Trans Fatty Acid Composition and Tocopherol Content in Vegetable Oils Produced in Mexico

L.A. Medina-Juárez[a,b,*], N. Gámez-Meza[a,b], J. Ortega-García[a], J.A. Noriega-Rodriguez[a], and O. Angulo-Guerrero[b]

[a]Departamento de Investigaciones Científicas y Tecnológicas de la Universidad de Sonora, 83000, Hermosillo, Sonora, México, and [b]Unidad de Investigación y Desarrollo en Alimentos, Instituto Tecnológico de Veracruz, 91860, Veracruz, México

ABSTRACT: The purpose of this study was to evaluate the *trans* fatty acid (TFA) composition and the tocopherol content in vegetable oils produced in Mexico. Sample oils were obtained from 18 different oil refining factories, which represent 72% of the total refineries in Mexico. Fatty acids and TFA isomers were determined by gas chromatography using a 100-m fused-silica capillary column (SP-2560). Tocopherol content was quantified by normal-phase high-performance liquid chromatography using an ultraviolet detector and a LiChrosorb Si60 column (25 cm). Results showed that 83% of the samples corresponded to soybean oil. Seventy-two percent of the oils analyzed showed TFA content higher than 1%. Upon comparing the tocopherol contents in some crude oils to their corresponding deodorized samples, a loss of 40–56% was found. The processing conditions should be carefully evaluated in order to reduce the loss of tocopherols and the formation of TFA during refining.

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KEY WORDS: Tocopherols, *trans* fatty acid, vegetable oils.

Traditionally, the purpose of refining oil is to eliminate all impurities present in the crude oil in order to obtain a good quality vegetable oil. The refining process improves flavor and appearance as well as chemical stability. However, in developed countries (Japan, the United States, and European Countries), the nutritional quality of the edible oils is now becoming considerably important. This means that refining has the purpose of not only eliminating impurities such as phospholipids, free fatty acids, peroxides, polymers, pigments and secondary oxidation products but also minimizing *trans* fatty acid (TFA) formation and tocopherol loss (1).

The typical oil refining process includes degumming, chemical or physical refining, bleaching, and deodorization (2). Deodorization is an important step in the oil refining process. During this step, steam at 1–6 mm Hg pressure and 220–260°C is injected into the oil in order to eliminate free fatty acids, aldehydes, unsaturated hydrocarbons, and ketones, which cause undesirable odors and flavors in the oil (3). However, during deodorization, thermal degradation of vegetable oils may occur, the double bond may isomerize from *cis* to *trans* (4), and tocopherols may be lost (5).

Several population studies have examined the relationship between TFA intake and coronary heart disease risk (6,7). In addition TFA inhibits desaturation of essential fatty acids (8).

Tocopherols are important lipid oxidation inhibitors in food and biological systems. These natural antioxidants are found in oilseeds in four different forms: α-tocopherol (α-T), β-tocopherol (β-T), γ-tocopherol (γ-T), and δ-tocopherol (δ-T) (9). They are found in higher concentration in crude soybean oil (1,200 ppm) than in corn oil (900 ppm) or cotton oil (790 ppm) (10). However, these important natural antioxidants are drastically reduced during the degumming, refining, bleaching, and especially deodorization of the oils (1,5).

In European countries the quality parameters for refined edible oils include low levels of TFA (<1%) and high retention of tocopherols because of their health implications (11, 12). Deodorization conditions have been set such that tocopherol loss never exceeds 25% (1).

The objective of this study was to evaluate the TFA composition and the tocopherol content in refined vegetable oils produced in Mexico.

EXPERIMENTAL PROCEDURES

Oil sample collection. The oil samples were selected from 18 Mexican refinery plants with integrated processing systems (extraction and refining), representing 72% of the total refineries in Mexico. Sample AV1 was processed by physical refining, while AV2 to AV18 were processed by chemical refining. All oil samples were deodorized using a semicontinuous process (240–260°C). Samples were collected from February to July (1998) and kept under nitrogen at –20°C in dark brown glass bottles.

Fatty acid profile. Oil samples were saponified and methylated according to AOCS procedure Ce 2-66 (13). Fatty acid methyl esters (FAME) were analyzed in a gas chromatograph (GC) (Model 3400, Varian, Mexico City, Mexico) equipped

*To whom correspondence should be addressed at Departamento de Investigaviones Cietíficas y Tecnológicas de la Universidad de Sonora, Apartado Postal #1819, 83000, Hermosillo, Sonora, México.
E-mail: amedina@guayacan.uson.mx

with flame-ionization detector and integrator (Model 1020, PerkinElmer, Mexico City, Mexico). A fused-silica capillary column coated with 100% biscyanopropyl polysiloxane as the stationary phase (SP-2560, 100 m × 0.25 mm i.d. 20 μm thickness, Supelco, Bellefonte, PA) was used. The carrier gas was nitrogen at a flow rate of 20 cm/s. The oven temperature was programmed: 140 (5 min), 140–170 (4°C/min), 170 (3 min), 170–200 (1.5°C/min), and 200°C (10 min). Injector and detector temperatures were held at 250°C. This single-step direct GC analysis was satisfactory for the reliable quantification of TFA (14–16).

FAME peaks were identified by comparison with the retention time of the respective standards (Sigma Chemical Co., St. Louis, MO). *Trans* isomers were identified from the linoleic acid methyl ester isomer mix and from the linolenic acid methyl ester isomer mix (Sigma Chemical Co.). Quantification was done using $C_{17:0}$ as an internal standard, taking into account the natural content of $C_{17:0}$ in deodorized soybean oils. Results are expressed as weight percentage of oil and are the mean of two replicates.

Tocopherol analysis. Tocopherols were analyzed by a high-performance liquid chromatography (HPLC) (Model 9050, Varian) equipped with an ultraviolet detector (Model 3400, Varian). A normal-phase LiChrosorb Si60 column (25 cm × 4 mm, 5 μ particle size; Supelco) was used, operating at room temperature. The mobile phase was hexane/isopropanol (99.5:0.5) at a 0.7 mL/min flow rate. The solvent mixture was filtered through a 0.45 μm filter before use. Wavelengths were programmed to detect each tocopherol at its maximal absorption: 294 nm (α-T) and 298 nm (γ-T and δ-T).

Two grams of sample were diluted in 25 mL of hexane and injected directly into the HPLC (17,18). The tocopherol peaks were identified by comparison with the retention times of the respective standards (Sigma Chemical Co.). Purity and stability of standards were defined by extinction coefficient ($E^{1\%}$) values measured in a spectrometer (Lambda 2S, PerkinElmer) (13,19). The technique was verified using a certified standard of coconut (NBS 1563-2, NIST, Gaithersburg, MD).

RESULTS AND DISCUSSION

Fatty acid profile. Ninety-four percent of all vegetable oils analyzed presented a fatty acid profile similar to soybean oil (20). One sample (AV9) had a fatty acid profile of a mixture of oils similar to canola and soybean.

Two *trans* isomers (18:2Δ9*c*,12*t* and 18:2Δ9*t*,12*c*) from the linoleic acid (18:2Δ9*c*,12*c*) and three groups of *trans* isomers (18:3Δ9*c*,12*t*,15*t* + 18:3Δ9*t*,12*c*,15*t*; 18:3Δ9*c*,12*c*,15*t* + 18:3Δ9*t*,12*t*,15*c*; and 18:3Δ9*c*,12*t*,15*c* + 18:3Δ9*t*,12*c*,15*c*) from the linolenic acid (18:3Δ9*c*,12*c*,15*c*) were identified in the oil samples (21). The linolenic acid *trans* isomers were calculated as groups because of incomplete separation between individual *trans* isomers (Fig. 1). On the other hand, the $C_{20:0}$ was eluted before *trans* isomers of linolenic acid, in agreement with the results reported previously (14,22).

The TFA content in oil samples varied from 0.90 to 2.93%

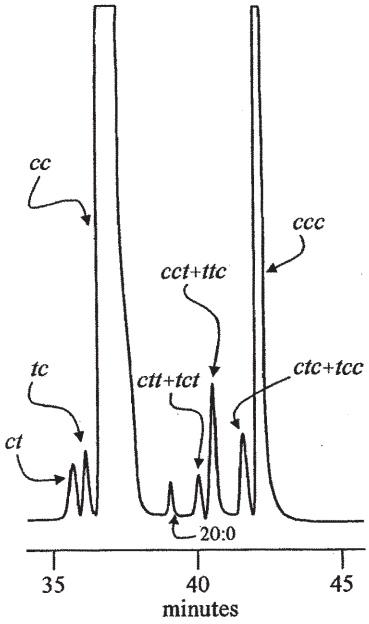

FIG. 1. Partial chromatogram of fatty acid methyl esters prepared with AV6 sample. Analysis on a SP-2560 (Supelco, Belloefonte, PA) capillary column with the temperature program, the carrier gas was nitrogen at a flow rate of 20 cm/min. *ct*, 18:2Δ9*c*,12*t*; *tc*, 18:2Δ9*t*,12*c*; *cc*, 18:2Δ9*c*,12*c*; *ctt* + *tct*, 18:3Δ9*c*,12*t*,15*t* + 18:3Δ9*t*,12*c*,15*t*; *cct* + *ttc*, 18:3Δ9*c*,12*c*,15*t* + 18:3Δ9*t*,12*t*,15*c*; *ctc* + *tcc*, 18:3Δ9*c*,12*t*,15*c* + 18:3Δ9*t*,12*c*,15*c*; *ccc*, 18:3Δ9*c*,12*c*,15*c*.

(Table 1). Similar results were reported for deodorized soybean oil in other countries, 0.16–2.99% TFA in France (21) and 0.9–3.5% TFA in Belgium (1). Four samples (AV1, 3, 6, and 9) showed the highest total TFA content (2.21–2.93%) and 72% of all samples analyzed had up 1% TFA content. The levels of α-linolenic *trans* isomers [degree of isomerization (DI): 11.49–22.33] were higher than those derived from linoleic acid (DI: 0.71–3.54), while *trans* isomers of oleic acid were not detected. These results are in accordance with Kellens (1) and Martin *et al.* (23), who found that α-linolenic acid is more sensitive to isomerization than linoleic acid, due to the number of double bonds and to the high temperatures (>220°C) that the oil is exposed to during deodorization.

During deodorization the geometrical isomerization of linoleic and linolenic acids increases with temperature and heating time (4). Total TFA content in all samples ranged from 0.9 to 2.93% (Table 1). This could indicate that the operation conditions used during deodorization vary among the different oil processing companies. However, the distribution pattern of linolenic acid geometrical isomers is almost the same for all samples, 18:3Δ9*c*,12*c*,15*t* + 18:3Δ9*t*,12*t*,15*c* > 18:3Δ9*c*,12*t*,15*c* + 18:3Δ9*t*,12*c*,15*c* > 18:3Δ9*c*,12*t*,15*t* + 18:3Δ9*t*,12*c*,15*t* (Table 1), and it is independent of the total content of TFA. Forty-four percent of the oil samples contained mono-*trans*-isomers of linoleic acid, and six samples (AV9, 6, 1, 3, 2, and 18) had a greater content of 18:2 Δ9*c*,12*t* than 18:2Δ9*t*,12*c* (Table 1).

Deodorization is the principal stage of oil refining that

TABLE 1
***Trans* Fatty Acid Composition (wt %) in Vegetable Oils Produced in Mexico**

Oil	Total *trans*	Linoleic acid				Linolenic acid				
		ct[a]	*tc*	*cc*	DI[b]	*ctt* + *tct*	*cct* + *ttc*	*ctc* + *tcc*	*ccc*	DI
AV9	2.93	0.61 ± 0.06	0.45 ± 0.01	28.85 ± 0.95	3.54	0.41 ± 0.05	0.87 ± 0.08	0.59 ± 0.04	8.05 ± 0.51	18.85
AV6	2.69	0.49 ± 0.05	0.47 ± 0.05	50.94 ± 0.98	1.85	0.37 ± 0.02	0.81 ± 0.05	0.55 ± 0.06	6.25 ± 0.28	21.68
AV1	2.42	0.55 ± 0.06	0.52 ± 0.05	52.91 ± 1.27	1.98	0.29 ± 0.02	0.63 ± 0.04	0.43 ± 0.04	6.20 ± 0.31	17.88
AV3	2.21	0.49 ± 0.04	0.33 ± 0.05	51.22 ± 1.71	1.58	0.25 ± 0.02	0.66 ± 0.07	0.48 ± 0.05	6.13 ± 0.21	18.48
AV2	2.14	0.52 ± 0.04	0.36 ± 0.02	52.65 ± 1.98	1.64	0.27 ± 0.02	0.59 ± 0.06	0.40 ± 0.06	6.56 ± 0.54	16.11
AV18	2.11	0.50 ± 0.03	0.38 ± 0.02	52.18 ± 1.05	1.66	0.26 ± 0.02	0.58 ± 0.03	0.39 ± 0.06	6.51 ± 0.38	15.89
AV4	1.78	ND[c]	ND	50.38 ± 1.03	ND	0.36 ± 0.03	0.78 ± 0.02	0.64 ± 0.08	6.19 ± 0.24	22.33
AV5	1.74	0.30 ± 0.01	0.32 ± 0.03	51.34 ± 1.58	1.19	0.25 ± 0.02	0.52 ± 0.06	0.35 ± 0.01	6.36 ± 0.17	14.97
AV7	1.54	0.16 ± 0.01	0.21 ± 0.02	51.78 ± 1.61	0.71	0.25 ± 0.03	0.55 ± 0.06	0.37 ± 0.03	6.08 ± 0.21	16.14
AV11	1.23	ND	ND	53.86 ± 0.67	ND	0.34 ± 0.01	0.54 ± 0.06	0.41 ± 0.02	6.65 ± 0.04	16.25
AV15	1.13	ND	ND	53.70 ± 0.51	ND	0.24 ± 0.03	0.56 ± 0.08	0.33 ± 0.01	6.78 ± 0.23	14.29
AV16	1.13	ND	ND	53.74 ± 1.83	ND	0.23 ± 0.03	0.49 ± 0.04	0.41 ± 0.02	7.12 ± 0.34	13.70
AV17	1.12	ND	ND	50.38 ± 1.52	ND	0.28 ± 0.03	0.49 ± 0.05	0.35 ± 0.06	5.98 ± 0.37	15.77
AV13	1.02	ND	ND	50.12 ± 0.79	ND	0.18 ± 0.02	0.49 ± 0.06	0.35 ± 0.02	5.41 ± 0.21	15.86
AV12	0.97	ND	ND	51.34 ± 1.26	ND	0.19 ± 0.03	0.42 ± 0.02	0.36 ± 0.01	5.63 ± 0.27	14.70
AV14	0.94	ND	ND	53.48 ± 1.98	ND	0.25 ± 0.02	0.39 ± 0.05	0.30 ± 0.02	7.24 ± 0.20	11.49
AV8	0.93	ND	ND	53.15 ± 1.55	ND	0.19 ± 0.03	0.41 ± 0.06	0.33 ± 0.03	5.74 ± 0.26	13.94
AV10	0.90	ND	ND	50.41 ± 0.32	ND	0.18 ± 0.02	0.39 ± 0.04	0.33 ± 0.02	5.90 ± 0.16	13.24

[a]*ct*, 18:2Δ9*c*,12*t*; *tc*, 18:2Δ9*t*,12*c*; *cc*, 18:2Δ9*c*,12*c*; *ctt* + *tct*, 18:3Δ9*c*,12*t*,15*t* + 18:3Δ9*t*,12*c*,15*t*; *cct* + *ttc*, 18:3Δ9*c*,12*c*,15*t* + 18:3Δ9*t*,12*t*,15*c*; *ctc* + *tcc*, 18:3Δ9*c*,12*t*,15*c* + 18:3Δ9*t*,12*c*,15*c*; *ccc*, 18:3Δ9*c*,12*c*,15*c*).
[b]DI, degree of isomerization for the indicated fatty acid ratio of total *trans* 18:2 (or *trans* 18:3) on total 18:2 (or 18:3) times 100.
[c]ND, not detectable (<0.01%).

contributes to increases in the content of TFA. Therefore, it is recommended that the severeity of the deodorizing conditions be reduced, in order to obtain TFA contents below 1% for rapeseed and soybean oil and below 0.5% for sunflower and corn oil (1).

Tocopherol quantification. The tocopherol content in analyzed oils is shown in Table 2. Eighty-three percent of the samples presented the distinguishing characteristics of a soybean oil in the distribution of tocopherols, γ-T > δ-T > α-T (10,17,24). It is desirable to preserve the high content of δ-T and γ-T in the soybean oil because of their intrinsic antioxidant activity, which is stronger than α-T (9). A recent study has shown that γ-T can prevent polymerization in oils during frying (25).

TABLE 2
Content (ppm) of Tocopherols (T) in Deodorized Vegetable Oils Produced in Mexico[a]

Oil	α-T	γ-T	δ-T	Total
AV1	62	511	129	702
AV2	221	396	101	718
AV3	110	521	163	794
AV4	94	472	233	799
AV5	ND	643	170	813
AV6	85	573	173	831
AV7	96	601	144	841
AV8	100	686	65	851
AV9	86	624	188	898
AV10	83	743	86	912
AV11	101	652	194	947
AV12	85	731	146	962
AV13	91	741	138	970
AV14	212	620	145	977
AV15	64	850	108	1,022
AV16	87	808	144	1,039
AV17	108	792	139	1,039
AV18	71	778	220	1,069

[a]ND, Not detectable (<10 ppm).

During deodorization, part of the tocopherol loss is a result of distillation removal, or a consequence of thermal degradation. The major factors affecting distillation losses are deodorization temperature, pressure, and stripping steam. On the other hand, thermal degradation depends more on time and temperature (>240°C) (1).

Tocopherol content for crude and deodorized soybean oil was compared in five samples (AV1, 3, 6, 7, and 4). Tocopherols lost varied from 40 to 56% (Table 3). Gutfinger and Letan (26) reported that 31–47% of the tocopherols were lost during soybean oil processing. According to Zehnder (3), when the deodorization operation is done at 260°C, tocopherol loss is 40 to 50%.

Taking into account the high antioxidant potency of tocopherols in vegetable oils (27) and the nutritional contribution of vitamin E (28), it is evident that careful attention should be paid to establish the optimal operating conditions in the refining process in order to reduce the tocopherol loss.

Kellens (1) reported that, in order to obtain deodorized oils with low levels of TFA (<1%) and modest tocopherol losses (<25%), chemical refining should be done at 230–235°C and physical refining at 235–240°C. In Mexico, the common deodorization temperature for chemical and physical refining is 240–260°C. This could explain the high content of TFA (>1%) and the high loss of tocopherols (>25%) in samples AV1, 3, 4, 6, and 7. However, oil samples presenting TFA < 1% (AV12, 14, 8, and 10) were deodorized at a temperature of 240°C.

TABLE 3
Content (ppm) of Tocopherols (T) in Crude Soybean Oil Produced in Mexico

Oil	α-T	γ-T	δ-T	Total
AV1				
Crude	98	671	412	1,181
Deodorized	62	511	129	702
AV3				
Crude	115	1,060	337	1,512
Deodorized	110	521	163	794
AV6				
Crude	286	785	523	1,595
Deodorized	85	573	173	831
AV7				
Crude	102	1,250	375	1,727
Deodorized	96	601	144	841
AV4				
Crude	112	1,267	431	1,810
Deodorized	94	472	233	799

The results of this study indicate that it is important to be aware of the nutritional quality of edible vegetable oils. In Mexico, it is recommended that adjustments in the temperature used in deodorization be made to attain international quality standards with respect to the TFA and tocopherol content.

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