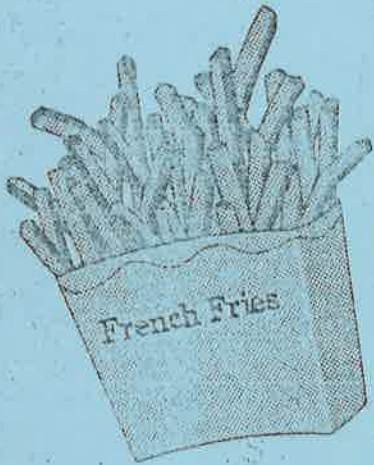
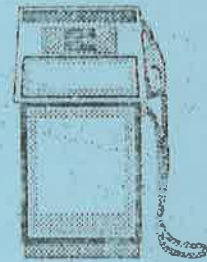
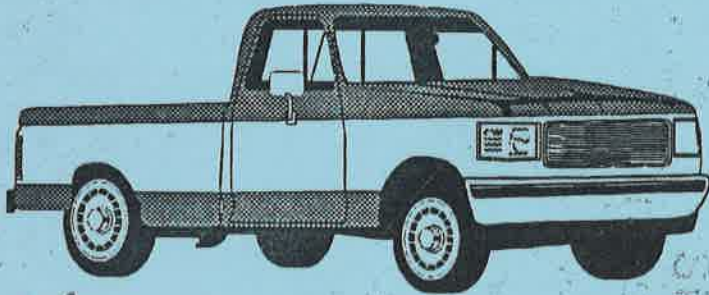


HySEE Preliminary Processing and Screening

**Making and testing a biodiesel fuel
made from ethanol and waste french-fry oil**



Idaho
Bioenergy Program



Idaho Department of Water Resources



Energy Division

July 1995

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DISCLAIMER

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HYSEE PRELIMINARY PROCESSING AND SCREENING

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HYSEE PRELIMINARY PROCESSING AND SCREENING

by

**Charles L. Peterson, Daryl L. Reece, Brian Hammond
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INTRODUCTION

Due to increasing environmental awareness, Biodiesel is gaining recognition in the United States as a renewable fuel which may be used as an alternative to diesel fuel without any modifications to the engine. Biodiesel fuels can be produced from ethanol and vegetable oil, both agriculturally derived products. As such, they provide several advantages: they are renewable, they are safer, they are biodegradable, they contain little or no sulfur and they reduce engine exhaust smoke. Currently, the cost of this fuel is a primary factor that limits its use. One way to reduce the cost of Biodiesel is to use a less expensive form of vegetable oil such as waste oil from a potato processing plant.

Idaho produces approximately 120 million cwt of potatoes from over 152,000 ha annually. Nearly 60 percent of these are processed, the vast majority being made into french fried potatoes. These operations use mainly hydrogenated soybean oil, some beef tallow and canola. It is estimated that there are several million pounds of waste vegetable oil from these operations each year. Additional waste frying oil is available from smaller processors, off-grade oil seeds and restaurants.

One of these processors, produces over 2 billion pounds of frozen potatoes per year at plants in Oregon, Idaho and North Dakota. This company built two ethanol plants in the late 1980's, which use potato waste as the feedstock. One plant provides an opportunity for a Biodiesel facility using waste vegetable oil and ethanol to produce hydrogenated soy ethyl esters (HySEE). The market value of waste frying oils is about \$0.11 per liter (\$0.40 per gallon). Ethanol has a plant value of about \$0.28 per liter (\$1.05 per gallon). It is projected that this facility could produce Biodiesel at only slightly over \$0.25 per liter (\$1.00 per gallon) making it economically comparable to diesel fuel.

Biodiesel is being demonstrated as a motor fuel in an ongoing project entitled, "Demonstration of the On-the-Road Use of Biodiesel." This project is a cooperative effort between the University of Idaho and the Idaho Department of Water Resources. Hydrogenated soy ethyl ester (HySEE) has good possibilities for use as a diesel fuel substitute because:

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as the baseline fuel for the engine performance testing and emissions testing, and D2 from a local vendor was used for the 300 hour endurance engine testing.

Fuel Characterization

The fuels were characterized by evaluating the parameters required in ASAE EP X552. The tests for specific gravity, viscosity, cloud point, pour point, flash point, heat of combustion, total acid value, catalyst, and fatty acid composition were performed at the Analytical Lab, Department of Agricultural Engineering, University of Idaho. The boiling point, water and sediment, carbon residue, ash, sulfur, cetane number, copper corrosion, Karl Fischer water, particulate matter, iodine number, and the elemental analysis were performed at Phoenix Chemical Labs, Chicago Illinois. The high performance liquid chromatograph (HPLC) and titration analysis for total and free glycerol, percent of oil esterified, free fatty acids, and mono-, di-, and triglycerides were performed by Diversified Labs Inc., Chantilly, Virginia.

Engine Performance Tests

All engine performance tests were conducted in the engine performance lab at the University of Idaho. The equipment used and tests conducted are described below. The short term tests were performed with an in-line four cylinder John Deere 4239T turbocharged, direct injected diesel engine. It has a displacement of 3.9 liters (239 cubic inches), a high RPM of 2650, 61 kW (82 hp) at 2500 RPM, and 290 Nm (214 ft lbf) torque at 1500 RPM. It is attached to a General Electric 119 kW (159 hp) cradled dynamometer. The engine was not modified in any way for use with renewable fuels.

A Hewlett Packard data acquisition unit (model 3497-A) and a personal computer were used to collect data every thirty seconds during each of the tests. Torque, power, opacity, fuel consumption, and temperatures of various engine parameters were monitored throughout the testing and saved into a data file.

Fuel Flow Equipment -- The fuel delivery and return lines were adapted with quick couplers for fast and clean changing of the fuels. Individual 19 liter (5 gallon) metal fuel tanks were modified with a fuel filter and flexible fuel lines which could be connected to the engine quick couplers. Fuel flow rate was determined by direct weighing. The fuel containers were placed on an electric 45.4 kg (100 lb) scale accurate to 23 grams (0.05 lb) with RS232 capability.

Opacity Meter -- A Telonic Berkley model 200 portable opacity meter was connected to the data acquisition unit. The opacity meter consists of a light source positioned on one side of the exhaust stream and a photo resistor mounted on the opposite side. The meter provides an output voltage ranging from 0 to 1.00 volts. One hundred percent opacity (1.00 volt) corresponds to no light transmission whereas 0 percent opacity (0.0 volts) corresponds to complete light transmission.

PROCEDURES

Fuel Production

The HySEE fuel production process utilizes 70 percent stoichiometric excess ethanol (absolute, 100 percent pure), or a molar ratio of 5.1:1 ethanol to oil ratio. The total free fatty acids are determined and neutralized with the calculated addition of catalyst. Based on the amount of input oil by weight, 1.3 percent of KOH is used plus the amount to neutralize the free fatty acids. The following equations were used for the quantities processed:

$$\text{EtOH} = 0.2738 \times \text{Oil} \qquad \text{KOH} = \text{Oil}/85$$

where: Oil = desired amount of oil, in liters

EtOH = amount of ethanol needed, in liters

KOH = amount of potassium hydroxide required, in kg

The waste hydrogenated soybean oil is heated to 49 degrees Celsius (120 degrees Fahrenheit). The catalyst is dissolved into the alcohol by vigorous stirring in a small reactor. The oil is transferred into the Biodiesel reactor and then the catalyst/alcohol mixture is pumped into the oil and the final mixture stirred vigorously for two hours. A successful reaction produces two liquid phases: ester and crude glycerol. Crude glycerol, the heavier liquid will collect at the bottom after several hours of settling. Phase separation can be observed within 10 minutes and can be complete within two hours after stirring has stopped. Complete settling can take as long as 20 hours. After settling is complete, water is added at the rate of 5.5 percent by volume of the oil and then stirred for 5 minutes and the glycerol allowed to settle again. After settling is complete the glycerol is drained and the ester layer remains. Washing the ester is a two step process which is carried out with extreme care. A water wash solution at the rate of 28 percent by volume of oil and 1 gram of tannic acid per liter of water is added to the ester and gently agitated. Air is carefully introduced into the aqueous layer while simultaneously stirring very gently. This process is continued until the ester layer becomes clear. After settling, the aqueous solution is drained and water alone is added at 28 percent by volume of oil for the final washing.

Engine warm-up and cool-down

Three different engine test protocols were followed using facilities at the University of Idaho. Each test started with a warm-up and ended with a cool-down period. The warm-up period consisted of a two minute interval on D2 at low idle. Then there was an eight minute interval with the fuel to be tested. During this eight minute period there is a gradual increase in load and RPM to the rated horsepower and load. The cool-down period consisted of 10 minutes on D2 at low idle. For both the warm-up and cool-down periods the return fuel line was placed into a separate container.

RESULTS

Fuel Production

Waste vegetable oil was obtained from the french fry plant owned by Simplot, Inc., Caldwell, Idaho. The waste oil was placed in drums and is solid at normal room temperatures. The oil is heated in the drums by electric heaters and is then transferred into the biodiesel reactor for transesterification. The ethanol-KOH mixture is added to the heated waste grease. The amount of ethanol and KOH must be adjusted upward to account for vaporization of the ethanol as it is heated and the free fatty acid content of the waste oil. Separation of the ester and glycerol is a constant problem. The final product produced in these tests was found to be 92.26% esterified and contained 0.3% glycerine, 0.99% total glycerine. Monoglycerides were 1.49%, diglycerides 4.23% and triglycerides 0.99%. Alcohol content was only 0.012%. The remaining catalyst measured 32 microg/gm.

Fuel Characterization

A complete summary of the fuel characterization data is listed in Table 1 for the HySEE and the reference diesel fuel used for this study. Some comparisons include:

Viscosity - HySEE had a viscosity 1.9 times that of D2.

Cloud and Pour Point - HySEE had a cloud point 19 degrees Celsius higher than D2 and a pour point 23 degrees higher than D2.

Sulfur - HySEE had 1.56 times less sulfur than the low sulfur diesel fuel used for comparison.

Heat of Combustion - HySEE has 12.3 percent less energy on a mass basis than D2. Since HySEE has a 4.1 percent higher specific weight, the energies average 8.2 percent lower on a volume basis.

HySEE has an apparent molecular weight of 306.95 compared to D2 at 198. As the molecular weight increases so do the cetane number and viscosity.

Injector Coking

A visual inspection of the injector tips would indicate no difference between the HySEE and diesel fuel. However, the numerical scales show that diesel has an injector coking index of one and HySEE has an index of 3.05 (for comparison in these tests, Rape Ethyl Ester had an injector coking index of 3.16) The coking index is an average of three runs, four injectors for the four cylinder engine, and two orientations for a total of 24 samples averaged for each fuel (Table 2). The overall injector coking is low, especially when compared with older tests that included runs with raw vegetable oil.

Engine Durability Screening Test

HySEE Engine -- The initial power was set at 2800 watts with the engine operating at 2100 RPM under a load condition and 2250 RPM under a no-load condition. During the second night of operation the ambient conditions were such that the HySEE gelled and shut the engine down for approximately two hours. A drum heater was added to the drum of fuel, a new fuel filter was installed and the engine was restarted. On June 30, the circuit breaker tripped and 19.4 hours were not logged on the hour meter. The engine was running at full RPM but was not loaded during this time. Ten hours, about one half of the time the circuit breaker was tripped, was added to the end of the test to compensate for the 19.4 hours for which there was no loading. On July 3 the engine shut down again with the probable cause being cold weather gelling the fuel.

During the first one half of the test, when the engines were set at the same load, the engine high RPM under no-load condition was 2300 and under load was 2200 RPM. The engine produced a load of 2820 watts and consumed fuel at a rate of 1.13 L/hr (0.299 gph).

During the second half of the test, both engines were set at the same high RPM's, the engine high RPM under no-load condition was 2270 and under load was 2140 RPM. The engine produced a load of 2950 watts and consumed fuel at a rate of 1.14 L/hr (0.3 gph). For the entire test a total of 340 L (90 gal) gallons of fuel were consumed and 309 hours were logged. The engine was shut down and restarted twice for oil changes.

After completing the 300 hour endurance testing the engine was disassembled and inspected for wear and compared to the engine operating on 100% diesel fuel.

Diesel Engine -- The initial power was set at 2800 watts with the engine operating at 2200 RPM under a load condition and 2300 RPM under a no-load condition.

During the first half of the test, when the engines were set at the same load, the engine high RPM under no-load condition was 2300 and under load was 2200 RPM. The engine produced a load of 2820 watts and consumed fuel at a rate of 0.84 L/hr (0.223 gph)..

During the second half of the test, both engines were set at the same high RPM's, the engine high RPM under no-load condition was 2270 and under load was 2160 RPM. The engine produced a load of 2860 watts and consumed fuel at a rate of 0.85 L/hr (0.224 gph). For the entire test 255 L (67.5 gal) of fuel were used and 302 hours were logged. The engine was shut down and restarted twice for oil changes.

The HySEE fueled diesel engine consumed 25% more fuel than that of the diesel fueled engine. The HySEE fueled engine shutdown twice, presumably due to cool weather, and produced a significantly greater amount of visible exhaust smoke.

On the average HySEE showed a slight reduction in NO_x, a significant reduction in HC and CO, and a slight increase in PM and CO₂. The PM data even though different was significantly variable that it was not significantly different from diesel.

CONCLUSIONS

A complete set of fuel characteristics for HySEE and diesel are presented. Performance tests demonstrated that HySEE can be used to successfully fuel a diesel engine. In general, the testing performed has shown that torque and power are reduced about 5 percent compared to D2 and fuel consumption is increased 7 percent.

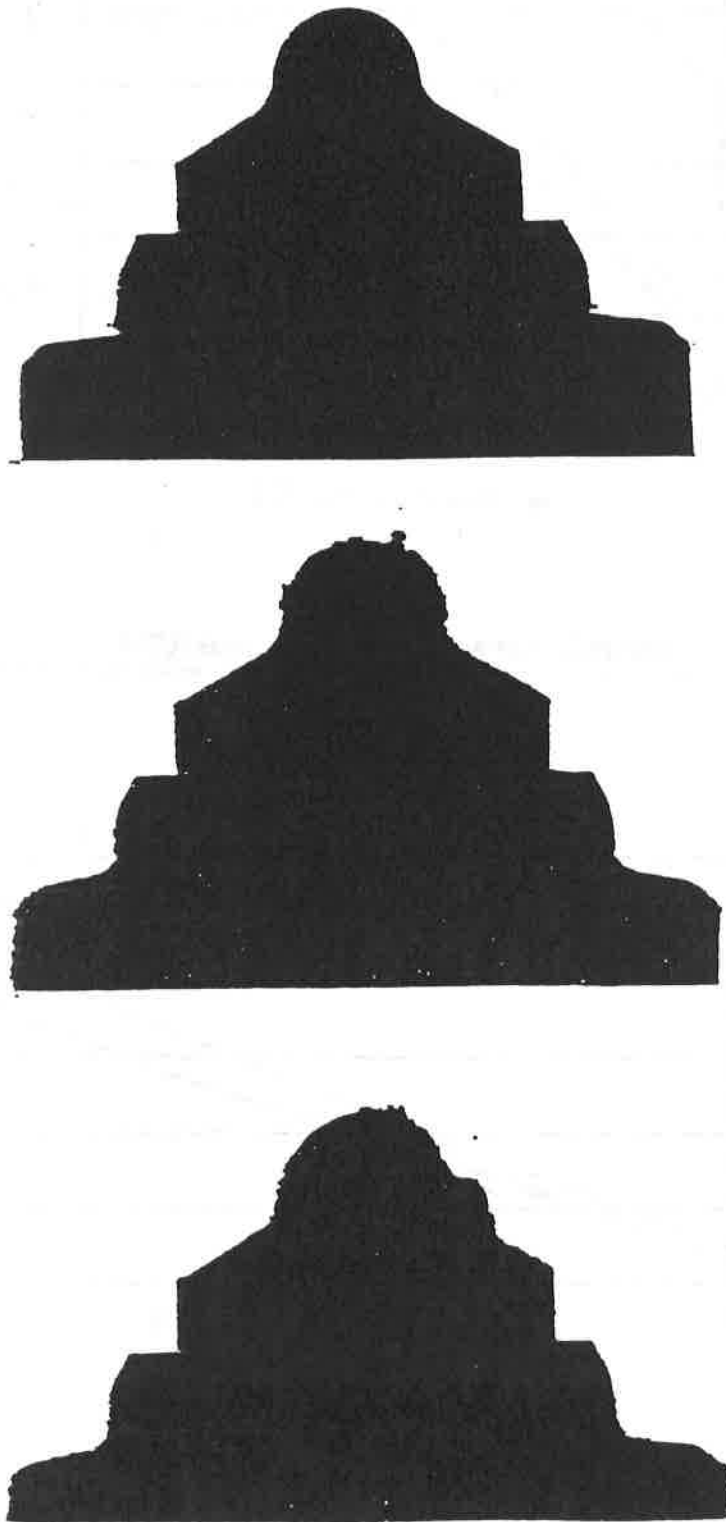
Specific conclusions of this study are:

1. Fuel characterization data show some similarities and differences between HySEE and D2. a) Specific weight is higher for HySEE, viscosity is 1.9 times that of D2 at 40°C (104°F), and heat of combustion is 12% lower than D2. b) Sulfur content For HySEE is 36% less than D2.
2. The average HySEE injector coking index was 3.07 and D2 was 1.00. Visually, all injector coking was low especially compared with older tests that included raw vegetable oils.
3. Opacity was decreased by as much as 71 percent compared to D2.
4. At rated load, engine power produced by HySEE decreased by 4.8 percent compared to D2.
5. Peak torque for HySEE at 1700 RPM was reduced by 6 percent compared to D2 while at 1300 RPM it was reduced only 3.2 percent, demonstrating a flatter torque curve characteristic of Biodiesel.
6. The average fuel consumption (g/s) on a mass basis was 7 percent higher than that of D2. The differences in fuel consumption and power reflect the differences in heat of combustion and density between the two fuels.
7. Thermal efficiencies for HySEE and D2 were not significantly different.
8. Emissions tests showed a 54 percent decrease in HC, 46 percent decrease in CO, 14.7 percent decrease in NO_x, 0.57 percent increase in CO₂ and a 14 percent increase in PM when HySEE was compared to D2. The HC, CO and NO_x differences were statistically significant.

Table 1
Fuel Characterization

	D2	HySEE
Fuel Specific Properties		
Specific Gravity, 60/60	0.8495	0.8716
Viscosity, cs @ 40°C	2.98	5.78
Cloud Point, °C	-12	9
Pour Point, °C	-23	8
Flash Point, PMCC, °C	74	124
Boiling Point, °C	191	273
Water and Sediment, % Vol.	<0.005	<0.005
Carbon Residue, % mass	0.16	0.06
Ash, % mass	0.002	0.002
Sulfur, %wt	0.036	0.014
Cetane Number	49.2	61
Heat of Combustion, MJ/kg		
Gross	45.42	40.51
Net	42.9	37.20
Copper Corrosion	1A	1A
Karl Fischer Water, ppm	38	877
Particulate Matter, mg/L		
Total	0.9	6.4
Non-Combustible	<0.1	1.5
Elemental Analysis		
Nitrogen, ppm		12
Carbon, %	86.67	77.72
Hydrogen, %	12.98	12.34
Oxygen, % (by difference)	0.33	9.92
Acid Value	0.128	0.165
Iodine Number	8.6	63.5
Ester Specific Properties		
Percent Esterified		92.26
Free Glycerine, %wt		0.3
Total Glycerine, %wt		0.99
Free Fatty Acids, %wt		0.38
Monoglycerides, %wt		1.49
Diglycerides, %wt		4.23
Triglycerides, %wt		1.42
Alcohol Content, % mass		<1
Catalyst, microgram/gram		32
Fatty Acid Composition, %		
Palmitic (16:0)		10.3
Stearic (18:0)		15.0
Oleic (18:1)		24.6
Linoleic (18:2)		48.6
Eicosenoic (20:1)		0.3

Figure 1. Typical injector coking photographs, clean (top), diesel (middle), HySEE (bottom).



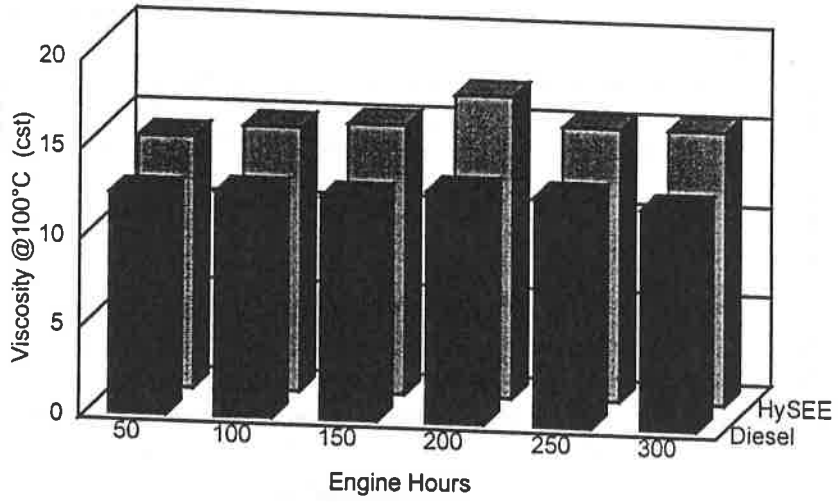


Figure 6. Engine oil viscosity at 50 hour oil change intervals

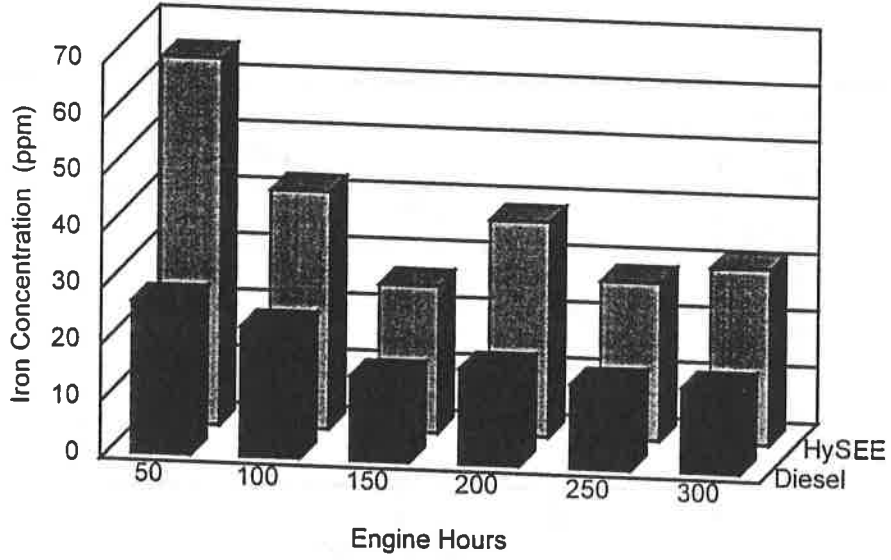


Figure 7. Concentration of iron in engine oil analysis

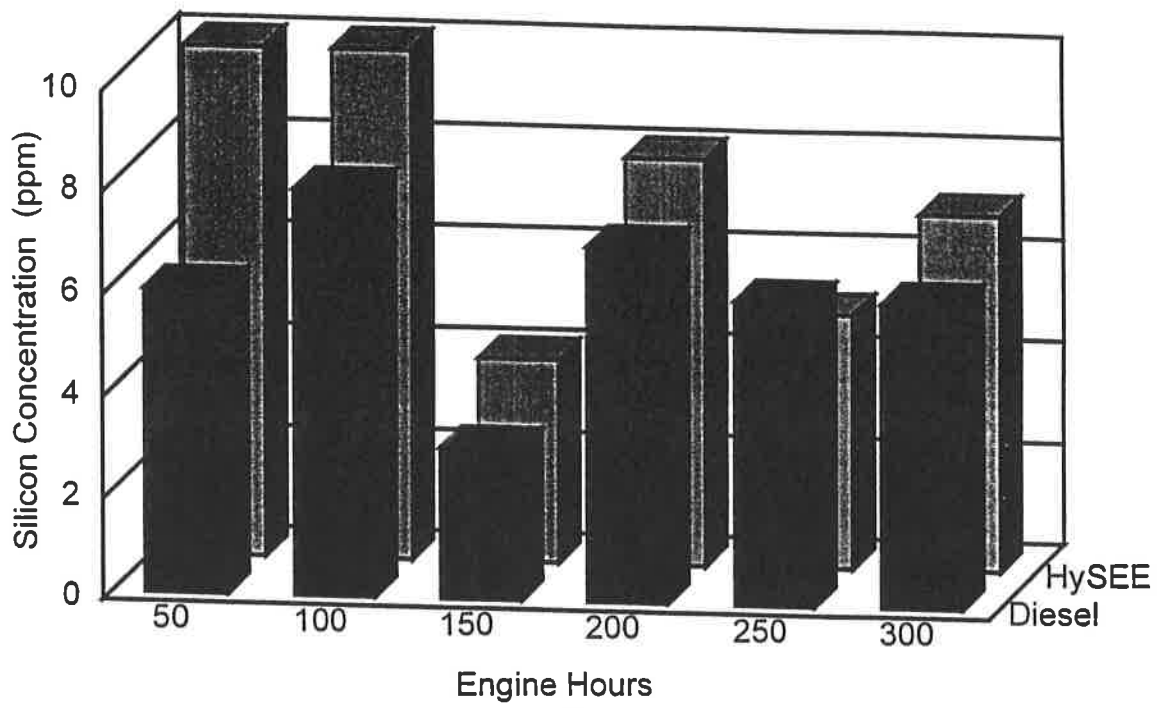


Figure 8. Concentration of silicon in engine oil analysis

