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Butter as a Feedstock for Biodiesel Production

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Fatty acid methyl esters (FAME) were produced from cow's milk (Bostaurus) butter by esterification/transesterification in the presence of methanol. The product was assayed according to the Standard Specification for Biodiesel Fuel Blend Stock (B100) for Middle Distillate Fuels (ASTM D 6751). The preparation failed to meet the specifications for flash point, free and total glycerin contents, total sulfur, and oxidation stability. Failures to meet the flash point and free/total glycerin specifications were determined to be due to interference with standard assays for these parameters by short-chain-length fatty acid esters. The oxidation stability of the butterfat FAME was improved by supplementation with a commercial antioxidant formulation. Approximately 725 ppm of antioxidant was required to meet the ASTM-specified stability value for biodiesel. This work indicates that, without further purification to reduce a slightly excessive sulfur content, fatty acid ester preparations produced from butter are unacceptable as sole components of a biodiesel fuel. However, it is possible that even without further purification a butter-based ester preparation could be mixed with biodiesel from other feedstocks to produce a blend that meets the current quality standards for biodiesel. The results presented here also illustrate some potential weaknesses in the accepted methods for biodiesel characterization when employed in the analysis of FAME preparations containing mid- and short-chain fatty acid esters.

KEYWORDS: Biodiesel; butter; fatty acid methyl ester

INTRODUCTION

Virtually worldwide, there is strong and growing interest in the development from renewable resources of liquid fuels for transportation. For use in compression ignition (diesel) engines, one renewable alternative to petroleum-derived fuel is "biodiesel", which consists of the simple alkyl esters of fatty acids (FAME) derived from vegetable and animal fats and oils. With governments setting ambitious production targets for renewable fuel production, and growing interest and use on the part of citizens, biodiesel production is growing rapidly. U.S. production in 2008 has been estimated at 2.65 billion liters (2.35 mmt), up from 7.57 million liters (0.0067 mmt) in 2000 (*1*). In the European Union, the world leader in biodiesel production, 2007 output was over 3.42 billion liters (3.03 mmt) (*2*). With the Renewable Fuels Standard component of the Energy Independence and Security Act of 2007 committing the United States to the production of a total of 35 billion gallons of biofuels by 2022 (*3*), continued growth in biodiesel output is anticipated, with annual production expected to soon exceed 3.79 billion liters (1 billion gallons, 3.36 mmt).

Such vigorous growth in the production and use of biodiesel has triggered concerns over the availability of feedstocks to meet anticipated future growth in demand. These concerns are heightened by the fact that refined edible oils are the traditional feedstocks for biodiesel production. Their costs are relatively high in the context of feedstocks for commodity materials and will rise with further demand, increasing fuel costs. Present and future feedstock prices are of interest because even now feedstock costs typically constitute at least 75% of final fuel costs when using refined lipids as feedstocks. Further increases in the price of biodiesel could render it economically noncompetitive. Considerations such as these have stimulated interest in the development of other lipid sources as feedstocks for biodiesel production. Among these nontraditional feedstocks are animal fats, inedible vegetable lipids, lipid byproducts of edible fat and oil refining, crude and refined lipids from food service applications, and lipids from nontraditional sources such as algae. It is pertinent to consider any material rich in acylglycerols as a potential biodiesel feedstock.

In terms of organoleptic qualities, market price, and widespread use in edible applications, butter is the premier lipid product of animal agriculture. Although annual U.S. production is considerable, slightly above 1 billion pounds (454 million kilograms, 498 million liters) (*4*), demand is also substantial. Butter prices on the commodity markets are on the order of $US 2.00/L ($2.20/kg). Given the approximately $US0.80−1.00/L retail cost of biodiesel, it is clear that food grade butter cannot be a significant feedstock for biodiesel production. However, spoilage, excessive storage, and other situations can render butter inedible

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and available at reduced cost. It is possible that in these cases it may be considered as a biodiesel feedstock. Toward this possibility, we have produced the fatty acid methyl esters of butter and report here the results of their characterization.

MATERIALS AND METHODS

Butter. Salted butter (316 kg, 348.2 L) was commercial edible grade material (Land O'Lakes, Inc., Arden Hills, MN) that had been manually packed and sculpted along the surface of a steel skeleton, forming a "butter sculpture" that was kept at approximately 6 °C for 9 days while on public display. The butter was then peeled manually from the steel scaffold and stored an additional 7 days at 4 °C before conversion to fatty acid methyl esters.

Production of Butter Methyl Esters. The butter was heated under reduced pressure (110 °C, 635 mmHg) to melt it and remove water by vaporization (47.3 L collected). The free and acylglycerol-linked fatty acids in the resulting dry lipid preparation were converted to methyl esters by acid-catalyzed esterification/transesterification in the presence of methanol at elevated temperature and pressure (*5*) and separated from nonlipid materials, yielding 287.7 L of FAME.

Analytical Methods. The acylglycerol content of the butter starting material was determined by high-performance liquid chromatography (HPLC) on a Lichrosorb Si60 silica column (Varian, Walnut Creek, CA). Peaks were eluted with gradients of isopropanol and water in hexane/0.6 vol % acetic acid, detected by evaporative light scattering, and quantitated by reference of peak areas to standard curves constructed with pure samples of tri-, di-, and monoacylglycerols, free fatty acids, and fatty acid methyl esters (*6*). The pure materials for use as standards were obtained from Sigma Chemical Co. (St. Louis, MO). The composition of butterfat FAME was determined by HPLC analysis on a Lichrosorb Diol column (Varian), which was developed isocratically with a mixture of hexane, isopropanol, and acetic acid (98.9:1.0:0.1 vol %) at a flow rate of 0.5 mL/min. Peaks were detected by evaporative light scattering and quantitated by reference to standard curves constructed with known pure materials as employed in the HPLC of the butter starting material, described above. These methods are group specific, allowing the detection and quantitation of, for example, tri-, di-, and monoacylglycerols, free fatty acids, and FAME without speciating them according to fatty acid component.

For analytical purposes, the acylglycerol fatty acids in the butter feedstock were converted to FAME by transesterification with sodium methylate (0.15 N in methanol, 50 °C, 1 h), isolated by solvent extraction, dried over sodium sulfate, and identified by gas chromatography (GC) on a Supelco SP2340 column, 30 m × 0.25 mm i.d., 0.2 μm film thickness (Sigma-Aldrich, St. Louis, MO) with flame ionization detection. The thermal profile was 10 min at 130 °C, linear ramp at 3 °C/min to 200 °C, followed by a hold at that temperature for 10 min. Fatty acid assignments were made by reference to the retention times of commercial FAME standards (NuCheck Prep, Elysian, MN). The fatty acid species in the FAME preparation produced from the butter were also determined and quantitated by this method.

The butterfat FAME preparation was analyzed according to ASTM D6751-08, the accepted U.S. Standard Specification for biodiesel (*7*). The oxidation stability component of ASTM D6751-08 was generously conducted by Dr. Bryan Moser (National Center for Agricultural Utilization Research, Peoria, IL). All other tests of D6751 were conducted by Midwest Laboratories, Inc. (Omaha, NE).

The following additional assays were also conducted on the butterfat FAME preparation:

ASTM D6751-08 specifies the determination of glycerin by GC according to the official method for glycerin in biodiesel, ASTM D6584-00 (*8*). To discriminate between free glycerin and other components in the sample, such as the methyl esters of C8:0 and C10:0 fatty acids, that exhibited GC retention times very similar to that of glycerin when subjected to ASTM D6584-00, the butterfat FAME preparation was also analyzed qualitatively at high resolution by a proprietary industrial GC method derived from D6584-00 (Archer Daniels Midland Co., unpublished data).

The glycerin content was also additionally determined by means of a glycerin-specific enzymatic method involving glycerin kinase, pyruvate kinase, and L-lactate dehydrogenase (catalog no. 10 148 270 035, R-Biopharm AG, Darmstadt, Germany).

Table 1. Fatty Acid Compositions of Butter and FAME Prepared from Butter (Determined by Gas Chromatography, Expressed as Mass Percent)

fatty acid (carbons: unsaturations)	butter (FAME feedstock)	FAME from butter	typical butter[a] (*Bos taurus*)
4:0	not determined	not determined	3.57
6:0	6.91	2.59	2.22
8:0	3.34	4.13	1.17
10:0	5.37	9.75	2.54
12:0	4.63	8.50	2.81
14:0	11.5	15.5	10.1
14:1	1.6	1.86	1.60
16:0	27.5	25.2	25.0
16:1	1.57	1.46	2.60
18.0	11.6	7.18	12.1
18:1	21.5	18.74	27.1
18:2	3.35	2.48	2.40
18:3	not detected	not detected	2.10

[a] From ref *16*.

Methanol was determined by headspace GC using a Restek REX-624 column (60 m × 0.25 mm i.d., 1.4 μm film thickness, Restek Corp., Bellefonte, PA), with flame ionization detection.

The commercial antioxidant product Bioextend 30 (Eastman Chemical Co., Kingsport, TN), which contains 2-*tert*-butylhydroquinone and a metal chelating agent, was employed to test the ability of added antioxidant to increase the oxidation stability of the butterfat FAME.

RESULTS AND DISCUSSION

Analysis by HPLC indicated that the lipid portion of the butter starting material consisted of 93.9 ± 0.6 mass % triacylglycerols, 3.0 ± 0.2 mass % diacylglycerols, and 3.1 ± 0.5 mass % free fatty acids and contained no detectable (i.e., < 0.8 mass %) FAME (averages of duplicate determinations).

Combined esterification/transesterification of the fatty acids and acylglycerols in 348.2 L (317.2 kg) of butter resulted in the recovery of 287.7 L (247.4 kg) of a clear, yellow material that was liquid at room temperature, was shown by HPLC to be essentially 100 mass % FAME, and had a density at room temperature of 0.86 g/mL. Given the detection sensitivities of the HPLC analytical system employed, the aggregate content of free fatty acids and tri-, di-, and monoacylglycerols was no more than 1.9 mass %. The density value is comparable to that of commercial soybean oil-based biodiesel. Given a typical lipid content of 80.0 wt % for commercial salted butter (*9*), a maximum theoretical FAME yield of 248.6 L (247.4 kg) from the input butter can be calculated. Thus, the combined efficiency of transesterification and recovery of the acylglycerols in the butter starting material was > 99%.

The fatty acid compositions of the butter used as feedstock and of the FAME prepared from it, determined by GC, are listed in **Table 1**. Also shown there is the typical fatty acid composition of butter. There is a generally acceptable degree of correspondence between the fatty acid content of the FAME preparation and that of its parent butter for fatty acids above 12 carbons in length, which constitute > 80% of the fatty acids in butter. However, there are discrepancies between the butter feedstock and its product FAME below this size and notable differences in the smaller chain length region between these materials and the values published for typical butter. The significance of differences in the short- and mid-chain fatty acid contents of the butter and butterfat FAME samples studied here and a "typical" butter (**Table 1**) is difficult to evaluate, because these components are present at low levels. Also, compositional differences between the butter used here and generic data for butter may originate in seasonal or regional effects that render any specific butter different in composition from the average of many samples. In addition, the

Table 2. Results of the Analysis of Butter Fatty Acid Methyl Ester Preparation According to ASTM D 6751-08, the Accepted Standard for S15 Grade Biodiesel in the United States[a]

parameter (ASTM or EN method)	specified limit (ASTM D6751-08)	butterfat FAME
free glycerin (D 6584)	0.02% mass (max)	3.493% mass
total glycerin (D 6584)	0.24 mass % (max)	3.506% mass
flash point (D 93)	93 °C (min)	91 °C
alcohol control (meet one)		
1. methanol content (EN 14110)	0.2% vol (max)	0.024%
2. flash point (D 93)	130 °C (min)	see above
water and sediment (D 2709)	0.05 vol % (max)	<0.01%
kinematic viscosity at 40 °C (D 445)	1.9−6.0 mm^2/s	3.097 mm^2/s
sulfated ash (D 874)	0.020% mass (max)	0.000 mass %
total sulfur (D 5453)	15 ppm (max)	19 ppm
copper corrosion (D 130)	no. 3 (max)	1a
cetane number (D 613)	47 (min)	61.8
cloud point (D 2500)	report	5.0 °C
carbon residue (D4530)	0.050% mass (max)	<0.010%
acid number (D 664)	0.5 mg KOH/g (max)	0.22 mg of KOH/g
phosphorus (D 4951)	0.001% mass (max)	<0.0005% mass
distillation under reduced pressure (D 1160)	90% recovered below 360 °C	99% recovered below 362 °C
sodium and potassium (EN14538)	5 ppm (max)	<5.00
calcium and magnesium (EN 14538)	5 ppm (max)	<2.00
oxidation stability (EN 14112)	3 h (min)	0.94 h
cold soak filtration (D7501-09)	360 s (max)	82 s

[a] From ref *7*.

butter was converted to methyl esters in an industrial pilot plant that had been processing other lipids. It was not possible to scrupulously clean all material from prior batches out of the system. It is likely that the butterfat FAME product became contaminated with traces of material remaining from previous reactions.

The GC method employed here to determine the fatty acid content of the FAME product was developed and optimized for determination of the relatively long-chain (≥C16) fatty acids characteristic of vegetable oils, with no intent to accurately detect and quantitate the shorter, more volatile, fatty acids also present in dairy lipids. Accurate determination of short-chain fatty acids by GC is known to require specialized approaches (S. Bloomer, perssonal communication). Using the methodology available, it was not possible to quantitatively determine the amounts of the four-carbon fatty acid butyrate present in our butter and butterfat FAME samples, and this is so indicated in **Table 1**.

The results of analysis of the butterfat FAME as per the current standard specifications for biodiesel are shown in **Table 2**. Notably, the cetane value was >60, indicating that this fuel would have excellent ignition qualities and good cold-starting performance and give reduced smoke emissions. Cetane value generally tracks with the degree of saturation of the alkane chains in a biodiesel preparation. The cetane value for butterfat FAME is on the order of those reported for other FAME preparations with high contents of saturated fatty acyl chains, such as biodiesels from palm oil, tallow, and lard (*10*), and is consistent with the high content of saturated fatty acids in the preparation (**Table 1**).

Also notable is the 82 s cold soak filtration value of the butterfat FAME, well below the 360 s maximum threshold value (**Table 2**). A low value is desirable, as it suggests that a fuel will not generate a persistent solid fraction upon storage at ambient temperatures. This low cold soak filtration value is consistent with the origin of this sample, in that animal fats such as butter lack the sterol glucosides that are substantial contributors to high values (*11*).

Typically, the cloud point of a biodiesel, the temperature at which visual turbidity appears as a sample is cooled, increases with increasing content of saturated fatty acid esters. Cloud point can be a predictor of low-temperature performance of a fuel, with high cloud points associated with filter plugging and poor engine operability. The 5 °C cloud point of butterfat FAME (**Table 2**) is notably lower than the 13−16 °C values for some other saturated methyl esters, such as from tallow and lard (*12*). This could be due to the shorter average chain length of the fatty acids in butter relative to those feedstocks (*13*) and suggests that butterfat FAME would have better low-temperature performance properties than biodiesels produced from other substantially saturated fats.

However, the preparation was outside allowed limits for flash point, free and total glycerin, and sulfur. The flash point specification in the ASTM Biodiesel Standard Specification was designed to signal the presence of potentially hazardous levels of unreacted methanol in a fatty acid ester preparation. However, other volatile organic materials can have low flash points and could contribute to the value determined here. For example, the flash point of the methyl ester of the 10-carbon fatty acid *n*-capric acid, which comprised nearly 10% of the butterfat FAME (**Table 1**), is 90 °C (*14*). Esters of even shorter acyl chain length (e.g., four, six, and eight carbons) are found in the butterfat FAME sample and have lower flash points.

As an alternative to a minimum flash point of 130 °C, ASTM D6751-08, the Standard Specificaton for Biodiesel, lists a demonstrated methanol content of no more than 0.2 vol % as acceptable (*7*). The butterfat FAME sample was analyzed by a headspace GC procedure designed to detect and quantitate methanol. A content of 2.65×10^{-5} vol %, several thousandfold below the maximum methanol level allowed by the Specification, was determined. Thus, the butterfat FAME sample met the specifications of ASTM D6751-08 in terms of methanol content. Its flash point was nonetheless 2 °C below the minimum acceptable value. Presumably this is due to the volatility of short-chain esters in the preparation. Whether this would cause a hazard in fuel-use applications of this preparation remains to be determined.

Analysis by the method specified by ASTM D6751-08 also indicated that the butterfat FAME sample contained free glycerin levels nearly 15 times above the acceptable maximum for biodiesel (**Table 2**). Glycerin detection by ASTM D6751 is conducted by GC (ASTM D6584). Materials exhibiting retention times within

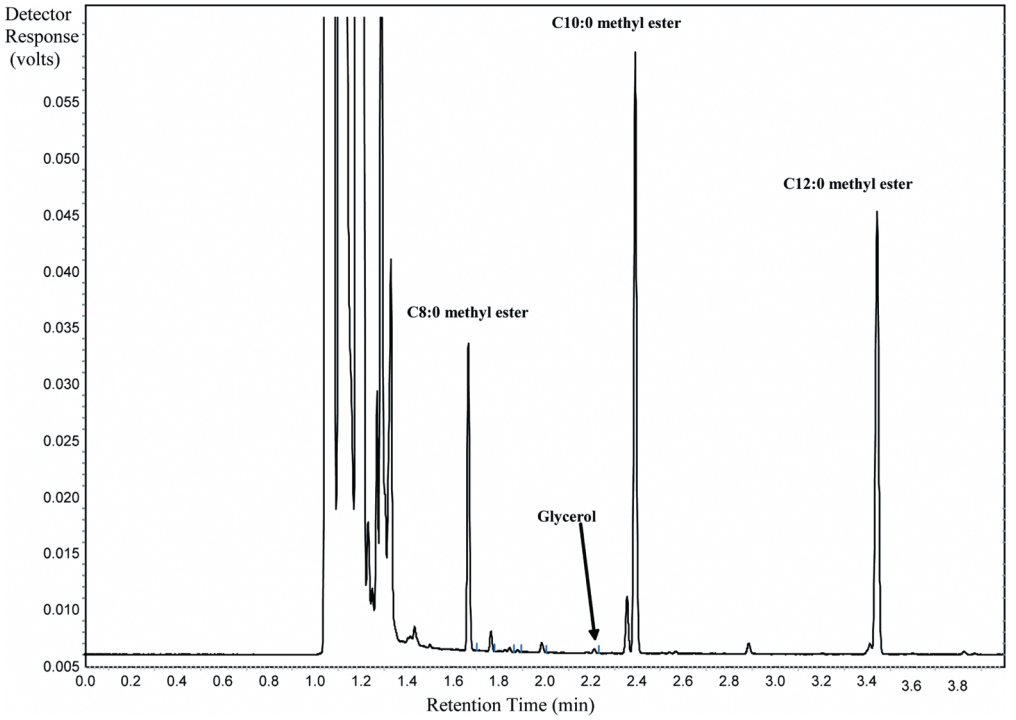

Figure 1. Short retention time region of a gas chromatogram of butterfat FAME collected under conditions optimized for the separation and detection of both mid- and long-chain-length fatty acid methyl esters. Peak identities were assigned on the basis of the retention times of known compounds.

an assigned range of values are designated glycerin in this method. The butterfat FAME showed a prominent peak in this retention time region when analyzed by a commercial testing laboratory using the ASTM D6584 method.

However, the Scope section of ASTM D6584 states that the procedure is not applicable to vegetable oil methyl esters obtained from lauric oils, such as coconut and palm kernel. Such feedstocks contain significant amounts of short- and mid-chain-length (C6−14) fatty acids in their acylglycerols. Butter also contains significant amounts of short- and mid-chain-length fatty acids (**Table 1**). Thus, it is inappropriate to attempt to determine the glycerin content of a FAME preparation derived from butter according to ASTM D6584. We therefore applied a selective enzymatic method known to accurately and specifically detect glycerin. This assay, routinely applied for the determination of glycerin in analytical and clinical settings, involves the one-pot (a) phosphorylation of glycerin by glycerin kinase in the presence of adenosine triphosphate (ATP), generating adenosine diphosphate (ADP) and glycerin-3-phosphate; (b) regeneration of ATP from the ADP by pyruvate kinase, coordinately converting phosphoenolpyruvate to pyruvate; and (c) reduction of the pyruvate to lactate by lactate dehydrogenase at the expense of reduced nicotinamide-adneine dinucleotide (NADH), which is converted to NAD. The amount of NADH oxidized, determined by following absorption at 340 nm, is stoichiometric to the amount of glycerin present. By this method it was determined that the free glycerin content of the butterfat FAME preparation was actually 0.016 mass %, which is within the limit set by ASTM D6751.

In addition, a high-resolution GC method able to separate glycerin from compounds of similar chromatographic mobility was applied to the butter-based FAME sample. The resulting chromatogram contained a peak with a retention time equal to that of a glycerin standard as well as a peak corresponding to a methyl caprate (C10:0) standard. The two peaks eluted less than 0.2 min from one another (**Figure 1**). Due to the proximity of these peaks in the region typically attributed to glycerin in the routine analysis of biodiesel, the area under the methyl caprate peak was most likely attributed to glycerin during initial analysis of the butter FAME, leading to an erroneous conclusion of excessive free glycerin levels in the butterfat FAME. Indeed, the GC trace generated upon initial testing of the butterfat FAME at a commercial testing laboratory showed a prominent peak within the region assigned as characteristic of glycerin (data not shown).

The total glycerin value of the butterfat FAME sample, which is the summation of the content of free glycerin and of that bound in acylglycerols present in the sample, was 3.506 mass %, substantially exceeding the maximum allowed value of 0.24 (**Table 2**). However, the erroneously high value determined for the free glycerin content (3.493 mass %), discussed above, contributes substantially to error in the total glycerin estimate. The bound glycerin content (i.e., that in acylglcycerols) was determined to be 0.049 mass % by the accepted ASTM assay. Confirmatory analysis in our laboratory by a high-performance liquid chromatographic method gave results consistent with this value (data not shown). The combination of this value with the actual free glycerin value, determined enzymatically (above), indicates a total glycerin content of 0.065 mass %, approximately one-fourth of the maximum allowed by ASTM D6751. Clearly, alternate official methods are needed to appraise the quality of ester preparations containing short-chain FAME.

The butterfat FAME sample also was out of specification with regard to total sulfur content, with a level 27% above the allowed maximum for the S15 grade of biodiesel that will be used as a liquid transportation fuel (**Table 2**). This is consistent with previous descriptions of the presence of methanethiol and dimethyl disulfide in butters (*15*). It is possible that these could be removed by water washing, adsorption, or vacuum treatment.

The oxidation stability index (OSI) of the butterfat FAME, determined in accordance with EN 14112, the recommended method for measuring this value in biodiesel, was 0.94 h. This does not meet the acceptable minimum value of 3 h (**Table 2**).

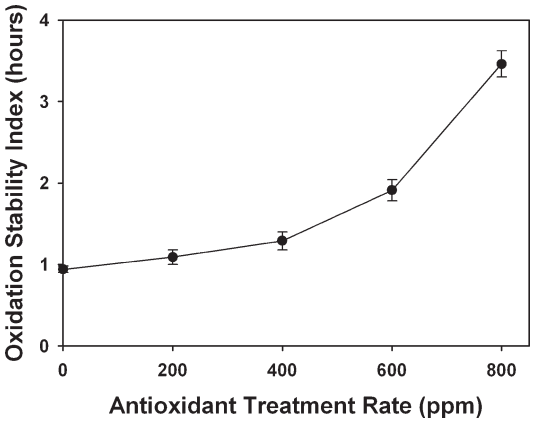

Figure 2. Effect of antioxidant (Bioextend 30) addition on the oxidation stability index of fatty acid methyl esters produced from butter, determined according to method EN14112: fat and oil derivatives − fatty acid methyl esters (FAME) − determination of oxidation stability (accelerated oxidation test; European Committee for Standardization, Brussels, Belgium, 2003). Data are the averages of triplicate determinations.

To explore the effect of antioxidant addition on the OSI value of the preparation, the assay was repeated on samples to which various amounts of the commercial antioxidant Bioextend 30 had been added. The results indicate that acceptable stability was conferred by a treatment rate of approximately 725 ppm (**Figure 2**). Other antioxidants would be expected to also be able to confer sufficient stability.

This study addresses the feasibility of producing quality biodiesel from butter and demonstrates the need for caution in assessing the quality of potential biodiesels via ASTM D6751, particularly in the case of samples containing the esters of fatty acids 10 or fewer carbons in length. Among such lipids are palm kernel and coconut oils, abundant lipids that are being assessed globally as biodiesel feedstocks.

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