Oil Stability Index Correlated with Sensory Determination of Oxidative Stability in Canola Oil

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ABSTRACT: The usefulness of the Oil Stability Index (OSI) as an accelerated oxidative stability test for canola oil was studied by correlating the OSI with the induction period as determined by sensory analysis. Canola oil was treated by holding it for differing times (0, 1, 2, 3, 4, and 6 d) at elevated temperature (60°C) in the dark with agitation. The sensory induction period (SIP) was determined by storing the five treatments of oil and the control at 60°C in the dark with agitation and removing aliquots of oil for a nine-member sensory panel to evaluate over a 9-d period. The time it took for a treatment to reach an average sensory score of 5 (10-point scoring scale) was defined as the treatment's SIP. OSI values were obtained on day 0 using a heating block temperature of 110°C and an air pressure of 6 psi. The relationship between SIP and OSI had a 0.89 coefficient of determination (r^2). This relationship may be sufficiently strong to warrant use of the OSI in industry applications but may not be ideal for more precise experimental studies of canola oil shelf life.

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KEY WORDS: Accelerated methods, canola oil, lipid oxidation, oil stability index, oxidative stability, peroxide value, rancidity, Rancimat, sensory evaluation, shelf life.

Lipid oxidation has long plagued food industries wishing to extend product shelf life. Estimating the shelf life of canola oil is of particular interest since canola oil is the second most widely consumed oil in the United States. However, because of its relatively high concentration of linolenic acid, canola oil is very susceptible to lipid oxidation. Oil shelf life can be defined as the amount of time for an oil to reach an unacceptable level of quality. Evaluating an oil's quality under ambient temperatures requires testing oil over an extended period of time. Evaluating shelf life using accelerated testing methods requires much less time.

Summaries of factors affecting the shelf life of canola oil have been compiled. Hawrysh (1) reviewed the degradation of canola oil *via* autoxidation, photooxidation, and thermal oxidation. Also assessed were the relative stability, the evaluation of current status, the efficacy of using antioxidants, and the effects on oil quality of volatile decomposition products in canola oil. Malcolmson *et al.* (2) reviewed temperature effects in accelerated autoxidation, Schaal oven studies, and odor stability of canola oil. Since that time, other sensory analysis studies have compared the consumer acceptance of regular canola oil to canola oil containing a lower concentration of linolenic acid (3,4).

Accelerated shelf life estimates may be made using sensory or chemical techniques (5). However, any chemical method used to evaluate shelf life must be closely correlated to sensory analysis. Only sensory analysis can detect flavors due to oxidative and nonoxidative degradation processes (6). No instrumental or chemical analysis can detect all off-flavors. Sensory analysis is ultimately the best method of determining an oil's quality and stability due to its unique sensitivity (7) and is critical to realistic shelf life evaluations and accelerated shelf life studies.

The widespread use of sensory analysis to evaluate oil has prompted AOCS and ASTM to adopt standard practices for evaluating vegetable oils (8–10). Standard practices describe such parameters as the panel room, presentation utensils, reagents, oil preparation, and presentation (Ref. 9, Cg 2-83; Ref. 10, E 1346-90 and E 1627-94).

The AOCS-recommended score sheet for evaluating edible oil intensity is a 10-point scoring scale based on the overall flavor intensity. Each number on the 10 to 1 scale is assigned a descriptor associated with the flavors and odors typical of each stage of oxidation (10 being bland and 1 being extreme). Although this ranking scale is sensitive, maintaining a trained sensory panel to accurately distinguish differences using the ranking scale is extremely time consuming and costly. Because of the challenges of conducting sensory analysis, researchers and oil producers have long sought faster alternatives in product shelf life testing.

Despite the usefulness of many chemical and instrumental tests designed to evaluate the current state of oxidation in a product, evaluating shelf life requires testing of a product over a period of time. Testing a product over time is the only way such a test can be used to evaluate how susceptible a lipid is to oxidation. Several relatively fast methods of evaluating a lipid's susceptibility to oxidation do exist (11). One method used in the past was the Active Oxygen Method (AOM; AOCS Official Method Cd 12-57). However, the AOM was labor-intensive, its reproducibility was relatively low, and it involved measurement of primary oxidation products (peroxides), which readily breakdown at elevated temperatures (7,12).

The Oil Stability Index (OSI; AOCS Official Method Cd 12b-92) has largely replaced the AOM as a means to measure

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an oil's susceptibility to oxidation. The Rancimat (Brinkmann Instruments, Inc., Westbury, NY) and the Oxidative Stability Instrument (Omnion, Inc., Rockland, MA) have gained acceptance for their ability to measure OSI (the point of maximal change of the rate of oxidation) because of their ease of use and high reproducibility (13). These instruments also record data continuously, so the length of time to reach the OSI is precisely determined. In addition, the OSI is determined by measuring stable secondary oxidation products formed at high temperatures (12).

Thus, OSI results are reproducible and can be collected quickly. However, because the criteria for lipid quality are ultimately based on sensory analysis, the relationship between the OSI and sensory analysis needs to be established (14). The purpose of this study was to determine the usefulness of OSI as an accelerated oxidative stability test for canola oil by correlating the OSI with the induction period as determined by sensory analysis of canola oil stored in the dark.

EXPERIMENTAL PROCEDURES

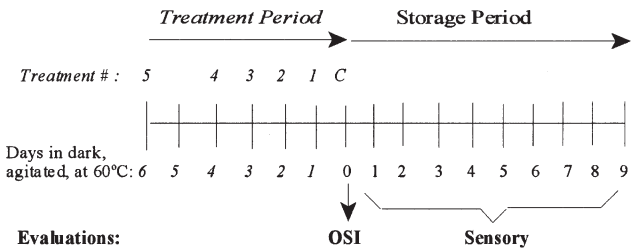
Freshly processed canola oil was obtained from a commercial processor (Archer Daniels Midland Company, Decatur, IL). Treatment and control samples were prepared by placing 1025 mL of commercially processed canola oil into Erlenmeyer flasks, flushing the flasks with nitrogen gas, capping them with saran-covered rubber stoppers, and holding them at −18 ± 2°C. Oil samples were removed from the freezer and placed in a 60°C orbital shaking (230 rpm) incubator (Innova 4000; New Brunswick Scientific) covered with aluminum foil to prevent light exposure. Samples were held in the 60°C agitated, dark environment for 0, 1, 2, 3, 4, and 6 d, which constituted the control and treatments 1–5, respectively. The experimental design is graphically depicted in Scheme 1. The control, removed from the freezer on day 0 of the storage period, was warmed at 60°C just long enough for the oil to become liquid before aliquots of oil were removed. The control and treatment samples were then stored under the same conditions: a dark, agitated, 60°C environment. The OSI was measured at day 0 of the storage period, and PV and sensory analysis were performed over the storage period of 9 d (see Scheme 1). PV was determined using AOCS Official Method Cd 8b-90 (9).

OSI. OSI values (AOCS Official Method Cd 12b-92) were obtained in duplicate for all oil treatments and the control using the Oxidative Stability Instrument (Omnion, Inc.). The instrument was run using a 5.00 ± 0.006 g oil sample and a heating block temperature of 110°C; purified air was bubbled through the oil at 6 psi. Probes, rubber stoppers, and connecting glass and tubes were washed prior to use in a 1% Alconox detergent solution (Alconox, White Plains, NY), boiled 45 min, scrubbed, and rinsed with distilled water followed by deionized water. The tubes were then placed in a deionized water bath for 1 h and rinsed again with deionized water before being allowed to air dry under cover (free from dust).

Sensory analysis. (i) Panel members and training. Nine panelists were selected for the descriptive sensory panel based on their previous experience in descriptive oil analysis. All panelists underwent a 3-d training session to become familiar with canola oil. During this training session, panelists received smelling and tasting instructions, reference standards to familiarize themselves with typical odors and flavors of oxidized canola oil, and testing samples (disguised reference standards and mixtures of reference standards) to evaluate their sensory ability. Evaluation instructions are shown in Scheme 2. Prior to evaluating each sample's overall flavor intensity, panelists were instructed to smell the oil and arrange the samples in order of increasing intensity. This was done to help reduce the possible sensory overload that may occur if panelists taste an intensely oxidized sample first. After each training session, panelists were told how they performed and coached on what odors and flavors were typical of each stage of oxidation.

(ii) Reference standards. Reference standards equivalent to bland, slight, moderate, and strong were provided to panelists during each evaluation session. These were, respectively, equal to 10, 7, 5, and 3 on a 10 to 1 scale. The slight, moderate, and strong reference standards were prepared by shaking oil at 100 rpm in a dark, 60°C environment until each had attained a PV of 5.1, 10.4, and 18.4 meq/kg, respectively. The bland reference standard was prepared immediately after receiving the oil; its PV was 0.4 meq/kg. After reaching the desired PV, each reference standard was divided into 200-mL portions (in 250-mL Erlenmeyer flasks), flushed with nitrogen gas, capped with saran-covered rubber stoppers and held at −18 ± 2°C. Reference standards were removed from the freezer and refrigerated (2 ± 1°C) for 12 to 15 h prior to being placed in 30-mL vials and treated identically to the oil samples.

(iii) Panel conditions. During the panel, each panelist received the same smelling and tasting instructions and reference standards as those utilized during training. Warm water and expectorating cups were also provided to panelists during each session. Each morning before the sensory panel began, samples were drawn from the Erlenmeyer flasks and maintained 1–3 h at room temperature (22 ± 2°C) until panelists evaluated the samples. Just prior to serving, samples were heated in a forced draft oven to 50 ± 3°C, and then immediately served in a styrofoam block (30.5 × 12.2 × 3.8 cm). Panelists evaluated 10 mL of each treatment of oil that had been placed in a 30-mL clear vial capped with a Teflon-lined, screw-cap lid. All vials and lids used during the panel were washed, double rinsed, and dried prior to each vial being labeled with a random three-digit



SCHEME 1

Sample panelist score sheet.

Initials & Sensory Code:_____ Date_____

Please refer to the standards provided as necessary to familiarize yourself with the scoring of the samples. It may be necessary to taste each standard until you are completely familiar with their characteristic taste. Even after becoming a skilled taster, please be sure to taste at least the "slightly oxidized" standard each tasting session.

Directions when tasting: take 75 to 100% of warm oil sample into the mouth, pull air through the oil and evaluate the flavor by exhaling through the nose (keeping the mouth closed). Expectorate the oil after your evaluation in the cup provided. Mark the **overall flavor intensity** for each sample using the intensity scale below.

	Quality	Overall Intensity Scores		
		Sample# 123	Sample# 456	Sample# 789
10	Bland	_____	_____	_____
9	Trace	_____	_____	_____
8	Faint	_____	_____	_____
7	Slight	_____	_____	_____
6	Mild	_____	_____	_____
5	Moderate	_____	_____	_____
4	Definite	_____	_____	_____
3	Strong	_____	_____	_____
2	Very strong	_____	_____	_____
1	Extreme	_____	_____	_____

SCHEME 2

code. A total of 48 sensory determinations was planned for each panelist (five treatments plus the control evaluated on 8 d of the storage period.) However, slightly fewer (43) determinations were completed because some treatments became too oxidized to evaluate. To avoid sensory fatigue, panelists evaluated oil samples in two sessions each day. Three of the treatments were randomly assigned to each session. No more than three samples were evaluated by any panelist in any session. Sessions were conducted in a sensory panel room (22 ± 2°C) equipped with individual booths. Panelists evaluated the samples under red light.

Relationship of OSI to sensory induction period (SIP). The averages of sensory panelists' flavor scores for each treatment were plotted against storage time. A best-fit line was determined for each treatment using linear regression. The point where this line intersected a sensory score of five was defined as the SIP or the time necessary to reach an unacceptable level of quality. The relationship between SIP values and OSI values was then determined using linear regression analysis (SigmaPlot®; Jandel Scientific, San Rafael, CA).

RESULTS AND DISCUSSION

PV for each treatment were obtained over time and can be seen in Figure 1. As expected, treatments that had been held at 60°C for shorter periods of time were slower to oxidize and form peroxides. Therefore, holding at 60°C in the dark with agitation for differing amounts of time was an effective treatment to obtain oil samples with varying amounts of rancidity.

Each treatment's OSI value (obtained on day 0 of the storage period) can be seen in Table 1. As expected, OSI values decreased with increased 60°C treatment time. From Table 1, it is

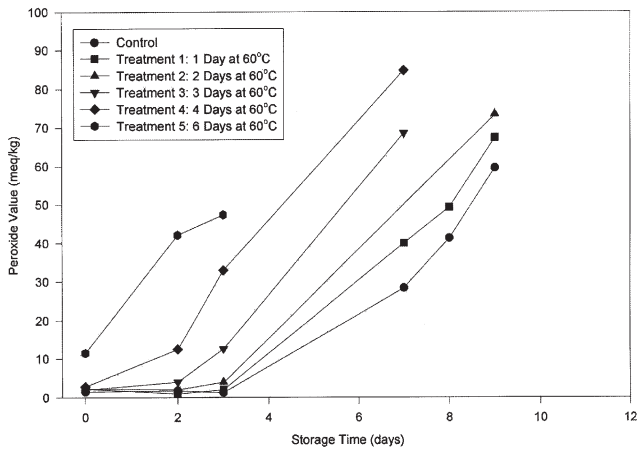


FIG. 1. Peroxide values of canola oil held at 60°C for various times.

TABLE 1
Oil Stability Index of Canola Oil Held at 60°C for Various Times[a]

	Control	Treatment 1	Treatment 2	Treatment 3	Treatment 4	Treatment 5
Rep 1	9.70	8.85	8.20	7.60	7.50	5.60
Rep 2	10.05	8.85	8.30	7.80	7.50	5.80
Avg	9.875	8.85	8.25	7.70	7.50	5.70

[a]Rep, replicate; Avg, average.

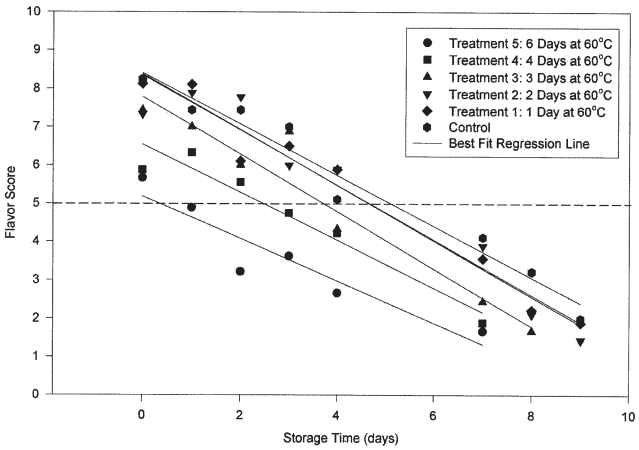


FIG. 2. Average sensory flavor scores for canola oil held at 60°C for various times.

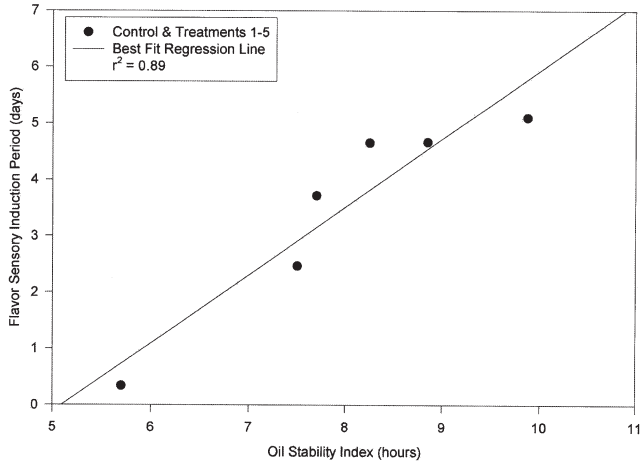


FIG. 3. Regression analysis line and coefficient of determination of Oil Stability Index and sensory induction period of canola oil held at 60°C for various times.

evident that OSI values obtained from the Oxidative Stability Instrument exhibit little variation (SD of the means ranged from 0 to 0.248). This agrees with previous research that showed OSI values can be obtained with little variation (13).

Figure 2 shows each treatment's average flavor score plotted over time. The average treatment SD was 1.63 (data not shown). As expected, average flavor scores decreased over time and according to treatment. That is, the longer the oil was held at 60°C, the lower the average sensory score. The regression lines in Figure 2 were used to determine each treatment's SIP.

Despite variation among sensory panelists, it is evident from Figure 2 that the average sensory scores are representative of oil quality. This agrees with an AOCS collaborative study (8) that indicated that within-laboratory variance of sensory panel scores tends to be large but that between-laboratory variance is significantly lower. This suggests that despite the variance inherent in sensory analysis, average sensory scores from a trained panel can be an accurate indication of oil quality.

The usefulness of the OSI in predicting shelf life as defined by the canola oil's SIP was determined using regression analysis. This is shown graphically in Figure 3, where average OSI values for each treatment are plotted against SIP values for each treatment. All five treatments and the control were used to determine the OSI/SIP relationship. For the line shown in Figure 3, the coefficient of determination (r^2) is 0.89. This value indicates that there is a moderate linear relationship between OSI and SIP.

The r^2 of 0.89 for canola oil stored in the dark is slightly lower than the r^2 of 0.92 reported previously for the relationship between OSI and SIP in light-exposed soybean oil (15).

Since the food industry often needs information about oil quality within a matter of hours instead of days or weeks, the results of this study suggest the use of OSI to predict oxidative stability is probably warranted. However, OSI may not be ideal for more exacting research, such as kinetic shelf life studies, regarding canola oil.

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