

TWO-YEAR STORAGE STUDY WITH METHYL AND ETHYL ESTERS OF RAPESEED

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ABSTRACT. *Methyl and ethyl esters, prepared from various vegetable oils by the process of transesterification, have shown much promise as fuels for all types of diesel engines. Very limited information is available on possible deterioration of biodiesel in storage. This project was designed to determine the extent of deterioration of Rape Methyl Ester (RME) and Rape Ethyl Ester (REE) in storage. The study involved triplicate samples of RME and REE stored in glass and steel containers at room temperature (inside) and at the local ambient outdoor temperatures (outside). The study was conducted for 24 months. At the beginning of the study and at three-month intervals, samples were taken for measurement of peroxide value, acid value, density, viscosity, and heat of combustion. At the conclusion of the study, engine performance tests were conducted with the two year stored REE and RME, new REE and RME, and low sulfur diesel reference fuel. On the average, the esters increased over time in all of the previously mentioned properties with the exception of heat of combustion, which decreased. Regression models are presented to predict the deterioration with time. Engine power varied less than 2% for both Biodiesel fuels compared to the stored counterparts while smoke density decreased 3.2% for the stored RME and increased 17.5% for stored REE.*

Keywords. *Biodiesel, Vegetable oil, Storage, Physical properties.*

Biodiesel is gaining recognition as a renewable alternative to diesel fuel. One step toward commercialization of biodiesel is establishing how well it stores and what affect storing for long periods has on the performance of the fuel. The problems of fuel deterioration with biodiesel during storage are expected to be more severe than for commercial diesel fuel. Although vegetable oils contain natural antioxidants, their high degree of unsaturation makes them susceptible to gum formation. Since fuel deterioration occurs mainly by oxidative polymerization leading to gum formations one purpose of this study was to determine the rate of oxidative polymerization for different fatty acid esters prepared from rapeseed, as effected by container types, and environments. Peroxide values measure the levels of the oxidation products in the samples. Another purpose of this study was to use short term engine coking and torque test procedures to determine whether the presence of the oxidation products affected the engine performance.

LITERATURE REVIEW

Previous work on the storage stability of vegetable oil fuels includes a three year study reported by Klopfenstein and Walker (1984, 1985, 1986). In that study, samples of soybean methyl esters were stored in three different environments (indoor, outside, and underground), two container types (steel and plastic) and two formulations (with and without antioxidants). Samples were analyzed initially and at four month intervals for fatty acid composition, peroxide value, density, and viscosity. Results favored underground storage, plastic-lined containers and the use of 0.5% butylated hydroxy toluene as an antioxidant.

A study performed at the University of Idaho by Korus (1983) and continued by Jo (1984) involved neat vegetable oils as fuels. Oil deterioration was measured as a function of storage conditions (aerobic and anaerobic at room temperatures) and vegetable oil composition (fatty acid saturation vs unsaturation). Parameters measured at six month intervals over a two-year period were peroxide values and fatty acid profiles. Engine testing was also performed using the stored oils to test injector coking as a function of fuel deterioration. Fuels tested were 50/50 blends of winter rapeseed, linoleic safflower and oleic safflower with diesel fuel and 100% 2-D as a reference. All vegetable oil fuel blends gave a statistically significant ($\alpha < 0.05$) increase in carbon deposits relative to diesel fuel with linoleic safflower having an injector coking area of 7.57 cm² relative to diesel fuel, 50% oleic safflower 5.01 cm² and 50% winter rapeseed 3.93 cm². Results indicated that deterioration was reduced by anaerobic storage and by high levels of saturated fatty acids in the oil.

OBJECTIVES

1. Store triplicate sets of vented glass and steel containers of RME and REE in flammable solvent

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storage cabinets at both inside and outside ambient temperatures and analyze fuel properties (peroxide value, acid value, density, viscosity, and heat of combustion) from each storage container every three months for a 24-month period and compare with the initial values.

2. Compare two-year stored RME and REE in short-term engine performance tests with new RME and REE.

MATERIALS AND METHODS

FUEL PREPARATION

One hundred-thirty liters of each fuel (RME and REE) were made at the beginning of the study. Samples of each fuel were taken for triplicate determinations in all of the five test procedures at the beginning of this test and at each three-month interval thereafter.

The Biodiesel fuels were processed in a batch type reactor. The methyl ester process utilizes 100% molar excess alcohol (anhydrous), or a molar ratio of 6:1 alcohol to oil ratio. Based on the amount of input oil by weight, 1.1% potassium hydroxide (KOH) was used. The ethyl ester process utilizes 70% stoichiometric excess ethanol (anhydrous), or a molar ratio of 5.1:1 ethanol to oil ratio. Based on the amount of input oil by weight, 1.3% of KOH was used. The transesterification procedure used has been reported in several other University of Idaho articles (Peterson et al., 1991, 1997).

STORAGE CONTAINERS

Fuel containers for the study were: 4 L brown glass reagent bottles and 8 L steel pails with crimp on lids. All containers were filled 3/4 full and vented to the atmosphere to simulate actual fuel storage. The study used triplicate samples of two esters, RME and REE, each fuel was arranged in four different configurations: glass and steel containers stored at room temperature at approximately 23°C (referred to as inside) and at the local outside ambient temperature which varies from an average of -2°C, with short periods going below -23°C, in December and January to an average high of 19°C in August, with short periods exceeding 37.8°C, (referred to as outside) for a total of 24 samples. The yearly average temperature at this site is 8.3°C; temperature data from Molnau (1997).

FUEL ANALYSIS

After each three-month storage period, samples were analyzed according to the following procedures (AOCS, 1987; ASTM, 1991a,b,c,d): Peroxide Value, AOCS Cd 8b-90; Acid Value, ASTM Test D974; Density, ASTM Test D1298; Viscosity, ASTM Test D445; and Heat of Combustion; ASTM Test D240.

The fuels were characterized initially and after the two year storage study by evaluating the parameters in ASAE EP552 (ASAE, 1995) The tests for specific gravity, viscosity, cloud point, pour point, flash point, heat of combustion, total acid value, peroxide value, catalyst, and fatty acid composition were performed at the Analytical Lab, Department of Biological and 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 by Phoenix Chemical Labs, Chicago, Illinois.

SHORT TERM ENGINE TESTS

The 24 samples from the storage study were combined into two large REE and RME samples, respectively, for use in the short-term engine testing. New batches of REE and RME were produced for comparative purposes. Phillips 66 low sulfur number two diesel fuel (2-D) was used as a reference. The test engine was a John Deere (4239T) direct-injection, turbocharged diesel. This is a four-cylinder engine with a bore of 106 mm, a stroke of 110 mm, a displacement of 3.917 L, and a compression ratio 16.2:1. Its advertised specifications are a high idle of 2650 rpm, with 61 kW (82 HP) at 2500 rpm and 290 N·m (214 lbf·ft) of torque at 1500 rpm. Attached to the engine was a General Electric 119 KW (159 HP) cradle-type dynamometer.

Two short-term engine performance test procedures were performed. The first was a rapid engine test to measure injector fouling in diesel engines using vegetable oil fuels (Korus, 1985). For this test the engine was operated at maximum power at 2500, 2300, 2100, 1900, 1700, and 1500 rpm for 10 min at each step. Readings of ambient air, opacity meter, exhaust, fuel, lube oil and intake air temperatures, and exhaust opacity were acquired every 30 s. After each fuel test the injectors were removed and the carbonaceous tips were measured using machine vision (Goodrum et al., 1996). There was only enough fuel for one replication of this test.

The second test was a SAE torque test (SAE J1349, 1990). This test was performed under full throttle and full load conditions from 2600 to 1300 rpm in 100 rpm steps. This test was replicated once also. Both test procedures were set up to run the fuels in random order. For more information on engine test procedures and equipment refer to (Hammond, 1996; Perkins et al., 1991).

STATISTICAL ANALYSIS

This experiment was set up as a randomized complete block with replication. Blocks were inside and outside for each time period. Treatments were fuels (RME and REE) and containers (glass and steel). Each fuel and container type were replicated three times for a total of 12 containers inside and 12 containers outside. Statistical analyses were performed with SAS (1996). This consisted of a statistical analysis (ANOVA) followed by a Tukey multiple range procedure to separate the means for each of the parameters, peroxide value, acid value, density, viscosity, and heat of combustion for each time period. All differences were accepted as significant when they exceeded a probability level of $\alpha = 0.05$.

In addition a regression model was formulated for each parameter also by using SAS. Initially a multiple regression was run on each parameter against the variables container, location, time, location by time, time squared and fuel, and then those parameters with non-significant coefficients were dropped and the analysis repeated. The variables in the models were coded as shown in table 1.

Table 1. Storage variables

Container	- Listed from 1 to 2, glass = 1, metal = 2
Location	- Listed from 1 to 2, inside = 1, outside = 2 (inside temp = 70°F, outside temp varied from 34°F to 72°F)
Time	- Listed from 1 to 9
Fuel type	- Listed from 1 to 2, RME = 1, REE = 2

RESULTS AND DISCUSSION

In the following discussion the nomenclature used is as follows: G = Glass, M = Metal, RME = Methyl Ester, REE = Ethyl Ester, I = Inside and O = Outside.

PEROXIDE VALUE

Peroxide values, measured in milliequivalents of peroxide per kilogram of sample for each sampling period, are shown in figure 1. There was a consistent increase in peroxides over time with an acceleration of that increase from the 6th through the 18th month. Peroxide values for either fuel were not significantly affected by the type of container. Statistical analysis of the interactions between fuel and location indicated peroxide values were significantly higher between inside and outside storage for REE. RME showed no significant difference for interactions between fuel and location for peroxide values. There was a significant increase for peroxide from initial readings with RME after six months. REE showed a significant increase after three months.

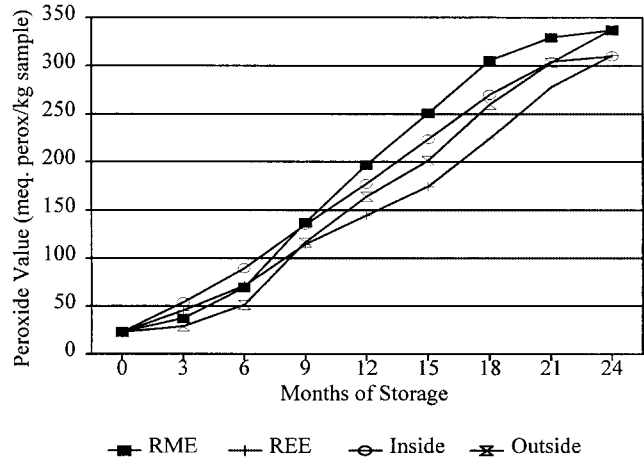


Figure 2—Peroxide value for the 24-month storage period for RME and REE, and inside and outside locations.

Figure 2 shows that after 6 months the peroxides in the RME increased at a faster rate than in the REE. Peroxide values at the outside location had a slower rate of increase than did the values at the inside location for the first six months, also figure 2.

An increase in peroxide value over time is observed. At 24 months, the peroxide value was 14.5 times higher for RME and 13.7 times higher for REE compared to the

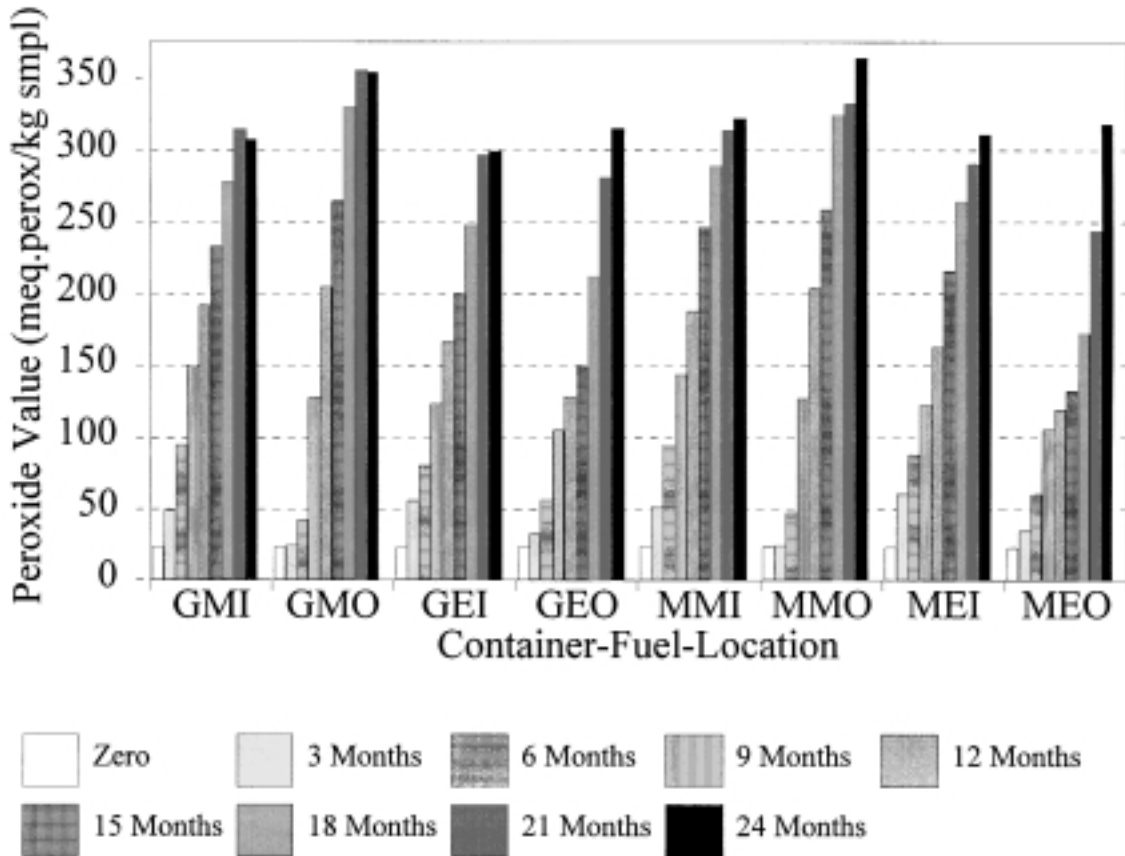


Figure 1—Peroxide value vs container type, fuel type, and location for 24 months of storage. Symbols used in the chart are: 1st letter is the container G = Glass, M = Metal; 2nd letter is the fuel type M = Methyl Ester, E = Ethyl Ester; 3rd letter is the location I = Inside and O = Outside; for example, GMI means glass container with methyl ester stored inside.

beginning value. Fuel stored outside had a peroxide value 14.7 times higher while fuel stored inside was 13.5 times higher compared to the beginning value. The best fit regression model for the change in peroxide value was as follows:

$$P_{\text{perox}} = -30.906*\text{loc} - 33.41*\text{fuel} + 35.19*\text{time} + 3.95*\text{loc}*\text{time} + 61.624$$

$$R^2 = 0.94$$

This equation, over the 24 months, shows that fuel type affected peroxide by a change of 33.41, each quarter of time increased peroxide by 35.19, outside location decreased peroxide value by 30.906, and there was a small location by time interaction.

ACID VALUE

The acid values, as shown in figure 3, measured in milligrams of KOH per gram of sample, displayed the same trend as did the peroxide values. Since both of these values are related to autoxidation, the acid values naturally increase with an increase in peroxides because the esters first oxidize to form peroxides which then undergo complex reactions including a split into more reactive aldehydes which further oxidize into acids. Acids can also

be formed when traces of water cause hydrolysis of the esters into alcohol and acids (Formo, 1979).

Acid values for either fuel were not significantly affected by the type of container. Statistical analysis of the interactions between fuel and location indicated REE and RME had a significant difference between inside and outside storage for acid values. The effects of time on REE and RME show that after nine months, there was a significant increase in acid values from initial readings and the increase accelerated toward the end of the 24-month period.

Figure 4 shows acid value versus time for both RME and REE. The acid values were fairly constant for the first six months then took a significant upward trend. The RME acid values increased at a faster rate than the REE values after six months as was the case with peroxides. Figure 4 also shows the acid value versus time for inside and outside samples. The acid value of the outside samples lagged behind the inside samples. At 24 months the acid value was 10.3 times higher for RME and 9.2 times higher for REE compared to the beginning values. Fuel stored outside had acid values 9.0 times higher while fuels stored inside had acid values 10.5 times higher compared to the beginning value. The best fit regression model for the change in acid levels was as follows:

$$P_{\text{acid}} = -0.064*\text{time} - 0.172*\text{fuel} - 0.0187*\text{loc}*\text{time} + 0.0209*\text{time}^2 + 0.473$$

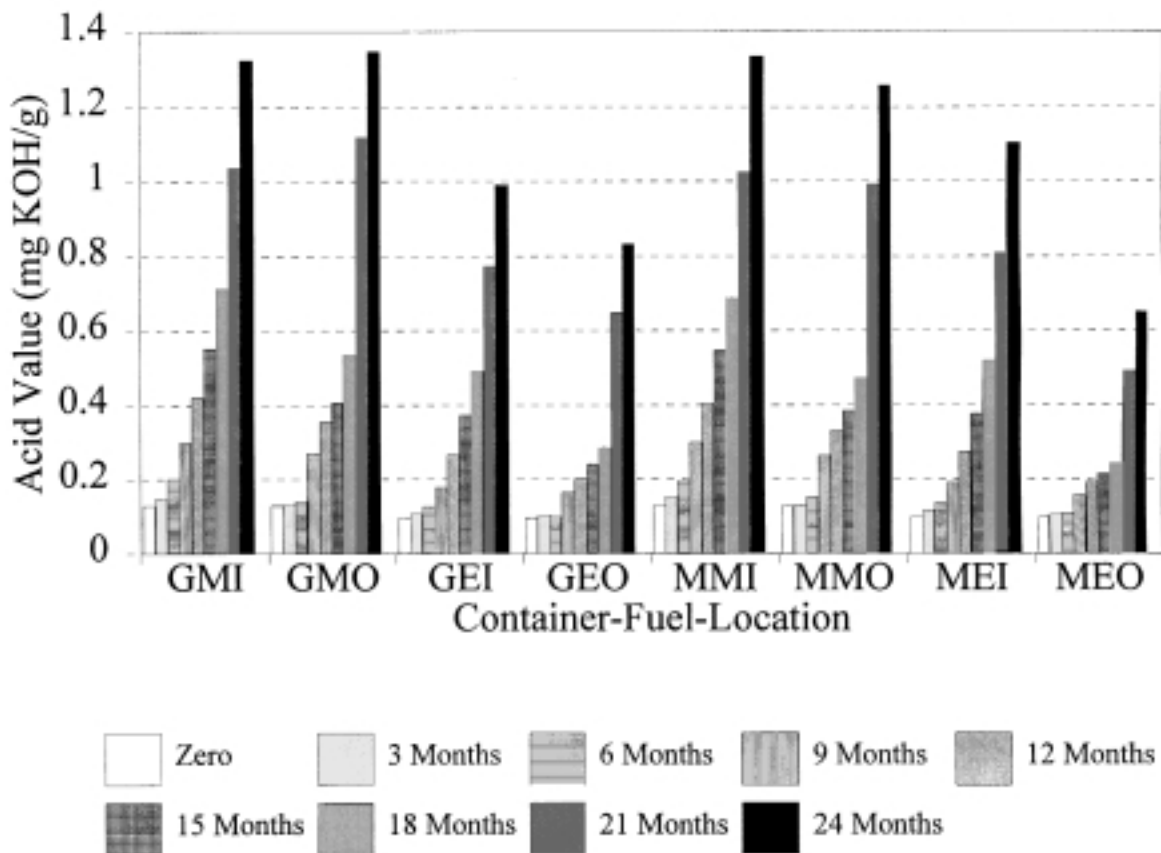


Figure 3—Acid value vs container type, fuel type, and location for 24 months of storage. Symbols used in the chart are: 1st letter is the container G = Glass, M = Metal; 2nd letter is the fuel type M = Methyl Ester, E = Ethyl Ester; 3rd letter is the location I = Inside and O = Outside; for example, GMI means glass container with methyl ester stored inside.

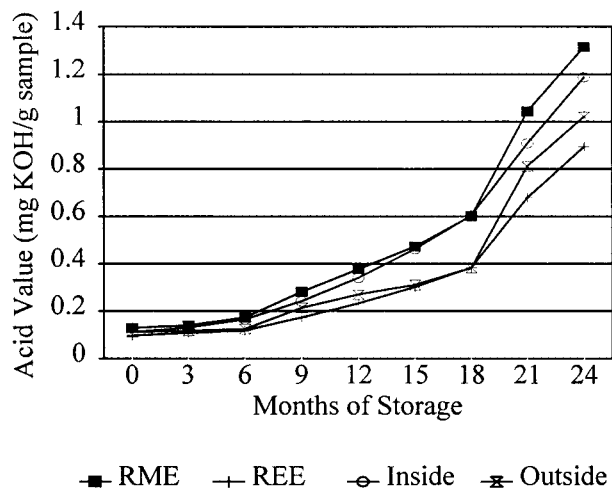


Figure 4—Acid value for the twenty-four month storage period for RME and REE, and inside and outside locations.

$$R^2 = 0.93$$

This model shows a significant time to location interaction and a quadratic relationship for time. The acid value was -0.172 higher for fuel type (the negative sign indicates that REE was lower than the RME) and the outside samples had a 0.06 lower acid value than the inside samples.

DENSITY

It was found that the density of the esters increased over time, as shown in figure 5. Density values for either fuel were not significantly affected by the type of container. Statistical analysis of the interactions between fuel and location indicated REE had a significant density increase between inside and outside storage. RME showed no significant density difference for interactions between fuel and location. The effects of time on RME show that after three months, there was a significant density difference from initial readings. REE showed a significant difference after six months. Figure 6 shows density versus time for both RME and REE. The density of the RME at the beginning of the study was higher than that of REE and increased at a faster rate after six months. Significant differences were found among means from each sampling period with the exception of the density values of REE between zero and three months. Location was not a major influence in changing density values over time, however, the inside samples were slightly higher density than the outside samples. A 1.05% increase of density with time was measured. RME density increased 1.22% and REE density increased 0.88%. Fuel stored outside increased 1.05% and fuel stored inside 1.04%. The best fit regression model for the change in density values was as follows:

$$P_{den} = -0.4815*loc - 4.61*fuel + 0.114*time^2 + 885.28$$

$$R^2 = 0.966$$

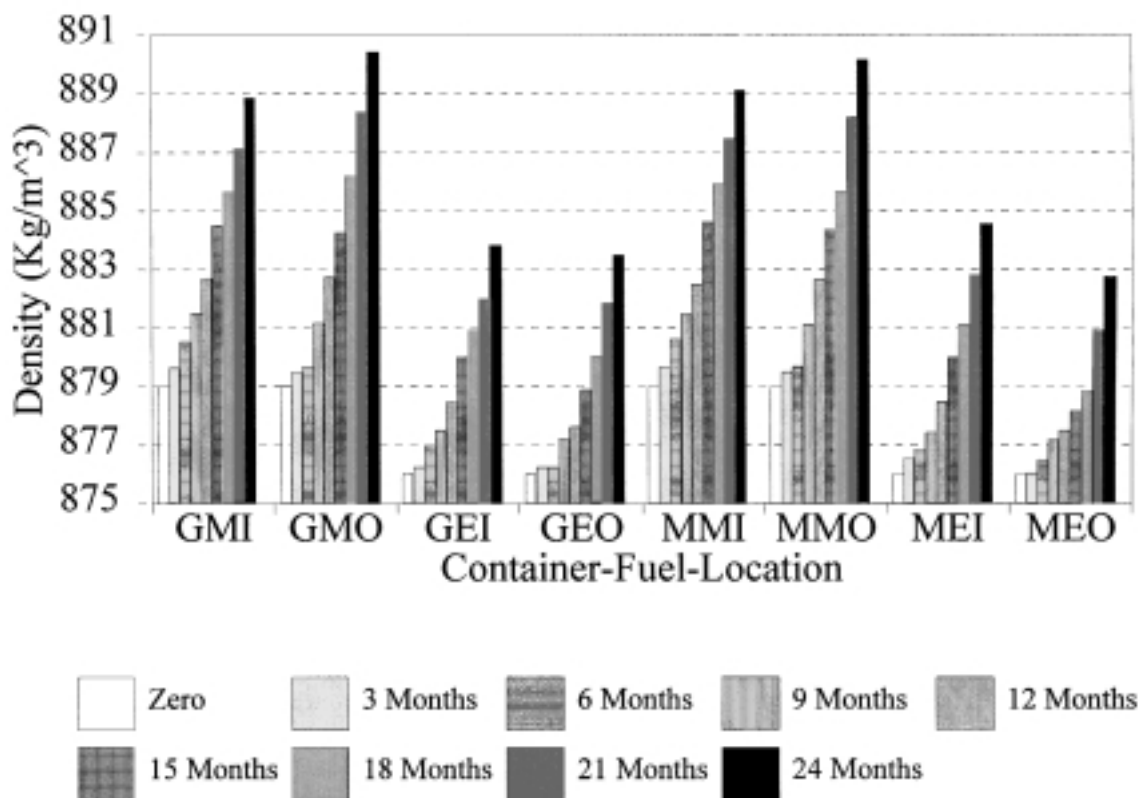


Figure 5—Density vs container type, fuel type, and location for 24 months of storage. Symbols used in the chart are: 1st letter is the container G = Glass, M = Metal; 2nd letter is the fuel type M = Methyl Ester, E = Ethyl Ester; 3rd letter is the location I = Inside and O = Outside; for example, GMI means glass container with methyl ester stored inside.

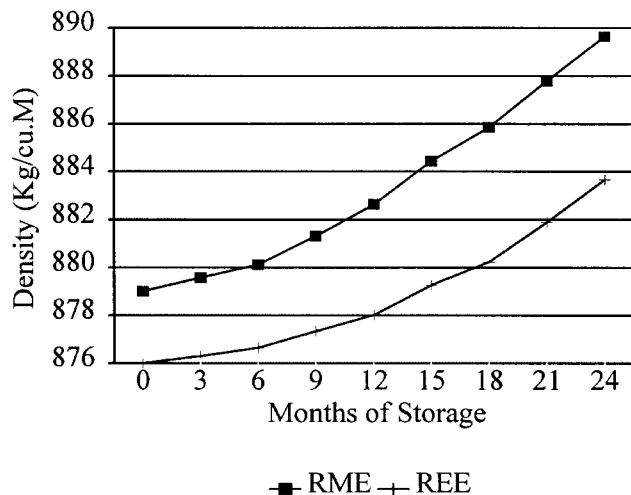


Figure 6–Density vs 24 months of storage for RME and REE.

This equation shows a quadratic relationship for time. The density increment for fuel type was -4.61 (the negative sign indicates a lower density for REE than RME) and a -0.48 reduction in density for the outside stored samples.

VISCOSITY

Viscosity tended to increase over time as shown in figure 7; however, the outside samples tended downward

during the first half of the study before increasing. This could be due in part to the lower average temperatures of the outside samples during the early period. Viscosity values for either fuel were not significantly affected by the type of container. The viscosity of REE as shown in figure 8 started out about 10% higher than that of RME but after a year of storage was only 5% higher. RME experienced a significant rise in viscosity after six months of storage and continued to rise significantly over the 9- to 24-month periods. REE stayed fairly constant for the first 12 months and then increased at about the same rate as RME for the final 12 months. The viscosity of RME increased 23.1% and viscosity of REE 16.8% over the 24 months. Inside and outside samples had nearly identical 19+% increases. The best fit regression model for the change in viscosity was as follows:

$$P_{\text{visc}} = -0.0807*loc + 0.0394*time + 0.5006*fuel + 0.0198*time^2 + 5.46$$

$$R^2 = 0.91$$

The model shows viscosity was 0.08 cSt lower for outside stored samples, the effect of fuel type changed viscosity by 0.5 cSt (REE higher than RME) and a quadratic effect of time.

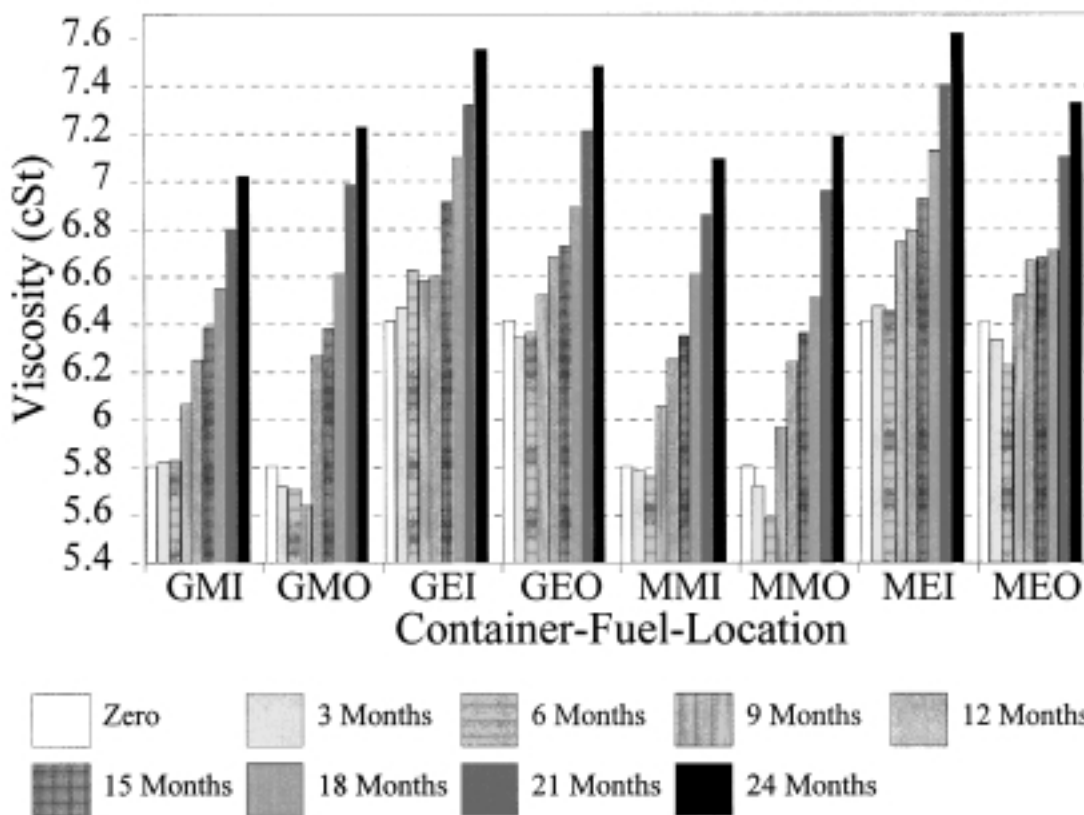


Figure 7–Viscosity @ 40°C vs container type, fuel type, and location for 24 months of storage. Symbols used in the chart are: 1st letter is the container G = Glass, M = Metal; 2nd letter is the fuel type M = Methyl Ester, E = Ethyl Ester; 3rd letter is the location I = Inside and O = Outside; for example, GMI means glass container with methyl ester stored inside.

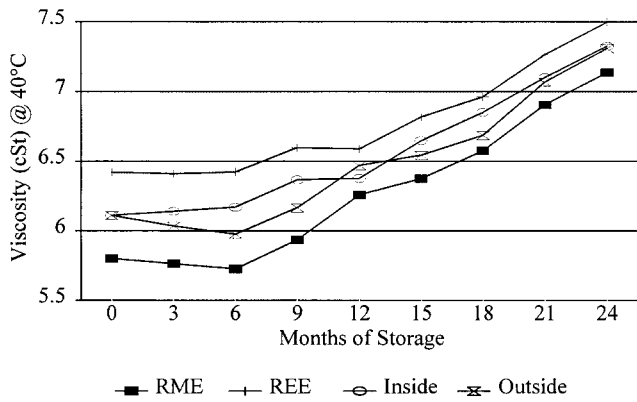


Figure 8—Viscosity @ 40°C for 24 months of storage for RME, REE, and inside and outside locations.

HEAT OF COMBUSTION

The values for heat of combustion, shown in figure 9, tended to decrease over time. This was most likely due to the breakdown of the fuel by oxidation which was verified by the increase in the peroxide and acid values. Heat of combustion values for either fuel were not significantly affected by the type of container. Statistical analysis of the interactions between fuel and location indicated REE had a significant difference and RME no significant difference between inside and outside storage for heat of combustion values. Figure 10 shows a decline in the heat of combustion of the two fuels with the exception of REE between month

6 and 9. The heat values for RME at 12 months were significantly lower than values at both zero and three months. The REE heat of combustion values on the other hand showed a significant drop at six months which corresponds with a rise in the other four parameters at six months, however it then increased at nine months although not significantly. The final reading at 24 months was significantly lower than the beginning value for heat of combustion. Heat of combustion declined about 1.4% over the 24 months of storage. RME declined 1.50% and REE 1.27%. Inside and outside samples declined at about the same 1.4% rate. The best fit regression model for the change in heat of combustion was as follows:

$$P_{HoC} = 46.4*loc + 235.5*fuel - 6.7* time^2 + 40132$$

$$R^2 = 0.747$$

The model shows a quadratic change in heat of combustion with time, 235 kJ/kg less energy for RME than for REE and 46.4 kJ/kg more energy for samples stored outside compared to the inside samples.

FUEL CHARACTERIZATION

Table 2 shows the fuel characteristics for the initial and two-year stored fuel as well as for the low sulfur diesel used for the control in the engine tests. The SREE and SRME are the REE and RME fuels, respectively, stored for two years. The cetane number increased more than 12%, viscosity increased more than 16%. Particulate matter,

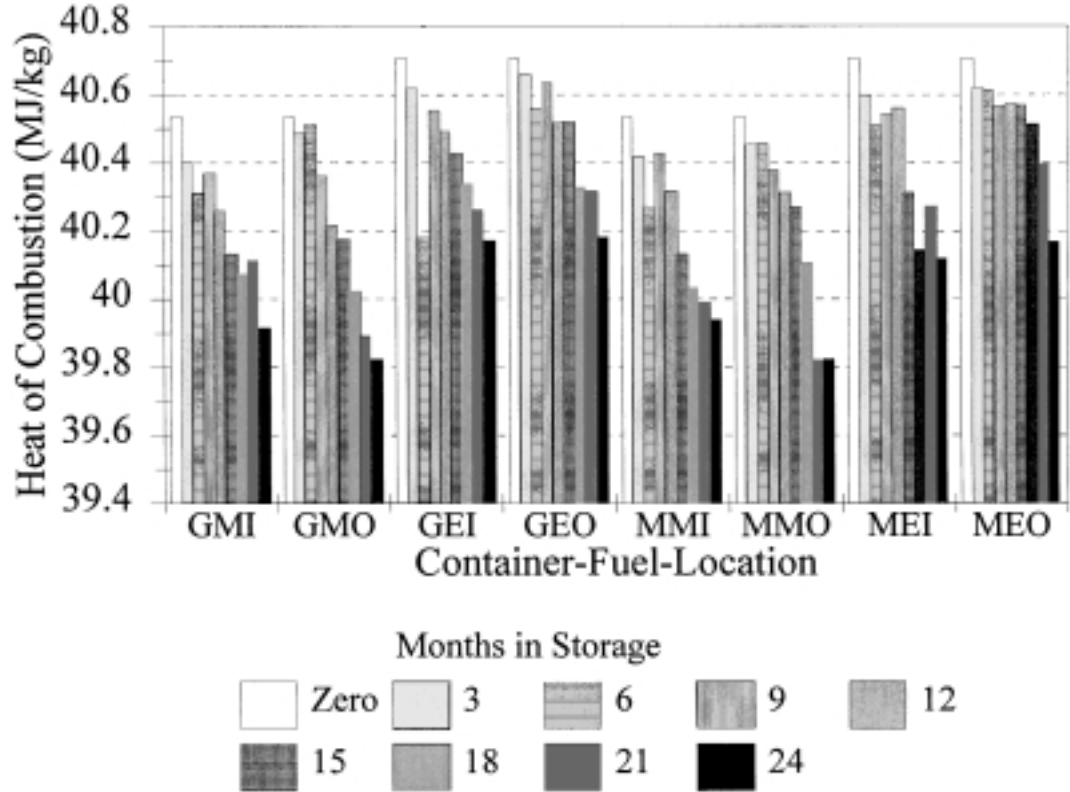


Figure 9—Gross heat of combustion vs container type, fuel type, and location for 24 months of storage. Symbols used in the chart are: 1st letter is the container G = Glass, M = Metal; 2nd letter is the fuel type M = Methyl Ester, E = Ethyl Ester; 3rd letter is the location I = Inside and O = Outside; for example, GMI means glass container with methyl ester stored inside.

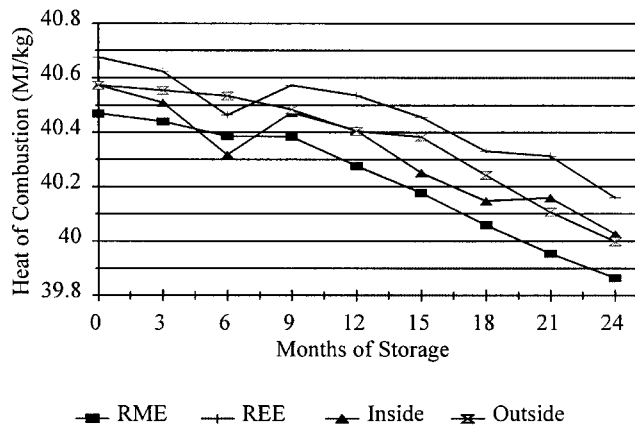


Figure 10—Gross heat of combustion for 24 months of storage for RME, REE, and inside and outside locations.

specific gravity, acid value, and peroxide value all increased during storage while flash point and heat of combustion decreased.

SHORT-TERM ENGINE TESTS

At maximum power output, the fuel stored for two years increased power 1.9% for REE and 1.5% for RME compared to newly produced fuel as shown in figure 11. The stored fuels also increased maximum torque by 0.9%. The new fuels had 3.4% less power than 2-D at maximum power and 1.4% less torque than 2-D at peak torque rpm.

Smoke density for the Biodiesel fuels varied from 1.7 to 3.6 times that of 2-D. The smoke density for the stored REE was 1.175 times more than that of the new REE and the stored RME produced 3% less smoke than the new RME, as shown in figure 12.

Fuel economy (kg/min) was compared at each rpm level, also shown in figure 12. For example, at 1500 rpm, the stored RME used 1.13% more fuel than did the new RME and the stored REE used 1.2% more fuel than the new REE.

The injector tip coking index for the 2-D, new RME and REE, and the stored RME and REE fuels were found by

dividing each of the fuels coking area by the diesel coking area. The coking on the injector tips for the stored fuels was 7.8% more for RME and 2.8% more for REE. The coking observed for RME in this test was extremely low and for REE was at or below other tests for Biodiesel reported by the authors recently (Peterson et al., 1997.) The effect due to storage was extremely small in both cases.

CONCLUSIONS

According to test results from this 24-month study of rapeseed methyl and ethyl esters, there was a slight deterioration of the fuel over time. The following trends became apparent as a result of this study. In general no effect was found for container type. Test values compare very closely for both metal and glass containers. Significant differences were, however, found in the values for each of the five measured parameters for both fuel type and storage location with the passage of time. For both fuels the peroxide and acid values, density, and viscosity tended to increase over time. The heat of combustion, on the other hand, tended to decrease slightly over time.

Fuels tended to degrade at a slightly faster rate, particularly as indicated by the peroxide and acid values, at

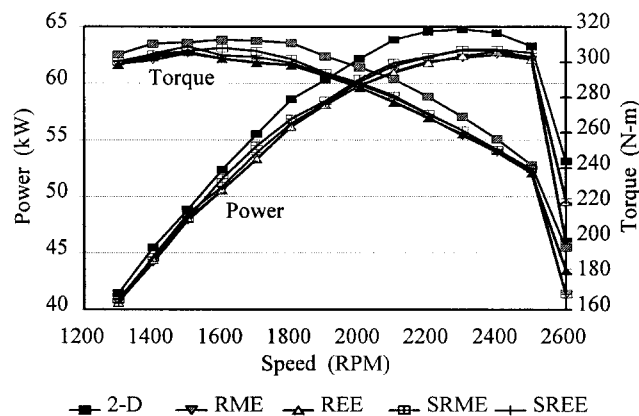


Figure 11—Power and torque plotted against engine speed for five fuels. The SRME and SREE are the RME and REE after 24 months of storage.

Fuel Specific Properties	2D	REE	SREE	RME	SRME
Specific gravity, 60/60	0.8495	0.8760	0.8837	0.8790	0.8897
Viscosity, cSt @ 40°C	2.98	6.42	7.50	5.80	7.14
Cloud point (°C)	-12	-2	0	0	1
Pour point (°C)	-20	-10	-9	-15	-15
Flash point (°C)	87	170	148	179	148
Boiling point (°C)	186	273	254	347	271
Water and sediment (% vol.)	< .005	< 0.005	< 0.005	< 0.005	< 0.005
Carbon residue (% mass)	0.16	0.06	0.12	0.08	0.07
Ash (% mass)	0.002	0.002	0.011	0.002	0.003
Sulfur (wt%)	0.036	0.014	0.008	0.012	0.009
Cetane number	46	64.9	> 72.7	61.8	> 72.7
Heat of combustion, gross (MJ/kg)	45.42	40.51	40.16	40.54	39.86
Copper corrosion	1A	1A	1A	1A	1A
Karl Fischer water (ppm)	38	761	949	757	1077
Particulate matter (mg/L)					
Total	0.9	1.9	48.45	1.1	57.00
Non-combustible	< 0.1	0.9	8.10	< 0.1	2.05
Elemental analysis					
Carbon (%)	86.67	76.83	78.85	78.7	77.76
Hydrogen (%)	12.98	11.8	12.49	12.66	12.49
Oxygen (%) (by difference)		11.36	8.64	9.22	9.74
Iodine number	9	96.7	90.1	91.9	93.9
Peroxide value		22.68	310.88	23.26	336.93
Acid value	0.002	0.097	0.894	0.128	1.315

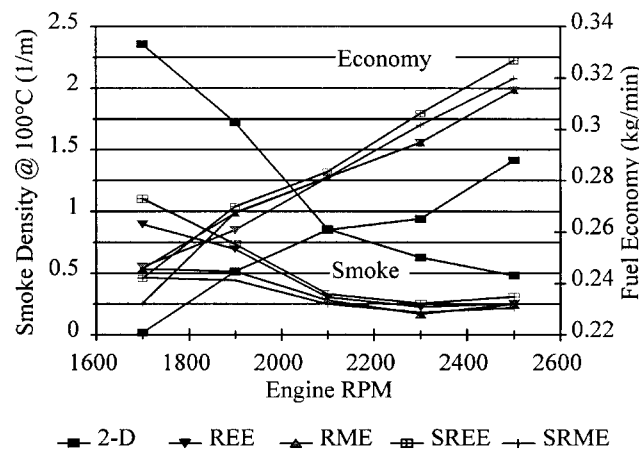


Figure 12—Smoke density and fuel economy for five fuels at five different engine speeds. The SRME and SREE are the RME and REE after 24 months of storage.

the indoor location. This was evidently due to the higher average temperature at the indoor location. Initially, and over the 24-month storage period, properties of the two esters displayed slightly different values. The ethyl esters were higher in viscosity and heat of combustion, but lower in density, peroxide and acid values than the methyl esters. Over time, degradation occurred at a different rate for the two fuels. The RME increased in peroxide value, acid value, and density at a faster rate than did the REE. Specific observations are noted below.

1. Peroxide values were increased 14.5 times for RME and 13.7 times for REE after 24 months compared to the beginning values.
2. Acid values were increased 10.3 times for the RME and 9.2 times for REE after 24 months compared to the beginning values.
3. Density increased 1.22% for RME and 0.88% for REE after 24 months compared to beginning values.
4. Viscosity increased 23.1% for RME and 16.87% for REE after 24 months compared to the beginning values.
5. Heat of Combustion declined 1.50% for RME and 1.27% for REE after 24 months compared to the beginning values.
6. The cetane number increased more than 12% and particulate matter increased from 1.5 mg/L to 53 mg/L during storage.
7. Regression models showed significant location effects (due to temperature differences in storage) for peroxide, density, viscosity, and heat of combustion.
8. Regression models showed second order time effects for acid value, density, and viscosity.
9. In general outside samples stored slightly better than indoor samples. It was noted that during the fall and winter the change in samples stored outdoors was less than those inside. In the summer when outside temperatures were higher the changes were more equal.
10. The short-term engine tests, including torque, power, fuel economy, and coking showed very small differences between the stored fuels and the new fuels. The increased peroxide and acid values of the stored fuels had little effect on the performance of the engine in short term tests. Long-term durability tests would be the next step needed to determine if these changes affect engine life. The small amount of fuel stored in this study precluded such testing.

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