The Production of Fatty Acid Isopropyl Esters and Their Use as a Diesel Engine Fuel

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ABSTRACT: Biodiesel is an alternative fuel for diesel engines that consists of the monoalkyl esters of vegetable oils or animal fats. Currently, most biodiesel consists of methyl esters, which have poor cold-flow properties. Methyl esters of soybean oil will crystallize and plug fuel filters and lines at about 0°C. However, isopropyl esters have better cold-flow properties than methyl esters. This paper describes the production of isopropyl esters and their evaluation in a diesel engine. The effects of the alcohol amount, the catalyst amount, and two different catalysts on producing quality biodiesel were studied. Both sodium isopropoxide and potassium isopropoxide were found to be suitable for use in the transesterification process. A 20:1 alcohol/TG molar ratio and a catalyst amount equal to 1% by weight (based on the TG amount) of sodium metal was the most cost-effective way to produce biodiesel fuel. The emissions from a diesel engine running on isopropyl esters made from soybean oil and yellow grease were investigated by comparing them with No. 2 diesel fuel and methyl esters. For nitrogen oxide emission, the difference between the biodiesel produced from soybean oil and yellow grease was greater than the difference between the methyl and isopropyl esters of both feedstocks. The other emissions from using isopropyl esters were about 50% lower in hydrocarbons, 10–20% lower in carbon monoxide, and 40% lower in smoke number when compared with No. 2 diesel fuel.

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KEY WORDS: Biodiesel, diesel fuel, isopropanol, isopropyl esters

Vegetable oils have been investigated as alternative diesel fuels for many years (1). This research has shown that long-term use of vegetable oils can lead to injector coking, thickening of the crankcase oil, and the formation of in-cylinder deposits, leading to piston ring sticking (2–5). Therefore, vegetable oils are not recommended for use in diesel engines.

By transesterifying the vegetable oils, the mono alkyl esters of the FA present in the vegetable oil can be obtained. These esters are commonly referred to as *biodiesel*. Biodiesel is an alternative fuel that is renewable in the sense that its primary feedstock has a sustainable source. In addition to vegetable oil, some other less expensive feedstocks that can be converted to biodiesel are restaurant waste grease and animal fat.

The emissions produced from biodiesel are generally lower than those of petroleum-based diesel fuel. Particulate emissions, soot, unburned hydrocarbons (HC), and carbon monoxide (CO) are lower since biodiesel is an oxygenated fuel. However, emissions of nitrogen oxides (NO_x) are higher (6,7). The cause of the rise in NO_x is unclear and is being studied (8–12).

One particular problem with biodiesel is its cold-flow properties. At low temperatures, crystallization can occur, which leads to plugging of the fuel filters and lines. Neat biodiesel made with methanol and soybean oil has a pour point (i.e., the lowest temperature at which the fuel is pourable) of −3°C (13). Typically in the United States, biodiesel is blended with diesel fuel. A B20 blend would be 20% biodiesel in diesel fuel. Such a blend would have better cold-flow properties compared with neat biodiesel. Johnson and Hammond (14) invented a method to reduce the crystallization temperature of a petroleum/biodiesel blend by using branched-chain esters.

The research done by Lee *et al.* (13) compares the crystallization onset temperatures for methyl and isopropyl esters made from soybean oil and tallow. For soybean oil, they found the crystallization onset temperature of methyl esters to be 5.2°C, and that of isopropyl esters to be −6.0°C; for tallow methyl esters, the crystallization onset temperature was 17.8°C, and for isopropyl esters it was 6.6°C. For both feedstocks, the crystallization onset temperature for the isopropyl esters was more than 11°C lower than for the methyl esters.

Biodiesel made from tallow has a higher crystallization onset temperature because it has a greater fraction of esters from saturated FA. The amount of saturated esters is the most important factor determining the biodiesel cold-flow properties. The m.p. for pure isopropyl and methyl esters from the two most common saturated FA in the feedstocks for this study, palmitic acid (16:0; hexadecanoic acid) and stearic acid (18:0; octadecanoic acid), are as follows (15): Palmitic acid methyl esters have a m.p. of 30°C, and for isopropyl esters it is 13°C; for stearic acid methyl esters, the m.p. is 39.1°C, and for isopropyl esters it is 28°C. These data confirm that isopropyl esters will have better cold weather performance.

Although the cold-flow benefits of using isopropyl esters are clear, the effects of this fuel on the performance of and emissions from a diesel engine have not been reported. Wu and colleagues (16) conducted engine performance tests using a 20% blend of isopropyl esters with diesel fuel. However, because of their limited fuel supply, the data collected in their study were very preliminary and their study was not definitive for the effects of isopropyl ester fuels on diesel engine performance. The U.S. Environmental Protection Agency (EPA) has released a draft technical report on the impact of biodiesel on exhaust

emissions (17). In this study, pre-existing data from various emissions test programs were analyzed. However, in the database that was collected, the fuels were not clearly distinguished as either methyl or ethyl esters, and isopropyl esters were probably not included.

Commonly, most biodiesel consists of methyl esters, and methanol is used since it is low cost and widely available. The contract price for methanol is $250.35–251.19/metric ton (MT) (18), whereas isopropanol is priced between $1,014–1,036/MT (19), which makes it more expensive to make isopropyl esters. However, the yield for isopropyl esters is about 10% more than for methyl esters because of their heavier M.W., so this partially compensates for the increased cost.

Research done by Lee *et al.* (13) utilized isopropyl esters made from a 66:1 ratio of alcohol/TG. Typically, a 6:1 molar ratio of methanol/TG is used for the transesterification process to produce methyl esters. The M.W. of methanol is 32.04 and that of isopropanol alcohol is 60.09. The heavier M.W. of isopropanol, compounded with the need for more moles of alcohol per mole of TG, increases the cost of producing isopropyl esters. Although the excess alcohol can be recovered and recycled, this is complicated for isopropanol because of the azeotrope it forms with water.

EXPERIMENTAL PROCEDURES

In this study, the amount of isopropanol was varied to examine the completeness of the transesterification process. The three different molar ratios tested were 20:1, 10:1, and 6:1. The catalyst amount was also varied for each of these alcohol amounts. The completeness of the reaction was determined using the total, free, and combined glycerol iodometric-periodic acid method (AOCS Official Method Ca 14-56) (20). The total glycerol was defined as the sum of the free glycerol and the glycerol portion of any unreacted or partially reacted oil or fat (20). Low levels of total glycerol (<0.240 %) meant that a high conversion of TG to monoalkyl esters had taken place (21).

Experimental batches were made using fully refined soybean oil (Baker and Chefs). The same amount of oil (100 g) was used for each transesterification process, and the reaction temperature was maintained between 70 and 86°C. To ensure no loss of alcohol, a reflux condenser was used. The setup is shown in Figure 1. All batches were reacted for at least 8 h. After the reaction, the contents of the reactor were placed in a separatory funnel. There was no separation of glycerol and biodiesel, so warm water (*ca.* 60°C, 50% of the volume of biodiesel) had to be added to aid the separation process and to wash the product. After several warm-water washes, clean biodiesel was obtained by gravity separation and its weight was measured to provide the yield.

Sodium isopropoxide was produced in the laboratory by dissolving sodium metal in isopropanol. To speed up this process, the alcohol was agitated and heated on a stir plate. The isopropanol was heated to about 80°C, just below its b.p. of 82.2°C (at 760.0 mmHg). Since sodium isopropoxide was not commercially available in the quantity needed for pilot-scale
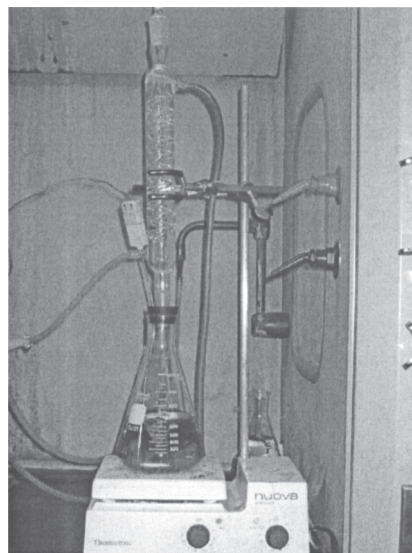

FIG. 1. Small reactor setup.

fuel production, potassium isopropoxide was investigated. The potassium isopropoxide was purchased from Callery Chemical Company (Evans City, PA) as a 20% (w/w) solution in isopropanol. Isopropanol was purchased from Barsol Solvents (Des Moines, IA).

The possibility of using isopropanol for the acid-catalyzed pretreatment of yellow grease was also investigated. This process converts the FFA to alkyl esters and is usually followed by a base-catalyzed transesterification of the TG. Methanol and ethanol were used for comparison. All reactions used a 20:1 molar ratio of alcohol to FFA and 5% sulfuric acid (based on FFA content). For this test, the base material was yellow grease from National By-Products (Des Moines, IA) with a FFA content of 13%. Agitation was provided by a magnetic stirrer and the reactions were carried out at the b.p. of the alcohol with a reflux condenser. The progress of the acid-catalyzed esterification reaction was determined using the acid value test (AOCS Official Method Cd 3d-63) (22).

The biodiesel pilot plant located at the Iowa Energy Center's Biomass Energy Conversion Center (BECON) in Nevada, Iowa, was used to produce a sufficient quantity of isopropyl and methyl esters for engine testing. Soybean oil was pressed within the facility itself, and yellow grease was obtained from National By-Products (Des Moines, IA). The yellow grease had an FFA content of 8.2% and was pretreated with sulfuric acid and methanol following the process described by Canakci and Van Gerpen (23,24). After pretreatment, the FFA level was reduced to 0.86%. The transesterification was performed using a 20:1 molar ratio of alcohol/TG and an amount of potassium isopropoxide that corresponded to 1.34% potassium metal based on the weight of the oil.

A John Deere 4045T, which is a 4.5-L, four-cylinder, four-stroke, turbocharged diesel engine, was used for the exhaust emission characterization portion of this research. The engine was coupled to a General Electric DC dynamometer (TLC 2544). To completely characterize the effects of the isopropyl es-

TABLE 1
Fuel Properties

Test property	No. 2 diesel fuel	Soybean oil methyl esters	Yellow grease methyl esters	Soybean oil isopropyl esters	Yellow grease isopropyl esters
Carbon (% mass)[d]	86.66[a]	77.00	76.66	77.90	77.40
Hydrogen (% mass)[d]	12.98[a]	12.18	12.33	12.13	12.49
Oxygen (% mass)[d]	—	10.82	11.01	9.97	10.11
C/H ratio	6.676	6.322	6.217	6.422	6.197
Sulfur (% mass)	0.034[a]	<0.005	<0.005	<0.005	<0.005
Typical formula	$C_{14.01}H_{25.00}$	$C_{18.74}H_{34.51}O_2$[d]	$C_{18.40}H_{35.26}O_2$[d]	$C_{20.76}H_{38.53}O_2$[d]	$C_{20.27}H_{38.99}O_2$[d]
Average M.W.	194.14[b]	291.73[d]	288.29[d]	320.14[d]	314.53[d]
Cetane number (ASTM D 613)[a]	42.2	50.4	62.6	51.5	65.6
Hydrocarbon type, FIA (ASTM D 1319)[a]	—	—	—	—	—
Saturates	56.6	—	—	—	—
Olefins	1.6	—	—	—	—
Aromatics	41.8	—	—	—	—
Gross heat of combustion[a] (Btu/lb)	19419	17183	17252	17324	17367
Net heat of combustion[a] (Btu/lb)	18235	16072	16209	16155	16239
Specific gravity[c] (@ 60°C)	0.8559	0.8796	0.8722	0.8704	0.8617
Kinematic viscosity[c] (@ 40°C, mm²/s)	2.8911	4.5926	5.9156	5.2649	6.0997
Cloud point[c] (°C)	−18	−2	8	−9	0
Pour point[c] (°C)	−30	−6	6	−12	−3
Total glycerin[c] (%) (AOCS Ca 14-56)	—	0.175	0.194	0.227	0.082

[a]Measured by Phoenix Chemical Laboratory Inc. (Chicago, IL).
[b]Calculated using Universal Oil Products (UOP) Method 375-86 (26).
[c]Measured at the Mechanical Engineering Department, Iowa State University (Ames, IA).
[d]Calculated from the FA profile unless stated otherwise. A dash (—) indicates items that are not applicable. FIA, fluorescent indicator adsorption.

ters of soybean oil and yellow grease in a diesel engine, the testing also included No. 2 diesel fuel and methyl ester-based biodiesel produced from the same soybean oil and yellow grease feedstocks as the isopropyl esters (25). The testing of the five fuels was conducted in random order, and each test point was repeated three times. The properties of the five fuels are shown in Table 1. A more detailed description of the test procedures and the equipment setup can be found in Reference 12.

The engine emissions are reported on a brake-specific basis (g/kW-h) for a steady-state operating condition of 352.5 N-m and 1400 rpm, which corresponds to peak torque for the engine. The measured emissions were CO, carbon dioxide (CO_2), unburned HC, NO_x, and the Bosch smoke number.

The objective of the engine testing was to observe whether there were any differences between the pollutants from the combustion of the four types of biodiesels and the No. 2 diesel fuel. An ANOVA was performed to see whether at least two of the means were unequal. This was followed by a statistical analysis using the Tukey method to determine whether there was any significant difference among the means of all possible pairs.

RESULTS AND DISCUSSION

The two most important variables in the transesterification reaction are the amount of excess alcohol and the amount of catalyst. The indicator of reaction completeness is the residual total glycerin in the biodiesel. The total glycerol values for three different ratios of isopropanol/TG and for differing amounts of catalyst are plotted in Figure 2 for soybean oil. The best combination for producing a quality fuel was determined

to be a 20:1 alcohol/TG ratio and 1% sodium metal by weight with respect to the TG. Lesser amounts of alcohol produced a fuel that had a higher total glycerol amount than that allowed by the standard ASTM D 6751-03a (21). The trends for the 10:1 and 6:1 ratios did not extend as far as the 20:1 ratio because when excess sodium metal was introduced, the sodium isopropoxide solidified. Although the reaction can take place by adding heat and maintaining strong agitation, the biodiesel is hard to separate from the alcohol and glycerol after the reaction is complete. The yields for lesser amounts of alcohol were not high since it was difficult to extract the biodiesel.

Table 2 shows the total amounts of glycerol for the different alcohol ratios when using potassium isopropoxide as a catalyst.
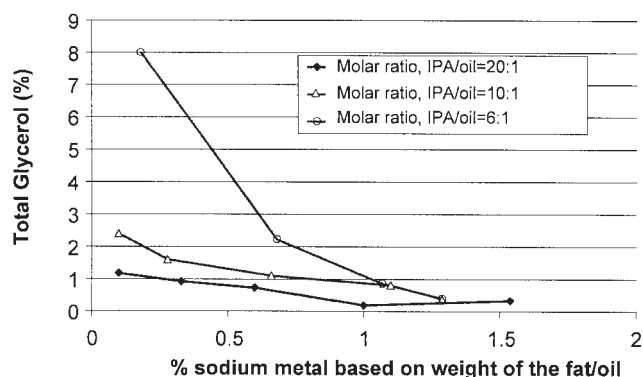

FIG. 2. Effects of alcohol and sodium amounts on completeness of reaction for soybean oil. IPA, isopropanol.

TABLE 2
Effects of Alcohol Ratio and Catalyst on the Completeness of the Reaction

Alcohol/TG ratio	Potassium (% wt)	Total glycerol (% wt)
6:1	1.34	0.64
10:1	1.34	0.70
20:1	0.67	0.33
20:1	1.34	0.04

As before, the alcohol ratio that was considered to work best was 20:1. To ensure completeness of the reaction, 20 mL of the 20% (w/w) potassium isopropoxide solution in isopropanol was added to 100 g of soybean oil, which corresponds to 1.34% potassium.

Figure 3 depicts how the acid value changed with time when acid-catalyzed esterification was used to convert the FFA in a high-FFA oil to the corresponding monoalkyl ester. As shown in Figure 3, methanol had the best reactivity, followed by ethanol, and isopropanol was the least effective in pretreating the yellow grease. Based on these results, methanol was chosen for the pretreatment because of its better reactivity. Once the FFA content of the yellow grease had dropped below 1%, the remaining unreacted TG could be converted to isopropyl esters using 1.34% potassium catalyst (as potassium isopropoxide) and a 20:1 isopropanol/TG ratio. The resulting fuel was an isopropyl–methyl ester blend. Since the fuel was mostly isopropyl esters (91.8% by wt), the yellow grease-based fuel blend is identified hereafter as yellow grease isopropyl esters.

An isopropyl–methyl ester blend retains a portion of the cold-flow properties of pure isopropyl esters, depending on the amount of methyl esters present. Measurements of the cold flow for soy-based biodiesel showed that 5% (w/w) methyl esters in 95% isopropyl esters raised both the cloud and pour points by 5°C above the corresponding temperatures for 100% isopropyl esters. Ten percent methyl esters in 90% isopropyl esters raised both temperatures by 7°C compared with 100% isopropyl esters. Figure 4 shows the brake-specific emissions for the various fuels normalized by the average brake-specific emission for the baseline No. 2 diesel fuel. In Figure 4, fuels that do not share the same letter or letters are significantly different. The error rate of α = 0.05 applied to the family of all paired comparisons, which corresponds to a 95% confidence level.

CO emissions. All four biodiesel fuels exhibited a reduction in CO emissions compared with the baseline fuel, with the soybean methyl esters having the largest drop, 33.7%. Isopropyl esters from soybean oil had the smallest reduction, with a 10.3% drop. Canakci (7) found the CO emissions for soy methyl esters and yellow grease methyl esters to be 18.2 and 17.8% lower compared with diesel fuel. Monyem (6) found a 15.7 and 15.8% drop for the same fuels when compared with No. 2 diesel fuel. The mean for the CO emission from No. 2 diesel fuel was significantly different from all of the biodiesel fuels. Additionally, the means of the isopropyl esters and methyl esters from the same source material were significantly different, whereas the methyl esters from soybean oil and yellow grease were not significantly different.
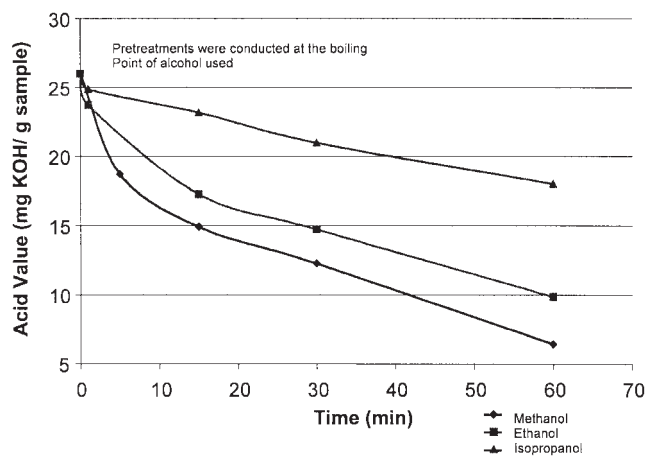

FIG. 3. Acid value of yellow grease under acid pretreatment.

CO_2 emissions. CO_2 emissions were not shown in Figure 4 because, according to the Tukey grouping, the means for the brake-specific CO_2 emissions were not significantly different for any of the fuels, which is consistent with previous research (6,7).

Unburned HC emissions. All four biodiesels showed statistically significant reductions in unburned HC when compared with No. 2 diesel fuel. Although the yellow grease methyl esters had a 53.3% drop in HC emissions and yellow grease isopropyl esters experienced a 42.9% drop, the means for all four biodiesels were not significantly different from each other. Canakci (7) reported drops of 46.3 and 42.5% in HC for yellow grease methyl esters and soy methyl esters, respectively, when compared with No. 2 diesel fuel. Monyem (6) found a decrease of 53.2% in HC when soy methyl esters were used.

NO_x emissions. Although the other emissions for the various biodiesel fuels were consistent, the NO_x emission results were more variable. As can be seen in Figure 4, the NO_x emissions were slightly lower for both yellow grease-based biodiesel fuels than for diesel fuel and were higher for the soy
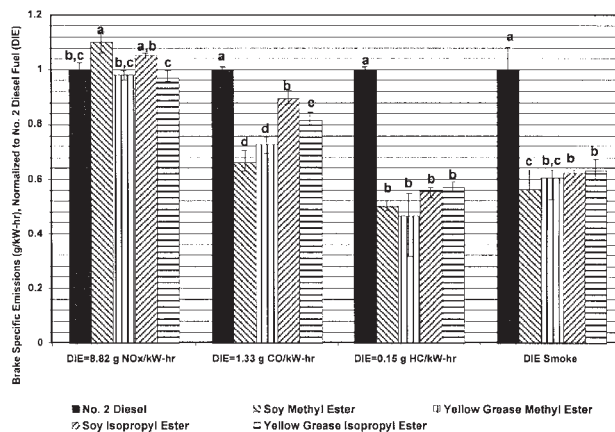

FIG. 4. Comparison of emissions from various biodiesels relative to diesel fuel. Error bars show the maximum and minimum values of three independent measurements. Fuels that do not share the same letter or letters are significantly different. DIE, diesel; NO_x, nitrogen oxides; CO, carbon monoxide; HC, hydrocarbons.

oil-based biodiesel fuels. When comparing the means using the Tukey test, the 10.3% rise in NO_x emissions for soy methyl esters was found to be statistically significant when compared with the base No. 2 diesel fuel. Although there was a 5.4 % rise in NO_x emissions when comparing soy isopropyl esters with No. 2 diesel fuel, this increase was not statistically significant. Likewise, the NO_x emissions of both yellow grease-based biodiesels were not significantly different from that of No. 2 diesel fuel. When comparing the means of the NO_x emissions for methyl and isopropyl esters originating from the same source material (i.e., comparing soy methyl esters to soy isopropyl esters), whether it was yellow grease or soy oil, the means were not statistically different. These results suggest that the source of the feedstock oil affected NO_x emissions more than the type of alcohol used in producing the biodiesel.

Others have noted that soy-based biodiesel can cause a rise in NO_x emissions (8). The rise in NO_x can at least partially be attributed to the properties of biodiesel causing changes in the engine's fuel injection and combustion timing (9–12). These timing changes have been shown to cause NO_x increases of the same magnitude as those observed between biodiesel and diesel fuel.

Bosch smoke numbers. All four biodiesels displayed significant reductions in Bosch smoke numbers. The smoke numbers were lower by 43.6, 39.4, 37.8, and 36.9% for soybean-based methyl esters, yellow grease-based methyl esters, soybean-based isopropyl esters, and yellow grease-based isopropyl esters, respectively. By comparison, Monyem (6) reported a 56.9% drop in smoke number when using soybean methyl esters. The Tukey tests for comparison of the means showed that the change from diesel fuel to biodiesel was statistically significant. The difference in the means for both of the isopropyl esters was not statistically significant. This was true for the methyl esters as well.

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