (3.81 cm^2) ports to the reactor cover made adding solid materials easier; Figure 6B: the depth of each port was 8 in (20.32 cm) for choking of the microwaves.

A special microwave leak detector designed to test microwaves at either household frequencies of 2450 MHz or industrial frequency of 915 MHz was used to ensure no leakage of microwave energy. Figure 7 shows a microwave leak detector being used.

Figure 7: Microwave Leak Detector

Finally, the reactor has performed well for manufacturing both lithium and lithium complex greases. However, several attempts at making aluminum or calcium complex greases have proved that the mixing mechanism was not sufficient to properly mix the product. To improve the physical mixing of products that tend to thicken up in their entirety, like aluminum complex greases, the shaft of the single propeller was extended and two additional propellers were added. This increases the mixing performance of the system. Since lithium greases tend to fully liquefy at higher temperatures, the current system has shown to be effective for manufacturing such greases because the metal propellers always remain below the level of fluid while microwaves are applied. Research is underway to incorporate methods of mixing where the entire product can be mixed regardless of its thickness, and at the same time, without exposing the arms of the mixers to the microwaves. Figure 8a and 8b show changes in the mixing mechanism for the reactor.

Figure 8a Single propeller; and 8b: modified with three propellers

Conclusions

To date the use of microwaves for manufacturing grease has moved from the laboratory to production. The first ten months of the operation has shown promising results, with large quantities of product produced. At the same time, actual operation has pointed to some needed changes to the design of the reactor. The smaller footprint of these microwave systems and their relative cost effectiveness makes them highly desirable manufacturing methods. Smaller manufacturers would potentially be able to produce specialized greases in either small or large quantities at significantly reduced cost.

Microwave energy is highly efficient and can be focused to apply to the exact space to be heated. The microwave transmitter can be physically placed away from the actual reaction process, and potential microwave energy leakage can be “choked off” to nearly zero emissions, to meet established international safety standards. Additionally, microwave transmitters can be controlled through PLCs for accurate temperature control, by pulsing or reducing the intensity of the energy input.

The need for science based vs. science/art based grease making has recently resulted in several new approaches to making grease, which include the use of preformed dehydrated soaps, preformed oily soaps, micro-particle dispersion of lithium hydroxide, the use of contactor/reactors to improve process efficiency and grease consistency, and the incorporation of microwaves for heating. Vegetable oils due to their polarity respond well to microwave energy for heating. The process time can be reduced considerably when heating for reaction is accomplished through the use of microwaves.

The research team is coordinating with tank manufacturers on the design of new reactors that can handle the mixing of thick products without the mixing arms acting as an antenna for microwaves (preventing microwave leaks). Also, future designs may include jacketed reactors to allow using the jackets as the holding space for heat transfer fluids, to be heated by biobased oils inside the reactor. Whatever the final design, the use of microwaves for manufacturing greases continues to show great promise for higher energy efficiency, greater ease of operation, and particularly improved product properties for the resulting biobased greases. This remains an on-going project with further research to continue.

Economic Analysis

- Biobased greases have been conventionally more expensive that mineral (petroleum) oil based greases. The UNI-NABL Researchers have been developers of a new process that uses microwaves for heating instead of conventional heat transfer oil heating. In a paper presented to the European Lubricating Grease Institute (ELGI), the primary investigator reported on the economic benefits of using microwave heating for processing biobased greases. This process is estimated to reduce the processing cost to less than 50% of the conventional methods of using hot heat transfer oils. At this point the conclusion is that due to advances in technology, the cost of biobased greases are in parity with mineral oil based greases. In a related research project performed for the US Department of Transportation, the UNI-NABL researchers purchased commercial rail curve greases from three biobased grease suppliers and three mineral oil based grease suppliers and the price of these greases were about the same. In one case the price of the biobased grease was lower than that of the mineral based rail curve greases. The biobased greases claimed to meet the USDA Biopreferred labeling requirements as Biobased. Biobased labeling requires that the products show the percentage of biobased content as well as a report of life cycle analysis as performed by the National Institute of Standards (NIST). To date the higher price of biobased greases has been considered a barrier to commercial success. But, the research at UNI-NABL Center has shown that with price parity, biobased greases offer additional environmental adders that would make them economically attractive for commercial success.

Acknowledgements: The authors thank the staff of the Environmental Lubricants Manufacturing and AMTek Microwave Systems http://www.4amtek.com/ for their continued support of this project.

Task C: FUNDAMENTAL VEGETABLE OIL RESEARCH ACTIVITIES

> NABL will conduct research targeting new methods of improving the inherent characteristics and fundamental properties of vegetable oils.

In order to comprehensively study the behavior of vegetable oils for use in lubricants applications, a number of vegetable oils were tested. These oils presented different fatty acid profiles and some could be considered exotic, but were available in different countries as major oils. The oils were tested for their physiochemical properties and their fatty acid make ups. Additionally, these oils were evaluated for their tribological properties. The results provide a comprehensive overview of properties of these vegetable oils. The data can be used to study various correlations between the fatty acid makes of the oils and their performance in biobased lubricants and greases.

The following is a report on the properties of those vegetable oils.

Table: Fatty Acid Profile Report on Tested Vegetable Oils

Oil Name	C8:0 Octanoic (Caprylic)	C10:0 Decanoic (Capric)	C12:0 Dodecanoic (Lauric)	C14:0 Tetradecanoic (Myristic)	C16:0 Hexadecanoic (Palmitic)	C16:1 Hexadecenoic (Palmitoleic)	C17:0 Heptadecanoic (Margaric)	C17:1 Heptadecenoic Margaroleic	C18:0 Octadecanoic (Stearic)	C18:1 Octadecenoic (Oleic)	C18:2 Octadecadienoic (Linoleic)	C18:3 Octadecatrienoic (Linolenic)	C18:4 Octadecatetraenoic	C20:0 Eicosanoic (Arachidic)	C20:1 Eicosenoic (Gadoleic)
Apricot Kernel Oil					5.30	0.27			2.96	63.86	25.75	0.14		0.23	0.22
Avocado Oil - refined			0.17	0.51	18.18	0.23			3.23	64.48	11.36	0.14		0.33	0.27
Caprylic Acid 99% pure	99.00														
Castor Oil					1.42				1.62	4.52	5.87	0.61			0.52
Coconut Oil - refined	7.32	5.83	46.57	18.54	9.28				3.07	6.71	1.84				
Corn Oil					10.94	0.18			1.86	28.24	56.02	0.93		0.41	0.34
Cottonseed Carrier Oil				0.86	19.11	0.58			2.65	23.00	51.50	0.21		0.31	0.17
Flaxseed (Linseed) Oil					5.04	0.11			3.67	25.94	17.75	28.10	0.26	0.28	0.39
Grapeseed Carrier Oil					8.02	0.14			3.65	22.35	63.83	0.49	0.10	0.30	0.36
Hempseed Oil					5.69	0.13			2.53	10.27	54.45	23.00	1.54	0.87	0.41
High Oleic Canola Oil					3.59	0.26		0.23	1.77	74.73	14.38	1.73		0.63	1.38
HOBO (82% Oleic Bean Oil)					6.07	0.16		0.12	3.47	82.28	3.53	2.61		0.36	0.44
Jojoba Oil Clear refined					0.99	0.28				9.23		0.20			72.01
Jojoba Golden Carrier Oil					1.03	0.33				9.80	0.11	0.21			71.81
Macadamia Nut Carrier Oil				0.78	7.91	17.18			3.69	60.39	2.61	0.17		2.79	2.67
Methyl Ester of Hydroxy-Fatty Acid Soy					8.99	0.14	0.11		4.71	34.97	43.94	4.35	0.11	0.45	0.44
Olive Oil - refined A					12.46	0.94	0.11	0.17	2.89	70.47	10.95	0.59		0.47	0.39
Palm Oil — refined			0.21	1.05	45.46	0.18	0.11		4.65	37.74	9.62	0.16		0.38	0.15
Poppyseed Carrier Oil					8.78	0.15			2.40	15.90	70.06	0.45		0.13	1.17
Ricebran Carrier Oil				0.20	13.08	0.19			2.31	46.44	34.26	0.67		0.62	0.49
Sesame Carrier Oil					8.92	0.16			4.33	38.50	46.16	0.34		0.52	0.28
81% high oleic Sunflower Oil					3.50				3.41	82.58	7.83	0.45		0.31	0.33
Sunflower Oil - organic					4.38	0.14			2.70	79.02	11.13	0.32		0.26	0.40
Walnut Carrier Oil					7.08	0.18			2.68	16.48	59.61	8.24	0.15	0.14	0.36

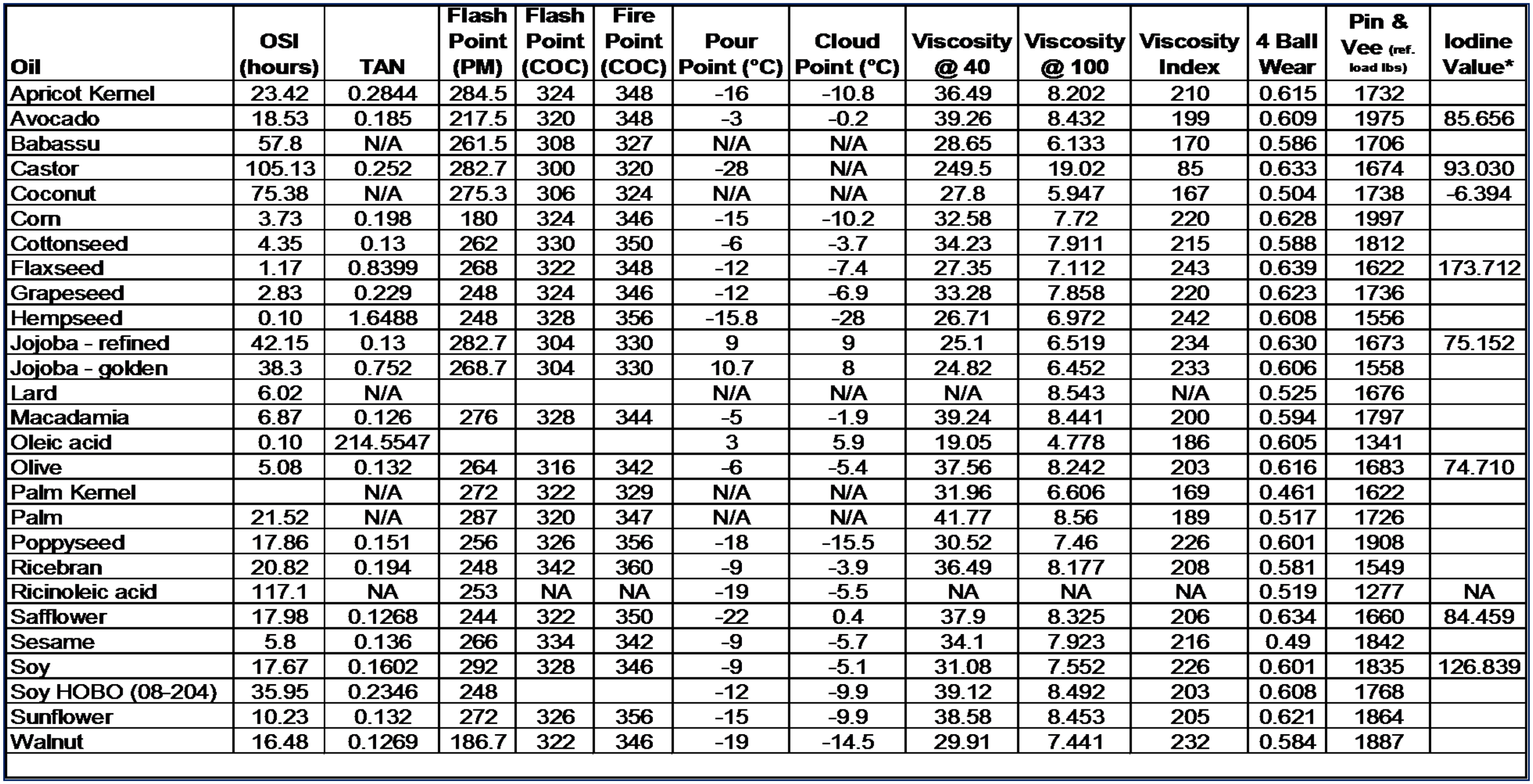
10% to avoid chart clutter

Relative Area Percent AOCS Ce 2-66

files with the following previously tested results:

xample oils high in certain fatty acids exhibiting higher dielectric strength.
ned chart the molecular weight against microwave absorption values.

Table: Selected Properties of Selected Vegetable Oils



Oil	OSI (hours)	TAN	Flash Point (PM)	Flash Point (COC)	Fire Point (COC)	Pour Point (°C)	Cloud Point (°C)	Viscosity @ 40	Viscosity @ 100	Viscosity Index	4 Ball Wear	Pin & Vee (ref. load lbs)	Iodine Value*
Apricot Kernel	23.42	0.2844	284.5	324	348	-16	-10.8	36.49	8.202	210	0.615	1732	
Avocado	18.53	0.185	217.5	320	348	-3	-0.2	39.26	8.432	199	0.609	1975	85.656
Babassu	57.8	N/A	261.5	308	327	N/A	N/A	28.65	6.133	170	0.586	1706	
Castor	105.13	0.252	282.7	300	320	-28	N/A	249.5	19.02	85	0.633	1674	93.030
Coconut	75.38	N/A	275.3	306	324	N/A	N/A	27.8	5.947	167	0.504	1738	-6.394
Corn	3.73	0.198	180	324	346	-15	-10.2	32.58	7.72	220	0.628	1997	
Cottonseed	4.35	0.13	262	330	350	-6	-3.7	34.23	7.911	215	0.588	1812	
Flaxseed	1.17	0.8399	268	322	348	-12	-7.4	27.35	7.112	243	0.639	1622	173.712
Grapeseed	2.83	0.229	248	324	346	-12	-6.9	33.28	7.858	220	0.623	1736	
Hempseed	0.10	1.6488	248	328	356	-15.8	-28	26.71	6.972	242	0.608	1556	
Jojoba - refined	42.15	0.13	282.7	304	330	9	9	25.1	6.519	234	0.630	1673	75.152
Jojoba - golden	38.3	0.752	268.7	304	330	10.7	8	24.82	6.452	233	0.606	1558	
Lard	6.02	N/A				N/A	N/A	N/A	8.543	N/A	0.525	1676	
Macadamia	6.87	0.126	276	328	344	-5	-1.9	39.24	8.441	200	0.594	1797	
Oleic acid	0.10	214.5547				3	5.9	19.05	4.778	186	0.605	1341	
Olive	5.08	0.132	264	316	342	-6	-5.4	37.56	8.242	203	0.616	1683	74.710
Palm Kernel		N/A	272	322	329	N/A	N/A	31.96	6.606	169	0.461	1622	
Palm	21.52	N/A	287	320	347	N/A	N/A	41.77	8.56	189	0.517	1726	
Poppyseed	17.86	0.151	256	326	356	-18	-15.5	30.52	7.46	226	0.601	1908	
Ricebran	20.82	0.194	248	342	360	-9	-3.9	36.49	8.177	208	0.581	1549	
Ricinoleic acid	117.1	NA	253	NA	NA	-19	-5.5	NA	NA	NA	0.519	1277	NA
Safflower	17.98	0.1268	244	322	350	-22	0.4	37.9	8.325	206	0.634	1660	84.459
Sesame	5.8	0.136	266	334	342	-9	-5.7	34.1	7.923	216	0.49	1842	
Soy	17.67	0.1602	292	328	346	-9	-5.1	31.08	7.552	226	0.601	1835	126.839
Soy HOBO (08-204)	35.95	0.2346	248			-12	-9.9	39.12	8.492	203	0.608	1768	
Sunflower	10.23	0.132	272	326	356	-15	-9.9	38.58	8.453	205	0.621	1864	
Walnut	16.48	0.1269	186.7	322	346	-19	-14.5	29.91	7.441	232	0.584	1887	

Table: Physiochemical Properties of Selected Vegetable Oils

Oil	OSI (hours)	TAN	Flash Point (PM)	Flash Point (COC)	Fire Point (COC)	Pour Point (°C)	Cloud Point (°C)
Apricot Kernel	23.42	0.2844	284.5	324	348	-16	-10.8
Avocado	18.53	0.185	211	320	348	-3	-0.2
Babassu	57.8	N/A	258	308	327	N/A	N/A
Castor	105.13	0.252	279	300	320	-28	N/A
Coconut	75.38	N/A	275	306	324	N/A	N/A
Corn	3.73	0.198	180	324	346	-15	-10.2
Cottonseed	4.35	0.13	262	330	350	-6	-3.7
Flaxseed	1.17	0.8399	268	322	348	-12	-7.4
Grapeseed	2.83	0.229	248	324	346	-12	-6.9
Hempseed	0.10	1.6488	248	328	356	-15.8	-28
Jojoba - refined	42.15	0.13	260	304	330	9	9
Jojoba - golden	38.3	0.752	265	304	330	10.7	8
Lard	6.02	N/A				N/A	N/A
Macadamia	6.87	0.126	276	328	344	-5	-1.9
Oleic acid	0.10	214.555				3	5.9
Olive	5.08	0.132	263	316	342	-6	-5.4
Palm Kernel		N/A	272	322	329	N/A	N/A
Palm	21.52	N/A	187	320	347	N/A	N/A
Poppyseed	17.86	0.151	256	326	356	-18	-15.5
Ricebran	20.82	0.194	248	342	360	-9	-3.9
Ricinoleic acid	117.1	NA	253	NA	NA	-19	-5.5
Safflower	17.98	0.1268	238	322	350	-22	0.4
Sesame	5.8	0.136	266	334	342	-9	-5.7
Soy	17.67	0.1602	292	328	346	-9	-5.1
Soy HOBO (08-204)	35.95	0.2346	248			-12	-9.9
Sunflower	10.23	0.132	272	326	356	-15	-9.9
Walnut	16.48	0.1269	186	322	346	-19	-14.5

Environmental Impacts of Biobased Greases

Tests of biodegradability are often performed on the base oils used to make greases. This is similar to tests of flash and fire points whereby grease cannot be tested for flash point and as a result the flash point of the base oil of the grease is required for specifications. Testing grease in the current biodegradability instruments is difficult because grease does not easily dissolve in water. The research team had worked with biobased and mineral based greases for a project sponsored by the US DOT. The UNI-NABL Center has tried a method with limited success. To test the biodegradability of grease products a quantity of grease is dissolved in a sample which along with some reference samples are placed in a controlled environment and then inoculated with standard specified bacteria. The test runs for 28 days and as bacteria consume food or the biodegradable materials they take up oxygen and release carbon dioxide. After consuming all the nutrients in the sample, the bacteria begin to die out thus oxygen consumption flattens out and then drops. The OECD 301 series tests corresponding to the ASTM tests and require monitoring either oxygen consumption or carbon dioxide evolution. The test method used was the OECD 301 test which is a 28 day test. The biobased grease showed to be biodegradable according to the test method; while the mineral based grease did not meet the percent oxygen consumption as required by the test and thus not considered biodegradable.

This test was run a little different than other tests. Normally this test is run using oil samples; but since this test required the use of grease samples it had to be modified to accommodate the change. The only change made to the test was how the sample was introduced to the water. Normally 100 mg of the sample is put directly in to the water. For grease, the team spread the required 100 mg weight of grease onto a half a piece of filter paper and placed it into the water. This helped in two different ways, the first being it helped keep the grease from accumulating on the side of the bottle and not being fully exposed to the bacteria. The second was getting an accurate weight of the sample, by using the filter paper it was possible to spread the sample on the paper and then weight it. All the other aspect of the test remained the same and it was run for 28 day at 22 degrees Celsius.

The results showed that samples containing sodium benzoate which is the reference food for the bacteria showed a rapid growth in the oxygen uptake. The test requires that within the first 10 days of the test, the oxygen uptake by the test sample to reach 60 percent of the reference sample. The test is then continued for 28 days and the oxygen uptake continues at these levels if the product can be consumed to sustain the test bacteria. The results indicated that sample of mineral greases did not result in oxygen uptakes approaching 60% of the reference samples; and in effect cannot be considered biodegradable.

Task D: CONTINUED PREPARATORY ACTIVITIES

Qualify the NABL Center to serve the bio-lubricants industry as a respected testing facility.

This UNI-NABL Center played a leading role in working groups that were focused on the performance of biobased lubricants and greases. As chairman of a Joint Working Group for both NLGI and ELGI, the primary Investigator helped to initiate a worldwide effort to determine the best test methods for biobased lubricants and greases. The UNI-NABL Center performed tests and coordinated the round robin tests by providing sample oils and greases for testing to various labs in the US and in Europe. The following slides present a sampling of the work performed. Vegetable oils and biobased derived esters as well mineral oils were sent to different laboratories to be tested in known oxidation tests according to US and European standards. The goal was to determine which test methods would best differentiate between a stable biobased oil and unstable biobased oil.

In a follow up series of tests, greases were made using the same oils that were tested earlier and were sent for a round robin testing using the same test methods for oxidation stability. One of the test methods identified for testing of biobased greases is the Rotary Pressure Vessel Oxidation Test (RPVOT) which is a ubiquitous test machine in the petroleum industry. But, since the test method is for liquid lubricants, the UNI-NABL Center developed a modified method which was used in the round robin tests. The modification included mixing the grease with silicone oils to make the grease behave like a liquid and thus test it in RPVOT. In earlier tests, the UNI-NABL Center staff had shown that silicone oils would show minimal degradation in stability when tested the RPVOT.

- The UNI-NABL Center staff, especially the primary investigator, Lou Honary, was actively involved with both the National Lubricating Grease Institute (NLGI) and the European Lubricating Grease Institute (ELGI) serving on the board of directors and as chairman of a Biobased Grease Performance Working Group for both organizations. The activities of the working group included educating the members about the differences between biobased and petroleum based greases; and performing round robin testing to determine which tests would be suitable for biobased greases. Oxidation stability of biobased greases is one of the most important performance aspects that are often questioned. As a result an attempt was made to identify which current oxidation test methods that are used for petroleum products can also be used effectively for biobased greases. The working group identified a number of tests methods that are used for oxidation stability. Then sample base oils were sent to different labs in the US and in Europe for tests of oxidation. Then base greases were prepared using the same base oils and those too were sent for round robin testing.
- The Working Group round robin tests indicated that the test methods most suitable for biobased greases would be the RPVOT which is commonly used by the petroleum industry; and the RSSOT. This work is continuing and the ELGI working group members will expand the testing to include tests for cold temperature performance of biobased greases.
- The following presents excerpts from a presentation at a meeting of the working group lead by the primary investigator.

Focus on Oxidation Stability

1. Determine which one(s) of the current conventional test methods work for biobased oils
2. Determine which one(s) of the current conventional test methods work for biobased greases
3. Choose test(s)
4. Proposed new or modified test(s)

OSI (Oxidative Stability Index),

AOCS Cd 12b-92

	OSI Results – Lou Honary, UNI-NABL	OSI Results – G. Dodos, ELDON'S SA
L1 Soybean Oil Neat	**0.23 hours**	**0.02**
L2 Soybean Oil w. Anti-Oxidant	**0.25**	**11.6**
L3 PAO 6	**62.00**	**62.7**
L4 Biobased Synthetic Oil	**131.55**	**>120**
L5 Synthetic Oil	**52.42**	**36.7**
L6 Saturated Biobased Ester	**N/A**	**>120**
L7 Canola Oil Neat	**8.62**	**8.7**
L8 Canola Oil w. Anti-Oxidant	**102.62**	**78.7**
L9 Biobased Synthetic Ester - Saturated	**103.57**	**86.5**
L10 Biobased Synthetic Ester - Unsaturated	**16.12**	**15.4**

PDSC (Pressurized Differential Scanning Calorimeter), ASTM D6186

	PSDC Results – E. Jisheng, GKN
L1 Soybean Oil Neat	**0.29 min**
L2 Soybean Oil w. Anti-Oxidant	**0.36**
L3 PAO 6	**3.7**
L4 Biobased Synthetic Oil	**10.98**
L5 Synthetic Oil	**3.41**
L6 Saturated Biobased Ester	**6.54**
L7 Canola Oil Neat	**0.51**
L8 Canola Oil w. Anti-Oxidant	**1.98**
L9 Biobased Synthetic Ester - Saturated	**6.48**
L10 Biobased Synthetic Ester -Unsaturated	**0.44**

RPVOT (Rotating Pressure Vessel),

ASTM D2272-11

	Stephanie Janeda, Rhein Chemie Fheinau GmbH	Lou Honary, UNI-NABL
L1 Soybean Oil Neat	**11 min**	**24 min**
L2 Soybean Oil w. Anti-Oxidant	**11**	**26**
L3 PAO 6	**34**	**39**
L4 Biobased Synthetic Oil	**110**	**124**
L5 Synthetic Oil	**25**	**36**
L6 Saturated Biobased Ester	**55**	**73**
L7 Canola Oil Neat	**11**	**24**
L8 Canola Oil w. Anti-Oxidant	**14**	**24**
L9 Biobased Synthetic Ester - Saturated	**26**	**40**
L10 Biobased Synthetic Ester - Unsaturated	**16**	**30**

RSSOT (Rapid Small Oxidation Test),

ASTM D7545-09

	RSSOT, George Dodos, ELDON'S
L1 Soybean Oil Neat	**7**
L2 Soybean Oil w. Anti-Oxidant	**25**
L3 PAO 6	**139**
L4 Biobased Synthetic Oil	**322**
L5 Synthetic Oil	**111**
L6 Saturated Biobased Ester	**326**
L7 Canola Oil Neat	**32**
L8 Canola Oil w. Anti-Oxidant	**75**
L9 Biobased Synthetic Ester - Saturated	**126**
L10 Biobased Synthetic Ester - Unsaturated	**35**

The following charts present the testing performed on various mixtures of silicone oil and grease in the RPVOT method. Each grease was mixed with silicone oil and tested in RPVOT.

Introduction:

Greases were made using preformed soap and the following oils:

ID	General Composition
L1	Soybean oil neat
L2	PAO 6
L3	Biobased synthetic
L4	Synthetic oil
L5	Saturated biobased ester
L6	HO canola oil neat
L7	Biobased derived synthetic ester saturated
L8	Biobased derived synthetic ester unsaturated

The greases were then tested for Penetrometer, Dropping Point, and RPVOT.

Formulations:

	Oil (g)	Preformed Soap (g)
Soybean oil neat	300	60
PAO 6	300	30
Biobased synthetic	300	30
Synthetic oil	300	30
Saturated biobased ester	300	100
HO canola oil neat	300	30
Biobased derived synthetic ester saturated	300	30
Biobased derived synthetic ester unsaturated	300	30

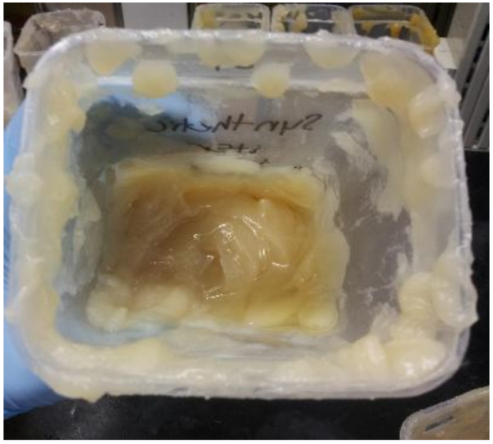
Each grease was first made with 10% preformed soap. If the grease was too thin, the amount of preformed soap was increased until the desired consistency was reached.

Soybean Grease

PAO 6 Grease

Biobased Synthetic Grease

Synthetic Ester Grease



Saturated Biobased Ester Grease

High Oleic Canola Grease

Biobased Derived Synthetic Ester Grease Saturated

Biobased Derived Synthetic Ester Grease Unsaturated

Procedure:

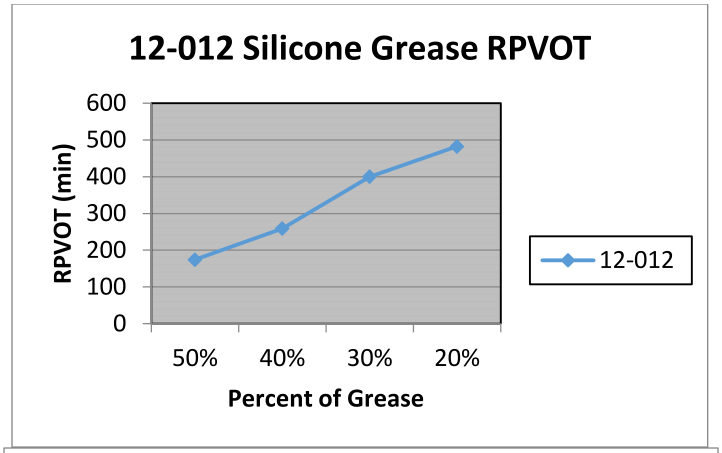
1) Using a microwave, heat each of the oils to 150˚C. The PAO 6 oil will not heat properly with a microwave, a hot plate must be used instead.
2) Add 10% preformed soap and gently mix. Increase the amount of preformed soap on an as need basis.
3) Microwave the oil again to increase the temperature to 200˚C.
4) Once the soap and oil is fully mixed, let it cool to room temperature.
5) Mill or high shear mix to homogenize.

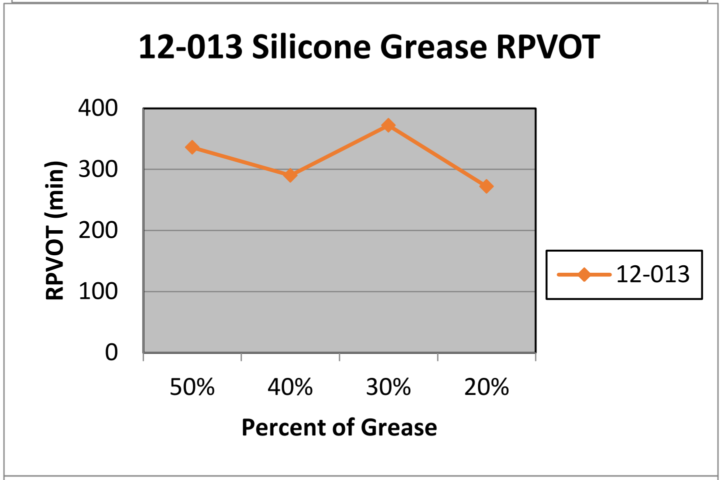
Testing:

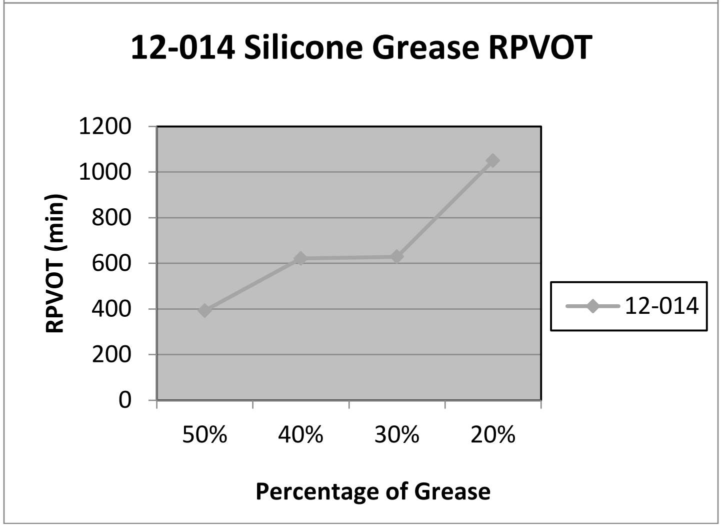
Unworked Penetrometer (small cup), Dropping Point, and RPVOT were preformed on the 8 greases.

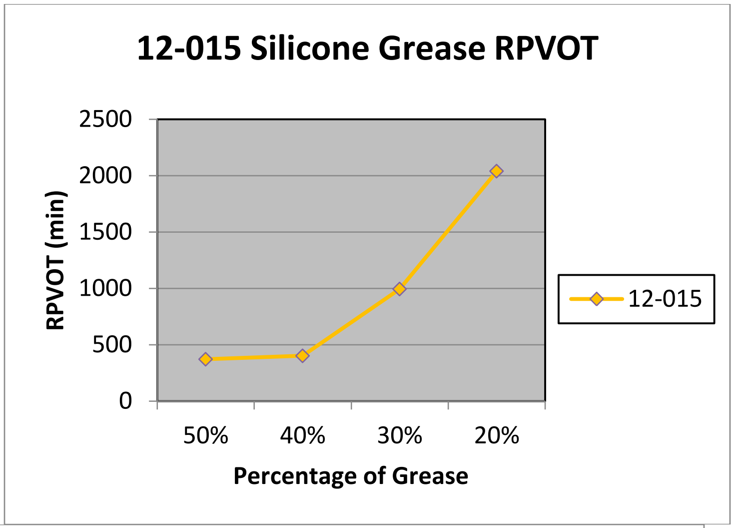
	Soybean Grease (20% soap)	**PAO6 Grease (10% soap)**	**Biobased Synthetic Grease (10% soap)**	**Synthetic Ester Grease (10% soap)**	**Saturated Biobased Ester Grease (30% soap)**	**HO Canola Grease (10% soap)**	**Biobased Derived Synthetic Ester Grease Saturated (10% soap)**	**Biobased Derived Synthetic Ester Grease Unsaturated (10% soap)**
RPVOT (min)	26	89	1796	706	261	31	150	51
Pentrometer	185	259	194	234	188	251	202	259
Dropping Point	200	211.33	227.34	195.34	195	190.34	196.67	192

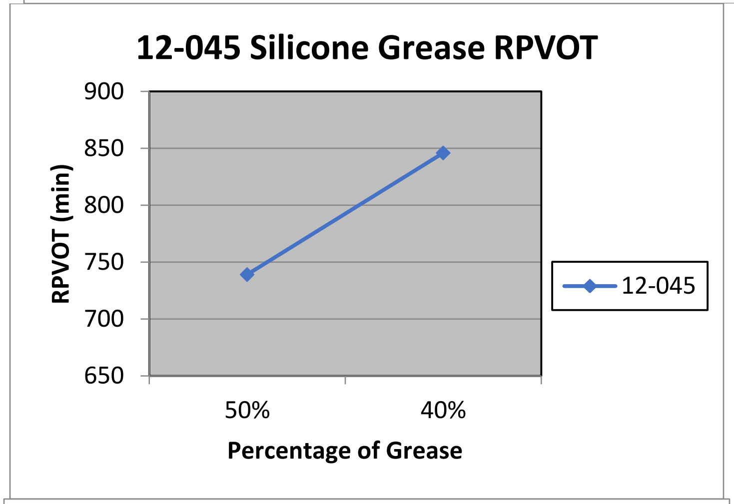
Note: When testing the RPVOT, a sample was prepped with 25% grease and 75% silicone oil.

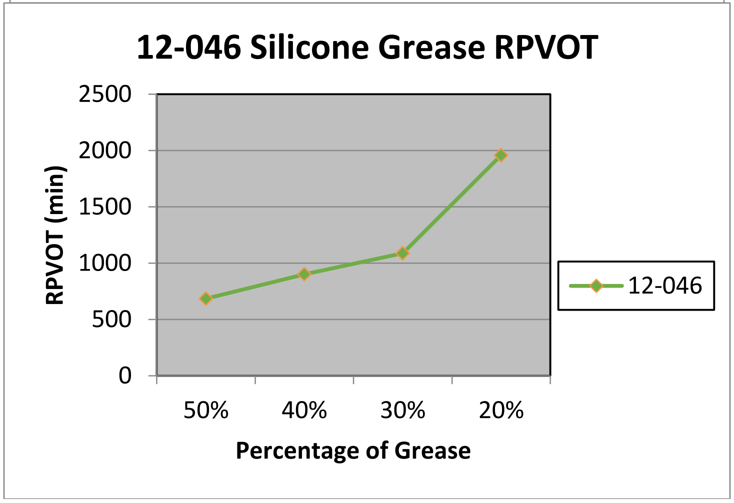

12-012 Silicone Grease RPVOT
RPVOT (min)
600
500
400
300
200
100
0
50%
40%
30%
20%
Percent of Grease
12-012

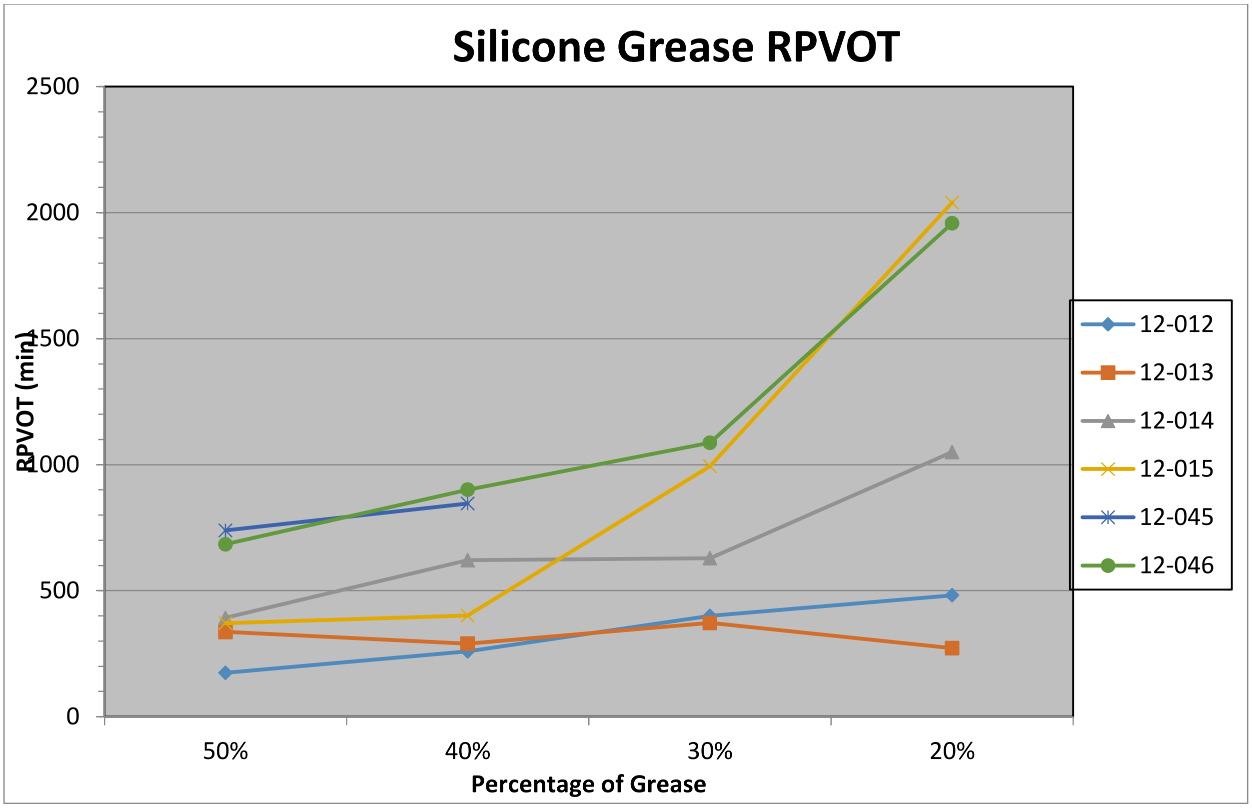

12-013 Silicone Grease RPVOT
RPVOT (min)
400
300
200
100
0
50%
40%
30%
20%
Percent of Grease
12-013


12-014 Silicone Grease RPVOT
RPVOT (min)
1200
1000
800
600
400
200
0
50%
40%
30%
20%
Percentage of Grease
12-014


12-015 Silicone Grease RPVOT
RPVOT (min)
2500
2000
1500
1000
500
0
50%
40%
30%
20%
Percentage of Grease
12-015


12-045 Silicone Grease RPVOT
RPVOT (min)
900
850
800
750
700
650
50%
40%
Percentage of Grease
12-045


12-046 Silicone Grease RPVOT
RPVOT (min)
2500
2000
1500
1000
500
0
50%
40%
30%
20%
Percentage of Grease
12-046


Silicone Grease RPVOT
RPVOT (min)
2500
2000
1500
1000
500
0
50%
40%
30%
20%
Percentage of Grease
12-012
12-013
12-014
12-015
12-045
12-046

Task E: CONTINUED RESEARCH ACTIVITIES

NABL will pursue various research activities to be continued from previous scopes of work, which were not completed previously due to either budgetary constraints, or limited staff time and technical capabilities, in the following sub-tasks:

NABL will continue laboratory work conducting fundamental vegetable oil based research, studying vegetable oil and vegetable oil derivatives, identifying desirable properties of various seed oils for use in lubricants, and relevant chemical structures.

New applications for the oils mentioned in E.2 will continue to be researched.

Significant amount of work was performed in this area. Specifically: a) Greases were made using Free Fatty Acids to determine if the resulting grease has more predictable performance at extreme cold or hot temperatures. Since vegetable oils are made from a number of fatty acids with different melting point, grease made from vegetable oils would present unpredictable dropping point or freezing point. Grease made from a specific fatty acid would show less variability and thus more predictable performance at extreme temperatures. The following report presents greases made from nine fatty acids and tested for basic properties.

Introduction

Nine fatty acid greases were made and tested among a full spectrum of grease tests. They yielded the following results:

Formulations

AN Number	Fatty Acid	FA (g)	PG (g)	LiOH (g)	Castor (g)
AN 11117	Palmitic Acid	604.2	110	102.3	716
AN 11119	Myristic Acid	806.5	119.4	155	704.5
AN 11137	Palmitic Acid	733.1	122.9	109.3	1061.6
AN 11138	Stearic Acid	784.2	122.9	69.3	1083.7
AN 11139	Oleic Acid	794.0	NA	100.0	994.3
AN 11140	Oleic Acid	728.5	NA	160.5	886.9
AN 11141	Lauric Acid	785.8	147.8	165.8	741.5
AN 11142	Lauric Acid	808	134.7	153.1	965.6
AN 11143	Stearic Acid	727.2	108.5	138.4	1038.3

The formulation of each of these greases was based on previous calculations reported in May 2011.

Procedure

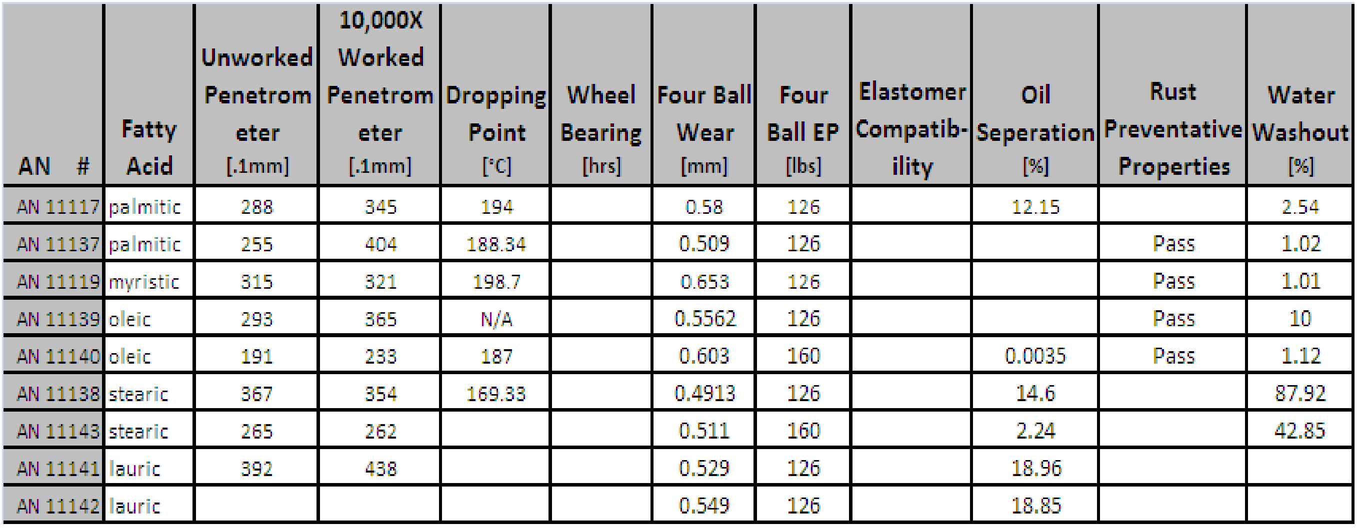
After zeroing the beaker on the scale, the solid fatty acid was poured in and weighed. That value was recorded and the scale was zeroed again. Propylene glycol was added and its weight was recorded. The two substances were mixed using a glass stir rod in order to coat the fatty acid with propylene glycol as much as possible. (Since propylene glycol more readily heats in the microwave than fatty acid, it is important that the two be mixed thoroughly before heating.) The beaker was placed in the microwave and heated in increments of 30-60 seconds, stirring between each increment. Usually about 5 increments of heating were necessary for the mixture reached 130°C. At that point, the mixture was entirely liquid and clear yellow.

At 130°C, the Lubrizol LiOH was added in increments of 10-15% of the total amount to ensure a slow and controlled reaction between LiOH and the fatty acid. As the LiOH was slowly added, the temperature of the soap was kept between 130-140°C using bursts of microwaves and frequent stirring. After all LiOH had been added, the soap was kept at that temperature for an additional 5 minutes to allow the entire saponification reaction to occur.

The soap was then brought to 200°C using ~1 minute bursts of microwave energy. Once temperature was reached, Castor cooling oil was added slowly while stirring to create a homogenous mixture between the soap and oil, or grease. While still warm, grease was homogenized using the blue handheld homogenizer. Grease allowed cooling with periodic stirring until it reached room temperature. It was then stored in clean paint cans until ready to be tested.

Testing

Unworked penetrometer, 10,000X worked penetrometer, dropping point, four ball wear, four ball EP, elastomer compatibility, oil separation, rust preventative properties, water washout, and wheel bearing tests were performed on the nine greases. The results of these tests are present in the following table:



AN #	Fatty Acid	Unworked Penetrometer [.1mm]	10,000X Worked Penetrometer [.1mm]	Dropping Point [°C]	Wheel Bearing [hrs]	Four Ball Wear [mm]	Four Ball EP [lbs]	Elastomer Compatibility	Oil Seperation [%]	Rust Preventative Properties	Water Washout [%]
AN 11117	palmitic	288	345	194		0.58	126		12.15		2.54
AN 11137	palmitic	255	404	188.34		0.509	126			Pass	1.02
AN 11119	myristic	315	321	198.7		0.653	126			Pass	1.01
AN 11139	oleic	293	365	N/A		0.5562	126			Pass	10
AN 11140	oleic	191	233	187		0.603	160		0.0035	Pass	1.12
AN 11138	stearic	367	354	169.33		0.4913	126		14.6		87.92
AN 11143	stearic	265	262			0.511	160		2.24		42.85
AN 11141	lauric	392	438			0.529	126		18.96		
AN 11142	lauric					0.549	126		18.85		

Observations

All of the greases have been observed at room temperature, refrigerator temperature, and freezer temperature. Results are as followed:

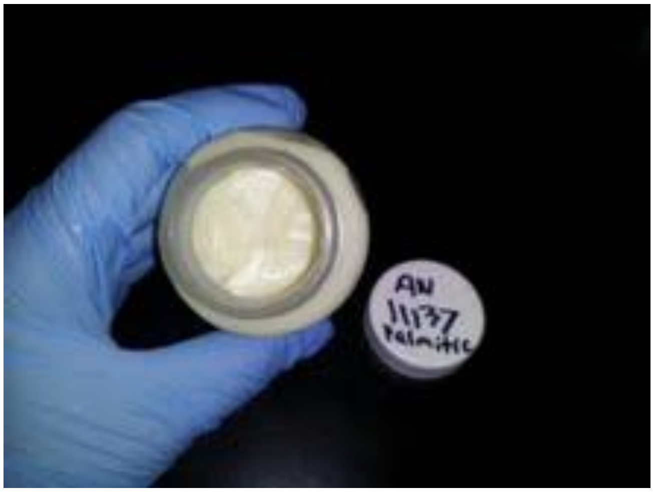
AN 11117

Room Temperature: Had a smooth temperature and thick consistency and showed good tackiness. Refrigerator Temperature (10.2 ˚C): Slightly hardened, very thick consistency almost like a balm. Freezer Temperature (0˚F): Froze completely solid.

AN 11119

Room Temperature: Mostly smooth texture but when it is left to sit, it becomes slightly chunky and the oil begins to separate.

Refrigerator Temperature (9˚C): Smooth at fridge temperature, thickened and hardened slightly. Freezer Temperature (0˚F): Froze completely solid.



AN 11137

Room Temperature: Thick and smooth, some small clumps scattered throughout. Refrigerator Temperature: (8.6 ˚C): Hard but not completely solid, still smooth

Freezer Temperature: (0˚C): Completely solid.

AN 11138

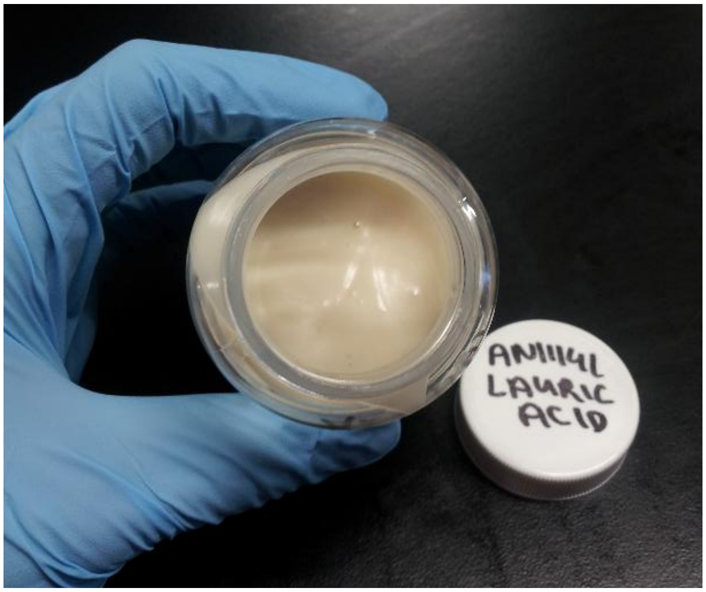
Room Temperature: Smooth texture, has a light and airy feel to it. Refrigerator Temperature: (8˚C): Smooth, creamy texture, light and airy feel. Freezer Temperature: (0˚C): Completely solid.

AN 11139

Room Temperature: Very smooth and thick consistency.
Refrigerator Temperature: Did not solidify at all. Kept same smooth and thick texture as it had at room temperature.
Freezer Temperature: (0˚C): Completely solid.

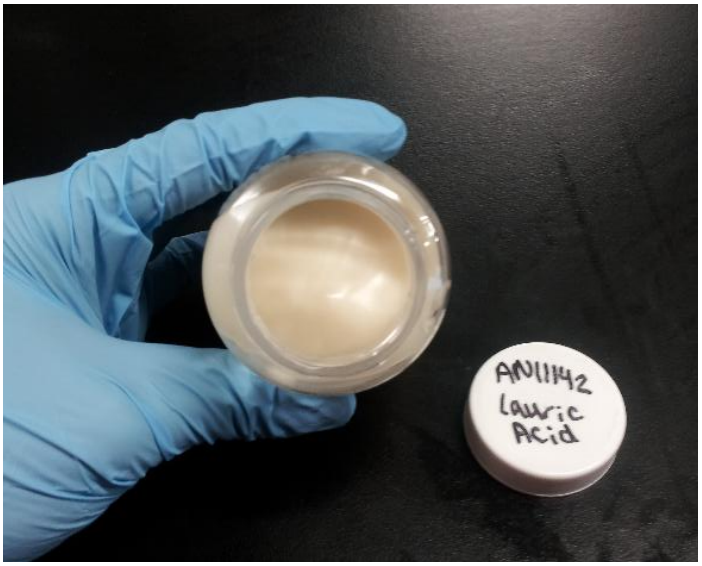
AN11140

Room Temperature: Very thick and extremely chunky.
Refrigerator Temperature: Did not solidify, hardened very slightly, still thick and chunky consistency.
Freezer Temperature: (0˚C): Completely solid, still chunky.



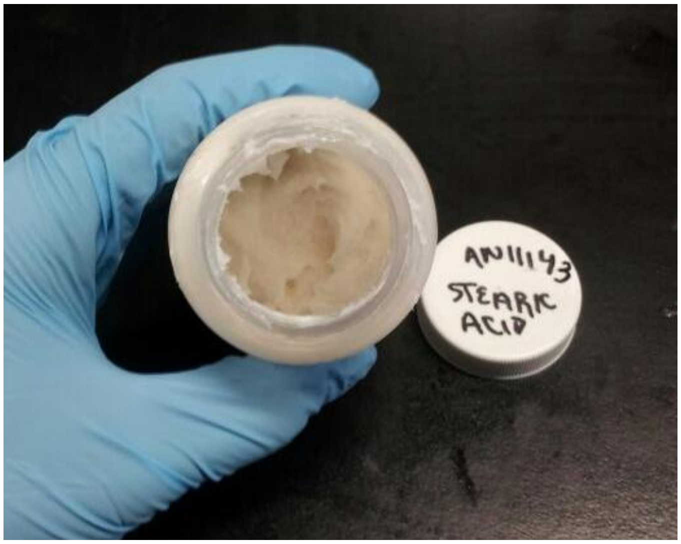
AN11141

Room Temperature: Thin, smooth texture.
Refrigerator Temperature: Did not solidify. Very smooth, but thickened slightly.
Freezer Temperature: Completely solid.



AN11142

Room Temperature: Thin, smooth texture.
Refrigerator Temperature: Did not solidify, grainy. Thickened a lot.
Freezer Temperature: Completely solid.



AN11143

Room Temperature: Very thick, slightly chunky.
Refrigerator Temperature: Hardened slightly, but not solid.
Freezer Temperature: Completely solid.

Additionally, an attempt was made to modify the base vegetable oils by esterifying them to determine if esterification helps with improving the performance of the biobased greases at extreme temperatures. The following report presents the results of experiments of making methyl esters from vegetable oil and then preparing biobased grease using those esters in the base oils of the grease.

Grease made using selected methyl esters

Introduction/ Purpose:

This report will attempt to answer the following questions: What are the characteristics of a HOBO methyl ester grease formulation? How good of a base or cooling material is HOBO methyl ester? Does the resulting have grease better properties or body/texture?

Procedure:

Our procedures were the same for each trial unless otherwise noted. A methyl ester was prepared using high oleic bean oil (HOBO) with a target viscosity of 4cst, resulting in a HOBO ME. The following blends were then prepared using the described procedure:

1) Thickened ME Blend #1:
    - Blend the HOBO ME with stearic acid to thicken to a target viscosity of about 250 cSt 40°C. It will need to be heated to above the melting point of stearic acid to mix.
2) Thickened ME Blend #2:
    - Blend the HOBO ME with Electrion 1500 to thicken to a target viscosity of about 250 cSt 40°C.

3) For each of the above blends, start by adding only 5% of the thickener to the ME. If the viscosity is below 250 cSt, increase thickener as necessary. To estimate the 250 cSt target viscosity, visually/manually compare the blends
to castor oil (which has about 250 cSt at 40 C viscosity).
4) Once the proportions of each thickened blend are known, make a 1200mL sample of each.

Next, using LiOH, 500ml of soap was made out of each ME blend (unthickened, stearic thickened, and Electrion thickened). Each soap was cooled with the same base oil used for the soap to make grease.

Each grease was tested on the following tests:

1) Un-worked cone penetrometer (B2)
2) Worked (60X) cone penetrometer (B3)
3) Bearing Oxidation (AKA Life Performance Wheel Bearing Grease B16)
4) Dropping point (B8)
5) Water washout at 100°F and 175°F (B12)
6) Cold temperature flowability (AKA Grease Mobility B13)

HIGH OLEIC (soy) BEAN OIL (hobo) METHYL ESTER Formulation

Before greases could be made, a large sample of HOBO methyl ester was necessary. First a small sample was made to ensure the formulation produced a quality methyl ester. The small sample was washed and analyzed and proved to be acceptable. Therefore, a larger batch was made using the following formulation and procedure:

HOBO Oil (10-088)	1000 mL
Methanol (09-228)	250.00g
KOH (09-260)	10.02g

1) The HOBO oil was heated to 60°C.
2) The KOH and methanol were combined and stirred together at room temperature until completely dissolved.
3) The methanol/KOH mixture was poured into the 60°C HOBO oil. The substance was stirred continuously while maintaining a 60-65°C temperature for 1 hour.
4) The mixture was removed from heat/motion and allowed to sit overnight to allow the glycerin to separate from the methyl ester.
5) The methyl ester was washed by filling half of a separatory funnel with the methyl ester (leaving the glycerin behind in the reaction beaker) and half with DI water. Using a combination of manual agitation and bubbling (seen below), the water and methyl ester was allowed to interact so the water could pull the impurities out. Occasionally, the agitation would subside and the water and methyl ester would separate again, with water on the bottom layer. The water was drained into solvent waste, new DI water was added on top, and agitation continued. At the beginning of the washing process, this drained water has a pH of about 8-9. The washing process was complete when the pH of the draining water was slightly acidic (~6) instead of basic.

Methyl ester in the washing step

This process was repeated many times to accumulate ME for trial batch greases as well as a large batch.

<u>Thickened ME Blend #1</u>: HOBO ME + Stearic Acid

Blend 1 Trial 1

HOBO ME (AN10840)	99.9g
Stearic Acid (SA) (09-325)	109.9g

As directed, 99.9g of HOBO methyl ester was measured and 5.0g (5%) of stearic acid was added at room temperature. The beaker was then placed in the microwave and the stearic acid melted at about 60°C. The mixture had not significantly thickened, so more stearic acid was added. Even after 109.9g of stearic acid had been added to the original 99.9g of HOBO ME, the blend did not appear to have the same viscosity as castor oil. Nevertheless, this blend was used to make grease to observe how a ME/SA blend would behave as base and cooling material.

ME/SA blend	100.8g
LiOH Emulsion (#10-049)	6.5g
ME/SA blend	117g

ME/SA blend was heated to 130°C. Lubrizol LiOH emulsion (33% LiOH) was added incrementally while maintaining temperature until 6.5g had been added. The same ME/SA blend was then used as a cooling material. Grease was liquid at this temperature so it was placed in the refrigerator to cool. At room temperature, grease was poor because it became a solid.

Blend 1 Trial 2

HOBO ME	101.0g
Stearic Acid	25.3g

The stearic acid was melted into the methyl ester to create the blend for the base/cooling oil.

ME/SA blend	50.4g
LiOH Emulsion	3.7g
ME/SA blend	50.4g

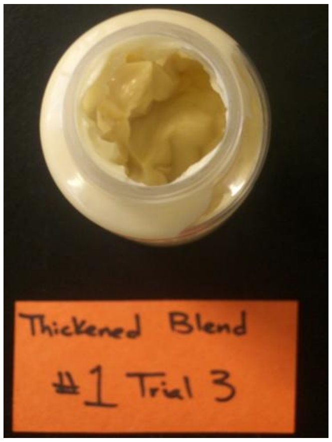
This grease was homogenized by hand blender. This grease had a large amount of oil separation. The body was very poor. It was whippy and slightly fibrous. Two more greases were planned. They were to contain more LiOH than this trial as seen in trials 3 and 4.

A blend of HOBO ME and stearic acid was made for these two trials. However the first attempt for trial 4 caught fire after a failed attempt to heat to 200°C.

HOBO ME	201.4g
Stearic Acid	51.66g

Blend 1 Trial 3

ME/SA blend	50.5g
LiOH Emulsion	4.8g
ME/SA blend	50.1g

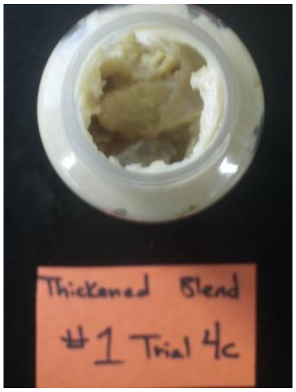


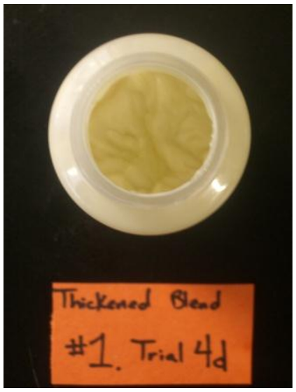
This trial seemed to have the smallest amount of oil separation. It had better body than the previous trial; however, it was still lightweight and had a whippy consistency. It was slightly fibrous and very smooth.

There were little to no chunks at all in this grease.

Blend 1 Trial 4

This trial is a set of failed trials. After increasing the LiOH emulsion amount by another gram, it was difficult to get the sample to a high temperature and then keep this blend at a high temperature. It required very careful monitoring to achieve the correct temperatures. During the first two trials, 4a and 4b, the soap caught fire before they were quite at 200°C due to slow heating up to 200°C and then a quick spike in their heating, causing very high temperatures for the soap, catching on fire.



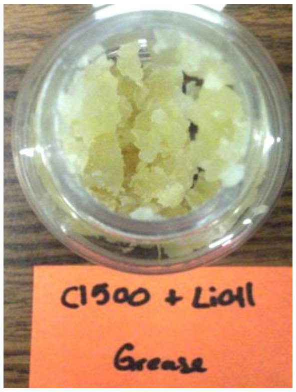


<u>Thickened Blend #2</u> : HOBO ME + Electrion 1500

<u>Pre-Trial experiments:</u>

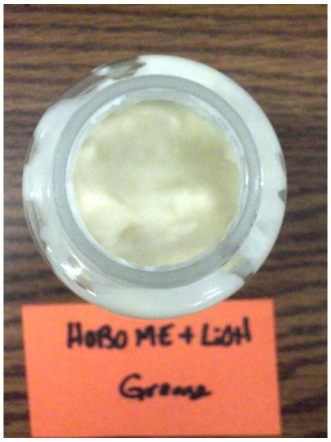
In order to determine what effects each component of the base/cooling oil has on the grease. The first grease was made from purely C1500 and another made from only HOBO methyl esters.

C1500 + LiOH



The grease made with the C1500 and LiOH (seen at the left) had very chunky and dried properties. It clumped up quite a bit. One variable that this may explain is the amount of C1500 in each blend for the greases. Perhaps changing the amount of the C1500 or ratio with the HOBO ME could prove beneficial.

HOBO ME + LiOH



This grease seemed to be rather smooth and liquid. Once cooled, the grease hardens, however it is not clumpy. It has a bar soap feel and is creamy. The hardness is problem when adding the cooling oil to the soap to make the grease because the grease will not be easily cooled. The cooling oil seems to turn solid very quickly after adding it to the 200°C soap.

lend 2 Trial 1

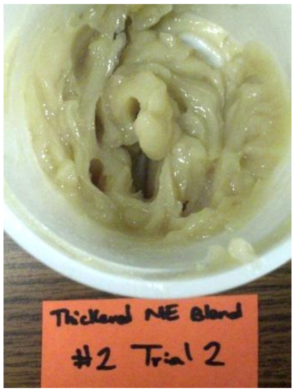
HOBO ME (AN10840)	100.0g
C1500 (08-013)	65.9g

Again, after nearly 66g of C1500 the blend still did not appear to have the viscosity of castor oil; however, experimentation proceeded to making grease using this blend.

ME/C1500 blend	50.2g
LiOH Emulsion (#10-049)	3.1g
ME/C1500 blend	50.0g

ME/C1500 blend was heated to 130°C. After the first small portion of LiOH emulsion (1.3g), the mixture gelled to a semisolid and was very difficult to stir. Despite this, more LiOH emulsion was added and was not able to mix in with the soap. The process was stopped.

Blend 2 Trial 2



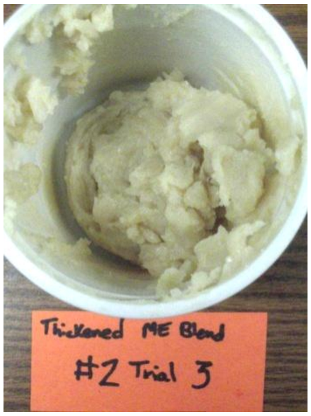
HOBO ME	101.1g
C1500	10.8g

This methyl ester and C1500 blend was very thin and watery.

ME/C1500 blend	50.0g
LiOH Emulsion	3.0g
ME/C1500 blend	50.0g

This blend of methyl ester and C1500 was heated to 130°C and the LiOH emulsion was added in three parts. The soap that formed was very foamy and gelled. After adding only three grams, no more lithium hydroxide was added because the soap was too hard to stir. This grease was homogenized after adding cooling oil and after refrigeration. After homogenizing, the grease looked appeared smooth; however, the body of the grease was very poor.

Blend 2 Trial 3



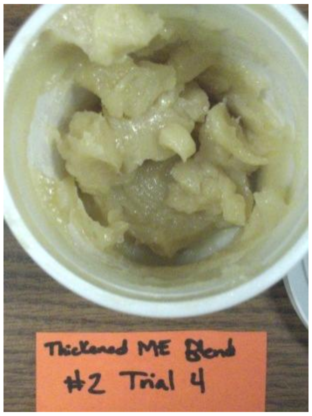
HOBO ME	100.0g
C1500	10.0g

Again this blend was not close to the viscosity of castor oil, specified in the procedure. In this trial, the six grams of LiOH emulsion was to be added instead of stopping after the hardening of the soap. The soap was to be kept at a liquid even if that meant heating to higher than 130°C. The soap after adding LiOH emulsion was usually liquid around 160 degrees.

ME/C1500 blend	50.8g
LiOH Emulsion	5.6g
ME/C1500 blend	51.8g

Once all of the lithium suspension had been added, the soap was heated to 200°C and the cooling oil was added. This grease was homogenized before and after refrigeration. It was much thicker than the previous trial. Like Trial 2, this grease had very poor body. It seemed to only “mush” between fingers.

Blend 2 Trial 4



HOBO ME	99.3g
C1500	19.7g

Although the amount of the C1500 was doubled in this blend, the body was still thin. By adding more of the C1500, it was hoped that a better body would be present in the grease. 3 grams of the LiOH Emulsion was used to recreate the smoothness of Trial 2.

ME/C1500 blend	49.9g
LiOH Emulsion	3.1g
ME/C1500 blend	50.0g

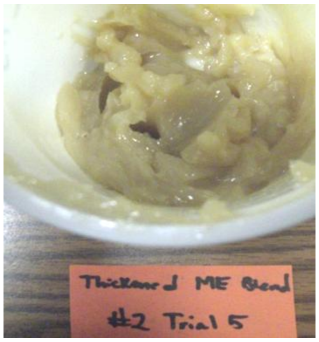
This grease seemed to be worse than trial 2; it could probably be homogenized further to look more like trial 2. Grease was a little thicker and had a little more substance to it.

Blend 2 Trial 5

HOBO ME	104.8g
C1500	31.8g

This grease was an experiment with adding more C1500 to create a thicker base/cooling oil.

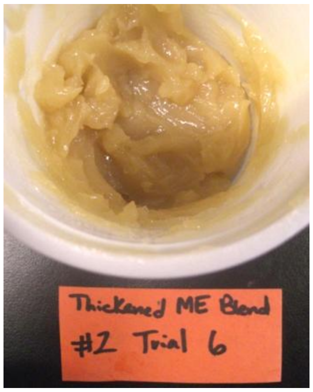
ME/C1500 blend	50.5g
LiOH Emulsion	2.9g
ME/C1500 blend	51.6g



This grease was by far the best grease made at this point. It was a light and fluffy feel but good lubricity. It has a good smooth look and has better body than the other trials. The grease was still slightly mushy.

Blend 2 Trial 6

HOBO ME	104.6g
C1500	41.2g



In this trial, more C1500 was added to the blend in order to increase the viscosity to match the castor oil. The amount of C1500 was increased by another 10 gram increment.

ME/C1500 blend	51.0g
LiOH Emulsion	2.8g
ME/C1500 blend	51.0g

This grease seemed to be worse than the previous trial. The previous trial sat for a couple of weeks which may have affected it during comparison. The body seemed to be better in trial 5. This grease was very fluffy and light weight. It almost had a whipped topping consistency.

Blend 2 Trial 7

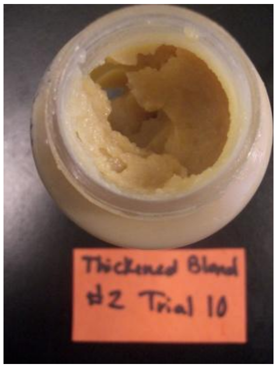
HOBO ME	81.2g
C1500	45.8g

This trial was designed to increase the ration of C1500 to ME in hopes of getting a thicker grease.

ME/C1500 blend	50.2g
LiOH Emulsion	2.82 g
ME/C1500 blend	51.5g

This grease was better than the previous trial. The grease was slightly thicker and tackier. Without very much improvement in the body of the grease in the past trials, the next three trials were made from 100g ME/ 10g C1500 blends with varying amounts of LiOH suspension. These greases were compared with Trial 2 and 3 above.

Blend 2 Trials 8, 9, and 10



A large mixture of the ME/C1500 was made for these three trials to share.

ME (AN 10840/ AN 10849)	305.2 g
C1500	36.6g

Difference in LiOH Emulsion Amounts

Trial 8	**Trial 9**	**Trial 10**
ME/C1500 blend 49.8g	ME/C1500 blend 50.3g	ME/C1500 blend 51.0g
LiOH Emulsion 3.4g	LiOH Emulsion 6.7g	LiOH Emulsion 4.7g
ME/C1500 blend 52.0g	ME/C1500 blend 50.0g	ME/C1500 blend 49.6g

Comparison of Trials 2, 3, 8, 9, and 10

Trial #	LiOH Emulsion Amount	Comments
2	3.0g	This grease is rather smooth and very whippy/ lightweight. It has poor body, but no chunks. There is oil separation.

8	3.4g	This grease has slightly stronger body than 2 and still has oil separation. It is still airy and smooth, but lacks tackiness.
10	4.7g	This grease is slightly grainy and not airy like 2 and 8. It is slightly fibrous but not tacky and there is no oil separation.
3	5.6g	This grease has the best body of these five trials. It is grainy, thick, gel-like mixture that is slightly sticky.
9	6.7g	This grease is very grainy and has a paste consistency. The tackiness/stickiness is poor.

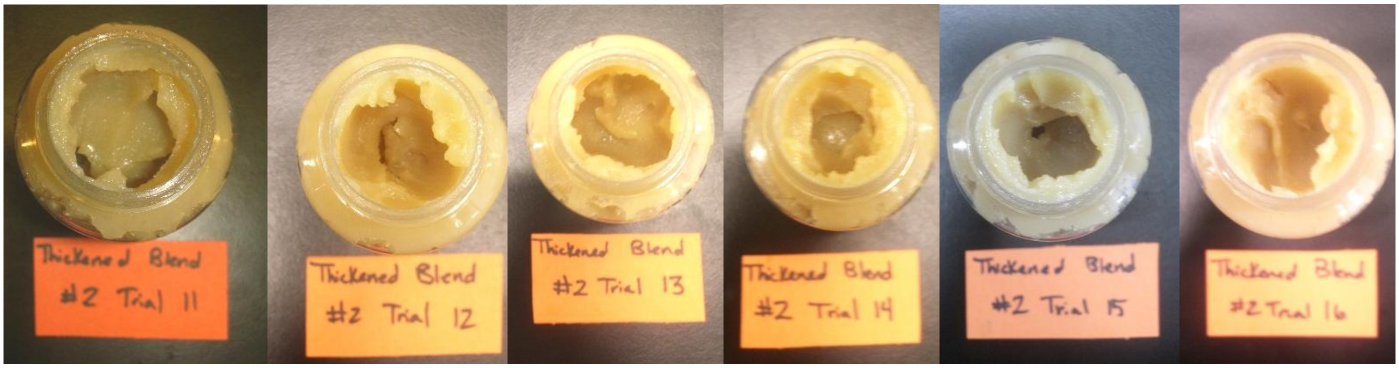
The chart above is a comparison of the five greases made with the 100g/10g mixture of ME/C1500. The qualities of these greases were not diversely different and were still poor quality. A decision was made to make the next five greases from a ME/C1500 mixture of 100g/25g based on the slightly better properties of the greases in trials 4 and 5 made with a slightly higher C1500 content. These five greases, like trials 8, 9, and 10, were to be made from the same mixture for base and cool oil but with varying LiOH suspension amounts added.

Blend 2 Trials 11, 12, 13, 14, 15, and 16

A large mixture of ME/C1500 was made for these trials to share.

HOBO ME	500.1g
C1500	128.2g

However, an extra trial was added (Trial 14) because Trial 13 failed to meet the preparation standards. (Too much cooling oil was added.) Therefore, more of the mixture was needed. 200 g of HOBO ME was added as well as 50g of C1500 to the remaining mixture before Trial 15 and 16 were made.



Trial #	11	12	13	14	15	16
ME/C1500 Blend	49.1g	49.7g	51.2g	49.8g	50.7g	48.5g
LiOH Emulsion	2.5g	3.5g	4.6g	4.4g	5.5g	6.5g
ME/C1500 Blend	53.3g	49.5g	~67g	50.4g	50.9g	49.1g

The chart below can be used to compare the greases made in this set. The goal of making this set was to see if the better greases are made with the higher LiOH emulsion amounts (4-6g) and if the greases made with this 100/25 blend are better than the greases made with the 100/10 blend.

Comparison of Blend 2 Trials 11-16

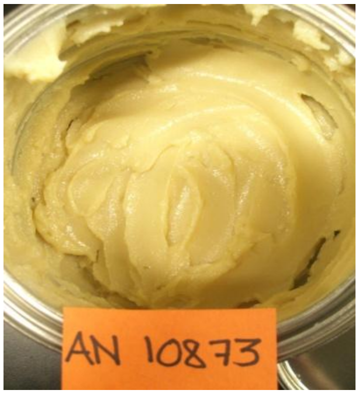
Trial #	LiOH Emulsion Amount	Comments
11	2.5g	This trial grease has a little bit of oil separation towards the bottom of the container and had a very weak body. It is more light and fluffy like previous greases made.
12	3.5g	This grease, like Trial 11, is fluffy, airy, and smooth, but it is slightly fibrous. It still has the whipped, lightweight feel and poor body.
13	4.6g	This grease had excess cooling oil added to it while being made. There were clumps but the grease was still smooth and not gritty.
14	4.4g	This grease was the “re-make” of Trial 13. It is also chunky and has a stronger body than 12 and 11. The grease was slightly fibrous.
15	5.5g	The grease was slightly chunky and not quite as smooth as the previous greases. It had stronger body and tackiness than the others before also.
16	6.5g	This grease became very grainy. It is very thick, pasty, and waxy. It is more “solid” than the previous two trials.

Overall, the greases made in this set were better than the greases made in trials 2, 3, and 8-10. The 100/25 ME/C1500 greases have better body and smoothness comparatively. The greases with the 4.5 and 5.5 gram LiOH suspension amounts seem to have a better consistency. Changing the amounts of LiOH suspension created greases with the consistency like that of whipped topping to wax. The 4.5 and 5.5 gram mixtures seemed to have the best properties in all trials. A large batch of Trial 15 was made given that it had been the best grease of the many trials and the ME/C1500 mixture used in this case worked better than the other mixtures.

Thickened Blend #2 AN 10873

This large batch was modeled from Trial 15. Below are the HOBO/C1500 blend amounts by part.

HOBO ME:	1600g	1616.1g
C1500:	400g	397.6g



Below are the grease component amounts used:

ME/C1500:	999.8g
LiOH suspension:	109.8g
ME/C1500:	1000.3g

Testing Data Results* and Comparison Charts:

Testing Results Raw Data Chart

Grease # and Blend	Penetration (0.1 mm @ 25°C)		Dropping Point by Aluminum Block (°C)	Water Washout (mass % loss)		Grease Mobility (g/min)			Wheel Bearing Grease (hr)
	Un-worked	60X Worked		38°C or 100°F	79°C or 175°F	At Temp. 1	At Temp. 2	At Temp. 3	
Blend #1 AN 10918	298	321	165	10.3	96	237 @ -15C	272 @ -13.5C	277 @ -13C	36
Blend #2 AN 10873	247	267	170	99.6	100	288 @ -11.5C	178 @ -10.4C	181 @ -8.3C	24

*See Appendix B for specific details about the repeatability of these tests.

Conclusions

The characteristics of the greases made with HOBO ME were tested and reported in the sections above. When comparing the greases made with HOBO ME to a standard HOBO LiOH grease, it appears the ME product has a much higher Grease Mobility rate (237 g/min at -15C vs. 21 g/min at -14C) however, overall, the methyl ester greases do not appear to have any advantageous product characteristics versus a standard HOBO LiOH grease as reported for WR #4.

Some options for further research and testing are to incorporate various additives to the greases to improve production characteristics and functionality.

Appendix A: Standard method of microwave grease making using oils

1. Measure oil in tared beaker on scale.
2. Microwave oil in 30-60s intervals, stirring occasionally, until the mixture is ~130°C.
3. Add LiOH (10-049 Lubrizol LiOH/oil emulsion) in increments of about 0.5-1.0 g increments by slowly pouring quantities into the beaker of hot oil.
    a. With each LiOH addition, stir the contents with a stir rod.
    b. Between each LiOH addition, microwave the soap to maintain a temperature of ~130°C .
4. Allow the saponification reaction to complete by maintaining a temperature of ~130°C for about 5 minutes after the final LiOH addition.
5. Increase the soap temperature to 200°C by microwaving in 1.0-1.5 minute intervals, stirring occasionally.
6. Maintain a temperature of 200-210°C for 20 minutes, stirring occasionally.
7. Slowly add cooling oil by pouring into the beaker of soap on scale, stirring continuously. Optional: blend with immersion blender using blade attachment.
8. Cool the grease in refrigerator and after cooling is complete, blend with immersion blender using blade attachment or the grease mill.

Appendix B:

Testing List for Work Request Greases

Test Name	Test Method	Repeatability Limit or Formula
Penetration (Unworked)	ASTM D217	85 to 475 8 units
Penetration (60X Worked)	ASTM D217	130 to 475 7 units
Dropping Point by Aluminum Block [Δ]	ASTM D2265	up to 116°C 6°C
		116°C up to 221°C 8°C
		221C° up to 277°C 6°C
		277C° up to 316°C 7°C
Water Washout 38°C / 100ºF	ASTM D1264	0.8 (x + 2) [†]
Water Washout 79°C / 175ºF	ASTM D1264	0.6 (x + 4.6) [†]
Cold Temperature Flowability AKA Mobility of Greases	U.S. Steel	none given in method
AKA Life Performance of Wheel Bearing Grease (up to 120 hr)	ASTM D3527	(0.8) x [†]

[Δ] as per the method, "The dropping point of some grease, particularly those containing simple soaps, are known to decrease upon aging, the change being much greater that the deviation permitted from results obtained by different laboratories. Therefore, comparative tests…should be made within a period of six days."

[†] where x = the average of the two test results

Note: Repeatability = the difference between successive results obtained by the same operator with same operating apparatus under constant operating conditions with identical test material…would exceed following values only in 1case in 20

Reproducibility = the difference between two single and independent results obtained by different operators working in different laboratories on identical test material

ASTM American Society for Testing and Materials International, 100 Barr Harbor Drive, West Conshohocken, PA19428-2959 USA
www.astm.org

Finally, an attempt was made to determine the effects of adding small amount of vegetable oil or fatty acids to both regular and low sulfur diesel fuel. The main property to measure was the improved anti-wear property. The following report presents the Effect of Adding Small Amount of Vegetable Oils into Diesel Fuel on its Anti-wear Property as measured by Four Ball test - ASTM 2266, “Wear Preventive Characteristics of Lubricating Grease”

The University of Northern Iowa’s
National Ag-Based Lubricants Center

Effect of Adding Small Amount of Vegetable Oils into Diesel Fuel on its Anti-wear Property as measured by Four Ball test - ASTM 2266, “Wear Preventive Characteristics of Lubricating Grease”

Supplemental Report for US Department of Energy

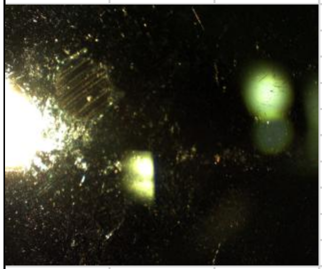
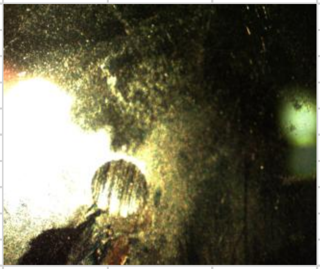
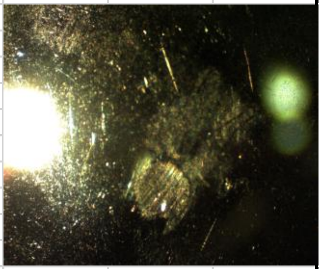
A. Effect of introducing a small amount of different vegetable oils into a B-100 commercial methyl ester on its anti-wear property as measured by four ball test - ASTM 2266, Wear Preventive Characteristics of Lubricating Grease:

Using four ball wear test to determine the effect of adding a small amount vegetable oil to diesel fuel, both methyl esters of vegetable oils and vegetable oils were tested. Scar diameters presented in millimeter are used as indictor of anti-friction property of the test fluid. The lower the average scar diameter the better the anti-wear property of the fluid.

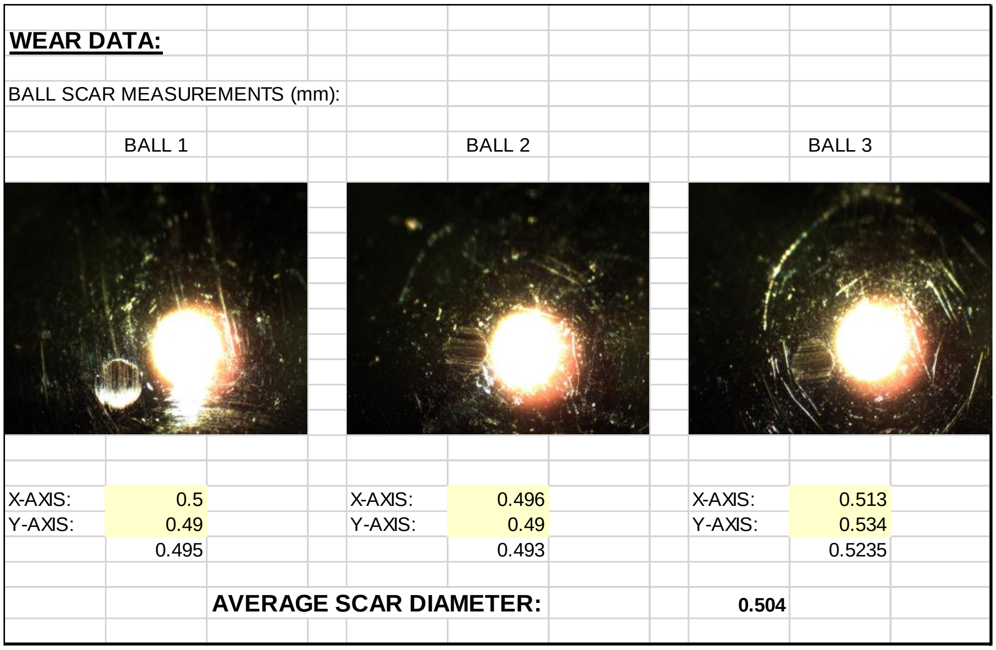
1. Safflower Oil in B-100:

WEAR DATA:

BALL SCAR MEASUREMENTS (mm):

	BALL 1		BALL 2		BALL 3
					
X-AXIS:	0.563	X-AXIS:	0.578	X-AXIS:	0.598
Y-AXIS:	0.637	Y-AXIS:	0.569	Y-AXIS:	0.614
	0.6		0.5735		0.606
AVERAGE SCAR DIAMETER:				**0.593**	

2. Coconut Oil in B-100



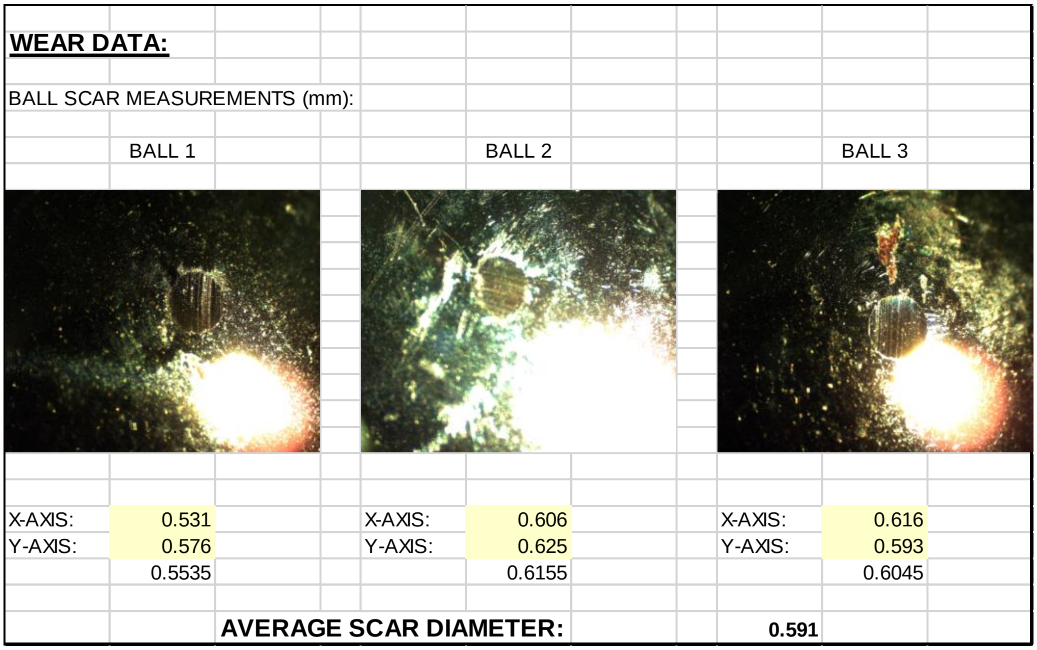
WEAR DATA:

BALL SCAR MEASUREMENTS (mm):

	BALL 1		BALL 2		BALL 3
X-AXIS:	0.5	X-AXIS:	0.496	X-AXIS:	0.513
Y-AXIS:	0.49	Y-AXIS:	0.49	Y-AXIS:	0.534
	0.495		0.493		0.5235
		AVERAGE SCAR DIAMETER:		**0.504**	

3. Coconut Oil in B-100

3. Commodity Soybean Oil in B-100



WEAR DATA:

BALL SCAR MEASUREMENTS (mm):

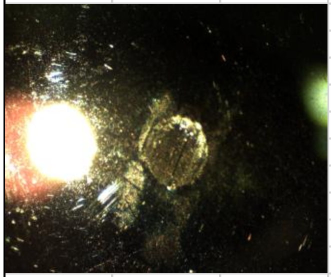
	BALL 1		BALL 2		BALL 3
X-AXIS:	0.531	X-AXIS:	0.606	X-AXIS:	0.616
Y-AXIS:	0.576	Y-AXIS:	0.625	Y-AXIS:	0.593
	0.5535		0.6155		0.6045
		AVERAGE SCAR DIAMETER:		**0.591**	

4. Olive Oil in B-100

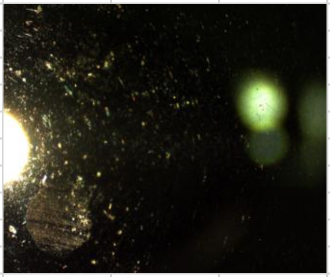
WEAR DATA:

BALL SCAR MEASUREMENTS (mm):

BALL 1



BALL 2

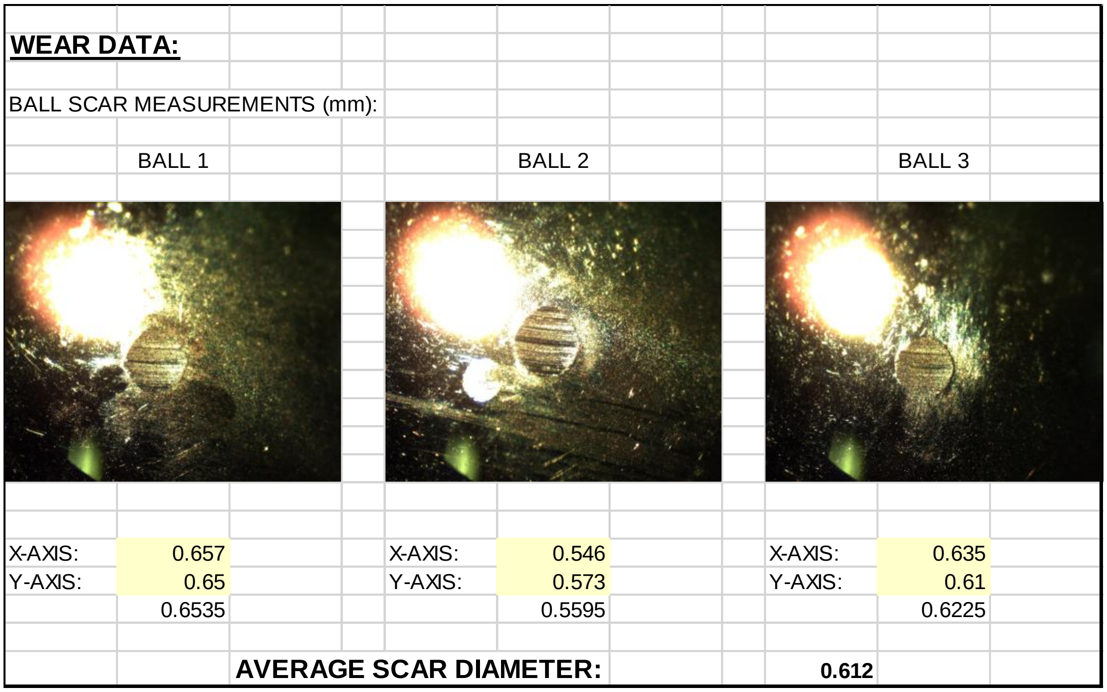


BALL 3

X-AXIS:	0.69	X-AXIS:	0.637	X-AXIS:	0.67
Y-AXIS:	0.66	Y-AXIS:	0.613	Y-AXIS:	0.65
	0.675		0.625		0.66

AVERAGE SCAR DIAMETER: **0.653**

4. Babasu Oil in B-100



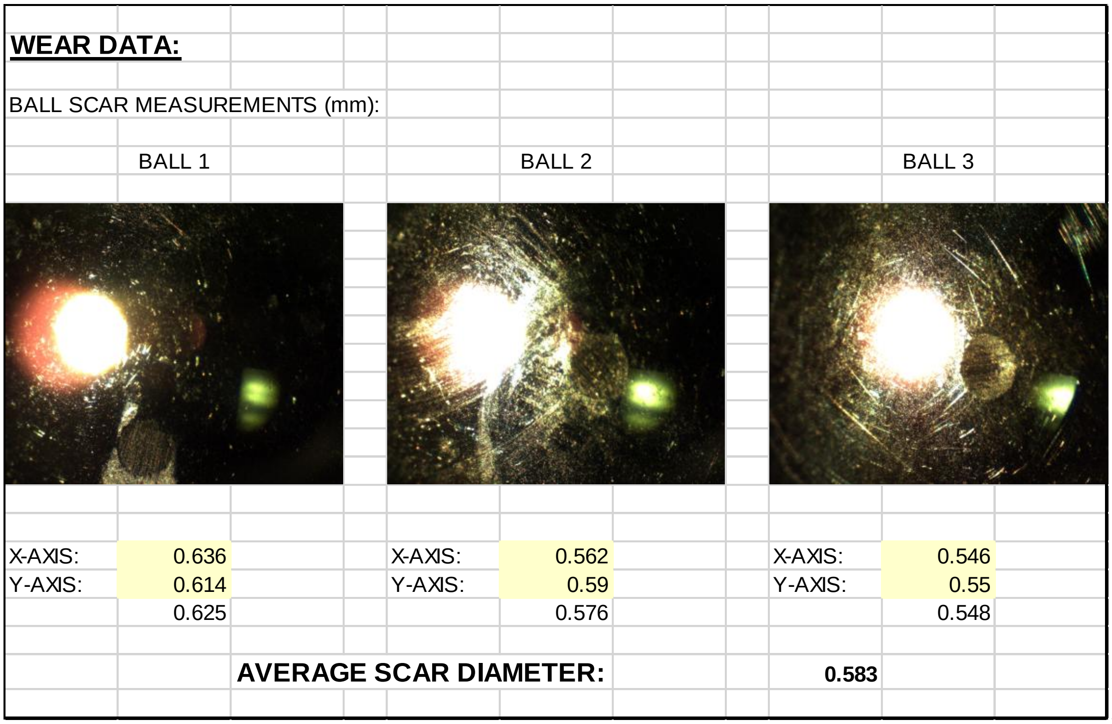
WEAR DATA:

BALL SCAR MEASUREMENTS (mm):

	BALL 1		BALL 2		BALL 3
X-AXIS:	0.657	X-AXIS:	0.546	X-AXIS:	0.635
Y-AXIS:	0.65	Y-AXIS:	0.573	Y-AXIS:	0.61
	0.6535		0.5595		0.6225

AVERAGE SCAR DIAMETER: 0.612

5. Refined Bleached Deodorized Soybean Oil in B-100



WEAR DATA:

BALL SCAR MEASUREMENTS (mm):

	BALL 1		BALL 2		BALL 3
X-AXIS:	0.636	X-AXIS:	0.562	X-AXIS:	0.546
Y-AXIS:	0.614	Y-AXIS:	0.59	Y-AXIS:	0.55
	0.625		0.576		0.548

AVERAGE SCAR DIAMETER: 0.583

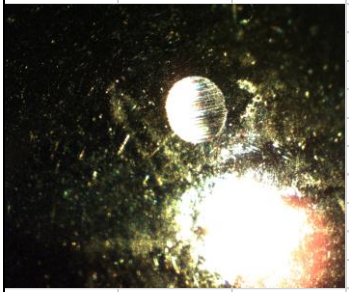
5.

6. Poppy Seed Oil in B-100

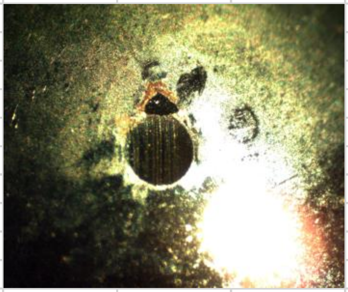
WEAR DATA:

BALL SCAR MEASUREMENTS (mm):

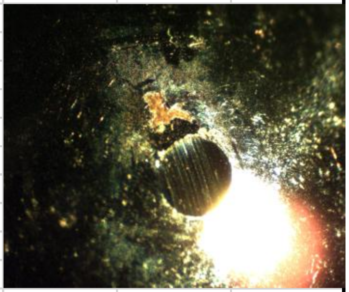
BALL 1



BALL 2



BALL 3



	BALL 1		BALL 2		BALL 3
X-AXIS:	0.656	X-AXIS:	0.69	X-AXIS:	0.611
Y-AXIS:	0.713	Y-AXIS:	0.674	Y-AXIS:	0.638
	0.6845		0.682		0.6245

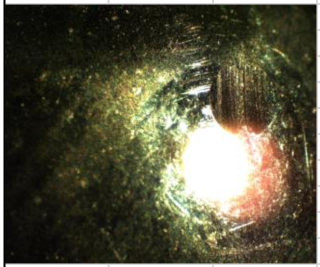
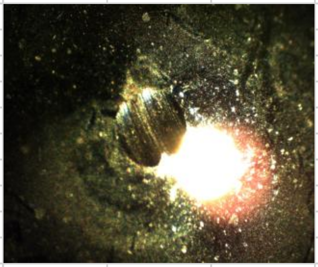
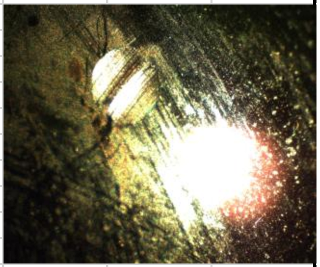
AVERAGE SCAR DIAMETER: **0.664**

B. Effect of introducing a small amount of different vegetable oils into regular diesel fuel on its anti-wear property as measured by four ball test - ASTM 2266, Wear Preventive Characteristics of Lubricating Grease:

7. Regular Diesel Fuel and .1% Safflower Oil

WEAR DATA:

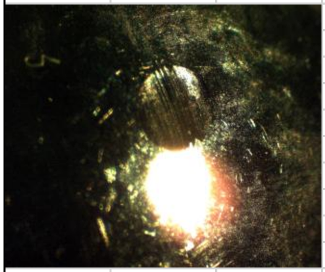
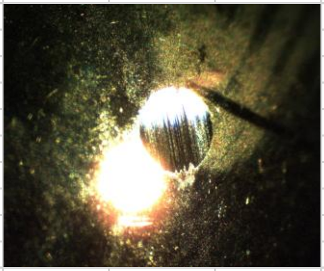
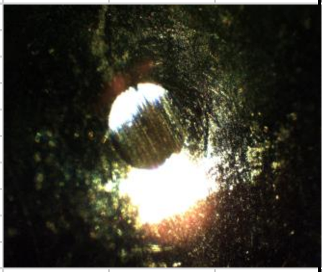
BALL SCAR MEASUREMENTS (mm):

	BALL 1		BALL 2		BALL 3
					
X-AXIS:	0.717	X-AXIS:	0.692	X-AXIS:	0.684
Y-AXIS:	0.743	Y-AXIS:	0.791	Y-AXIS:	0.698
	0.73		0.7415		0.691
	AVERAGE SCAR DIAMETER:			**0.721**	

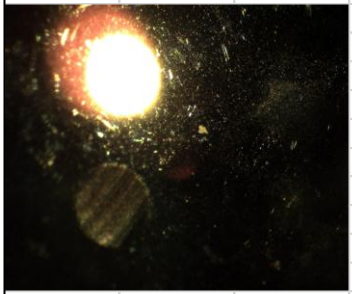
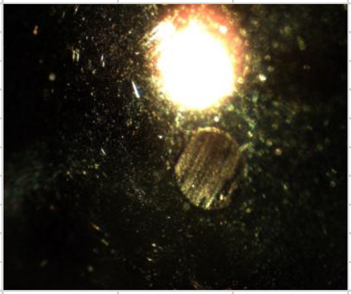
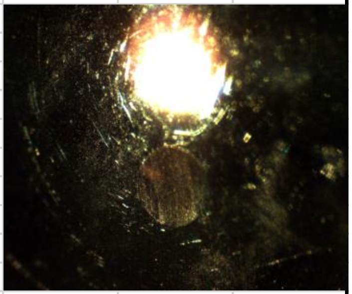
8. Regular Diesel Fuel and .5% Safflower Oil

WEAR DATA:

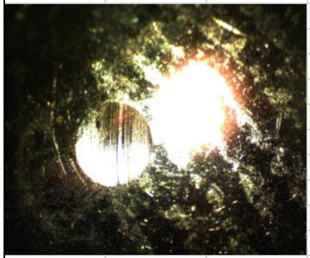
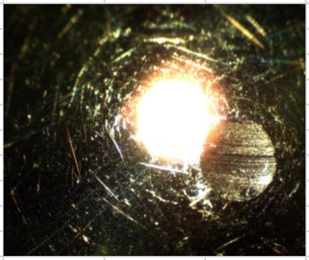
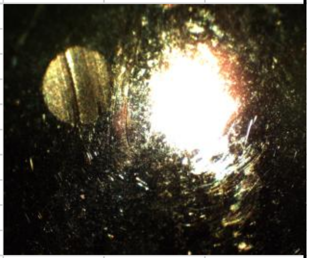
BALL SCAR MEASUREMENTS (mm):

	BALL 1		BALL 2		BALL 3
					
X-AXIS:	0.818	X-AXIS:	0.823	X-AXIS:	0.815
Y-AXIS:	0.777	Y-AXIS:	0.807	Y-AXIS:	0.795
	0.7975		0.815		0.805
	AVERAGE SCAR DIAMETER:			**0.806**	

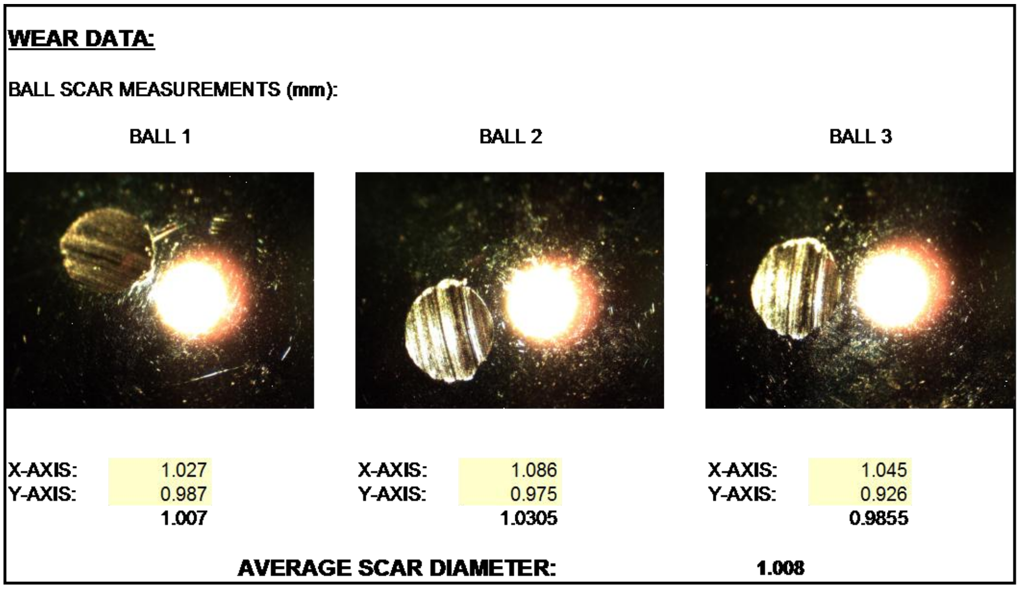
9. Low Sulfur Diesel Fuel and .5% Safflower Oil

WEAR DATA:					
BALL SCAR MEASUREMENTS (mm):					
BALL 1		BALL 2		BALL 3	
					
X-AXIS:	0.703	X-AXIS:	0.722	X-AXIS:	0.749
Y-AXIS:	0.704	Y-AXIS:	0.673	Y-AXIS:	0.719
	0.7035		0.6975		0.734
	AVERAGE SCAR DIAMETER:			**0.712**	

10. 0.05% methyl ester made from Olive Oil mixed in Regular Diesel Fuel

WEAR DATA:					
BALL SCAR MEASUREMENTS (mm):					
BALL 1		BALL 2		BALL 3	
					
X-AXIS:	0.841	X-AXIS:	0.793	X-AXIS:	0.869
Y-AXIS:	0.827	Y-AXIS:	0.809	Y-AXIS:	0.852
	0.834		0.801		0.8605
	AVERAGE SCAR DIAMETER:			**0.832**	

11. 0.1% methyl ester made from Olive Oil mixed in Regular Diesel



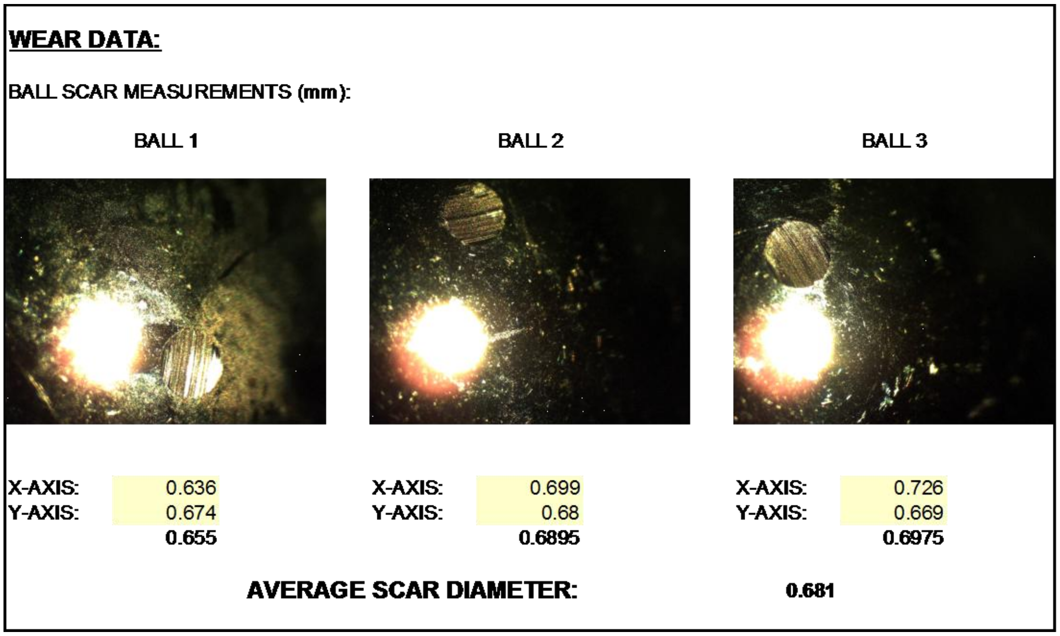
WEAR DATA:

BALL SCAR MEASUREMENTS (mm):

	BALL 1	BALL 2	BALL 3
X-AXIS:	1.027	1.086	1.045
Y-AXIS:	0.987	0.975	0.926
	1.007	**1.0305**	**0.9855**

AVERAGE SCAR DIAMETER: **1.008**

12. 0.5% methyl ester made from Olive Oil mixed in Regular Diesel Fuel



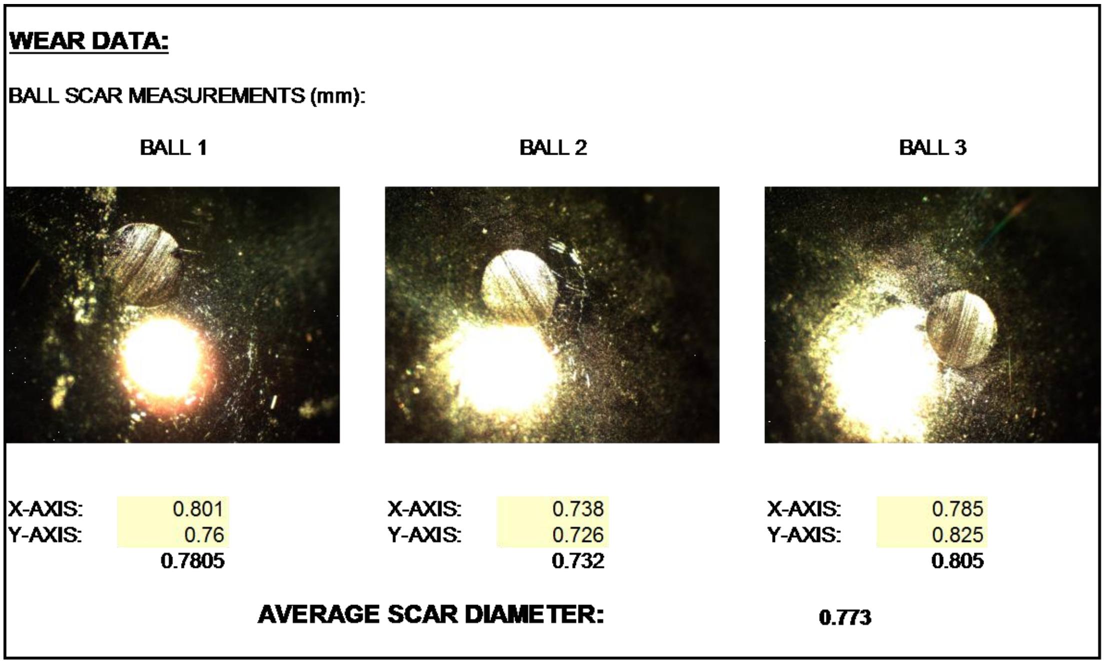
WEAR DATA:

BALL SCAR MEASUREMENTS (mm):

	BALL 1	BALL 2	BALL 3
X-AXIS:	0.636	0.699	0.726
Y-AXIS:	0.674	0.68	0.669
	0.655	**0.6895**	**0.6975**

AVERAGE SCAR DIAMETER: **0.681**

13. 0.05% methyl ester made from Olive Oil mixed in Low Sulfur Diesel Fuel



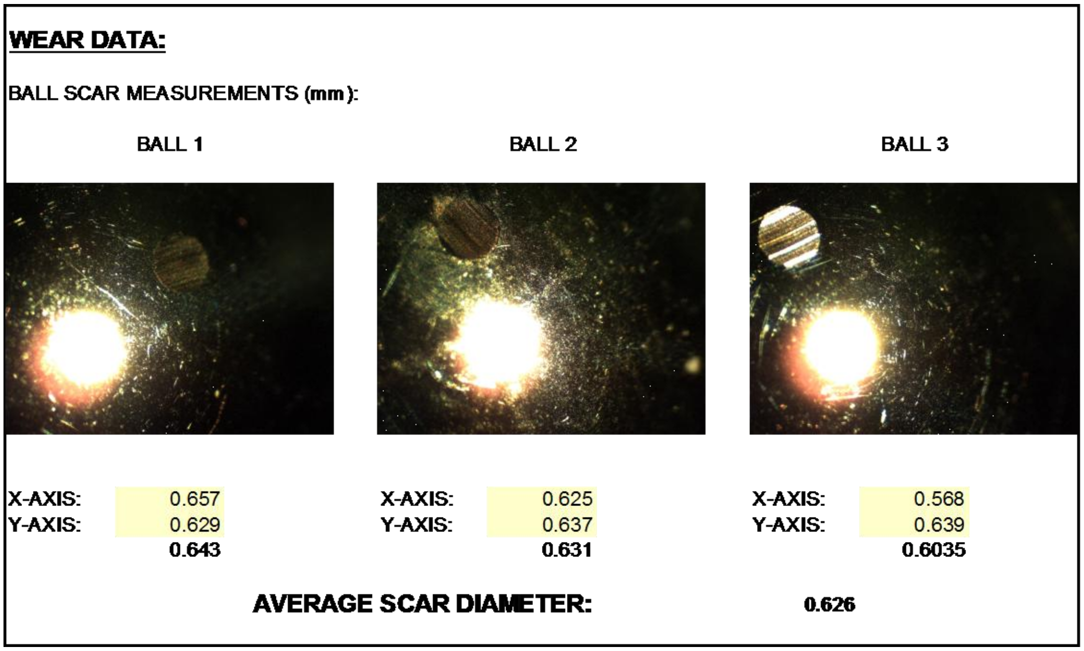
WEAR DATA:

BALL SCAR MEASUREMENTS (mm):

	BALL 1		BALL 2		BALL 3
X-AXIS:	0.801	X-AXIS:	0.738	X-AXIS:	0.785
Y-AXIS:	0.76	Y-AXIS:	0.726	Y-AXIS:	0.825
	0.7805		**0.732**		**0.805**

AVERAGE SCAR DIAMETER: **0.773**

14. 0.1% methyl ester made from Olive Oil mixed in Low Sulfur Diesel Fuel



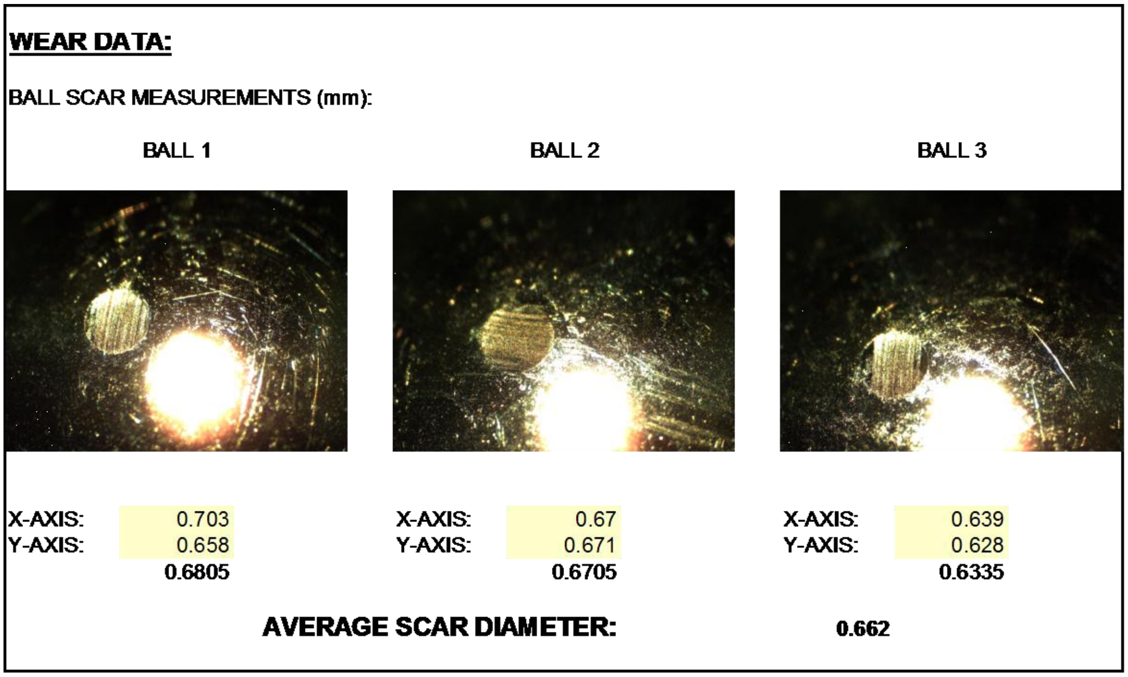
WEAR DATA:

BALL SCAR MEASUREMENTS (mm):

	BALL 1		BALL 2		BALL 3
X-AXIS:	0.657	X-AXIS:	0.625	X-AXIS:	0.568
Y-AXIS:	0.629	Y-AXIS:	0.637	Y-AXIS:	0.639
	0.643		**0.631**		**0.6035**

AVERAGE SCAR DIAMETER: **0.626**

15. 0.5% methyl ester made from Olive Oil mixed in Low Sulfur Diesel Fuel



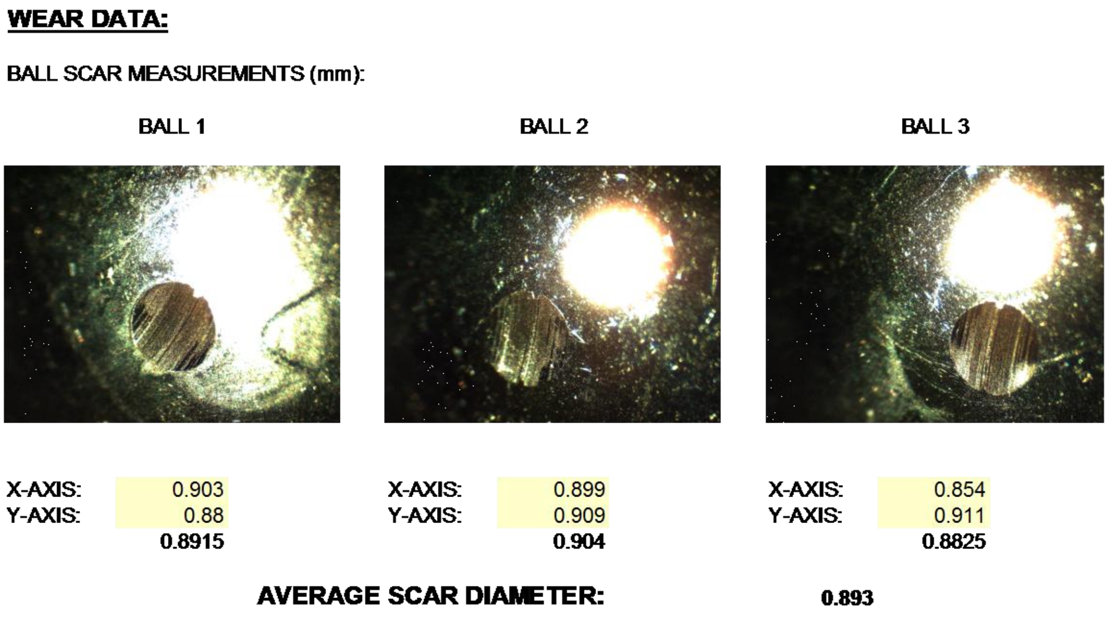
WEAR DATA:

BALL SCAR MEASUREMENTS (mm):

	BALL 1	BALL 2	BALL 3
X-AXIS:	0.703	0.67	0.639
Y-AXIS:	0.658	0.671	0.628
	0.6805	**0.6705**	**0.6335**

AVERAGE SCAR DIAMETER: **0.662**

16. 0.05% methyl ester made from Coconut Oil mixed in Regular Diesel Fuel



WEAR DATA:

BALL SCAR MEASUREMENTS (mm):

	BALL 1	BALL 2	BALL 3
X-AXIS:	0.903	0.899	0.854
Y-AXIS:	0.88	0.909	0.911
	0.8915	**0.904**	**0.8825**

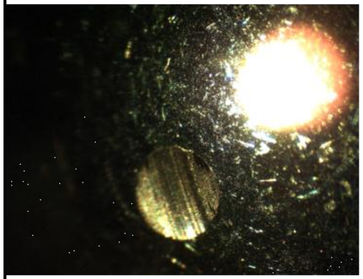
AVERAGE SCAR DIAMETER: **0.893**

17. 0.1% methyl ester made from Coconut Oil mixed in Regular Diesel Fuel

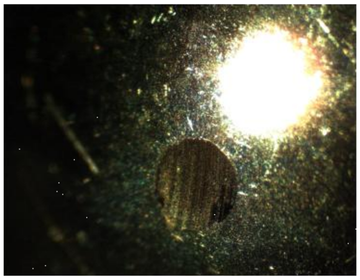
WEAR DATA:

BALL SCAR MEASUREMENTS (mm):

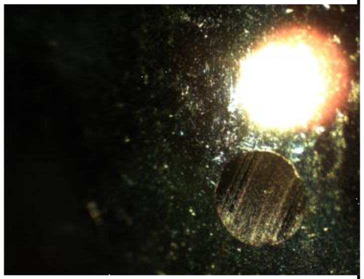
BALL 1



BALL 2



BALL 3



	BALL 1	BALL 2	BALL 3
X-AXIS:	0.859	0.888	0.888
Y-AXIS:	0.815	0.9	0.798
	0.837	**0.894**	**0.843**

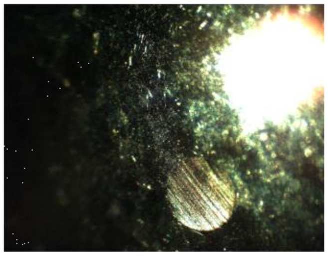
AVERAGE SCAR DIAMETER: **0.858**

18. 0.5% methyl ester made from Coconut Oil mixed in Regular Diesel Fuel

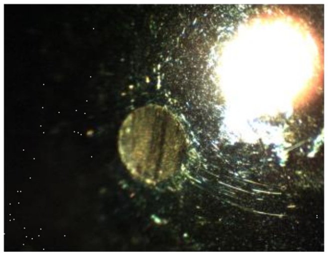
WEAR DATA:

BALL SCAR MEASUREMENTS (mm):

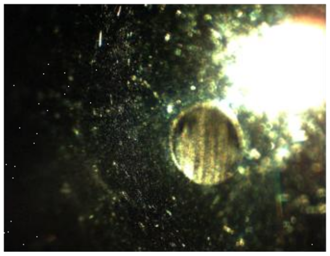
BALL 1



BALL 2



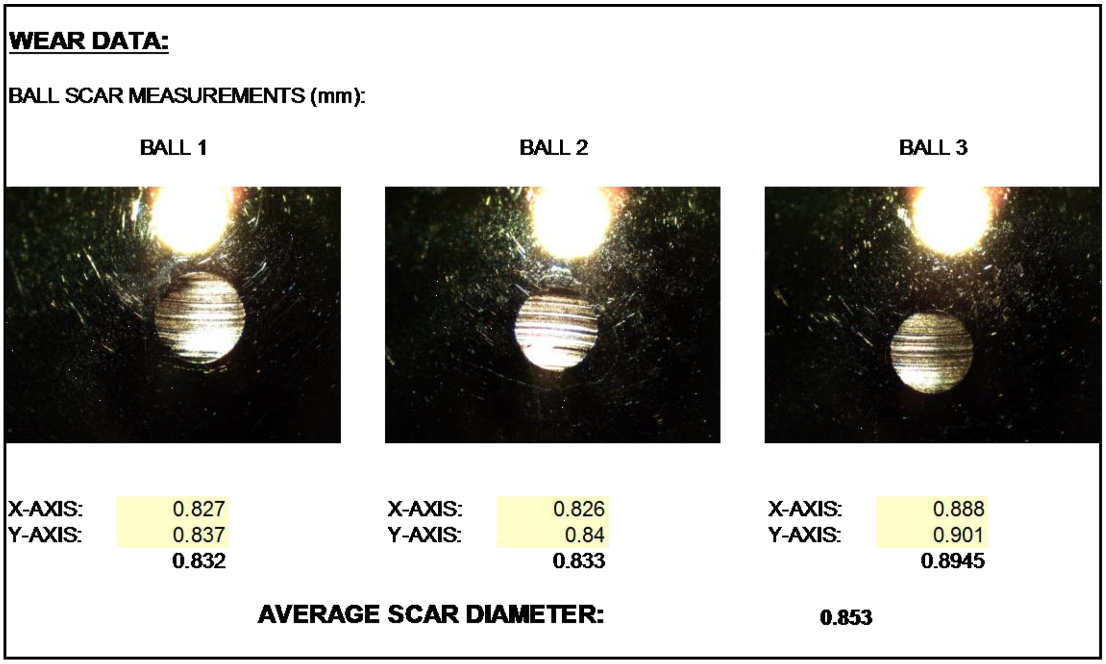
BALL 3



	BALL 1	BALL 2	BALL 3
X-AXIS:	0.806	0.842	0.793
Y-AXIS:	0.746	0.805	0.713
	0.776	**0.8235**	**0.753**

AVERAGE SCAR DIAMETER: **0.784**

19. 0.05% methyl ester made from Coconut Oil mixed in Low Sulfur Diesel Fuel



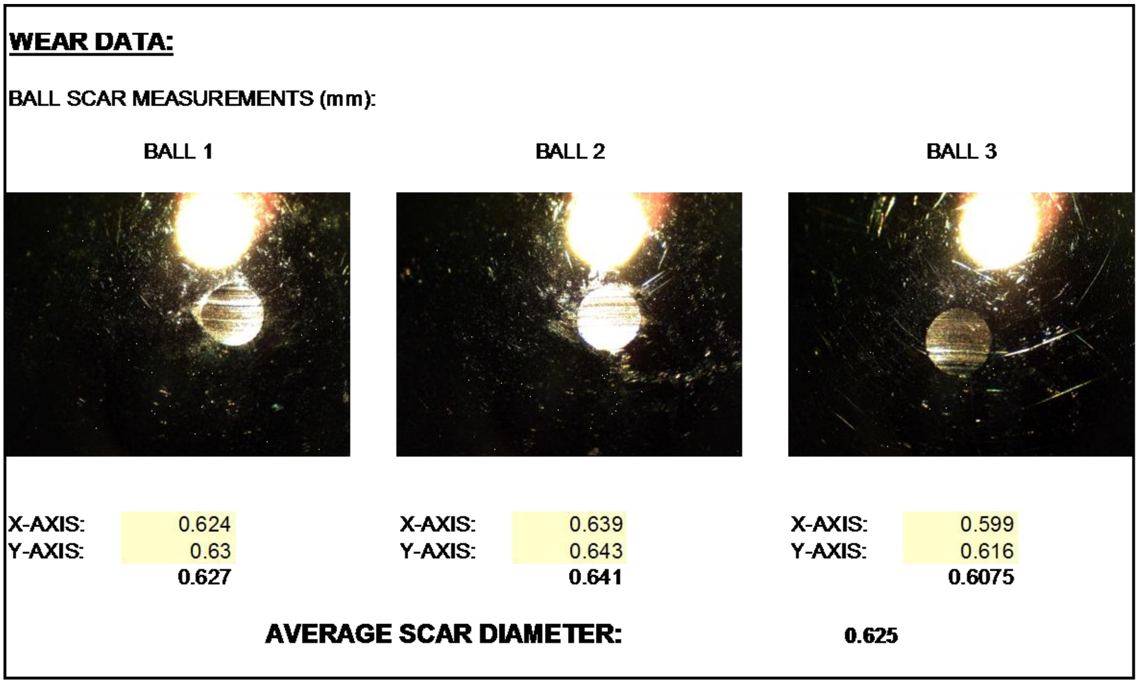
WEAR DATA:

BALL SCAR MEASUREMENTS (mm):

	BALL 1	BALL 2	BALL 3
X-AXIS:	0.827	0.826	0.888
Y-AXIS:	0.837	0.84	0.901
	0.832	**0.833**	**0.8945**

AVERAGE SCAR DIAMETER: **0.853**

20. 0.5% methyl ester made from Coconut Oil mixed in Low Sulfur Diesel Fuel



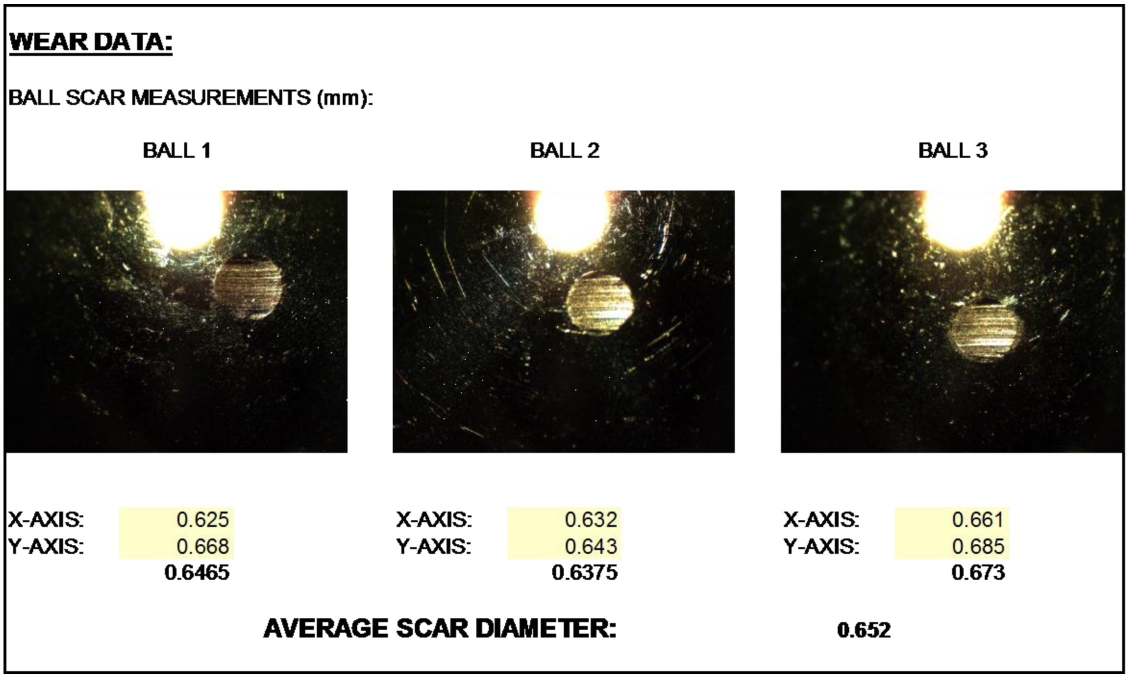
WEAR DATA:

BALL SCAR MEASUREMENTS (mm):

	BALL 1	BALL 2	BALL 3
X-AXIS:	0.624	0.639	0.599
Y-AXIS:	0.63	0.643	0.616
	0.627	**0.641**	**0.6075**

AVERAGE SCAR DIAMETER: **0.625**

21. 0.1% methyl ester made from Castor Oil mixed in Regular Diesel Fuel



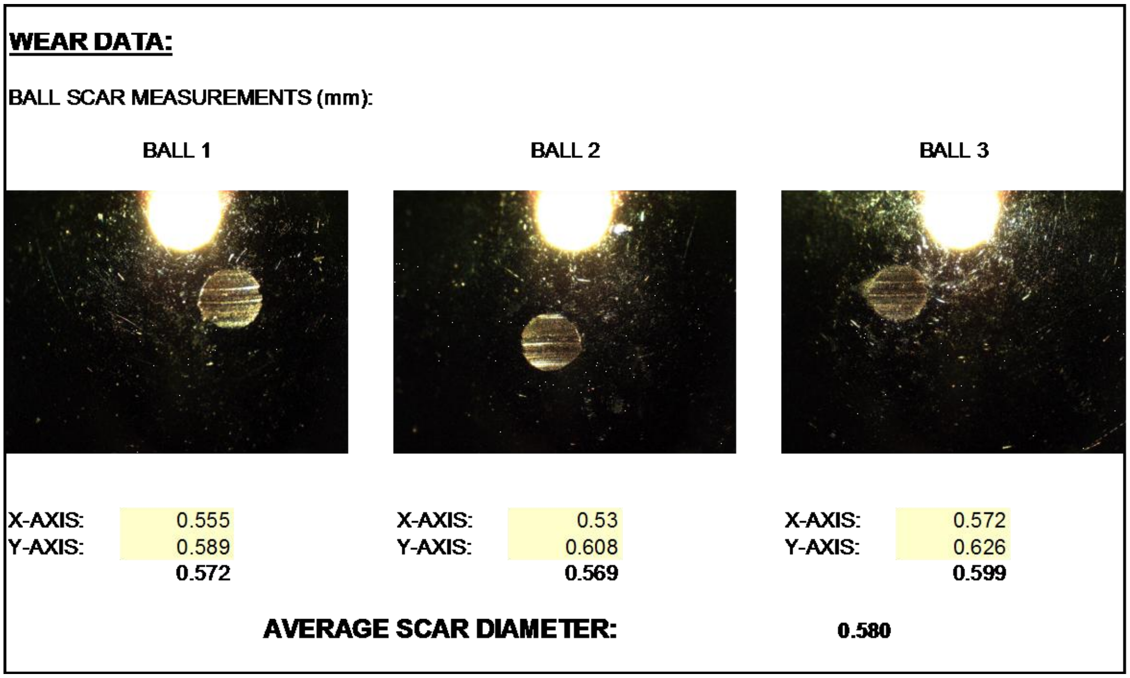
WEAR DATA:

BALL SCAR MEASUREMENTS (mm):

	BALL 1		BALL 2		BALL 3
X-AXIS:	0.625	X-AXIS:	0.632	X-AXIS:	0.661
Y-AXIS:	0.668	Y-AXIS:	0.643	Y-AXIS:	0.685
	0.6465		**0.6375**		**0.673**

AVERAGE SCAR DIAMETER: **0.652**

22. 0.5% methyl ester made from Castor Oil mixed in Regular Diesel Fuel



WEAR DATA:

BALL SCAR MEASUREMENTS (mm):

	BALL 1		BALL 2		BALL 3
X-AXIS:	0.555	X-AXIS:	0.53	X-AXIS:	0.572
Y-AXIS:	0.589	Y-AXIS:	0.608	Y-AXIS:	0.626
	0.572		**0.569**		**0.599**

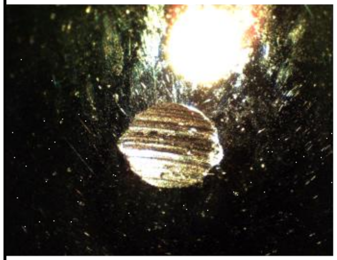
AVERAGE SCAR DIAMETER: **0.580**

23. 0.05% methyl ester made from Castor Oil mixed in Low Sulfur Diesel Fuel

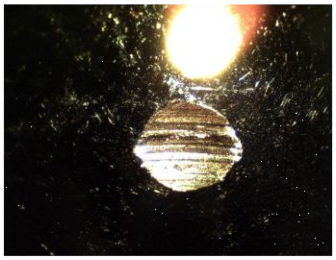
WEAR DATA:

BALL SCAR MEASUREMENTS (mm):

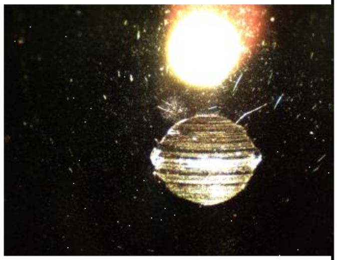
BALL 1



BALL 2



BALL 3



X-AXIS:	0.906	X-AXIS:	0.916	X-AXIS:	0.856
Y-AXIS:	1.115	Y-AXIS:	1.14	Y-AXIS:	1.096
	1.0105		**1.028**		**0.976**

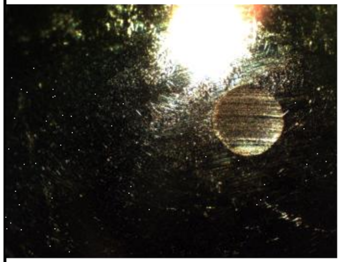
AVERAGE SCAR DIAMETER: **1.005**

24. 0.1% methyl ester made from Castor Oil mixed in Low Sulfur Diesel Fuel

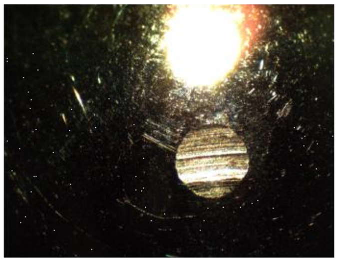
WEAR DATA:

BALL SCAR MEASUREMENTS (mm):

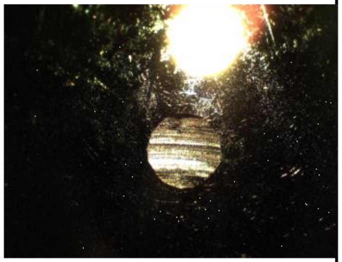
BALL 1



BALL 2



BALL 3



X-AXIS:	0.745	X-AXIS:	0.744	X-AXIS:	0.724
Y-AXIS:	0.753	Y-AXIS:	0.753	Y-AXIS:	0.732
	0.749		**0.7485**		**0.728**

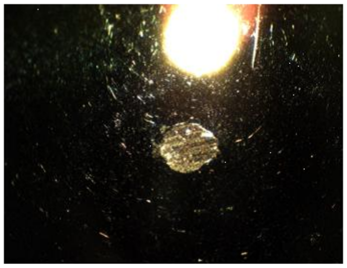
AVERAGE SCAR DIAMETER: **0.742**

25. 0.5% methyl ester made from Castor Oil mixed in Low Sulfur Diesel Fuel

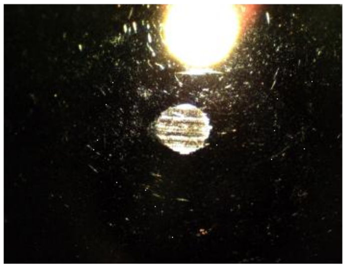
WEAR DATA:

BALL SCAR MEASUREMENTS (mm):

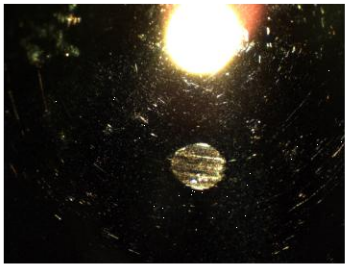
BALL 1



BALL 2



BALL 3



	BALL 1		BALL 2		BALL 3
X-AXIS:	0.485	X-AXIS:	0.457	X-AXIS:	0.488
Y-AXIS:	0.553	Y-AXIS:	0.549	Y-AXIS:	0.567
	0.519		**0.503**		**0.5275**

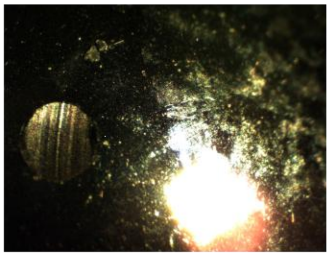
AVERAGE SCAR DIAMETER: **0.517**

26. 0.5% methyl ester made from Safflower Oil mixed in Low Sulfur Diesel Fuel

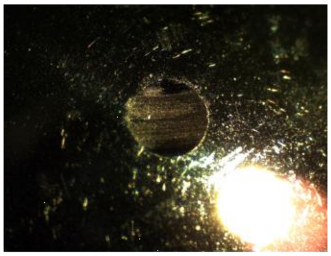
WEAR DATA:

BALL SCAR MEASUREMENTS (mm):

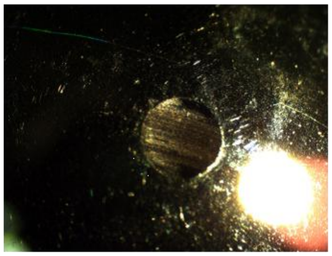
BALL 1



BALL 2



BALL 3



	BALL 1		BALL 2		BALL 3
X-AXIS:	0.851	X-AXIS:	0.848	X-AXIS:	0.79
Y-AXIS:	0.792	Y-AXIS:	0.834	Y-AXIS:	0.826
	0.8215		**0.841**		**0.808**

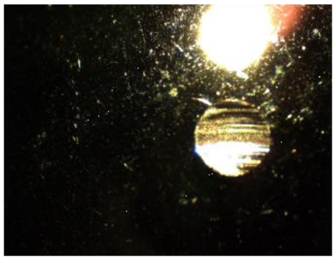
AVERAGE SCAR DIAMETER: **0.824**

27. 0.1% methyl ester made from Safflower Oil mixed in Low Sulfur Diesel Fuel

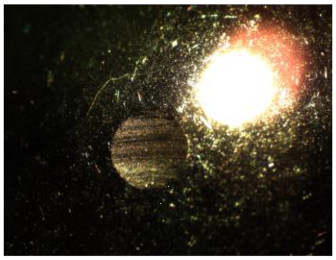
WEAR DATA:

BALL SCAR MEASUREMENTS (mm):

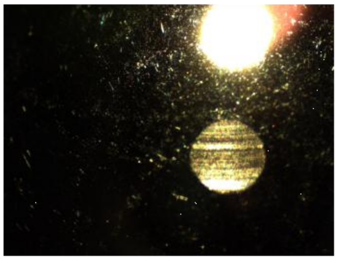
BALL 1



BALL 2



BALL 3



X-AXIS:	0.793	X-AXIS:	0.755	X-AXIS:	0.788
Y-AXIS:	0.788	Y-AXIS:	0.767	Y-AXIS:	0.792
	0.7905		**0.761**		**0.79**

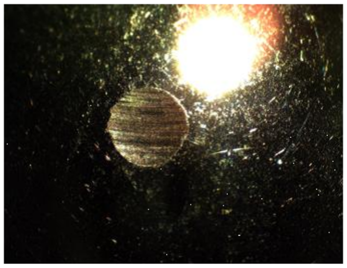
AVERAGE SCAR DIAMETER: **0.781**

28. 0.05% methyl ester made from Coconut Oil mixed in Low Sulfur Diesel Fuel

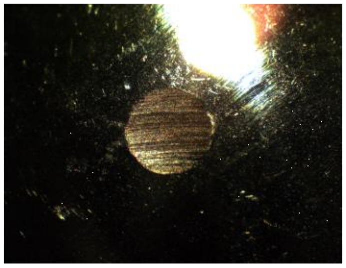
WEAR DATA:

BALL SCAR MEASUREMENTS (mm):

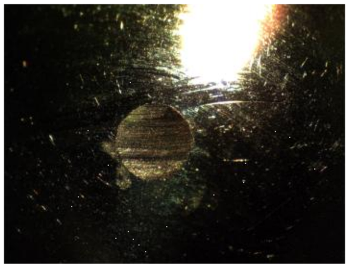
BALL 1



BALL 2



BALL 3



X-AXIS:	0.879	X-AXIS:	0.763	X-AXIS:	0.78
Y-AXIS:	0.846	Y-AXIS:	0.773	Y-AXIS:	0.806
	0.8625		**0.768**		**0.793**

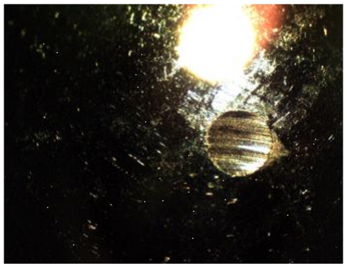
AVERAGE SCAR DIAMETER: **0.808**

29. 0.1% methyl ester made from Coconut Oil mixed in Low Sulfur Diesel Fuel

WEAR DATA:

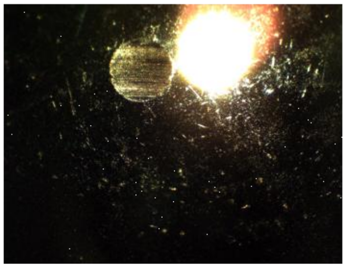
BALL SCAR MEASUREMENTS (mm):

BALL 1



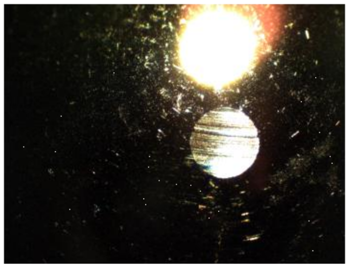
X-AXIS: 0.623
Y-AXIS: 0.641
0.632

BALL 2



X-AXIS: 0.688
Y-AXIS: 0.692
0.69

BALL 3



X-AXIS: 0.67
Y-AXIS: 0.645
0.6575

AVERAGE SCAR DIAMETER: **0.660**

Patents:

Honary, Lou A.T. *Soybean Oil Impregnation Wood Preservative Process and Products.* 2003. U.S. Pat. 6,641,927.

Patent applied for (April 2009) by Lou Honary on Continuous Oil Recycling Syste (CORS).

Patent applied for (April 2009), by Lou Honary and Wes James on Microwave Based Grease Manufacturing Process.

International patent applied for (April 2010), by Lou Honary and Wes James on Microwave Based Grease Manufacturing Process.

Publications / Presentations:

September 2012: Honary, L., and James, W. "Manufacturing Biobased Grease Using Microwaves."; NLGI Spokesman, September/October 2012, Vol. 76, No 4, pp. 38-46.

June 2012: Honary, L. "Microwave-based manufacturing for lower-cost biobased lubricants and chemicals"; AOCS Inform magazine, June 2012, Vol. 23, pp. 388-394.

April 29, 2012 - Lou Honary chaired the Biobased Grease Performance Working Group Meeting at the annual meeting of the European Lubricating Grease Institute in Munich, Germany.

Presentation at the Society of Tribology and Lubrication Engineers (Chicago Chapter) March 2012 - Topic: Biobased Lubricants

November 20, 2011 - Lou Honary chaired the Biobased Grease Performance Working Group Meeting in Amsterdam for the European Lubricating Grease Institute

Paper and Presentation at the National Lubricating Grease Institute's Annual Meeting June 20111 - Topic: A status update on manufacturing biobased grease with microwaves

Paper and Presentation with Cassandra Boevers at the National Lubricating Grease Institute's Annual Meeting June 20111 - Topic: A study of compatibility of fully formulated biobased and conventional greases

June, 2011, Lou Honary chaired the Biobased Grease Performance Working Group Meeting in California for the National lubricating Grease Institute

April 2011 – Book published: Honary and L.A. and Richter, E. (2011) Biobased Lubricants and Greases: Technology and Products. Wiley and Sons, UK

April 2011: Book Published: Honary, L. A. and Conconi, C. (eds.) (2011) Journal of ASTM International Selected Technical Papers STP 1477: Biofuels