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ORIGINAL PAPER

Formation of Insolubles in Palm Oil-, Yellow Grease-, and Soybean Oil-Based Biodiesel Blends After Cold Soaking at 4 °C

Haiying Tang · Rhet C. De Guzman · Steven O. Salley · K. Y. Simon Ng

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Abstract The insolubles formed in biodiesel blends can cause operation problems because they can plug the fuel lines and filters. The formation of insolubles in soybean oil (SBO-), yellow grease (YG-), and palm oil (PO-) based biodiesel blends after cold soaking at 4 °C was investigated. PO-based biodiesel blends displayed a much higher time to filter (TTF) and greater insoluble mass, compared to SBO-, and YG-based biodiesel blends. Fourier transform infrared (FTIR) spectra and gas chromatography-flame ionization detector (GC-FID) chromatograms indicated that PO-based biodiesel insolubles can be attributed to monoglycerides, while SBO-based biodiesel insolubles are due to steryl glucosides (SG). A simple analytical method for identification of SG in biodiesel samples was established by GC-FID.

Keywords Biodiesel · Biobased products

Introduction

Biodiesel is an alternative diesel fuel consisting of alkyl monoesters of fatty acids from vegetable oils or animal fats. Annual biodiesel production has dramatically increased in recent years [1] because of benefits associated with biodiesel, including renewability, domestic feedstocks, lower toxicity, and biodegradability. However, the performance of biodiesel under cold weather conditions is markedly worse than that of petroleum diesel because wax crystals, gels, and insolubles may form at lower temperatures. In general, wax crystals (or gels) formed at low temperatures from saturated and unsaturated fatty acid methyl esters (FAME) can be re-dissolved when warmed to room temperature [2], while insolubles formed from minor components often remain as precipitates in the fuel at room temperature [3]. Therefore, these solids may have serious implications, such as clogging of fuel filters in engine fuel delivery systems, forming deposits on injectors, and causing major operability problems.

There are many sources of vegetable oil or animal fat for biodiesel, such as, soybean oil, cottonseed oil, canola oil, rapeseed oil, palm oil, corn oil, yellow grease, brown grease, and poultry fat [4]. The most common source of oil for biodiesel in the US is soybean oil. Rapeseed and palm oil are widely used feedstocks in the European Union, Malaysia, Indonesia, Thailand, Nigeria, and Colombia. However, because of recent upward trends in soybean oil prices, less expensive yellow grease has been widely used in the US. Our previous study [3] investigated the effect of low temperature on insolubles formation for soybean oil- (SBO-), cottonseed oil- (CSO-), and poultry fat- (PF-) based biodiesel blends and found that cold soak temperature, cold soak time, biodiesel blend level, and feedstock affect the mass of insolubles formed. This study reports our findings on the impact of cold soaking at 4 °C on the formation of insolubles for yellow grease- (YG-) and palm oil- (PO-) based biodiesel, as well as the nature of the insolubles formed. The effect of SBO-based biodiesel from different suppliers on the formation of insolubles is also described.

H. Tang · R. C. De Guzman · S. O. Salley · K. Y. S. Ng (✉)
Department of Chemical Engineering and Materials Science, Wayne State University, 5050 Anthony Wayne Drive, Detroit, MI 48202, USA
e-mail: sng@wayne.edu

To understand the formation of insolubles better, reliable analytical methods to determine minor components, such as, free and bonded glycerin, sterols, and natural antioxidants, in biodiesel are necessary. Sterols include free sterols, steryl ester, steryl glucosides (SG), and esterified steryl glucosides (ESG) [5, 6]; while bonded glycerin is composed of mono-, di-, and tri-glycerides. The free and bonded glycerin in biodiesel can be determined using gas chromatography with a flame ionization detector (GC-FID) according to ASTM 6584 (GC-FID method) [7]; while Moreau et al. [8] reported that SG in precipitates can be quantified by high performance liquid chromatography (HPLC) method, but the detection limit of SG is about 100 ppm. Bondioli et al. [9] found that SG in biodiesel could be identified and quantified by GC, this indirect method requires a pretreatment step with acidic methanolysis. The methanolysis completely cleaves the glucosidic bonds of SG, thus allowing the quantification of the SG concentration based on the concentration of free sterols. Van Hoed et al. [10] also found the GC can be used to quantify the SG in biodiesel as well as in filter residues. However, up until now, no ASTM standard analytical methods to determine sterols in biodiesel have been established.

The objective of this study was to investigate fuel properties and insolubles formation of YG-, PO-, and SBO-based biodiesel (FAME) and their blends after cold soak at 4 °C. Moreover, the development of a direct analytical method to evaluate free sterols, steryl ester, SG, ESG, and mono-, di-, and tri-glycerides in biodiesel is reported.

Experimental

Materials

PO-based biodiesel was obtained from Golden Jomalina Food Industries Sdn. Bhd. (Kuala Langst, Selangor Darul Ehsan, Malaysia), YG-based biodiesel was obtained from Biodiesel Industries (Denton, TX), and SBO-based biodiesel was bought from G. E. Wacker Inc. (Manchester, MI). Certification #2 ultra low sulfur diesel (ULSD) was purchased from Haltermann Products (Channelview, TX). The blends were made on a volume basis and stored in glass bottles at room temperature.

Both SG and ESG standards were purchased from Matreya LLC (Pleasant Gap, PA). Stigmastanol (>95%), cholesteryl stearate (96%), heptane (>99% capillary GC), pyridine (>99%), *N*-Methyl-*N*-trimethylsilytrifluoroacetamide (MSTFA), and α-tocopherol (1000 IU/g) were purchased from Sigma Aldrich Inc. (St Louis, MO). 1, 2, 4 butanetriol [1 mg/mL, internal standard 1 (ISTD1)], tricaprin [8 mg/mL, internal standard 2 (ISTD2)] in pyridine, mono-, di-, and tri-glycerides were obtained from Supelco (Bellefonte, PA).

Fuel Properties

Composition

The FAME composition of each biodiesel was determined using a PerkinElmer Clarus 500 GC-mass spectrometer (GC-MS) with a split automatic injector and an Rtx-WAX (Restek, Bellefonte, PA) column (length: 60 m; ID: 0.25 mm; coating: 0.25 μm). Details of the procedure have been described elsewhere [3]. The column was held at 120 °C for 1 min and ramped to 240 °C at 20 °C/min, and it was then held at 240 °C for 13 min. The transfer line between GC and MS was kept at 240 °C.

Kinematic Viscosity, Acid Number, and Oxidative Stability

The viscosity of biodiesel at 40 °C was determined following ASTM D 445 [11] using a Rheotek AKV8000 automated kinematic viscometer (Poulten Selfe & Lee Ltd., Essex, England). The acid number of biodiesel was determined according to ASTM D 664 [12] using a Brinkmann/Metrohm 809 Titrando (Westbury, NY). The oxidative stability of biodiesel was determined as the induction period (IP) according to EN14112 [13] using a Metrohm 743 Rancimat instrument (Herisau, Switzerland).

Cold Flow Properties

The cloud point (CP), pour point (PP), and cloud filter plugging point (CFPP) measurements were done as per ASTM standards, D 2500-05 [14], D 97-96a [15], and D 6371-05 [16], respectively. A Lawler model DR-34H automated cold properties analyzer (Lawler Manufacturing Corporation, Edison, NJ) was used to measure the cold flow properties.

Cold Soak Test

The PO-based biodiesel used in this study had a white precipitate at room temperature. Before the cold soaking, the PO-based biodiesel was heated to 40 °C for 3 h under an inert atmosphere, and then maintained for 24 h at room temperature (23 °C). SBO- and YG-based biodiesel was used without any treatment.

As previously reported [3], 300 mL of fuel in 500 mL media bottles were stored in a refrigerator at 4 °C for 24 h. The bottles were then removed and allowed to return to room temperature before filtering to obtain the time to filter (TTF) and the insolubles mass.

Minor Components Analysis

To obtain insoluble-concentrated SBO-based biodiesel, fuel samples were centrifuged at 4,000 rpm (Eppendorf centrifuge 5804R, Germany) for 10 min, and then 95% of upper layer of fuel was removed. Moreover, insolubles were isolated by filtering with a 0.7-μm glass fiber filter (GF/F, Whatman) for further GC-FID and FTIR analysis.

GC-FID Analysis

A PerkinElmer Clarus 500 GC-FID was used to quantify minor components in the biodiesel. Standard solutions of stigmastanol (free sterol), SG, cholesteryl stearate (steryl ester), and ESG were prepared with pyridine (2 mg/mL). A standard 2 mg/mL solution of α-tocopherol was prepared in heptane. The concentrated insolubles from SBO-based biodiesel (~100 mg) or insolubles (~2 mg) were dissolved in 200 μL of pyridine. Then, 200 μL of standard mixture or the insolubles solution was mixed with 20 μL of ISTD2, and 100 μL of MSTFA in a vial, and allowed to sit for 30 min at room temperature. Finally, 2 mL of heptane was added to the vial. To determine the free and total glycerin concentration, the 100 μL of ISTD1 and ISTD2 were added based on ASTM D 6584. Details of the procedure have been reported [3]. A PE-5HT column was held at 50 °C for 1 min and then ramped to 180 °C at 15 °C/min, 230 °C at 7 °C/min, and 380 °C at 30 °C/min. Finally, it was held at 380 °C for 10 min. The useful range of masses that were quantifiable is from 10 to 1,000 amu. The detection limit for SG was found to be below 10 mg/kg; which agreed with a previous GC study of below 15 mg/kg [10].

FTIR Analysis

A PerkinElmer spotlight 400^{TM} spectrometer equipped with an attenuated total reflectance (ATR) sampling accessory (with diamond/ZnSe crystal) was employed to obtain Fourier-transform infrared spectroscopy (FTIR) spectra in the 4,000–650 cm^{-1} range. All spectra were obtained at 23 °C using an average of 4 scans, with a spectral resolution of 4° cm^{-1}, and air as the reference spectrum.

Results and Discussion

Characterization of YG-, PO-, and SBO-Based Biodiesel

Table 1 shows FAME compositions for the biodiesel produced from different feedstocks. The levels of methyl palmitate (C16:0, 47.2%) and methyl oleate (C18:1, 40.8%) in PO-based biodiesel are much higher than that in YG- and SBO-based biodiesel, while methyl linoleate (C18:2) is the predominant FAME in YG- and SBO-based biodiesel (more than 46%).

Table 1 Fatty acid methyl esters (FAME) compositions of PO-, YG-, and SBO-based biodiesel

Fatty acid	PO	YG	SBO
C14:0	0.6	0.1	0
C16:0	47.2	16.1	11
C16:1	0	0	0
C18:0	3	4	4.2
C18:1	40.8	31.4	22.6
C18:2	8.2	46.1	55
C18:3	0.2	2.3	7.2
∑SFA (%)	50.8	20.2	15.2
∑UFA (%)	49.2	79.8	84.8
Oxidizability (OX) (%)	9.4	51.3	69.9

OX = [0.02(% C18:1) + (% C18:2) + 2 (% C18:3)]/100 (according to [10])

The viscosity, acid number, IP, free and total glycerin of PO-, and SBO-based biodiesel met ASTM D6751-07b specifications (Table 2), however, the acid number (0.52 mg KOH/g) and IP (2.3 h) of YG-based biodiesel did not meet ASTM D6751-07b, suggesting that the YG-based biodiesel had partially oxidized. The oxidative stability of biodiesel in general depends on both FAME compositions and antioxidant concentration. Biodiesel is more susceptible to oxidation and results in shorter induction times with a high level of unsaturated FAME, especially methyl linolenate (C18:3) [17]. The coefficients of unsaturated FAME, which is proportional to the relative rates of oxidation, were reported to be 0.02 for methyl oleate (C18:1), 1 for C18:2 and 2 for C18:3 in the oxidizability (OX) of ester [18]. In this study, the PO-based biodiesel has much less C18:2 and C18:3 than YG- and SBO-based biodiesel and the IP is the highest. Although SBO-based biodiesel has a higher OX than YG-based biodiesel, the IP of SBO- is higher. This apparent contradiction can be attributed to the effect of antioxidant on oxidative stability of biodiesel: SBO-based biodiesel contains natural (~67 ppm), while no antioxidant was detected in YG-based biodiesel. It should be note that SBO-based biodiesel had a low tocopherol content (69 ppm), but it has a high IP, suggesting the presence of a synthetic antioxidant.

Cold Flow Properties

The CP, PP, and CFPP of PO-, YG-, and SBO-based biodiesel as a function of biodiesel concentration are shown in Fig. 1a–c, respectively. In general, the CP, PP, and CFPP

Table 2 Physical properties of PO-, YG-, and SBO-based biodiesel

	ASTM method	ASTM specification	PO	YG	SBO
Viscosity, 40 °C (mm^2/s)	D 445	1.9–6.0	4.52	4.55	4.06
Acid number (mg KOH/g)	D 664	0.5	0.25	0.52	0.21
Free glycerin (mass %)	D 6584	0.020	0.006	0	0
Total glycerin (mass %)	D 6584	0.24	0.22	0.184	0.078
Oxidative stability (IP, h)	EN 14112	3 minimum	11.1	2.3	7.2

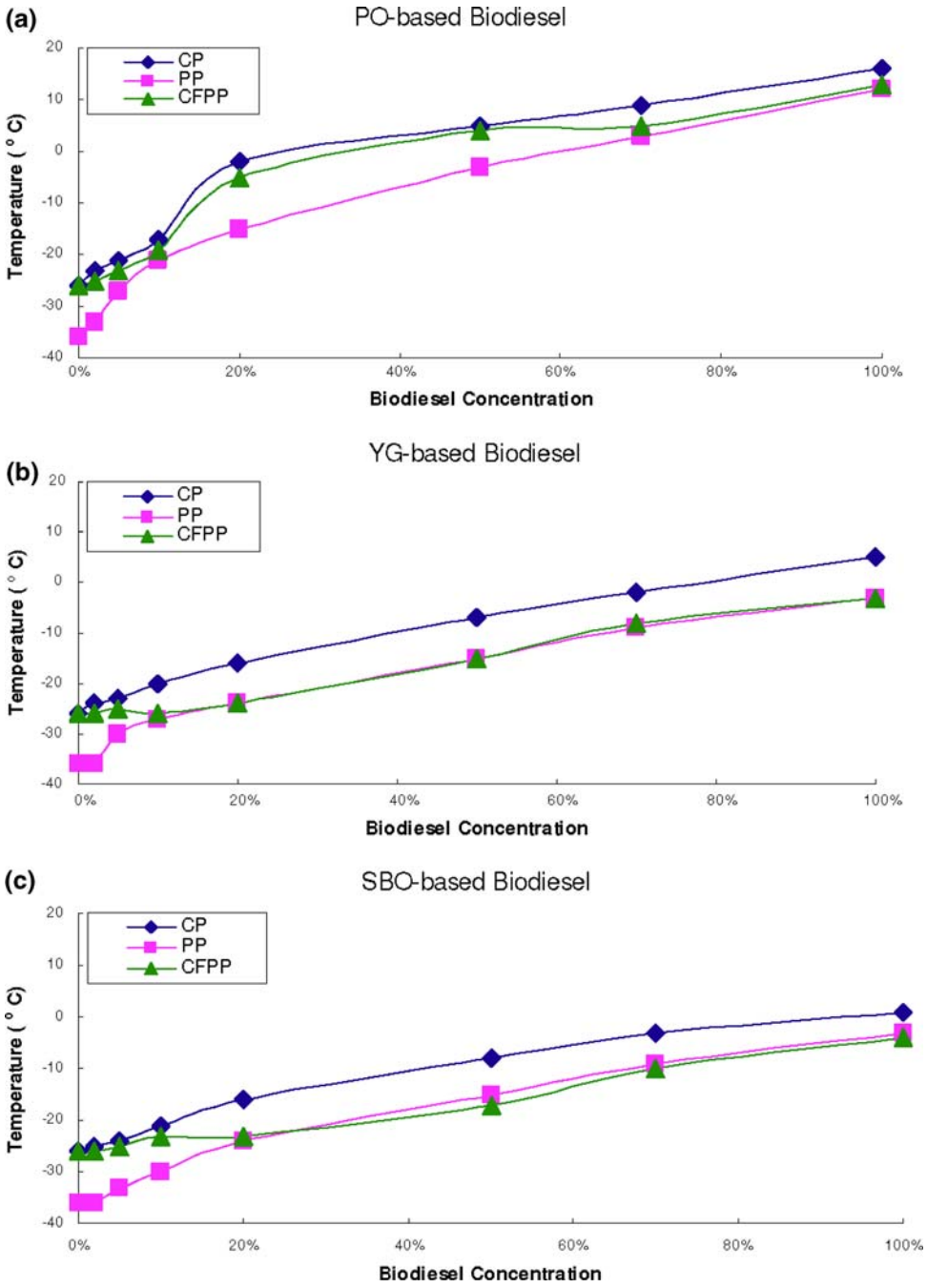

Fig. 1 The cloud point, pour point, and cold filter plugging point of **a** PO-, **b** YG-, and **c** SBO-based biodiesel and their blends with ULSD fuel as a function of biodiesel concentration (volume percent)

increased with biodiesel concentration. The CP of PO-, YG- and SBO-B100 is 15, 5, and 2 °C; while the PP is 12, −3, and −3, respectively. The more saturated FAME for PO-based biodiesel, the higher the CP and PP, as compared with both YG- and SBO-based biodiesel (Table 1). This is consistent with Knothe's finding that significant saturated

fatty compounds in biodiesel display higher CP and PP [19]. Interestingly, the CFPP is almost the same as the CP for PO-B20 and PO-B50, while the CFPP is close to the PP for YG- and SBO- based samples. This can be attributed to the higher insoluble formation for PO-B20 and PO-B50 than the other types of biodiesel at lower temperature. This finding is consistent with our previous study on SBO-, CSO-, and PF-based biodiesel that CFPP can indicate the relative extent of the insoluble formation [3].

Cold Soak Test

The PO-B100 used in this study contained insolubles in the form of white round particles even at room temperature, while no insolubles in YG- and SBO-B100 were visually observed. After cold soaking at 4 °C for 24 h, white wax crystal-like insolubles were observed in B10, B20, B50, B70, and B100 of PO-based biodiesel. These white wax crystal-like insolubles are quite different from the irregularly shaped agglomerates embedded with cloud-like materials formed in SBO-based biodiesel blends in our earlier study [3]. In contrast to the results from our previous study, no insolubles were visually observed for SBO-based biodiesel blends after cold soaking. The source of SBO-based biodiesel was different in this study, suggesting different pretreatment and processing conditions can have a great effect on insolubles formation. Interestingly, no insolubles were observed visually for YG-based biodiesel blends.

Time to Filter (TTF)

The TTF is a measure of the fuel filter blocking potential. Figure 2 shows the TTF of PO-, YG-, SBO-based biodiesel blends samples stored at 4 and 23 °C for 24 h. In general, the PO-based biodiesel blends have a significantly higher TTF than YG- and SBO-based biodiesel blends after storage at 4 and 23 °C. The TTF of PO-based biodiesel blends exhibit a relative linear increase as a function of biodiesel concentration at both temperatures. For PO-based samples soaked at 4 °C, the TTF of B70 and B100 were higher than that at room temperature. In contrast, there is no significant difference for TTF of YG-based samples as a function of concentration at both temperatures, except there is a jump in TTF for the YG-B100 at 4 °C storage as compared to room temperature storage. For SBO-based biodiesel, there is also no significant difference in TTF at different blend levels and storage temperature (4 and 23 °C). It should be noted this finding on SBO-based biodiesel is different from our previous study [3]: TTF for SBO-base biodiesel blends (B10, B20, and B50) increased significantly (up to 38 min) at 4 °C storage as compared to room temperature (within 3 min). As noted earlier, the SBO-based biodiesel for each study were obtained from different suppliers. There have been reports suggesting the pretreatment/refining process of the oil before biodiesel production might have an impact on the final SG concentration [9, 20], which should affect the amount of insolubles formation.

Insoluble Mass

The insoluble mass of PO-, YG-, and SBO-based biodiesel blends after storage at 4 and 23 °C is displayed in Fig. 3a–c, respectively. For PO-based samples, the insoluble mass has a relatively linear increase with concentration with a maximum amount of about 1,000 ppm for PO-B100 at 4 °C. The amount of insolubles at 4 °C is about 45% higher than at room temperature for B20. These results indicated that cold soak temperature had an impact on the insoluble mass. However, the amounts of insolubles from YG- and SBO-based samples were found to be much lower than PO-based samples (less than 35 ppm). The difference can be attributed to different natures of insolubles from different feedstocks. Moreover, for SBO-based biodiesel, the insoluble mass of B20 blends show a significant increase after cold soaking compared to room temperature. The increase amount of insolubles may be attributed to the fact that ULSD is a poor solvent (solvency effect) as compared to biodiesel. This is consistent with our previous finding that SBO-based biodiesel blends (B20) displayed a solvency effect with cold soaking [3].

Nature of Insolubles

Identifying insolubles such as sterols and SG compounds in biodiesel has been a challenge. Although chromatograms of SG, which is extracted and purified from biodiesel, using GC-FID have been reported [9, 10], however, to the best of our knowledge, no analytical methods have been reported in identifying sterol and SG compounds in biodiesel directly. In this study, a new sample preparation procedure was developed for GC-FID, with pyridine as a solvent, MSTFA as a derivatization agent, and tricaprin as an internal standard. Using this new procedure, free sterols, SG, steryl ester, and tocopherols peaks in biodiesel can be successfully resolved. It should be noted that the GC-FID operating conditions to identify the sterols and natural antioxidants are the same as determination of free and total glycerin in biodiesel (ASTM D 6584), suggesting that sterols, natural antioxidants, and total glycerin can be quantified using GC-FID method.

The chromatogram of standards reference of sterols using GC-FID is shown in Fig. 4a, where peaks at 18.03, 20.36, 20.40, 20.48, 21.01, and 21.87–22.33 min are due to stigmastanol (free sterol), campesterol glucoside,

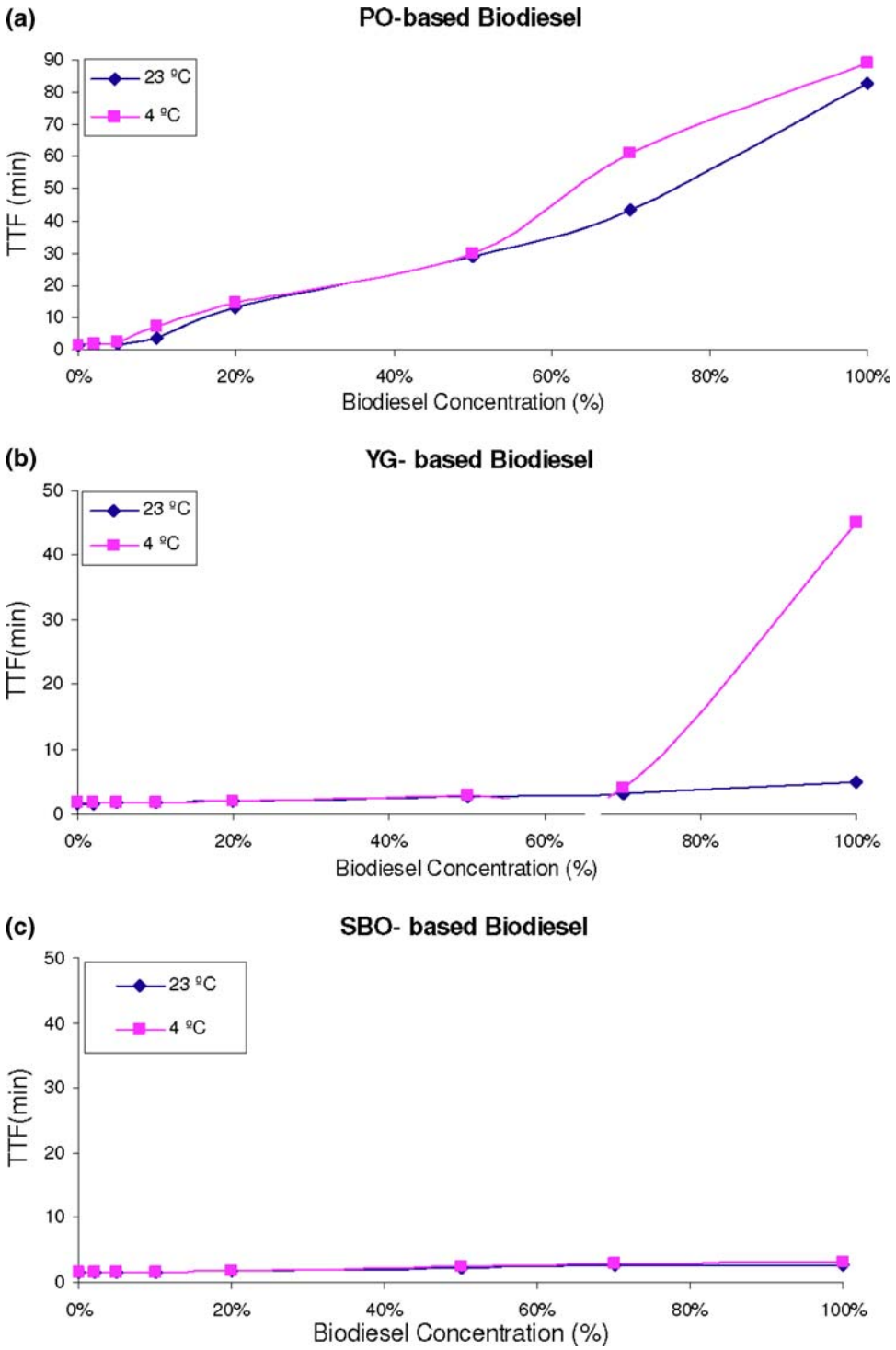

Fig. 2 Time to filter (*TTF*) for a 300-mL volume of ULSD, B2, B5, B10, B20, B50, B70, and B100 **a** PO-, **b** YG-, and **c** SBO-based biodiesel stored at 4 °C and 23 °C for 24 h (the blends were made based on volume percent)

stigmasterol glucoside, β-sitosterol glucoside, cholesteryl stearate (steryl ester), and ESG, respectively, while the peak at 18.60 is ISTD2. The chromatogram of SG is in good agreement with the GC-FID results of Bondioli et al. [9]. Figure 4b shows the chromatogram of a mixture of mono-, di-, tri-glycerides, α-tocopherol, and sterols. Typical peaks of monopalmitin, monoolein, and monostearin were found at 13.24, 14.64, and 14.9 min, respectively; with ISTD2 at 18.60 min, diolein at 19.91–19.98 min, and triolein at 21.89 min. Moreover, the peak displayed at 17.4 min is α-tocopherol, and the peaks corresponding to free sterol, SG, and steryl ester are also observed. Figure 4c shows the chromatogram of concentrated insolubles from SBO-based biodiesel, where peaks of mono-glycerides,

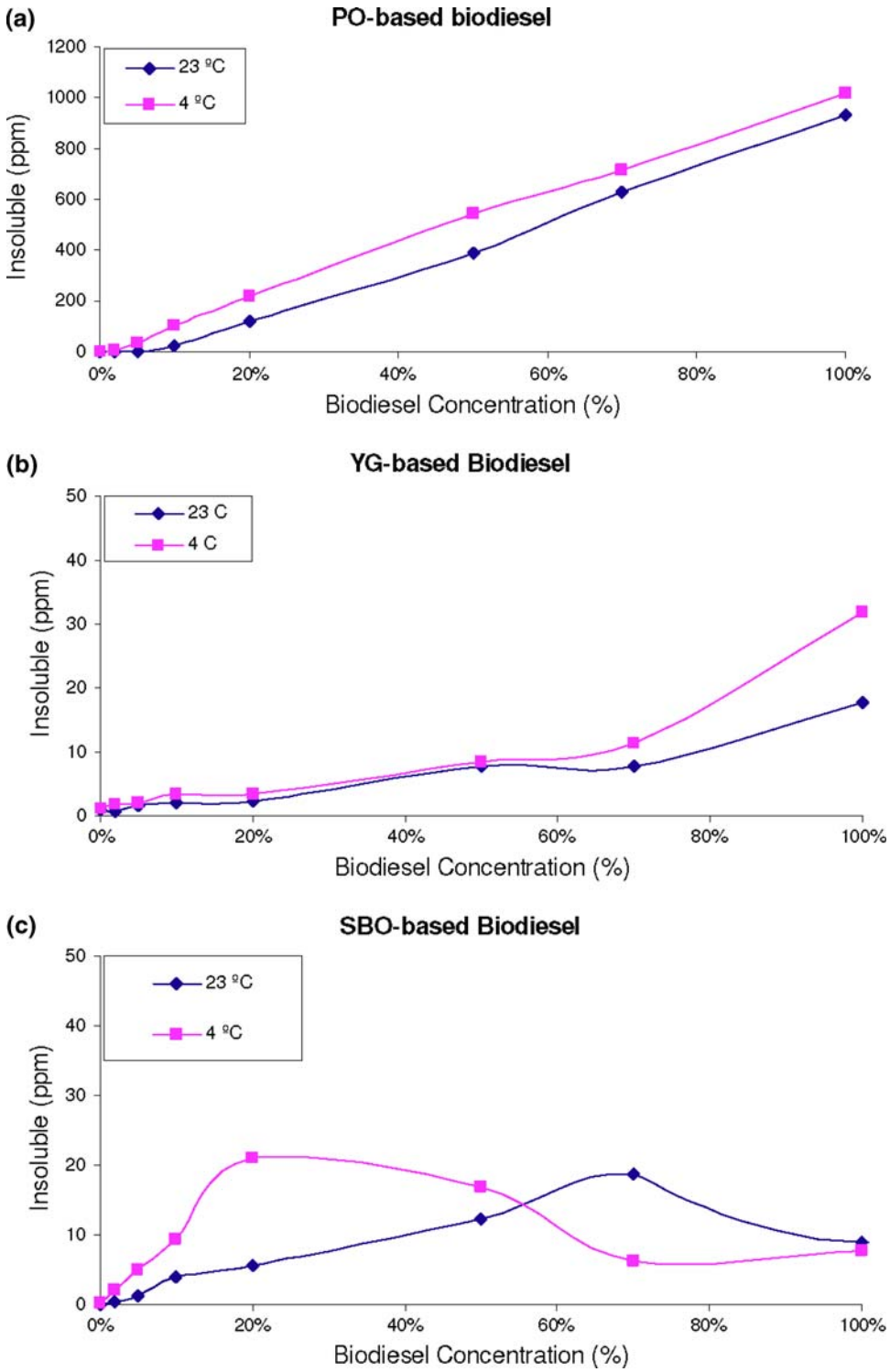

Fig. 3 The mass of precipitates formed by **a** PO-, **b** YG-, and **c** SBO-based fuel stored at 23, 4, and −15 °C for 24 h as a function of biodiesel concentration (volume percent)

free sterols, ISTD2, di-glycerides, SG, and steryl ester are found, based on these reference chromatograms. The results demonstrate that minor components in biodiesel, including glycerides, sterols, and tocopherol can be determined by GC-FID at the same time. It should be noted that the retention time of ESG is close to triglycerides, so it is difficult to identify ESG in the biodiesel.

Figure 5 shows a typical GC-FID chromatogram of insolubles from SBO- B100 and PO-B100. For insolubles formed from SBO-B100 (Fig. 5a), the peaks attributed to ISTD2, campesterol glucoside, stigmasterol glucoside, β-sitosterol glucoside were clearly observed. On the other hand, the peaks attributed to monopalmitin, monosterin, monoolein, and ISTD2 were found on the chromatograms

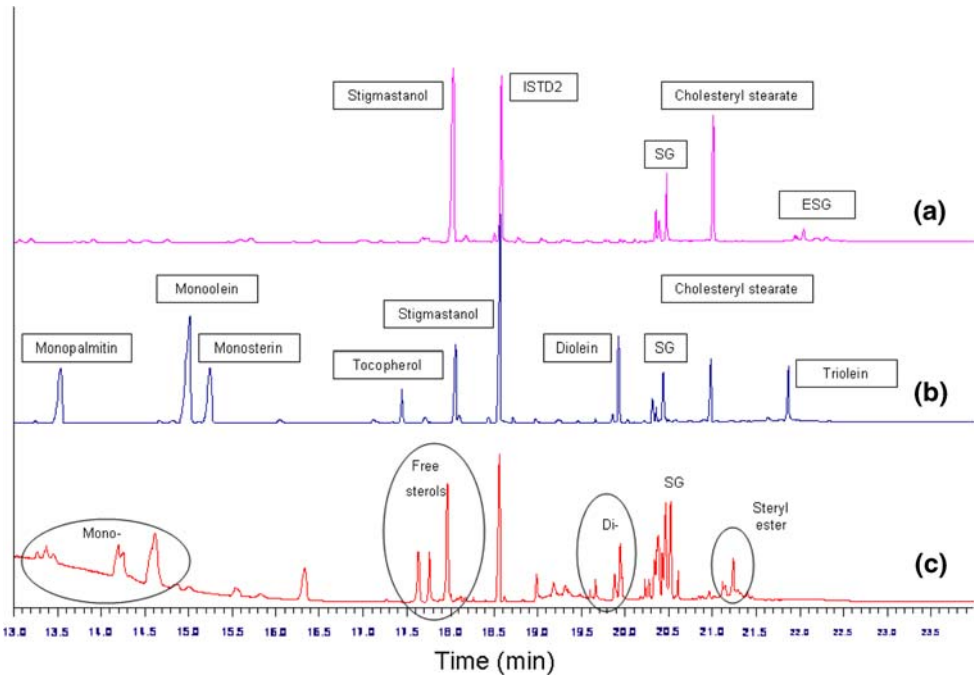

Fig. 4 Representative GC-FID chromatograms of **a** standard reference mixtures of free sterol, ISTD2, campesterol glucoside, stigmasterol glucoside, β-sitosterol glucoside, steryl ester, and ESG, **b** reference standard mixtures of mono-, di- and tri-glycerides, α-tocopherol, ISTD2, and sterols, and **c** the concentrated insolubles from SBO-based biodiesel

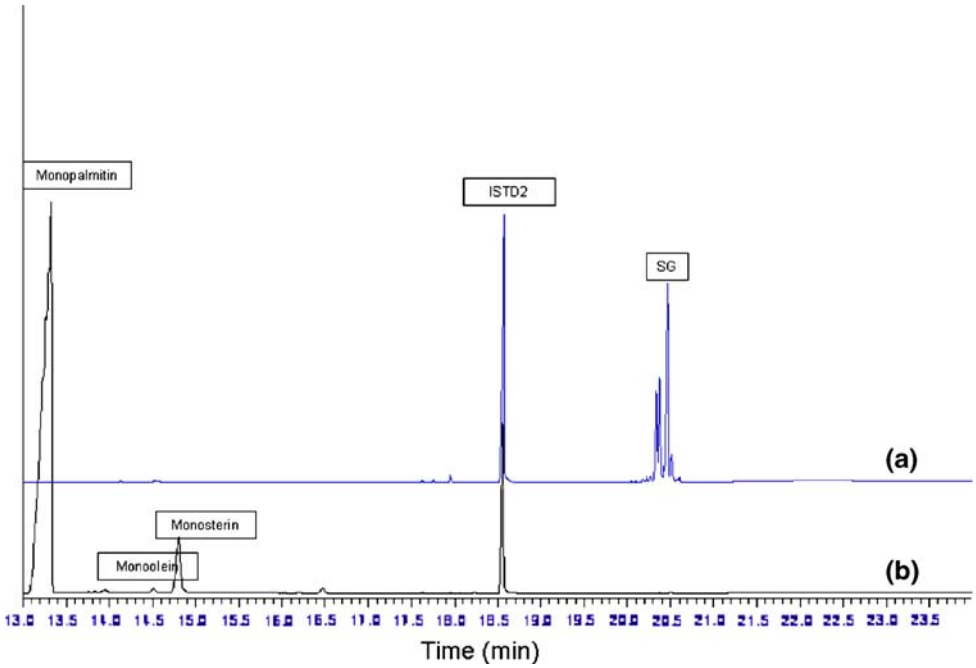

Fig. 5 Representative GC-FID chromatograms of insoluble formed from **a** SBO-B100, and **b** PO-B100

of insolubles from PO-B100 (Fig. 5b). These results indicate that SBO-B100 insolubles are due to SG, where PO-B100 insolubles are due to mono-glycerides.

Figure 6 shows the FTIR spectra of SBO-B100, PO-B100, and their insolubles, compared with standards spectra of monoolein and SG. A broad –OH, asymmetric and symmetric stretch of CH_2, bending vibration of CH_2, and C–O–C absorption peak at 3,391, 2,933 and 2,868, 1,462, and 1,019 cm^{-1}, respectively, were found in the spectrum of standard reference SG (Fig. 6a-1). Similar strong absorption peaks at 3,386, 2,928 and 2,854, 1,459, and 1,019 cm^{-1} were also observed on the spectrum of SBO-B100 insolubles (Fig. 6b-1), while no peak at 3,391 cm^{-1} exists for SBO-B100 (Fig. 6b-2). Thus, SBO-B100 insolubles can be attributed to SG. The spectrum of standard monoolein (Fig. 6a-2) displays the –OH, –CH_2,

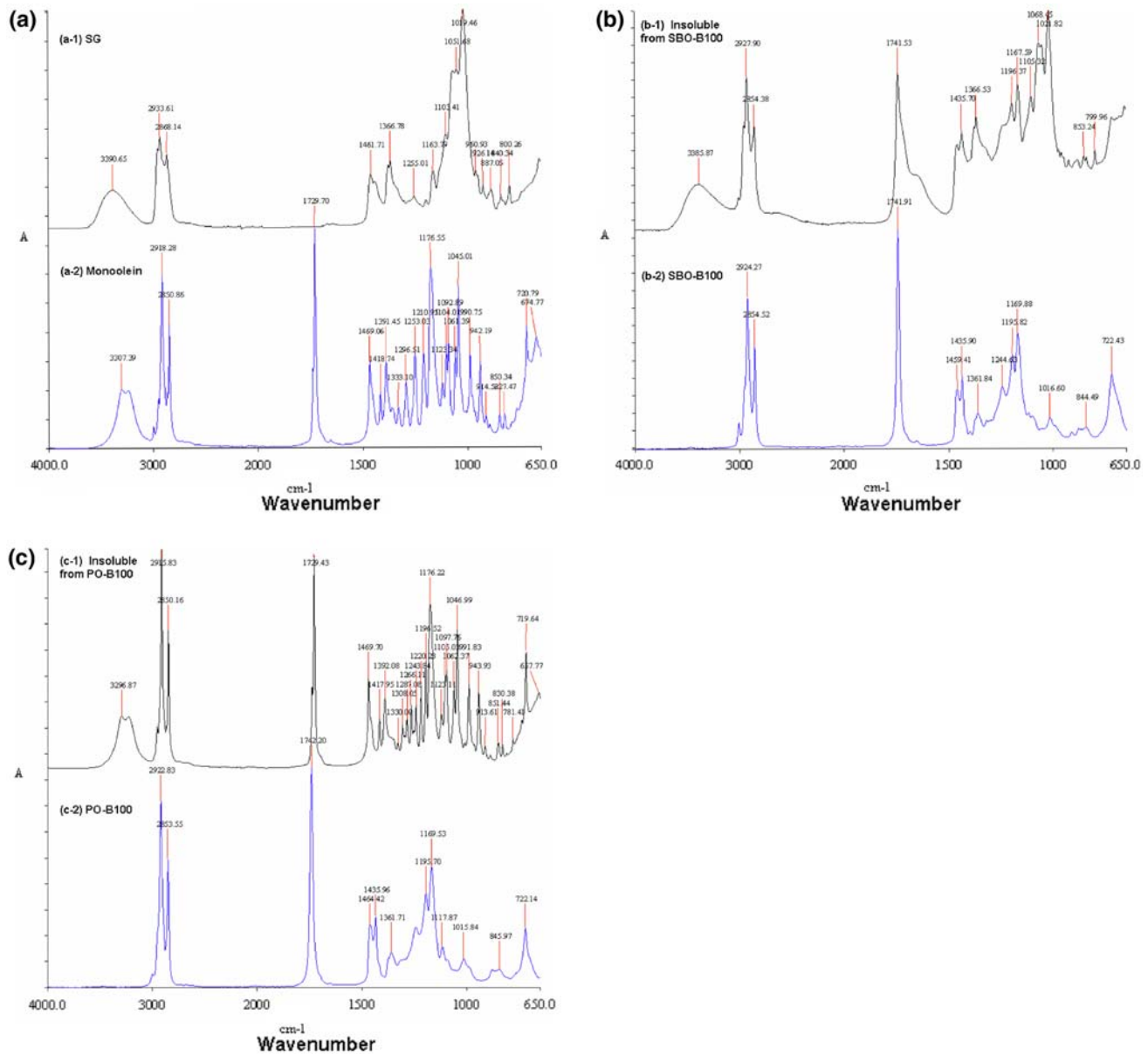

Fig. 6 FTIR spectra from 800 to 4,000 cm^{-1} **a** reference standard SG and monoolein, **b** SBO-B100 and its insolubles, and **c** PO-B100 and its insolubles

–COO absorption peaks at 3,307, 2,918 and 2,851, 1,730 cm^{-1}. The spectrum of insolubles from PO-B100 (Fig. 6c-1) also had strong absorption peaks at 3,297, 2,916 and 2,850, 1,729 cm^{-1}, corresponding to –OH, $–CH_2$, –COO functional groups respectively; however, no –OH absorption was found in the PO-B100 spectrum (Fig. 6c-2). This suggests that the insolubles from PO-B100 can be attributed to monoglycerides. These results are consistent with GC-FID analysis, further confirming that insolubles from PO-B100 are due to monoglycerides, not to SG; while SBO-B100 insolubles are due to SG. Our previous study [3] also showed that SG were the major components of insolubles in SBO-based biodiesel under low temperature storage. However, previous studies [9, 10] reported that the nature of insoluble residue isolated during the production of biodiesel in soy and palm oil-based biodiesel was the same (a mixture of SG); which contradicts our finding. One possibility is that the oil pretreatment and biodiesel production techniques determine the nature of insolubles that are retained in the final product.

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