

Cetane Number Testing of Biodiesel

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ABSTRACT

Reported values of the cetane number for biodiesel vary widely. For soybean oil-derived biodiesel, the values range from 48 to as high as 67. Some of this variation may be due to differences in the fatty acid composition of the soybean oil but other factors are also important. This paper reviews the historical development of the cetane number test procedure and its current status. It discusses the existing data for the cetane number of biodiesel and presents new data for the effect of the fatty acid composition and for fuel contaminants such as unsaponifiable matter and hydroperoxides. Data are presented for the cetane number increase resulting from blending small amounts of pure fatty esters with diesel fuel. These data indicate that the cetane number is most strongly affected by the saturation level of the fatty acids. The unsaponifiable matter does not affect the cetane number but higher levels of hydroperoxides increase the cetane number.

Keywords. Diesel, Diesel Engine, Diesel Fuel, Methyl Esters

INTRODUCTION

Biodiesel, as used in this paper, is defined to be the alkyl monoesters of vegetable oils or animal fats. It is produced by chemically reacting the oil or fat with an alcohol in the presence of a catalyst. The products of this reaction are the monoesters, known as biodiesel, and glycerin, which is a high value byproduct. Because of its low cost, methanol is the most common alcohol used but ethanol is used in some cases. In Europe, rapeseed oil is the most common vegetable oil feedstock and in the United States, soybean oil is generally used. Biodiesel produced by reacting these two oils with methanol is called rapeseed methyl ester (RME) and soybean methyl ester (SME) (or methyl soyate). These more specific terms will be used when it is necessary to distinguish between biodiesels from different feedstocks.

The *cetane number* is one of the most commonly cited indicators of diesel fuel quality. It measures the readiness of the fuel to autoignite when injected into the engine. It is generally dependent on the composition of the fuel and can impact the engine's startability, noise level, and exhaust emissions.

The cetane number of biodiesel is generally observed to be quite high. Data presented below will show values varying between 45 and 67. In the United States, No. 2 diesel fuel usually has a cetane number between 40 and 45. The objective of this paper is to discuss the possible reasons for the wide range in reported values of the cetane number of biodiesel. Suggestions for reporting the fuel properties of biodiesel that correlate to cetane number are also provided.

Background of the Cetane Number Test

The cetane number test procedure has been subject to much criticism in recent years. Most of this criticism is based on the differences between the cetane test engine configuration and operating conditions and the configurations and operating conditions of modern engines. The cetane number test procedure now designated as ASTM D 613 grew out of a number of research programs to determine the ignition quality of diesel fuel in a similar manner to the octane test for gasoline. As a result of work to determine the causes of knock in spark-ignition engines, researchers in the 1920s were aware of the relationship between fuel type and a property known as the *spontaneous ignition temperature* (S.I.T.) (Callendar et al., 1926). They observed that this temperature was very

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dependent on the technique used to calculate it, and on other details of the measurement such as the size of the compression machine, whether air or oxygen was used, and the local turbulence level. Callendar (1926) observed that with fuels subject to compression ignition, the time is as important a variable as the temperature. The ignition temperature of a fuel will be different depending on the time it is held at that temperature.

The primary argument for an engine-based test appears to have been the inability to convert analytical data about the fuels, such as the S.I.T., to a practical judgment about the suitability of the fuel in a diesel engine. In 1933, a small group consisting primarily of representatives of petroleum-refining companies and calling itself the *Volunteer Group for C.I. Fuel Research*, met to expedite the development of a standardized method for measuring ignition quality. Acting on the recently published work of Boerlage and Broeze (1931, 1932) and Pope and Murdock (1932), the Volunteer group published their first report in 1935 (Rendel, 1935) that described a modified cylinder head and piston for the diesel C.F.R. (Cooperative Fuel Research) engine developed by Pope and Murdock. This design, incorporating a separate high-swirl combustion chamber with a volume that could be changed to vary the engine's compression ratio, is essentially the same as that used for cetane testing today.

Boerlage and Broeze (1931, 1932) can also be credited with the proposal to use blends of two reference fuels as standards for ignition quality in a manner that was similar to the then recently developed gasoline octane test. They proposed the alpha isomer of cetene and mesitylene (1,3,5-trimethylbenzene) as the primary reference fuels. It was found to be difficult to control the location of the double bond in cetene and this apparently caused significant variation in ignition quality. For this reason, normal-cetane (n-hexadecane), a straight-chain saturated hydrocarbon, was substituted for cetene. Alpha methylnaphthalene was also substituted for mesitylene and was itself replaced much later by heptamethylnonane which was assigned a cetane number of 15. The matching of the ignition quality of the unknown fuel to the ignition quality of a blend of reference fuels was intended to minimize the influence of engine variables. The Volunteer group presented detailed operational procedures and validation data in later reports (Rendel, 1936; Baxley and Rendel, 1938). The current ASTM D 613 method is essentially the same as that described in their final report presented in January 1938 (Baxley and Rendel, 1938).

RESULTS AND DISCUSSION

Table 1 shows the range of reported values for the cetane number of four different types of biodiesel. The range of values for SME varies from 45.0 to 67.0. The numbers in the brackets are the references from which the cetane numbers were taken.

The cetane number test procedure is complex and inherently involves considerable uncertainty so some of the variation can be attributed to experimental error. ASTM Test method D 613 states that for fuels with cetane numbers in the range of 44 to 56, the repeatability should be within 0.7 to 0.9 cetane number units (American Society for Testing and Materials, 1988). The repeatability is the difference between two measurements conducted by the same operator with the same apparatus, under the same test conditions, and using the same test material. ASTM says these limits should be exceeded by no more than 1 test in 20. However, the reproducibility limits, defined as the difference between two measurements conducted by two different operators, using different apparatus, but the same test material, are between 2.5 and 3.3. Clearly, the variation in cetane numbers shown in Table 1 cannot be entirely explained by experimental error.

Some of the cetane number variation shown in Table 1 might also be attributed to variations in feedstock. Soybean oil produced in different parts of the United States will have slightly different fatty acid compositions. Even seasonal weather differences can cause composition changes. However, these differences are much less than those between oils from different plant sources. For example, the variation between reported measurements of SME is much larger than the differences between SME and RME. To further investigate this issue, a series of tests were conducted to determine the effect of different fatty acids on the cetane number.

Table 1. Reported values of the cetane number for biodiesel

Soybean Methyl Ester	Soybean Ethyl Ester	Rapeseed Methyl Ester	Rapeseed Ethyl Ester
45.0 [1]	48.2 [2]	51.9 [9]	64.9 [6]
46.2 [2]	52.7 [6]	48.0 [10]	59.7 [16]
54.7 [3]		54.4 [11, 12]	
67.0 [4]		49.9 [13]	
45.0 [5]		54.5 [14]	
54.8 [6]		54-65 [15]	
60.0 [7]		61.2 [6]	
51.9 [8]		61.8 [16]	
48.6 [8]			

[1] Reed, 1993.

[2] Wagner, et al, 1984.

[3] McDonald, et al, 1995.

[4] Marshall, et al, 1995.

[5] Pischinger, et al, 1982.

[6] Peterson, et al, 1994.

[7] Sharp, 1994

[8] Midwest Biofuels, 1993

[9] Gragg, 1994

[10] Rantanen, et al, 1993.

[12] Peterson, et al, 1993.

[13] Mittelbach, et al, 1985

[14] Reece and Peterson, 1993b.

[15] Vellguth, 1983.

[16] Peterson and Reece, 1994.

Cetane Effect of Esters

This section presents the results of a study of the impact of adding pure esters to diesel fuel on the cetane number of the blend. Seven different pure esters or ester blends were used for this study. Methyl palmitate and methyl stearate are the two esters derived from the most common saturated fatty acids in soybean oil. The isopropyl esters of these fatty acids were also tested. Methyl oleate was produced from a commercial grade of oleic acid. Since linoleic and linolenic acids are very expensive in their pure forms, a high linoleic safflower oil and linseed oil were used as sources of these fatty acids. The purity of the esters is shown in Table A-1 of the appendix.

In order to study how each ester affects the cetane number, cetane tests were conducted on blends of the esters from 0% to 50% in No. 2 diesel fuel. In a few cases, 100% ester data was included, also. This cetane number data was collected from three different testing laboratories using ASTM D613. A linear regression was performed on the cetane number data for each ester. The regression equation used was as follows:

$$\text{Cetane Number} = a + b [\% \text{ ester}] \quad (1)$$

Figure 2 is provided as an example of the cetane number regression lines and illustrates how the cetane number varied for the blends. Some of the variation in the y intercept, a, is due to different diesel fuels used for blending with the biodiesel. Due to fuel storage limitations, it was not possible to use the same diesel fuel for all of the experiments.

There is considerable variation in the cetane numbers from different laboratories and even between duplicate samples to the same laboratory. This variation is reflected in the low correlation coefficients for some fuels. Blind samples of ASTM check fuel were sent to each laboratory and no significant bias was detected.

The cetane response, which is b, the slope of the regression line, changes for each ester. Three methyl soyate cases are shown corresponding to different diesel fuels used for blending. The methyl soyate blends are all quite close with a slope between 0.10 and 0.13. These curves include data for 100% methyl soyate that show its cetane number to be between 54 and 56. The stearate esters have a higher cetane response than the palmitates. Methyl oleate has a lower cetane response than the saturated esters, but it is higher than methyl soyate. The cetane response of the methyl ester of safflower oil, which is high in methyl linoleate, is 0.1533 which is close to that for methyl soyate. This is not surprising since methyl linoleate is the predominant ester in methyl soyate. The methyl ester of linseed oil, which is high in methyl linolenate, has the lowest cetane response of all the esters

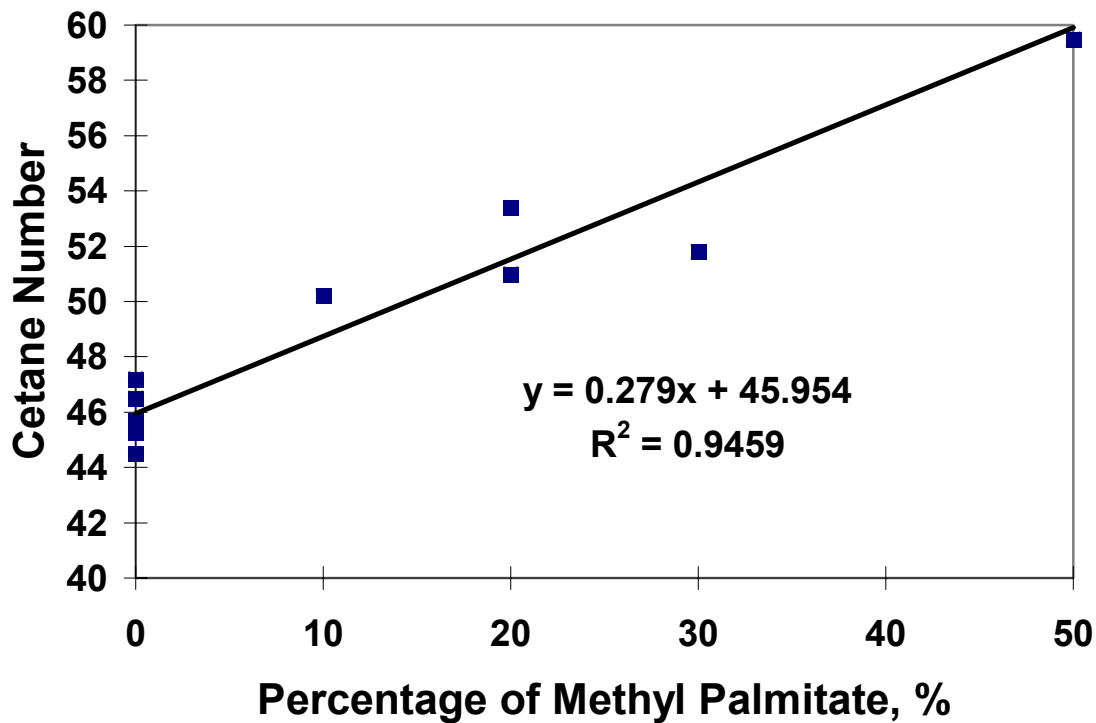


Figure 2. Cetane response for different levels of unsaturation

Table 2. Linear regression coefficients for cetane numbers of fatty acid ester blends.

Fuel	a	b	R ²
Methyl Soyate	46.151	0.1013	0.4889
Methyl Soyate	44.040	0.1293	0.8083
Methyl Soyate	47.586	0.1245	0.9536
Methyl Palmitate	45.954	0.2790	0.9450
Iso-propyl Palmitate	45.723	0.2394	0.9114
Methyl Stearate	43.125	0.3550	0.8905
Iso-propyl Stearate	43.452	0.2488	0.9034
Methyl Oleate	43.194	0.1930	0.7399
Methyl Safflower	41.695	0.1533	0.7688
Methyl Linseed	41.278	0.0837	0.6790
Octadecane	43.090	0.5978	0.9965

tested. Octadecane was included for comparison and had the highest cetane response of any fuel tested. Figure 3 shows the cetane response of the esters derived from 18-carbon chain length fatty acids. The decreasing cetane response with increasing unsaturation is clear. Methyl stearate, the saturated ester, has the highest cetane response and methyl oleate, with only one double bond, has the next highest. The methyl esters of safflower oil and linseed oil, with two and three double bonds, respectively, have correspondingly lower cetane response.

These data suggest that longer chain length compounds have higher cetane response than shorter chain length compounds and saturated compounds have higher cetane response than unsaturated compounds. This confirms observations by other researchers (Freedman and Bagby, 1990).

If the regression equations are extrapolated to 100% ester, admittedly a questionable procedure given the uncertainty in the values, the predicted values of cetane number of methyl palmitate and methyl stearate are 73.9 and 78.6, respectively. These values correspond well to the

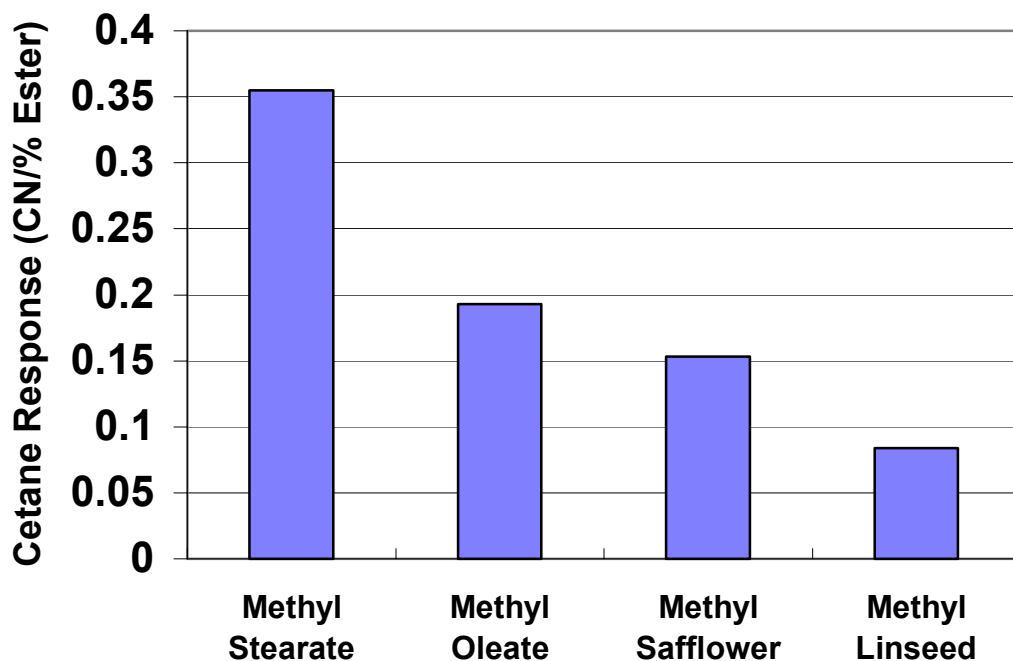


Figure 3. The effect of unsaturation on the cetane response of methyl esters

values observed by other researchers. Freedman and Bagby (1990) have reported cetane numbers for methyl palmitate and methyl stearate as 74.3 and 75.6, respectively, and Klopfenstein (1985) reported 74.5 and 86.9.

These data illustrate one of the main reasons for the variation in reported cetane numbers for biodiesel. When different feedstocks are used to produce biodiesel, the distribution of esters changes, also. Biodiesel produced from tallow will be high in saturated fatty esters and have a high cetane number. Biodiesel produced from unsaturated vegetable oils, such as soybean oil, will have a lower cetane number. The *Iodine Value*, a measure of unsaturation, was used in a cetane number correlation by Krisnangkura (1986). Krisnangkura's equation indicates that each unit of Iodine Value increase lowers the cetane number by 0.225. However, this does not explain the large variations in reported cetane number for samples of biodiesel from the same feedstock oil. Other factors must be important.

Effect of Unsaponifiable Matter

One major difference between different samples of biodiesel may be whether the fuel contained the unsaponifiable matter that was originally present in the soybean oil. The unsaponifiable matter in soybean oil consists of a variety of trace compounds that have very low volatility and are stable toward bases. It consists of sterols, alcohols, and high molecular weight hydrocarbons. A typical soybean oil might contain from 0.5% to 1.5% of this material.

Most of the soybean-based biodiesel used during 1993 and 1994 in the United States was processed using a distillation process that removed the unsaponifiable matter along with the glycerin and all other materials with higher boiling temperatures than the esters themselves. This fuel, commonly identified as "distilled fuel," may have different properties than undistilled fuel due to the removal of the unsaponifiable matter.

To investigate the effect of unsaponifiable matter on the cetane number, a mixture of materials normally found in the unsaponifiable matter in soybean oil was produced and blended with both diesel fuel and distilled methyl soyate. This "synthetic" unsaponifiable matter contained 70% sterols derived from soybean oil, 14% α -tocopherol, 8% squalane, and 8% squalene. This material was blended with

diesel fuel at 1% and 2% levels (by weight). It was not possible to keep more than 2% of this

Table 3. Effect of Unsaponifiable Matter on Cetane Number

Fuel	Cetane Number (ASTM D613)
Diesel Fuel	45.9
1% Unsap in Diesel Fuel	44.2
2% Unsap in Diesel Fuel	45.6
Methyl Soyate	52.7
1% Unsap in Methyl Soyate	53.7
2% Unsap in Methyl Soyate	53.4
3% Unsap in Methyl Soyate	52.8

material in solution with diesel fuel. The unsaponifiable matter was blended with methyl soyate at 1%, 2%, and 3% levels.

Table 3 shows the effect of unsaponifiable matter on the cetane number of diesel fuel and methyl soyate. The data show that the presence of unsaponifiable matter in the fuel has no effect on the cetane number of the fuel, either for diesel fuel or for methyl soyate. The slight changes in cetane number are generally less than the 0.7-0.8 ASTM repeatability specification for cetane number and are always less than the 2.6-3.1 reproducibility specification.

Oxidation of Methyl Soyate

Esters are subject to oxidation through contact with the oxygen in the air. When this process occurs at ordinary temperatures, the initial products are hydroperoxides formed by the addition of oxygen to a carbon atom adjacent to a carbon-carbon double bond (Bailey, 1945). Generally, at this step, the double bond is left intact. The extent of this level of oxidation can be characterized by the *peroxide value*. As oxidation proceeds, the peroxides may split and form aldehydes and short chain acids.

Oxidation of biodiesel occurs through a series of steps. Most vegetable oils contain natural anti-oxidants, such as Vitamin E (tocopherol), that inhibit oxidation until the antioxidant is consumed. When the natural anti-oxidants are depleted, oxidation proceeds rapidly. The period of slow oxidation that precedes rapid oxidation is often called the *induction period*.

Bailey (1945) states that if two or more double bonds are present in one fatty acid chain, they have a *mutually activating effect*. For example, linoleic acid, with its two double bonds, oxidizes more readily than oleic acid with only one double bond. In addition, the tendency to oxidize is greater than can be explained by the increase in the number of double bonds.

In the food industry, the result of oxidation is rancidity which is characterized by an unpleasant odor. When esters are used as fuel, an important consequence of oxidation is that the hydroperoxides are very unstable and tend to attack elastomers. In addition, the hydroperoxides can induce polymerization of the esters and form insoluble gums and sediments. Sediment and gum formation can be a problem with diesel fuel also, although the unsaturated nature of soybean-based biodiesel may aggravate the problem.

Recent engine durability testing with soybean-based biodiesel has shown that biodiesel may be subject to fuel filter plugging problems caused by sediment and gum formation (Ortech, 1995). The currently accepted explanation for the presence of sediment and gum is that the fuel changes chemically to produce these compounds and this is identified as a *fuel stability* problem. Biodiesel's oxidation is accelerated by heat and light. Generally, when air is present, oxidation will proceed.

When biodiesel has been distilled to remove the high boiling point materials such as monoglycerides and glycerin, the natural antioxidants such as tocopherol are also removed. Figure 4 shows the increase of the peroxide value with time between two samples of biodiesel. Both samples were prepared from the same soybean oil but one was distilled and the other was not. To oxidize the esters, both samples were stirred at room temperature while exposed to fluorescent light. Obviously, the distilled fuel oxidizes much more quickly than the undistilled fuel. While this was an accelerated

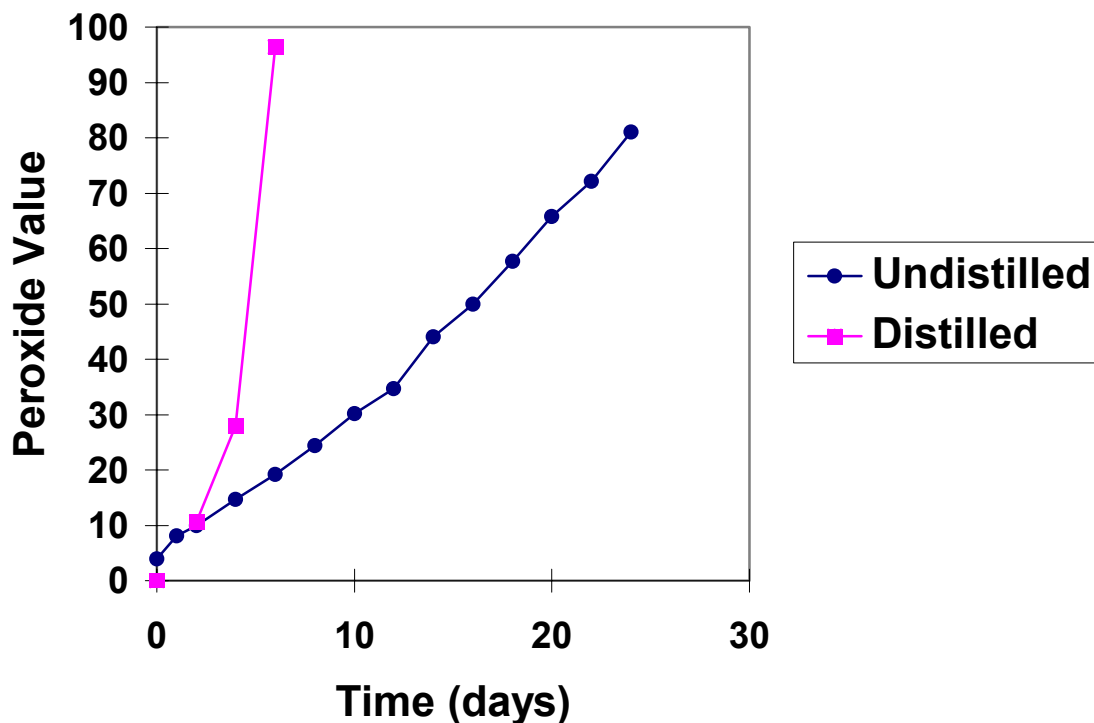


Figure 4. Rate of increase of peroxide value for distilled and undistilled esters.

test, it is expected that even under typical diesel fuel storage conditions, the distilled fuel will oxidize more quickly.

It was suspected that changing the oxidation level of the biodiesel would have an impact on cetane number since peroxide compounds have been proposed as cetane improvers (Liotta, 1993). Table 4 shows the results of cetane number measurements on the samples described in Figure 4, before and after oxidation.

As can be seen in the table, when the freshly prepared esters were oxidized to a peroxide value of 81, the cetane number increased from 47.8 to 54.6. A soybean oil with a peroxide value of 81 would generally be considered to be rancid. However, methyl soyate with a high peroxide value does not always have the unpleasant smell usually associated with rancidity.

The cetane testing of the distilled esters showed that when the cetane number was determined before oxidation occurred, the cetane number was the same as before distillation. The decrease from 47.8 to 46.8 is not considered to be significant. However, as with the undistilled esters, there was a large increase in cetane number as the esters were oxidized, from 46.8 to 55.4. While the reason for the cetane number increase is not entirely certain, it is likely that the hydroperoxides formed during the oxidation of the esters are responsible. Dialkylperoxides have been shown to be strong cetane enhancers (Liotta, 1993) and hydroperoxides are likely to increase the cetane number also.

To further investigate the effect of oxidation on the cetane number of biodiesel, samples of methyl soyate at six different levels of peroxide value were prepared. To produce these samples, freshly prepared, undistilled methyl soyate was mixed in varying amounts with highly oxidized,

Table 4. Effect of oxidation and distillation on cetane number

Sample	Rep 1	Rep 2	Average
Freshly prepared methyl esters (PV=6)	46.8	48.7	47.8
Oxidized methyl esters (PV=81)	54.6	54.5	54.6
Distilled methyl esters (PV=0)	46.5	47.2	46.8
Distilled and oxidized methyl esters (PV=82)	55.5	55.2	55.4

distilled methyl soyate. Since the previous testing had established that distillation did not affect the cetane number, any changes in cetane number between these samples were assumed to be due to the difference in oxidation. Each of the six samples was split into three parts and the 18 test specimens were submitted as blind samples to a commercial laboratory for cetane testing. The results of this testing are shown in Figure 5.

Figure 5 shows the three values of cetane number at each of the six levels of peroxide value. ASTM D613 states that since these measurements were conducted by the same operator, on the same equipment, and on the same test material, the difference between the highest and lowest value should be within 0.7 to 0.8 cetane numbers. However, the differences in this data were as large as 3.9. In spite of the experimental variation, the trend toward increasing cetane number with increasing peroxide value is clear.

The equation for a least-squares-fit cubic polynomial is shown in the figure. The predicted cetane number for methyl soyate with a peroxide value of zero is 46.17. This value corresponds well to the low peroxide value cetane number of 47.8 and 46.8 given in Table 5. It appears that the cetane boosting effect of the hydroperoxides becomes minimal above a peroxide value of 70.

CONCLUSIONS

1. The cetane number of biodiesel depends on the distribution of fatty acids in the original oil or fat from which it was produced. The longer the fatty acid carbon chains and the more saturated the molecules, the higher the cetane number.
2. The unsaponifiable matter in biodiesel does not appear to have an impact on the cetane number.
3. Fuel which has been distilled oxidizes much more quickly than undistilled fuel. While the distillation process does not affect the cetane number, the oxidation results in a cetane number increase. When the fuel was oxidized to a peroxide value of 82, the cetane number increased between 7 and 8 points. Further increases in the peroxide value did not increase the cetane number.

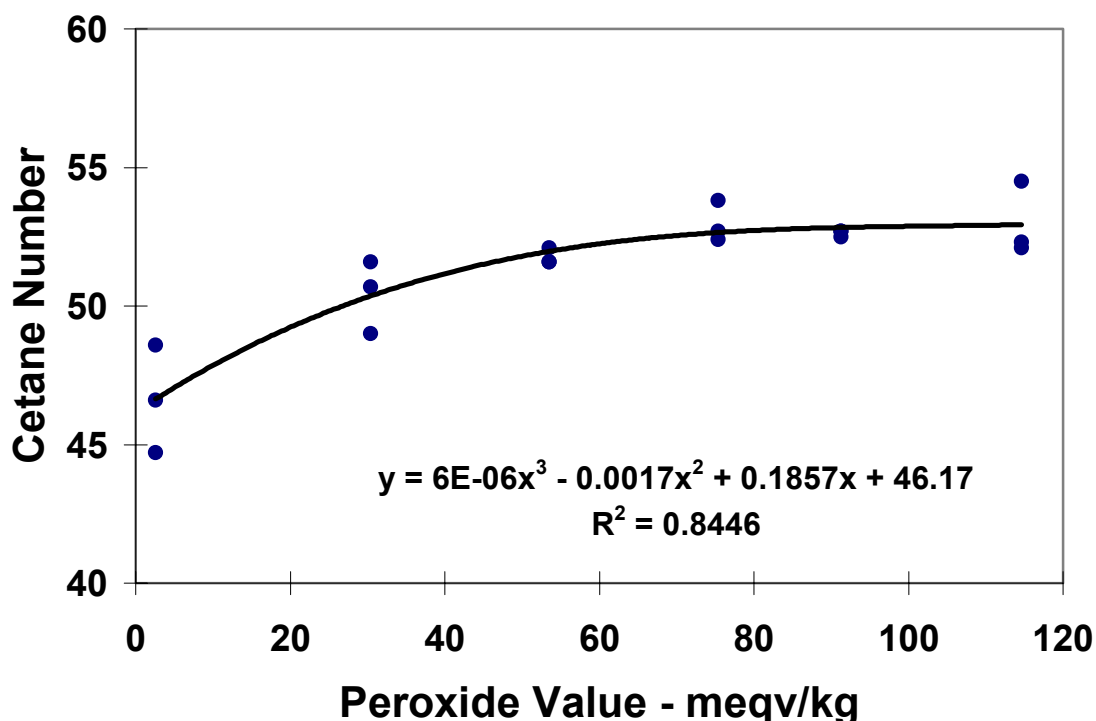


Figure 5. The effect of peroxide value on cetane number

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APPENDIX

Table A-1. Fatty acid composition of ester fuels.

Ester	14:0	16:0	18:0	18:1	18:2	18:3	20:0
Methyl Palmitate	1.3	97.5	1.2				
Methyl Stearate		1.6	97.2				1.2
Methyl Oleate		0.8	2.9	91.9	4.5		
Methyl Safflower		6.6	2.4	13.4	77.1	0.2	0.3
Methyl Linseed		5.3	3.1	18.7	16.7	56.2	
Methyl Soyate		11.2	4.0	22.8	53.4	8.3	0.3
Isopropyl Palmitate	1.1	96.2	2.8				
Isopropyl Stearate		1.2	97.7				1.1