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ORIGINAL PAPER

Determination of Deep Frying Soybean Oil Disposal Point by a Sensory Method

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Abstract Measurements of degradation in frying oils are mainly based on physico-chemical properties. Total polar compounds (TPC) and free fatty acids (FFA) content in frying oils are used as a guide for discarding used oils. The purpose of this study was to evaluate the efficacy of a sensory method in detecting degradation in soybean oils used in potato chips deep frying. The sensory evaluation of oil samples was determined by a trained panel; after rigorous selection and training steps. Free fatty acid, TPC and Rancimat induction period (IP) were quantified in the same samples. The proposed sensory method was sensitive to small differences in rancidity. The selected and trained sensory panel discarded oil samples with 0.175% FFA as oleic acid, 18.92% TPC, and 0.20 h IP. According to the results achieved in this research sensorial trained panel response is sensitive and accurate in refusing deteriorated frying oils. Besides this, soybean oil can be used for deep frying procedures and safely discarded according to the panel response, although presenting up to 7% linolenic acid.

Keywords Oxidation · Sensory analysis · Soybean oil · Polar compounds · Deep fat frying

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Introduction

Lipid oxidation is the cause of important deteriorative changes in edible oils and fatty foods since it affects their chemical, sensory, and nutritional properties [1]. The consumption of fried foods has increased in recent years, causing a higher intake of fats and oils that have been subjected to high temperatures in the frying process. This choice has been influenced by social and economic reasons, as people have less time to prepare their food and frying provides a fast alternative in food preparation, adding pleasant flavors [2]. Part of the oil used as a means of heat transfer is absorbed by the food, ranging from 5 to 40%, making it an ingredient of the final product [3]. Studies have shown that overconsumption of fried foods is a health risk, possibly by the toxicity of products formed during frying [4]. Abused oils intake can provide a source of *trans* fatty acids and loss of essentials fatty acids which promote higher risks of atherosclerosis [5]. The assessment and identification of numerous compounds formed in fats and oils during deep frying is of great interest to researchers, consumers, and industries.

The correct identification of the disposal point of frying oil is economically important because it implies a higher cost in the food preparation due to early discharge or quality loss of fried food when disposal occurs after the desirable endpoint. The first concerns on the quality of frying oil occurred in the mid-1970s, by means of the German Society for Fat Research pointing out the quantification of oxidized fatty acids in different materials. The point of discharge of oil or fat was determined by subjective features, such as the presence of unacceptable odors and flavors, high viscosity or a smoke point lower than 170 °C [6, 7]. Subsequently several studies have been made and the determination of total polar compounds

(TPC), free fatty acids (FFA), polymers of triglycerides and the smoke point are widely used today [8].

Countries like Belgium, France, Germany, Switzerland, Holland and Chile already have regulations for oils used in frying. The European parameters, with minor modifications, follow the same principles adopted by the German legislation for frying oil disposal: 25–27% maximum polar compounds, a 16 units reduction in the iodine value, a peroxide index higher than 15 mequiv kg^{-1} and a free fatty acids content from 1 to 2.5% [6, 9]. Brazil has not established rules and regulations for the disposal of oils used in frying. However, the National Health Surveillance Agency [10] recommends a limit of 0.9% FFA (as oleic acid) and 25% TPC for the disposal of oil used in frying. The deep frying process is most commonly performed in Brazil with refined, bleached and deodorized soybean oil, due to its ready availability and lower prices.

Sensory attributes of edible oils must be carefully monitored to maintain the flavor quality of the oil. The AOCS-recommended score sheet for evaluating edible oil intensity is a 10-point scoring scale based on the overall flavor intensity [11]. Each number on the 10–1 scale is assigned a descriptor associated with the flavors and odors typical of each step of oxidation (10 being bland and 1 being extreme). Such scales are designed to detect large differences in flavor among oils. However, perceptible differences may sometimes be small but still important. Besides this, it is necessary to maintain a trained sensory panel to accurately distinguish differences using the ranking scale [12]. This paper presents a method for the evaluation of flavor, taste, color, and viscosity of soybean oils used in deep frying in order to determine the disposal point. Relations between the sensory results and analytical index were established.

Experimental Procedures

Materials

Freshly refined soybean oil was obtained from a commercial processor (Cargill, SP, Brazil). Raw potatoes (var. Binjen) were peeled, cut into 1-mm slices, and rinsed in cold water. Slices were dried and fried. Electric stainless steel fryers with a capacity of 3.5 and 30 L oil in direct contact with heating device without water in the bottom (Tedesco model FE 1B and FAO 30E, respectively) were used in the assays.

Thermoxidation of Soybean Oil

A 30-L quantity of oil was heated at 170 °C without food. Samples of oxidized oil were collected during the heating period and frozen in amber glass bottles until the sensory evaluation was carried out. The objective was to achieve TPC levels adopted as standard for the panelist selection phase.

Deep Frying Procedure

Potato chip portions (400–550 g) were fried in 3.5 L soybean oil at 170 °C. The frying time of each portion ranged from 8 to 15 min, according to the degree of oil use and final color of potato and oil TPC content. Fresh oil was added as makeup oil after each frying to maintain the original amount of oil in the fryer. Data of the frying process, such as oil turnover, potato chips initial and final weight, and total oil turnover were recorded. Experiments were carried out in replicate and oil samples were taken after 1 h 05 min, 2 h 05 min, 3 h 01 min, 3 h 57 min and 5 h 19 min of frying, in mean (approximately for 1, 2, 3, 4 and 5 h). Samples were stored under freezing until sensory analysis.

Analytical Methods

The free fatty acids content (determined by titration); peroxide value (determined by iodometric assay) in oil samples; and the oil content and water content in potato chips were evaluated according to AOCS methods [11]. Oil stability was determined at 110 °C on a 743 Rancimat apparatus (Metrohm, Herisau, Switzerland) according to the method 12b-92 [11]. Quantification of the total polar compounds was determined according to the 2.507 IUPAC method [13] modified by Pérez-Camino et al. [14]. Nonpolar and polar fractions were separated from a 1-g of oil sample by silica column chromatography (20 g silica/H_2O, 95:5 w/w). The nonpolar fraction content was eluted with 150 mL *n*-hexane/diethyl ether (90:10, vol/vol). A second fraction, comprising the total polar compounds, was eluted with 150 mL diethyl ether.

Sensory Analysis

Panel Selection

Two steps were implemented using a simple triangular test to evaluate color, aroma, viscosity and flavor of thermo-oxidized oil samples with different contents of total polar compounds. The analyses were performed from the highest to the lowest oxidation degree, so that all candidates for panelists ($n = 126$) were subjected to six possible combinations among the samples [15]. Fresh oil was comparatively analyzed against two different thermo-oxidized oils (12.50% TPC and 6.60% TPC). Each attribute (color, aroma, viscosity and flavor) was analyzed separately.

Questionnaires were applied and each panelist's response was recorded in a simple triangular test, when tasting from left to right. During the panelist selection, each person received the same instructions. For the evaluation of color, the oil samples were presented in the Petri dishes in equal volumes, covered with aluminum foil, at room temperature over a blank sheet under white light, properly labeled with random three-digit numbers. The aroma, viscosity and flavor of oil samples were evaluated under red light, in order to mask the visible differences between the samples, and served at 43 °C, in order to favor differentiation. Aroma evaluation samples were served in 125-mL glass flasks, covered with aluminum foil and labeled with random three-digit numbers. For the evaluation of viscosity and flavor, the samples were served in 50 mL clear glasses covered with aluminum foil, labeled with random three-digit numbers, accompanied by a glass of water, paper napkin and a bland salted biscuit at room temperature for cleaning the palate. Expectorating cups were also provided to panelists during each session. Sessions were conducted in a sensory panel room (22 ± 2 °C) equipped with individual booths. At each phase, the panelist evaluated samples on four characteristics (color, aroma, viscosity and flavor) on the same day, being invited to return to the Laboratory of Sensory Analysis for the same test five more times. Responses of both phases of tests were used for evaluate the panelist's performance (Fig. 1).

Panel Training

The selected panelists for the sensory panel ($n = 15$) underwent a 3-day training session to become familiar with

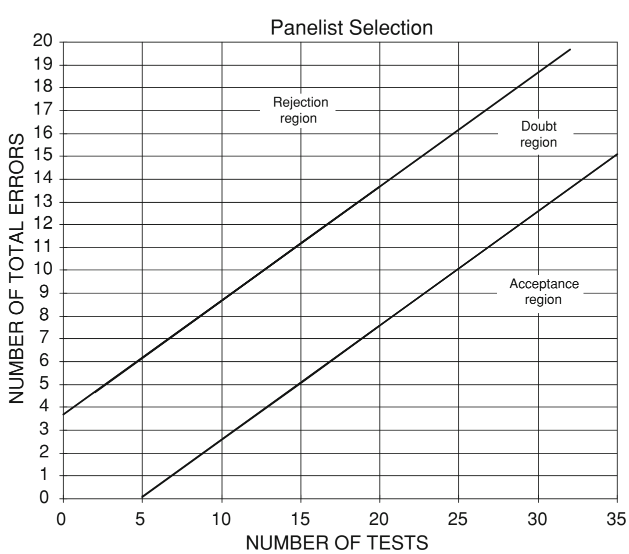

Fig. 1 Chart for selection of panelists by the sequential analysis ($\alpha = 0.01$; $\beta = 0.05$; $P0 = 35\%$; $P1 = 65\%$)

soybean oil with different degrees of oxidation. During the training session, panelists received smelling and tasting instructions. Samples of fresh oil and potato chips frying oils with different TPC were used to familiarize themselves with typical odors and flavors of oxidized soybean oil and to evaluate their sensory ability. Panelists were instructed to evaluate the oil and arrange the samples in order of increasing intensity. This was done for each attribute under the same conditions used in the selection phase. For all the characteristics oil samples were placed in the lower level of sensory perception to the highest level (Fresh, 1, 2, 3, 4, 5 h), which indicated the time of frying of each sample, enabling consumers to assess and differentiate the period in which the oil presents sensory rancidity and which would be suitable for disposal. Total polar compounds (% TPC) were not displayed during training. The samples were initially compared as follows: (1) × (2), (2) × (3), (3) × (4), (4) × (5) to assess if the panelist was able to perceive difference between the oxidized samples. Then the samples of frying oil were compared to fresh, as follows: (fresh) × (1), (fresh) × (2), (fresh) × (3), (fresh) × (4), (fresh) × (5). The samples were always evaluated from right to left.

Determining the Level of Perceived Difference of Oxidation of Deep Frying Soybean Oil

The paired test of simple difference and the directional paired test were applied. Samples were presented to panelists in pairs, to evaluate color, aroma, viscosity and flavor separately (Fig. 2) under the same conditions as were presented during the selection phase. Tests were applied with fresh oil (*F*) and frying oils (1, 2, 3, 4, and 5 h) in the following order: (*F*) × (3); (*F*) × (1); (*F*) × (2); (*F*) × (4) and (*F*) × (5), with three replicate for each test. To avoid sensory fatigue, panelists evaluated oil samples in two sessions each day. Results were noted in a control sheet and evaluated according to Roessler et al. [16]. The evaluation of discharge was considered in terms of the percentage of consumers that discarded the sample and the frequency at 5% probability.

Results and Discussion

Analytical parameters of soybean oil used in deep frying of potato chips are presented in Table 1. Alteration in water and oil content in potato chips during continuous oil frying time are shown in Table 2. In the first step of panelist selection only 15 out of 126 persons detected the differences between the samples of fresh oil with 4.40% TPC (A) and thermo-oxidized oil with 12.50% TPC (C) for the six possible combinations (AAC, CAA, CCA, ACC, ACA,

Fig. 2 Sample panelist sheet

SIMPE PAIRED TEST - DIFFERENCE

Name__________________ **DATA**___/___/___

Within this pair, do you realize difference in:

COLOR

SAMPLE N⁰ Yes No

FEEDBACK :______________________________

IF THERE IS DIFFERENCE CONTINUE THE TEST, IF NOT, PLEASE STOP

DIRECTIONAL PAIRED TEST

Within this pair, please make a circle in the most OXIDIZED sample.

SAMPLE N⁰

FEEDBACK:______________________________

PAIRED TEST

Would you discard that sample?

YES NO

Table 1 Free fatty acids (FFA), total polar compounds (TPC) and induction period of soybean oil subjected to deep frying at 170 °C

Parameter	Fresh oil	Thermo-oxidized oil	Frying oil (h)				
	0 h	15 h	1	2	3	4	5
TPC[a] (%)	4.36	12.52	7.62	11.23	13.16	18.92	19.85
FFA (as oleic acid)[a] (%)	0.078	0.136	0.112	0.117	0.154	0.175	0.155
Induction period (h)	6.19	2.73	0.21	0.35	0.17	1.64	<1.60

[a] Mean values of three determinations of replicate experiments

CAC). However, after the tests, the results evaluated by sequential analysis showed that the perceived difference between the samples was easily detected. Thus, the second step of selection, in which the candidates for panelist evaluated the difference between fresh oil samples (A) and thermo-oxidized oil with 6.60% TPC (B), was performed following the six possible combinations (AAB, BAA, BBA, ABB, ABA, BAB). To select panelists, the sequential analysis was applied according to Della Modesta [15] with $\alpha = 0{,}01$, $\beta = 0{,}05$, $P\ 0 = 35\%$, $P\ 1 = 65\%$, where $\alpha =$ error type 1 (risk of selecting a bad panelist), $\beta =$ error type 2 (risk of rejecting a good panelist), $P\ 0 =$ probability the panelist to miss, $P\ 1 =$ probability the panelist to set. Figure 1 shows the graph of the sequential analysis constructed by calculating limits of acceptance and rejection. Panelists were finally selected after this second phase.

Panelists have to be highly trained, so that they are very sensitive to differences that might seem small to ordinary people. For panel training, oil samples with different concentrations of total polar compounds were used. Thus, one can verify that the panelists acknowledged differences between the oils, the time of initial oxidation perception and the time when oil should be discarded. These samples were presented to each panelist during training to verify if there was a consensus on the sensory perception of the oxidation of oils by color, aroma, viscosity and flavor evaluation. For increases in color of soybean oil, the panelists concluded, sensorially, in the ascending sequence, fresh sample (F) < thermo-oxidized sample (A) < (1 h) < (2 h) < (5 h) < (3 h) < (4 h). The sensory panelists reached a consensus that the deterioration of the oils studied, i.e. the characteristic of rancidity, was only present after 4 h of frying. Therefore, when asked about the disposal point based on the color evaluation, the immediate and only response was sample (4 h). The viscosity was then evaluated during panel training and samples were ranked in the same order observed for color. The 5 h frying

Table 2 Characteristics of potato chips fried in soybean oil

Parameter	Total frying period (h)					
	0	1	2	3	4	5
Water content (%)[a]	84.6	7.64	9.45	9.08	3.86	3.02
Oil content (%)[a]	0.52	36.29	35.87	42.39	43.59	38.76

[a] Mean values of two experiments

assay had higher turnover and the resulting oil showed a less strong color and lower viscosity. For aroma evaluation, after training the panelists ranked samples in the following order of preference: (F) < (1 h) < (A) < (2 h) and (5 h) < (3 h) < (4 h). For flavor, samples were ranked from best to worst (F) < (A) < (1 h) < (2 h) < (3 h) < (4 h) < (5 h). Thus, flavor appears to be the most suitable attribute to be used for determining the disposal point for used soybean oil. Besides this, it was the only characteristic directly correlated to TPC.

In the following test, trained panelists identified differences among fresh oil, thermo-oxidized oil and oil from potato chips deep fat frying; the first perception of rancidity, and the disposal point of an oil sample. Table 3 presents the results of the simple paired test (H_0: $P = 1$ h H_1: $P \neq 1$ h, H_0: $P = 2$ h H_1: $P \neq 2$ h, H_0: $P = 3$ h H_1: $P \neq 3$ h, H_0: $P = 4$ h H_1: $P \neq 4$ h, H_0: $P = 5$ h H_1: $P \neq 5$ h), with significant difference ($P < 0.001$) between fresh oil (F) and deep frying oil samples. When panelists compared fresh oil (F) to sample (1 h), for the characteristics of aroma, flavor and viscosity, it was observed that the frequency of yes was 29, 28 and 28, respectively, a significant difference at $P < 0.001$ between these oils [16].

The difference is proved by the frequency analysis, shown in Table 4, as result of a rigorous selection and training phase. The directional paired test (H_0: $P = 1$ h H_1: $P < 1$ h, H_0: $P = 2$ h H_1: $P < 2$ h, H_0: $P = 3$ h H_1: $P < 3$ h, H_0: P 4 h $= H_1$: $P < 4$ h, H_0: $P = 5$ h H_1: $P < 5$ h), which compare the fresh oil (F) to the frying oil samples (1 h), (2 h), (3 h), (4 h) and (5 h) showed that despite variation among sensory panelists response it is clear that sensory evaluations provide good indication of oil quality.

According to Roessler et al. [16] within 30 sensory tests carried out, the requirement is that the frequency (the darkest color, the most oxidized aroma, the most viscous and the most oxidized flavor) is 20 ($P < 0.05$), 22 ($P < 0.01$) and 24 tests ($P < 0.001$). There was a significant difference ($P < 0.001$) between fresh oil (F) and the frying oil samples, since frequency of the four characteristics evaluated was greater than 24. Regarding color and viscosity, a total of 30 analyses performed in each oil sample confirmed that used oils are darker and more viscous than fresh oil.

A larger number of panelists identified sample (4 h) as a disposable one, through the color, aroma, viscosity, and flavor. As the time of frying increased, the number of discard recommendations also increased. Thus, the time of disposal of soybean oil used in deep frying of potato chips at a maximum temperature of 180 °C was defined as 4 h by the selected and trained panelists. These samples presented 0.175% FFA, 18.92% TPC and the induction period of

Table 3 Simple paired difference test used in the sensory evaluation of potato chip deep frying oil samples by panelists

Comparison	Color				Aroma			
	Total tests	Yes frequency	No frequency	S/NS	Total tests	Yes frequency	No frequency	S/NS
Fresh oil (F) × 1 h frying oil (1 h)	30	30	0	***	30	29	1	***
Fresh oil (F) × 2 h frying oil (2 h)	30	30	0	***	30	30	0	***
Fresh oil (F) × 3 h frying oil (3 h)	30	30	0	***	30	30	0	***
Fresh oil (F) × 4 h frying oil (4 h)	30	30	0	***	30	30	0	***
Fresh oil (F) × 5 h frying oil (5 h)	30	30	0	***	30	30	0	***

Comparison	Viscosity				Flavor			
	Total tests	Yes frequency	No frequency	S/NS	Total tests	Yes frequency	No frequency	S/NS
Fresh oil (F) × 1 h frying oil (1 h)	30	28	2	***	30	28	2	***
Fresh oil (F) × 2 h frying oil (2 h)	30	29	1	***	30	30	0	***
Fresh oil (F) × 3 h frying oil (3 h)	30	30	0	***	30	30	0	***
Fresh oil (F) × 4 h frying oil (4 h)	30	30	0	***	30	30	0	***
Fresh oil (F) × 5 h frying oil (5 h)	30	29	1	***	30	30	0	***

S significant (*$P < 0.05$; **$P < 0.01$; ***$P < 0.001$), NS not significant ($P > 0.05$)

In 30 tests the frequency must be 20 ($P < 0.05$), 22 ($P < 0.01$) and 24 ($P < 0.001$)

Table 4 Directional paired difference test used in the sensory evaluation of potato chip deep frying oil samples by panelists

Comparison	Color				Aroma			
	Total tests	Frequency darker	S/NS	Discard	Total tests	Frequency oxidized	S/NS	Discard
Fresh oil (*F*) × 1 h frying oil (1 h)	30	30 (1)	***	01 S	30	30 (1)	***	02 S
Fresh oil (*F*) × 2 h frying oil (2 h)	30	30 (2)	***	07 S	30	29 (2)	***	05 S
Fresh oil (*F*) × 3 h frying oil (3 h)	30	30 (3)	***	03 S	30	30 (3)	***	08 S
Fresh oil (*F*) × 4 h frying oil (4 h)	30	30 (4)	***	20 S	30	30 (4)	***	20 S
Fresh oil (*F*) × 5 h frying oil (5 h)	30	30 (5)	***	03 S	30	30 (5)	***	09 S

Comparison	Viscosity				Flavor			
	Total tests	Frequency oxidized	S/NS	Discard	Total tests	Frequency oxidized	S/NS	Discard
Fresh oil (*F*) × 1 h frying oil (1 h)	30	30 (1)	***	0 S	30	29 (1)	***	0 S
Fresh oil (*F*) × 2 h frying oil (2 h)	30	30 (2)	***	01 S	30	28 (2)	***	0 S
Fresh oil (*F*) × 3 h frying oil (3 h)	30	30 (3)	***	03 S	30	29 (3)	***	01 S
Fresh oil (*F*) × 4 h frying oil (4 h)	30	30 (4)	***	18 S	30	30 (4)	***	23 S
Fresh oil (*F*) × 5 h frying oil (5 h)	30	30 (5)	***	05 S	30	30 (5)	***	10 S

S significant (*$P < 0.05$; **$P < 0.01$; ***$P < 0.001$), *NS* not significant ($P > 0.05$), $S =$ yes, $N =$ no

In 30 tests the frequency must be 20 ($P < 0.05$), 22 ($P < 0.01$) and 24 ($P < 0.001$)

0.20 h. For the color and aroma, the discharge frequency of sample (4 h) was 66.7%; for the viscosity, 60%; and for the flavor, 76.7%.

The analytical index of frying oil that best related to the results of sensory analysis was the FFA and the sensory characteristics most representative for assessing the degree of oxidation of frying oil was the taste. Oils with long periods of frying, with replacement of pure oil, presented similar acceptance to oils with short periods of frying.

The results shown in this paper clearly indicate that it is possible to apply sensory analysis to frying oils quality. This is the first paper that has reached this conclusion. This research strongly indicates that European Standards for frying oils disposal point determination, based on analytical indexes like total polar compounds and acidity, could be reviewed. The proposed sensorial method can be used as a reference for training people who prepare food, thus assuring food safety for frying procedures.

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