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Physicochemical and Sensory Quality of Crude Brazilian Pecan Nut Oil during Storage

Tatiana Oro · Helena Maria André Bolini · Daniel Barrera Arellano · Jane Mara Block

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Abstract The physicochemical and sensory characteristics of pecan nut [*Carya illinoensis* (Wangenh.) K. Koch] oil obtained by cold pressing and stored at room temperature (average of 22.5 °C) in amber glass flasks for 120 days were determined. The pecan nut oil had an iodine value of 98.4; a refraction index at 40 °C of 1.469; saponification value of 184.3 g/100 g; a peroxide value (PV) of 0.55 mequiv O_2/kg; an Oil Stability Index (OSI), i.e. an oxidative stability of 9.8 h and an acid value of 0.13 mg KOH/g. During the storage period, changes in the peroxide value (PV), specific extinction (SE) at 232 nm, acid value, and color of the oil were observed but, after 120 days, the oil still had adequate quality characteristics according to the Brazilian legislation for crude oils. Sensory analysis (descriptive profile and acceptance) showed that the sensory characteristics of pecan nut oil were unaltered for up to 60 days of storage. After 90 days, there was a significant increase in oxidized taste and bitterness, with a reduction in nut taste and acceptability of the oil ($p \leq 0.05$). After 120 days of storage, sensory changes became more pronounced and were unacceptable. It was concluded that crude pecan nut oil stored in amber glass flasks at room temperature had a shelf life of 90 days.

Keywords Pecan nut oil · Oxidation · Sensory analysis · Shelf life

T. Oro · J. M. Block (✉)
CCA, Departamento de Ciência e Tecnologia de Alimentos, UFSC, Rodovia Admar Gonzaga, 1346, Caixa Postal: 88034-000, Itacorubi, Florianópolis, SC, Brazil
e-mail: jmblock@cca.ufsc.br

H. M. A. Bolini
Department of Food and Nutrition, Faculty of Food Engineering, University of Campinas, UNICAMP, Campinas, SP, Brazil

D. B. Arellano
Department of Food Technology, Faculty of Food Engineering, University of Campinas, UNICAMP, Campinas, SP, Brazil

Introduction

Nuts have been considered an important component of the Mediterranean diet. Epidemiologic studies indicate that nut consumption may have cardioprotective effects, as a function of their low saturated fatty acid content and high levels of monounsaturated and polyunsaturated fatty acids, phytosterols and tocols [1]. The pecan nut [*Carya illinoensis* (Wangenh.) K. Koch], due to its agreeable flavor and texture, is much appreciated and utilized in a wide variety of foods, including bakery and confectionery products, desserts, ice-creams and snacks (raw, toasted or salted). The nuts are commercialized with or without shells, in halves, in pieces of different sizes or even as flour [2].

In Brazil, pecan production is still very modest, but with the increasing consumption of nuts, the market is expanding and creating space for the production of the oil and of byproducts such as the shell and defatted flour. In many parts of the world, such as the Middle East and Asia, nut trees are grown for the use of the oil, especially as gourmet oil, but also to produce essential oils, cosmetic products, and medicines [1, 3, 4].

After harvesting, the nuts are exposed to a series of environmental conditions that can affect their physicochemical and sensory quality [5]. Pecan nut oil is known as a gourmet oil due to its unique sensory characteristics. The

lipid content of pecan nut ranges from 60 to 75%, depending on the variety, location, year of production, soil composition, and harvest period [6]. Unsaturated fatty acids represent approximately 92% of the triacylglyceride composition, with oleic acid account for 60–70% of the fatty acids [5, 7]. There are few studies regarding pecan oil stability and changes in quality during storage. Thus, the objective of this work was to study the changes in physicochemical and sensory quality of crude pecan nut oil during storage.

Materials and Methods

Raw Material

Pecan nuts composed of a mixture of different varieties: Barton (approximately 50%), Shoshone, Shawnee, Choctaw and Cape Fear from the 2006 harvest were kindly donated by Divinut Indústria de Nozes Ltda. (Cachoeira do Sul—RS, Brazil).

Methods

Production and Storage of Crude Pecan Nut Oil

Pecan nut oil was produced by cold pressing of 4 kg nuts from three different batches, amounting to 12 kg of pressed nuts, using a Te 098 model hydraulic press (Tecnal, Ourinhos, SP, Brazil). Pecan nuts were pressed in small quantities (200 g) and the oil obtained (90 mL) was filtered and stored in amber glass flasks that were sealed with a transparent plastic top and a dark screw top. The oil was stored in a room with an average temperature and relative humidity of 22.5 °C and 55.6%, respectively, for a period of 120 days. A control oil was stored under freezing during the storage time available for comparison.

Characterization and Quality Evaluation of Crude Pecan Nut Oil during Storage

The oil was characterized using official American Oil Chemists' Society [8] methodology that included acid value (Ca 5a-40); moisture and volatile compound content (Ca 2c-25); peroxide value (Cd 8-53); saponification value (Cd 3-25); iodine value (Cd 1-25); Lovibond color (Cc 13b-45); specific extinction (Ch 5-91); unsaponifiable matter (Ca 6a-40); refraction index (Cc 7-25) and oxidative stability (Oil Stability Index—OSI) at 110 °C (Cd 12b-92). Moisture and volatile compounds; peroxide value; acid value; specific extinction and Lovibond color [8] were also evaluated every 15 days over the 120-day period. Oil characterization and quality evaluation analyses were performed in duplicate.

Evaluation of Sensory Quality of Crude Pecan Nut Oil during Storage: Quantitative Descriptive Analysis

Sensory analysis of the oil was carried out every month during the 120 days through quantitative descriptive analysis by a team of trained panelists. Training for the formation of sensory memory and equalization among panelists was carried out by direct contact of the individuals with the references. Each of the descriptive terms was represented by two references, which defined the extreme points of the scale: to the left, minimum intensity, and to the right, maximum intensity. The references used in the experiment are described in Table 1.

Judges generated six descriptor terms with definitions and references through Kelly's Repertory Grid Method. References were determined by a consensus of all the judges and then panelists were further trained on the product attributes using identified references. Analysis of variance (ANOVA) for each panelist and each attribute was employed and 10 panelists out of 18 were chosen for participation according to their discriminating capability ($p < 0.30$) and repeatability ($p > 0.05$), while an individual consensus was also considered [9]. Panelist performance was evaluated through descriptive analysis of four samples of Brazilian pecan nut oil. The judges analyzed oxidation levels for each sample four times in monadic presentations. Four samples were produced for this purpose by heating at 50 °C (0, 1, 2, and 3 h) in an electric heater to create the levels of oxidation used in the training.

The trained and selected panelists participated in the sensory analysis of each Brazilian pecan nut oil sample during the storage period, at 0, 30, 60, 90 and 120 days. At each time point, the panelists analyzed each sample in monadic presentations, using a 9-cm non-structured linear scale. Each sample was evaluated by each panelist four times.

Sensory Analysis of Pecan Nut Oil through Consumer Acceptance Analysis

The affective tests involved the participation of 120 consumers who liked nuts, and the tests were conducted in laboratory booths. The consumers received the samples monadically, in glasses coded with three-digit numbers and covered with a watch glass, which was removed at the moment of the test. The participants were asked to smell first and then taste the samples. They sipped the Brazilian pecan nut oil samples directly from the glasses, and marked their acceptance in relation to aroma, taste and global impression, using a 9-cm non-structured linear hedonic scale with the anchor points "dislike extremely" on the left and "like extremely" on the right.

Table 1 Descriptive terms, definitions and references used for the descriptive sensory training

Descriptive term	Definition	References for the extremes of the intensity scales
Apparent homogeneity	Homogeneity of the oil detected visually	*Low*: RBD Canola oil, Cargill Agricola SA, 10 days after production date, with 5% added sodium chloride
		High: RBD Canola oil, Cargill Agricola SA, 10 days after production date
Pecan nut aroma	Characteristic aroma of fragmented pecan nut, at the beginning of its shelf life, very similar to traditional nut	*Weak*: 5 g fragmented pecan nut in 100 mL distilled water
		Strong: 20 g pecan nut in pieces, at the beginning of its shelf life
Vegetable oil aroma	Characteristic aroma of commercial vegetable oils	*Weak*: RBD Soybean oil, Cargill Agricola SA, 10 days after production date, with 60% added commercial mineral oil
		Strong: RBD Soybean oil, Cargill Agricola SA, 10 days after production date
Pecan nut taste	Characteristic taste of fragmented pecan nut, at the beginning of its shelf life, very similar to traditional nut	*Weak*: mixture of 5 g pecan nut, at the beginning of its shelf life, ground with 30 g French bread crumb
		Strong: 20 g pecan nut in pieces, at the beginning of its shelf life
Oxidized taste	Taste associated with oil oxidation, rancid	*None*: Distilled water
		Strong: RBD Soybean oil, Cargill Agricola SA, in clear PET flask, left for 10 hours in sunlight
Bitterness	Characteristic taste of caffeine solution	*None*: RBD Soybean oil, Cargill Agricola SA, 10 days after production date
		Strong: RBD Soybean oil, Cargill Agricola SA, 10 days after production date with 0.025% added caffeine p.a. Merck

Statistical Analysis

Statistical analyses were carried out using the Statistica program, version 6.0, at a level of significance of $p < 0.05$. Simple linear correlation analysis was used for all the variables analyzed for the treatments during the period studied. Pearson's correlation was used to verify dependence between the variables.

Data obtained from the sensory analysis of pecan nut oil were submitted to ANOVA, the Tukey test and principal component analysis. Simple linear correlation analysis was used to determine the correlation between the trained panel sensory profile and consumer acceptance [10]. These statistical analyses were carried out using the SAS program, v. 8.12 [11].

Table 2 Physicochemical characterization of crude pecan nut oil

Physicochemical characteristics	Value
Peroxide value (mequiv O_2/kg oil)	0.55 ± 0.07
Acid value (mg KOH/g oil)	0.13 ± 0.01
Moisture and volatile compounds (%)	0.05 ± 0.02
Iodine value (Wijs)	98.4 ± 0.86
Refraction index (40 °C)	1.469 ± 0.011
Saponification value (g/100 g)	184.3 ± 1.8
Unsaponifiable matter (g/100 g)	1.99 ± 0.04
Lovibond Color (5 ¼″ cuvette)	20.0 Y/3.0 R[a]
Oxidative Stability (OSI at 110 °C) (h)	9.8 ± 0.4

[a] *Y* yellow, *R* red

Results and Discussion

Physicochemical Characteristics and Oxidative Stability of Pecan Nut Oil

Physicochemical characteristics of crude pecan nut oil are shown in Table 2. Peroxide (0.55 mequiv O_2/kg) and acid (0.13 mg KOH/g) values are within the limits (i.e. 15 meq O_2/kg and 4.0 mg KOH/g, respectively) established by the National Health Surveillance Agency (ANVISA) of Brazil [12] for cold-pressed non-refined oils. The iodine value (IV) of 98.4 and the saponification value (SV) of 184.3 g/100 g were slightly lower than the values (i.e. 100–106 for IV and 190–198 g/100 for SV) reported for pecan nut oil

by Firestone [13]. However, the value obtained for the refraction index (i.e. 1.469 at 40 °C) was identical. The unsaponifiable matter of 1.99 g/100 g was higher than the values (i.e. 0.4–1.5 g/100 g) reported by Firestone [13]. The oil had a Lovibond color of 20.0 yellow units and 3.0 red units, indicating a light yellow color. The oxidative stability (OSI) of pecan nut oil was 9.8 h. This stability is considered high and reflects the fatty acid composition of the oil under study, rich in oleic acid (62.55%) and low in linolenic acid (1.23%) as reported by Oro et al. [5]. Toro-Vásquez et al. [6] reported induction periods of 8.5–10.8 h for pecan nut oil produced in Mexico.

Quality Evaluation of Crude Pecan Nut Oil during Storage

The results of the analyses of peroxide value, specific extinction at 232 nm and specific extinction at 270 nm, acid value, moisture and volatile compounds and Lovibond color, carried out during pecan nut oil storage, are presented in Table 3. The peroxide value (PV) of pecan nut oil varied from 0.55 mequiv O_2/kg at the initial time of analysis up to a maximum of 7.23 mequiv O_2/kg oil, after 75 days storage. After 75 days, a reduction in the PV of the oil was observed, probably due to hydroperoxide decomposition and, after 120 days storage the oil had a PV of 2.16 mequiv O_2/kg. According to the linear regression analysis, it was possible to observe that the linear variation for peroxide value as a function of time was not significant ($p \geq 0.05$).

Specific extinction at 232 nm of crude pecan nut oil ranged from 0.833 to 2.926 after 120 days storage. The regression analysis indicated a significant linear increase ($p < 0.0001$) during the storage period ($R^2 = 0.58$). Specific extinction at 270 nm ranged from 0.033 to 0.356 after 120 days storage. According to the regression analysis, there were no significant variations in the specific extinction coefficient with time ($p > 0.05$).

The acid value of pecan nut oil varied between 0.13 mg KOH/g at the beginning of storage and 0.170 mg KOH/g after 120 days storage. According to the regression analysis, the free fatty acid content of the oil increased significantly and linearly as a function of time ($p \leq 0.0001$ and $R^2 = 0.72$). Moisture and volatile compound content of pecan nut oil remained low throughout all the storage period. According to the regression analysis, and as expected, oil moisture did not present significant variations during the period of analysis ($p > 0.05$).

Regarding the stability of pecan nut oil color during storage, there was an increase in the intensity of yellow during the 120 days of storage of the oil. According to the regression analysis, these alterations were significant ($p < 0.05$ and $R = 0.96$). Statistically significant changes ($p > 0.05$) were not observed in the red hue of the oil.

Evaluation of Sensory Quality of Pecan Nut Oil during Storage

Descriptive Sensory Analysis

The mean values attributed to the descriptive terms of pecan nut oil as a function of storage time can be seen in Table 4. According to the analysis of variance, only nut aroma, oxidized taste, and bitter taste were altered with time. It is possible that the nut taste was unchanged because the sensory attribute vegetable oil was the predominant characteristic in the product, which also did not change significantly during the storage time.

According to the Tukey test, at 90 days storage, it was possible to observe a significant increase in oxidized taste. After 120 days storage, it was possible to observe a

Table 3 Evolution of quality parameters of crude pecan nut oil during storage

Time (days)	PV[a]	SE 232[b]	SE270[b]	AV[a]	M[b]	LC
0	0.55 ± 0.07	0.833 ± 0.004	0.033 ± 0.023	0.13 ± 0.02	0.05 ± 0.00	20.0Y/3.0R
30	1.53 ± 0.03	1.181 ± 0.016	0.029 ± 0.026	0.13 ± 0.01	0.05 ± 0.01	–
45	4.18 ± 0.01	1.260 ± 0.141	0.231 ± 0.004	0.13 ± 0.01	0.05 ± 0.01	26.6Y/2.6R
60	5.62 ± 0.00	1.426 ± 0.000	0.291 ± 0.000	0.13 ± 0.00	0.05 ± 0.00	26.6Y/2.6R
75	7.23 ± 0.07	1.439 ± 0.015	0.343 ± 0.023	0.14 ± 0.00	0.05 ± 0.01	30.0Y/2.0R
90	4.79 ± 0.07	1.089 ± 0.015	0.130 ± 0.000	0.14 ± 0.01	0.05 ± 0.00	30.0Y/2.0R
105	2.41 ± 0.21	1.272 ± 0.031	0.118 ± 0.015	0.15 ± 0.01	0.06 ± 0.00	30.0Y/3.1R
120	2.16 ± 0.07	2.926 ± 0.038	0.356 ± 0.061	0.17 ± 0.00	0.06 ± 0.01	36.7Y/2.8R

PV peroxide value (mequiv O_2/kg oil), *SE232* specific extinction at 232 nm, *SE270* specific extinction at 270 nm, *AV* acid value (%), *M* moisture and volatile compounds (%), *LC* Lovibond color (*Y* yellow, *R* red)

[a] Results represent means of two repetitions

[b] Results represent means of three repetitions

Table 4 Mean obtained for the descriptive terms of crude pecan nut oil as a function of storage time

Time (days)	Apparent homogeneity	Nut aroma	Nut taste	Vegetable oil	Oxidized taste	Bitterness
0	8.8 a	8 a	8.2 a	6.5 a	0.5 c	0.5 b
30	8.7 a	7.8 a	8.3 a	6.4 a	0.4 c	0.5 b
60	8.5 a	7.9 a	8.1 a	6.4 a	0.5 c	0.7 b
90	8.7 a	7.8 a	8.0 a	6.5 a	1.5 b	0.8 b
120	8.7 a	6.9 b	8.2 a	6.3 a	2.7 a	2.5 a

Mean followed by the same letters in columns do not differ significantly; Tukey test ($p \leq 0.05$)

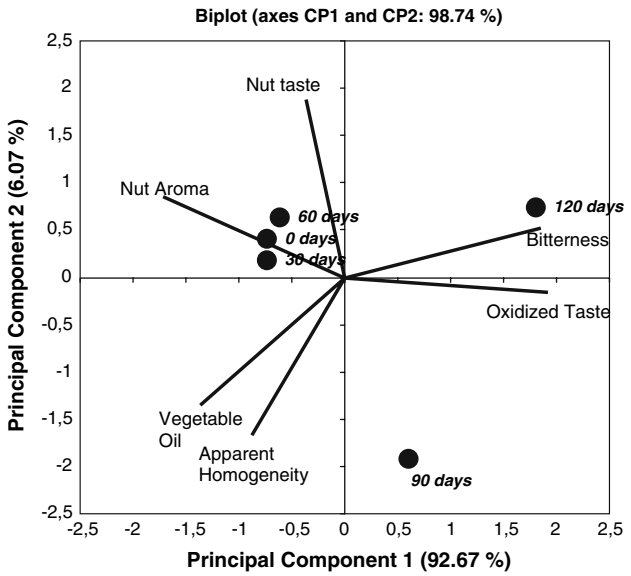

Fig. 1 Two-dimensional sensory analysis diagram of the intensity of the attributes of pecan nut oil as a function of time

significant decrease in nut aroma and a significant increase in oxidized taste and bitterness ($p \leq 0.05$). Despite the significant decrease in nut aroma, changes in nut taste were not detected by the trained panelists.

Multivariate principal component analysis is represented in Fig. 1, where it can be seen that the samples formed three different groups. These consist of a first group formed by the samples stored for 0, 30 and 60 days, which were sensorially very similar, as they are located close to each other, and are characterized mainly by nut aroma and taste. The second group is formed by the sample stored for 90 days, in an intermediate point, and without any outstanding sensory characteristic. The third group is formed by the sample stored for 120 days, which was characterized mainly by an oxidized taste and bitterness.

Table 5 Mean for acceptance of pecan nut oil as a function of storage time

Time (days)	Aroma acceptance	Taste acceptance	Global acceptance
0	7.8 a	8.0 a	8.0 a
30	7.6 a	7.9 a	8.1 a
60	7.8 a	7.9 a	7.9 a
90	7.0 b	6.5 b	6.0 b
120	6.5 c	5.2 c	5.0 c

Mean followed by the same letters in columns do not differ significantly; Tukey test ($p \leq 0.05$)

Consumer Acceptance Analysis

The mean for the acceptance of pecan nut oil with respect to aroma, taste and global acceptance as a function of the storage period can be seen in Table 5. Acceptance of pecan nut oil began to decrease significantly ($p \leq 0.05$) at 90 days, and was accentuated at 120 days.

Correlation Between Sensory Profile and Acceptance

Figure 2 shows the correlation between the sensory attributes and acceptance of pecan nut oil as a function of storage time. Global acceptance means were analyzed in relation to the descriptive terms. It was possible to verify that there was a significant negative linear correlation ($p \leq 0.05$) between consumer acceptance and the descriptive panel indicators for bitterness and oxidized taste. Thus, there was a notable decline in acceptance after 90 days storage and an increase in bitter taste and oxidized taste in the same period.

According to the sensory analysis results, it can be concluded that pecan nut oil remained sensorially unaltered up to 60 days of storage. By 90 days, there was a significant increase in oxidized taste and bitterness and a significant decrease in the nut taste ($p \leq 0.05$), which occurred simultaneously with the significant decline ($p \leq 0.05$) in the acceptance of the oil. After 120 days storage, the changes became more pronounced.

Conclusion

Although not indicated by the physicochemical analyses, according to the sensory analysis, crude pecan nut oil presented a shelf life of 90 days, when stored in amber glass flasks at room temperature (average of 22.5 °C). The storage period (120 days) was limited by the sensory analysis, when global acceptance was 5.0. Physicochemical

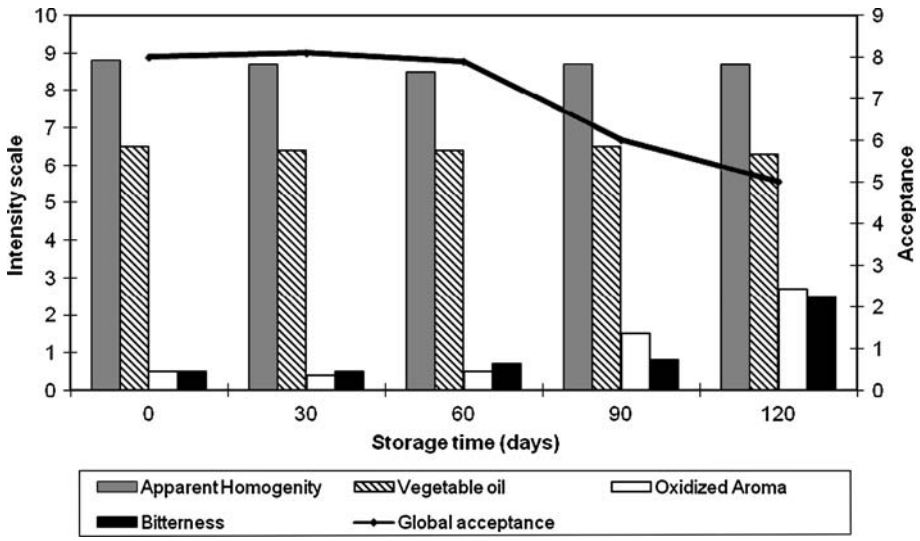

Fig. 2 Mean of sensory attributes and acceptance of crude pecan nut oil as a function of storage time

analyses—like Headspace or Anisidine Value—that measure secondary oxidation products may provide additional information about the stability of pecan nut oil and could be correlated to sensory analysis.

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