Low-Temperature Property and Engine Performance Evaluation of Ethyl and Isopropyl Esters of Tallow and Grease[1]

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ABSTRACT: Three monoalkyl fatty acid esters derived from tallow and grease were prepared by lipase-catalyzed transesterification and evaluated as prospective diesel engine fuels. The low-temperature properties of the esters, both neat and as 20% blends in No. 2 diesel fuel, were evaluated. Those properties included cloud point, pour point, cold filter plugging point, low-temperature flow test, and crystallization onset temperature. Other properties of the esters, such as kinematic viscosity, heating value, and calculated cetane number, also were determined. All three esters had acceptable physical and low-temperature properties, as well as acceptable fuel properties at the 20% level in diesel blends. Engine performance and emissions for the ester blends were determined in a direct-injection, matched two-cylinder diesel engine. Among the monoalkyl esters studied, ethyl greasate had better properties and engine performance characteristics than the two tallow esters. For the latter esters, isopropyl tallowate had better properties than ethyl tallowate.

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KEY WORDS: Biodiesel, carbon buildup, cetane number, cloud point, cold filter plugging point, crystallization onset temperatures, emissions, engine performance, low-temperature flow test.

There is an increasing interest in the development of alternative fuels to reduce the dependency of the United States on imported petroleum and to reduce the environmental burden from petroleum-based fuels. Considerable effort has been spent on the use of renewable fats and oils (triacylglycerols) as alternative diesel engine fuels because heavy-duty diesel engines have been identified as significant sources of pollutants (1–3). However, the high viscosity of vegetable oils in general has been recognized as one major impediment to their use as neat diesel fuels. Previous studies have shown that the viscosity of fats and oils can be reduced when they are converted to their respective monoalkyl esters (4–8). More recently, additional evidence has suggested other beneficial effects of monoalkyl esters in the form of lower emissions (9–11), improved biodegradability, and minor contributors to the greenhouse effect when used as fuels known as biodiesel (10,11). In addition to biodiesel, lubricants and lubricant additives also can be derived from fats and oils.

Tallow was chosen as one material in our study because the United States produces more tallow than the rest of the industrialized world (12). The demand for tallow in the global food market has gradually decreased due to health concerns and competition from other fats and oils. Value-added products, such as nutraceuticals, cleaning solvents and biofuels, have to be developed from tallow to improve its commercial value. Recycled restaurant grease was incorporated in our study because it can be less expensive than tallow.

Previously, Nelson and coworkers (13) demonstrated the application of lipase-catalyzed transesterification to the production of alkyl esters—including methyl esters from soybean oil, rapeseed oil, tallow, and recycled restaurant grease—that could be used as biodiesel. They also showed that the low-temperature properties of monoalkyl esters derived from tallow and grease were significantly improved when branched alcohols were used for transesterification (14). Among the large varieties of alkyl esters synthesized, three alkyl esters, namely ethyl tallowate, isopropyl tallowate, and ethyl greasate, were selected in the present study for scale-up production and diesel engine performance tests because preliminary data suggested their potential as biodiesel fuels (14). The physical and low-temperature properties determined for the three selected monoalkyl esters included: cloud point (CP), pour point (PP), cold filter plugging point (CFPP), low-temperature flow test (LTFT), and onset temperature of crystallization (T_{co}), obtained with a differential scanning calorimeter. The chemical composition, residual free glycerol, and moisture content of the three alkyl esters prepared in this study also were determined. Engine performance of 20% (vol/vol) blends in No. 2 diesel fuel also was evaluated.

MATERIALS AND METHODS

Materials. Beef tallow with less than 0.75% free fatty acid was obtained from HRR Enterprise (Chicago, IL). Recycled restaurant grease was supplied by Kaluzny Bros., Inc. (Joliet,

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IL) and contained 8.5% free fatty acid on receipt. The fatty acid compositions of the tallow and grease used in the study are given in Table 1. Selected monoalkyl esters, including ethyl tallowate, isopropyl tallowate, and ethyl greasate, derived from the tallow and grease, were prepared in sufficient quantity (2.8 kg) by solvent-free lipase-catalyzed transesterification described previously (13). For the ethyl esters, Lipozyme IM60 of *Mucor miehei* (Novo Nordisk, Franklinton, NC) was used, and for the isopropyl ester, the lipase was SP435 of *Candida antarctica* (Novo Nordisk). Diesel fuel No. 2 was purchased from Phillips Petroleum (Bartlesville, OK). The 20% (vol/vol) alkyl ester blends in diesel fuel were prepared in volumetric flasks.

Chemical composition of alkyl esters. Percentage esters, free fatty acids, monoacylglycerols, diacylglycerols, and triacylglycerols in alkyl esters prepared from tallow and grease were determined by a high-performance liquid chromatography method developed earlier (15). Reaction mixtures were separated on a cyanopropyl (CN) column (PHENOMENEX, Torrance, CA) with a binary mobile phase of hexane and methyl *t*-butyl ether, both containing 0.4% acetic acid, and detected by an evaporative light scattering detector (Varex, Burtonsville, MD). Fatty acid profiles of the esters were determined by gas–liquid chromatography (GLC) after conversion to methyl esters by methylation with BF_3/MeOH (Sigma Chemical Co., St. Louis, MO). The residual free glycerol in the alkyl esters was measured by a gas chromatography/mass spectrometry (GC/MS) procedure described by Mittelbach (16). A Karl Fischer coulometer (Mettler Toledo, Hightstown, NJ) was used to determine the moisture content of the alkyl esters.

Physical and low-temperature properties of alkyl esters. Kinematic viscosity of the esters was measured at 40°C with Cannon-Fenske opaque reverse-flow viscometers, obtained from Koehler Instrument Co., Inc. (Bohemia, NY), according to ASTM method D 445 (17). CP and PP were determined by ASTM methods D2500 and D97, respectively. LTFT (ASTM D4539) and CFPP were performed at the National Center for Agricultural Utilization Research (Peoria, IL) as described by Dunn *et al.* (18). T_{co} is defined as the lowest temperature at which the sample was completely liquid, as determined by melting curves obtained in a differential scanning calorimeter (DSC7; Perkin-Elmer, Norwalk, CT) at a scanning rate of 5°C/min by following the protocol of Lee *et al.* (19). Sulfur content and heating values of the neat alkyl esters were measured at Phoenix Chemical Labs (Chicago, IL).

Engine performance and emissions tests. A direct-injection, water-cooled and naturally aspirated Lister-Petter Model LPW2 (Lister-Petter, Gloucestershire, England) diesel engine, equipped with two cylinders (465 mL each), was used to evaluate the 20% ester-diesel blends. Because of the limited quantity of fuel available for engine tests, cylinder 1 was supplied with neat reference diesel fuel, and cylinder 2 was supplied with the 20% fuel blend. Previous work with this engine arrangement showed that there can be a bias of results, even with identical injection timing in both cylinders (20). To determine what bias, if any, was present, a run with both cylinders fueled with the reference fuel was made. The bias was then subtracted out of all test results to give a better indication of the actual performance of the test fuels. The limited amount of test fuels also required changes to the fuel supply system of the engine. Fuel was supplied to the engine from two tanks pressurized with air. Ordinarily, each tank supplied one cylinder with fuel, and the supply line had a positive displacement flow meter between the tank and the engine. However, a large quantity of fuel was required for purging the lines of air and diesel fuel, which was used in both cylinders while the engine was warmed up. There was insufficient test fuel to purge the lines and still have enough for the full test. Therefore, the fuel system was modified with a short bypass fuel line that ran directly from the tank supplying the test fuel to the fuel pump for cylinder 2. This arrangement prevented measuring the fuel consumption of the engine directly. The intake air pressure was maintained at 101.3 ± 0.5 kPa. The engine oil was preheated to 85°C to accelerate the warm-up of the engine for all tests. The engine was controlled with a General Electric Model TLC-7.5 DC dynamometer (Schenectady, NY), controlled by a Dyn-Loc IV system and a DTC-1 digital throttle controller. The Dyn-Loc IV held the engine at a constant speed of 2200 rev/min, while the DTC-1 was set to maintain the engine at 40.0 N-m of torque during tests. Cylinder pressure measurements were taken *via* AVL 8QP-500ca piezoelectric pressure tranducers (AVL North America, Plymouth, MI), located in the head of each cylinder. The trans-

TABLE 1
Fatty Acid Profile of Alkyl Ester Derivatives of Tallow and Grease

	Composition[a] (%)				
Fatty acid	Tallow[b]	Ethyl tallowate	Isopropyl tallowate	Grease[b]	Ethyl greasate
14:0	3.05	3.38	3.40	0.68	0.72
16:0	27.55	26.97	27.09	14.44	14.33
16:1	2.45	2.29	2.39	1.08	1.33
17:0	1.25	1.22	1.26	—	—
18:0	24.95	24.56	24.31	9.23	9.22
18:1	38.64	39.06	39.00	48.57	47.01
18:2	2.11	2.53	2.55	23.74	25.27
18:3	—	—	—	2.26	2.32

[a]Determined as fatty acid methyl esters.
[b]Composition of tallow and grease used in preparation of alkyl esters.

ducers were mounted over the piston bowls, and the faces of the transducers were covered with high-temperature silicone gasket material to protect them. The transducers were connected to either a Kistler model 504E or 503 charge amplifier (Kistler Instrument Corporation, Amherst, NY). For each cylinder, the specific transducer–amplifier combination was calibrated, and that pairing was maintained for all tests. Needle lift data were also recorded to provide information on injection timing and duration. The fuel injectors were standard Lister-Petter parts, which were then modified by Wolff Controls Corporation (Winter Haven, FL). Each injector had a hall-effect sensor installed to detect the needle lift. Each injector's sensor was connected to a Wolff Controls Universal Signal Conditioner. Timing for the acquisition of both injection pressure and needle lift was provided by a shaft encoder, mounted to the crankshaft of the engine. The encoder was a BEI model XH25D-SS-1440-ABZC-7406R-LED-EM18-S rotary shaft encoder (BEI Sensors & Systems Company, Goleta, CA). It provided a pulse every time cylinder 1 reached top dead center (TDC) on one output channel and a pulse every 0.25° on a second channel.

Data acquisition (except emissions) was accomplished with a Hewlett-Packard 3852A data acquisition computer (Hewlett-Packard Company, Santa Clara, CA). Static temperatures and pressures were measured with a model 44701A 5 1/2 to 3 1/2 digit integrating voltmeter module, fed by a model 44708A 20-channel relay multiplexer module. High-speed cylinder pressure and needle lift data were measured by two model 4470B 13-bit high-speed voltmeter modules. The high-speed data were multiplexed so that one voltmeter measured the pressure in cylinder 1 and the needle lift in cylinder 2, while the other did the converse. The two data streams were separated out in the postprocessing software. The control program for the data acquisition was a custom-written QuickBASIC (Microsoft Corporation, Redmont, WA) program that ran on a MS-DOS-based computer.

For carbon buildup characteristics, measurements were taken of the cross-sectional area of the injector nozzles before and after each test run, along with the growth in this area. Measurements were also taken of the deposit depths on the cylinder head and on the piston face. The exhaust emissions were measured by a Beckman Industrial Model 402 HC analyzer (Beckman Corp., LaHabra, CA), a Rowemount Analytical Model 955 NO/NO_x analyzer (Rosemount Analytical, LaHabra, CA), two Beckman Industrial Model 880 analyzers for CO and CO_2, and a Beckman Industrial OM-11 O_2 analyzer.

RESULTS AND DISCUSSION

The fatty acid profiles of the three monoalkyl esters derived from tallow and grease in general reflected the fatty acid profiles of the materials from which they were prepared (Table 1). Fatty acid profiles of the two tallowates were similar to each other, with oleic acid being the predominant fatty acid, followed by palmitic and stearic acids. Both tallowate esters had iodine values (IV) between 50 and 60. On the other hand, ethyl greasate, with iodine number of approximately 100, contained less saturated fatty acids than the two tallowates because it was a recycled restaurant grease that contained a high level of vegetable oil, presumably hydrogenated soybean oil. Although IV, a measure of unsaturation, is included in the German specification for biodiesel as an indicator for stability, it is not considered in proposed ASTM specifications because it is possible for two samples to have the same IV but different stability performance (21). The U.S. biodiesel industry currently is planning further work to determine appropriate measurement techniques for stability and the correlation to engine performance. Nevertheless, limitation of unsaturated fatty acids (e.g., linolenic acid) in biodiesel has been suggested because heat polymerization of polyunsaturated esters has been implicated in the formation of deposits and deterioration of the lubricating oil (22). The high saturated fatty acid level of the tallowates may in fact prove to be advantageous in terms of storage stability compared to the more unsaturated vegetable oil-based fuels, which are more susceptible to chemical deterioration (e.g., autoxidation and polymerization) under certain conditions of storage temperature, moisture, ultraviolet radiation, and container construction.

Of the three preparations, ethyl tallowate contained nearly 100% alkyl ester and almost no glyceride or free fatty acid impurities (Table 2). Isopropyl tallowate had about 97.5% alkyl ester, a total of 1.4% glycerides, and 1.1% free fatty acids. The preparation of ethyl greasate showed higher residual glycerides (a total of 2.4%) and free fatty acids (2.4%) compared to the two tallowates, but this was not unexpected considering the starting material for this preparation, which contained nearly 13% partial glycerides (Table 2). No triacylglycerols were detected in any of these preparations. Residual free glycerol in the three monoalkyl esters met the 0.02% proposed ASTM standard (21). Moisture contents of the three monoalkyl esters were 0.02, 0.05, and 0.02% for ethyl tallowate, isopropyl tallowate, and ethyl greasate, respectively, equal to or less than the proposed ASTM standard of 0.05% (21) (Table 2).

Physical and low-temperature properties of the three monoalkyl esters and their 20% blends in No. 2 diesel fuel are shown in Table 3. Properties of methyl soyate, the main form of alkyl esters currently available in the U.S. biodiesel market, also are included for comparison. Kinematic viscosities of the three monoalkyl esters were close to the proposed ASTM specification for biodiesel (1.9–6.0 mm^2/s) (21). Viscosity values for the ester-diesel blends (20:80, vol/vol) were in the acceptable range of 3.1 to 3.3 mm^2/s, compared to 3.0 mm^2/s for 20% methyl soyate-No. 2 diesel blend and 2.8 mm^2/s for No. 2 diesel fuel. Viscosity of isopropyl tallowate (6.4 mm^2/s) was higher than ethyl tallowate (5.2 mm^2/s) due to the increased molecular weight of the isopropyl esters, which paralleled previous findings (14). In general, monoalkyl esters derived from grease seemed to have better low-temperature properties than the tallow esters (Table 3). T_{co} was determined because it can be used to predict CP from

TABLE 2
Chemical Composition (%) of Alkyl Esters of Tallow and Grease[a]

	Tallow	Ethyl tallowate	Isopropyl tallowate	Grease	Ethyl greasate
Alkyl esters	0	100	97.5	0	95.2
Monoacylglycerols	<0.01	<0.1	0.6	0.8	1.6
Diacylglycerols	<0.01	<0.1	0.8	12.3	0.8
Triacylglycerols	100	<0.1	<0.1	78.2	<0.1
Free fatty acids	<0.75	<0.1	1.1	8.7	2.4
Free glycerol	—	0.02	0.01	—	0.01
Moisture	0.05	0.02	0.05	0.42	0.02

[a] Alkyl esters, mono-, di-, triacylglycerols, and free fatty acids determined by high-performance liquid chromatography, glycerol by gas chromatography/mass spectrometry, and water by Karl-Fischer. See Materials and Methods section for detail.

the melting curve (23). However, caution must be taken when using this correlation in that T_{co} from cooling curves may be significantly affected by the rate of cooling (19). Although the low-temperature properties of the three neat monoalkyl esters did not compare favorably with the properties of diesel fuel, the data in Table 3 indicate that the low-temperature properties of the ester-diesel blends were acceptable. In particular, low-temperature properties of the 20% ester-blend of ethyl greasate were similar to those of the 20% methyl soyate-No. 2 diesel blend.

The heating values of the three neat esters also are shown in Table 3. The heating value of a fuel is important because it is a measure of the energy content of the fuel and hence will influence fuel economy. The heating value is also the basis for calculating the thermal efficiency of an engine running on that fuel (24). All three esters had approximately the same level of gross heat as methyl soyate, around 40,000 kJ/kg. These values are also close to the reported heating value of No. 2 diesel fuel (Table 3). The ignition delay time of a fuel, when injected into the combustion chamber of a diesel engine, is indicated by its cetane number (24). The standard method, ASTM D975, calls for a minimum cetane number of 40 for conventional diesel fuel, which also is the proposed ASTM standard for biodiesel (21,25). The lowest reported cetane number for methyl soyate is 46.2 (26). Cetane numbers of the monoalkyl esters could not be determined in a Cooperative Fuel Research engine in the present study owing to the limited availability of the esters. However, cetane numbers of the three neat esters were estimated by using a spreadsheet calculation (Goering, C.E. unpublished) based on the weighted average cetane number for each individual fatty ester (27). This was done to facilitate heat-release calculations for the ester blends. The calculated cetane numbers were 65.9, 62.8, and 54.3 for ethyl tallowate, isopropyl tallowate, and ethyl greasate, respectively. The relatively high cetane numbers of the two tallow esters probably resulted from their high saturated ester ($C_{16:0}$ and $C_{18:0}$) content. Similarly, cetane number for ethyl greasate was closer to 50 because this ester contained much less saturated ester than the two tallowates and higher amounts of polyunsaturated fatty ester. All three monoalkyl esters were low in sulfur (<0.005%), as expected, and below the proposed ASTM biodiesel standard of 0.05% (21).

Results from the diesel engine performance and emissions tests for the ester-diesel blends are summarized in Table 4. By comparing performance of the two cylinders of the test diesel engine, we found that the ester-diesel blends resulted in a 1 to 3% higher indicated mean effective pressure than those from the diesel fuel and therefore provided 1 to 3% more power than the diesel fuel. These data were determined from the cylinder pressures recorded by the data acquisition system every 5 min of running. Twenty cycles of cylinder pressure were averaged to produce each datapoint. The injection duration was determined directly from the needle lift data, which also provided the start of injection information. Owing to limited fuel availability, it was not possible to directly measure the fuel consumption of the engine running on the ester-diesel blend. The fuel consumption was thus compared rela-

TABLE 3
Physical and Low-Temperature Properties of Alkyl Esters and Ester-Diesel Blends[a]

Fuel	Viscosity (mm^2/s, 40°C)	LTFT (°C)	CFPP (°C)	CP (°C)	PP (°C)	T_{co} (°C)	Gross heat (kJ/kg)
Ethyl tallowate (ET)	5.2	13	12	15	3	17.8	39,623
Isopropyl tallowate (IPT)	6.4	19	5	9	3	10.6	40,268
Ethyl greasate (EG)	6.2	9	0	5	–1	9.4	39,984
Methyl soyate (MS)[b]	4.3	2	–3	0	–2	—	39,800[c]
No. 2 Diesel fuel (D)	2.8	–14	–27	–16	–23	–8.5	45,200[c]
ET/D (20:80 vol/vol blend)	3.1	–1	–10	–6	–12	3.7	—
IPT/D (20:80 vol/vol blend)	3.2	12	–8	–10	–19	–5.3	—
EG/D (20:80 vol/vol blend)	3.3	–3	–12	–12	–21	–4.7	—
MS/D (20:80 vol/vol blend)[b]	3.0	–12	–14	–14	–21	—	—

[a] LTFT, low-temperature flow test; CFPP, cold filter plugging point; CP, cloud point; PP, pour point; T_{co}, crystallization onset temperature.
[b] Source: Reference 3, except for the heating values.
[c] Source: Reference 28.

TABLE 4
Diesel Engine Performance and Emissions Tests for Ester-Diesel Blends[a]

Test	Summary of observations (blends vs. diesel fuel)
Performance	1 to 3% higher indicated mean effective pressure; shorter injection durations (EG/D and IPT/D blends); increased torque and power.
Carbon buildup	All three ester-diesel blends showed modest improvement over diesel in buildup characteristics.
Emissions	0.25 to 0.5% reduction in CO_2; less than 1% increase in O_2; no apparent change in CO, HC, or NO_x.

[a]Ester-diesel blends (20:80, vol/vol) evaluated were ethyl tallowate (ET), isopropyl tallowate (IPT), and ethyl greasate (EG) in No. 2 diesel fuel (D).

tive to diesel fuel by using the injection duration data. Although this is only an approximate measure, relative effects should be valid because the engine was timed such that the duration was identical for both cylinders when both were running on diesel fuel. With this method, the isopropyl tallowate- and ethyl greasate-diesel blends showed shorter injection durations and thus lower fuel consumption than diesel fuel. The two blends also had higher combustion efficiency than diesel fuel because they provided equal or better power output. On the other hand, the ethyl tallowate-diesel blend had longer injection durations and higher fuel consumption for essentially equal power output as the diesel fuel.

Examination of selected areas of injector nozzles, cylinder heads, and piston faces after running the engine for 5 h showed that all three ester-diesel fuel blends generated less carbon buildup than did No. 2 diesel. Emissions for all three ester-diesel blend fuels were similar, with slightly lower CO_2 emissions and slightly greater O_2 emissions than those from the reference No. 2 diesel fuel, while no apparent changes in CO, HC, or NO_x emissions were found between the ester-diesel blends and the No. 2 diesel fuel. It has to be kept in mind, however, that engine durability and exhaust emissions data collected in this study are preliminary, owing to the short duration of testing and the indirect methods used to measure fuel flow.

In this study, we have demonstrated that selected monoalkyl esters of tallow and recycled grease have acceptable low-temperature properties and diesel engine performance when blended in diesel fuel. Furthermore, all three ester-diesel blends provided adequate performance as diesel extenders with no apparent negative impacts on power output, fuel consumption, combustion efficiency, carbon buildup, or CO_2, CO, O_2, HC, and NO_x emissions, according to the preliminary results from diesel engine tests conducted in this study. Among the three monoalkyl esters tested, ethyl greasate had the best potential for use as diesel fuel extender. We are currently investigating different approaches to improve the effectiveness and efficiency of production of biodiesel through enzymatic transesterification, such as the reuse of biocatalysts, immobilization of selective lipases, and the optimization of reaction parameters, for large-scale production of these esters for further long-term testing as biodiesel fuels.

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