

**PROCESSING, CHARACTERIZATION & PERFORMANCE
OF EIGHT FUELS FROM LIPIDS**

by

Charles L. Peterson
Professor

Daryl L. Reece
Engineering Technician

Brian J. Hammond
Graduate Research Assistant

Joseph Thompson
Engineering Technician

Sidney M. Beck
Professor Emeritus of Bacteriology

**Department of Agricultural Engineering
University of Idaho
Moscow, Idaho 83844-2040**

**Written for Presentation at the
1994 ASAE International Winter Meeting
Sponsored by ASAE**

**Atlanta, Georgia
December 13-16, 1994**

Summary:

Test quantities of ethyl and methyl esters of rapeseed oil, soybean oil, canola and tallow were produced. A complete set of fuel properties and a comparison of each of these fuels in short term engine performance tests were studied. Seventy gallons of ethyl and methyl esters of each feedstock were produced. Each quantity of fuel was characterized using the ASAE EP X552 for reporting of fuel properties. Short term engine performance tests were conducted in a John Deere 4239T engine connected to an electric dynamometer.

Keywords:

Biodiesel, Rapeseed Canola, Soybean, Tallow

The author(s) is solely responsible for the content of this technical presentation. The technical presentation does not necessarily reflect the official position of ASAE, and its printing and distribution does not constitute an endorsement of views which may be expressed.

Technical presentations are not subject to the formal peer review process by ASAE editorial committees; therefore, they are not to be presented as refereed publications.

Quotation from this work should state that it is from a presentation made by (name of author) at the (listed) ASAE meeting.

EXAMPLE – From Author's Last Name, Initials. "Title of Presentation." Presented at the Date and Title of meeting, Paper No. X. ASAE, 2950 Niles Rd., St. Joseph, MI 49085-9659 USA.

For information about securing permission to reprint or reproduce a technical presentation, please address inquiries to ASAE.

Processing, Characterization & Performance of Eight Fuels from Lipids

C. L. Peterson, D. L. Reece, B. L. Hammond, J. Thompson, S.M. Beck¹

ABSTRACT

Test quantities of ethyl and methyl esters of four renewable fuels were processed, characterized and performance tested. Canola, rapeseed, soybean oils, and beef tallow were the feedstocks for the methyl and ethyl esters. Previous results have shown methyl esters to be a suitable replacement for diesel fuel; however, much less has been known about the ethyl esters. A complete set of fuel properties and a comparison of each fuel in engine performance tests are reported. The study examines short term engine tests with both methyl and ethyl ester fuels compared to number 2 diesel fuel (D2). Three engine performance tests were conducted including an engine mapping procedure, an injector coking screening test, and an engine power study.

The gross heat contents of the Biodiesel fuels, on a mass basis, were 9 to 13 percent lower than D2. The viscosities of Biodiesel were twice that of diesel. The cloud and pour points of D2 were significantly lower than the Biodiesel fuels. The Biodiesel fuels produced slightly lower power and torque and higher fuel consumption than D2.

In general, the physical and chemical properties and the performance of ethyl esters were comparable to those of the methyl esters. Ethyl and methyl esters have almost the same heat content. The viscosities of the ethyl esters is slightly higher and the cloud and pour points are slightly lower than those of the methyl esters. Engine tests demonstrated that methyl esters produced slightly higher power and torque than ethyl esters. Fuel consumption when using the methyl and ethyl esters are nearly identical. Some desirable attributes of the ethyl esters over methyl esters were: significantly lower smoke opacity, lower exhaust temperatures, and lower pour point. The ethyl esters tended to have more injector coking than the methyl esters and the ethyl esters had a higher glycerol content than the methyl esters.

INTRODUCTION

Vegetable oil as an alternative fuel has been under study at the University of Idaho since 1979 (Peterson et al., 1990). Since then researchers at Idaho have pioneered the use of rapeseed oil as a diesel fuel substitute. Although short term tests using neat

¹ The Authors are professor of Agricultural Engineering, Engineering Technician, Graduate Research Assistant, Engineering Technician, and Professor Emeritus of Bacteriology respectively from the University of Idaho Department of Agricultural Engineering, Moscow, Idaho 83844.

catalyst during the reaction was necessary to get satisfactory results. The application of heat during the reaction is not economically sound because of the additional cost and reduced energy efficiency.

Nye and Southwell (1983) were the only workers to report a successful process for the transesterification of rapeseed oil at room temperature by systematically optimizing the other variables. In Idaho, a considerable number of graduate students have investigated the optimization of the reaction variables temperature, agitation time, catalyst amount, ratio of alcohol to rapeseed oil and degree of lipid conversion (Bam, 1991; Feldman, 1991; Jo, 1984; Madsen, 1985; Melville, 1987; Mosgrove, 1987; Perkins et al., 1991). They have confirmed the works of Nye and Southwell. Based on their bench-scale results, workers at the UI Agricultural Engineering Department developed a small pilot plant system for rapeseed methyl and ethyl ester production (Peterson et al., 1991). The reactor is also utilized as a washing tank for the ester. A separate alcohol-catalyst mixer, made of a 208 liter plastic barrel, serves as an accessory to the reactor. The reactor and the oil press constitute the farm-scale rapeseed oil and Biodiesel processing plant.

Ethanol will produce a more environmentally benign fuel. The Dangerous Properties of Industrial Materials (Sax, 1975) reports,

The systemic effect of ethyl alcohol differs from that of methyl alcohol. Ethyl alcohol is rapidly oxidized in the body to carbon dioxide and water, and in contrast to methyl alcohol no cumulative effect occurs. Methyl alcohol...once absorbed is only very slowly eliminated. ...in the body the products formed by its oxidation are formaldehyde and formic acid, both of which are toxic. Because of the slowness with which it is eliminated, methyl alcohol should be regarded as a cumulative poison.

Ethanol is also a preferred alcohol in this process compared to methanol because it is derived from agricultural products and is renewable and biologically less objectionable in the environment. Success of rapeseed ethyl ester (REE) production would mean that Biodiesel's two main raw materials would be agriculturally produced, renewable and environmentally friendly.

Engine Performance Tests: In a summary of 22 short term engine tests conducted at 12 locations worldwide (Peterson, 1986) in which vegetable oil was compared to diesel as a fuel, peak engine power on the vegetable oil fuels ranged from 91 to 109 percent of that produced when the same engine was operated with diesel fuel. In these tests, 16 of the 22 reported peak power equal to or exceeding that when the engines were operated on diesel. Fuel consumption was generally slightly higher, reflecting the reduced energy content of the vegetable oil. Thermal efficiencies are also generally reported to be slightly higher than for diesel fuel. Peterson et al. (1987) ran a series of short term engine tests to evaluate the effects of transesterification of winter rapeseed

OBJECTIVES

1. Produce test quantities of ethyl and methyl esters of rapeseed oil, soybean oil, canola, and tallow using the two procedures currently developed.
2. Determine the complete set of fuel specifications on each of the fuels according to the requirements set forth in the proposed ASAE Engineering Practice, ASAE EP X552.
3. Compare the performance of each of these fuels in short term engine performance tests.

MATERIALS AND METHODS

Seventy gallons of each of the esters were produced using the process developed by University of Idaho researchers. The feedstocks for these fuels were as follows: rapeseed from Dwarf Essex variety seed; canola from Stonewall variety seed; beef tallow purchased from Iowa Beef Products in Kennewick, Washington; and soybean oil purchased from Foodservices Brokerage Co. in Spokane Washington. In addition to these eight fuels, seventy five gallons of methyl soyate were purchased from Interchem, Inc., Overland Park, Kansas (Midwest Biofuels). The rapeseed and canola oils were expelled at the University of Idaho's Agricultural Engineering farm scale process facility. Each fuel, excluding the methyl soyate, was processed at this facility. Phillips 66 Company low sulfur diesel reference fuel was used as the baseline fuel.

The nomenclature for these fuels is as follows: R - rapeseed, C - canola, T- tallow, S - soybean, with the following letters ME for methyl ester and EE for ethyl ester. MWF represents Midwest Biofuels methyl soyate, and D2 - Phillips low sulfur diesel reference fuel.

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 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.

Vegetable Oil Fuels" (Korus et al, 1985). The engine was operated for ten minutes at each interval for data collection.

Torque Tests

The torque tests were performed with the engine operating at 2600 RPM to 1300 RPM in 100 RPM increments with the same data collection procedure as previously described. The engine was operated for 2 1/2 minutes at each interval for data collection.

Mapping Engine Performance

The engine mapping tests were performed using the procedure described in "Procedure for Mapping Engine Performance-Spark Ignition and Compression Ignition Engines" (SAE J1312, 1990). The mapping tests were performed at 2500, 2250, and 2000 RPM with loadings of 100, 75, 50, 25, and 0 percent of maximum power. The engine was operated for 5 minutes at each data collection interval.

Experimental Design

The engine performance data was collected using a randomized complete block experimental design. Each fuel was tested once in each block in random order for each of the three blocks. This resulted in a total of 30 injector coking tests, 30 torque tests, and 30 fuel mapping studies.

PROCEDURES

Fuel Preparation

The eight Biodiesel fuels were processed in a batch type reactor. The methyl ester process utilizes 100 percent molar excess alcohol (preferably absolute or 100 percent pure), or a molar ratio of 6:1 alcohol to oil ratio. Based on the amount of input oil by weight, 1.1 percent of potassium hydroxide (KOH) is used. The following equations were used for the quantities processed:

$$\text{MeOH} = 0.225 \times \text{Oil}$$

$$\text{KOH} = \text{Oil}/100$$

where : Oil = desired amount of oil, in liters
 MeOH = amount of methanol needed, in liters
 KOH = amount of potassium hydroxide required, in kg

RESULTS

A total of over 150 hours were logged on the John Deere diesel engine and 2,250 liters of fuel were consumed during the performance testing.

Fuel Characterization

A complete summary of the fuel characterization data is listed in table 1 for each of the fuels used in this study. Comments on each parameter would be excessively lengthy; however, some deserve attention.

Viscosity -The Biodiesel fuels had viscosity from 1.3 to 2.1 times that of D2. SME and MWF had the lowest viscosities of the biodiesels and RME and REE the highest viscosities.

Cloud and Pour Point - All the biodiesels have higher cloud and pour points than D2. RME and REE had the lowest pour points only 1 and 5 degrees respectively higher than D2 while the tallow esters were 28 and 32 degrees higher than D2. The soy esters were 13 to 19 degrees higher than D2.

Sulfur - All of the Biodiesel fuels contain considerably less sulfur than even the low sulfur diesel fuel used for comparison. The Biodiesel fuels were 0.55 to 0.22 that of D2.

Heat of Combustion - All of the Biodiesel fuels are lower in heat content than D2 by an average of 11.8 percent on a mass basis. Since the Biodiesel fuels have a 4.1 percent higher specific weight, the energies average 8.2 percent lower on a volume basis.

Percent Esterified - The methyl esters were 97.5 percent esterified while the ethyl esters were only 94.3 percent esterified. SME and RME had the highest esterified values of the methyl ester and TEE the highest of the ethyl esters. CEE was the lowest level.

Total Glycerol - Glycerol levels were consistently higher than the 0.25 percent allowed in the proposed ASTM standard based on the analysis provided. SEE was highest at 1.88 percent and TME lowest at 0.6 percent. The average total glycerol was 0.87 percent for the methyl esters and 1.4 percent for the ethyl esters. Note that the commercial Biodiesel had a total glycerol content of 1.25 percent.

Alcohol and Catalyst - All of the Biodiesel fuels had less than one percent alcohol. Residual catalyst varied from 11 to 36 parts per million (ppm).

Figure 8 is a summary of the brake mean effective pressure (bmep) versus fuel consumption for all of the Biodiesel fuels compared to D2. Bmep is useful for comparing performance parameters in engines. The scatter of dots is each Biodiesel data point. A line is drawn through the average of the Biodiesel fuel consumption. The average Biodiesel fuel consumption is 7 percent higher than that of diesel fuel. Figure 9 compares thermal efficiencies versus bmep for the Biodiesel fuels compared to D2. Biodiesel fuels have a slightly higher thermal efficiency compared to D2 on the mid power range.

CONCLUSIONS

A complete set of fuel characteristics for a variety of Biodiesel fuels and D2 are presented. Performance tests demonstrated that these fuels are similar to diesel fuel. In general, the testing performed has shown that torque and power are similar to D2 and as the molecular weight of the Biodiesel decreases so does the torque and power. Injector coking is greater for the ethyl esters which are also higher in total glycerol, even though with linear regression there is no correlation based on glycerol content alone. As the heat of combustion for the Biodiesel fuels increases so does the viscosity and molecular weight.

In general, the physical and chemical properties and the performance of ethyl esters are comparable to those of the methyl esters. Ethyl and methyl esters have almost the same heat content. The viscosity of ethyl esters are slightly higher and the cloud and pour points are slightly lower than methyl esters. Engine tests demonstrate that methyl esters produced slightly higher power output and torque than ethyl esters. Fuel consumption when using the two different esters is nearly identical. Some desirable attributes of the ethyl esters over methyl esters are significantly lower smoke opacity, lower exhaust temperatures, and lower pour point.

Specific conclusions of this study are:

1. Fuel characterization data show some similarities and differences between Biodiesel fuels and diesel. a) Specific weight is higher for Biodiesel, heat of combustion is lower, viscosities are 1.3 to 2.1 times that of D2. b) Pour points for Biodiesel fuels vary from 1 to 25 degrees Celsius higher for Biodiesel fuels depending on the feedstock. c) Sulfur content for Biodiesel is 20 to 50 percent that of D2.
2. The percent oil esterified as determined by an outside lab was lower than expected. Methyl esters averaged 97.5 percent and ethyl esters 94.3 percent esterified.
3. Total glycerol was higher than expected averaging 1.1 percent. Methyl esters averaged 0.87 percent and the ethyl esters 1.4 percent.

REFERENCES

- Bam, N.B. 1991. Process Development of Rapeseed Oil Ethyl Ester as a Diesel Fuel Substitute. M.S. Thesis. Chem Engr Dept, University of Idaho, Moscow, ID 83843.
- Chancellor, W.J., and N. Reubach. 1985. Vegetable Oil Methyl Esters for Fuel. Unpublished manuscript, Univ. of California. Davis, CA 95616.
- Clark, S.J., L. Wagner, M.D. Schrock, and P.G. Piennaar. 1984. Methyl and Ethyl Soybean Esters as Renewable Fuels for Diesel Engines. *J of Am Oil Chemist Soc.* 61(10):1632-1637.
- Du Plessis, L.M., and J.B.M. De Villiers. 1985. Stability Studies on Methyl and Ethyl Fatty Esters of Sunflower seed Oil. *J of Am Oil Chemist Soc.* 62(4):748-751.
- Einfalt, J., C.E. Goering. 1985. Methyl Soyate as a Fuel in a Diesel Tractor. *Transactions of the ASAE* 28(1):70-74.
- EMA. 1982. 200-Hour Screening Test for Alternate Fuels. *Energy News.* Northern Agricultural Energy Center, Peoria, IL 61064.
- Feldman, M. 1991. Optimization of a Direct Injection Diesel Engine for Operation on Winter Rape Methyl Ester. M.S. Thesis, Ag Engr Dept, University of Idaho, Moscow, ID 83843.
- Feuge, R.O., and A.T. Gros. 1949. Modification of Vegetable Oil VII: Alkali catalyzed Interesterification of Peanut Oil with Ethanol. *J of Am Oil Chemist Soc.* 26:97-102.
- Formo, M.W. 1954. Ester Reactions of Fatty Materials. *J. of Am. Oil Chemist Soc.* 31:548-559.
- Freedman, B., and E.H. Pryde. 1982. Fatty Esters from Vegetable Oils for Use as a Diesel Fuel. *Vegetable Oil Fuels, Proc. of the international conference on plant and vegetable oil as fuels.* ASAE publication 4-82. pp 117-122.
- Freedman, B., E.H. Pryde, and T.L. Mounts. 1984. Variables Affecting the Yields of Fatty Acid Esters from Transesterified Oils. *J of Am Oil Chemist Soc.* 61:1638-1645.
- Harrington, K.J., and C. D'Arcy-Evans. 1985. A Comparison of Conventions and Insitu Methods of Transesterification of Seed Oil from a Series of Sunflower Cultivators. *J of Am Oil Chemist Soc.* 62: 1009-1018.
- Jo, Jaiduc. 1984. Improving Storage and Use of Vegetable Oil for Fuel. M.S. Thesis. Chemical Engr Dept, University of Idaho, Moscow, ID 83843.
- Kaufman, K.R., and M. Ziejewski. 1984. Sunflower Methyl Ester for Direct Injected Diesel Engines. *Transactions of the ASAE* 27(6): 1626-1633.
- Korus, R.A., J. Jo, and C.L. Peterson. 1985. A rapid engine test to measure injector fouling in diesel engines using vegetable oil fuels. *JAOCs, Vol.62 no. 11.*
- Kusy, P.F. 1982. Transesterification of Vegetable Oils for Fuels. *Vegetable Oil Fuels, Proc. of the international conference on plant and vegetable oil as fuels.* ASAE publication 4-82. pp 127-137.

Sax, N. Irving. 1975. *Dangerous Properties of Industrial Materials*, 4th Ed., Van Nostrand Reinhold.

Stern, R., G. Hillion, P. Gateau, and J.C. Guibet. 1986. Preparation of Methyl and Ethyl Esters from Crude Vegetable Oils and Soapstone. In: *Proc of the World Conference on Emerging Technol. Fats, Oils Ind* (A. Richard Baldwin, Editor), Am Oil Chemist Soc., Champaign, IL. pp 420-422.

Vinyard, S., E.S. Rendl, J.S. Goodling, L. Hawkins, and R.C. Bunt. 1982. Properties and Performance Testing with Blends of Biomass Alcohols, Vegetable Oils and Diesel Fuel. ASAE publication 4082. ASAE, St. Joseph, MI 49085.

Wagner, L.E., S.J. Clark, and M.D. Schrock. 1984. Effects of Soybean Oil Esters on the Performance, Lubricating Oil, and Wear of Diesel Engines. SAE Technical Paper Series 841385. 400 Commonwealth Drive, Warrendale, PA 15096.

Zhang, Q., M. Feldman and C. L. Peterson. 1988. Diesel Engine Durability when Fueled with Methyl Ester of Winter Rapeseed Oil. ASAE paper no. 88-1562, ASAE, St. Joseph, MI 49085-9659.

Table 2
Hypothetical Formulas, Apparent Molecular Weights and
Fatty Acid Compositions of the Fuels Tested.

Fuel	Hypothetical Formula	Molecular Weight
CEE	$C_{20}H_{37}O_2$	309.4
CME	$C_{19}H_{35}O_2$	295.3
MWF	$C_{19}H_{34}O_2$	292.2
REE	$C_{22}H_{43}O_2$	340.1
RME	$C_{21}H_{38}O_2$	323.4
SEE	$C_{20}H_{36}O_2$	306.4
SME	$C_{19}H_{34}O_2$	292.4
TEE	$C_{19}H_{38}O_2$	300.8
TME	$C_{18}H_{36}O_2$	286.7

Table 3
Injector Coking Compared with Viscosity, Percent Esterified, Total Glycerol,
and Heat of Combustion for the fuels tested.

	Injector Coking	Viscosity @ 40°C	Percent Esterified	Total Glycerol	Heat of Combustion
CEE	2.88	4.89	92.31	1.18	40.03
CME	2.17	4.75	96.35	0.87	39.90
MWF	2.15	3.90	97.11	1.25	39.61
REE	3.16	6.17	94.75	0.93	40.15
RME	3.08	5.65	98.02	0.86	40.54
SEE	2.18	4.49	94.54	1.88	39.96
SME	2.14	3.89	98.17	0.75	39.77
TEE	3.06	5.04	95.62	1.42	40.09
TME	2.18	4.81	97.80	0.60	39.92
DIESEL	1.00	2.98			45.42

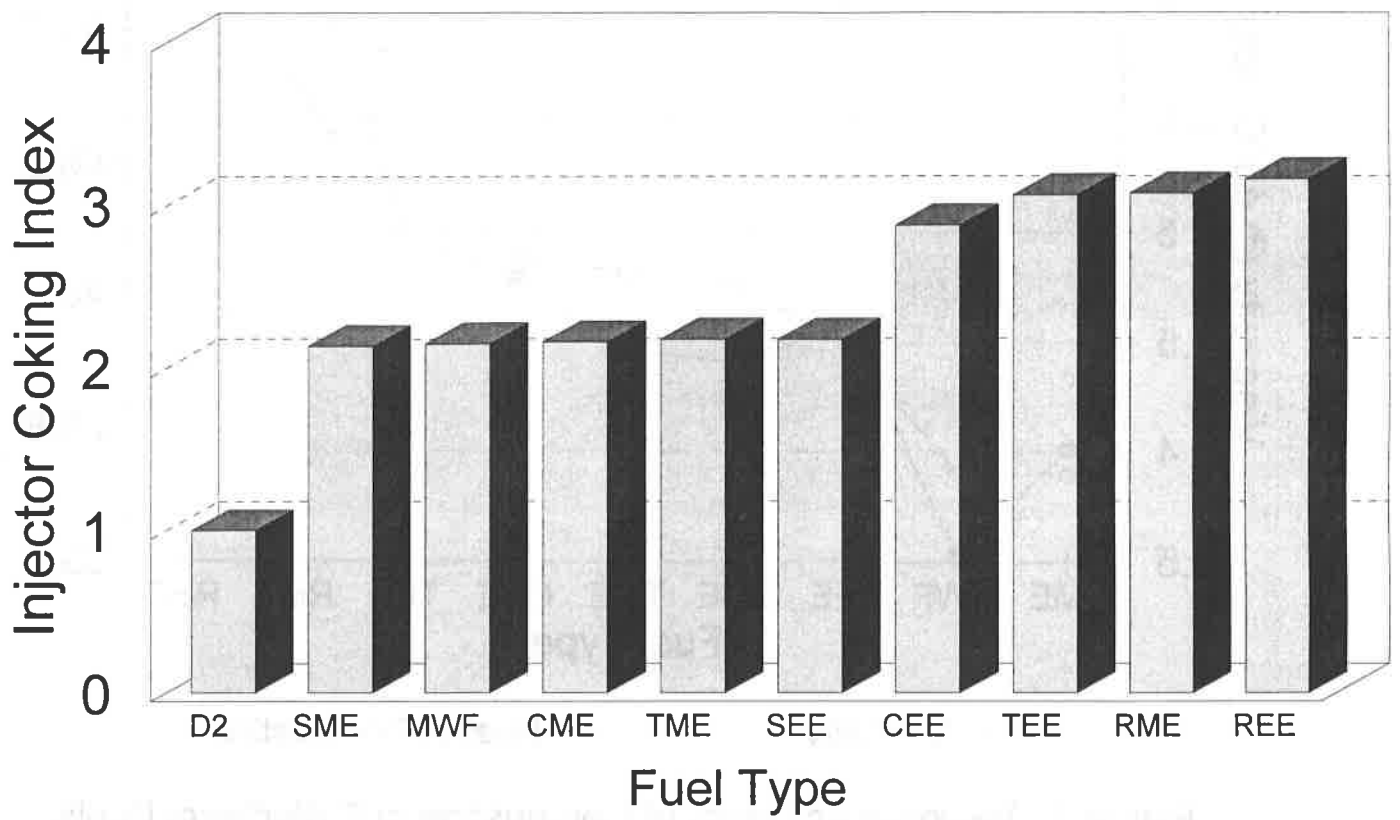


Figure 2. Injector coking versus fuel type for 9 biodiesel fuels and D2.

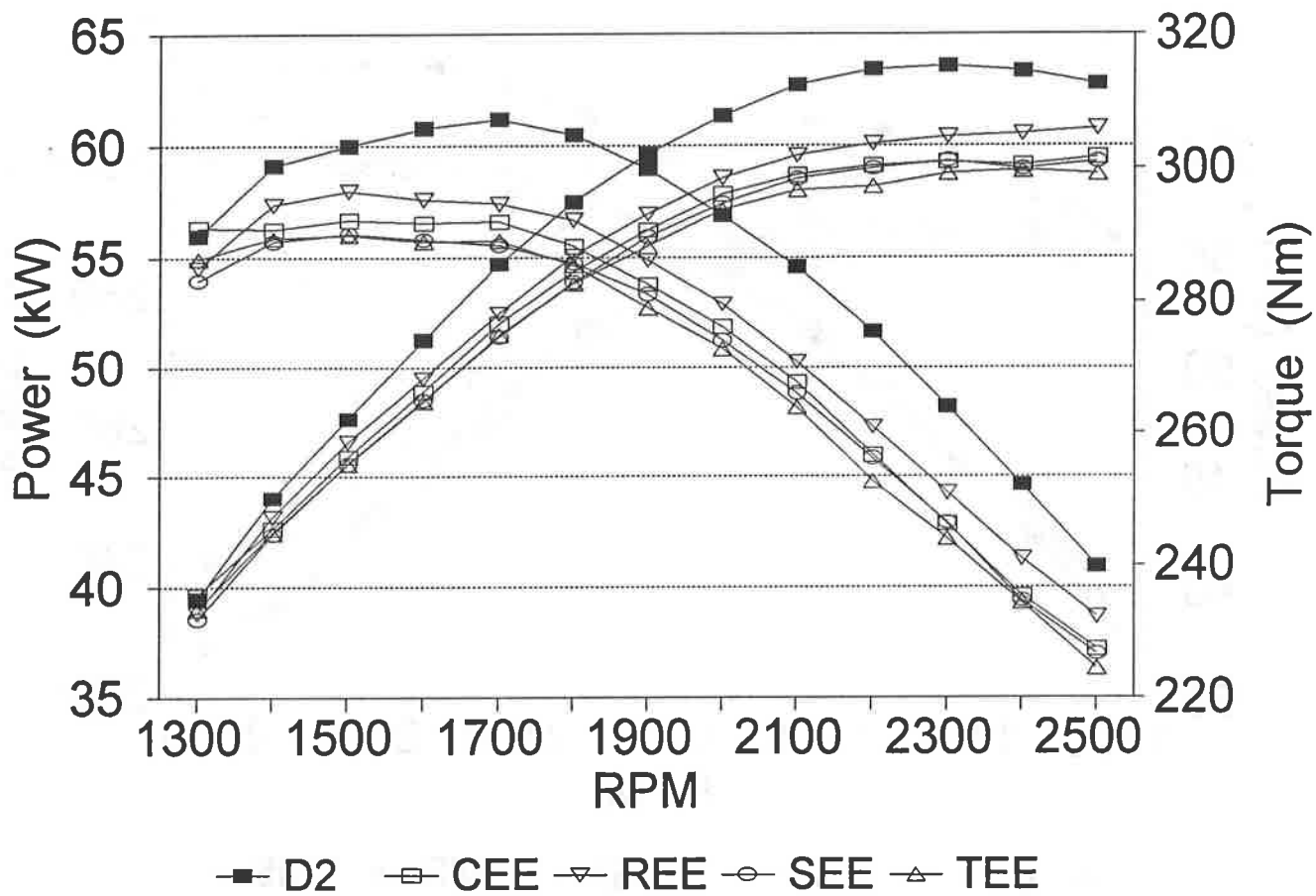


Figure 4. Power and Torque for Ethyl Ester Biodiesel Fuels and D2.

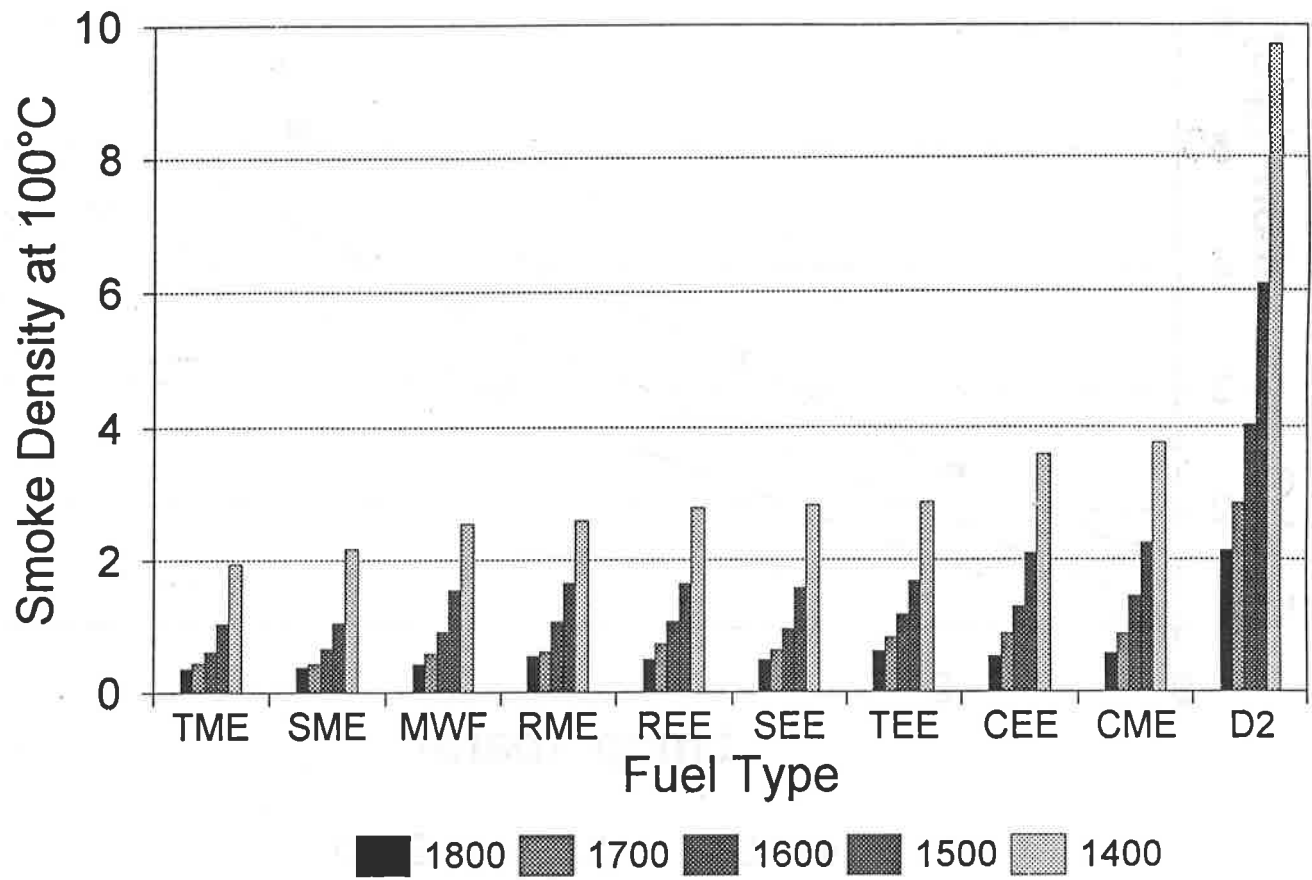
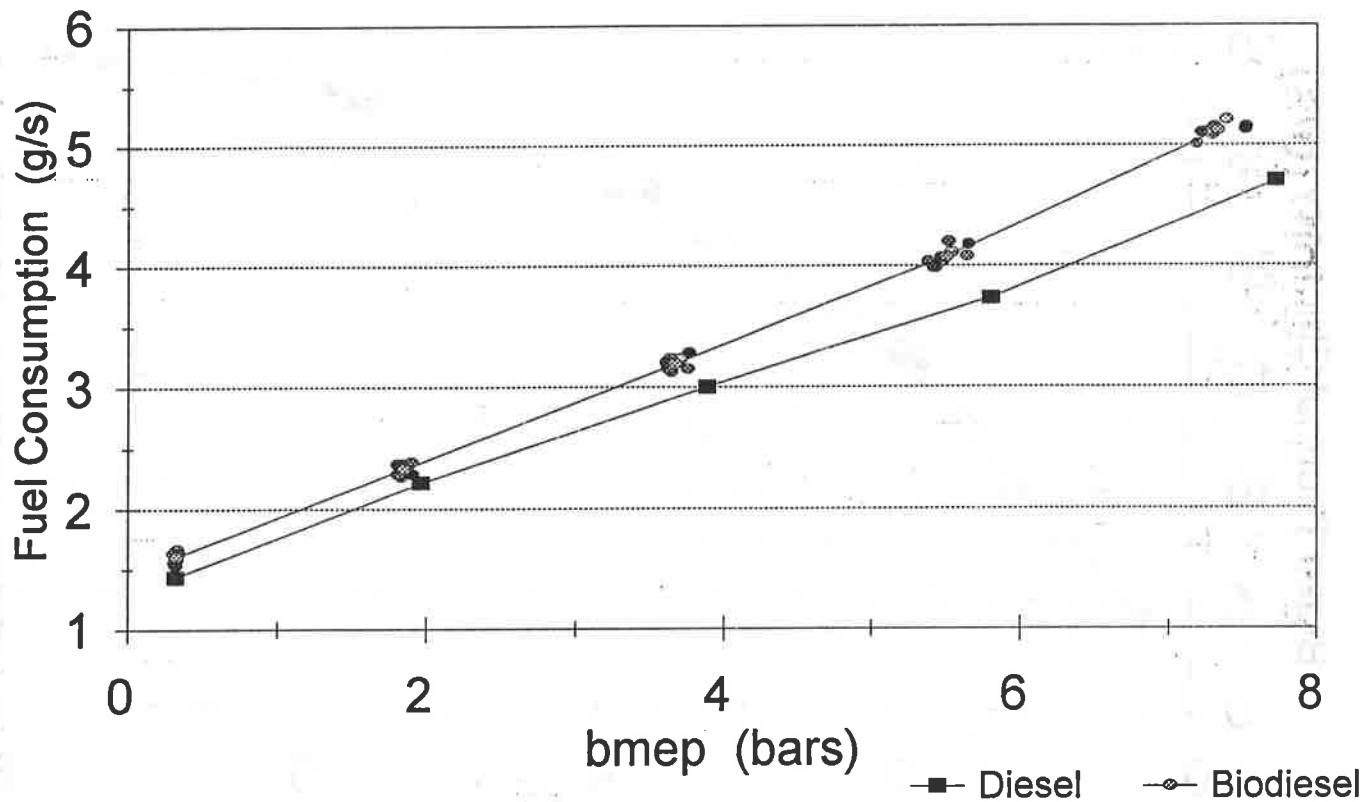


Figure 6. Smoke Density from 9 Biodiesel Fuels and D2 as Measured in a Torque test. Data shown is for 1800 to 1400 RPM.



Average Biodiesel fuel consumption = 107% of diesel at the same % load

Figure 8. Fuel Consumption versus bmep for the 9 Biodiesel Fuels and D2.
Data from mapping test at 2500 RPM.