J Am Oil Chem Soc (2008) 85:77–82
DOI 10.1007/s11746-007-1159-9

ORIGINAL PAPER

Melting Points and Viscosities of Fatty Acid Esters that are Potential Targets for Engineered Oilseed

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Received: 9 July 2007 / Revised: 28 September 2007 / Accepted: 12 October 2007 / Published online: 14 November 2007

Abstract Our previous isolation of branched-chain fatty acid (BCFA) methyl esters from lanolin was improved and scaled up. Also, oleate esters of isopropanol, oleyl alcohol and normal alcohols of 1–12 carbons chain lengths were prepared. Esters were made by interesterification with sodium alcoholates and by esterification with *Candida antarctica* lipase. It proved easier to obtain pure esters by the enzymatic synthesis. Melting points and viscosities over the range of 0–70 °C were determined in order to better identify potential lubricant targets that might be produced by genetically modified oilseed crops. Isopropyl and butyl oleate have melting points of −33 and −32 °C, respectively and viscosities that range from ~17 cp (0 °C) to ~2.5 cp (70 °C). They should have suitable stability for lubricants. BCFA esters had viscosities similar to their straight chain analogs. Viscosities increased with alcohol chain length and decreased with temperature. The dependence of viscosity on temperature was fit with an equation based on Erying's rate equation. Some esters with branched acid or branched alcohol moieties, and some oleate esters might be utilized as biolubricants or biofuels on the basis of their melting points and viscosities.

Keywords Branched chain fatty esters · Melting point · Oleate esters · Viscosity

Introduction

Lipids used as lubricants should have suitable viscosities, low melting points and good stability to oxidation. For maximum economic advantage they should require only extraction and limited refining to be ready for use. Low melting-lipids with good oxidative stability can be achieved by using esters of monounsaturated fatty acids. An alternate solution is to use branched-chain fatty acids (BCFAs) and alcohols. By expressing surface coat genes that control wax synthesis in oil seed tissue the biosynthesis of such esters might be achieved. Physical property data for such esters is limited, which prevents rational target selection. Previously, we reported the melting points of methyl and isopropyl esters of BCFAs with chain lengths of 13–18 [1]. Their attractive melting points encouraged us to measure the viscosity of these molecules, so we scaled up and refined our previous methods to produce larger quantities of some branched esters from lanolin. Methyl oleate melts at −20 °C and has fairly good oxidative stability [2], which suggests that oleate esters might be suitable candidates for biolubricants.

In this study, we studied the effect on melting points and viscosities of oleate esters with alcohols of various chain lengths and branching.

Experimental procedures

Materials

Lanolin acids were purchased from Rita Corp. (Crystal Lake, IL, USA) with the brand name-RITALAFA. Silica gel (40–140 Mesh) was from J.T.Baker (Phillipsburg, NJ, USA). Lipase acrylic resin from *Candida antarctica* (EC

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3.1.1.3), methyl oleate, oleic acid and alcohols of various chain lengths, methyl pentadecanoate and methyl palmitate were purchased from Sigma-Aldrich (St Louis, MO, USA). Aminopropyl extract-clean cartridges and silica gel G Uniplates (250 μm) with inorganic binder for thin layer chromatography (TLC) were purchased from Alltech (State college, PA, USA).

Bench-top Scale-up Procedure for Isolation and Purification of BCFAs

Methyl esters of lanolin free fatty acids (FFA) were made by Downing's procedure [3]. Lanolin FFA was mixed with benzene, methanol and concentrated sulfuric acid in a ratio of 5:50:50:1 (g:ml:ml:ml), and refluxed for 4–5 h. The benzene solution was extracted once with 2% sodium bicarbonate solution and twice with water. The intermediate emulsion phase was separated by centrifugation, and the benzene solution was filtered through a Buchner funnel containing sodium sulfate. The benzene was evaporated, and residual benzene was removed with a stream of nitrogen gas. The crude FAME was then distilled at 0.5 Torr pressure in a simple insulated still, and the distillate was collected up to a vapor temperature of 160 °C using an air condenser to cool the distillate. The distillate was further fractionated by chain length in a spinning band column as described previously [1].

Urea counter-current distribution of methyl esters was used to separate branched, normal and hydroxy compounds with similar boiling points according to their ease of forming urea complexes [1]. Since ~3 g urea is required to complex 1-g long-chain FAME, we added 6 g FAME obtained from a spinning band distillation fraction, to a 500-ml filter flask (designated flask #1) and then added 12–15 g urea and 200 ml of methanol 50%-saturated with urea. Then the mixture in flask #1 was heated to dissolve the FAME and urea and slowly cooled it to room temperature for crystallization. A series of 500-ml filter flasks, each containing 7 g urea, were prepared. A filter stick was used to transfer the liquid from flask #1 to flask #2. Another portion of 200 ml 50%-saturated urea–methanol solution was added to flask #1. The mixtures in flask #1 and #2 were both heated to dissolve and then cooled to crystallize. This process was repeated until 15–18 flasks were filled with the complex. The FAME was released by heating the mixture at 60 °C, and then extracted with water and hexane.

Chemical Synthesis and Purification of Oleate Esters

Methyl oleate (99% pure) was transesterified with an excess of the desired alcohol (1:200 molar ratio) for about 1.5 h at room temperature. For alcohols with a chain length longer than 10, the reactants were heated enough to keep them in a liquid state. The alkali catalyst was generated by reacting sodium metal with the various alcohols before the methyl oleate was added. The alcohols were freed of carbon dioxide before adding the sodium by sparging with nitrogen or with reduced pressure. Catalyst was used at ~0.19 mequiv/g of FAME [4]. After the reaction, adequate acetic acid was added to neutralize the catalyst. The esters were dissolved in hexane and washed with 2% aqueous sodium bicarbonate solution until the acetic acid was removed, usually three times. Residual alcohols that had a carbon chain length less than 5 could be removed by evaporation under dry nitrogen gas or with a rotary evaporator. Longer chain alcohols were mostly removed by distillation in a water bath at ~100 °C and at ~0.1 Torr. Residual alcohol, usually 1–2 ml, was removed by passage through a silica gel (1 g silica/0.1 g reaction mixture) column with hexane–diethyl ester (90:10 v/v).

Enzymatic Synthesis and Purification of Oleate Esters

When doing transesterifications with a large excess of the alcohol, it was not economic to remove the residual alcohol with a silica gel column. To avoid this problem and improve the purity of products, enzymatic syntheses were performed with oleic acid and alcohol at a 1:2 molar ratio in a solvent free system. *Candida antarctica* lipase was added at 2% by weight. Molecular sieve in an amount which was calculated to absorb the water generated in the reaction, was added at a ratio of 1 g molecular sieve to 1.8 g water at 3 and 24 h after the reaction started. The reaction took place at room temperature (~25 °C) with constant stirring and was stopped after 4 days with the addition of water. The product was centrifuged to remove the enzyme and molecular sieve, and then extracted with hexane and 2% sodium bicarbonate solution. Since alcohols were added in smaller excess than in the chemical syntheses, silica gel chromatography was applied to the product after extraction to remove residual alcohol and FFA. If subsequent gas chromatography (GC) or TLC detected FFA, these samples were further purified by passing through an amino cartridge with hexane, and the neutral esters were eluted with chloroform-isopropanol (2:1 v/v).

GC, TLC and NMR

The purities of oleate esters and BCFA esters were examined by GC. Esters in hexane solution were injected into a HP 5890 Series II instrument (Hewlett-Packer, PA) with a SP-2330 fused silica column (15 m × 0.25 mm and a 0.20 μm film thickness) (Supelco, Bellefonte, PA, USA). The injector and flame detector were at 220 °C, and the oven temperature was programmed from 100 to 220 °C at a rate of 5 °C/min.

Free fatty acid and residual alcohol also were detected by TLC on silica plates. Hexane–diethyl ether–acetic acid (80:20:1) was used as developing solvent. The separation was visualized under ultraviolet light after spraying with 0.1% (w/v) 2′, 7′-dichlorofluorescein in methanol.

The structures of the oleate esters in $CDCl_3$were confirmed by ^{13}C nuclear magnetic resonance (NMR) with a Varian VXR-400 NMR spectrometer (Varian Inc., Palo Alto, CA, USA).

Melting Points

Melting points of the oleate esters were measured with a DSC7 differential scanning calorimeter equipped with an Intracooler System I (Perkin Elmer, Norwalk, CT, USA). Indium and *n*-decane were used for calibration. The melting onset temperatures of standards were calibrated with melting points listed in the DSC7 operation manual (Perkin Elmer, Norwalk, CT, USA). Methyl stearate was used as a secondary standard, of which the onset melting temperature (T_{onset}), peak temperature (T_{p}), and completion of melting (T_{com}) are 38.9, 41.9 and 43.1 °C, respectively. About 3–4 mg of each sample was weighed to 0.01 mg in an alumina pan, and an empty pan was used as a reference. The thermal program was in accordance with our previous study and AOCS recommendations [1, 5]. After equilibration at 25 °C for 1 min, the sample was rapidly heated to 80 °C at 40 °C/min and held for 10 min for complete melting of crystal nuclei, cooled to −60 °C at 10 °C/min rate and equilibrated for 20 min. When reheated at 5 °C/min, T_{onset}, T_{p}, and T_{com} were recorded.

Viscosity

The viscosities of the various oleate esters and BCFA esters were determined with a Brookfield DV II + viscometer (Brookfield Inc., Stoughton, MA, USA) using a CP42 cone spindle. The cup was filled with 1 ± 0.03 ml of ester. Temperature was controlled with an Isotemp 3016 refrigerated circulator (Fisher Scientific, Pittsburgh, PA, USA). Temperature was monitored with a certified partial immersion Fisherbrand thermometer (Fisher Scientific, Pittsburgh, PA, USA) with a range of −1 to 101 °C calibrated in 0.1° divisions, which was inserted in a tube on the return line of the circulating water bath. Air pressure was used to keep the water level in the tube at the proper immersion level of the thermometer. Data was collected between 0 and 70 °C at 5 °C intervals. Viscosities were recorded after equilibrating at the desired temperature for 10 min and at the maximum possible torque.

Results and Discussions

Bench-top Scale-up Procedure

The yield of crude methyl ester from the commercial lanolin FFA was quantitative based on a MW of 256 for lanolin FFA and 270 for FAME. They had nearly the same FAME composition by GC as those of our previous research [1]. The preliminary distillation removed longer chain compounds as well as small amounts of sterol esters. The distillate was rich in FAMEs with chain lengths shorter than 21 C and the distillation pot residual was rich in FAMEs with chain length longer than 18 C. Since no new peaks were observed from GC of the distillate or residual, we concluded that the distillation did not bring any undesired reaction. The distillate amounted to ∼38.2% of the crude FAME by weight. From the crude distillations, 374 g was collected and fractionated through the more efficient spinning band column.

The scaled-up counter-current urea separation of the spinning band fractions yielded gram quantities of methyl 12-methyltetradecanoate (15a) and methyl 14-methylpentadecanoate (16i) with 95% purity. The urea separation also removed the hydroxy FAME and avoided the expensive alumina chromatographic purification step used in our previous study [1].

Oleate Esters Synthesis

Oleate esters of isopropanol, oleyl alcohol and normal alcohols of 1–12 carbons chain lengths were synthesized with enzyme as well as with sodium alcoholate catalyst. The yields of oleate esters by chemical transesterification averaged 85%, and some batches were as high as 94%. The purity of the esters by GC was >95%. The major impurity always was methyl oleate. Enzymatic syntheses gave yields of ∼75% but improved the product's purity to 99% since the excess oleic acid was easily removed from the nonpolar oleate esters with an amino cartridge or TLC. The ^{13}C-NMR chemical shifts of oleate esters were assigned to various carbon atoms by comparing observed values with calculated values obtained by ACD/ChemSketch Predictor (Advanced Chemistry Development Inc., Toronto, ON, Canada) and Vieville et al.'s [6] result. The ^{13}C-NMR spectra of the oleate esters all showed a carbonyl group at 174.4–175.3; the methylene adjacent to the carbonyl group at 61.5–66.6, except for the isopropyl at 68.3; the methylene β to the carbonyl at 35.1–35.8, double bond carbons at 130.3–131.3; terminal methyl groups at 11.2–15.4 except for the isopropyl ester at 22.9; a carbon α to the terminal carbon at 14.7–15.6, a carbon β to the terminal carbon at 23.2–24.1; a methylene carbon adjacent to the ester bond at 61.5–66.6 for the normal chains and 68.4 for the isopropyl ester; other methylene carbons ranged from 23.7 to 33.3. In most instances the extreme values for the straight-chain esters belong to the ethyl, propyl or butyl ester values, which is undoubtedly a reflection the terminal groups of the alcohols.

Melting Point

The esters from enzymatic synthesis were used for melting points measurements since they had higher purity than those of alkali catalyzed reaction. The melting points of oleate esters are shown in Table 1. Of all the esters examined, the lowest melting point was that of isopropyl oleate at −33.0 °C. Among even chain normal alcohols, butyl oleate melted lowest at −31.7 °C; and among the odd chains, propyl oleate was lowest at −27.2 °C. Pentyl and hexyl oleate's melting points were very close at −25.2 and −25.8 °C, respectively. The equipment manufacture's precision claim is better than ±0.1%, and temperature accuracy is ±0.1 °C; however, the AOCS has reported that the standard deviation of determination among laboratories was ±1.8 °C for the onset temperature and 0.8 °C for the completion [5]. The melting points of methyl through pentyl oleate agreed with the results of Candy et al. [7]. Plots of the melting points of the straight chain ester versus the number of carbon atoms in the alcohol moiety showed a decreasing melting point until carbon number four, and then an increasing value (Fig. 1). Literature values of corresponding esters of various saturated fatty acids as well as FFAs show similar melting patterns, but at considerably higher temperatures than the oleate esters [8, 9].

Table 1 Melting onset (T_{onset}), peak (T_p) and completion (T_{com}) temperatures of oleate esters

Alcohol moieties	T_{onset} (°C)	T_p (°C)	T_{com} (°C)
Methyl	−20.3	−17.3	−16.3
Ethyl	−23.1	−19.0	−18.1
Propyl	−32.5	−28.3	−27.2
Butyl	−35.7	−32.8	−31.7
Pentyl	−29.2	−26.3	−25.2
Hexyl	−30.3	−27.0	−25.8
Heptyl	−18.3	−14.7	−13.1
Octyl	−9.3	−4.1	−2.9
Nonyl	−9.4	−5.6	−4.4
Decyl	2.8	6.8	8.2
Dodecyl	12.9	17.3	18.4
Oleyl	−7.1	−2.9	−1.5
Isopropyl	−37.7	−34.2	−33.4

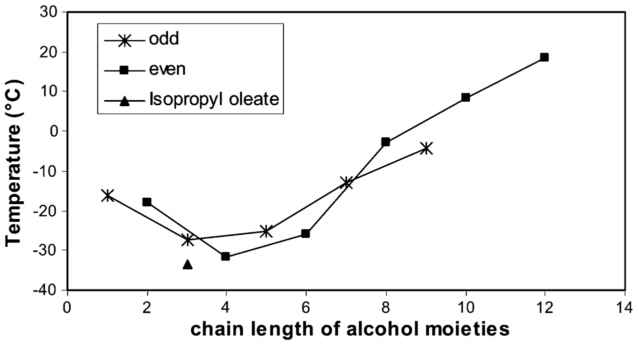

Fig. 1 Effect of chain length on the melting points (T_{com}) of oleate esters

King and Garner gave an explanation on the melting point pattern of saturated fatty acids [8, 10] based on crystal diffraction studies and heats of melting. According to this explanation, the terminal methyl and carboxyl groups in crystals of fatty acids actually are further apart than in the liquid state. Thus, when a crystal melts, these terminal groups come closer together, and their melting is exothermic. The crystal is held together by the sidewise attraction of methylene groups in the fatty acid chains, and their melting is endothermic. The methylene group attraction increases with the number of these groups per molecule. As the alcohol chain lengthens from methyl through butyl, the additional methylene groups make the end groups take more and more unfavorable positions in the crystal, making the crystal less stable and lowering their melting point. In esters with alcohol groups greater than butyl, the methylene groups have increasing attraction, and their attraction is greater than the effect of the end groups. So the crystal stability and melting point begin to increase as the alcohol chain grows longer. The same theory could apply to oleate esters.

Viscosity

The esters from enzymatic synthesis were used for viscosity measurements since they had higher purity than those of the alkali catalyzed reaction. The viscosities of oleate esters with normal alcohol chain lengths of 1–10 and BCFAs, plus branched isopropyl oleate in the temperature range of 0–70 °C are given in Table 2. The melting points of the methyl esters of BCFAs were reported in a previous paper [1]. Methyl esters of 15a and 16i melt at −9 and 16 °C, respectively. Although the melting point of these branched derivatives are not particularly low, they were chosen for

Table 2 Viscosities of oleate esters, BCFA methyl esters and saturated straight chain FAME

Temperature (°C)	Viscosity (cp)														
	Methyl	Ethyl	Propyl	Butyl	Pentyl	Hexyl	Heptyl	Octyl	Nonyl	Decyl	Isop	15a	15n	16i	16n
0.00	12.30	13.60	15.70	17.20	19.20	21.80	25.00	27.45	30.60	34.60	15.40	7.97	–	–	–
5.05	10.20	11.30	13.00	14.10	15.65	17.80	20.20	22.25	24.60	27.70	12.60	6.85	–	–	–
10.00	8.64	9.60	10.95	11.80	13.00	14.70	16.60	18.20	20.10	22.50	10.50	5.90	–	9.86	–
14.95	7.39	8.17	9.25	9.98	10.95	12.30	13.90	15.20	16.70	18.60	8.89	5.11	–	8.33	–
20.00	6.37	7.05	7.96	8.55	9.32	10.40	11.70	12.80	14.30	15.55	7.63	4.48	5.05	7.06	–
25.00	5.66	6.25	6.98	7.48	8.23	9.12	10.15	11.10	12.10	13.30	6.57	4.03	4.42	5.96	–
30.00	4.97	5.48	6.08	6.52	7.15	7.88	8.77	9.53	10.40	11.40	5.67	3.61	3.91	4.94	4.48
35.00	4.39	4.83	5.36	5.75	6.25	6.89	7.63	8.30	9.03	9.91	4.98	3.22	3.49	4.31	3.94
40.00	3.91	4.30	4.75	5.08	5.50	6.05	6.67	7.27	7.89	8.62	4.40	2.91	3.11	3.79	3.53
45.00	3.50	3.84	4.24	4.54	4.88	5.36	5.88	6.39	6.95	7.54	3.89	2.65	2.8	3.36	3.14
49.95	3.17	3.46	3.80	4.10	4.39	4.78	5.23	5.67	6.15	6.64	3.50	2.42	2.54	3.03	286
54.95	2.87	3.14	3.43	3.69	3.92	4.26	4.68	5.06	5.51	5.91	3.15	2.22	2.32	2.75	2.6
59.80	2.62	2.85	3.12	3.34	3.56	3.84	4.18	4.55	4.91	5.28	2.86	2.05	2.13	2.49	2.38
64.80	2.40	2.62	2.84	3.04	3.22	3.49	3.79	4.11	4.44	4.76	2.59	1.89	1.96	2.29	2.19
69.70	2.21	2.42	2.62	2.78	2.95	3.19	3.46	3.71	4.00	4.29	2.38	1.76	1.81	2.11	2.02

15a, 12-methyltetradecanoic acid methyl ester; 15n, pentadecanoic acid methyl ester; 16i, 14-methylpentadecanoic acid methyl ester; 16n, hexadecanoic acid methyl ester; isop, isopropyl oleate; the viscosities of 15n, 16i, and 16n were measured above their individual melting points

comparison with the oleate esters. The viscosities of dodecyl oleate and oleyl oleate were not measured because of limited amounts of materials. Viscosity increased with the chain length of the esterified alcohol and with decreasing temperature. The viscosity of the branched isopropyl oleate was close to its straight chain counterpart, propyl oleate. The branch on the alcohol moieties does not affect viscosity very much. This agrees with Knothes's study [11]. Viscosities of methyl esters of 15a and 16i had viscosities close to those of their straight chain counterparts as well as that of isopropyl oleate. Such branches on the acid moieties of these esters did not greatly affect the viscosity.

The oleate esters and BCFAs methyl esters all behave like Newtonian fluids. At 25 °C, the viscosities remain constant with an increasing shear rate from 20 to 200 rpm.

Numerous equations have been proposed to fit the change in viscosity with temperature [12]. Many of these equations are based on the idea that energy of activation has to be added to the molecules of a liquid for them to flow past each other. Such equations are analogous to the Arrhenius equation for the rate of a chemical reaction and take the form:

$$\ln \eta = \ln A - \frac{E_a}{RT} \tag{1}$$

where η is the viscosity and A is a constant, E_a is the energy of activation, T is the temperature in degrees Kelvin and R is the gas constant. Ln η calculated by versions of this equation gives a straight line for ln η versus $1/T$ that poorly fit the experimental data, which gives a very slightly curved line for ln η versus $1/T$. We considered that the fit for long linear molecules such as oleate esters might be improved by considering the spatial orientation of the activated molecules as in Erying's rate equation [13]. This equation is often simplified by assuming changes in entropy for the activated state are negligible. Then

$$\ln \eta = \ln\left(\frac{R}{Nh}\right) + \ln T - \frac{E_a}{RT} \tag{2}$$

where N is Avogadro's number, and h is Planck's constant.

The fit of the logarithm of the viscosity data was improved by the inclusion of the ln T term, but the fit was still imperfect. By using our viscosity data and appropriate values of R, N and h, we were able to calculate E_a at the various temperatures for our esters. Such a treatment revealed that E_a was not a constant but was almost a linear function of T. Further study showed that the fit of the data by this linear equation, like that of Eq. [1] was poorer at the extreme temperatures and that the fit could be improved from $R^2 = 0.9999$ to 1 by using a polynomial, such that

$$E_a = B + CT + DT^2 \tag{3}$$

where B, C and D are constants. Thus,

$$\ln \eta = \ln\left(\frac{R}{Nh}\right) + \ln T - \left[\frac{B + CT + DT^2}{RT}\right] \tag{4}$$

It may be that for long linear molecules such as these esters, E_a is a function of T because as temperature increases, the

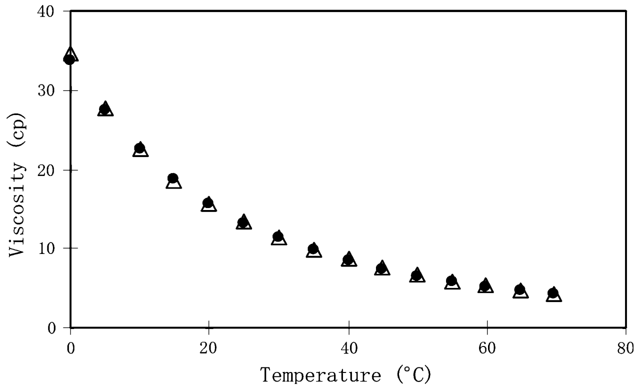

Fig. 2 Fit of observed viscosity data of decyl oleate at various temperatures with calculated data of Eqs. 2 and 5. *Triangle* observed data, *filled circle* calculated data

probability of the molecule departing from linearity increases. Departure from linearity entails a corresponding increase in the energy needed to move one molecule past another. This equation's DT^2 term also succeeded in fitting the curve of the experimental ln η versus $1/T$ data.

The fit of data by Eq. 4 is not strongly dependent on the absolute value of $\ln[R/(Nh)]$, as B, C and D can change to make the equation fit when various values of this constant are used. We used 28.3651 for $\ln[R/(Nh)]$ when η was in centipoise.

Plots of B, C, and D versus n, the chain length of the alcohol portion of the esters are generally linear functions but show considerable scatter. This may be the result of varying amounts of impurities in the preparations that we could not detect by the methods we used. Alternatively this scatter may reflect differences in the favored configuration of the various esters possibly reflecting the interplay of methylene and end groups noted by King and Garner [8, 9] in melting points. However, the errors caused by scatter in the B, C, and D plots are relatively insignificant. If one fits these plots with the best straight line for the three plots one gets the following equation:

$$E_a = -(833.31n + 33089) + (3.1648n + 419.28)T - (0.0039n + 0.1297)T^2 \quad (5)$$

where n is the chain length of the alcohol in the oleate esters. Using this equation to obtain E_a and substituting it in Eq. 2 allow one to calculate the viscosity of the esters with less than 1% deviation from the experimental value. The fit is worst for the 0 °C values but even these values differ from the experimental values by an average of 2.2% and do not exceed 4%. The viscometer has an error of ±1% under the conditions used for our measurements.

An example of the fit of decyl oleate data is given in Fig. 2. Thus, these equations allow prediction of the viscosity at other temperatures and longer chain lengths. This equation was developed for the oleate esters of straight-chain alcohols, but fit equally well for isopropyl ester, and the methyl esters of BCFAs. The equations for isopropyl oleate, methyl esters of 16i and 15a, respectively, are:

$$E_a = -38561 + 445.57T - 0.1632T^2 \quad (6)$$

$$E_a = -53717 + 541.21T - 0.3103T^2 \quad (7)$$

$$E_a = -30266 + 408.02T - 0.117T^2 \quad (8)$$

Acknowledgments We acknowledge James Cornette for advice about the viscosity equation, and to the Center for Crops Utilization Research and the Plant Science Institute at Iowa State University for financial support.

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