Fatty Acid Composition and Its Relationship with Physicochemical Properties of Pecan (*Carya illinoensis*) Oil

J.F. Toro-Vazquez*, M.A. Charó-Alonso, and F. Pérez-Briceño

[a]Facultad de Ciencias Quémicas-CIEP, Universidad Autónoma de San Luis Potosí, San Luis Potosí 78210, México

ABSTRACT: The composition and physicochemical properties of pecan (*Carya illinoensis*) kernels and oils from different native trees of the central region of Mexico were investigated. The main compositional characteristic of the kernel was the high lipid content (70–79% w/w on dry basis) with elevated concentration of oleic acid (55–75% w/w). The results confirmed the relationship in the biosynthesis of linoleic and linolenic acids from oleic acid existing in oilseeds. Our results indicate that in pecans such relationship is a function of pecan tree age. The proportion of oleic, linoleic, and linolenic fatty acids determined the oxidative stability, viscosity, and melting/crystallization behavior of pecan oil. In general, these properties in pecan oils were similar or superior to extra-virgin olive oil and unrefined sesame oil. Although all native pecan oils studied showed a significant concentration of oleic acid, a particular group of native Mexican pecan trees produces an oil with a fatty acid composition with the nutritional appeal that consumers demand nowadays (i.e., very high oleic acid, 60–75%), with excellent natural oxidative stability (i.e., induction time for oxidation between 8.5 and 10.8 h), and substantially higher concentrations of α-, γ-, and δ-tocopherol than in pecan varieties previously reported in the literature.

Paper no. J9046 in *JAOCS 76*, 957–965 (August 1999).

KEY WORDS: Oil viscosity, oleic acid, oxidative stability, pecan oil, tree age.

As consumers become more aware of the health implications of their dietary patterns, their food choices become more selective. The nutritional effect of dietary lipids is nowadays a major consumer issue, affecting consumption habits and food choices. Well-known by consumers is that polyunsaturated fatty acids as well as monounsaturated fatty acids reduce the blood levels of low-density lipoproteins (LDL) when compared to saturated fatty acids. However, Mattson and Grundy (1) reported a decrease in high-density lipoprotein (HDL) levels in subjects following a polyunsaturated diet, while subjects following a monounsaturated fat diet were less able to do so. However, it is important to point out that the results of Mattson and Grundy (1) have not been consistently demonstrated by other investigators. Some of the problems that might be associated with this inconsistency of results have been pointed out (2,3). The HDL are the lipoproteins responsible for transporting cholesterol to the liver where it is catabolized. Then, a reduction in the blood levels of HDL is not advisable. The so-called Mediterranean diet, with a lipid source mainly based on olive oil (55–83% oleic acid), obtained considerable attention, supported by additional claims of lower colon cancer mortality rates and lower incidence of breast cancer in countries that follow this diet (e.g., Spain, Greece, Italy) (2,4).

Another vegetable source high in monounsaturated oil is the pecan. Pecan kernels from the genus *Carya* contain 65–75% oil, depending on growing conditions, maturity, variety, and past productivity of the tree. In general, pecan oil is mainly composed of oleic acid (60–70%) and linoleic acid (19–30%) with small concentrations of palmitic, stearic, and linolenic fatty acids (3,5–9). Pecan oil is very low in saturated fatty acids (<9%)—only high-oleic safflower (4–8%) and canola (<7.0%) oils are lower—and is exceeded in monounsaturated fatty acids concentration by olive oil and other less-commercial oils like hazelnut (≈80–81%) and almond (60–80%).

The objective of this study was twofold: to determine the general composition of pecan kernels (*Carya illinoensis*) and pecan oil and to investigate the relationships between the fatty acid oil profile and some physicochemical properties (i.e., viscosity, stability to oxidation, and melting/crystallization profiles) of pecan oil. In this study we utilized oil extracted from pecan kernels from native trees growing in the central region of Mexico.

MATERIALS AND METHODS

Pecan collection and storage. The pecans from 22 trees were collected. The trees grow in Queretaro (n = 12), Guanajuato (n = 7), and San Luis Potosí (n = 3), states geographically located in central Mexico. The pecan trees were selected under the basis of kernel color (ΔE = 20.6) and agronomic parameters such as pecan yields (112–314 pecans/kg of pecan) and percentage of kernel present in the pecan nut (38.4–49%, wet

*To whom correspondence should be addressed at Facultad de Ciencias Químicas-CIEP, Universidad Autonoma de San Luis Potosí, Av. Dr. Manuel Nava 6, Zona Universitaria, San Luis Potosí, SLP, 78210, México.
E-mail: toro@deimos.tc.uaslp.mx

basis). The ΔE value, a parameter known as color difference, corresponds to one of the six categories defined by Thompson *et al.* (10) and transformed from the Munsell color notation system for pecan color determination. The whole pecans were stored in sealed plastic bags (400 g) at −25°C until their analysis.

Although the age was recorded for each tree collected, this variable was not considered to perform the pecan tree selection. Tree age was determined by counting the tree rings in a transverse section of the tree log (i.e., a core of wood) with a stereoscopic microscope. The transverse section of the tree log was obtained at a height of 1.30 m from the ground with a borer (i.e., Pressler drill) of 14 inches of bit length and 0.169 inches of core diameter.

Chemical determinations. Before the chemical determination, the kernels were manually separated from the shell. The water content was determined by drying (70°C/18 h) of milled kernel (≈2 g). The total lipid content was quantified in dry-milled kernels (≈2 g) through Soxhlet extraction (6 h) with hexane [high-performance liquid chromatography (HPLC) grade]. Protein was determined by micro-Kjeldhal (150–200 mg of dry-milled kernel) using a conversion factor of 5.3. The fatty acid composition of pecan oil was determined by gas chromatography utilizing a Shimadzu chromatograph GC-9A (Shimadzu Corp., Kyoto, Japan) with flame-ionization detector; the conditions utilized in the chromatograph were reported previously (11). The α-, γ-, and δ-tocopherol was determined by HPLC (Waters 600E; Water Millipore Co., Milford, MA) with ultraviolet detection utilizing a Nova-Pak column (3.9 × 150 mm). A solvent composed of hexane/acenonitrile/2-isopropanol (98:1:1, vol/vol/vol) was utilized at a flow rate of 0.9 mL/min. The identification and quantity of particular tocopherols in the oil were achieved by determining the elution time and areas obtained with standards (Sigma, St. Louis, MO) eluted in pure form and in mixtures at different proportions. The exact concentration of the standards was determined through spectrophotometry utilizing an extinction coefficient in ethanol ($E^{1\%}_{1cm}$) of 71 for α-tocopherol at 294 nm, 92.8 for γ-tocopherol at 298 nm, and 91.2 for δ-tocopherol at 298 nm (9). For comparison purposes, samples of unrefined and refined sesame seed oil (Aceitera San Juan, S.A., Salvatierra, Gto., México) as well as samples of extra-virgin olive oil (Ybarra Hermanos S.A., Seville, Spain) were analyzed for fatty acid profile. In all cases, duplicate determinations were performed and the mean reported.

Physicochemical determinations. The oil utilized in these analyses was extracted from milled pecan kernels (≈60 g) with hexane (>99.6 purity) in a Soxhlet apparatus (600 mL) for 12 h. The solvent was evaporated under vacuum in a rotating evaporator at 50°C and the resulting oil centrifuged (3500 rpm/30 min) to remove the remaining aqueous phase. The pecan oil obtained was stored in dark glass bottles under nitrogen at−25°C until utilized in the physicochemical determinations.

The viscosity (η) at 10, 20, and 30°C (η_{10}, η_{20}, and η_{30}) was measured in a cone-and-plate geometry Brookfield DV-III viscometer (Brookfield Instruments, Stoughton, MA), utilizing the spindle CP-40 and a volume of 0.5 mL. The shear stress (Γ) was obtained within the shear rate interval (γ) of 22.5 to 450 s^{-1}. The η at each temperature was calculated by determining the slope of the linear regression of Γ on γ utilizing the statistical package STATISTICA v. 5.1 (StatSoft, Tulsa, OK). Duplicate determinations were performed.

The crystallization and melting profiles were obtained by differential scanning calorimetry (DSC-7; Perkin-Elmer, Norwalk, CT); the equipment was previously calibrated with indium, utilizing an empty aluminum pan as inert reference material. First, the oil sample in a sealed pan (≈12 mg) was heated at 80°C during 10 min to eliminate crystallization memory; then the system was cooled to −40°C at a rate of 10°C/min (dynamic section), keeping the oil at this temperature for 30 min (isothermal section). Afterward, the melting thermogram was obtained by heating the system to 80°C at a rate of 5°C/min. From the dynamic crystallization thermogram, the peak temperature in the crystallization exotherm (T_{Cr}) was calculated with the DSC-7 library software. In the same way, the induction time of crystallization (T_i) (i.e., time where the heat capacity of the sample had a significant departure from the baseline) was determined from the isothermal thermogram, and the temperature at the melting peak (T_M) was calculated from the melting thermogram of the oil crystals.

The oxidative stability index (OSI) in the oils and milled pecan kernels was determined at 110°C in an oxidative stability instrument (Omnion Inc., Rockland, MA), utilizing 5 ± 0.2 g of sample. The induction time for oxidation in hours (OSI_{110}) was calculated with the software of the equipment.

As in the chemical analysis, samples of unrefined and refined sesame seed oil as well as samples of extra-virgin olive oil were physicochemically analyzed for comparison purposes.

RESULTS AND DISCUSSION

The present investigation was an observational study in which no variable was controlled. The pecan oils studied were obtained from native species grown under the natural variation of the environmental conditions found in the central region of Mexico.

The general composition and physicochemical properties of the pecan kernels and pecan oils investigated are shown in Table 1. The main characteristic of pecan kernels from *C. illinoensis* is the high lipid content with an elevated concentration of oleic acid. These observations are in agreement with the results obtained in other studies (6,7,12,13). Table 1 presents the results disregarding the compositional differences existing among pecan kernels collected from different trees within the same state (i.e., Queretaro, Guanajuato, and San Luis Potosí). Nevertheless, a low standard error was found for oil concentration within each state (percentage of coefficient of variation, CV%, of 2.2, 6.6, and 3.1 for Guanajuato, Queretaro, and San Luis Potosí, respectively) which was an indication of the similarity in growing conditions for the pecan trees selected, and very probably also in the extent of maturity of the kernels since pecans do not accumulate lipids

TABLE 1
Composition (% w/w) and Physiocochemical Properties of Kernel and Pecan Oil[a]

	State of central region of Mexico		
	Guanajuato (n = 7)	Queretaro (n = 12)	San Luis Potosí (n = 3)
Kernel			
Water	3.63 ± 0.28	3.17 ± 0.13	2.86 ± 0.22
Protein[b]	8.66 ± 0.17	8.49 ± 0.15	8.15 ± 0.24
Lipid[b]	72.66 ± 0.58	72.71 ± 1.37	76.49 ± 1.38
OSI_{110} (h)	25.88 ± 2.17	25.78 ± 1.99	25.63 ± 2.37
Oil			
Palmitic	5.12 ± 0.15	5.11 ± 0.15	5.36 ± 0.14
Stearic	2.87 ± 0.23	2.77 ± 0.09	2.51 ± 0.02
Oleic	67.47 ± 2.02	65.65 ± 1.68	60.55 ± 2.73
Linoleic	22.10 ± 1.81	23.61 ± 1.45	27.51 ± 1.32
Linolenic	2.14 ± 0.15	2.31 ± 0.11	2.19 ± 0.22
Tocopherol (ppm):			
α-	83.21 ± 3.53	89.98 ± 9.32	N.D.[b]
δ-	31.40 ± 14.85	45.13 ± 14.10	78.77 ± 4.31
γ-	160.27 ± 38.74	182.81 ± 17.60	109.59 ± 34.58
OSI_{110} (h)	6.67 ± 0.59	7.21 ± 0.53	6.13 ± 0.15
T_{Cr} (°C)[d]	–33.79 ± 0.75	–34.51 ± 0.38	–34.19 ± 0.41
T_M (°C)[e]	–19.37 ± 1.84	–23.54 ± 1.81	–29.56 ± 1.80
T_i (min)[f]	15.97 ± 2.58[g]	9.45 ± 1.29[g]	—[h]
η_{10} (cps)[i]	105.36 ± 3.49	110.65 ± 2.52	102.18 ± 3.48
η_{20} (cps)	63.65 ± 3.43	65.91 ± 1.30	61.67 ± 2.70
η_{30} (cps)	35.01 ± 2.10	42.70 ± 2.73	39.59 ± 2.45

[a]The results (mean ± the standard error) are summarized per state of central region of Mexico involved in this study.
[b]Determination on dry basis.
[c]Not detected.
[d]Peak temperature in the crystallization exotherm.
[e]Peak temperature in the melting endotherm.
[f]Induction time for isothermal crystallization at –40°C.
[g]Some oils did not show a detectable T_i under the time–temperature conditions utilized.
[h]Not detected under the time–temperature conditions utilized.
[i]Viscosity at 10, 20, and 30°C. OSI, oxidative stability index.

at a constant rate during growth (14). In the same way the standard error for protein concentration in the pecan kernels (Table 1) provided CV% of and 9.8, 6.1, and 5.1 for Guanajuato, Queretaro, and San Luis Potosí, respectively. In contrast, water concentration presented a higher degree of variability (CV% of 8.7, 14.21, and 13.32 for Guanajuato, Queretaro, and San Luis Potosí, respectively) which pointed out the susceptibility of this parameter to different growing conditions.

In some vegetables, like soybeans, an inverse linear relationship between protein and oil concentration was observed (15,16). However, the magnitude and type of this relationship (i.e., linear or curvilinear) vary with geographic location and interval of temperature studied during the oil development in soybean seed (15–18). Additionally, in these studies particular cultivars were grown under controlled conditions to minimize the effect of variation of reproductive development in seed composition. The control of such variables was not established in the present investigation, since native varieties of pecans were analyzed. Consequently, the relationship between the concentration of protein and oil for all 22 samples was negligible and not statistically significant (r = 0.242, P = 0.28). The same results were obtained when the statistical analysis was performed, partitioning the data by state of pecan tree origin. On the other hand, the tree age showed a significant quadratic regression on the concentration of lipids in the pecan kernel (r = 0.65, $P < 0.008$), and a less strong one with water concentration (r = 0.535, P = 0.048) (Fig. 1). These relationships indicated that lipid accumulation in pecan kernel decreases in a curvilinear fashion as a function of the tree age, while water concentration increases. Different soil composition among the areas where the pecan trees were growing and drying loss associated to date of harvest were excluded as variables affecting the relationship of lipid and water content in pecan kernels with tree age. Heaton *et al.* (19) determined that total lipid accumulated by 45 varieties grown in six different locations was not significantly influenced by the level of fertilizer applied for 9 yr previous to the analysis. Additionally, the pecan kernels were collected in the same harvest period and stored under the same conditions. On the other hand, protein concentration in the Mexican varieties was higher than the variety utilized in the United States Department of Agriculture report in 1984 for dry-roasted pecans (7.97%) and raw pecans (7.75%) (20). In contrast with lipid and water, the concentration of protein in pecan kernels was not significantly affected by tree age (r = 0.385, P = 0.237).

The OSI_{110} of the pecan kernel was higher than the OSI_{110} of the oil (Table 1). Part of this difference was accounted for by the different lipid concentration in each system (i.e., 70–80% in the kernel and >99% in the oil), and the lower susceptibility to oxidation of the lipid within a solid matrix (e.g.,

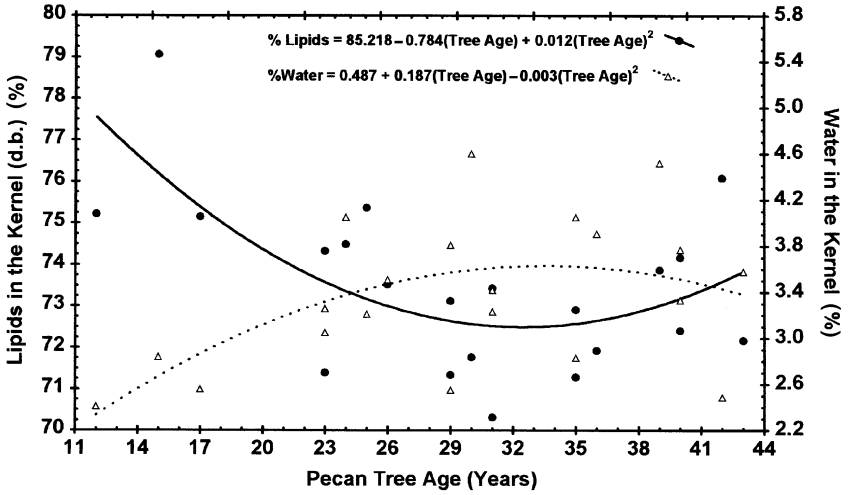

FIG. 1. Quadratic regression of pecan tree age on lipid (d.b., dry basis) and moisture content in pecan kernels. The respective equations are indicated.

pecan kernels) than in the liquid phase (e.g., pecan oil). The OSI_{110} and fatty acid composition of both kernel and oil did not show significant relationships with α-, δ-, or γ-tocopherol (data not shown). As for pecan varieties Stuart, Desirable and Schley (9), γ-tocopherol was the predominant homolog in Mexican pecans (Table 1). However, the concentration of α- and δ-tocopherol was substantially higher than in the Mexican varieties (Table 1). It is well-known that tocopherol content is strongly influenced by varietal differences, different stages of maturity of the vegetable at harvest, as well as geographic and climatic conditions under which the plant is grown (21,22). Also tocopherol synthesis is affected by temperature, but the results are contradictory in establishing the extent of the temperature effect on tocopherol synthesis as well as on its relationship to fatty acid desaturation by the vegetable tissues (22,23). Five out of the 22 pecan oils analyzed showed similar or higher OSI_{110} values (8.76–10.78 h) than extra-virgin olive oil and unrefined sesame oil (Table 2), and even similar stability as that of the cold-pressed sesame oil reported in the literature (OSI_{110} = 9.05 h) (24). It is important to point out that a higher degree of unsaturation, mainly due to the concentration of linoleic and linolenic acids, was present in pecan oil (Table 1) than in olive and sesame oils (Table 2). Olive oil and sesame oil contain, in addition to tocopherols, other natural antioxidants like tyrosol, hydroxytyrosol, and caffeic acid, in the case of olive oil (25), and sesamol, sesamin, and sesamolin, in sesame oil (26). Then, the fatty acid and tocopherol compositions do not completely explain the high oxidative stability observed in some pecan oils (and kernels). This might indicate the presence of natural antioxidants in addition to the tocopherols in these particular pecan oils, which still retain some activity at the temperature of the OSI analysis.

The OSI_{110} of the pecan kernel (Table 1) showed a limited

TABLE 2
Fatty Acid Profile (%w/w), OSI_{110}, Viscosity, and Crystallization/Melting Parameters for Unrefined Sesame (*Sesamum indicum*) Oil (USO), Refined Sesame Oil (RSO), and Extra-Virgin Olive Oil (OO)

	Oil source		
	USO	RSO	OO
Fatty acids			
Palmitic	9.60 ± 0.01	9.15 ± 0.04	9.88 ± 0.04
Stearic	5.16 ± 0.01	5.01 ± 0.04	3.72 ± 0.01
Oleic	39.26 ± 0.18	37.26 ± 0.01	76.27 ± 0.16
Linoleic	45.28 ± 0.17	46.79 ± 0.01	7.72 ± 0.01
Linolenic	N.D.[a]	N.D.	N.D.
Others	1.12 ± 0.37	1.77 ± 0.01	2.40 ± 0.11
OSI_{110}	6.23 ± 0.04	3.60 ± 0.07	8.50 ± 0.07
η_{10} (cps)	123.10 ± 0.01	121.70 ± 0.01	149.30 ± 0.01
η_{20} (cps)	73.80 ± 0.01	74.20 ± 0.00	87.30 ± 0.00
η_{30} (cps)	47.60 ± 0.00	47.60 ± 0.00	55.30 ± 0.00
T_{Cr} (°C)	–25.78	–19.19	–23.42
T_M (°C)	–20.33	–19.02	–16.69
T_i (min)	—	—	2.99

[a]The results are shown in mean ± standard error. The remaining notes are as in Table 1.

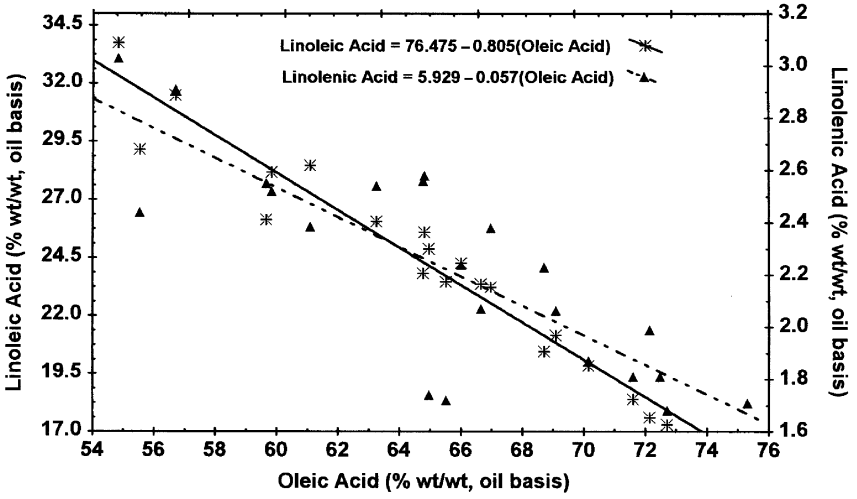

FIG. 2. Linear regression of oleic acid concentration on the concentration of linoleic and linolenic acids in pecan oil. The respective equations are indicated.

but significant inverse linear relationship with the water content of the kernel (slope = –4.09, $r = 0.436$, $P = 0.042$). The slope of this relationship indicated that a hydrolytic effect of water combined with the heat action on triacylglycerides might release fatty acids which are more susceptible to oxidation than when esterified to glycerol (27). Pecans are a semiperishable product, and a high moisture content accelerates chemical reactions associated with kernel discoloration (9,28). Since color is an important quality parameter that determines market price, an appropriate moisture level within 4–5% was recommended to maintain color quality in intact pecan kernels (28). The mean moisture content (±standard error) of the pecan kernels collected in each state of the central region of Mexico was within the recommended moisture interval. It is important to point out that the OSI_{110} was measured in milled pecan kernels which made the pecan components highly available to react. Thus, in intact pecan kernels, higher oxidative stability than the one reported here for milled kernels is expected.

Significant inverse linear relationships were observed between linoleic ($r = 0.976$, $P < 0.0001$) and linolenic acids ($r = 0.820$, $P < 0.0001$) with oleic acid (Fig. 2). Thus, an approximate 1% (w/w) increase in the concentration of oleic acid in the pecan oil occurs parallel with a decrease in the concentration of linoleic and linolenic fatty acids of the order of 0.805 and 0.057%, respectively. Consequently, the increase in the concentration of oleic acid resulted in an overall decrease in the unsaturation degree of the pecan oil and, subsequently, the oil was less susceptible to oxidation (i.e., a higher OSI_{110}). The magnitude of the slopes of the regressions of oleic (slope = 0.178, $r = 0.64$, $P = 0.0014$), linoleic (slope = –0.214, $r = 0.64$, $P = 0.0013$), and linolenic (slope = –1.829, $r = 0.45$, $P = 0.034$) fatty acids with the OSI_{110} of the oil demonstrated that linolenic acid had a higher prooxidant activity than linoleic acid, an effect obviously associated with its higher degree of unsaturation (18:3) in comparison with linoleic acid (18:2).

The association among the concentrations of oleic, linoleic, and linolenic acids in the pecan oil investigated is the result of the biosynthetic pathway for fatty acids in vegetable cells, which is performed in both the plastid and the endoplasmic reticulum. In most crops the primary fatty acid synthesized is palmitic acid (16:0), which is partially elongated to stearic acid (18:0) by the isozyme β-ketoacyl-acyl carrier protein synthetase. Furthermore, the 18:0 can then be desaturated to oleic acid (18:1) by a desaturase which locates the unsaturation between the carbons 9 and 10 (29). All these reactions occur within the plastid, and since higher plants do not have 9-desaturase in the cytoplasm or endoplasmic reticulum once 18:0 leaves the plastid, it is not further desaturated (30). Further desaturations of 18:1 in carbons ω6 and ω3 occur by desaturases present in the endoplasmic reticulum, synthesizing the linoleic (18:2) and linolenic (18:3) fatty acids, respectively. In consequence, the biosynthesis of linoleic and linolenic acids utilizes the oleic acid as primary substrate, decreasing its concentration as they are produced (Fig. 2). However, the biosynthesis of linoleic and linolenic acids from oleic acid is at least partly associated with climate temperature during vegetable growth, i.e., a high climate temperature is strongly associated with greater percentages of oleic acid and lesser percentages of linoleic and linolenic acids (16,31,32). For instance, in soybean seeds it was demonstrated that oleoyl desaturase, the enzyme responsible for the conversion of oleic acid to linoleic, and linoleoyl desaturase, involved in the conversion of linoleic to linolenic acid, have negligible activities in seeds grown in culture at 35°C as compared to the ones grown at 20°C (33). Our data indicated that the production of these two fatty acids in pecans is interrelated since a direct linear relationship between linoleic and linolenic acids was obtained ($r = 0.836$, $P < 0.0001$) (Eq. 1). However, climate

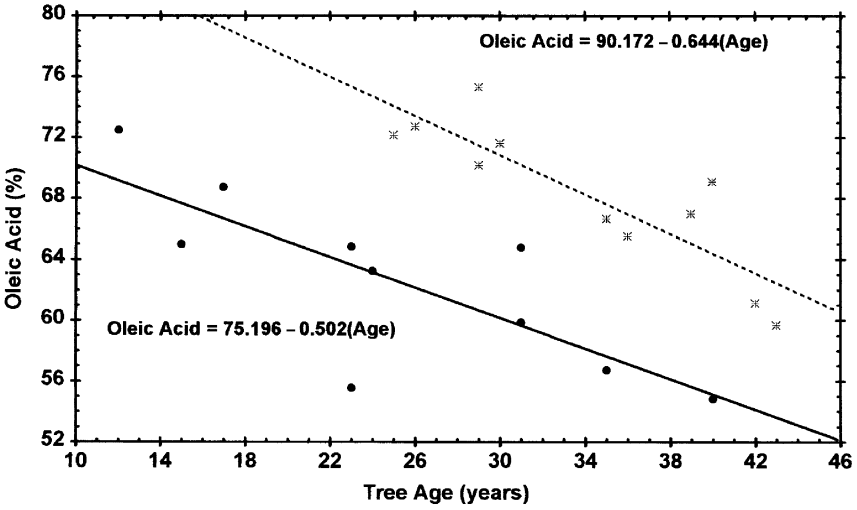

FIG. 3. Linear regression of pecan tree age on oleic acid concentration. The dotted line represents the regression for the high-oleic pecan oils (n = 11), while the solid line represents the regression for the low-oleic pecan oils (n = 10).

$$\text{linolenic acid} = 0.581 + 0.069\ (\text{linoleic acid}) \quad [1]$$

temperature history during the growing season of the pecans utilized in this investigation was not available, and the effect of climate temperature on the concentration of oleic, linoleic, and linolenic fatty acids was not determined.

On the other hand, the age of the tree formed a significant linear regression ($P < 0.005$) with oleic, linoleic, and linolenic acid concentrations in the pecan oil (Figs. 3–5). This regression analysis divided the pecan trees into two families, one with a mean oleic acid concentration of 68.3% (w/w), identified as high-oleic pecan oil (HOPO), and the other with a mean oleic acid concentration of 62.6% (w/w) identified as low-oleic pecan oil (LOPO) (Table 3). These results indicated that the biosynthetic association among the concentrations of oleic, linoleic, and linolenic acids in pecan kernels is a function of tree age. As a result, the proportion among these three fatty acids is constantly affected by tree age and subsequently influencing the physicochemical properties of the oil, in spite of the lipid accumulation in the pecan kernel seeming to achieve a constant value after the tree is 30 yr of age (Fig. 1). In the same way, our results indicated that two different varieties of pecans were involved in this study (i.e., HOPO and LOPO, Figs. 3–5). As a result of the relationship in the biosynthesis of linoleic and linolenic acids from oleic acid (Fig. 2), the HOPO group showed a lower degree of unsatu-

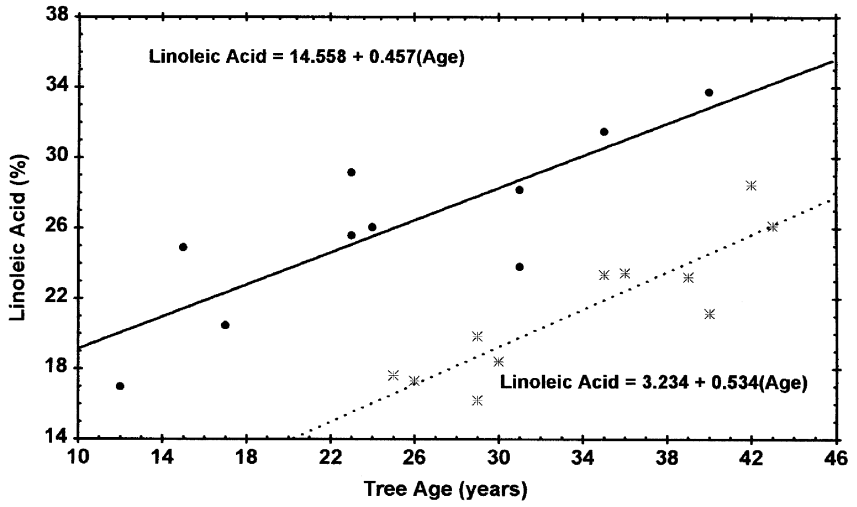

FIG. 4. Linear regression of pecan tree age on linoleic acid concentration. The dotted line represents the regression for the high-oleic pecan oils (n = 11), while the solid line represents the regression for the low-oleic pecan oils (n = 10).

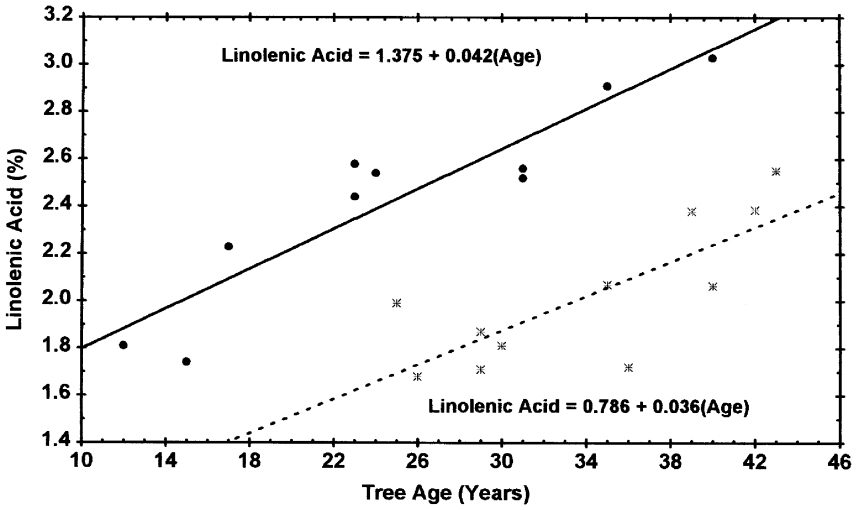

FIG. 5. Linear regression of pecan tree age on linolenic acid concentration. The dotted line represents the regression for the high-oleic pecan oils (n = 11), while the solid line represents the regression for the low-oleic pecan oils (n = 10).

ration than the LOPO group. Consequently, the LOPO group had lower oxidative stability (i.e., lower OSI_{110}) than the HOPO group (Table 3).

As previously mentioned, the OSI_{110} and fatty acid composition of all pecan oils investigated did not show significant relationships with α-, δ-, or γ-tocopherol. This lack of relationship was also observed within the LOPO and HOPO groups. Some authors (22,34) observed a positive correlation between the concentration of particular tocopherols and the degree of unsaturation of vegetable oils. However, we were unable to corroborate such results in the pecan oils investigated. On the other hand, the increase in the concentration of oleic acid resulted in an overall decrease in the unsaturation degree of the pecan oil (Fig. 2), and subsequently in an increase in the η of pecan oil. This effect was independent of the temperature of viscosity measurement. In contrast, an increase in the concentration of linoleic acid (i.e., increasing the unsaturation degree of the oil) decreased the η of the pecan oil. The particular effect of oleic and linoleic acids on η for pecan oil at 30°C (η_{30}) was described by Equations 2 and 3 with regression coefficients of 0.49 and 0.52, respectively ($P < 0.025$). In general, the higher degree of unsatura-

TABLE 3
Compositional (% w/w) and Physicochemical Characteristics of the High-Oleic (HOPO) and Low-Oleic (LOPO) Pecan Oil[a]

	LOPO (n = 10)[a]			HOPO (n = 11)		
	Interval	Mean	S.E.	Interval	Mean	S.E.
Palmitic	4.77–5.89	5.40	0.07	4.26–5.52	4.92	0.11
Stearic	2.38–3.45	2.77	0.05	2.23–3.27	2.76	0.10
Oleic	54.83–72.48	62.60	0.84	59.67–75.31	68.27	1.47
Linoleic	16.99–33.75	26.04	0.77	16.21–28.46	21.38	1.18
Linolenic	1.74–3.03	2.44	0.06	1.68–2.55	2.02	0.09
Tocopherol (ppm):						
α-	70.03–85.09	78.06	0.66	71.71–122.40	84.55	4.40
δ-	0.00–90.37	31.22	12.85	0.00–112.17	55.81	13.95
γ-	40.74–289.08	202.92	27.28	60.32–260.29	182.66	18.59
OSI_{110} (h)	3.93–8.76	6.58	0.54	4.23–10.78	6.88	0.51
T_{Cr} (°C)	–35.66–(–30.82)	–34.01	0.50	–37.24–(–32.65)	–34.66	0.47
T_M (°C)	–32.17–(–14.90)	–25.65	1.87	–33.15–(–15.24)	–20.44	1.79
T_i (min)	13.40–14.88[b]	14.14	0.74	3.55–27.02[c]	12.40	2.36
η_{10} (cps)	95.00–120.00	104.90	2.26	94.00–123.00	109.36	3.11
η_{20} (cps)	58.00–72.00	63.50	1.26	56.00–73.00	65.06	1.76
η_{30} (cps)	36.00–46.00	40.60	0.87	37.00–47.00	42.09	1.07

[a]The HOPO and LOPO are referred to the groups of oil resulting from the linear regression of tree age on oleic, linoleic, and linolenic acid concentrations in the pecan oil, indicated in Figures 3–5 as dotted line and solid line, respectively; n represents the number of pecan trees included in each group. The rest of the legend is in Table 1.
[b]Only two of the pecan oils within this group had an induction time for crystallization.
[c]Only two of the pecans oils within this group did not have an induction time for crystallization.

tion of pecan oil (Table 1) provides it with the lowest viscosity at the three temperatures investigated, in comparison to sesame and olive oils (Table 2).

$$\eta_{30} = 23.624 + 0.2732 \text{ (oleic acid)} \quad [2]$$
$$\eta_{30} = 49.733 - 0.3465 \text{ (linoleic acid)} \quad [3]$$

The crystallization thermograms showed a single peak that in all cases had $T_{Cr} < -30°C$ (Table 1 and 3). This parameter was not a function of the fatty acid composition. In contrast, an effect associated with the overall unsaturation degree of pecan oil was observed during the isothermal crystallization at −40°C. Thus, during the isothermal crystallization, after the dynamic section of the thermogram, the HOPO group (i.e., oils with lower degree of unsaturation) developed further crystallization with $T_i > 3.0$ min. In contrast the oils, mainly the ones included in the LOPO group (i.e., oils with the higher degree of unsaturation), did not develop additional crystallization within the time interval utilized in the isothermal crystallization (i.e., 30 min) (Table 3).

As in the case of viscosity, the T_M calculated from the melting thermograms of the pecan oils (data not shown) showed significant correlations with the fatty acid composition (Eqs. 4–7). The magnitude of the correlation coefficient in Equations 4–7 confirmed that the most important fatty acids involved in determining the physicochemical characteristics of pecan oil are oleic, linoleic, and linolenic.

$$T_M = -49.703 + 9.665 \text{ (stearic acid)} \quad r = 0.492, P < 0.0001 \quad [4]$$
$$T_M = -82.179 + 0.901 \text{ (oleic acid)} \quad r = 0.838, P < 0.00001 \quad [5]$$
$$T_M = 3.249 - 1.113 \text{ (linoleic acid)} \quad r = 0.807, P < 0.0001 \quad [6]$$
$$T_M = 0.695 - 10.703 \text{ (linolenic acid)} \quad r = 0.686, P < 0.0004 \quad [7]$$

On the other hand, sesame oil, unrefined and refined, and olive oil exhibited T_{Cr} of −25.78, −19.19, and −23.42°C, respectively, and sesame oil did not show isothermal crystallization at −40°C, whereas olive oil had a single peak (not shown) with T_i of 2.99 min. In the same way, the T_M values for sesame oil and olive oil (Table 2) were similar to or higher than those for pecan oils (Tables 1 and 3). These results pointed out the higher unsaturation degree of the pecan oils investigated in contrast to sesame and olive oils, opening up the possibility of utilizing pecan oil as a speciality oil in salad dressings since triacylglycerol crystallization is not developed under ambient and refrigeration temperatures. Although all native oils studied showed a significant concentration of oleic acid, a particular group of native pecan trees (i.e., HOPO group) presented a fatty acid composition with the nutritional appeal that consumers demand (i.e., very high oleic acid), and some of them with excellent natural oxidative stability (i.e., OSI_{110} values between of 8.48 and 10.78 h). The effect of variables such as the stage of maturity and pecan tree age on pecan yield and kernel yield, as well as the effect of climate conditions (i.e., temperature), on fatty acid, tocopherol, and tocotrienol composition must be investigated.

ACKNOWLEDGMENTS

The present work was supported by CONACYT through the grant #485100-5-0658PB and by CONACYT-SHIGO through the grant ALIM-33.

REFERENCES

1. Mattson, F.H., and M.S. Grundy, Comparison of Effect of Dietary Saturated, Monounsaturated and Polyunsaturated Fatty Acids on Plasma Lipids and Lipoproteins in Man, *J. Lipid Res. 26*:194–202 (1985).
2. Haumann, B.F., Olive Oil, Mediterranean Product Consumed Worldwide, *INFORM 7*:890–903 (1996).
3. Hegsted, D.M., Dietary Fatty Acids, Serum Cholesterol and Coronary Heart Disease, in *Health Effects of Dietary Fatty Acids*, edited by G.J. Nelson, American Oil Chemists' Society, Champaign, IL, 1990, pp. 50–68.
4. Reddy, B.S., Omega-3 Fatty Acids as Anticancer Agents, *Ibid.*, pp. 157–166.
5. Beuchat, L.A., and R.E. Worthington, Fatty Acids Composition of Tree Nut Oils, *J. Food Technol. 13*:355–358 (1978).
6. Santerre, C.R., Pecan Composition, in *Pecan Technology*, edited by C.R. Santerre, Chapman and Hall, New York, 1994, pp. 98–110.
7. Senter, S.D., and R.V. Horvat, Lipids of Pecan Nutmeats, *J. Food Sci. 41*:1201–1203 (1976).
8. Senter, S.D., and R.V. Horvat, Minor Fatty Acids from Pecan Kernel Lipids, *Ibid. 43*:1614–1615 (1978).
9. Yao, F., G. Dull, and R. Eitenmiller, Tocopherol Quantification by HPLC in Pecans and Relationship to Kernel Quality During Storage, *Ibid. 57*:1194–1197 (1992).
10. Thompson, T.E., L.J. Grauke, and E.F. Young, Jr., Pecan Kernel Color: Standards Using the Munsell Color Notation, *J. Am. Soc. Hort. Sci. 121*:548–553 (1996).
11. Dibildox-Alvarado, E., and J.F. Toro-Vazquez, Isothermal Crystallization of Tripalmitin in Sesame Oil, *J. Am. Oil Chem. Soc. 74*:69–76 (1997).
12. Braddock, J.C., C.A. Sims, and S.F. O'Keefe, Flavor and Oxidative Stability of Roasted High Oleic Acid Peanuts, *J. Food Sci. 60*:489–493 (1995).
13. Heaton, E.K., R.E. Worthington, and A.L. Shewfelt, Pecan Nut Quality. Effect of Time of Harvest on Composition, Sensory and Quality Characteristics, *Ibid. 40*:1260–1263 (1975).
14. Wood, B.W., and J.L. Mcmeans, Carbohydrates and Fatty Acids in Developing Pecan Fruit, *J. Am. Soc. Hort. Sci. 107*:47–50 (1982).
15. Dornbos, D.L., Jr., and R.E. Mullen, Soybean Seed Protein and Oil Contents and Fatty Acid Composition Adjustments by Drought and Temperature, *J. Am. Oil Chem. Soc. 69*:228–231 (1992).
16. Gibson, L.R., and R.E. Mullen, Soybean Seed Composition Under High Day and Night Growth Temperatures, *Ibid. 73*: 733–737 (1996).
17. Wolf, R.B., J.F. Canvins, R. Kleiman, and L.T. Black, Effect of Temperature on Soybean Seed Constituents: Oil, Protein, Moisture, Fatty Acids, Amino Acids and Sugars, *Ibid. 59*:230–232 (1982).
18. Howell, R.W., and J.L. Cartter, Physiological Factors Affecting Composition of Soybeans: I. Response of Oil and Other Constituents of Soybean to Temperature Under Controlled Conditions, *Agron. J. 50*:664–667 (1958).
19. Heaton, E.K., J.E. Marion, and J.G. Woodroof, Pecan Oil Is Highly Unsaturated, *Peanut J. Nut World 45*(12):36–38 (1966).
20. United States Department of Agriculture, Composition of Foods: Nut and Seed Products, *Agriculture Handbook No. 8–12*,

U.S. Government Printing Office, Washington, DC, 1984.
21. Bauernfeind, J.C., and I.D. Desai, The Tocopherol Content of Food and Influencing Factors, *CRC Crit. Rev. Food Sci. 8*:337–382 (1977).
22. Vlahakis, Ch., and J. Hazebroek, Phytosterols and Tocopherols in Soybean Seeds: Effect of Temperature, presented at the 89th American Oil Chemists' Society Annual Meeting, Chicago, 1998.
23. Marquard, von R., Investigation on the Influence of Genotype and Location on Tocopherol Content on the Oil from Different Oil Crops, *Fat Sci. Technol. 92*:452–455 (1990).
24. De Panfilis, F., T. Gallina Toschi, and G. Lercker, Quality Control for Cold-Pressed Oils, *INFORM 9*:212–221 (1998).
25. Ranalli, A., and G. De Mattia, Characterization of Olive Oil Produced with a New Enzyme Processing Aid, *J. Am. Oil Chem. Soc. 74*:1105–1113 (1997).
26. Kamal-Eldin, A., and L.Å. Appelqvist, Variations in the Composition of Sterols, Tocopherols and Lignans in Seed Oils from Four *Sesamum* Species, *Ibid. 71*:149–156 (1994).
27. Miyashita, K., and T. Takagi, Study on the Oxidative Rate and Prooxidant Activity of Free Fatty Acids, *Ibid. 63*:1380–1384 (1986).
28. Senter, S.D., and W.R. Forbus, Jr., Leucoanthocyanidin Oxidation in Pecan Kernels: Relation to Discoloration and Kernel Quality, *J. Food Sci. 43*:128–134 (1978).
29. McKeon, T.A., and P.K. Stumpf, Purification and Characterization of the Stearoyl-acp Desaturase and the Acyl-acp Thioesterase from Maturing Seed of Safflower, *J. Biol. Chem. 257*:1211–1217 (1982).
30. Somerville, C.R., Prospects for Genetic Modification of the Composition of Edible Oils from Higher Plants, *Food Biotechnol. 5*:217–288 (1991).
31. Carver, B.F., J.W. Burton, T.E. Carter, Jr., and R.F. Wilson, Response to Environmental Variation of Soybean Lines Selected for Altered Unsaturated Fatty Acid Composition, *Crop Sci. 26*: 1176–1191 (1986).
32. Wilcox, J.R., and J.F. Cavins, Normal and Low-Linolenic Acid Soybean Strains: Response to Planting Date, *Ibid. 32*:1248–1251 (1992).
33. Cheesbrough, T.M., Changes in the Enzymes for Fatty Acid Synthesis and Desaturation During Acclimation of Developing Soybean Seed to Altered Growth Temperature, *Plant Physiol. 90*:760–764 (1989).
34. Kamal-Eldin, A., and R. Andersson, A Multivariate Study of the Correlation Between Tocopherol Content and Fatty Acid Composition in Vegetable Oils, *J. Am. Oil Chem. Soc. 74*:375–379 (1997).

[Received October 22, 1998; accepted March 29, 1999]