March 2003 • NREL/SR-510-31460

Subcontractor Report

Production of Biodiesels from Multiple Feedstocks and Properties of Biodiesels and Biodiesel/Diesel Blends

Final Report
Report 1 in a series of 6

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SUMMARY

In a project sponsored by the National Renewable Energy Laboratory, the Institute of Gas Technology is conducting an investigation of biodiesels (methyl esters) produced from vegetable- and animal-based feedstocks.

Soy biodiesel is predominantly used in the United States, while canola biodiesel is dominant in Europe. The high price of biodiesel (over double the price of diesel) is in large part due to the high price of the feedstock. However, biodiesel can be made from other feedstocks, including beef tallow, pork lard, and yellow grease. This project sought to understand the impact of the different feedstocks by generating biodiesel from each feedstock through one producer, to minimize the variation in production techniques, and by subjecting the samples to a series of tests to quantify the differences between them.

The production of methyl esters identified a number of conclusions

- Biodiesel is easier to produce and cleaner with equivalent amounts of processing when starting with clean vegetable oil (e.g., soy and canola). The tallow, lard, and yellow grease biodiesels need additional processing at the end of transesterification to achieve acceptable biodiesel properties, and would benefit from processing before transesterification to reduce or eliminate components that may interfere, as for the high free fatty acid yellow grease.

- Additional investigation should be considered that would focus on methods of economically handling free fatty acids and other components, at least to reduce their interference, and ideally, contribute to the production of methyl esters.

- Adequate testing is required during and after the processing to know the state of the fuel. Without it, the producer cannot ensure that "fuel grade" methyl esters are produced, instead of "commercial grade".

The properties testing of the biodiesels confirmed that the known advantages and disadvantages of soy and canola biodiesels are the same as the biodiesels based on the other feedstocks, and that the changes in properties expected from the feedstock variations occur.

- The test results show that there are problems in operating at lower temperatures (cloud point, cold filter plugging point, and pour point), and that the minimum temperatures at which biodiesels are usable increase as they move from vegetable to animal sources, due to the greater degree of saturation. Yellow grease, although originally vegetable oils, display intermediate temperatures. Additives to reduce cloud/plugging/pour point problems would help the adoption of biodiesels in areas with low ambient temperatures (much of the United States for the tallow and lard biodiesels).

- Viscosities show the same trends as temperatures, with the lard and tallow biodiesels higher than the soy and canola biodiesels. Because of the effect that high viscosities can have on injector spray performance, this property should be monitored in biodiesel production.

- The biodiesels have high boiling points, flash points, and extremely low vapor pressure, as well as an inability to smoke under the smoke point test. These results indicate a high level of safety for handling biodiesels.

The specifications used to identify methyl esters that are acceptable as biodiesel were reviewed in light of the properties testing. The biodiesel specifications do not need any additions from the test suite conducted in this project. The tests would contribute additional information about the composition and properties, but the tests in the specifications adequately identify whether the quality of the fuel is acceptable.

The biodiesel specification and the ASTM D 975 diesel specification can be partially applied to B20, with problems when the limiting values differ significantly between the two.

Biodiesel is mixed with diesel to bring many of the beneficial characteristics to diesel equipment, while reducing the overall cost of the fuel. Because biodiesel is usually used blended, the focus was to determine the characteristics of biodiesels from different feedstocks in varying concentrations with diesel when tested on a consistent basis. It was expected that some properties would vary in a non-intuitive manner, e.g., not varying linearly with respect to blend fraction.

The results add data to concentration ranges that have previously been overlooked in the study of the potential of biodiesel blends. It also adds information concerning feedstocks that have been considered only superficially.

The properties tested were those that would most affect operation of diesels, and consisted of: viscosity, pour point, cloud point, cold filter plugging point, Cetane number, scuffing load BOCLE (lubricity), and oxidation stability. Cetane number did not exhibit any unusual characteristics, with essentially linear variation with respect to blend fraction. Fuel oxidation stability tests show a predominantly linear relation between biodiesel fraction and insolubles produced, moving toward the higher levels of the biodiesels. However, two biodiesels showed peaks with 35% biodiesel that were significantly higher than pure biodiesel.

Of particular interest for people considering the use of biodiesels in other than B20 or pure applications is the shift in temperature for various properties tested. The biodiesel blends exhibited a viscosity depression at low concentrations. Conversely, most biodiesels significantly increased pour point, cloud point, and cold filter plugging point at low concentrations (<10-20%), then proceeded more linearly above that. Care should be taken in handling and use due to the temperature increases that occurred for pour point, cloud point, and cold filter plugging point.

The effect on lubricity cited by advocates of biodiesel was verified by the test results. Significant lubricity increases occur with concentrations at 3% or less of the biodiesels, seeming to confirm the concept that biodiesel additives can improve the operation of diesels and extend the life of their components. It is fortunate that these concentrations avoid the ranges where temperature increases occur. In particular, further study may be warranted to improve the processing of the beef tallow and pork lard, with a goal of reducing cost while enhancing the characteristics of the methyl esters that improve lubricity.

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INTRODUCTION

In a project sponsored by the National Renewable Energy Laboratory, the Gas Technology Institute (formerly Institute of Gas Technology) conducted an investigation of biodiesel fuels produced from various vegetable- and animal-based feedstocks. Due to the varying nature of the feedstocks and the processes for converting them into suitable biodiesel (methyl esters), an important part of the study has been the laboratory testing of the properties to determine the characteristics of each fuel.

The effort was conducted in three parts. The first segment of the project involved the production of methyl esters from the various feedstocks and a review of the issues encountered with each feedstock. In the second part, the biodiesels were subjected to a series of tests to document the properties of the methyl esters. With an understanding of the values and importance of each test, the specifications for acceptance of biodiesel were reviewed.

In the third part, the effect of mixing biodiesel and diesel was studied, because an understanding of the basic properties does not necessarily indicate what will happen as a result of mixing it with diesel fuel. Due to the cost, biodiesel usually is mixed with diesel (instead of used straight, or "neat") to bring many of the beneficial characteristics to diesel equipment, while reducing the overall cost of the fuel. Because of the different compositions and basic differences between biodiesel and diesel, some properties may vary in a non-intuitive manner, e.g., not varying linearly with respect to blend fraction. The testing quantifies these effects of blending the multi-feedstock biodiesels with diesel over a range of varying compositions.

PRODUCTION OF BIODIESEL FROM MULTIPLE FEEDSTOCKS

Biodiesel Processing Background

At the beginning of this project, there were three common types of biodiesel production technology, the use of which depended on feedstock quality and the sophistication of the facility. Combinations of some technologies are possible.

Fats and oils are composed of molecules called triglycerides. Each triglyceride is composed of three long-chain fatty acids of 8 to 22 carbons attached to a glycerol backbone. Biodiesel is composed of fatty acid chains that are chemically bonded to one methanol molecule. The glycerol molecules are almost completely removed from the final biodiesel product. Biodiesel is sometimes called fatty acid methyl esters or FAME. The glycerin byproduct has thousands of industrial chemical uses in common household products and foods. When the fatty acid chains break off the triglyceride, they are known as free fatty acids. Free fatty acids are desirable biodiesel feedstocks, but require different conversion processes compared to triglycerides.

Biodiesel feedstock are classified based on their free fatty acid content as follows:

- Refined oils, such as soy bean or refined canola oil (FFA <1.5%)
- Low free fatty acid yellow greases and animal fats (FFA<4%)
- High free fatty acid greases and animal fats (FFA ≥ 20%)

There are other potential feedstocks available at this time, namely trap and sewage grease and other very high free fatty acid greases whose FFA exceed 50%. Technology improvements need to be developed before these feedstocks can be used for biodiesel production.

Commercial biodiesel technologies can be grouped as follows:

- Base catalyzed transesterification with refined oils
- Base catalyzed transesterification with low free fatty acid greases and fats
- Acid esterification followed by transesterification of low or high free fatty acid greases and fats

Technologies can be run as batch or continuous processes. Given the limited size of the domestic market for biodiesel, most U.S. firms have used batch technology. Continuous processes used in Europe and in industrial processes in the U.S. (to produce methyl esters for uses other than as fuel) can use raw or may require refined oils. Batch processes provide excellent opportunities for quality control if variations in feedstock quality are common, such as with yellow grease and animal fats.

The goal of all the technologies is to produce a fuel grade biodiesel whose properties meet ASTM PS 121. The key quality control issues involve the complete (or nearly complete) removal of alcohol, catalyst, water, soaps, glycerine, and unreacted or partially reacted triglycerides and free fatty acids. Failure to remove or minimize these contaminates causes the methyl ester product to fail one or more fuel standards.

There are a wide variety of commercial methyl ester products made for the industrial lubricant and chemical processing industries. Processing requirements for these products may not be sufficiently robust to produce a fuel-grade biodiesel. The most common problem is the presence of high levels of unreacted or partially reacted triglycerides and glycerine, which increases viscosity, raises cloud and pour points, causes product separation, and may lead to engine damage. Only methyl esters that meet ASTM PS 121 are considered “biodiesel.”

A brief overview of each major technology follows.

Base catalyzed transesterification using refined oils has high efficiencies, up to 99.9%, and produces good to high quality fuels after removing excess methanol, base catalyst, and glycerine. A description of the technology can be found in Sheehan, J.; Camobreco, V.; Duffield, J.; Graboski, M.; Shapouri. H. (1998) **Life Cycle Inventory of Biodiesel and Petroleum Diesel for Use in an Urban Bus**: Final Report. 314 pp.; NICH Report No. SR-580-24089.

The basic chemistry of the reaction requires 3 molecules of methanol (or other alcohol) for every molecule of triglyceride (e.g., oil or fat), which corresponds roughly to about 10% the weight of methanol per mass of oil processed. The catalyst ratio is roughly 10% of the methanol mass. Glycerine is the major byproduct of the process.

A typical input/output stream is shown below.

Input streams:

Refined oil	1,000 kg
Methanol	107 kg
KOH 88%	10 kg
Acid (sulfuric, acetic, HCL)	8 kg
Water	17 kg
Electricity	20 kWh

Output Streams:

Biodiesel	1,000 kg
Glycerine 88%	125 kg
Fertilizer	23 kg
Byproduct chemicals	nil

Small amounts of FFA (less than 1.5%) are converted into soaps. These soaps typically are removed with the glycerine or removed during the crude oil refining process. Other commonly used base catalysts include sodium hydroxide or sodium methoxide. Sodium catalysts do not produce fertilizer byproducts. The acids are used to break emulsifications in the glycerine byproducts for partial processing and to neutralize the base catalysts.

There are numerous variations of this basic technology. Different catalysts, including non-alkaline catalysts can be used. Anhydrous ethanol, isopropyl or butyl alcohols can be substituted for methanol, but reaction times are slower, and yields may be lower, resulting in more rigorous quality control measures and additional processing. Various grades of glycerin can be produced, depending on the scale of the facility. Crude glycerine is often shipped to larger facilities for refining. The basic transesterification process is run at standard atmosphere and temperatures around 60°C. However, some continuous technologies use higher temperatures and elevated pressures, typically in the super critical range of methanol. Distillation is sometimes used for quality control, but not always necessary.

Base catalyzed transesterification using low free fatty acid feedstocks is a simple variation of the technology described above. Typically, a small amount of base catalyst is added to the feedstock to react with the free fatty acids and form soaps. The soaps are removed, and the transesterification process proceeds. Back yard producers and very small plants tend to recover the soap for novelty products or compost it. An equal amount of clean oils tends to be lost with the soaps, leading to significant yield losses depending on the free fatty acids level of the feedstock. Larger facilities can react the soaps back into free fatty acids and market them as agricultural products. The attractiveness of this variation depends on the availability of local agricultural markets for the free fatty acids and the value of those products (should be higher than biodiesel).

A more efficient approach to high free fatty acid feedstocks is the last technology to be reviewed—acid esterification followed by transesterification. The free fatty acids are reacted with methanol (1:1 ratio) and acid catalysts such as sulfuric acid, to form methyl esters. Yields on this reaction are typically less than 96%, which means that roughly 4% of the available free fatty acids remain in the feedstock stream and will react with the base catalyst in the next step to form soaps. Some foaming control processes may be required if the soaps are not removed prior to transesterification. The rest of the process is similar to those described above. Resulting yields can exceed 99%, depending on the amount of free fatty acids in the original feedstock, and the variety of byproducts produced.

Feedstocks Considered For Biodiesel

The previous experience with biodiesels has focused on fuel derived from soybean or canola (rapeseed) oil. These oils are available in a consistently high quality form, and are the easiest to process for making biodiesels. Most of the biodiesel available in the United States is based on soybean oil. Soybean and canola oil were included in this project as a reference for the other biodiesels.

In addition to plant oils, three animal (livestock) feedstocks were included in this project. Beef tallow consists of the renderings from meat packing and is available in either an edible or inedible form. The difference between the two forms is based on what is considered acceptable for food products. As a result, there is traditionally a larger variation in the composition of inedible beef tallow compared to the edible tallow. Pork lard is the swine-derived counterpart to beef tallow, also as a result of the meat packing process. The advantages of tallow and lard are their lower cost compared to soybean and canola oil. However, a disadvantage is that they may require additional processing to produce an acceptable biodiesel.

The third class of feedstocks is waste yellow grease. It is soybean oil (or equivalent) which has been used for cooking. Characteristic of the feedstock are the larger amounts of free fatty acids that have been liberated during the cooking process. Because of seasonal variations, the free fatty acid content can range from 2% to 20% or more. However, 4% to 10% is a more typical range, and was selected for the two yellow greases. Like the tallows and lard, the variation in composition is much wider than the plant oils.

Acidulated soapstock was considered as another feedstock, but was not available because the group developing the process was unable to produce methyl esters in sufficient quantity for testing.

Biodiesel Production and Problems

Methyl esters were required to meet the specifications shown in Table 1 (which were being evaluated by ASTM at the time) to be considered acceptable for use as biodiesels at the start of this project. The subsequently released ASTM PS121-99, **Provisional Specification for Biodiesel Fuel (B100) Blend Stock for Distillate Fuels**, is substantially similar to the draft standard used in this project. To determine whether the biodiesels were potentially acceptable, individuals from IGT, NREL, and the National Biodiesel Board had decided that an Acid Number screening test would be used to ensure that each of the biodiesel samples met at least this minimum fuel quality requirement before investing money into the complete suite of analyses.

Table 1. Biodiesel Specifications (July 1996)
(For pure mono alkyl esters of long chain fatty acids
derived from renewable lipid feedstocks)

Property	ASTM Method	Limits	Units
Flash Point	D93	100.0 min.	°C
Water & Sediment	D1796	0.050 max.	vol. %
Carbon Residue, 100% sample	D524[1]	0.050 max.	wt. %
Sulfated Ash	D874	0.020 max.	wt. %
Kinematic Viscosity, 40°C	D445	1.9 — 6.5	$mm^{2/}$sec (cSt)
Sulfur	D2622	0.050 max.	wt. %
Cetane	D613	40 min.	
Cloud Point	D2500	By Customer	°C
Copper Strip Corrosion	D130	No. 3b max.	
Acid Number	D664	0.80 max.	mg KOH/g
Free Glycerin	GC[2]	0.020 max.	wt. %
Total Glycerin	GG[2]	0.240 max.	wt. %

[1] or equivalent ASTM testing method.
[2] Austrian (Christina Planc) update of USDA test method.

Chemol Company, Inc., agreed to manufacture biodiesel from the feedstocks as part of this project. They applied their expertise with producing methyl esters for other purposes to deliver biodiesel. The soy, canola, inedible beef tallow, and pork lard feedstocks were supplied by Chemol. The low free-fatty-acid yellow grease (nominal 2% to 5%) was from Simplot (Idaho), and the edible beef tallow was from HRR Enterprises (Chicago, IL). In processing the feedstocks, they used some of their smaller equipment in batch mode.

There were initial problems in that only two methyl esters passed the Acid Number test (less than 0.8 mg KOH/g). These preliminary results indicated that "commercial grade" methyl esters may not meet "fuel grade" requirements. It had then been decided that Chemol would reprocess the rejected biodiesels.

The initial soy methyl ester that passed the Acid Number test did not meet the complete biodiesel fuel specification. Although the free glycerin level was within the 0.02 wt. % limit, the total glycerin value of 0.798 wt. % reported in the analysis considerably exceeded the 0.24 wt. % maximum value of the specification. The test results for the methyl tallow indicated it passed. The total glycerin was 0.102 wt. %, well within the biodiesel requirements. Other samples had similar problems during the production of the biodiesel fuels.

After additional processing and modifications to their facility, Chemol was able to deliver adequate methyl esters from each of the feedstocks except for the high free fatty acid yellow grease. The high concentration of free fatty acids interfered with their ability to process the yellow grease.

A review of the process indicated a number of problems in producing the methyl esters. At Chemol, the heated oil is dried under vacuum since water deactivates the catalyst. Transesterification then occurs when the ester reacts with carboxylic acid or alcohol; methanol is used when the goal is methyl esters. Sodium methoxide catalyst is added (0.1% to 0.3%) and the mixture is allowed to react. After the reaction, the catalyst is removed by water washing or acidification. The mixture is then filtered to remove trace soaps and heated under vacuum to remove small amounts of fatty acid methyl esters.

When processing feedstocks with free fatty acids (FFAs), additional steps must be taken, because the FFAs will deactivate the catalyst during reaction. Higher FFA concentrations result in more oil that is lost in processing. The FFAs, such as that in yellow grease, are formed from triglycerides (principal component of fats and oils). These form naturally from enzymes in oils, oxidation, or acids/bases/moisture and heat reactions. Removal of FFAs at Chemol involves alkali refining. In this process, the oil is mixed with caustic soda (sodium hydroxide) to form soaps. The soap is dispersed in aqueous phase together with phospholipids, pigments, and other compounds. Some oil is removed with the soaps (loss) as the soap is separated. The processing parameters are a function of FFA content, phospholipids (which act as surfactants), and caustic concentration. Soap must be removed to prevent filter clogging in the bleaching steps. The soapstock is commonly acidified and then sold as animal feed. The remaining material can then be processed into methyl esters. The limitations in the Chemol pilot scale process revolve around the composition of the oil after the FFAs are substantially removed. Since Chemol’s process requires that less than 1% FFAs enter the transesterification process; the full benefits of utilizing inexpensive waste greases may not be realized in this environment.

After the basic transesterification process, the cleanup is equally important in providing methyl esters suitable for use as fuel. Some of the biodiesels had high total glycerin values, which was considered not surprising by Chemol. This value is tied to the conversion efficiency of their process and the degree of reprocessing. Chemol's ability to control this value was based on the reprocessing procedure as well as their ability to accurately determine the total glycerin value. Since Chemol was not initially using the C. Planc GC method to determine the free and total glycerin levels, they cannot precisely determine the levels of free and total glycerin. Adequate testing during the processing was introduced as the project continued.

CIFER identified a supplier of a suitable high free fatty acid yellow grease, who was also willing to produce the biodiesel. The biodiesel was produced in the following steps:

1. Acid catalyzed pre-esterification of free fatty acids.
2. Washing and prep.
3. Conventional Transesterification.
4. Washing and prep.

The washing and preparation steps, especially, are part of a patented process described in U.S. Patent Nos. 5,399,791 and 5,434,279. By working to convert the free fatty acids into a form that wouldn't interfere with the basic transesterification, CIFER's supplier was able to avoid the problems that had occurred at Chemol.

Production Issues and Potential Solutions

In summary, the basic problems and potential solutions are the following:

1. Free fatty acids and other compounds interfere with transesterification, reacting with the base to form soaps. One approach is to supply additional base to compensate for the amount lost and to ensure processing completion in an acceptable period of time. However, this increases processing costs. In addition, water is formed during the base and free fatty acid reaction, which will interfere with transesterification. The alternatives to this are:
    - Preprocessing to eliminate the free fatty acids. This step can incur additional cost, but the cost may be offset by increasing the overall useful yield. It proved successful in the production of biodiesel from the high free fatty acid yellow grease.
    - Separation by either physical or chemical means. Again, this can increase cost unless the fatty acids can be recovered and sold for higher value products.
    - Adequate processing at the end is necessary to remove components considered detrimental to biodiesel. For example, it was felt that additional washing would have reduced the total glycerin that was present in the early samples.
2. Adequate testing is required to ensure completeness of each stage of the process.

In addition, some processors have suggested the overall process could be improved if it were moved from batch to a more continuous mode. However, if the feedstock varies significantly and continuous monitoring is not possible, batch mode may better permit adjusting the process to the feedstock. Both economies of scale and recycling of some components such as catalysts are considered possible.

Alternate Feedstock Production

A number of other organizations have been considering the use of alternate feedstocks for biodiesel / methyl ester production. The focus has been on acidulated soapstocks. Acidulated soapstock is a byproduct created when hexane and other industrial substances are used to extract and refine edible oil from soy, canola, cotton, safflower, and sunflower. Cottonseed processors generate 60-120 million pounds of soapstock annually. It has traditionally been used by fatty acid producers, soap makers, foundries, and animal feed manufacturers. Other uses have recently been studied, e.g., it has been considered for dust suppression on highways, surfactants, and as a biodegradable coating for various materials.

IGT investigated the possibility with Agricultural Utilization Research Institute and the University of Nebraska. However, they were unable to provide any material.

NOPEC, a producer of soy-based biodiesel in Florida, investigated the possibility of processing acidulated soapstock at IGT's request. They were unable to process the feedstock without significant modification to their pilot reactor.

IGT also contacted Mr. Bob Riley of Feed Energy (Des Moines IA), as a potential source of methyl esters produced from acidulated soapstock. In a project with Iowa State, they were able to deliver 98% conversion, in small samples. Batch quantities had been produced that were measured on the order of pints, not gallons. One focus was to produce methyl esters that meet biodiesel specifications, but they were not able to meet those with their initial attempts. The concept was being pursued because of the significantly reduced cost of the feedstock, on the order of 55% that of soybean oil. Although it was originally indicated that there might be enough methyl ester for testing (in exchange for the results), nothing has been delivered.

Recent activities by the USDA (Philadelphia) have looked at processes that utilize biocatalysts to optimize production and improve the economics of enzymatic biodiesel production from fats, oils, and greases. A simple two-step chemical procedure for the converting soapstock into simple esters for use as biodiesel was developed and a patent application has been filed for this technology. Large-scale production of soapstock-based biodiesel was undertaken to evaluate soapstock esters as fuels for diesel engines.

PROPERTIES OF BIODIESELS FROM MULTIPLE FEEDSTOCKS

Fuel Properties Tests

The properties selected for study as part of this effort support feedstock-neutral specifications for biodiesel. Also of interest were the differences in temperature-dependent properties that would affect low-temperature fuel handling as a result of the different feedstocks. The tests that were conducted are shown in Table 2, and are discussed in the following section.

Table 2. Methods For Testing Properties of Biodiesels

ASTM	Title
D 86	Distillation of Petroleum Products
D 93	Flash-Point by Pensky-Martens Closed Cup Tester
D 97	Pour Point of Petroleum Products
D 130	Detection of Copper Corrosion from Petroleum Products by the Copper Strip Tarnish Test
D 240	Heat of Combustion of Liquid Hydrocarbon Fuels by Bomb Calorimeter
D 445	Kinematic Viscosity of Transparent and Opaque Liquids (the Calculation of Dynamic Viscosity)
D 482	Ash from Petroleum Products
D 524	Ramsbottom Carbon Residue of Petroleum Products
D 613	Cetane Number of Diesel Fuel Oil
D 664	Acid Number of Petroleum Products by Potentiometric Titration (for dark, opaque liquids)
D 971	Interfacial Tension of Oil Against Water by the Ring Method
D 1091	Phosphorus in Lubricating Oils and Additives
D 1094	Water Reaction of Aviation Fuels
D 1160	Distillation of Petroleum Products at Reduced Pressure
D 1298	Density, Relative Density (Specific Gravity), or API Gravity of Crude Petroleum and Liquid Petroleum Products by Hydrometer Method
D 1322	Smoke Point of Kerosene and Aviation Turbine Fuel
D 1796	Water and Sediment in Fuel Oils by the Centrifuge Method (Laboratory Procedure)
D 1959	Iodine Value of Drying Oils and Fatty Acids
D 2274	Oxidation Stability of Distillate Fuel Oil (Accelerated Method)
D 2500	Cloud Point of Petroleum Products
D 2622	Sulfur in Petroleum Products by X-Ray Spectrometry
D 2624	Electrical Conductivity of Aviation and Distillate Fuels
D 3231	Phosphorus in Gasoline

Table 2. Methods For Testing Properties of Biodiesels, cont.

ASTM	Title
D 3241	Thermal Oxidation Stability of Aviation Turbine Fuels (JFTOT Procedure)
D 3242	Acidity in Aviation Turbine Fuel (Total Acid Number)
D 4539/ IP 309	Filterability of Diesel Fuels by Low-Temperature Flow Test (LTFT) (Cold Filter Plugging Point)
D 4629	Trace Nitrogen in Liquid Petroleum Hydrocarbons by Syringe/Inlet Oxidative Combustion and Chemiluminescence Detection
D 5191	Vapor Pressure of Petroleum Products (Mini Method)
D 5291	Instrumental Determination of Carbon, Hydrogen, and Nitrogen in Petroleum Products and Lubricants
D 6078	Standard Test Method for Evaluating Lubricity of Diesel Fuels by the Scuffing Load Ball-on-Cylinder Lubricity Evaluator (SLBOCLE)
D 6217	Standard Test Method for Particulate Contamination in Middle Distillate Fuels by Laboratory Filtration
	Christina Planc — determination of glycerides
	Total Fatty Acids
	Carbon, Hydrogen, Nitrogen, Oxygen Analysis
	Gas Chromatographic Analysis of Fatty Acids

Properties of Biodiesels

Throughout the following, values from ASTM D 975 — Standard Specification for Diesel Fuel Oils — are included in the discussion of test results when they are available as a reference for comparison. For some tests, the measured values for a low sulfur 2D diesel is included; these values are from the testing conducted on diesel-biodiesel mixtures covered in the following section.

The majority of the testing was conducted by System Lab Services, a division of Williams Pipe Line Company (Kansas City, KS). Galbraith Laboratories, Inc. (Knoxville, TN) conducted the carbon, hydrogen, nitrogen, oxygen analysis, and Phoenix Chemical Laboratory, Inc. (Chicago, IL) conducted the phosphorus, total fatty acids, fatty acid analysis, and retested electrical conductivity.

ASTM D 86 — Distillation of Petroleum Products (Table 3) produces a picture of the volatility characteristics of the petroleum product being tested. Fuels with similar distillation characteristics indicate that the fuels will have similar automotive equipment performance.

The boiling ranges of the biodiesels are within a fairly limited range, around 600-675°F. It is at the upper end of the range of diesel. This may be related to the increased formation of coke (also indicated by Ramsbottom Carbon Residue, ASTM D 524). The higher boiling range (indicating a greater amount energy required to vaporize the biodiesels), results in a larger soluble organic fraction (SOF) in the emissions of biodiesel-fueled engines; testing by others has shown that these emissions are composed mostly of unburned biodiesel.

This distillation test serves as a indication for the fuel composition (as do other tests), which is normally difficult to obtain for diesel. Biodiesel differs in comparison to diesel because it is a "manufactured" fuel, and can be assessed with fatty acid composition.

Diesel that meets D 975 (Grade 2-D and 2-D Low Sulfur) has a 90% distillation fraction between 282°C and 338°C (540°F–640°F). The test results indicated that all but one of the biodiesels were above the maximum value; one was 1°C below, while the rest went up to 16°C above the maximum. For D 975 to be used with biodiesel, it would need to be amended for biodiesel only, based on these results.

Table 3. Test Results for ASTM D 86 — Distillation of Petroleum Products

Observation Point	**Soy ME**	**Canola ME**	**Lard ME**	**Edible Tallow ME**	**Inedible Tallow ME**	**LFFA Yellow Grease ME**	**HFFA Yellow Grease ME**
IBP, °F	613	600	580	616	611	624	594
10%, °F	643	615	612	634	631	633	623
20%, °F	649	628	616	637	634	635	627
50%, °F	654	636	624	646	639	640	632
90%, °F	669	645	638	665	658	655	647
EP, °F	673	671	670	682	675	665	667
IBP, °C	323	316	304	324	322	329	312
10%, °C	340	324	322	334	333	334	328
20%, °C	343	331	324	336	334	335	331
50%, °C	346	336	329	341	337	338	333
90%, °C	354	341	337	352	348	346	342
EP, °C	356	355	354	361	357	352	353
Recovery, ml	98	99	98.5	98.2	99	99.5	99
Residue, ml	1.1	0.5	0.5	1.8	0.4	0.4	0.7
Loss, ml	0.7	0.5	1	0	0.6	0.1	0.3

ASTM D 93 — Flash-Point by Pensky-Martens Closed Cup Tester (Table 4) measures the lowest temperature at which application of the test flame causes the vapor above the sample to ignite. It is used to assess the overall flammability hazard of a material. Specifically, flash point is used in safety regulations to define "flammable" and "combustible" materials. Higher values indicate materials that are less likely to ignite accidentally. A typical value for Number 2 diesel is 70°C, which is considered safe under normal conditions; D 975 requires a minimum of 52°C (126°F). The biodiesels would be considered significantly safer with temperatures between 128°C and 167°C.

Table 4. Test Results for ASTM D 93 — Flash-Point by Pensky-Martens Closed Cup Tester

	Soy ME	Canola ME	Lard ME	Edible Tallow ME	Inedible Tallow ME	LFFA Yellow Grease ME	HFFA Yellow Grease ME
Flash Point, °F	333	326	263	344	276	320	297
Flash Point, °C	167	163	128	173	136	160	147

ASTM D 97 — Pour Point of Petroleum Products (Table 5) measures the lowest temperature at which the oil is observed to flow. It is important because this defines the lowest temperature at which the fuel can still be moved, before it has gelled. Fuels with high pour points are more difficult to use in areas with lower temperatures because the fuel must be kept warm by some method, e.g., electric heaters with insulated tanks. All the biodiesels have significantly higher pour points compared to diesel; over 20°C higher or more. The animal and yellow grease feedstocks resulted in significantly higher values due to the higher proportion of saturated fatty acids.

Table 5. Test Results for ASTM D 97 — Pour Point of Petroleum Products

	Soy ME	Canola ME	Lard ME	Edible Tallow ME	Inedible Tallow ME	LFFA Yellow Grease ME	HFFA Yellow Grease ME	2D Diesel
Pour Pt, °F	30	25	52	56	46	54	46	-17
Pour Pt, °C	-1	-4	11	13	8	12	8	-27

ASTM D 130 — Detection of Copper Corrosion from Petroleum Products by the Copper Strip Tarnish Test (Table 6) measures the degree to which the fuel can have a corroding effect on various metals. The values obtained with each of the biodiesels are the lowest level of corrosiveness, and indicate that corrosion would not be considered a problem (in lieu of any specific information). The value of 1A is the lowest indication on the defined scale for this test. It should be noted that high levels of free fatty acids or residual levels of acids may results in lower values for this test. Because of this, tests like D 664 — Acid Number should be used to ensure that the biodiesel does not have those components which would mask the true values of this test. D°975 permits a maximum corrosion rating of 3; biodiesel easily meets this diesel requirement.

Table 6. Test Results for ASTM D 130 — Detection of Copper Corrosion from Petroleum Products by the Copper Strip Tarnish Test

Soy ME	Canola ME	Lard ME	Edible Tallow ME	Inedible Tallow ME	LFFA Yellow Grease ME	HFFA Yellow Grease ME
1A	1A	1A	1A	1A	1A	1A

ASTM D 240 — Heat of Combustion of Liquid Hydrocarbon Fuels by Bomb Calorimeter (Table 7) is a measure of the energy available from the fuel. The heat of combustion for Number 2 diesel is around 18,600 Btu/lb; each of the biodiesels vary from 0.6% below to 0.5% above the average (17,161 Btu/lb), which is lower than the diesel by 7.7%. Less energy would be carried in weight-constrained applications. When combined with the specific gravity, the lower heat of combustion results in a lower volumetric fuel efficiency (126,000 Btu/gal vs. 132,000 Btu/gal).

Table 7. Test Results for ASTM D 240 — Heat of Combustion of Liquid Hydrocarbon Fuels by Bomb Calorimeter, Btu/lb

Soy ME	Canola ME	Lard ME	Edible Tallow ME	Inedible Tallow ME	LFFA Yellow Grease ME	HFFA Yellow Grease ME
17153	17241	17165	17144	17061	17215	17154

ASTM D 445 — Kinematic Viscosity of Transparent and Opaque Liquids (Table 8) measures the flow resistance of the fuel, e.g., the time for a volume of liquid to flow under gravity through a calibrated glass capillary viscometer. Viscosity is important to diesels and biodiesels because it impacts the operation of components such as the fuel pump. Higher viscosity interferes with injector operation, resulting in poorer atomization of the fuel spray, and has been associated with increased engine deposits. If engines are expected to use higher fractions of biodiesel (instead of the 20% in B-20), they would benefit from redesigned injectors that would accommodate the higher viscosity to improve spray patterns and atomization. All the biodiesels have viscosities significantly higher than diesel. Because of the high values, biodiesels would be expected to have problems in this area on diesel engines, if measures (such as blending with diesel) are not taken. Acceptable values in D 975 are from a minimum of 1.9 to a maximum of 4.1; biodiesels would not be able to meet these requirements. The significantly higher value of the Low Free Fatty Acid Yellow Grease Biodiesel is probably a result of the problems in processing, and would not be expected in general. This is also indicated because of the lower value of the High Free Fatty Acid Yellow Grease Biodiesel.

Table 8. Test Results for D 445 — Kinematic Viscosity of Transparent and Opaque Liquids

	Soy ME	Canola ME	Lard ME	Edible Tallow ME	Inedible Tallow ME	LFFA Yellow Grease ME	HFFA Yellow Grease ME	2D Diesel
Visc, cSt	4.546	4.63	4.85	4.908	4.93	5.62	4.66	2.45

ASTM D 482 — Ash from Petroleum Products (Table 9) measures the amount of ash left after a sample is burned. The presence of ash may indicate undesirable impurities or contaminants. As such, it provides one measure of the suitability of a product for a given application. Both the low and high free fatty acid yellow grease methyl esters produced a significant amount of ash. Specifically for use with biodiesels, this can indicate the presence of processing catalysts remaining in the fuel, possibly indicating that additional cleanup was needed. The maximum acceptable value for diesel meeting D 975 requirements is 0.01%, which should be easily met with most of the biodiesels. The higher values for both the yellow grease biodiesels (which were produced by two different groups) may indicate that yellow grease processing needs are greater, or that greater ash is simply inherent in that biodiesel.

Table 9. D 482 — Ash from Petroleum Products

	Soy ME	Canola ME	Lard ME	Edible Tallow ME	Inedible Tallow ME	LFFA Yellow Grease ME	HFFA Yellow Grease ME
Ash, %	0	0.003	0	0.001	0	0.01	0.04

ASTM D 524 — Ramsbottom Carbon Residue of Petroleum Products (Table 10) determines the amount of carbon residue left after evaporation and pyrolysis of an oil, indicating its relative propensity to form coke. It is a potential indicator of the likelihood that a fuel would form deposits from carbon in an engine's combustion chamber. However, experience with additives has shown that this is not an absolute indicator. Specifically, some detergent additives contribute to ash formation but generally reduce the tendency of the oil to form deposits. The tendency of biodiesels to act as a solvents (resulting in plugging of fuel filters with residue when first introduced to a fuel system previously run on diesel) may be similar to detergent additives. However, use of biodiesels has been associated in the past with forming deposits on engine parts. These results indicate that the canola, animal, and yellow grease biodiesels are more likely to form deposits than the soy biodiesel. Given that deposits have occurred with soy biodiesel, it would be expected that engines using the others would be more likely to form deposits. (The value for the edible tallow methyl ester could not be determined by Williams Lab, for unspecified reasons.) The D 975 maximum of 0.35% can be easily met by the biodiesels.

Table 10. Test Results for D 524 — Ramsbottom Carbon Residue of Petroleum Products

	Soy ME	Canola ME	Lard ME	Edible Tallow ME	Inedible Tallow ME	LFFA Yellow Grease ME	HFFA Yellow Grease ME
Carbon Residue, %	0.01	0.04	0.04	N/A	0.08	0.04	0.05

ASTM D 613 — Cetane Number of Diesel Fuel Oil (Table 11) is a measure of the fuel's ignition delay. Higher Cetane numbers indicate shorter times between the injection of the fuel and its ignition. Higher numbers have been associated with reduced engine roughness and with lower starting temperatures for engines. 2D diesel's Cetane numbers are in the range of 40-50; the biodiesels all have higher values, so they would tend to improve operation of the engine with respect to pure diesel (based on this value alone). The inedible tallow's value is higher than the others, but may be as low as it is because of early production problems. Table 11a contains the results from Williams Lab, while Table 11b contains the results from CSM. There is no clear pattern between the results, or a reasonable explanation for the differences; however, the results for soy ME from CSM are more in-line with expected values. The minimum acceptable Cetane of 40 from D 975 is met by the biodiesels.

Table 11a. Test Results for ASTM D 613 — Cetane Number of Diesel Fuel Oil
(results from WIlliams Lab)

	Soy ME	Canola ME	Lard ME	Edible Tallow ME	Inedible Tallow ME	LFFA Yellow Grease ME	HFFA Yellow Grease ME	2D Diesel
Cetane No.	59	53.9	N/A	64.8	54.3	52.2	53.2	47

Table 11b. Test Results for ASTM D 613 — Cetane Number of Diesel Fuel Oil
(results from CSM)

	Soy ME	Canola ME	Lard ME	Edible Tallow ME	Inedible Tallow ME	LFFA Yellow Grease ME	HFFA Yellow Grease ME
Cetane No.	47.2	55	63.6	62.9	61.7	57.8	52.9

ASTM D 664 — Acid Number of Petroleum Products by Potentiometric Titration (for dark, opaque liquids) (Table 12) determines the acidic or basic constituents in petroleum products and lubricants. For biodiesels, the acid number is an indicator of the quality of the product. Specifically, it detects the presence of any unreacted fatty acids still in the fuel, or of any acids that were used in processing. This is also an indication of the condition of the stability of the fuel, because the acid number increases as the fuel ages.

Table 12. Test Results for ASTM D 664 — Acid Number of
Petroleum Products by Potentiometric Titration

	Soy ME	Canola ME	Lard ME	Edible Tallow ME	Inedible Tallow ME	LFFA Yellow Grease ME	HFFA Yellow Grease ME
Acid No., mg KOH/g	0.32	0.13	0.76	0.32	0.44	0.41	0.2

ASTM D 971 — Interfacial Tension of Oil Against Water by the Ring Method (Table 13) covers the measurement of the interfacial tension of mineral oils against water, under nonequilibrium conditions. This has been shown to reliably indicate the presence of hydrophilic compounds. The missing readings could not be obtained by Williams Lab due to the "consistency of the samples".

Table 13. Test Results for ASTM D 971 — Interfacial Tension
of Oil Against Water by the Ring Method

Soy ME	Canola ME	Lard ME	Edible Tallow ME	Inedible Tallow ME	LFFA Yellow Grease ME	HFFA Yellow Grease ME
11.32	15.52	12.19	31.74	N/A	N/A	N/A

ASTM D 1091 — Standard Test Methods for Phosphorus in Lubricating Oils and Additives (Table 14) covers the determination of phosphorus in unused lubricating oils, lubricating oil additives, and their concentrates. The methods that determine the amount of phosphorus are independent of the type of phosphorus compounds present, because all are converted to orthophosphate ions during analysis. In all cases, the amount of phosphorus present was below detectable limits of 0.02 wt %. The other phosphorus test conducted, D 3231, confirmed the low values that were identified by this test.

Table 14. Test Results for ASTM D 1091 — Standard Test Methods for Phosphorus in Lubricating Oils and Additives, weight percent

Soy ME	Canola ME	Lard ME	Edible Tallow ME	Inedible Tallow ME	LFFA Yellow Grease ME	HFFA Yellow Grease ME
< 0.02	< 0.02	< 0.02	< 0.02	< 0.02	< 0.02	< 0.02

ASTM D 1094 — Water Reaction of Aviation Fuels (Table 15) determines the presence of water-miscible components in aviation gasoline and turbine fuels. For biodiesels, it would indicate the presence of relatively large quantities of partially water soluble contaminants such as surfactants. It may indicate incomplete cleanup at the end of production. Nothing was indicated in this test, suggesting that there were no contaminants, either left from the processing or otherwise. (N/D = not detectable)

Table 15. Test Results for ASTM D 1094 — Water Reaction of Aviation Fuels

Soy ME	Canola ME	Lard ME	Edible Tallow ME	Inedible Tallow ME	LFFA Yellow Grease ME	HFFA Yellow Grease ME
N/D	N/D	N/D	N/D	N/D	N/D	N/D

ASTM D 1160 — Distillation of Petroleum Products at Reduced Pressure (Table 16) is an alternative procedure for determining the volatility characteristics of a petroleum product. ASTM acknowledges that these results are not directly comparable to the results from D86 (see above). This procedure accommodates products that decompose when distilled at atmospheric pressure.

Table 16. Test Results for ASTM D 1160 — Distillation of Petroleum Products at Reduced Pressure

	Soy ME	Canola ME	Lard ME	Edible Tallow ME	Inedible Tallow ME	LFFA Yellow Grease ME	HFFA Yellow Grease ME
IBP, °F	658	658	629	628	606	650	616
5%, °F	662	662	648	646	642	664	650
10%, °F	663	663	650	648	648	664	653
20%, °F	665	663	652	652	653	667	656
30%, °F	666	663	653	656	655	667	659
40%, °F	667	664	655	660	659	670	660
50%, °F	669	664	659	662	662	670	660
60%, °F	670	664	661	666	665	672	663
70%, °F	671	666	664	669	667	674	665
80%, °F	674	668	669	674	670	678	668
90%, °F	687	672	672	680	678	687	673
95%, °F	849	685	679	689	695	720	681
EP, °F	883	814	768	793	895	902	832
IBP, °C	348	348	332	331	319	343	324
5%, °C	350	350	342	341	339	351	343
10%, °C	351	351	343	342	342	351	345
20%, °C	352	351	344	344	345	353	347
30%, °C	352	351	345	347	346	353	348
40%, °C	353	351	346	349	348	354	349
50%, °C	354	351	348	350	350	354	349
60%, °C	354	351	349	352	352	356	351
70%, °C	355	352	351	354	353	357	352
80%, °C	357	353	354	357	354	359	353
90%, °C	364	356	356	360	359	364	356
95%, °C	454	363	359	365	368	382	361
EP, °C	473	434	409	423	479	483	444
Recovery, %	97	99	99	98	99	99	99
Residue + Loss, %	3	1	1	0.2	1	1	1

ASTM D 1298 — Density, Relative Density (Specific Gravity), or API Gravity of Crude Petroleum and Liquid Petroleum Products by Hydrometer Method (Table 17) is a measure of the specific gravity of the biodiesels. They are about 3.5% higher than 2D diesel, which was not considered significant by Williams Labs.

Table 17. Test Results for D 1298 — Specific Gravity - by Hydrometer Method

Soy ME	Canola ME	Lard ME	Edible Tallow ME	Inedible Tallow ME	LFFA Yellow Grease ME	HFFA Yellow Grease ME
0.8877	0.8811	0.8762	0.8708	0.8767	0.8789	0.8767

ASTM D 1322 — Smoke Point of Kerosene and Aviation Turbine Fuel (Table 18) measures the maximum flame height that can be achieved without smoking. This test involves soaking a wick in the sample, then burning the wick. Because of the characteristics of the biodiesels, there were problems getting the prescribed wicks to absorb biodiesel to be burned, and once soaked, they did not burn well. (N/A indicates not available). This result may have been expected, because the test had been formulated for lighter fuels.

Table 18. Test Results for ASTM D 1322 — Smoke Point of Kerosene and Aviation Turbine Fuel

Soy ME	Canola ME	Lard ME	Edible Tallow ME	Inedible Tallow ME	LFFA Yellow Grease ME	HFFA Yellow Grease ME
N/A	N/A	N/A	N/A	N/A	N/A	N/A

ASTM D 1796 — Water and Sediment in Fuel Oils by the Centrifuge Method (Laboratory Procedure) (Table 19) determines the amount of water and sediment in fuel oils using a centrifuge. The presence of either water or sediment in biodiesel can indicate incomplete washing or filtering. The relatively large amount in the lard methyl ester indicated problems in processing the lard into methyl ester, in part due to the large amount of sediment in the lard which is normally not an issue for users of lard. Chemol indicated that additional filtering would have eliminated it; in a commercial process, this may incur additional expense compared to the other feedstocks. Although the lard ME was above the acceptable value, in discussions with NREL it was decided to continue to use it.

Related to this is a note from Industrial Uses of Rendered Fats and Oils, Vol. 3 Edible Oil and Fat Products, Products and Application Technology, in Bailey's Industrial Oil and Fat Products, Fifth Ed., ed. Y.H. Hui, 1996, John Wiley and Sons:

> "Some metalworking formulators have noted 'fallout' problems with methyl lardate and methyl tallowate if their products are stored in drums or tanks without agitation, especially in cooler weather. Over time, the saturated fatty acid esters tend to crystallize and settle, leaving a cloudy or opaque substance in the bottom of the storage container. In sample bottles, this settled opaque material can appear to be as much as 50% of the volume of the total sample.
>
> It should be noted that the saturated fractions are a natural portion of the product and that this fallout is a natural characteristic of the product. Some users have requested methyl lardate with lower levels of fallout or no fallout. In an attempt to provide this product, manufacturers have blended certain crystal inhibitors…"

Past experience with biodiesel, including soy biodiesel, has indicated that visible sediments can form, especially if the temperature drops below 45°F; this is also true if the biodiesel is mixed with cold diesel. Heating the fuel above 100°F causes the solids to go completely back into solution. The sediment would be measured by D 1976, so any samples that were subjected to lower temperatures should be heated to dissolve the crystallized "sediment", and any fuel should be kept at higher temperatures to keep “sediment” from forming before testing. One of the possible reasons for the high value for the lard methyl ester was the presence of this sediment.

The current biodiesel standard ASTM PS 121 recommends ASTM D 2709 — Standard Test Method for Water and Sediment in Middle Distillate Fuels by Centrifuge — for detecting water and sediment. This method has been designed specifically for fuels with the viscosities and densities of the various grades of diesels, including biodiesel. D 1796 is intended for higher viscosity fuel oils (per the D 2709 scope).

Table 19. Test Results for ASTM D 1796 — Water and Sediment in Fuel Oils by the Centrifuge Method (Laboratory Procedure)

	Soy ME	**Canola ME**	**Lard ME**	**Edible Tallow ME**	**Inedible Tallow ME**	**LFFA Yellow Grease ME**	**HFFA Yellow Grease ME**
Sediment, %	0	0	0.6	0.05	0	0	0.03

ASTM D 1959 — Iodine Value of Drying Oils and Fatty Acids (Table 20) measures the amount of iodine required to saturate the olefinic bonds. The iodine value is an indicator of the unsaturation of the fuel, which has been linked with formation of engine deposits and problems in storing the fuel. It has been suggested that values over 115 may be unacceptable; the biodiesels easily meet this requirement. The problems with this test has resulted in ASTM 5550 being selected as the preferred method, after this project was running.

Table 20. Test Results for ASTM D 1959 — Iodine Value of Drying Oils and Fatty Acids

	Soy ME	**Canola ME**	**Lard ME**	**Edible Tallow ME**	**Inedible Tallow ME**	**LFFA Yellow Grease ME**	**HFFA Yellow Grease ME**
Iodine, ppm	2.98	66	53	4.68	17.9	16.8	16.8

ASTM D 2274 — Standard Test Method for Oxidation Stability of Distillate Fuel Oil (Accelerated Method) (Table 21) measures the inherent stability of middle distillate petroleum fuel under accelerated oxidizing conditions. In the method, the fuel is aged at 95°C (203°F) for 16 hours while oxygen is bubbled through the sample at a rate of 3 L/h. The sample is then cooled to room temperature before filtering to obtain the filterable insolubles. Adherent insolubles are removed from the oxidation cell with trisolvent, which is evaporated to determine the quantity. The sum of the two is the total insolubles. Higher numbers indicate that more of the fuel has oxidized, and that it is less stable than a sample that produces a lower value. The biodiesels were inherently less stable than the 2D diesel sample that was used as a base (and the 0% value for the biodiesel/diesel mixtures reported in the next section). This is in agreement with previous experience, specifically that biodiesel does not age well, degrading faster than diesel. It is interesting to note that the iodine value (D 1959) shown above shows similar results, i.e., the canola, lard, and inedible tallow methyl esters have significantly higher numbers than the soy and edible tallow methyl esters. However, the values for the yellow grease methyl esters do not show this pattern.

Table 21. Test Results for ASTM D 2274 — Standard Test Method for Oxidation Stability of Distillate Fuel Oil (Accelerated Method), mg/100 mL

Soy ME	Canola ME	Lard ME	Edible Tallow ME	Inedible Tallow ME	LFFA Yellow Grease ME	HFFA Yellow Grease ME	2D Diesel
16.0	44.9	72.0	8.1	41.0	6.2	8.2	2.3

ASTM D 2500 — Cloud Point of Petroleum Products (Table 22) measures the temperature at which wax crystals or other small crystals of fuel (in the case of biodiesel) begin to form in the liquid causing a haziness as the sample is cooled. It is an indicator of the utility of a petroleum oil for some applications. In the case of biodiesel, the haze is made up of crystallized fuel molecules, specifically crystallized stearic and/or palmitic methyl esters. Using a product below its cloud point may reduce the lubricating properties, and may plug filters. For biodiesel, the settled material may not cause lubrication problems, but the remaining liquid may have lower properties relative to the fully mixed fuel.

In D 975, the cloud point is referenced but not specified by a single number. Instead, it references the tenth percentile of the ambient temperature for the area being used. Use of flow-improvement additives modifies these values. However, using the temperatures shown for the states in the standard (continental U.S.), 18 states are at or below the lowest cloud point in October, while over 41 of the 48 states are at or lower for November through March (Nov-45, Dec-46, Jan-48, Feb-46, Mar-42). This implies that any of the biodiesels would have problems with clouding in most states during these times. Blending with diesel would lessen this problem, as would the use of additives.

Table 22. Test Results for ASTM D 2500 — Cloud Point of Petroleum Products

	Soy ME	Canola ME	Lard ME	Edible Tallow ME	Inedible Tallow ME	LFFA Yellow Grease ME	HFFA Yellow Grease ME	2D Diesel
Cloud Pt, °F	36	27	57	68	73	108	46	0
Cloud Pt, °C	2	-3	14	20	23	42	8	-18

ASTM D2622 — Test Method for Sulfur in Petroleum Products by X-Ray Spectrometry (Table 23) measures the amount of sulfur. As part of the fuel, sulfur is converted to sulfur oxides and sulfuric acid, affecting the emissions of the engine. The lack of detectable sulfur in the biodiesels would result in a reduction in the particulate emissions, in comparison to diesel. (<DL = below detectable limits). Subsequent to this project, ASTM D 5453 — Total Sulfur by Ultraviolet Florescence — has been adopted for testing biodiesel because of its ability to detect lower sulfur levels than D 2622. The maximum value in D 975 for low-sulfur diesel 1D & 2D is 0.05 % mass. Based on the inability to detect at a level lower than the D 975 requirement, which references D 2622 for detecting sulfur, these biodiesels would meet D 975.

Table 23. Test Results for D 2622 — Test Method for Sulfur in Petroleum Products by X-Ray Spectrometry

	Soy ME	Canola ME	Lard ME	Edible Tallow ME	Inedible Tallow ME	LFFA Yellow Grease ME	HFFA Yellow Grease ME
Sulfur, wt%	<DL	<DL	<DL	<DL	<DL	<DL	<DL

ASTM D 2624 — Electrical Conductivity of Aviation and Distillate Fuels (Table 24) indicates the conductivity of a fuel. The conductivity is a measure of the ability of a fuel to dissipate any electrical charge that has been generated during pumping and filtering. No problems are expected from any of the biodiesels. (The retested value was required because of the extremely high value initially obtained.)

Table 24. Test Results for ASTM D 2624 — Electrical Conductivity of Aviation and Distillate Fuels

	Soy ME	Canola ME	Lard ME	Edible Tallow ME	Inedible Tallow ME	LFFA Yellow Grease ME	HFFA Yellow Grease ME
Conductivity, pS/m	181	147	122	809	264	663	>9000
(retested)							470

ASTM D 3231 — Standard Test Method for Phosphorus in Gasoline (Table 25) indicates the amount of phosphorus. This method was developed to be able to detect the levels of phosphorus, because it will damage catalytic converters used in automotive emission control systems. It detects levels of phosphorus lower than the test reported in a previous section (D 1091).

Table 25. Test Results for ASTM D 3231 — Standard Test Method for Phosphorus in Gasoline, ppm

Soy ME	Canola ME	Lard ME	Edible Tallow ME	Inedible Tallow ME	LFFA Yellow Grease ME	HFFA Yellow Grease ME
20.0	13.3	18.4	44.0	19.6	18.7	28.0

ASTM D 3241 — Thermal Oxidation Stability of Aviation Turbine Fuels (JFTOT Procedure) (Table 26) establishes a procedure to rate the tendencies of gas turbine fuels to form deposits within the fuel system, e.g., heated surfaces. When used with biodiesels, it indicates the same tendency when the fuel would come into contact with components such as valves, pistons, and cylinders. Only the edible tallow methyl ester showed up as higher than the minimum rating (tube rating); modifications in processing should be able to improve its rating.

Table 26. Test Results for ASTM D 3241 — Thermal Oxidation Stability of Aviation Turbine Fuels (JFTOT Procedure)

	Soy ME	Canola ME	Lard ME	Edible Tallow ME	Inedible Tallow ME	LFFA Yellow Grease ME	HFFA Yellow Grease ME
Pressure drop	1	0	0	2	0	0	4
Tube rating	1	1	1	4P	1	1	1

ASTM D 3242 — Acidity in Aviation Turbine Fuel (Total Acid Number) (Table 27) describes the procedure used to determine the acidity of aviation turbine fuel. It provides a measure of the acidity in biodiesel, as used here, and may indicate the quality of the biodiesel in a manner similar to D 664. Comparison with the values of the D 664 testing (Table 12) shows a high correlation between the two sets. Although there are differences in values, the relative position of each remained the same. Further study is required to identify whether the differences in value are due to a property of the biodiesels, or are a result of differences in technique or reporting.

Table 27. Test Results for ASTM D 3242 — Acidity in Aviation Turbine Fuel (Total Acid Number)

	Soy ME	Canola ME	Lard ME	Edible Tallow ME	Inedible Tallow ME	LFFA Yellow Grease ME	HFFA Yellow Grease ME
Neutralization Number, mg KOH/g	0.322	0.12	0.76	0.35	0.646	0.492	0.238

ASTM D 4539 — Filterability of Diesel Fuels by Low-Temperature Flow Test (LTFT) (Cold Filter Plugging Point), also known as IP 309, (Table 28) identifies the temperature at which wax or other crystals (first observed at the Cloud Point) can stop the flow of fuel by plugging the engine's fuel filter. The biodiesels have significantly higher temperatures than diesel, a minimum of 16°C above the diesel value. The lard and tallow biodiesels are the highest (as they were for cloud point and pour point).

Table 28. Test Results for D 4539 — Filterability of Diesel Fuels by Low-Temperature Flow Test (LTFT) (Cold Filter Plugging Point)

	Soy ME	Canola ME	Lard ME	Edible Tallow ME	Inedible Tallow ME	LFFA Yellow Grease ME	HFFA Yellow Grease ME	2D Diesel
CFPP, °F	28	24	52	58	50	52	34	-4
CFPP, °C	-2	-4	11	14	10	11	1	-18

ASTM D 4629 — Trace Nitrogen in Liquid Petroleum Hydrocarbons by Syringe/Inlet Oxidative Combustion and Chemiluminescence Detection (Table 29) indicates the amount of nitrogen present in the fuel. It could indicate material left from processing, and would be a measure of the quality. The edible tallow was high as might have been expected based on the comments about processing. The reason for the relatively high levels from the high free fatty acid yellow grease could also be processing, or incomplete cleanup, although the other tests did not seem to indicate this.

Table 29. Test Results for ASTM D 4629 — Trace Nitrogen in Liquid Petroleum Hydrocarbons by Syringe/Inlet Oxidative Combustion and Chemiluminescence Detection

	Soy ME	**Canola ME**	**Lard ME**	**Edible Tallow ME**	**Inedible Tallow ME**	**LFFA Yellow Grease ME**	**HFFA Yellow Grease ME**
Total Nitrogen, ppm	3	0	3	77	5	4	48

ASTM D 5191 — Vapor Pressure of Petroleum Products (Mini Method) (Table 30) is a measurement of the vapor pressure of the petroleum. The test results indicated that none of the biodiesels had a measurable vapor pressure at room temperature. This is in agreement with what was observed with the two distillation tests, D 86 and D 1160, and with the flash point test, D 93. The high temperatures recorded for those tests indicated that essentially no vapor would be expected at lower temperatures. (N/D = no vapor pressure detected)

Table 30. Test Results for ASTM D 5191 — Vapor Pressure of Petroleum Products (Mini Method)

Soy ME	**Canola ME**	**Lard ME**	**Edible Tallow ME**	**Inedible Tallow ME**	**LFFA Yellow Grease ME**	**HFFA Yellow Grease ME**
N/D	N/D	N/D	N/D	N/D	N/D	N/D

ASTM D 5291 — Instrumental Determination of Carbon, Hydrogen, and Nitrogen in Petroleum Products and Lubricants (Table 31) results in a breakdown of the carbon, hydrogen, and nitrogen in a fuel sample. Because of the small amounts of nitrogen in the biodiesels, as shown in D 4629 testing, no values were reported for this test. Comparing the carbon and hydrogen shows that the vegetable oil-derived methyl esters (soy and canola) are less hydrogenated than the others, which are all about 5% higher. This is in agreement with the cloud point (D 2500), pour point (D 97), and cold filter plugging point (D 4539), because more hydrogenated oils have higher melting points.

Table 31. Test Results for ASTM D 5291 — Instrumental Determination of Carbon, Hydrogen, and Nitrogen in Petroleum Products and Lubricants

	Soy ME	Canola ME	Lard ME	Edible Tallow ME	Inedible Tallow ME	LFFA Yellow Grease ME	HFFA Yellow Grease ME
Carbon, wt%	77.95	77.68	77.36	77.07	75.88	76.2	76.03
Hydrogen, wt%	11.98	12.25	12.50	12.50	12.69	12.46	12.50

ASTM D 6078 — Standard Test Method for Evaluating Lubricity of Diesel Fuels by the Scuffing Load Ball-on-Cylinder Lubricity Evaluator (SLBOCLE) (Table 32) is an indication of the lubricating quality of the fuel. It is reported as the weight that can be maintained on a lubricating film in a SLBOCLE test rig Higher lubricity had been linked with less wear, resulting in longer engine component life. In all but one case, the biodiesels essentially doubled the weight that could be maintained. This supports the benefit claims of biodiesel advocates in improving the life of diesel engines. The soy methyl ester is almost double the recommended minimum of 3100 by the Engine Manufacturers Association, and may not be higher only for this batch.

Table 32. Test Results for ASTM D 6078 — Standard Test Method for Evaluating Lubricity of Diesel Fuels by the Scuffing Load Ball-on-Cylinder Lubricity Evaluator (SLBOCLE)

	Soy ME	Canola ME	Lard ME	Edible Tallow ME	Inedible Tallow ME	LFFA Yellow Grease ME	HFFA Yellow Grease ME	2D Diesel
wt, g	6050	>7000	>7000	>7000	>7000	>7000	>7000	3600

ASTM D 6217 — Standard Test Method for Particulate Contamination in Middle Distillate Fuels by Laboratory Filtration (Table 33) indicates the amount of contaminants in fuels by weighing the residue left after filtering the product. The original test for particulates planned for biodiesel was D 2276. Then D 5452 had been established from the laboratory portion of D 2276 as a separate standard. However, the thickness of the biodiesel resulted in extremely long filtration times, so the test was switched to D 6217, which had been established as the middle distillate equivalent. D 6217 is similar to D 1796, indicating contaminants in the fuel, but could be significantly different in value because this test ignores the presence of water. Because of the sensitivity, it is more likely to detect particulates in very small quantities than D 1796. Alternatively, biodiesel has been known to dissolve test filters, and so may have produced the results shown, though nothing was mentioned by the lab.

Table 33. Test Results for ASTM D 6217 — Standard Test Method for Particulate Contamination in Middle Distillate Fuels by Laboratory Filtration

	Soy ME	Canola ME	Lard ME	Edible Tallow ME	Inedible Tallow ME	LFFA Yellow Grease ME	HFFA Yellow Grease ME
mg/L	2.56	11	789	74	1,154	N/A	0

The ***Christina Planc method*** is a more sensitive test than other techniques to detect the presence of glycerides (Table 34). It had been adopted by the biodiesel industry as a method of determining the quality of the biodiesel. The presence of unexpectedly large amounts of triglycerides after transesterification would indicate that the process had not been complete. As noted in the previous section, it also can be used to identify when further cleanup of the methyl ester is required. The high free fatty acid yellow grease methyl ester shows either the most conversion or the greatest cleanup, and is probably a result of the second supplier's process more than an inherent characteristic. The reason for the higher values for the soy and low free fatty acid yellow grease methyl esters is unknown; the results of the other tests do not necessarily correlate with these.

Table 34. Test Results for Christina Planc method

	Soy ME	Canola ME	Lard ME	Edible Tallow ME	Inedible Tallow ME	LFFA Yellow Grease ME	HFFA Yellow Grease ME
Free Glycerin	0.001	0.001	0	0	0	0	0.004
Monoglycerides, wt%	0.87	0.738	0.563	0.320	0.572	0.856	0.250
Diglyrcerides, wt%	1.358	0.02	0.093	0.120	0.070	0.233	0.076
Triglycerides, wt%	3.542	0.01	0.005	0.014	0	0	0
Total Glycerin, wt%	0.798	0.196	0.16	0.102	0.159	0.256	0.080

Total Fatty Acids (Table 35) is the analysis of the amount of fatty acids present in the biodiesel. It is an indication of the conversion efficiency of the original feedstock; these were considered good by Williams Labs.

Table 35. Total Fatty Acids, %

Soy ME	Canola ME	Lard ME	Edible Tallow ME	Inedible Tallow ME	LFFA Yellow Grease ME	HFFA Yellow Grease ME
94.2	92.3	94.4	90.6	90.6	94.3	90.9

Carbon, Hydrogen, Nitrogen, Oxygen Analysis (Table 36) results described below were determined by Organic Elemental Analysis. It is obtained by flash combustion and GC separation. The values are in relative agreement with the values obtained by D 5291, with the added benefit of obtaining oxygen.

Table 36. Carbon, Hydrogen, Nitrogen, Oxygen Analysis

	Soy ME	Canola ME	Lard ME	Edible Tallow ME	Inedible Tallow ME	LFFA Yellow Grease ME	HFFA Yellow Grease ME
Carbon, wt %	76.88	76.84	76.01	75.96	76.40	76.39	76.56
Hydrogen, wt %	11.67	12.03	12.11	12.40	12.46	12.26	12.15
Nitrogen, wt %	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02
Oxygen, wt %	11.40	10.79	11.60	11.31	11.61	11.15	11.72

Gas Chromatographic Analysis of Fatty Acids (Table 37) indicates the composition of each of the biodiesels. The largest fractions of fatty acids for each of the biodiesels is a potential indication of the rest of the properties. The "unknown" entries represent isomers of the fatty acids that the lab did not have a standard reference for and so could not be listed, but are understood to be connected with the following listed fatty acids.

The methyl esters follow some patterns. The soy, canola, and two yellow grease ME are mostly oleic and linoleic acid (C18, one and two double carbon bonds), while the two largest components of the lard, edible tallow, and inedible tallow ME are oleic and palmitic acid (C18, one C:C bond, and C16, saturated).

Table 37. Gas Chromatographic Analysis of Fatty Acids

	Soy ME	**Canola ME**	**Lard ME**	**Edible Tallow ME**	**Inedible Tallow ME**	**LFFA Yellow Grease ME**	**HFFA Yellow Grease ME**
Lauric $C_{12}H_{24}O_2$	(trace)	(trace)	0.12	0.06	0.08	(trace)	(trace)
Unknown	(trace)	(trace)	(trace)	(trace)	0.05	(trace)	(trace)
Myristic $C_{14}H_{28}O_2$	0.09	0.07	1.86	2.91	2.08	(trace)	1.08
Unknown	(trace)	(trace)	0.09	1.57	1.18	(trace)	(trace)
Palmitic $C_{16}H_{32}O_2$	10.54	5.25	24.49	24.34	23.93	11.53	17.3
Unknown	(trace)	(trace)	(trace)	(trace)	(trace)	0.18	(trace)
Palmitoleic $C_{16}H_{30}O_2$	0.13	.22	2.80	3.44	2.79	(trace)	2.23
Unknown	0.14	.23	0.89	3.01	2.34	(trace)	0.97
Stearic $C_{18}H_{36}O_2$	3.75	2.46	14.39	19.10	19.54	13.36	9.54
Unknown	(trace)	(trace)	(trace)	(trace)	(trace)	(trace)	(trace)
Oleic $C_{18}H_{34}O_2$	23.18	58.09	38.32	40.23	38.54	60.67	45.28
Unknown	(trace)	(trace)	0.07	0.24	0.24	12.02	(trace)
Linoleic $C_{18}H_{32}O_2$	48.92	21.79	13.44	2.58	6.43	0.62	14.48
Unknown	7.08	6.63	0.70	0.32	0.52	(trace)	0.57
Linolenic $C_{18}H_{30}O_2$	1.16	0.41	0.33	0.33	0.32	(trace)	1.3
Unknown	1.47	(trace)	(trace)	(trace)	(trace)	(trace)	(trace)
Arachidic $C_{20}H_{40}O_2$	0.24	1.04	0.45	0.29	0.34	0.41	1.06
Unknown	0.39	0.12	0.10	0.51	0.18	(trace)	(trace)
cis-eicosanoic $C_{20}H_{38}O_2$	0.32	1.57	0.67	0.51	0.46	0.21	1.33
Unknown	1.76	0.80	0.86	0.36	0.62	0.49	2.58
Behenic $C_{22}H_{44}O_2$	0.12	0.37	0.18	0.05	0.06	0.32	0.75
Unknown	0.10	(trace)	(trace)	(trace)	(trace)	(trace)	1.54
Erucic $C_{22}H_{42}O_2$	0.08	0.37	0.06	0.09	0.06	(trace)	(trace)
Unknown	0.52	0.42	0.19	0.05	0.25	(trace)	(trace)
Nervonic $C_{24}H_{46}O_2$	(trace)	0.18	(trace)	0.00	(trace)	0.17	(trace)

EVALUATION OF BIODIESEL SPECIFICATION

The specifications for acceptable biodiesels were to be reviewed as part of this project, based on the results of the comprehensive testing on the samples. The specifications are shown in Table 38.

Table 38. Biodiesel Specifications (July 1996)
(For pure mono alkyl esters of long chain fatty acids
derived from renewable lipid feedstocks)

Property	ASTM Method	Limits	Units
Flash Point	D93	100.0 min.	°C
Water & Sediment	D1796	0.050 max.	vol. %
Carbon Residue, 100% sample	D524[1]	0.050 max.	wt. %
Sulfated Ash	D874	0.020 max.	wt. %
Kinematic Viscosity, 40°C	D445	1.9 — 6.5	mm^2/sec (cSt)
Sulfur	D2622	0.050 max.	wt. %
Cetane	D613	40 min.	
Cloud Point	D2500	By Customer	°C
Copper Strip Corrosion	D130	No. 3b max.	
Acid Number	D664	0.80 max.	mg KOH/g
Free Glycerin	GC[2]	0.020 max.	wt. %
Total Glycerin	GG[2]	0.240 max.	wt. %

[1] or equivalent ASTM testing method.
[2] Austrian (Christina Planc) update of USDA test method.

The first step would be identifying tests that may not be needed.

- Flash point is an important measure and should remain a part of the specifications, because it ensures the safety of the fuel handlers and provides a measure of the quality of the fuel. A high flash point is one indication that the methyl esters have been properly washed, eliminating any remaining alcohols.

- Water and Sediment is another measure of the quality of the fuel, and should remain in the specification. High values indicate a problem in the cleanup of the fuel, and may cause problems with the flow of the fuel through filters and other components.

- The Ramsbottom Carbon Residue is an indicator of the tendency of a fuel to form engine deposits. Because the other fuel samples had four or more times the value of the soy biodiesel, and soy biodiesel has been linked with engine deposits in the past, even though the maximum is one-seventh the maximum specified by D 975 for diesel, further experience should be gained in a production environment. Experience with a number of different engine and injector types would assist in understanding in the direction the biodiesel maximum needs to be adjusted, if at all.

- Sulfated ash is a measure of the residue from oils and additives. The sulfated ash is the residue remaining after the sample has been carbonized, then treated with sulfuric acid and heated to constant weight. It is typically used to indicate the concentration of known metal-containing additives in new oils, including barium, calcium, magnesium, zinc, potassium, sodium, and tin. ASTM recommends that D 482 should be used instead of D 874 for non-additive lubricating oils; this would be closer to biodiesel. The production of biodiesel uses sodium, potassium, and other metallic catalysts; therefore, this test (or D 482) should remain in the specifications.

- Kinematic viscosity is an important measure of the biodiesel's flow properties. Because high viscosity has been associated with poor atomization of the fuel spray from the injectors and engine deposits, viscosity should remain a part of the specification. Tracking this number ensures that the fuel has been processed to achieve an acceptable viscosity (i.e., as low as possible with the fuel), and that nothing has been left behind from the processing.

- Sulfur is a measure of the sulfur in the fuel. The biodiesels produced as part of this project did not contain detectable amounts of sulfur. However, because some processing methods use sulfur catalysts to utilize high free fatty acid feedstocks, it should be kept in the set.

- Cetane is a measure of the ignition delay of the fuel, and is a good indicator of the quality of the biodiesel. All of the biodiesels were at least 5 greater than the Cetane number of the 2D diesel tested and experience in the industry has shown that biodiesels are generally at the higher end of diesels. It should continue as part of the specifications because of its gauge of quality of the biodiesel.

- Cloud point, as the highest of the three temperatures indicating the potential for flow problems (the other two are cold filter plugging point and pour point), should be maintained in the specifications.

- Copper strip corrosion indicates the corrosiveness of the fuel on various metals. All measurements from the biodiesels indicated the minimum level of corrosiveness. Unless this test also serves as a measure of processing quality on a component that is undetected by others (e.g., flash point, water & sediment, Cetane number), or there is experience in the industry that indicates otherwise, this test is a candidate for removal from the specification.

- Acid number is a measure of the acidic or basic constituents in a fuel. Because it is a good indicator of the quality of the fuel (processing or cleanup), it should remain as part of the specifications.

- Free glycerin and total glycerin (by the Christina Planc method) is a good indicator as to the cleanup of the fuel, or of the degree of completion during the reaction. As such, it should remain in the specifications for biodiesel.

In addition to reviewing the existing specifications, the question of whether any other tests should be included was addressed. In discussions with biodiesel providers and with testing laboratories, and in reviewing the purpose and focus of the complete test suite, the recommended set of specifications should continue to be able to qualify biodiesel acceptance. Other tests provide additional insight into the properties of the fuel, but it is thought that many could be inferred from other results, at least generally. Specifically, there is no test conducted that would serve as an indicator of acceptability better than the existing specifications.

COMPARISON OF BIODIESEL AND DIESEL SPECIFICATIONS

ASTM D 975 specifies acceptable properties of diesel. The biodiesel samples produced for this project met most of the specifications. Their Flash Points were at least 76°C (137°F) above the minimum. Ash for the samples were half or less of the maximum acceptable value, except for the yellow grease biodiesels (which may have been due to processing and could be corrected now that it is known). The Ramsbottom Carbon Residue maximum of 0.35% was met by the biodiesels, and all but one met the biodiesel specification of 0.05%. The biodiesels all met the requirement of a Cetane number of 40 by at least 7. Sulfur levels in biodiesel were below detectable limits for the test method specified by D 975.

However, the biodiesels have problems meeting D 975 requirements in three areas. For the biodiesels, the 90% distillation fraction was at or above the maximum temperature specified by D 975. The viscosity of all the biodiesels was above the maximum value of 4.1 centiStokes. Cloud Point is a problem for all of the biodiesels because the temperatures are significantly higher than the tenth-percentile ambient temperature specified by D 975. Because each of these is related to the composition of the biodiesels, D 975 may need to be changed to reflect this.

Table 39 compares the biodiesel specifications with those of D 975. The applicability of these specifications for B20 (20% biodiesel / 80% diesel) can be considered.

Table 39. Comparison of Biodiesel Specifications and ASTM D 975 Specifications

Property	Biodiesel Limits	D 975 Limits	Units
Flash Point	100.0 min.	52.0 min.	°C
Water & Sediment	0.050 max.		vol. %
Carbon Residue, 100% sample	0.050 max.	0.35 max.	wt. %
Sulfated Ash	0.020 max.	0.010 max.	wt. %
Kinematic Viscosity, 40°C	1.9 — 6.5	1.9 — 4.1	$mm^{2/}$sec (cSt)
Sulfur	0.050 max.	0.050 max.	wt. %
Cetane	40 min.	40 min.	
Cloud Point	By Customer	By Customer	°C
90% Distillation Fraction Temp		282 — 338	°C
Copper Strip Corrosion	No. 3b max.		
Acid Number	0.80 max.		mg KOH/g
Free Glycerin	0.020 max.		wt. %
Total Glycerin	0.240 max.		wt. %

In general, the properties of B20 would be expected to be between the pure components, and closer to the pure diesel values than biodiesel values.

- For sulfur and Cetane number, the limits are the same.

- The flash point of B20 could be significantly lower than the biodiesel limit, given that diesel needs to be at least 52°C, compared with the 100°C biodiesel limit. In B20, the diesel components that produce the flash point would be present around the same temperature as pure diesel, indicating that the flash point of B20 would be very close to that of the diesel used in the mixture.

- B20 may not meet the biodiesel carbon residue maximum, because diesel can be 7 times the maximum of biodiesel and still meet the D 975 specification.

- The D 975 limit of 0.01% sulfated ash could be exceeded with B20, because the biodiesel maximum is twice the value of D 975.

- Viscosity of B20 could exceed the D 975 maximum, because the biodiesel maximum is 58% greater than the diesel limit.

- There is no value in the biodiesel specification for the temperature of 90% distillation fraction, but as noted in the discussion for D 86, biodiesel was above the maximum temperature for diesel in D 975. To meet D 975, most of the diesel would have to be vaporized, along with over 50% of the biodiesel at the maximum temperature of 338°C. Since the 50% distillation fraction temperatures were around the maximum temperature, it is possible for B20 to be outside the temperature range from D 975.

Based on this, B20 limits would be expected to be closer to diesel limits, but could exceed the limitations of D 975 in those values for sulfated ash, viscosity, and distillation fraction temperature. B20 could fail on biodiesel specification limits in flash point and carbon residue.

BIODIESEL/DIESEL BLEND TESTING

Testing Methodology

The biodiesels used in the blended testing were the fuels produced for this program: the soy methyl ester, canola methyl ester, pork lard methyl ester, edible and inedible beef tallow methyl esters, low free fatty acid (<4%) yellow grease methyl ester, and high free fatty acid (10%) yellow grease methyl ester.

It was considered important to use a diesel that would represent diesel fuel in general, and that could be utilized in other projects as a common baseline. To meet this requirement, IGT identified a suitable diesel fuel for use as a reference and as the base for mixing with the biodiesels. The reference fuel for this test program was certification diesel fuel obtained from Phillips Petroleum (Lot D434). It is the low-sulfur diesel that is currently used for testing engine emissions, and because of this use, is known in terms of its quality and characteristics. The properties of this fuel are shown in Table 40.

Table 40. Certification Diesel Fuel Lot D-434 Properties

Property	Lot D-434	ASTM Method
API Gravity	36.28	D-287
Viscosity, cS 40°C	2.5	D-445
Net Heating Value, BTU/lb	18456	D-3338
Cetane Number	46.0	D-613
Carbon, wt%	86.6	D-5291
Hydrogen, wt%	13.4	D-5291
Oxygen, wt%	0	D-5291
Sulfur, ppm	300	D-2622
Nitrogen, ppm	--	D-4629
IBP, F	353.9	D-86
T50, F	498.7	D-86
T90, F	583.7	D-86
EP, F	646.4	D-86
Aromatics, vol%	29.2	D-1319
Olefins, vol%	2.0	D-1319
Saturates, vol%	68.8	D-1319

The tests that were selected for the blended fuels were the ones that were expected to vary in a non-intuitive fashion, or that may not be proportional with respect to blend rates. They also are the tests that have the most direct correlation to handling and use with diesel engines. The tests are shown in Table 41.

Table 41. Physical and Chemical Tests of Biodiesel Blends

Property	ASTM Method	Importance
Viscosity, mm^2/s @ 40°C	D 445	Fuel flow resistance; higher viscosity associated with poorer fuel atomization from injectors and increased engine deposits; also impacts energy requirements and wear of fuel pump and injectors;
Pour Point, °C	D 97	Minimum temperature above which fuel can be poured, i.e., is still a liquid and can be pumped; affecting use in cold climates
Cloud Point, °C	D 2500	Temperature at which fuel begins to cloud (i.e., increases in turbidity), indicating wax is beginning to form (potential for plugging)
Cold Filter Plugging Point, °C	D 4539 / IP 309	Temperature at which fuel will plug a fuel filter
Cetane number	D 613	Measure of ignition delay of a compression ignition fuel; higher values indicate shorter ignition lags, fewer deposits, lower starting temperatures, reduced engine roughness
Lubricity	D 6078	Measure of the lubricating quality of the fuel; higher values indicate better lubrication
Oxidation Stability	D 2274	Measure of change in fuel oil quality; indicator of "shelf life" of fuel

The specific fuel blend fractions were selected to complement the tests that had been conducted on the pure biodiesels. They also took into account the range of low-concentration biodiesel blends (in the range of diesel fuel additives) that advocates of biodiesel emphasize are beneficial, and that present little possibility for unusual changes in properties once the biodiesel becomes the dominant component. The fractions of biodiesel that were tested were: 0.25%, 0.50%, 1%, 3%, 5%, 10%, 20%, 35%, and 50%. In addition, the pure diesel fuel was tested to establish the 0% biodiesel point.

The testing was conducted by outside agencies. The D 6078 (Scuffing Load Ball On Cylinder Lubricity Evaluator) tests were conducted by Engineering Test Services, and the other tests were conducted by Williams Lab Services.

Biodiesel/Diesel Blend Test Results

The following tables display the results of the testing on the various biodiesel / diesel blends. The 0% biodiesel concentration is the test result of the pure diesel fuel, and the 100% biodiesel concentration has been carried from the section of the pure biodiesel test results. The discussion of the results is presented in the following section.

Table 42 contains the results of the viscosity testing.

Table 42. Biodiesel Blend Viscosity Test Results

Biodiesel Concentration	SME	CME	LME	ETME	ITME	LYGME	HYGME
0%	2.453	2.453	2.453	2.453	2.453	2.453	2.453
0.25%	2.505	2.461	2.453	2.453	2.453	2.453	2.453
0.50%	2.453	2.453	2.453	2.453	2.453	2.476	2.461
1%	2.461	2.461	2.468	2.468	2.476	2.468	2.490
3%	2.392	2.400	2.400	2.487	2.409	2.418	2.400
5%	2.418	2.418	2.444	2.435	2.444	2.452	2.435
10%	2.461	2.496	2.522	2.513	2.522	2.556	2.513
20%	2.743	2.743	2.859	2.894	2.930	3.011	2.876
35%	2.936	2.988	3.145	3.208	3.262	3.387	3.172
50%	3.189	3.308	3.459	3.531	3.593	3.826	3.495
100%	4.546	4.63	4.85	4.908	4.93	5.62	4.66

Figure 1 shows the results of the viscosity tests for each of the biodiesel blends, while Figure 2 shows an expanded view of the 0-10% biodiesel range. Viscosity affects fuel atomization by the injectors, engine deposits (increased viscosities associated with increased deposits), and energy use (higher viscosities require more energy by the fuel pump). Figure 2 shows that, at low concentrations (<10%), there appears to be a moderate level of viscosity reduction between 1% and 5%, returning back to a blend fraction-dependent mix of the two fuels' viscosity by the 10% level. The maximum amount of reduction occurs with the soy methyl ester at 3%, with a total reduction of approximately 2.5%. The fact that this occurs with each of the biodiesels indicates that it is a general artifact, independent of any particular feedstock-derived characteristic. However, because the reduction is minimal and occurs in a small range, it may not be worthwhile to take advantage of it.

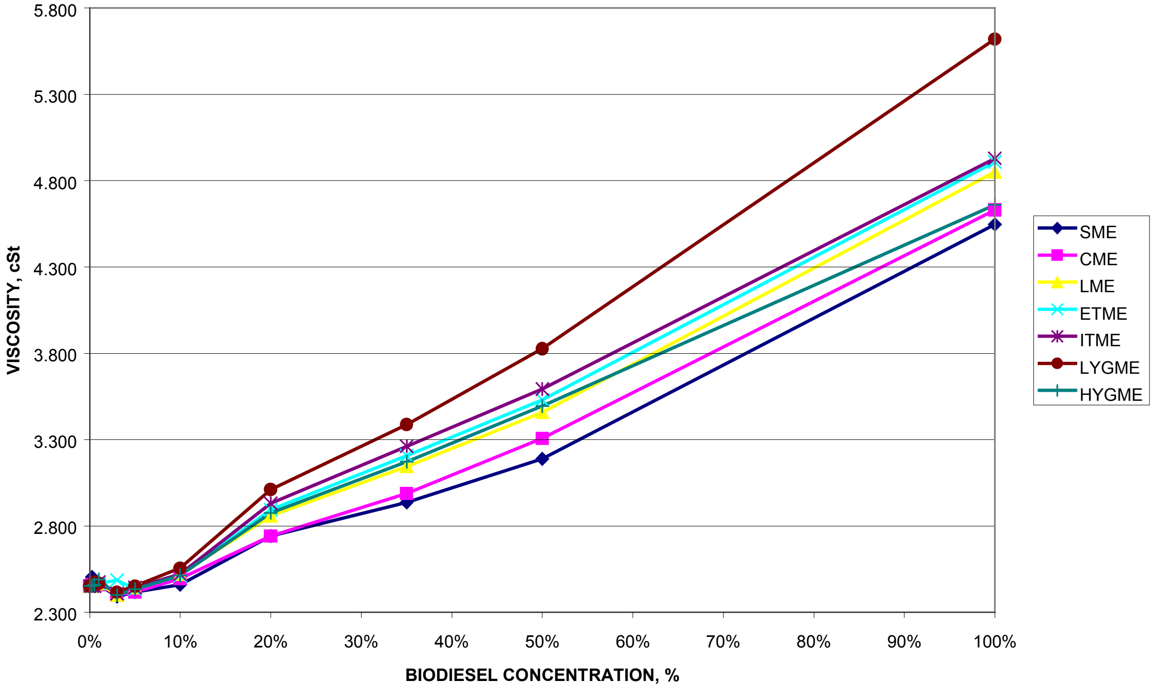

Figure 1. Biodiesel/Diesel Blend Viscosity Test Results

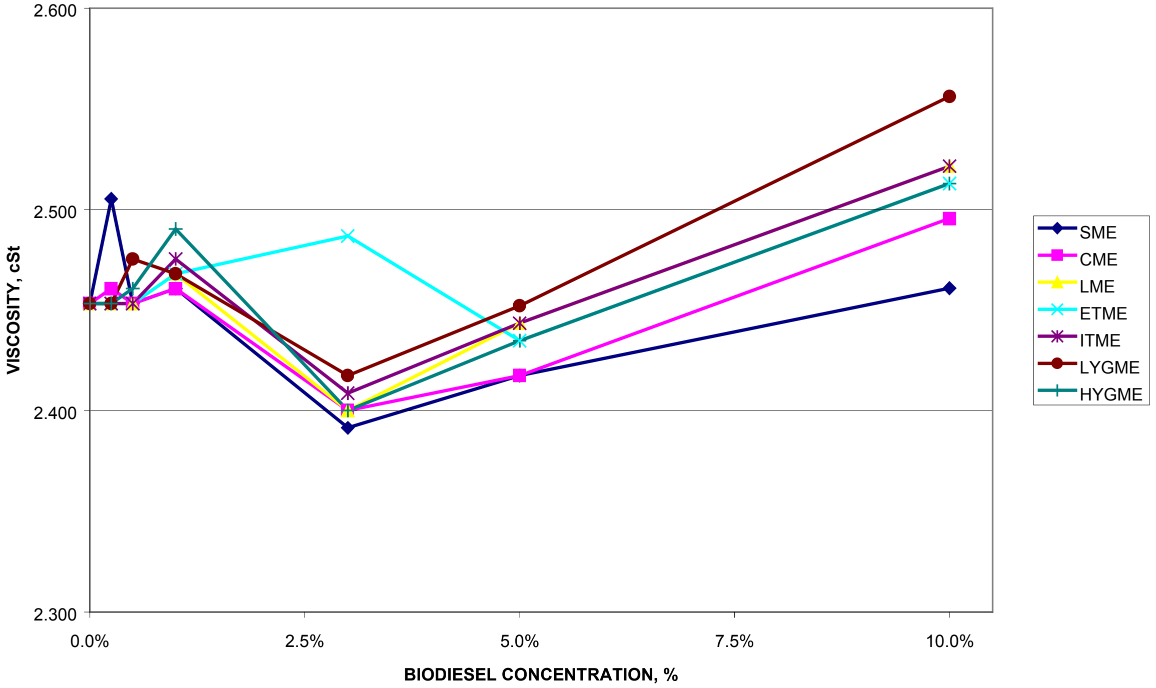

Figure 2. Biodiesel/Diesel Blend Viscosity Test Results (0–10% biodiesel blend range)

Table 43 shows the results from the pour point testing.

Table 43. Biodiesel Blend Pour Point Test Results, °C

Biodiesel Concentration	**SME**	**CME**	**LME**	**ETME**	**ITME**	**LYGME**	**HYGME**
0%	-27	-27	-27	-27	-27	-27	-27
0.25%	-27	-21	-24	-24	-24	-24	-24
0.50%	-27	-24	-24	-24	-24	-24	-24
1%	-24	-24	-24	-21	-24	-24	-24
3%	-24	-24	-21	-21	-21	-21	-21
5%	-21	-21	-18	-18	-15	-18	-18
10%	-18	-21	-15	-12	-12	-18	-18
20%	-18	-18	-9	-9	-9	-9	-12
35%	-15	-18	0	-6	-3	-6	-6
50%	-9	-15	3	3	3	0	-3
100%	-1	-4	11	13	8	12	8

Figure 3 shows the test results for pour point, and Figure 4 shows the expanded view of the 0%-10% biodiesel range. Pour point, the temperature above which the fuel will pour, is important because it directly affects the usability of the fuel in colder climates. The high pour points of many of the biodiesels significantly limit their use in the pure form in cold weather. Even though the blends show much better pour point temperatures at lower concentrations, handling the pure biodiesels before mixing will require insulation and heating of the tanks to ensure that the liquid can be moved prior to mixing. It also affects the way in which mixing occurs, so that an adequate temperature of the biodiesel is maintained until thorough blending can occur. Unlike the viscosity results, the pour point results demonstrate a fairly rapid rise in pour point up to a 10% concentration, at which time the trend is more linear with respect to blend fraction. This indicates that the presence of biodiesel, even at the low concentrations when used as a diesel additive, will increase the pour point of the blend by approximately 5°C. The steps that are seen in Figure 3 are a result of the step size when the temperature is adjusted while conducting the test.

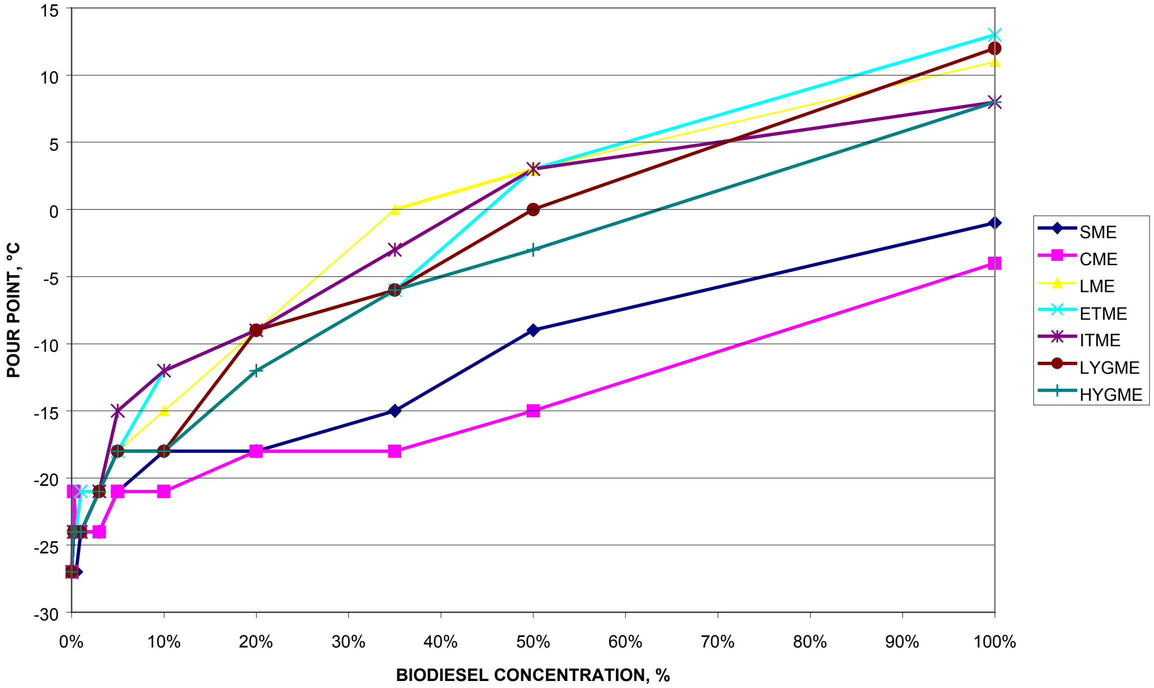

Figure 3. Biodiesel/Diesel Blend Pour Point Test Results

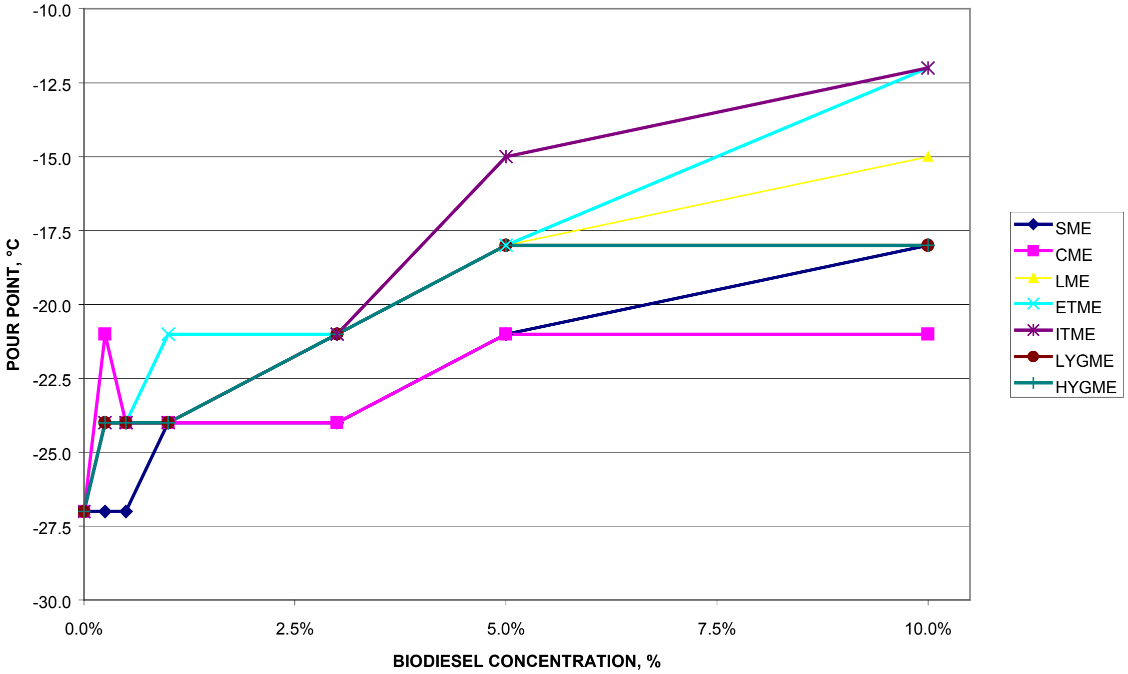

Figure 4. Biodiesel/Diesel Blend Pour Point Test Results (0–10% biodiesel blend range)

Table 44 contains the results of the cloud point testing of the biodiesel blends.

Table 44. Biodiesel Blend Cloud Point Test Results, °C

Biodiesel Concentration	SME	CME	LME	ETME	ITME	LYGME	HYGME
0%	-18	-18	-18	-18	-18	-18	-18
0.25%	-20	-18	-18	-16	-16	-15	-18
0.50%	-17	-18	-17	-16	-17	-14	-15
1%	-16	-18	-17	-15	-17	-16	-15
3%	-16	-17	-16	-13	-14	-16	-15
5%	-16	-17	-15	-12	-13	-16	-14
10%	-15	-17	-14	-9	-10	-13	-13
20%	-14	-15	-3	-2	-6	-6	-8
35%	-9	-12	-3	0	0	5	-6
50%	-9	-10	-2	3	4	13	-3
100%	2	-3	14	20	23	42	8

Figure 5 shows the results of the cloud point testing, and Figure 6 shows an expanded view of the 0%-10% biodiesel range. The cloud point is an important measure because it is the temperature at which components of the fuel begin to crystallize, forming a visible clouding of the liquid. When circulating in the fuel system, the components that produce the clouding can be captured in filters, or cause components to wear due to the solidification of the lubricants. The cloud point exhibits a larger increase at low concentrations of biodiesel, but to a lesser extent than the pour point tests. In general, avoiding complications due to this effect will require measures similar to those for addressing the high pour point. The fluctuations in the 0.25% and 0.5% biodiesel measurements may be more noise.

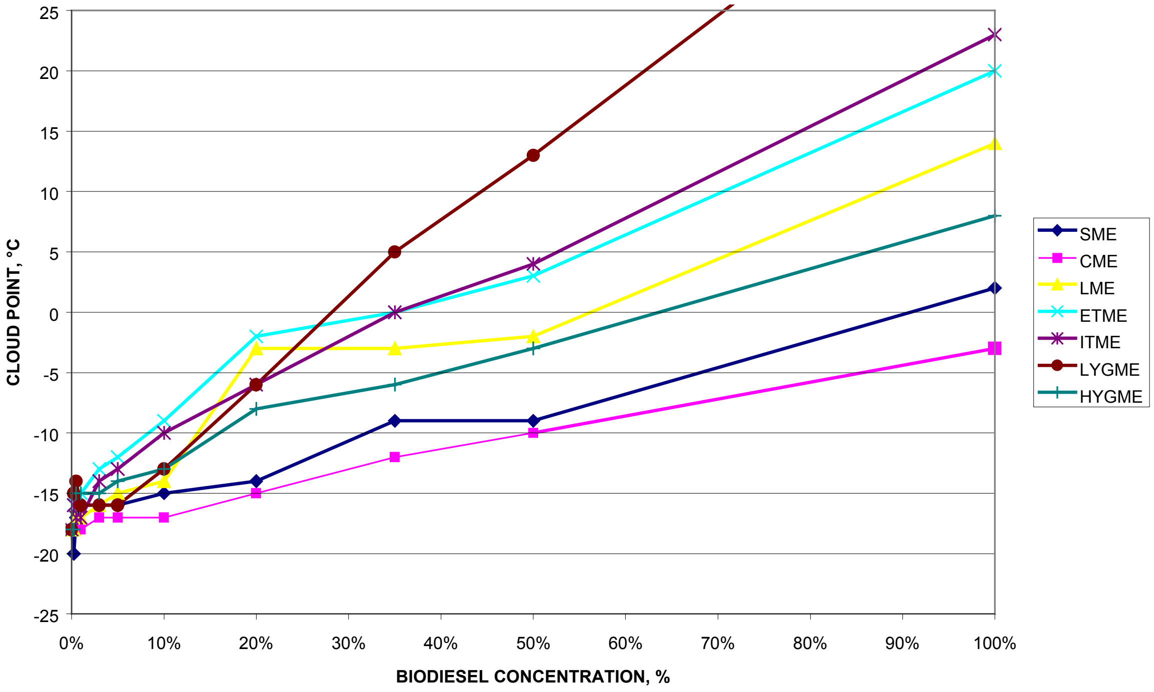

Figure 5. Biodiesel/Diesel Blend Cloud Point Test Results

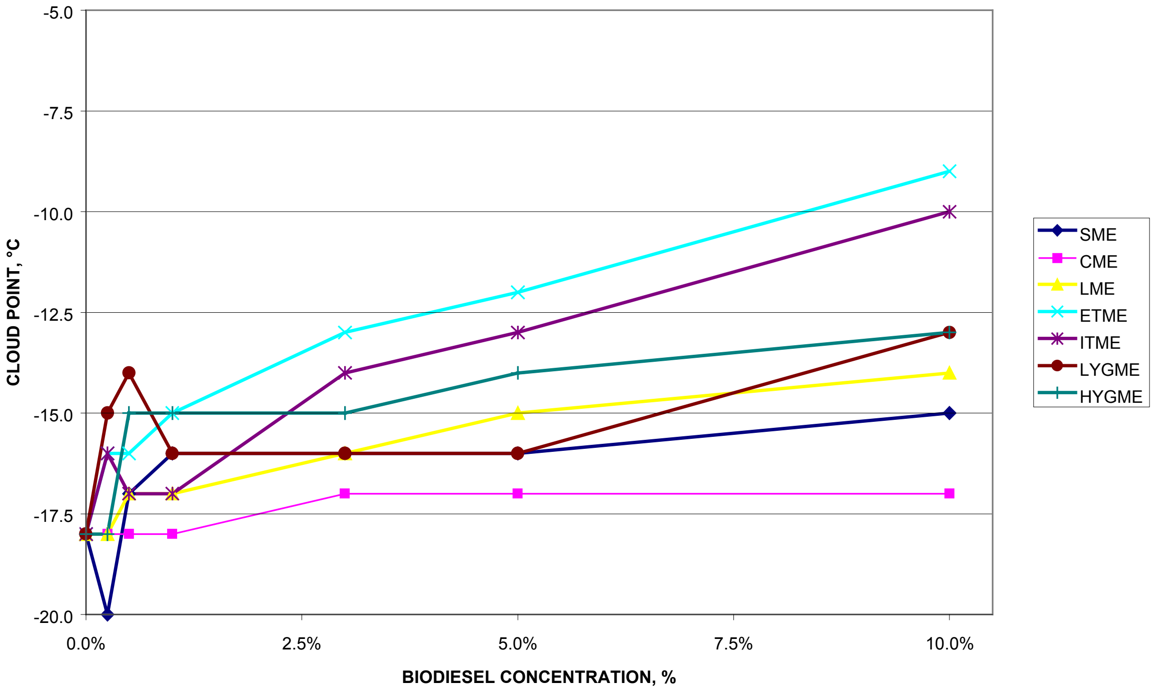

Figure 6. Biodiesel/Diesel Blend Cloud Point Test Results (0–10% biodiesel blend range)

Table 45 holds the testing results for cold filter plugging point.

Table 45. Biodiesel Blend Cold Filter Plugging Point Test Results, °C

Biodiesel Concentration	**SME**	**CME**	**LME**	**ETME**	**ITME**	**LYGME**	**HYGME**
0%	-20	-20	-20	-20	-20	-20	-20
0.25%	-20	-20	-20	-20	-21	-20	-20
0.50%	-20	-20	-20	-20	-20	-20	-21
1%	-21	-20	-20	-20	-20	-19	-20
3%	-19	-18	-19	-19	-21	-18	-20
5%	-19	-18	-18	-17	-18	-17	-19
10%	-18	-18	-17	-14	-15	-14	-18
20%	-17	-18	-8	-3	-11	-1	-14
35%	-17	-17	4	5	-6	7	-12
50%	-17	-16	6	6	-1	9	-6
100%	-2	-4	11	14	10	11	1

Figure 7 shows the results of the cold filter plugging point testing, and Figure 8 shows the expanded 0%-10% biodiesel range. The test measures the temperature at which a cold filter would be plugged when attempting to handle the fuel. The relationship between increase in temperature and biodiesel fraction is more linear than in the pour point or cloud point results for most of the biodiesels at the lower concentrations. However, the pork lard, edible beef tallow, and low free fatty acid yellow grease methyl esters exhibited higher than expected increases in the 20% to 50% concentration range. Since these also had the highest cold filter plugging point measurements, it appears that this characteristic is dominant until low biodiesel concentrations are reached. Conversely, the soy and canola methyl esters show a temperature depression through the same concentration range (20%-50%). These blends were close to the pure diesel temperature (within 5°C) through 50%. The biodiesels significantly varied in their effect on cold filter plugging point, where three inflated, two depressed, and two had a linear effect on cold filter plugging point. Since the two sets of biodiesels from similar sources showed differing actions (tallow and yellow grease biodiesels), it does not appear to be feedstock dependent. It is possible that processing differences caused the effect, especially since processing of the high free fatty acid yellow grease was handled in two steps, unlike the others.

Researchers have investigated the effectiveness of additives to improve flow characteristics[1,2]. Their results indicate that the additives can keep the crystals from combining, which results in lower cold filter plugging points. In addition, the gelling, identified by the pour point, can be lowered by appropriate additives.

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[1] Dunn, R. O., Shockley, M. W., and Bagby, M. O. "Improving the Low-Temperature Properties of Alternative Diesel Fuels: Vegetable Oil-Derived Methyl Esters." Journal of Amer. Oil Chem. Soc., 73 (1996): 1719-1728.

[2] Midwest Biofuels Inc. *Biodiesel Pour Point and Cold Flow Study.* Report to National Soydiesel Development Board, September 30, 1993, St. Louis, MO.

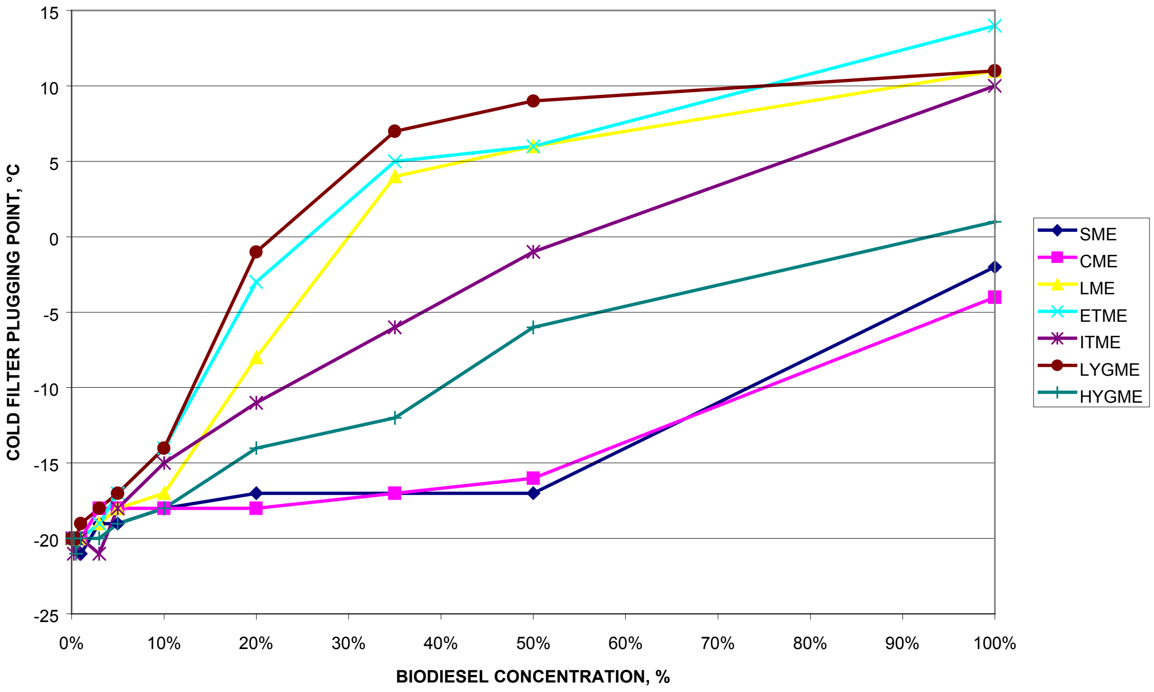

Figure 7. Biodiesel/Diesel Blend Cold Filter Plugging Point Test Results

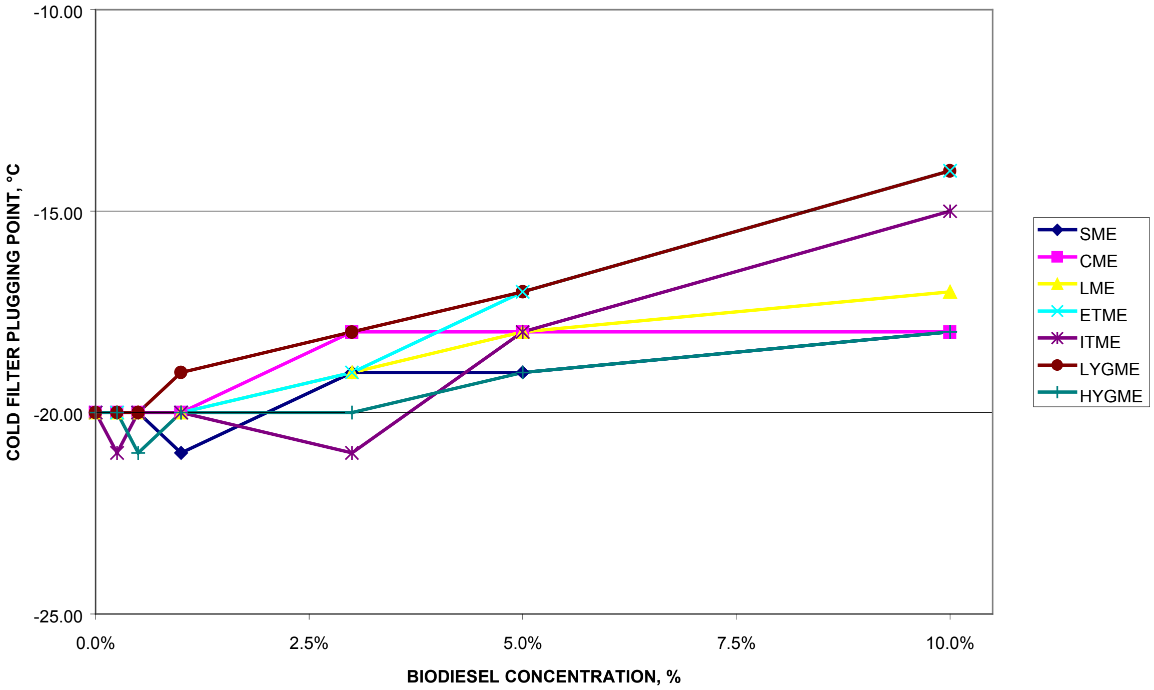

Figure 8. Biodiesel/Diesel Blend Cold Filter Plugging Point Test Results
(0–10% biodiesel blend range)

Table 46 contains the results of the Cetane number testing of the biodiesel blends.

Table 46. Biodiesel Blend Cetane Number Test Results

Biodiesel Concentration	SME	CME	LME	ETME	ITME	LYGME	HYGME
0%	47	47	47	47	47	47	47
0.25%	47.2	47.1	47	47	47.1	46.8	47.1
0.50%	47.1	46.9	47	47.1	47.1	47.1	47
1%	47.2	47.3	47.3	47.1	47.3	47.2	47.2
3%	47.3	47.2	47.4	46.9	47.5	47.3	47.2
5%	47.4	47.6	47.4	47.3	48	47.5	47.7
10%	47.7	47.9	48	47.7	48.4	47.6	47.8
20%	49.2	48.8	47.9	48.4	49.8	48.7	49.3
35%	50.4	49.6	50	49.2	50.9	50	51.4
50%	51.4	50.6	51.2	49.8	51.9	50.8	51.6
100%	59	53.9		64.8	54.3	52.2	53.2

Figure 9 shows the results of the Cetane number testing, and Figure 10 shows the expanded 0%-10% biodiesel range. Higher Cetane numbers indicate higher ignition rates, which tend to reduce carbon and lacquer formation and engine deposits, and decrease engine roughness. It appears to be linear with respect to biodiesel concentration, except for the outlying points for the soy and edible tallow methyl esters.

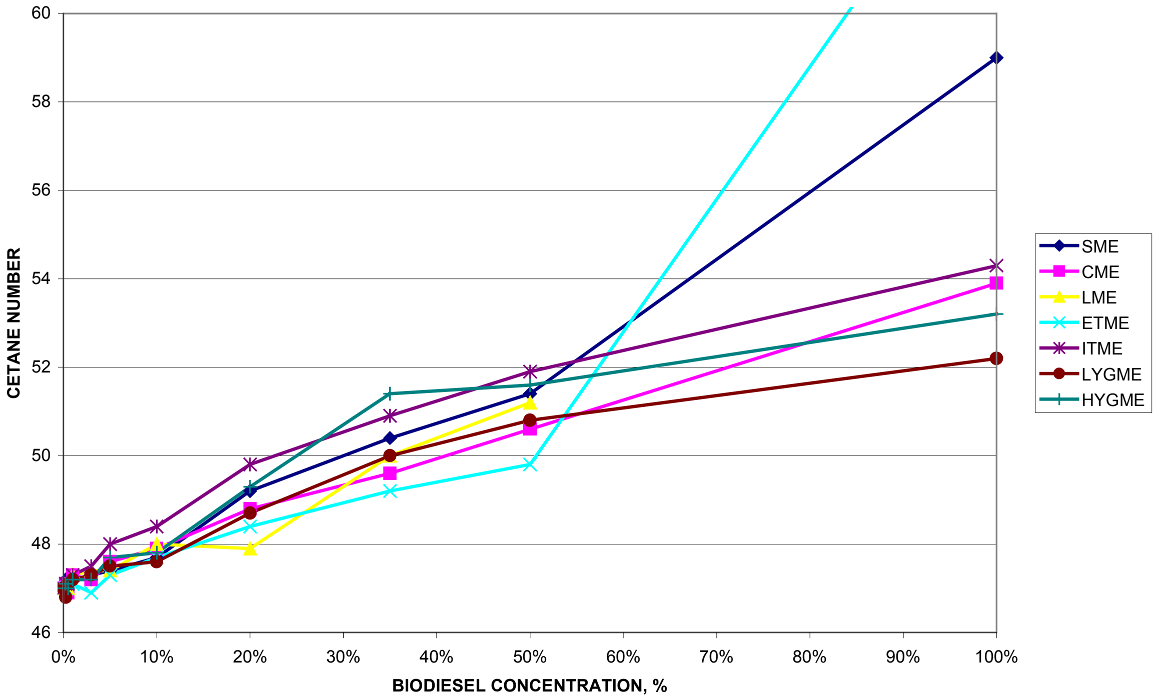

Figure 9. Biodiesel/Diesel Blend Cetane Number Test Results

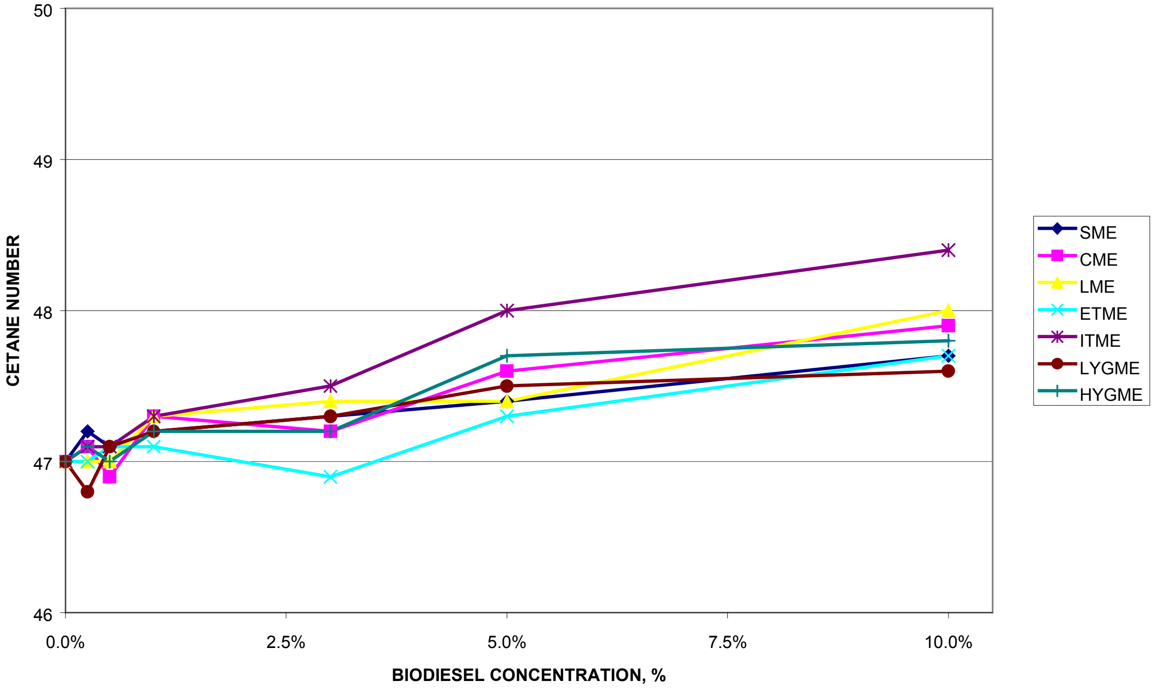

Figure 10. Biodiesel/Diesel Blend Cetane Number Test Results
(0–10% biodiesel blend range)

Table 47 contains the results for scuffing load BOCLE lubricity testing. The high and low reference results are those required to verify the accuracy of the SLBOCLE test.

Table 47. Biodiesel Blend Scuffing Load BOCLE Test Results

Methyl Ester Concentration	**Soy**	**Canola**	**Inedible Tallow**	**Edible Tallow**	**Lard**	**Low FFA Yellow Grease**	**High FFA Yellow Grease**
0%	3600	3600	3600	3600	3600	3600	3600
0.25%	4700	4600	4050	3800	4400	4700	4650
0.50%	4950	4750	4700	4300	4700	5100	4850
1%	5600	4950	5550	4900	5500	5500	5450
3%	5600	5300	5850	5950	6150	6050	5650
5%	5400	5950	6550	6100	6500	6550	5800
10%	6100	6550	>7000	6350	6700	6650	6150
20%	6150	>7000	>7000	6400	>7000	>7000	>7000
35%	5850	>7000	>7000	6500	>7000	>7000	>7000
50%	6000	>7000	>7000	>7000	>7000	>7000	>7000
100%	6050	>7000	>7000	>7000	>7000	>7000	>7000
High Reference Cat 1K	5950	6200	6250	6250	6000	6000	6100
Low Reference Isopar M	1900	2200	2000	2000	2100	1900	2050

Figure 11 shows the results of the scuffing load BOCLE tests, and Figure 12 shows the expanded view. Although the chart indicates that the blends stopped at 7000 grams, they were in fact higher. Due to limitations of the equipment, testing was stopped at 7000 grams. The reference line of 3100 grams indicates the minimum acceptable level for EMA specification of diesel. The test on the reference fuel from Philips was at 3600 grams. Figure 12 highlights the dramatic increase in lubricity with even small amounts of biodiesel present, which is the primary reason that advocates recommend the use of biodiesel as an additive even if it is not being used as a substantial fraction of the fuel, e.g., as B20. In general, the lubricity improved by 10% on average with just 0.25% biodiesel present, and by 30%+ at 0.5%. Most of the blends had reached 50% improvement with 1% biodiesel, and all exceeded 50% at 3%. The first blend to reach the maximum value was the edible beef tallow at 10%, followed by the pork lard, canola, and both yellow grease methyl esters at 20%. Although the soy methyl ester also increased the lubricity of the diesel by over two-thirds, it did not reach the maximum test value that the others encountered. If the biodiesels derived from tallow, lard, or yellow grease can be produced at a cost lower than the soy or canola biodiesels, while retaining their impact on lubricity, they could favorably impact the biodiesel additive market.

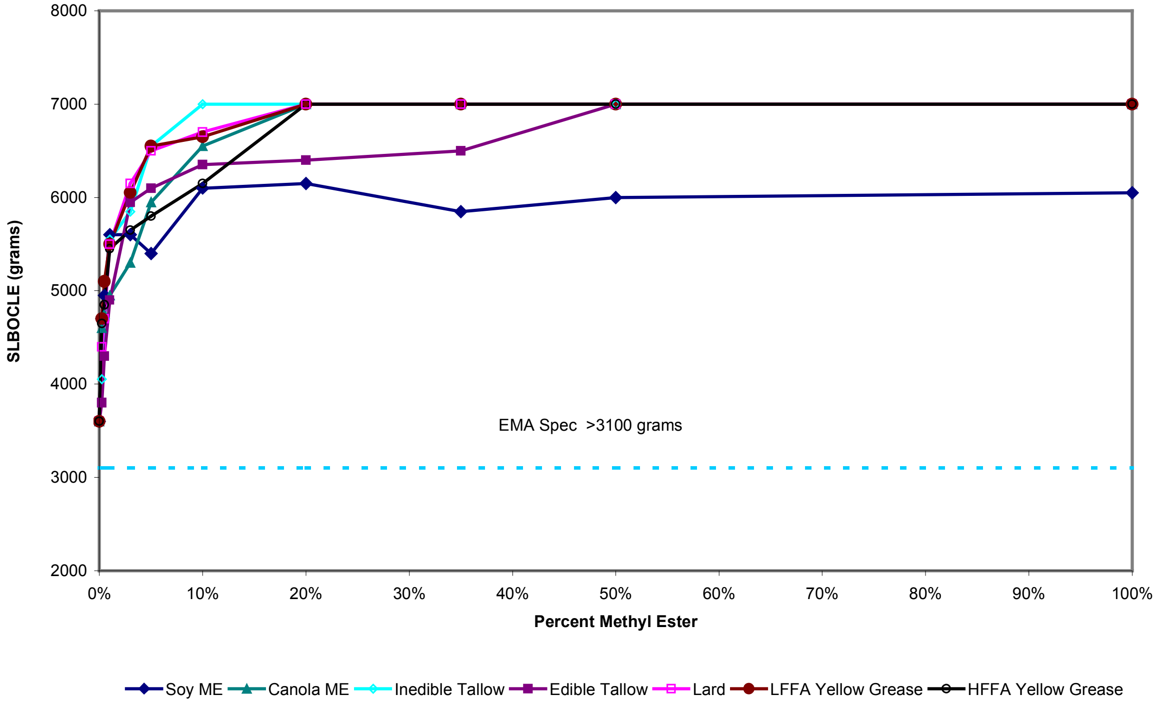

Figure 11. Biodiesel/Diesel Blend SLBOCLE Test Results

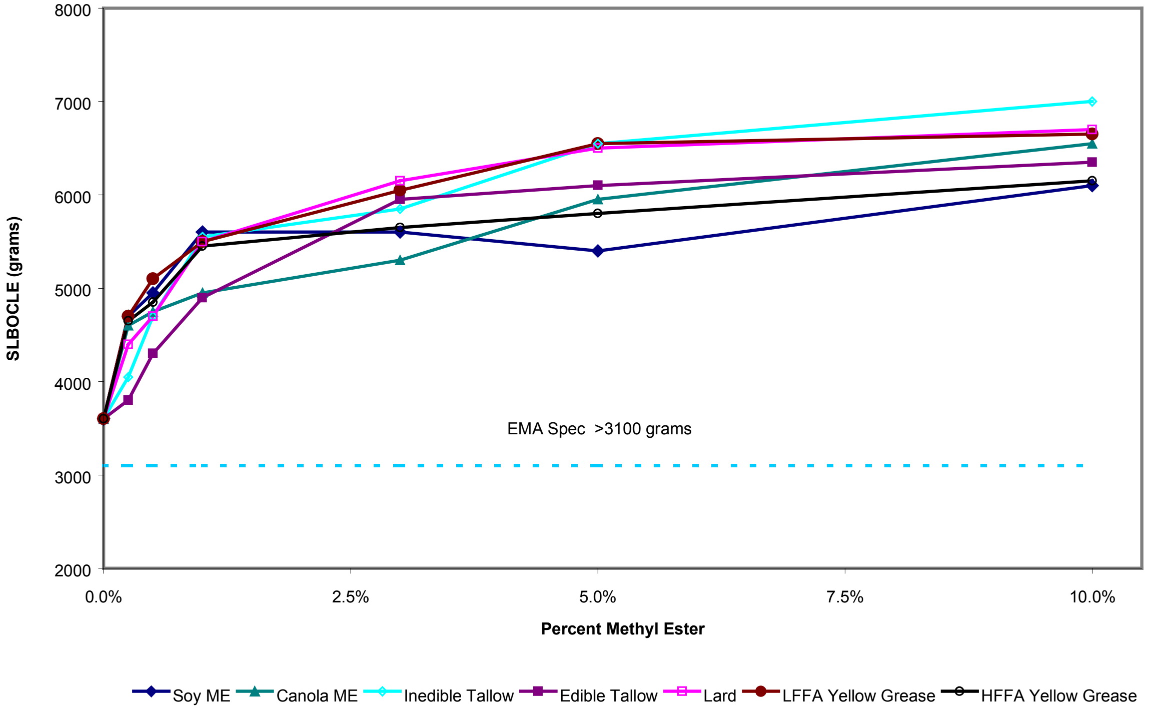

Figure 12. Biodiesel/Diesel Blend SLBOCLE Test Results
(0–10% biodiesel blend range)

Table 48 contains the results of the Oxidation Stability testing of the biodiesel blends.

Table 48. Biodiesel Blend Oxidation Stability Test Results

Biodiesel Concentration	**SME**	**CME**	**LME**	**ETME**	**ITME**	**LYGME**	**HYGME**
0%	2.34	2.34	2.34	2.34	2.34	2.34	2.34
0.25%	3.2	3.6	2.3	1.3	2.4	2.2	0.4
0.50%	2.3	3.7	2.1	2.4	1.5	2.0	0.8
1%	2.6	1.2	2.8	1.9	1.8	2.6	1.3
3%	1.3	4.1	0.7	1.4	3.4	2.9	1.5
5%	1.0	0.9	1.1	4.8	1.2	1.3	2.8
10%	0.8	0.9	1.2	25.7	0.7	1.0	1.7
20%	1.6	83.4	14.0	4.4	0.8	0.7	2.2
35%	3.0	132.9	110.3	7.6	1.8	0.9	1.8
50%	7.4	91.8	83.1	4.0	4.5	1.6	1.2
100%	16.0	44.9	72.0	8.1	41.0	6.2	8.2

Figures 13 and 14 show the results of the oxidation stability tests. The results appear to indicate that the amount of biodiesel affects the degree of aging fairly linearly for most of the biodiesels. The peaks shown for the canola methyl ester and the lard methyl ester are of similar shape, and occur at the same biodiesel fractions. This might indicate that there was something present that reacted with the diesel when the diesel:biodiesel ratio was about 2:1. Further study would be required to determine whether this is a general trait or something specific to these batches of biodiesel. Given that the other biodiesels did not show the same pattern, it is more likely to be unique to these samples.

Figure 14 shows that, at the lower concentrations (under 10%), the biodiesel does not add proportionally to the insolubles, and that it may actually have a slight suppression effect on the aging of the fuel. Alternatively, the distribution may be a measure of the noise in this test method. Further study would be required to identify the cause of the fluctuations.

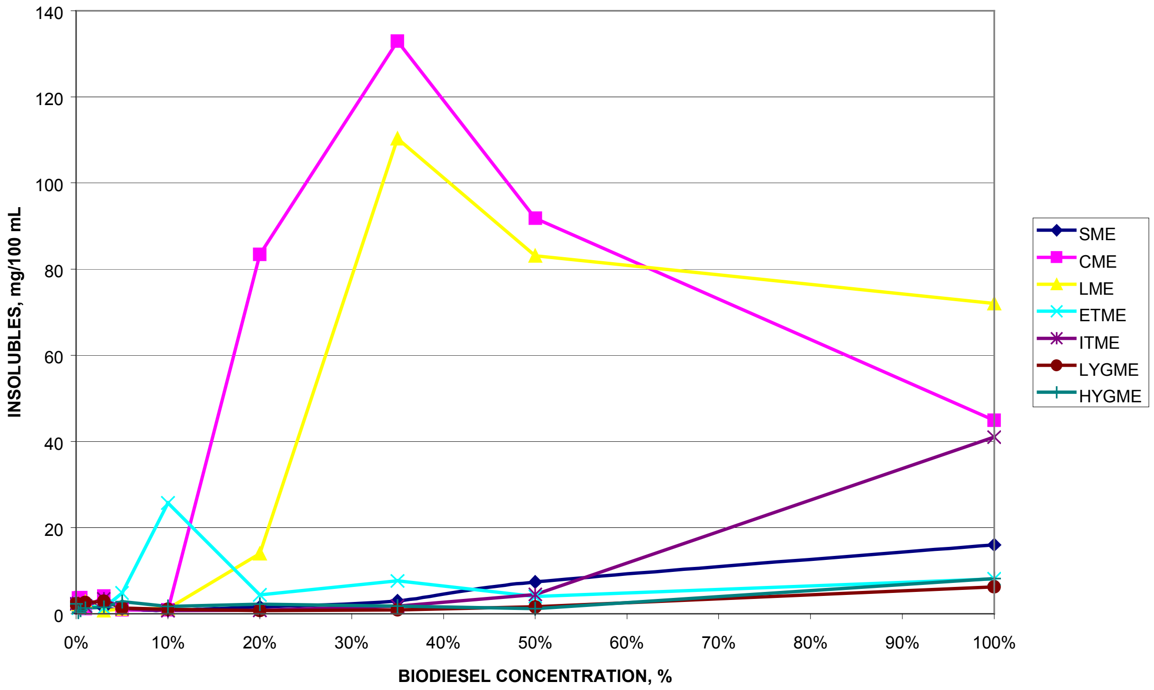

Figure 13. Biodiesel/Diesel Blend Oxidation Stability Test Results

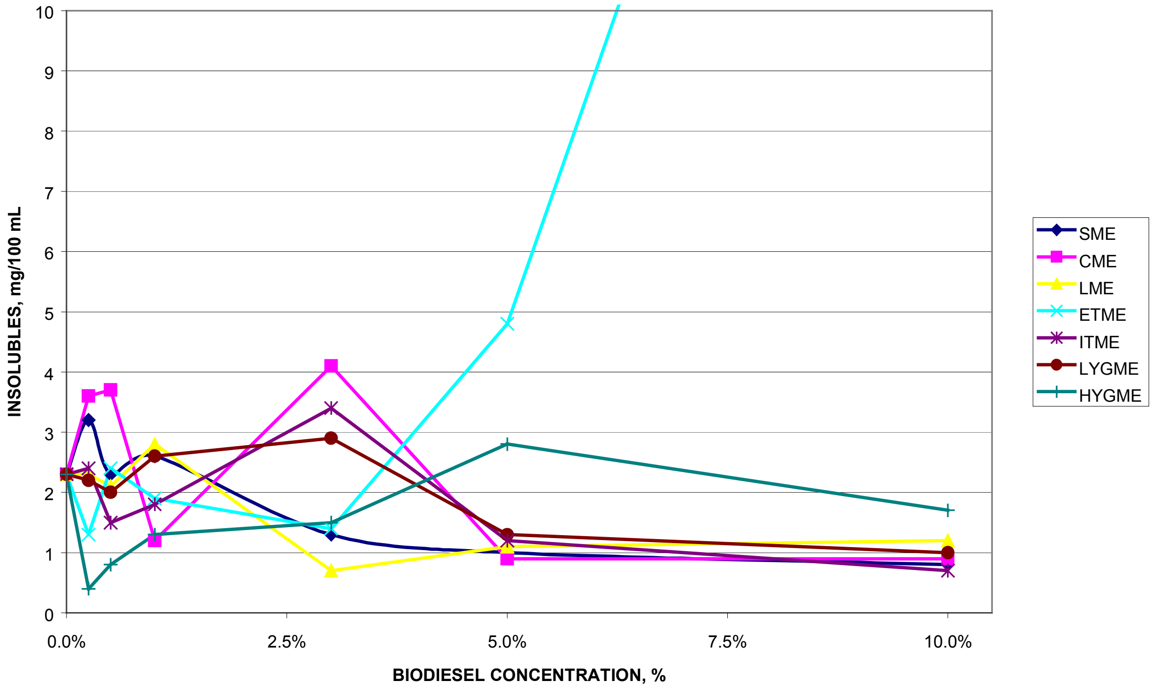

Figure 14. Biodiesel/Diesel Blend Oxidation Stability Test Results
(0–10% biodiesel blend range)

CONCLUSIONS

The first set of conclusions concerns the production of methyl esters. Starting with clean vegetable oil (e.g., soy and canola) leads to biodiesel that is easier to produce and cleaner with equivalent amounts of processing. The animal-based and waste grease-based biodiesels have additional needs at the end of transesterification to ensure acceptable properties of the biodiesel, and may benefit from processing before transesterification to reduce or eliminate components that may interfere, as in the case with the free fatty acids in the yellow grease. Further study should identify methods of economically handling free fatty acids and other components, so that they do not interfere with processing at a minimum, and ideally, that they contribute to the production of methyl esters. It is important to provide adequate testing throughout processing to know the state of the fuel. Care must be taken to ensure that "fuel grade" methyl esters are produced, instead of the more common "commercial grade."

Testing indicated there are problems in operating with low temperatures (cloud point, cold filter plugging point, and pour point), and that the minimum temperatures at which biodiesels are usable increase with the move from vegetable to animal sources, probably due to the greater degree of saturation. Yellow grease, having some of the properties of the animal biodiesels although they were originally vegetable oils, display intermediate temperatures. The higher viscosities showed the same trends as the temperatures, with the lard and tallow biodiesels higher than the soy and canola biodiesels. Because of the potential effect that high viscosities can have on injector spray performance, this property should be watched in producing biodiesel. However, the biodiesels have high boiling points, flash points, and low vapor pressure, as well as an inability to smoke under the smoke point test, indicating a high level of safety for handling biodiesels. In addition, biodiesels show low levels of reactiveness with other materials (e.g., copper corrosion). A focus on additives to forestall the cloud/plugging/pour point problems would help the adoption of biodiesels in areas with low ambient temperatures (much of the United States for the tallow and lard biodiesels).

In considering the range of tests performed in this project, the specifications used to identify acceptable biodiesels do not need any additions. Other tests would contribute additional information about the composition and properties, but the tests in the specifications adequately identify whether the quality of the fuel is acceptable.

The biodiesel specification and the ASTM D 975 specification can be partially applied to B20. B20 could exceed D 975 values for sulfated ash, viscosity, and distillation fraction temperature, and could exceed biodiesel limits in flash point and carbon residue.

The objective of the biodiesel/diesel blend testing was to determine if biodiesels from different feedstocks exhibit different characteristics in varying concentrations with diesel when tested on a consistent basis. Testing covered blending ranges that have previously been overlooked in the study of the potential of biodiesel blends. Of particular interest for people considering use of biodiesels in ratios other than as B20 or straight are the shifts in temperature for various properties tested. Care should be taken in handling and use due to the temperature increases that occurred for pour point, cloud point, and cold filter plugging point. In terms of lubricity, the significant increase that occurs with even concentrations at 3% or less of the biodiesels appears to confirm that biodiesel additives could improve operation of diesels and extend life of their components. In particular, further study may be warranted to improve the processing of the beef tallow and pork lard, with a goal of reducing cost while enhancing the characteristics of the methyl esters that improve lubricity.

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13. ABSTRACT *(Maximum 200 words)*

In a project sponsored by the National Renewable Energy Laboratory, the Institute of Gas Technology is conducting an investigation of biodiesels produced from vegetable and animal based feedstocks. This subcontract report presents their findings.

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