Investigation of Biodiesel Thermal Stability under Simulated In-Use Conditions

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Summary:

Biodiesel is an alternative diesel fuel produced by transesterification of vegetable oils or animal fats. While biodiesel provides numerous environmental benefits such as reduced exhaust emissions, it is more prone to oxidation than petroleum-based diesel fuel and this can alter its properties. When oxidation occurs at ordinary temperatures, the initial products are hydroperoxides. Sediment and gums are formed through polymerization of the peroxides and can cause fuel filter plugging. The objective of this study was to relate the chemical and physical processes associated with biodiesel oxidation to the conditions that affect diesel fuel system performance. A relationship was sought between the test that is used by the engine industry to define engine fuel stability requirements (ASTM D2274) and the tests used by the fats and oils industry to characterize oxidation (Peroxide Value and Acid Value). While oxidation causes the fuel viscosity to increase, fuel filter plugging was not necessarily a natural consequence of biodiesel oxidation even when the fuel was oxidized to a level beyond what would be observed in practice. The effect of fuel temperature and blending with diesel fuel on the oxidation was investigated and the interrelationship between the fuel's acid value and viscosity is shown.

Keywords: diesel, energy, engines, fuel, oil, oilseed, soybean oil.

INTRODUCTION

Many researchers have investigated vegetable oils as an alternative fuel for diesel engines (Goering, et al., 1982; Bagby, et al., 1987; Engelman, et al., 1978; Hawkins, et al., 1983). Most of this research has shown that using raw vegetable oils for diesel engines can cause numerous problems [Korus, et al., 1982; Lague, et al., 1988]. The increased viscosity, low volatility, and poor cold flow properties of vegetable oils lead to severe engine deposits, injector coking, and piston ring sticking (Perkins, et al., 1991; Pestes and Stanislao, 1984; Clark, et al., 1984; Vellguth, 1983). However, these effects can be reduced or eliminated through transesterification of the vegetable oil to form monoesters (Perkins et al., 1991; Zhang et al., 1988). The process of transesterification removes glycerol from the triglycerides and replaces it with radicals from the alcohol used for the conversion process (Kusy, 1982). This process decreases the fuel's viscosity but maintains its cetane number and heating value. These alkyl monoesters of vegetable oils or animal fats have come to be known as *biodiesel*. The most common form of biodiesel in the United States is made with methanol and soybean oil and is known as soy methyl ester, or methyl soyate.

One drawback of biodiesel is that there is an inverse relationship between biodiesel's tendency to oxidize and its cold flow properties. Saturated compounds are less prone to oxidation than unsaturated compounds but they crystallize at unacceptably high temperatures. Vegetable oils are oxidized through contact with molecular oxygen in the air. Oxidation reactions that occur without any outside influence are called *autoxidation*. Autoxidation is initiated and propagated by free radicals reacting with methylene -CH_2- groups that are adjacent to double bonds (Stauffer, 1996). This is why the rate of oxidation is so strongly affected by the degree of saturation of the oil. Some vegetable oils contain natural antioxidants, such as vitamin E (tocopherol). These antioxidants react with the active free radicals producing a species of low reactivity that does not initiate new reaction chains. However, as free radicals continue to form, eventually all the antioxidant will be consumed, and then oxidation will proceed rapidly.

Hydroperoxides are the initial products of oxidation at ordinary temperatures. They are very unstable and have a tendency to attack elastomers. In addition, hydroperoxides can polymerize and form insoluble gums and sediments that increase viscosity and plug fuel filters (Clark et al., 1984). These peroxides split and form aldehydes, ketones, and finally shorter chain acids. The level of these acids is measured by the acid value. However, no research has yet been conducted to determine the maximum degree of oxidation allowable for fuel to be used in diesel engines.

The objective of this study was to monitor the oxidation of biodiesel under conditions that are similar to those encountered in use. Then the chemical and physical processes associated with biodiesel oxidation were related to the conditions that affect fuel system performance. To support this objective a connection was sought between the ASTM (American Society for Testing and Materials) D 2274 fuel stability tests (ASTM, 1998) and the AOCS (American Oil Chemists' Society) tests for peroxides and acids. ASTM D2274 measures sediment and gum formation in diesel fuels and AOCS Cd 8-53 and Cd 3a-63 measure the peroxides and acids formed by fuel oxidation (AOCS 1998a, 1998b). The *Peroxide Value* is expressed as milliequivalents of peroxide per kilogram of sample. The *Acid Value* characterizes the amount of organic acids produced by oxidation and is expressed as the amount of potassium hydroxide required to neutralize the acids.

During the course of this project it was found that ASTM D2274 was not appropriate for use with biodiesel. As will be described later, the technique used to isolate gums and sediment in this method does not adequately distinguish between these compounds and biodiesel. It was also found that fuel filter plugging is not an inevitable consequence of biodiesel oxidation. In contrast to an earlier project by other researchers (Clark et al., 1984), no tendency to fuel filter plugging was found in this project, even with highly oxidized biodiesel.

This paper describes the chemical changes that were observed in the biodiesel as it oxidized under simulated in-use conditions. The effect of temperature and blending with diesel fuel is also discussed. Since ASTM D2274 is not able to characterize the stability of biodiesel a new test procedure is needed. The data presented here will support efforts to develop this procedure. A companion paper presents the results of biodiesel oxidation under controlled laboratory conditions where phenomena occur that were not observed during the simulated in-use testing (Canakci et al., 1999).

EQUIPMENT AND PROCEDURE

To simulate the in-use conditions experienced by fuel in a diesel fuel system, a test stand following Society of Automotive Engineers (SAE) standard J905 (SAE, 1995) for fuel filter testing was constructed. A schematic of the test stand is shown in Figure 1. A Holley model 12-802 electric fuel pump followed by a Fleetguard model FS1001 fuel filter was attached to the top of a 208 liter (55 gallon) barrel. The biodiesel fuel was pumped from the barrel and pushed through the fuel filter before returning to the barrel. A three-way valve was used to take fuel samples at a point downstream of the filter. An electric heating tape was wrapped around the barrel to control the fuel temperature.

In this experiment, a total of four test rigs using 208 liter (55 gallon) barrels and a single test rig with a 19 liter (5 gallon) stainless steel container were used. Two of the barrels were maintained at a controlled temperature at 60°C (140°F) and the other two were at room temperature. The 19-liter test rig was maintained at 60°C. The flow rate was kept constant for all of the tests at 5 kg/min (11 lbm/min), a typical flow rate for an engine circulating pump. The fuel flow rate was measured periodically by switching the 3-way valve following the filter to direct the flow into a graduated cylinder rather than returning it to the barrel. A stop watch was used to determine the average flow rate.

The conditions in the test rig were intended to simulate the conditions that fuel would be exposed to while circulating through a diesel fuel system. The temperature of 60°C was chosen because this is a reasonable value for fuel circulating through a diesel engine fuel system. The test should be considered as severe because the fuel is recirculated for a longer time than would normally be encountered in practice. Originally, the test duration was intended to correspond to the actual length of time that fuel could be expected to recirculate before being injected and burned in the engine. However, when no indication of fuel filter plugging was found, the tests were extended in an attempt to identify conditions that might cause filter plugging.

A data logging system was used to measure the time, temperature, and differential pressure across the filter every five minutes for each test rig. Fuel samples were initially taken every day, however, after a few days, the samples were taken every two days.

The biodiesel used for these tests was purchased from NOPEC Corporation of Lakeland, FL. The diesel

fuel was a commercial No. 2 low sulfur diesel fuel. The laboratory analysis of these two fuels is shown in Tables 1 and 2. As mentioned earlier, the peroxide value was measured with AOCS method Cd 8-53 and the acid value was measured with AOCS method Cd 3a-63. These methods are both based on titration and were assumed to be applicable to petroleum-based fuels. Since similar ASTM methods are available for petroleum fuels (D664, D3703) this assumption should be valid.

In order to understand the effect of oxidation on the properties of biodiesel fuel, five tests were conducted using the fuel filter test apparatus. Two of them were with 20% and 50% biodiesel in No. 2 diesel fuel (by weight) at 60°C (140°F). Two other tests were conducted with 20% and 50% biodiesel at room temperature (23°C, 73°F). These four tests were conducted in 208 liter (55 gallon) barrels each containing 125 liters (33 gallons) of fuel. The final test was 100% biodiesel at 60°C in the 19 liter (5 gallon) stainless steel container with 5 kg (11 kg) of biodiesel. In general, all of the tests were continued until the fuel pump failed, usually by seizure. The duration varied from 38 days for the 60°C test of 100% biodiesel to 190 days for the room temperature test of 20% biodiesel. The failure of these pumps should not be interpreted as an indication of the quality of the pumps or any incompatibility between the pumps and biodiesel. During the test the flow rate was monitored and as it dropped due to increased fuel viscosity, the voltage applied to the pump was raised to compensate. Generally, by the end of the test, the voltage applied to the pump was above the manufacturers recommendations and this was the probable cause of failure.

RESULTS AND DISCUSSION

In this section, the effect of oxidation on the peroxide value, acid value, and viscosity of biodiesel are presented. These properties characterize the chemical changes occuring in the biodiesel as it oxidizes. One expected product of the fuel oxidation was the production of sediment and gum. This sediment and gum should be collected by the fuel filter and cause the pressure drop across the filter to rise. A sample case of 100% biodiesel at 60°C (140°F) is shown in Figure 2. The increase in the pressure drop across the filter was relatively small for all of the cases studied in this project.

Effect of oxidation on the peroxide value of biodiesel
The peroxide values measured for all five tests are shown in Figure 3. In all cases, the peroxide value (PV) rises to a level between 300 and 400 meq/kg ester and then drops off. The three cases at 60°C went through this process of rise and fall of PV within the first 1000 hours with the rise in PV for the 50% and 100% biodiesel occuring within the first 50 hours. The 20% blend rose more slowly but followed the same general process. The two runs at room temperature were similar but much slower, both for the initial rise in PV and the subsequent decline. The total test duration here is clearly beyond what would occur in actual practice. The test was continued to gain a complete understanding of the processes that may occur in the fuel as it progresses from unoxidized to highly oxidized fuel. The oxidation process in an engine fuel system may be accelerated compared with this experiment if there is a higher degree of aeration. Other test results have shown that with a high level of aeration, the fuel can oxidize in a few hours at 60°C. Another reason for the extended testing was to determine whether fuel filter plugging would occur. No instances of plugged fuel filters were observed in this test program.

Miyashita et al. (1988) investigated the autoxidation rates of various esters of safflower oil. They found that the peroxide value increased rapidly after the induction period, but then decreased as was found in this study. However, they found the maximum peroxide value to be about 2000 meq/kg ester. The

reason for the higher PV is not known but they did use a different method, the Calorimetric Iodine method (Takagi et al., 1978), to measure the peroxide value. Gan et al. (1995) investigated the effects of oxidation on the methyl ester of palm olein. They recorded the maximum peroxide value to be about 300 meq/kg ester after 288 hours, which was close to that observed in this experiment.

The reason the PV reaches a maximum and then drops off may be due to the solubility of oxygen in the fuel. When the induction period is over, the oxidation proceeds rapidly consuming all of the oxygen that is dissolved in the ester. The peroxides formed are subsequently converted to aldehydes, ketones, and acids. When the dissolved oxygen is consumed, the peroxide formation rate drops off to the rate that can be sustained by diffusion of O_2 into the ester from the air surface. If this rate is below the rate at which peroxides are converted to aldehydes and ketones, the concentration of peroxides will remain low.

Effect of oxidation on the acid value of biodiesel
The results of the acid value measurements for the tests described earlier are shown in Figure 4. The acid value for all the blends increased with time, but the rates of increase were different. It can be seen in Figures 3 and 4 that the acid value starts to increase at a slightly later time than when the peroxide value starts to rise. The rate of increase of the acid value is higher until the point where the peroxide value returns to a low value. Beyond this point the acid value increases more slowly indicating a slower rate of oxidation. This behavior supports the hypothesis proposed earlier for an oxidation rate controlled by oxygen diffusion.

Effect of oxidation on the viscosity of biodiesel
Figure 5 shows that the viscosity of all the blends increased with time. The blends containing higher fractions of biodiesel and at higher temperatures increased more rapidly. The 50% and 100% biodiesel at 60°C had rapid increases in viscosity until the time where the rate of increase of acid value was a maximum. Then the viscosity increased more slowly. The data for the 50% blend at room temperature demonstrated the unusual behavior of having a peak at about 1300 hrs and then leveling off. This anomalous behavior has not been explained.

Interrelationship between acid value and viscosity
Figure 6 shows the correlation between acid value and viscosity. For the 20% biodiesel at 60°C (140°F) and at room temperature, the viscosity vs. acid value curves coincide, but the acid value did not increase to as high a value for the room temperature case by the time the test was terminated. The coincidence of the 20% blend lines supports the hypothesis that there is a coupling between the reactions that form the acid compounds and the polymerization reactions that increase the viscosity. The 50% blend lines also coincide for the initial portion of the oxidation process but when the anomaly mentioned earlier occurred, and the viscosity decreased, the curves diverged.

ASTM D2274-94 test method for oxidative stability test
The standard method to measure the thermal and oxidative stability of diesel fuels is ASTM (American Society for Testing and Materials) D2274 (ASTM, 1998). The method uses accelerated oxidizing conditions to determine the mass of insoluble material formed by the fuel oxidation. A 300 ml (0.079 gallon) sample of fuel is held at 95°C (203°F) for 16 hours while pure oxygen is bubbled through it at the rate of 3 L/h. Then the sample is cooled to approximately room temperature before filtering to collect insoluble material. Adherent insolubles are removed from the oxidation cell and associated glassware with a solvent that is evaporated to obtain the quantity of adherent insolubles. The sum of the

filterable and adherent insolubles is the total insolubles and is expressed as milligrams per 100 ml of fuel. The prescribed filter media are 47mm (1.85 inch) diameter cellulose ester membranes with a pore size of 0.8 micrometer (3.15×10^{-5} inches). The method requires two filters with one stacked on top of the other. The bottom filter is used as a blank and the difference between the top and bottom filters is the filterable insoluble. The ASTM procedure needed to be modified for this test because finding a matched pair of filters is very difficult. This problem was accommodated by weighing the top (sample) and bottom (blank) filters before and after filtration of the sample.

ASTM D2274 was never fully applied to biodiesel in this study because preliminary testing showed that the filter washing procedures were not effective. In an effort to validate the method, 300 ml of previously filtered biodiesel from a newly opened barrel of methyl soyate was poured through the filters. This was clean fuel that should have contained no insoluble material. After filtration, three separate 50 ml volumes of isooctane were used to rinse the filter assembly. The two filters were dried at 80°C for 30 minutes and then cooled at room temperature for another 30 minutes before weighing. The results of this initial validation test are shown as Test No. 1 in Table 3. The top and bottom filters had weight gains of 0.01478 g and 0.01501 g, respectively. The total filterable insoluble for this test would be the difference between these two weights and was –0.00023 g or –0.00008 g/100 ml fuel which could be taken as an indication of little or no sediment formation. However, the large increase in weight for the blank filter is suspicious because it indicates that the biodiesel was not removed from the filters. Subsequent repetitions with biodiesel gave approximately the same results.

Test No. 2 shown in Table 3, used the same procedure as Test No. 1 except that the sample was clean No. 2 diesel fuel. Instead of gaining weight as occurred during the methyl soyate test, both the top and bottom filters lost a small amount of weight. This expected result was probably caused by the isooctane washing a small amount of organic material from the filter medium. It was the primary purpose for including the blank. The weight change of the top filter was -0.00051 g and for the bottom was –0.00023 g so the filterable insoluble was –0.00009 g/100 ml fuel. These results indicate that the isooctane washing procedure was effective in removing the diesel fuel from the filters. This positive result with diesel fuel also showed that the laboratory techniques used for the test were valid.

Additional testing was performed to determine whether the filter weight gain with the biodiesel could have been caused by contaminants. The amount of biodiesel was varied, and different techniques were used to filter and preclean the biodiesel before it entered the filters. Different solvents, including hexane and toluene were tried with the same results. Other, more polar, solvents might be more effective at removing the biodiesel but they would also tend to remove the gums and sediments the test is used to measure.

Another approach was to increase the number of times the filters were washed with isooctane from 3 to 10. When this was performed the weight change of the filters was much smaller, although it was still positive. This further confirmed that inadequate filter washing was the problem. Washing the filters with this much isooctane was not considered to be an acceptable method because it was a significant departure from the ASTM procedure and this amount of isooctane could also remove a portion of the gums and sediments that are to be measured. The type of filter used was also changed. Gelman type AE glass fiber filters were used and retained even more biodiesel than the membrane filters specified in the ASTM method. At this point it was determined that ASTM D2274 was not appropriate for use with biodiesel and further testing was terminated.

CONCLUSIONS

The original objective of this project was to relate the ASTM test procedures for fuel oxidation and stability to the AOCS test procedures and to the needs of the engine. During the course of the project it was found that the standard ASTM procedure for characterizing the oxidative stability of diesel fuel (ASTM D2274) is not appropriate for biodiesel. The original expectation that the primary obstacle to using oxidized biodiesel in an engine would be fuel filter plugging was also found to be incorrect. Long term testing with biodiesel that had been oxidized well beyond what would be encountered in practice showed no instances of fuel filter plugging.

Quantitative results showing the chemical and physical changes that occur in the fuel when it oxidizes were presented. The following conclusions can be drawn about the oxidation process of biodiesel.

1. The oxidation of biodiesel (SME) occurs more rapidly at higher temperatures. Dilution with diesel fuel slows the biodiesel oxidation rate.
2. The maximum peroxide value observed for any of the fuels was between 300 and 400 meq/kg ester. The peroxide value rose to the maximum level and then dropped off.
3. The induction period varies for different blend levels with 100% biodiesel having a shorter induction period than the blends.
4. The initial rate of increase of acid value was higher until the point where the peroxide value dropped off, then the rate of increase of the acid value was lower.
5. A strong correlation was found between acid value and viscosity. The viscosity increased with time for all blends of biodiesel and diesel fuel.

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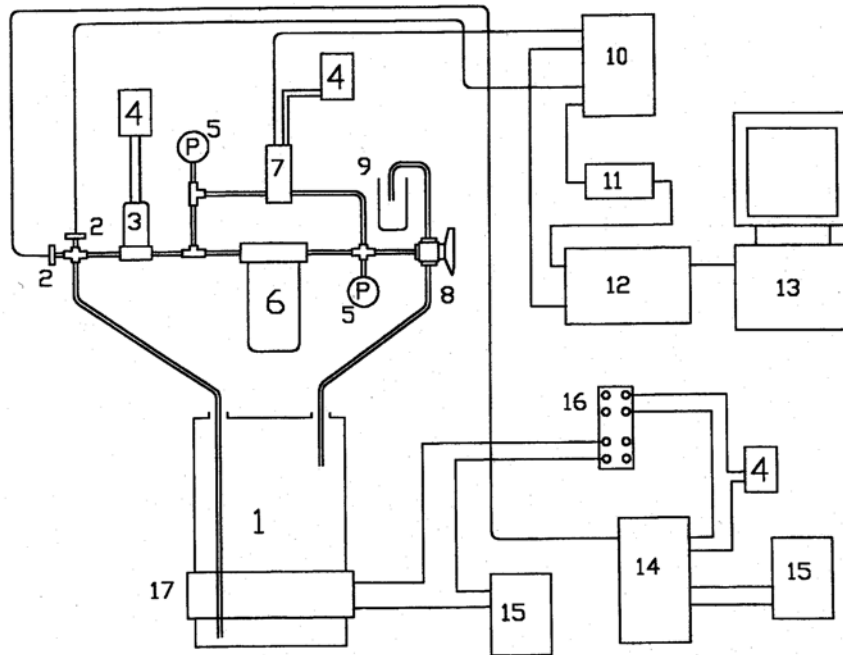
1. 208-liter barrel 2. K-type thermocouples 3.Holley model 12-802 electric fuel pump   4. 12 Volt DC supply 5. Pressure gauge 6. Fleetguard model FS1001 fuel filter   7. Differential pressure transducer 8. Three-way valve 9. Sample container 10. Scanner 11. Ice point compensator 12. Volt meter 13. Computer 14. Omega Model CN9000A temperature controller 15. 110 Volt AC supply 16. Relay 17. Barrel heating tape

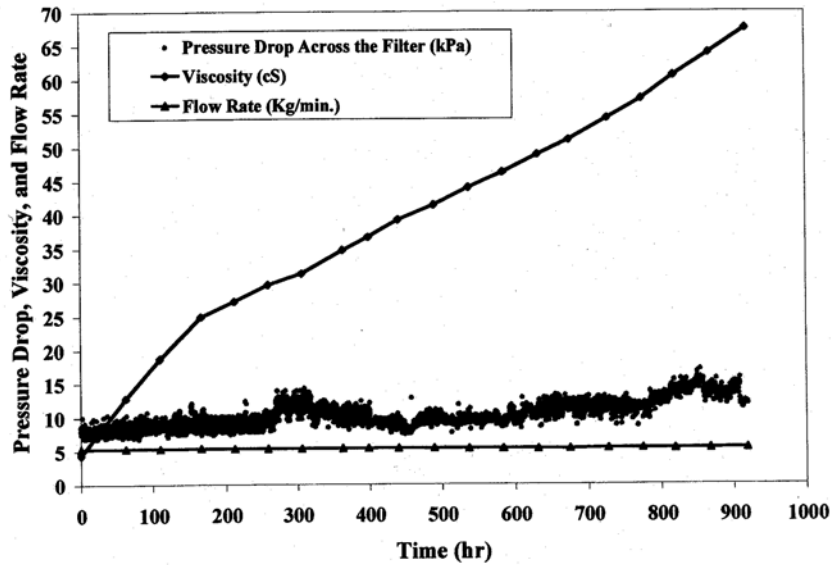**Figure 1. Schematic diagram of the fuel filter test rig**

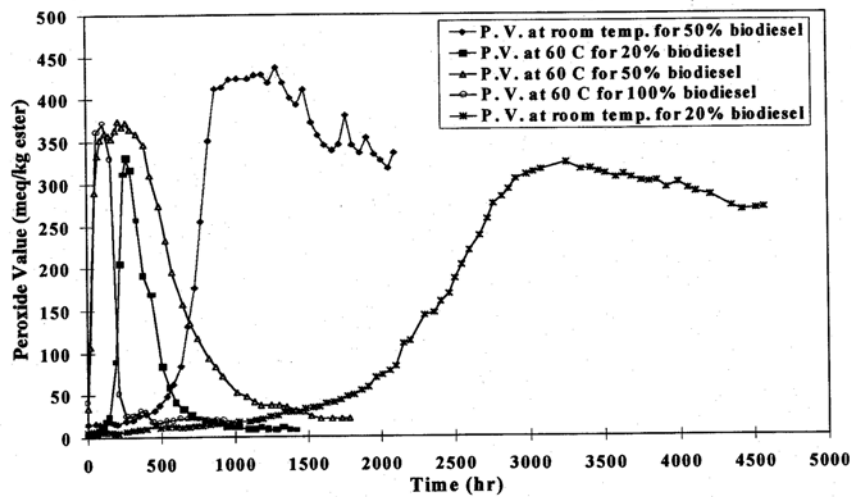Figure 2. Pressure drop across the fuel filter for 100% biodiesel at 60 °C

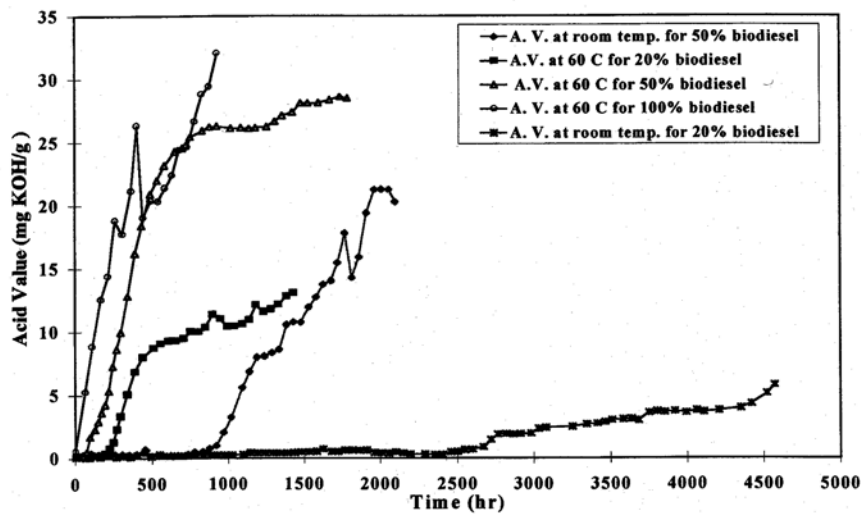Figure 3. Effect of oxidation on the peroxide value of biodiesel

Figure 4. The effect of oxidation on the acid value of biodiesel

Legend:
- A. V. at room temp. for 50% biodiesel
- A.V. at 60 C for 20% biodiesel
- A.V. at 60 C for 50% biodiesel
- A. V. at 60 C for 100% biodiesel
- A. V. at room temp. for 20% biodiesel

Y-axis: Acid Value (mg KOH/g)
X-axis: Time (hr)
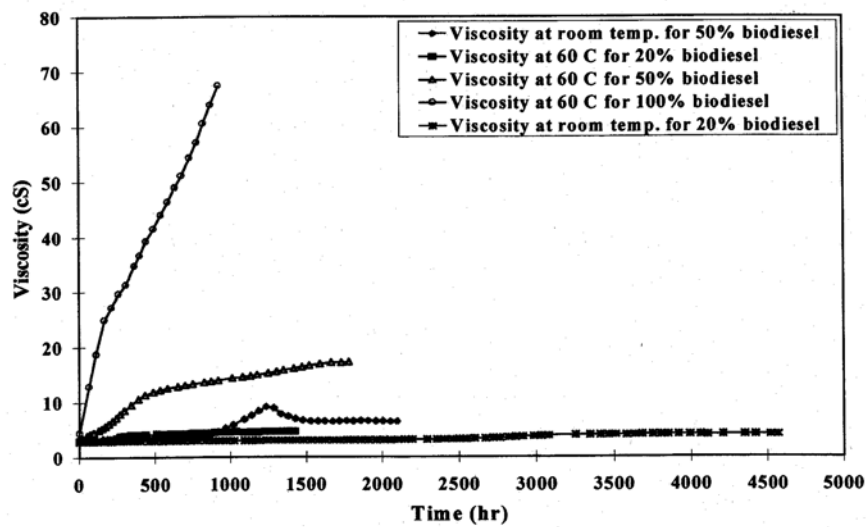
Figure 5. The effect of oxidation on the viscosity of biodiesel

Legend:
- Viscosity at room temp. for 50% biodiesel
- Viscosity at 60 C for 20% biodiesel
- Viscosity at 60 C for 50% biodiesel
- Viscosity at 60 C for 100% biodiesel
- Viscosity at room temp. for 20% biodiesel
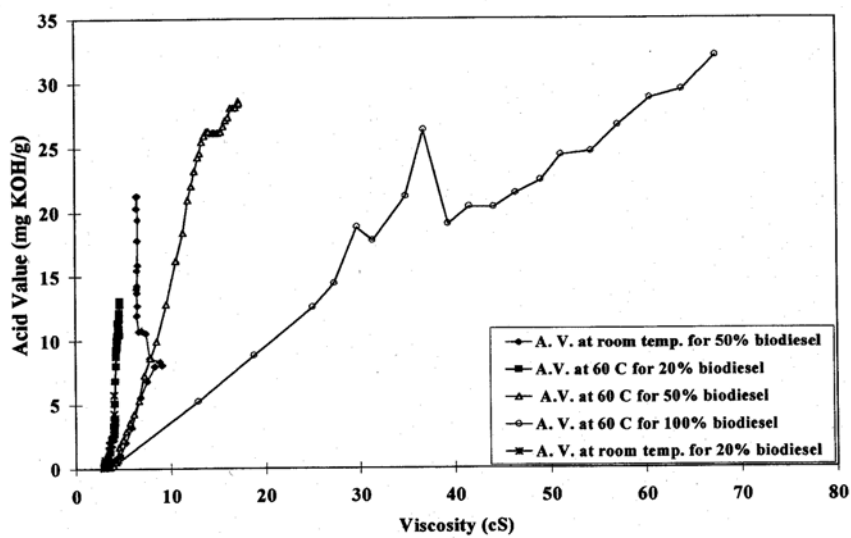
Y-axis: Viscosity (cS)
X-axis: Time (hr)

Figure 6. The relation between viscosity and acid value for biodiesel blends

Table 1. Analysis of the biodiesel fuel

Fatty Acid Composition	
Palmitic (16:0)	10.76%
Stearic (18:0)	4.37%
Oleic (18:1)	24.13%
Linoleic (18:2)	51.83%
Linolenic (18:3)	6.81%
Free glycerin, %	0.004
Monoglycerides, %	0.352
Diglycerides, %	0.132
Triglycerides, %	0.152
Total glycerin, %	0.131
Cetane Number (D613)	51.1
Lower Heating Value, kJ/kg	37272
% Sulfur	0.005
% Carbon	76.14
% Hydrogen	11.75
Viscosity, cS	4.63

All percentages at wt%.

Table 2. Analysis of No. 2 diesel fuel

% Carbon	86.23%
% Hydrogen	13.14%
% Sulfur	0.034%
Heat of Combustion	
Gross, kJ/kg	45504
Net, kJ/kg	42715
Hydrocarbon types	
Saturates, %	64.1
Olefins, %	4.9
Aromatics, %	31.0

All percentages are wt%.

Table 3. Filterable insoluble for different fuels

		Test No. 1	Test No. 2
Fuel		Biodiesel	No. 2 Diesel
Amount of Fuel Filtered (ml)		300	300
Washing Fluid		Isooctane	Isooctane
No. of Washing Time (each time 50 ml)		3	3
Vacuum Pressure (kPa)		83	83
Top Filter Weight (g)	After	1.09388	1.07566
	Before	1.07910	1.07617
	Difference	0.01478	-0.00051
Bottom Filter Weight (g)	After	1.09335	1.08359
	Before	1.07834	1.08382
	Difference	0.01501	-0.00023
Total Filterable Insoluble (g)		-0.00023	-0.00028
Filterable Insoluble (g/100ml)		-0.00008	-0.00009