Fractionation of Oligomeric Triacylglycerides and the Relation to Rejection Limits for Used Frying Oils

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ABSTRACT: Precipitates enriched in oligomeric triacylglycerides were separated from thermally oxidized olive residue oil, conventional and high-oleic sunflower oils, and soybean oil by solvent fractionation in methanol/acetone at 4–5°C for 16 h. Different fractionation conditions were evaluated in an effort to isolate the oligomeric triacylglycerides (OTG). OTG, formed in frying oils upon heating at low concentations, were not detectable with conventional methods to determine polymeric compounds. The best conditions found from the different assays were the following: (i) weight of oil sample-to-solvent volume ratio of 1:20; and (ii) solvent system methanol/acetone 10:90 (vol/vol) for monounsaturated oils and 15:85 (vol/vol) for polyunsaturated oils. Precipitates, enriched in oligomers, were formed when heated oils and used frying oils contained more than 27% polar compounds, a value which is widely accepted as the upper limit for use of frying oils.
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KEY WORDS: Fractionation, frying oil assessment, oligomeric compounds, polar compounds, polymeric compounds, thermal oxidation.

Deep-fat frying is one of the most commonly used procedures, both in the food industry and household culinary practices. Analyses of frying fats in different countries (1–3) indicate that a significant number of used frying fats and oils surpass the alteration limit set by legislation in several countries (4). As a consequence, a more systematic quality assessment of frying fats and oils is required by food processors and food-service institutions.

Two of the most widely used analytical procedures for quality assessment of frying fats and oils are the determination of polar compounds (5) and of polymeric triacylglycerides (6). Values for polar compounds in the range of 25–30% and polymeric triacylglyceride levels of 10 or 16% have been established in different regulations as the upper limit for oil rejection. Although these methods provide a good indication of the compounds that are responsible for oil deterioration, they cannot be applied when laboratory facilities are not available.

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On the other hand, quick tests are available for rapid assessment of oil quality in the fried-food industry, food services, or restaurants. The Fritest®, based on colorimetric measurement of carbonyl compounds formed during thermal oxidation (7), the Spot test (8) and the Shortening Monitor test (9), based on colorimetric measurement of free fatty acid content as an indicator of hydrolytic rancidity, the Food Oil Sensor (10), based on changes in the dielectric constant of heated oils, Oxifritest or RAU-test (11), based on the total amount of oxidized compounds and the TPM test (12), based on a measurement of the total polar materials, are useful tests that can be carried out in fast-food shops or restaurants. However, the accuracy of the above tests is limited because the compounds involved may not be good indicators of the degree of degradation that occurs in the complex process of frying (11,13). Consequently, additional efforts are necessary to develop new tests that may be used in the food industry or food-service institutions. In particular, such a test should be capable of accurately determining the point of needed frying fats and oils rejection.

In an effort to standardize a rapid method to determine the rejection point of a frying oil, fractionation between certain polymeric compounds and all other constituents was carried out previously (14). In that work, a precipitate enriched in polymeric compounds was obtained by solvent fractionation at 0°C in methanol/acetone (10:90, vol/vol), when oil degradation was higher than the limit set for oil rejection (25–30% polar compounds) in samples of corn and cottonseed oils. A small amount of a solid-phase precipitate, consisting of the more saturated triacylglycerides of the oils (15), also was observed. A higher temperature (4–5°C) has been used in this work to minimize the precipitation of the saturated triacylglycerides and to maximize the percentage of polymeric compounds in the precipitate.

The objectives of this paper were: (i) to select new fractionation conditions at a higher temperature (4–5°C), than previously reported to isolate the abovementioned polymeric compounds; (ii) to determine the composition of these polymeric compounds by means of high-performance size-exclusion chromatography (HPSEC); (iii) to examine if the previously mentioned precipitate forms when the amount of polar compounds reaches the limit for oil rejection in other types of

oils (high-oleic sunflower oil, olive residue oil, sunflower oil, and soybean oil).

EXPERIMENTAL PROCEDURES

Materials and treatments. High-oleic sunflower seed (*Helianthus annuus*) oil (HOSO), olive residue oil (*Oleae europeae*) (or olive pomace oil, PO), sunflower seed (*H. annuus*) oil (SO), and soybean seed (*Glycine max*) oil (SBO), supplied by Spanish manufacturers, were subjected to thermal oxidation. Samples (50 mL) were placed in open beakers of 100 mL and heated at 170 ± 10°C in a sand bath for different periods of time, to obtain samples of increasing polar compound content, within a range that is normally found in frying fats.

Used frying oils from a number of restaurants and fried-food outlets in Southern Spain were supplied by local Food Inspection Services. According to information provided to us, oil samples were olive oil (OO), PO, SBO, and SO.

Solvent fractionation in methanol/acetone. Fractionation was carried out as follows: Oil samples of 1 ± 0.001 g were weighed in a test tube of known weight, and the suitable solvent, consisting of variable mixtures of methanol/anhydrous acetone, was added to the sample. The test tube was then sealed, and the contents were thoroughly mixed and placed in a refrigerator at 4–5°C overnight. After a period of 16 h, the insoluble fraction formed was separated by removing the soluble fraction with a Pasteur pipette. The insoluble fraction was rinsed with 10 mL of cold solvent at the same temperature, dissolved in tetrahydrofurane, and analyzed by HPSEC under the conditions described below.

Determination of polar compounds and polar compound distribution. Total polar compounds were determined in oil samples by silica column chromatography, following the method proposed by IUPAC (5), with two slight modifications (16). Distribution of polar compounds was determined by HPSEC in a Konik model 500 A liquid chromatograph (Barcelona, Spain), equipped with a 10-μL sample loop. Separation was achieved on two 100 and 500 Å Ultrastyragel columns (Waters Associates, Milford, MA), connected in series and operating at 35°C. The columns were 25 cm × 0.77 cm i.d. and packed with a porous, highly crosslinked styrene-divinylbenzene copolymer (10 μm). A refractive index detector (Hewlett-Packard, Pittsburg, PA) was used for detection. High-performance liquid chromatography-grade tetrahydrofuran served as the mobile phase at a flow rate of 1 mL/min, and the sample concentrations were between 15 and 20 mg/mL in tetrahydrofuran.

RESULTS AND DISCUSSION

Table 1 shows percentages of polar compounds and main groups of altered compounds in thermoxidized oils. The methodology used allows separation of five peaks that correspond to polymeric triacylglycerides (PTG), dimeric triacylglycerides (DTG), oxidized monomeric triacylglycerides (oxMTG), diacylglycerides (DG), and fatty acids (FA). The last peak also included the polar unsaponifiable fraction (16). Chromatograms A and B in Figure 1 are representative of the separation of the total sample by HPSEC (A) and of the polar compounds obtained after separation of nonpolar triacylglycerides (B).

TABLE 1
Total Polar Compounds (wt% in oil) and Polar Compound Distribution (wt% in oil) in Thermoxidized Oils

		Distribution[c]				
Sample[a]	PC[b]	PTG	DTG	oxMTG	DG	FA
HOSO-1	16.7	2.1	5.7	6.9	1.5	0.5
HOSO-2	24.4	5.2	7.6	8.7	2.3	0.6
HOSO-3	27.0	5.7	8.4	9.6	2.6	0.7
HOSO-4	34.4	8.3	10.8	12.7	2.0	0.6
HOSO-5	37.4	10.4	11.6	12.9	1.9	0.6
HOSO-6	41.5	12.9	12.0	14.0	2.0	0.6
PO-1	12.8	0.8	3.2	5.8	2.4	0.6
PO-2	24.5	4.0	7.3	9.4	3.2	0.6
PO-3	27.0	6.0	6.9	9.9	3.7	0.5
PO-4	32.1	6.7	9.7	11.2	3.9	0.5
PO-5	33.3	6.3	10.2	13.4	3.0	0.4
PO-6	37.1	9.4	10.9	13.5	3.3	0.4
SO-1	16.8	1.8	7.9	5.6	1.1	0.4
SO-2	27.9	5.2	12.1	9.0	1.1	0.5
SO-3	32.3	7.2	12.3	10.9	1.3	0.5
SO-4	35.9	8.4	15.0	10.7	1.1	0.6
SO-5	36.6	8.7	14.4	12.0	1.1	0.5
SO-6	42.2	11.8	15.8	13.0	1.2	0.5
SBO-1	22.4	3.5	8.9	7.9	1.6	0.5
SBO-2	28.5	5.9	11.0	9.5	1.6	0.5
SBO-3	29.4	6.3	11.4	9.6	1.6	0.5
SBO-4	30.7	7.5	10.9	9.6	2.0	0.6
SBO-5	32.7	7.9	11.5	11.1	1.7	0.5
SBO-6	35.1	9.1	12.8	11.0	1.6	0.6

[a]HOSO, high-oleic sunflower oil; PO, olive residue oil; SO, sunflower oil; SBO, soybean oil.
[b]PC, polar compounds.
[c]PTG, polymeric triacylglycerides; DTG, dimeric triacylglycerides; oxMTG, oxidized monomeric triacylglycerides; DG, diacylglycerides; FA, fatty acids.

Samples were selected with increasing polar compound content. The first of each oil type—HOSO, PO, SO, SBO—contained only moderate amounts of polar compounds. Samples 2 and 3 of each oil type (with the exception of SO-3) contained 24–30% polar compounds, which is near the point of oil discard. Finally, the last group of samples (4–6) had percentages of polar compounds (PC) above the acceptable limit.

In general, results shown in Table 1 indicate the lower tendency of PO and HOSO to polymerize as compared to polyunsaturated oils—SO and SBO. For example, at the same polar compound content (27%), HOSO-3 and PO-3 gave a lower level of PTG + DTG, (approximately 14%), than SO-2 and SBO-2 (approximately 17%). The importance of oxMTG as intermediates in the polymerization reactions also is deduced because the higher the level of polar compounds, the higher the proportional increase in polymerization compounds. For example, going from HOSO-1 to HOSO-6 PTG + DTG tripled, whereas oxMTG doubled. DG and FA

remained at levels similar to the initial values. Also, the level of PTG was approximately 6% in samples at levels close to the limit for oil rejection, although the sum of PTG + DTG ranged between 13 and 17% .

Selection of fractionation conditions. In a previous paper (14), it was demonstrated that an oily precipitate was obtained in oils close to the limit for oil rejection, after solvent fractionation at 0°C. The precipitate contained a great concentration of polymeric compounds, as deduced by viscosity measurements and nonelution materials from the gas-chromatographic column. In an attempt to determine classes of the precipitated polymeric compounds, HPSEC was used to separate these classes according to their molecular weight.

One sample of each oil, among those included in Table 1, was chosen to establish solvent fractionation conditions.The selection took into account that the abovementioned precipitate was obtained from used frying oils with PC percentages close to the limit for oil rejection (25–30% PC). Samples HOSO-4, PO-4, SO-3, and SBO-3 were chosen, which contained polar compounds in the range of 30–34%.

The main variables that influenced precipitation at low temperature were cooling temperature, cooling time, solvent polarity, and oil weight-to-solvent volume ratio. As mentioned earlier, cooling temperature and time were chosen *a priori* to minimize precipitation of the more saturated triacylglycerides, usually occurring in some polyunsaturated oils, such as cottonseed oil (15). Thus, 4–5°C, achievable in a typical refrigerator, and 16 h (overnight) were established. However, the precipitate, which was oily in nature for all oils used in this work, was already formed after 2–6 h, depending on oil degradation. The lower the alteration, the more time needed for precipitation. Also, the influence of temperature was tested in the temperature range from 0 to 8°C; a slight difference in solvent polarity was needed to obtain the same amount of precipitate for the extreme temperatures (0 and 8°C). However, fluctuations in temperature of 1 or 2°C had no appreciable effect on the quantity or composition of the precipitates. All these experiments were carried out at an oil weight/solvent volume ratio of 1:20 (wt/vol), conditions that were established in a previous paper (14).

Effect of solvent polarity was examined by increasing the concentration of methanol in the solvent used for fractionation. After precipitation of the four selected samples in methanol/acetone 5:95, 10:90, 15:85, 20:80, 25:75, and 30:70, precipitates were weighed and analyzed by HPSEC.

Figure 1 shows representative HPSEC chromatograms of a monounsaturated thermoxidized oil sample (A), polar compounds separated by silica column (B), and precipitates obtained at two different solvent polarities (C and D). In Figure 1A, the thermoxidized oil sample contained a high concentration of monomeric triacylglycerides (MTG). In Figure 1B, the polymeric triacylglyceride (PTG) and dimeric triacylglyceride (DTG) peaks were significantly increased due to elimination of the nonpolar MTG. In Figure 1C, the precipitate obtained from the thermoxidized oil after fractionation in methanol/acetone (10:90) contained a high concentration of compounds of high molecular weight (MW) that corresponded to higher-than-trimeric triacylglycerides. Thus, retention time of the main broad peak in chromatogram Figure 1C was shorter than that of PTG corresponding to compounds with MW higher than DTG. This group of compounds was not observed either in the unfractionated sample (Fig. 1A) or in the polar compounds fraction (Fig. 1B) and is referred to as oligomeric triacylglycerides (OTG) throughout the paper. In Figure 1D, the precipitate obtained after fractionation in methanol/acetone (15:85) is shown. By comparing chromatograms C and D, the effect of increasing the amount of methanol in the solvent used can be deduced. Thus, the concentration of MTG was low in chromatogram C,whereas a considerable amount was found at a higher methanol-to-acetone ratio (chromatogram D), which indicates precipitation of MTG present in the oil sample.

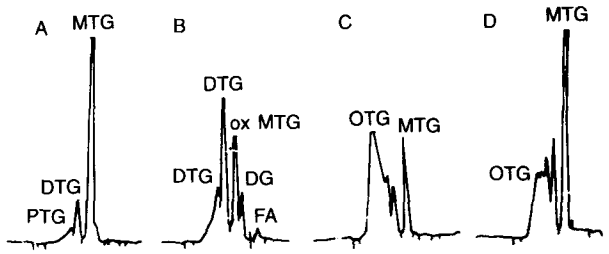

FIG. 1. Significant part of high-performance size-exclusion chromatography chromatograms corresponding to: A, thermoxidized monounsaturated oil; B, polar compounds obtained from A (after separation of nonpolar triacylglycerides by silica column); C, precipitate obtained from A after fractionation in methanol/acetone 10:90 (vol/vol); and D, precipitate obtained from A after fractionation in methanol/acetone 15:85 (vol/vol). FA, fatty acids; DG, diglycerides; DTG, dimeric triacylglycerides; PTG, polymeric triacylglycerides; MTG, monomeric triacylglycerides; oxMTG, oxidized monomeric triacylglycerides; OTG, oligomeric triacylglycerides.

Quantitative results of the weights of precipitates and concentrations of OTG are shown in Table 2 for the four samples selected. As can be observed, an increase in solvent polarity,

TABLE 2
Solvent Fractionation in Methanol/Acetone of Thermoxidized Oils: Influence of Increasing Percentages of Methanol on the Amount of Precipitate (wt% in oil) and Concentration of Oligomeric Triacylglycerides (wt% in precipitate)

Methanol	HOSO-4		PO-4		SO-3		SBO-3	
%	Prec.[a]	OTG[b]	Prec.	OTG	Prec.	OTG	Prec.	OTG
5	0	—	0	—	0	—	0	—
10	2.2	39	2.6	50	0	—	0	—
15	10.4	18	11.9	16	1.0	36	0.6	35
20	23.5	n.d.[c]	21.7	n.d.	3.7	20	4.5	n.d.
25	33.5	n.d.	35.0	n.d.	13.6	n.d.	10.7	n.d.
30	50.8	n.d.	56.4	n.d.	24.8	n.d.	26.9	n.d.

[a]Prec., precipitate.
[b]OTG, oligomeric triacylglycerides.
[c]n.d. , not detectable. See Table 1 for other abbreviations.

through the addition of methanol, clearly increased the amount of precipitate and reduced the concentration of OTG.

A small amount of a precipitate, high in OTG, was obtained with 10% methanol for monounsaturated oils (HOSO-4 and PO-4). The amount of the precipitate was increased, and the concentration of OTG was reduced with 15% methanol. Moreover, OTG were not detectable in the precipitates obtained with 20, 25, and 30% methanol, which indicates that selectivity in the precipitated OTG was practically lost, because a large amount of MTG was simultaneously precipitated (the chromatograms obtained were similar to Fig. 1A). At lower solvent polarity (5% methanol), the small amount of OTG formed (% OTG in oil = % weight of precipitate × % OTG in precipitate = approximately 1%) was dissolved.

On the other hand, 15% methanol was necessary to obtain a precipitate enriched in OTG from polyunsaturated oils (SO-3 and SBO-3). At lower solvent polarity (10% methanol), the small amount of OTG (approximately 0.5%) was solubilized. At higher solvent polarity (20, 25, and 30% methanol), the selectivity of the precipitated OTG was practically lost because a large amount of MTG simultaneously precipitated.

These results emphasize the importance of triacylglyceride composition in solvent fractionation and the convenience of slightly modifying solvent polarity, depending on oil unsaturation, to optimize the fractionation conditions for the isolation of OTG.

Finally, the influence of the oil weight-to-solvent volume ratio was also defined. Oil samples were subjected to fractionation from 1:15, 1:20, 1:25, and 1:30 (wt/vol) solutions in two mixtures of methanol in acetone (10 and 15%). The precipitates formed were subsequently examined by HPSEC. The analysis showed that the precipitate was mostly enriched in OTG at dilute solutions. At the ratio 1:15, the precipitate contained a lower amount of OTG, and at the ratios 1:25 and 1:30, the small amount of precipitate, formed at the crucial point for oil rejection, partly dissolved. Considering the above observations, it was decided to use the ratio 1:20 (wt/vol) in further assays.

Fractionation of thermoxidized oils. Based on the preliminary results obtained on selection of fractionation conditions, all experiments on thermoxidized oils were carried out with 1:20 (wt/vol) solutions of the samples in methanol/acetone mixtures of both 10:90 and 15:85 (vol/vol).

Table 3 shows the absence or presence of a precipitate, along with its OTG content. PC have also been included for a direct comparison between PC and the presence of a precipitate enriched in OTG. As can be clearly observed, the best results were obtained with methanol/acetone mixtures (10:90) for monounsaturated oils and (15:85) for polyunsaturated oils.

By using methanol/acetone (15:85) for monounsaturated oils, the selectivity of OTG was decreased because MTG precipitated (Fig. 1D). Precipitates were also obtained for samples of HOSO-1 and PO-1 with intermediate levels of polar compounds, which consisted of all other constituents except OTG (Fig. 1A).

TABLE 3
Solvent Fractionation in Methanol/Acetone Mixtures of Thermoxidized Oils; Total Polar Compounds (wt% in oil) and Oligomeric Triacylglycerides (wt% of precipitate)

		Methanol/acetone (10:90)		Methanol/acetone (15:85)	
Sample	PC[a]	Prec.[b]	OTG[c]	Prec.	OTG
HOSO-1	16.7	No	—	Yes	n.d.[d]
HOSO-2	24.4	No	—	Yes	n.d.
HOSO-3	27.0	No	—	Yes	21.9
HOSO-4	34.4	Yes	39.1	Yes	18.1
HOSO-5	37.4	Yes	64.1	Yes	21.6
HOSO-6	41.5	Yes	46.1	Yes	26.4
PO-1	12.8	No	—	Yes	n.d.
PO-2	24.5	No	—	Yes	n.d.
PO-3	27.0	No	—	Yes	17.2
PO-4	32.1	Yes	50.0	Yes	16.0
PO-5	33.3	Yes	48.7	Yes	18.3
PO-6	37.1	Yes	45.4	Yes	21.9
SO-1	16.8	No	—	No	—
SO-2	27.9	No	—	No	—
SO-3	32.3	No	—	Yes	35.8
SO-4	35.9	Yes	41.1	Yes	46.2
SO-5	36.6	Yes	46.2	Yes	46.9
SO-6	42.2	Yes	45.7	Yes	50.0
SBO-1	22.4	No	—	No	—
SBO-2	28.5	No	—	No	—
SBO-3	29.4	No	—	Yes	34.6
SBO-4	30.7	No	—	Yes	41.5
SBO-5	32.7	No	—	Yes	43.3
SBO-6	35.1	Yes	35.8	Yes	36.7

[a]PC, polar compounds.
[b]Prec., precipitate.
[c]OTG, oligomeric triacylglycerides.
[d]n.d., not detectable. See Table 1 for other abbreviations.

On the contrary, by using methanol/acetone 10:90 for polyunsaturated oils, the precipitate was observed at high levels of PC, although high amounts of OTG were found for both concentrations of methanol. This indicates that the low amount of OTG initially formed solubilizes at lower solvent polarity than that corresponding to 15% methanol. Thus, a slightly different solvent polarity is needed to fractionate the OTG that originate from different types of mono- and polyunsaturated oils. In view of these results, 10 and 15% methanol were selected for mono- and polyunsaturated oils, respectively, to isolate the OTG formed upon heating.

Interestingly, the precipitate enriched in OTG was obtained from samples with polar compound levels higher than 27% which, in turn, contained more than 6% PTG or 13–17% PTG + DTG (see Table 1). This indicates that a good correlation exists between the formation of the precipitate and the acceptable limit for oil rejection.

Fractionation of used frying oils. Tables 4 and 5 list the results obtained for monounsaturated oils with methanol/acetone 10:90 and for polyunsaturated oils with methanol/ace-

TABLE 4
Used Frying Monounsaturated Oils: Total Polar Compounds (wt% in oil), Polymeric Triacylglycerides (wt% in oil) and Results of Solvent Fractionation in Methanol/Acetone (10:90, vol/vol)

Sample	PC[a]	PTG[b]	Precipitate
A1	11.4	0.8	No
A2	12.3	1.0	No
A3	14.5	1.4	No
A4	17.3	2.7	No
A5	22.7	5.1	Yes
A6	23.6	5.0	No
A7	25.2	5.0	No
A8	25.8	7.2	Yes
A9	26.1	5.5	Yes
A10	28.6	6.4	Yes
A11	29.7	7.0	Yes
A12	32.9	8.0	Yes
A13	33.4	6.1	Yes
A14	33.8	7.0	Yes
A15	34.7	4.8	No
A16	34.7	8.5	Yes
A17	36.8	10.5	Yes
A18	38.8	9.8	Yes
A19	38.9	11.0	Yes
A20	49.1	14.9	Yes

[a]PC, polar compounds.
[b]PTG, polymeric triacylglycerides.

tone 15:85. Oils were classified according to the information provided and confirmed by analysis of FA composition. In addition to the results of the test application, PC and PTG concentrations in the oils also were included.

In general, formation of a precipitate enriched in OTG occurred at PC levels that were close to those established for rejection of used frying fats. Thus, in monounsaturated oils (Table 4), a precipitate was visually detected in samples with PC content above 25.8%, with the exception of A5 and A15. In polyunsaturated oils (Table 5), appearance of a precipitate corresponded to a PC level of 23.8%. Therefore, simply by keeping the used frying oil at 4–5°C for 16 h in a methanol/acetone solution, valuable information was obtained regarding discarding the oil.

TABLE 5
Used Frying Polyunsaturated Oils: Total Polar Compounds (wt% in oil), Polymeric Triacylglycerides (wt% in oil) and Results of Solvent Fractionation in Methanol/Acetone (15:85, vol/vol)

Sample	PC[a]	PTG[b]	Precipitate
B1	10.4	1.8	No
B2	12.5	1.1	No
B3	15.1	2.0	No
B4	15.6	2.3	No
B5	15.8	2.4	No
B6	16.5	2.1	No
B7	17.9	2.1	No
B8	21.0	4.0	No
B9	23.8	5.1	Yes
B10	25.7	6.1	Yes
B11	30.1	7.1	Yes
B12	30.2	7.7	Yes
B13	30.5	9.5	Yes
B14	35.9	11.7	Yes
B15	37.3	10.6	Yes
B16	37.5	9.7	Yes
B17	39.5	9.7	Yes
B18	49.7	15.1	Yes
B19	50.0	8.8	Yes
B20	53.6	20.6	Yes

[a]PC, polar compounds.
[b]PTG, polymeric triacylglycerides.

Additional information can be deduced on the relationship between the appearance of the precipitate and the level of PTG. The false results obtained for A5 and A15 might have an explanation once PTG were quantified. As can be observed, A15 had a low level of PTG in spite of its high concentration in PC. A more detailed evaluation confirmed that the sample corresponded to an olive oil with a high level of DG. On the other hand, sample A5 seemed to have a higher level of PTG than that found for other samples with similar PC percentages. In conclusion, results from frying oils indicated that a minimum level of PTG of around 5% would be necessary for the formation of OTG, which precipitated under the selected fractionation conditions.

Finally, it is important to comment that this test would be applicable only to liquid oils. In saturated fats or hydrogenated oils, a large amount of triacylglycerides of the type SSU or SSS (S, saturated fatty acid; U, unsaturated fatty acid) would crystallize to produce a solid-phase precipitate at low temperature. Such hydrogenated oils and fats, however, are stable to thermoxidation and rarely result in oil discard. The low amount of saturated TG contained in some polyunsaturated oils, such as cottonseed oil (15), becomes neglible at higher fractionation temperatures established in this work and does not interfere in the analysis.

In conclusion, under the specific fractionation conditions established in this work, it is possible to isolate oligomeric TG for futher analysis. The fractionation of OTG is based on different solubility properties between compounds with higher-MW (oligomeric TG) and lower-MW (trimeric, dimeric, and monomeric TG). The low amount of OTG formed upon heating, which is not detectable with conventional methods used for determination of polymeric compounds in frying oils and fats, can be determined this way. In addition, the formation of the OTG precipitate correlated well with the upper limit of oil rejection (25–30% PC, 6% PTG, and 13–16% PTG + DTG). Thus, fractionation would be a useful test for quality assessment of frying oils in the food industry, where the type of oil is known. Additional modifications are needed to deal with replacement of the flammable solvents and with measuring the sample volumetrically instead of gravimetrically, so that the test becomes applicable in food services without laboratory facilities.

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