Characterization of Stored Regular and Low-Linolenic Canola Oils at Different Levels of Consumer Acceptance¹

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ABSTRACT: A ten-member trained sensory panel evaluated regular (RCO) and low-linolenic (LLCO) canola oils that had been stored at 60°C to four levels of consumer acceptance identified in a prior study. These levels were 70, 60, 50, and 40% acceptance for RCO and 80, 70, 60, and 50% acceptance for LLCO. Painty odor intensity increased as consumer acceptance decreased. This same trend was found for chemical measurements of peroxide values, total volatiles, total carbonyls, unsaturated carbonyls, and dienals. These chemical indices were significantly correlated with each other, suggesting that they can be used to monitor related changes in oil quality with respect to lipid oxidation. Values for 19 individual volatiles at each consumer acceptance level were also reported. The data collected in this study provide chemical and sensory characterization of stored RCO and LLCO at distinct levels of consumer acceptance.

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KFY WORDS: Canola oil, consumer, low-linolenic canola oil, sensory, shelf life, stability, storage, volatiles.

Quality assessment of vegetable oils routinely involves measurement of the chemical and sensory characteristics of oil samples that are subjected to accelerated storage to determine their relative stability to oxidation (1). Work by Hawrysh *et al.* (2) suggests that regular canola oil with a sensory induction period of 2-4 d at $60-65^{\circ}$ C may be expected to remain perceptibly unchanged in flavor quality for at least 16 wk if protected from light at room temperature (3). This speculation is consistent with earlier work by Evans *et al.* (4) who found flavor scores for soybean oil, aged four days at 60^oC, were equivalent to those for oils aged four months at ambient temperatures.

While such relationships help to define the point at which changes occur in oils during storage, they lack the consumer acceptance perspective that is useful in predicting shelf life. Wan (1) has defined the keeping quality of an oil as the length of time that it resists significant change in its acceptable characteristics. Food practices suggest that consumers accept some degree of oxidation in edible oils, although the limits have not been known. Defining these limits could help to address concerns that surround the uncertain health consequences of ingesting lipid oxidation products (5).

A forerunner to the present study established a consumer acceptance gradient that can be used to monitor the change in consumer acceptance of canola oils during accelerated storage (6). Regular (RCO) and low-linolenic (LLCO) canola oils (12.5% and 2.5% 18:3, respectively) were examined by 92 consumers for odor acceptability during accelerated storage at 60°C. In keeping with earlier findings that the stability of canola oil is linolenic acid-dependent (7), storage times with the common levels of 70, 60, and 50% consumer acceptance differed between the two types of canola oils. For example, the threshold level of 50% consumer acceptance, calculated from logistic regression analysis, was reached at 12.5 d of storage at 60° C for RCO but not until 34.3 d for LLCO.

The present experiment was designed to characterize RCO and LLCO, stored to established levels of consumer acceptance. Characterization was done by means of sensory evaluation by a trained panel, chemical measurement of peroxides, and gas-chromatography assessment of flavor and off-flavor volatiles. Although peroxide values of oils are used routinely to monitor the oxidative state of edible oils, their predictive value is limited because peroxides are susceptible to decomposition during storage and heating (8). Analysis of the volatiles in stored oil appears to be the most suitable approach to mimic odor and flavor assessments, particularly when gaschromatographic methods, such as direct injection, are used where lower temperatures produce fewer artifacts (9).

EXPERIMENTAL PROCEDURES

Oils. The origin, processing, and accelerated storage conditions of RCO and LLCO have been described previously (6). The fatty acid compositions of the fresh oils are provided in Table 1. The largest difference can be seen in the levels of 18:3, with modest differences in 18:2 and 18:1 levels.

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 a ¹V = iodine value calculated as triglycerides, RCO, regular canola oil; LLCO, low-linolenic canola oil.

 b Other fatty acids each <0.3%.

Ninety-gram oil aliquots were stored at 60° C for the number of days that correspond to four levels of consumer acceptance (6). These were 3, 8, 12, and 17 d for RCO, representing consumer acceptance levels of 70, 60, 50, and 40%, respectively, and 9, 18, 27, and 34 d for LLCO, representing acceptance levels of 80, 70, 60, and 50%. Thus, there were three consumer acceptance levels common to both oils. In addition, samples of RCO stored 0 and 1 d, and LLCO stored 0, 4, and 14 d were included to provide a baseline for fresh oils and to reexamine storage days where data in the consumer study had been most variable. Accordingly, there were six storage times for RCO and seven for LLCO. Upon removal from storage, all sample jars were flushed with nitrogen, capped with Teflon-lined plastic lids, and held at -18° C until required (maximum 180 d).

Samples for odor evaluation consisted of 10 g of oil and 30 g of 4-mm glass beads in 125-mL glass jars, capped with Teflon-lined plastic screw lids. Samples were equilibrated to 50° C for sensory testing (10) by holding jars in a water bath on a Coming hotplate (PC-300).

Sensory evaluation. A ten-member trained panel (two men, eight women) was selected from 20 candidates on the basis of superior performance in odor discrimination among stored samples of RCO and LLCO, assessed in four triangle tests. Nine training sessions were held over a four-week period to familiarize panelists with the odor attributes of buttery and painty, the use of unstructured 15-cm line scales, and the reference samples. Panelists agreed that RCO stored at 60° C for 20 d was a suitable endpoint reference to represent strong/intense painty odor, and that fresh LLCO, containing 0.002% w/w Nat&Art Flavor #3059 Butter (Danisco Ingredients, USA Inc., New Century, Kansas), was a suitable endpoint reference to represent strong/intense buttery odor. Practice sessions were also held during training to give panelists experience in rating intensities of both odor attributes from a range of stored oils. The buttery parameter was always scored before paintiness. Samples were coded with three-digit numbers and were served in a randomized order. During each test session, panelists evaluated four stored samples of one type of oil. Thus four sessions were required to evaluate all storage days for both oils. Two replications were completed for a total of eight test sessions. The order of oil type was alternated between replications.

Sensory scores of 0 to 15 (none to strong/intense) for each parameter were assigned according to the point on the line scale marked by the panelist. Within each oil and parameter, scores for both replications were combined within panelists. A weighted least-squares analysis was performed with the weights proportional to the variability of the values assigned on a particular day. All analyses were performed on the square root-transformed dependent variables, which were arranged as a factorial design, where the main effects were days and panelists (RCO = 6×10 ; LLCO = 7×10), and their interaction served as the error term (11).

Odor examinations were carried out under red light in a sensory panel facility with eight computer-equipped booths. The software program CSA Computerized Sensory Analysis System (Compusense Inc., Guelph, Canada) was used to input and record the sensory responses.

Chemical analyses. Fatty acids were measured as methyl esters, separated on a capillary column (30 m \times 0.25 mm i.d.) coated with DB-225 (J&W Scientific, Folsom, CA), by using the AOCS procedure (10) as adapted by Przybylski *et al.* (7). The column temperature was isothermal at 200° C.

Peroxide values (PV) and volatile compounds were determined in duplicate on each of two replications of stored oils: those stored for the present study and samples from matching storage times reserved from the prior consumer study (6). PV were determined with the AOCS standard method (10).

Volatile compounds in the oils at each storage interval were determined by gas chromatography (GC) by using dynamic headspace analysis, modified to include aspects of direct injection (12). The oil sample was placed onto a glass wool plug, prepared inside an injector glass insert, which was then heated to 100° C, and the volatiles were purged for 15 min. Volatiles were collected on a trapping pre-column, which was a deactivated wide-bore capillary column immersed in liquid nitrogen. After purging, the liquid nitrogen was removed, and volatiles were transferred directly from the pre-column into the analytical capillary column at 45° C, where both columns were in the GC oven and connected through a splitter. Volatiles were separated on a capillary column (0.32 i.d. \times 60 m) with DB-5 (J&W Scientific, Folsom, CA). The column temperature was programmed from 45 to 235° C at a rate of 3° C/min. Starting and upper temperatures were held for 2 and 35 min, respectively. Individual peaks were quantitated with dodecane as an internal standard and grouped as total volatiles (TV), total carbonyls (TC), unsaturated carbonyls (UC), and dienals (DE).

^aSignificant $P < 0.05$; df, degrees of freedom; MS, mean square. See Table 1 for other abbreviations. b Significant $P < 0.01$.

 c Significant $P < 0.001$.

RESULTS AND DISCUSSION

TABLE 2

Sensory characteristics. Table 2 summarizes the analysis of variance of the trained panel's assessments of buttery and painty odor intensities in RCO and LLCO over storage. Although significant panelist effects were observed, these can be attributed to the use of the scoring line rather than disagreement among panelists in ordering the samples, because significant panelist-by-day interactions were not found in most cases. No significant day effect in butteriness was found for either oil. A significant day effect, however, was observed for painty odor intensity in both oils.

The buttery characteristic in canola oil, which has also been noted by Warner *et al.* (13), was found at low intensity in both oils (Fig. 1). Painty scores increased as storage time increased, but the rate was slower in LLCO than in RCO (Fig. 1). The end of the induction period, which typically precedes a sudden rise in oxidation rate (I), was observed at Day 1 in RCO but not until Day 9 in LLCO. To determine significant differences between successive storage days, contrasts were performed on mean painty scores for each oil. Significant differences were observed between Day 27 and 34 of LLCO and Day 1 and 3, Day 3 and 8, and Day 8 and 12 of RCO ($P < 0.07$). Similar distinctions had been observed in consumer acceptance estimates (6). Thus, it appears that sensory paintiness scores are a useful indicator of consumer acceptance.

Chemical characteristics. Initial oxidation products, estimated by PV, increased as storage time progressed (Fig. 2), as did their degradation products, measured as TV and the subgroups TC, UC, and DE (Figs. 3 and 4). These findings confirmed those of Przybylski *et al.* (7) but extended the accelerated storage time during which data were gathered from 12 to 17 d for RCO and from 12 to 34 d for LLCO. The time extension permitted definition of the products of LLCO oxidation, a process that was just beginning within the 12 d limit of Przybylski *et al. 's* study (7). For RCO, the rate and magnitude of peroxide accumulation at 60° C over 17 d appeared similar to values reported by Hawrysh *et al.* (2) when RCO was stored at 65° C for 16 d.

From Figures 2 and 3, an RCO oxidation induction period of 1 and/or 3 d was observed for PV, TV, and TC in keeping with that observed earlier for sensory paintiness (Fig. 1). On the other hand, sharp changes in slope in UC and DE pattems in RCO were not evident until after 8 d (Fig. 4). In the LLCO series, where oxidation proceeded more slowly, there was more than one inflection point in the painty storage time curve (Fig. 1). The first inflection point occurred at 9 d, followed by

FIG. 1. Effect of storage on the sensory characteristics of canola oils; \triangle = buttery regular canola oil (RCO), \bigcirc = painty RCO, ∇ = buttery lowlinolenic canola oil (LLCO), \blacksquare = painty LLCO.

FIG. 2. Changes in peroxide values of canola oils during storage; $O = RCO$, $\square = LICO$. See Figure 1 for abbreviations.

FIG. 3. Total volatiles (TV) and total carbonyl (TC) compounds in canola oils during storage; $O = TV RCO$, $\square = TC RCO$, $\blacktriangle = TV LICO$, $\nabla = TC$ LLCO. See Figure 1 for other abbreviations

a sharper inflection points at Days 19 and 27. For PV, the inflection point occurred at Day 9 (Fig. 2) but not until Day 14 for TV and TC (Fig. 3), and Day 27 for UC and DE (Fig. 4). Accordingly, there were few commonalities among the indicators of LLCO induction periods until Day 27, when the patterns for paintiness and all chemical descriptors except TV exhibited a marked increase in slope.

Correlation analysis was performed between all chemical indicators (PV, TV, TC, UC, DE) for both oils and yielded coefficients (r) that ranged from 0.85 to 0.99 (Table 3). Thus, the chemical indices measured in this study appeared to be tracking related changes in oil quality with respect to lipid oxidation.

Individual volatiles at specific levels of consumer acceptance. The amounts of 19 individual volatiles from stored

FIG. 4. Unsaturated carbonyl compounds (UC) and dienals (DE) in canola oils during storage, $O = UC$ RCO, $\Box = DE$ RCO, $\blacktriangle = UC$ LLCO, \blacktriangledown = DE LLCO. See Figure 1 for other abbreviations.

RCO and LLCO at specific consumer acceptance levels are shown in Table 4. They are compounds from the decomposition, through free-radical oxidation, of oleate, linoleate, and linolenate hydroperoxides as described by Przybylski and Eskin (9). Amounts reported by Snyder *et al.* (14) for eight of these volatiles (pentane, the five- to nine-carbon aldehydes, and 2,4-decadienal) from RCO, stored at 60°C for 8 and 16 d, were higher than those from RCO stored in the present study for 8 d (60% acceptance) and 17 d (40% acceptance), respectively. Because the fatty acid composition of RCO was similar in both studies, the quantitative differences may be attributed to novelties in analytical methods. Snyder and Mounts (15) later reported lower amounts than in Table 4 for the same eight volatiles, from RCO after 8 days' storage at 60° C, measured by either single headspace sampling or multiple headspace extraction; in this case the 18:3 content was lower than in the present study (9% vs. 12.5%).

The effect of fatty acid composition on the extent of volatile formation during canola oil storage is illustrated by comparing the data in Table 4 for RCO and LLCO at similar storage times. For example, in comparing RCO at 8 d vs. LLCO at 9 d, or RCO at 17 d vs. LLCO at 18 d, the amounts of all 19 volatiles were appreciably higher in RCO, and the difference was greater at the longer storage time. This reflects the fact that RCO had five times more 18:3, less linoleic acid $(18:2)$ and less oleic acid $(18:1)$ than LLCO (Table 1). Oxidation rates increase with unsaturation, with 18:3 oxidizing twice as fast as 18:2 and 25 times faster than 18:1 (16).

While it might be expected that individual volatiles in stored canola oils at the same levels of consumer acceptance would be similar in quantity, over half of the 19 compounds were higher in LLCO than in RCO within each of the 70, 60, and 50% consumer acceptance pairs (Table 4). However, of the five volatiles present in the greatest amounts in both oils, there were four in common at 60 and 50% consumer accep-

	RCO				LLCO			
	TC	UC	DE	PV	TC	UC	DE	PV
т٧	0.99	0.99	0.99	0.88	0.92	0.91	0.87	0.95
TC		0.99	0.99	0.90		0.99	0.99	0.88
UC			0.99	0.91			0.99	0.88
DE				0.87				0.85

Correlation Coefficients (r) of Chemical Indicators for Both Canola Oils^a

TABLE 3

^aTV, total volatiles; UC, unsaturated carbonyls; DE, dienals; PV, peroxide value; see Table 1 for other abbreviations.

 $a_n = 2$; mean of two storage replications. See Table 1 for abbreviations.

bMixture of *cis* and *trans* isomers.

CMixture of *cis, cis* and *cis, trans* isomers.

tance: pentane, hexanal, propenal, and 2,4-decadienal. Three of these, pentane, hexanai, and 2,4-decadienal, have been used as satisfactory indices of lipid oxidation (9). Tokarska *et al.* (17) identified 2,4-decadienal as a major off-flavor volatile in stored RCO, and Ullrich and Gross (18) recognized it as one of the most intense flavor compounds of autoxidized linoleic acid.

Sensory impact of volatile oxidation products, Table 5 cites published odor thresholds and qualitative descriptors for most of the 19 volatiles that were quantitated during this study of canola oil storage. Odor thresholds are measures of the concentration at which a compound is first detected when evaluated singly in a neutral medium. While they provide a point of reference in evaluating contributors to a particular sensation, it is risky to project suprathreshold impact from threshold concentrations. Dixon and Hammond (19) showed that threshold values of aldehydes and ketones common to oxidized oils failed to predict a compound's relative importance at higher concentrations. Furthermore, the sensory qualities of a compound may vary with concentration change, as has

been documented by Laing and Willcox (20) for hexanal, octanal, decanal, and their *trans* unsaturates.

Of the qualitative descriptors in Table 5, the terms "heavy," "oily (fatty)," "painty," "rancid," "sour, sharp," and "metallic" were among those mentioned by consumers as criticisms of stored canola oils at 50% acceptance (6). Such terms could implicate many of the volatiles measured in this study in the acceptance decision of consumers. Our trained sensory panel elected paintiness as the most appropriate descriptor of oxidation in canota oils. Painty odors have been associated previously with three of the volatile oxidation products found in our stored canola oils: pentanal, hexanal, and heptanal (Tables 4 and 5). In addition, the geometric isomers of two others have been characterized as painty, i.e., t-2-decenal (20) and t,t-2,4-decadienal (21). These observations suggest that paintiness is an integrated response to a combination of volatile stimuli.

A variety of interactions can occur in mixtures of sensory stimuli: suppression, additivity, and even synergy, depending

aReference 26. **bReference** 21 (cooked **with ground** beef). **CReference** 27. **dReference** 28. **eReference** 29 (in breads). **fReference** 20 (in di-2-ethy[hexylphthalate).

gReference 24 (in dioctylphthalate). **hReference** 30. **iReference** 31.

on their odor/flavor qualities and proportions. Combinations of subthreshold quantities of odorants have been shown to be additive (22). Suprathreshoid mixtures generally have shown only partial additivity. For example, Laing and Willcox (20) demonstrated partial additivity in the total odor intensity of mixtures of two fat oxidation products, t-2-hexenal (green) and t-2-decenal (painty). However, the quality of their mixture's aroma was not predominantly rancid, suggesting that a mixture more complex than two components may be responsible for the rancid odor of oxidized oils.

In the present experiment, the only odorant mixtures examined by panelists were the total volatiles of stored oils, and the single negative quality measured by the trained panel was paintiness. Exploring the relationships between perceived paintiness and TV in stored RCO and LLCO showed power functions of $S = 0.94C^{0.56}$ and $S = 0.71C^{0.73}$, respectively. The power function $(S = kC^n)$, describes the linear relationship in **log-log coordinates between a physical stimulus and the psychological response as follows (23):**

 $\log S = \log k + n \log C$ [1]

where: $S =$ sensory response; $C =$ physical r agnitude (concentration); $n =$ the exponent or slope; $k =$ the intercept.

The exponents (n) of the power functions for RCO and LLCO of 0.56 and 0.73, respectively, were in keeping with the range of 0.42 to 0.72 for the exponents of power functions reported by Hall and Andersson (24) for 14 individual volatile fat oxidation products. They considered that, within such a range of exponents, there would be similar rates of growth in sensory intensity with increases in concentration. An exponent less than one indicates that the sensory response compresses the intensity Of stimulus increments, For example, a 10-fold increase in stimulant concentration would be perceived as a 10-fold increase in sensory intensity when $n = 1$, whereas when $n = 0.5$, only a 3.2-fold increase in the inten**sity of the perceived sensation could be expected (25).**

Sensory and chemical indices at specific consumer acceptance levels. **Table 6 summarizes the sensory and chemical**

^aFor paintiness $n = 20$ (10 panelists \times 2 panel replications) and for all chemical measures $n = 2$ (2 storage replications). See Tables 1 and 3 for abbreviations.

characteristics of RCO and LLCO at the four consumer acceptance levels (6). According to Wan (1), it is advisable to measure a selected group of properties rather than any single one when defining the quality of an oil. The data collected in this study add to the database needed to describe oxidizing canola oils (9) and, uniquely, provide a chemical and sensory profile of RCO and LLCO at each acceptance level. For example, the odor of RCO with TV measuring around 3.87 mg/kg and with a PV of 18 meq/kg and a painty odor score of 1.8 would be considered acceptable to about 70% of the consumers. Such extrapolations must be viewed for what they are, approximations. Nevertheless, they provide a way of attaching consumer acceptance meaning to laboratory measurements of canola oil quality.

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TABLE 6

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