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Degradation and Nutritional Quality Changes of Oil During Frying

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Abstract The changes in regular canola oil as affected by frying temperature were studied. French fries were fried intermittently in canola oil that was heated for 7 h daily over seven consecutive days. Thermo-oxidative alterations of the oil heated at 185 ± 5 or 215 ± 5 °C were measured by total polar components (TPC), anisidine value (AV), color components formation, and changes in fatty acid composition and tocopherols. Results showed that TPC, AV, color and *trans* fatty acid content increased significantly ($P < 0.05$) as a function of frying temperature and time. The oil polyunsaturated fatty acids (PUFA) decreased in direct proportion to frying temperature and time. After 7 days of frying, the amount of PUFA was reduced by half and the *trans* isomers contribution increased 2.5 times during frying at 215 °C. Of the parameters assessed, total polar component and color had the highest correlation, with correlation coefficients of 0.9650 and 0.9302 for frying at 215 and 185 °C, respectively. TPC formation correlated inversely with the reduction of tocopherols.

Keywords Canola oil · Frying performance · Total polar component · Anisidine value · Color · Frying temperature · Tocopherols · French fries · Fatty acids

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Introduction

Deep-fat frying is probably one of the most dynamic processes in all of food processing. Essentially, the process involves immersing a food item in a large quantity of heated oil or fat, which is normally replenished and reused several times before being disposed. Deep-fat frying produces a product with desired sensory characteristics, including fried food flavor, golden brown color, and a crisp texture [1].

Most frying operations are conducted at temperatures of 175–195 °C, nevertheless German regulations allow maximal frying temperatures of up to 165 °C, to limit formation of acrylamides [2]. Extruded products and pellets are typically fried at 190–215 °C [2]. This high temperature requirement and the presence of air and moisture, from the food, initiate several chemical and physical changes affecting oxidative degradation of oil used. Published studies described chemical reactions involved and various volatile and non-volatile oxidation products were identified [3–6]. The chemical changes in the frying fats also affect the physical characteristics of the oil and fried product [7]. For instance, the color of frying oil was reported to darken as a result of oxidation and the formation of browning pigments when potato chips were fried [8, 9].

A number of studies have been undertaken to assess various chemical reactions and extent of oxidative deterioration as affected by frying temperature, many of the published data were obtained by heating an oil but not during actual frying [10–12]. Meanwhile, it has been observed that the chemical reactions that take place during deep-fat frying are different from those during continuous heating [13, 14]. Besides, different oils have been found to behave differently regarding the rate of formation of polar components and secondary oxidation products. Guillen and Cabo [15] reported that secondary products were formed

immediately after hydroperoxide formation in olive and rapeseed oils, whereas in sunflower and safflower oils, secondary products were formed when the concentration of hydroperoxides reached level of 180 and 270 meq/kg, respectively. Consequently, the need to study the frying performance of individual oil as a function of frying temperature during actual frying of food becomes imperative.

Oxidized short-chain fatty acids are secondary oxidation products formed through thermal degradation of lipid hydroperoxides. Recently, much concern has been on the biological effects of oxidized lipids, and there is increasing evidence that they may be detrimental to health, especially in connection with the development of atherosclerosis, liver damage, and promotion of intestinal tumors [16].

The objective of this study was to evaluate the effect of frying temperature on the degradation of canola oil by monitoring the accumulation of total polar components, oxidized short-chain fatty acids, polymers formation, *p*-anisidine value, color components formation, changes in fatty acid composition, and tocopherol contents.

Materials and Methods

Materials

Oil and French Fries

Commercially refined regular canola oil without antioxidants added was obtained from Richardson Oilseed Processing (Lethbridge, Canada). Frozen par-fried French fries in an institutional pack were obtained from a local food store.

Chemicals

All solvents and chemicals of analytical grade used in this study were purchased from Sigma–Aldrich (St Louis, MO). Standards of tocopherols were obtained from Calbiochem–Novabiochem (San Diego, CA). Standards of fatty acid methyl esters were purchased from Nu-Check-Prep (Elysian, MN).

Frying Procedure and Oil Sampling

The frying was simultaneously conducted in two 8-L capacity restaurant style stainless steel deep fryers (General Electric Company, New York, USA). Regular canola oil (3.75 L) was heated at 185 ± 5 and 215 ± 5 °C for 7 h daily for 7 days. A batch of 200 g of frozen French fries was fried for 5 min for a total of eight batches per frying day. At the end of each frying day, fryers were shut off and left to cool overnight. Two 25-mL samples of oil from each of the fryers were taken daily and kept frozen at −16 °C until analyzed. Before commencing frying each day, oils were filtered to remove solid debris. Oil was replenished every second day of frying with 500 mL of fresh oil.

Fatty Acid Analysis

Fatty acids were methylated following the AOCS Official Method Ce 1-62 [17]. The resulting fatty acid methyl esters (FAME) were analyzed on Trace GC Ultra gas chromatograph (Thermo Electron Corporation, Rodano, Italy) using a Trace TR-FAME fused silica capillary column (100 m × 0.25 mm × 0.25 μm; ThermoFisher Scientific, Waltham, MA, USA). Hydrogen was used as the carrier gas with a flow rate of 1.5 mL min^{-1}. The column temperature was programmed from 70 to 160 °C at 25 °C min^{-1} and held for 30 min, and further programmed to 210 °C at 3 °C min^{-1}. Starting and final temperatures were held for 5 and 30 min, respectively. Splitless injection was made using a PTV injector. Detector temperature was set at 250 °C. FAME samples, 1 μL, were injected with an AS 3000 autosampler (Thermo Electron Corporation, Rodano, Italy). Fatty acids were identified by comparison of retention time with authentic standards. Oxidized short-chain fatty acids methyl esters (OFAME) were identified as a group as described by Velasco et al. [18]. *Trans* isomers of fatty acids were assessed according to ISO method 15304.

Total Polar Compounds and Anisidine Value

TPC were determined by gravimetric method after column chromatography separation of non-polar fraction following AOAC Method 982.27 [19]. Polar components were eluted from the column with diisopropyl ether and further analyzed for polar components composition by size exclusion chromatography.

Anisidine values (AV), a measure of secondary oxidation products, was determined according to ISO Method 6885:2004 [20].

Tocopherols

Tocopherols were analyzed by AOCS Official Method Ce 8-89 [17]. Briefly, oil samples (75 mg) were weighed directly into vials and dissolved in 1.5 mL hexane. Analysis was performed on a Finnigan Surveyor liquid chromatograph (LC) (Thermo Electron Corporation, Rodano, Italy) with a Finnigan Surveyor Autosampler Plus and Finnigan Surveyor FL Plus fluorescence detector, set for excitation at 292 nm and emission 394 nm. The column was a normal-phase Microsorb 100 silica column (3 μm; 250 × 4 mm; Varian, CA). Of each sample, 10 μL was injected. Mobile phase consisted of 7% methyl-*tert*-butyl-

ether in hexane with a flow rate of 0.6 mL/min. The amounts of tocopherols were quantified using calibration curves for each isomer separately.

Size Exclusion Chromatography

The composition of polar components was analyzed using high performance size exclusion chromatography (HPSEC) according to ISO Method 16931 [21]. Separation was performed on a Finnigan Surveyor LC. Components were separated on three size exclusion columns in series (Phenogel 500 Å, 100 Å and 50 Å, 5 µm, 300 × 4.60 mm; Phenomenex, Torrance, CA), with tetrahydrofuran (THF) as the mobile phase at a flow rate of 0.3 mL/min, and column temperature of 30 °C. A 10 µL sample was injected, and components were detected with a Sedex 75 evaporative light scattering detector (Sedere, Alfortville, France), operated at 30 °C with an air pressure of 2.5 bar. Polar components were identified according to the method described by Marquez-Ruiz et al. [22].

Color Analysis

Color of the frying oils was determined according to AOCS Official method Cc 13c-50 [17] using a DU®-65 spectrophotometer (Beckman, Fullerton, CA).

Statistical Analysis

Samples from three repetitions of frying at each temperature were collected and were analyzed in triplicate. Data are presented as mean values ± SD. Data were analyzed by single factor analysis of variance (ANOVA) and regression analyses using Minitab 2000 statistical software (Minitab Inc., PA, ver. 13.2). Statistically significant differences between means were determined by Duncan's multiple range tests. Statistically significant differences were determined at the $P < 0.05$ level.

Results and Discussion

The fresh oil had 0.06% of free fatty acids (FFA), 1.0 meq/kg of peroxide value (PV), 4.2% of polar components and an AV at the level of 4.2, indicating good quality oil [23]. Thus, changes in these values during frying would indicate a degradation in oil quality.

Total Polar Compounds

The determination of TPC in frying oil provides the most reliable measure of the extent of oxidative degradation [14, 24]. In this study, the contents of TPC increased almost linearly with the frying time at a rate affected by frying temperature (Fig. 1). Total polar content during frying at 185 °C was 19.8% at the end of frying time, which was still below the 24% oil discard level set in many European countries [25]. However, the total amount of polar components reached the discard level after 4 days of frying at 215 °C. The TPC reached 38% by the end of the frying time. The extent of oxidative deterioration, as measured by TPC formation, was 2.6 times faster during frying at 215 °C compared to 185 °C.

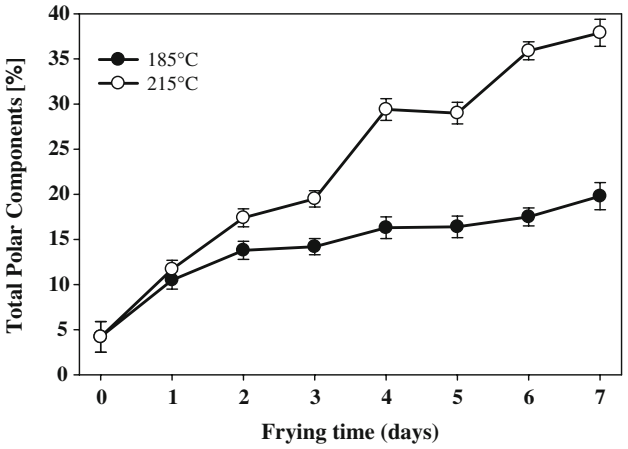

Fig. 1 Changes in polar components during frying at different temperatures. *Error bars* indicate significant differences ($P < 0.05$) in polar compounds between temperatures

Composition of Polar Components

The composition of polar compounds formed during frying was analyzed using HPSEC. Diacylglycerides (DG), oxidized triacylglycerides (OTG), dimers and polymers were separated, and their contribution calculated using peak area. The contribution of polymers in total polar material increased consistently with frying time at both frying temperatures achieving maximum values of 8 and 15.6% for frying at 185 and 215 °C, respectively (Figs. 2 and 3). The amount of polymers generated at 185 °C at the end of the 7 days frying period was comparable to the third day of frying at 215 °C. Comparable increase in the amount of dimers for oil fried at 185 °C was observed throughout the frying period (Fig. 2). However, when frying at 215 °C, a 16-fold increase in the contribution of dimers at the end of first day followed by slight increase for the next 2 days of frying, then decreased until the end of the frying period (Fig. 3). This is probably due to the conversion of the dimers to polymers and thermal degradation of these components [16]. As expected, the contribution of OTG decreased consistently over the frying period at both tested temperatures, as a consequence of thermal degradation. However, a more pronounced decrease in the contribution

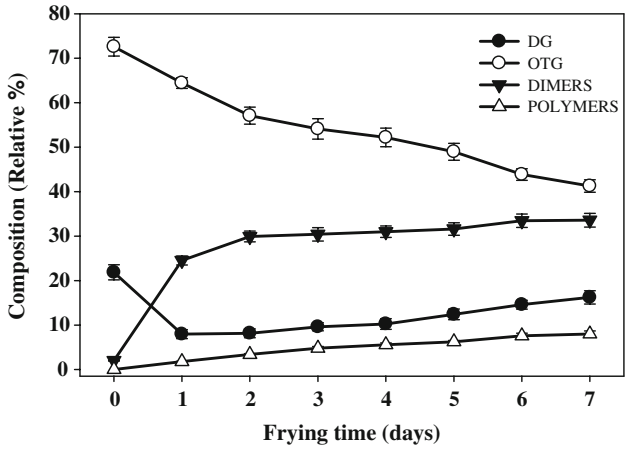

Fig. 2 Changes in composition of polar components during frying at 185 °C. *DG* diglycerides, *OTG* oxidized triacylglycerols, *dimers* dimers of triacylglycerols. *Error bars* indicate significant differences ($P < 0.05$) in polar compounds between temperatures

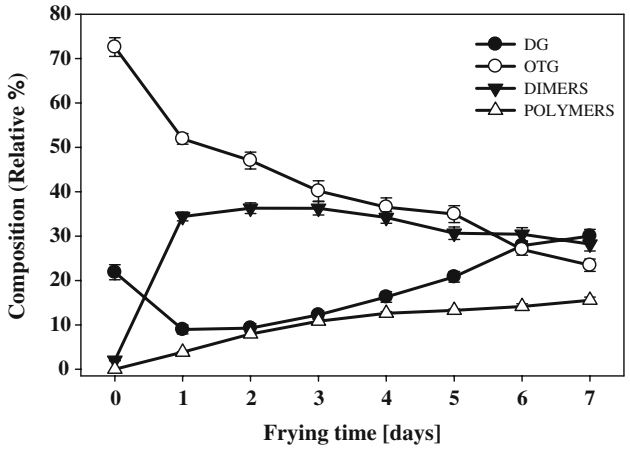

Fig. 3 Changes in composition of polar materials during frying at 215 °C. For abbreviations see Fig. 2. *Error bars* indicate significant differences ($P < 0.05$) in polar compounds between temperatures

of OTG, 1.5 times faster degradation was observed during frying at 215 °C (Fig. 3).

Anisidine Values

Aldehydes formed during oxidative degradation are secondary decomposition products, and the non-volatile portion of carbonyls remains in the frying oil [4, 13]. At the two testing temperatures, AV was not well correlated with frying time (Fig. 4). The maximum was reached on the second day of frying for both frying temperatures and then decreased consistently until the end of frying time. Apparently, oil replenishment played some role in the changes in carbonyls content but elevated temperatures was probably the main cause for the reduction in the amount of these labile and reactive components with time [26]. This result could be

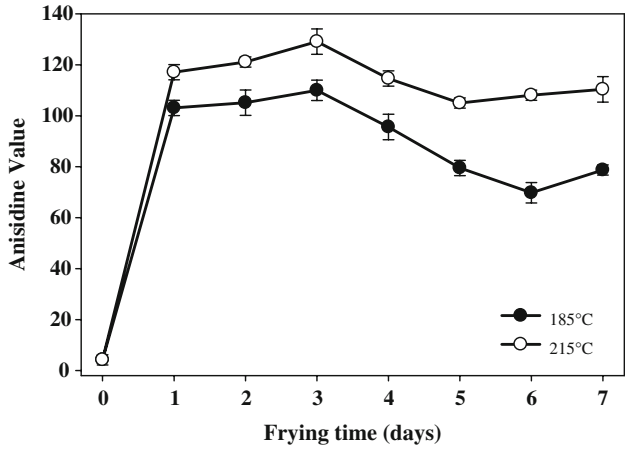

Fig. 4 Changes in anisidine values during frying at different temperatures. *Error bars* indicate significant differences ($P < 0.05$) in polar compounds between temperatures

explained by the thermal degradation of the aldehydes formed at higher temperature, which results in a lower accumulation in the oil at the higher frying temperature. Regardless of the general knowledge that the decomposition of hydroperoxides increases with increasing temperature and potentially the amount of carbonyls, AV followed an opposite trend in the frying tests. Carbonyl chemical reactivity, involvement in the formation of other compounds and thermal decomposition explains decreasing in AV [10, 25]. Houhoula et al. [27] reported a significant increase in AV as a function of temperature during frying potato chips in cottonseed oil. Thus, the initial increase in AV observed (Fig. 4) agrees with these authors. Furthermore, the increase was also observed as a function of temperature.

Fatty Acid Composition

The fatty acid composition of the fresh canola oil and the resulted changes during the 7 days of frying at 185 and 215 °C are presented in Table 1. The results indicate a progressive decrease in both linoleic and linolenic acids contributions throughout the frying period [24]. Linoleic acid decreased by 8.5 and 13.3% during frying at 185 and 215 °C, respectively. The deterioration of linolenic acid was more pronounced and was reduced by 24.0 and 47.1% during frying at 185 and 215 °C, respectively. White et al. [24] reported decreases of 7–11.5% in linoleic acid and 27–46% in linolenic acid when soybean oils were heated at 180 °C for 40 h.

The amount of *trans* fatty acids formed during frying increased when temperature and time increased (Figs. 5 and 6). At the lower frying temperature applied, the amount of *trans* isomers increased from 2.4 to 3.3%. The increase in frying temperature to 215 °C caused extensive *trans* isomerization of fatty acids (Figs. 5 and 6). The total

Table 1 Changes in contribution of canola oil fatty acid at different frying temperatures

Frying time (h)	Contribution[a] (relative percentage)						
	$C_{16:0}$	$C_{18:0}$	$C_{18:1}$	$C_{18:2}$	$C_{18:3}$ α	$C_{18:2}/C_{16:0}$	$C_{18:3}/C_{16:0}$
185 °C							
0	4.00 ± 0.01	1.82 ± 0.02	60.03 ± 0.72	18.91 ± 0.16	8.40 ± 0.09	4.73 ± 0.04	2.10 ± 0.02
7	4.14 ± 0.03	1.91 ± 0.06	61.15 ± 0.61	18.10 ± 0.10	7.46 ± 0.05	4.37 ± 0.02	1.80 ± 0.04
14	4.22 ± 0.02	2.01 ± 0.03	61.35 ± 0.86	18.01 ± 0.16	7.16 ± 0.13	4.26 ± 0.05	1.70 ± 0.03
21	4.24 ± 0.07	2.01 ± 0.04	61.78 ± 0.64	17.90 ± 0.21	7.11 ± 0.11	4.22 ± 0.08	1.68 ± 0.02
28	4.25 ± 0.08	2.02 ± 0.08	61.96 ± 0.95	17.84 ± 0.21	6.85 ± 0.09	4.20 ± 0.05	1.61 ± 0.03
35	4.27 ± 0.06	2.02 ± 0.03	61.97 ± 0.91	17.85 ± 0.18	6.78 ± 0.08	4.18 ± 0.08	1.59 ± 0.02
42	4.30 ± 0.08	2.02 ± 0.04	61.98 ± 0.84	17.81 ± 0.22	6.56 ± 0.11	4.14 ± 0.08	1.53 ± 0.03
49	4.46 ± 0.05	2.03 ± 0.04	61.98 ± 0.68	17.27 ± 0.17	6.39 ± 0.10	3.87 ± 0.06	1.43 ± 0.02
215 °C							
7	4.19 ± 0.03	1.93 ± 0.04	61.20 ± 0.78	17.92 ± 0.19	6.79 ± 0.08	4.28 ± 0.05	1.62 ± 0.03
14	4.27 ± 0.05	1.99 ± 0.05	61.77 ± 0.97	17.37 ± 0.17	5.68 ± 0.06	4.07 ± 0.06	1.33 ± 0.04
21	4.34 ± 0.10	2.02 ± 0.03	62.37 ± 1.01	17.03 ± 0.18	5.13 ± 0.08	3.93 ± 0.04	1.18 ± 0.02
28	4.37 ± 0.09	2.04 ± 0.06	62.43 ± 0.77	16.43 ± 0.19	4.47 ± 0.05	3.76 ± 0.09	1.01 ± 0.01
35	4.45 ± 0.11	2.06 ± 0.05	63.16 ± 0.63	16.25 ± 0.17	4.37 ± 0.07	3.65 ± 0.05	0.98 ± 0.01
42	4.61 ± 0.06	2.11 ± 0.06	63.46 ± 0.72	15.90 ± 0.18	3.84 ± 0.05	3.44 ± 0.06	0.83 ± 0.01
49	4.81 ± 0.09	2.18 ± 0.09	63.50 ± 0.98	15.54 ± 0.23	3.59 ± 0.04	3.23 ± 0.03	0.75 ± 0.01

[a] All values are averages of triplicate analyses

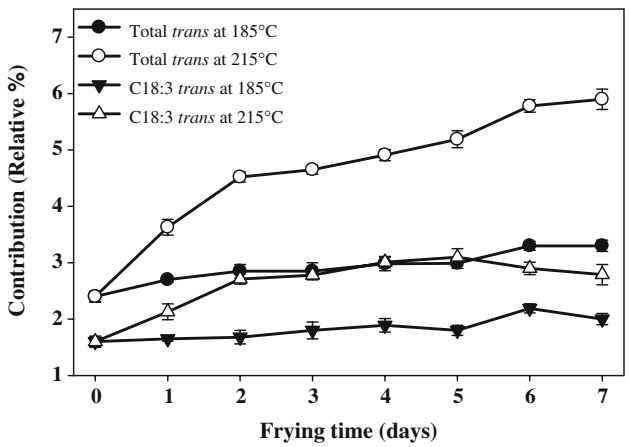

Fig. 5 Changes in total and linolenic acid *trans* isomers amounts during frying at different temperatures. *Error bars* indicate significant differences ($P < 0.05$) in polar compounds between temperatures

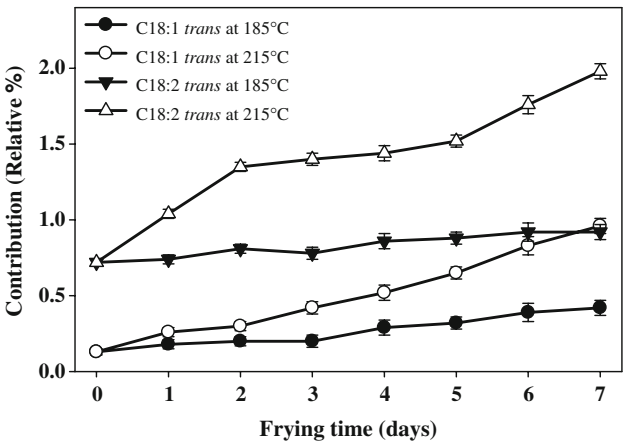

Fig. 6 Changes in oleic and linoleic acids *trans* isomers contribution during frying at different temperatures. *Error bars* indicate significant differences ($P < 0.05$) in polar compounds between temperatures

contribution of *trans* isomers in oil increased 2.5-fold, from 2.4 to 5.9% (Fig. 5). This indicates the importance of temperature on *trans* isomers formation during frying, and explains the amount of *trans* isomers observed in the initial oil (Fig. 5). The deodorization of canola oil is usually performed at temperatures above 200 °C under vacuum, where the main amount of *trans* isomers is formed [28]. The amount of individual *trans* fatty acids decreased in the following order: linolenic > linoleic > oleic (Figs. 5, 6). The quantity of *trans* isomers formed at elevated temperature indicates that a specific amount of energy is required to transfer double bonds from *cis* to *trans* configuration. Data from this work are supported by published results, confirming that activation energy for isomerization decreasing when the numbers of *cis* double bonds increases [29].

Increasing amounts of *trans* isomers during frying at higher temperature, can have practical implications related to nutritional claims about zero *trans* content in a serving portion of fried products. When the amount of these isomers increases 2.5-fold during frying at the higher temperature, then the amount of *trans* isomers in fried products will increase by the same amount and may exceed

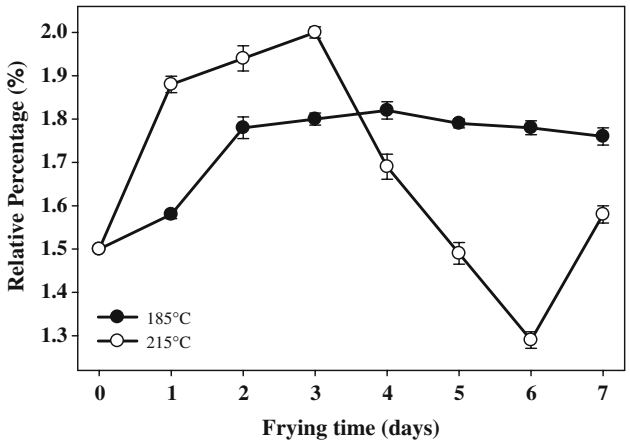

Fig. 7 Changes in oxidized fatty acids content during frying at different temperatures. *Error bars* indicate significant differences ($P < 0.05$) in polar compounds between temperatures

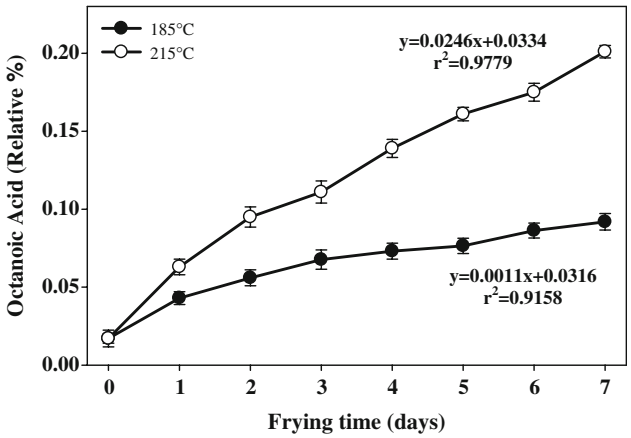

Fig. 8 Changes in octanoic acid content during frying at different temperatures. *Error bars* indicate significant differences ($P < 0.05$) in polar compounds between temperatures

the specified definition limit, making the claim for zero *trans* fat invalid. The amount of *trans* isomers in oil can affect the *trans* level in fried product due to fast exchange of fats during frying. We observed that the oil in fried food had a similar composition of fatty acids as frying oil, even when par-frying was done in different oil (data not included). These data clearly indicate the importance of controlling frying temperature and keeping it below 190 °C.

The ratio of linoleic acid to palmitic acid ($C_{18.2}/C_{16:0}$) has been suggested as a valid indicator of the level of PUFA deterioration [34]. Our result showed a decrease in this ratio from 4.73 to 3.87 and 4.28 to 3.23 during frying at 185 and 215 °C, respectively (Table 1). This implies that the decrease in this ratio was 1.2 times greater in oil heated at 215 °C as compared to 185 °C. Örnal and Ergin [30] reported a decrease in the ratio from 4.04 to 3.49 at the end of frying time. Houhoula et al. [27] reported a reduction of the ratio from 2.39 to 2.03 for cottonseed oil heated at 185 °C for 12 h. The decrease in the ratio of linolenic acid to palmitic acid was more pronounced, reducing it 1.9 times faster in oil heated at 215 °C compared to 185 °C (Table 1).

Short-chain glycerol-bound aldehydes, acids, ketones and alcohols are non-volatile secondary oxidation products formed during oxidative degradation of lipids [18]. They are of particular chemical and nutritional interest since they remain in the frying oil, and are absorbed and subsequently ingested. Analysis of the oxidized short-chain fatty acid methyl esters (OFAME) as a group revealed a consistent increase in their contribution for the first 5 days of frying at 185 °C, reaching a maximum at 1.83% (Fig. 7). However, for the oil heated at 215 °C, the amount of oxidized fatty acids increased in the first 3 days of frying with the maximum at 2.20%. Thereafter, a decrease for the next 3 days of frying was observed. Velasco et al. [31] observed that the amount of polar FAME decreased drastically as compared to the amount of total polar compounds in used frying fats.

Peers and Swoboda [32] suggested the quantification of methyl octanoate, product of linoleic acid degradation, as an oxidation index during frying. In this study, the amount of octanoic acid increased significantly ($P < 0.05$) as a function of frying temperature (Fig. 8). The increase in frying temperature from 185 to 215 °C resulted in a twofold higher contribution of octanoic acid at the end of frying time compared to lower frying temperature. Slope of regression shows that oxidative degradation of linoleic acid was 2.5 times faster at 215 °C, compared to 185 °C.

Color Analysis

A significant ($P < 0.05$) effect of frying temperature on formation of color components in the oil was observed. Over 100% increase in the optical density of frying oil was observed with the 30 °C increase in frying temperature (Fig. 9). The result indicated that the color at the seventh day of frying at 185 °C was comparable to the color of the oil at the fourth day of frying at 215 °C. After 70 h frying at 170, 180 and 190 °C in different oils, it was observed that color changes were influenced by frying temperature rather than frying medium [33].

Tocopherols

The tocopherol profile of the fresh canola oil used in this study was found to be: 214 μg/g of α-tocopherol, and 347 μg/g of γ-tocopherol. Tocopherols degradation increased as a function of frying temperature. At the end

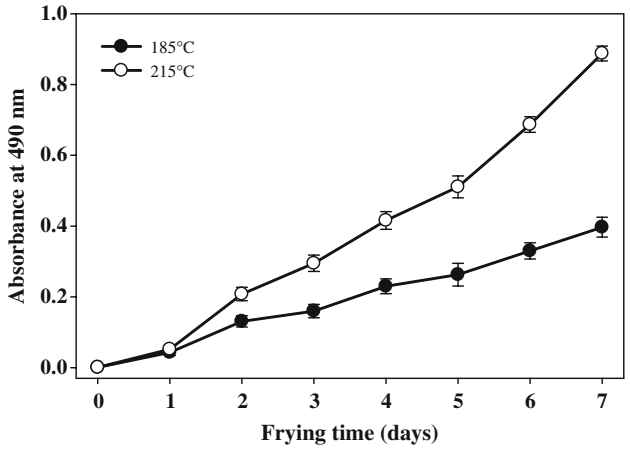

Fig. 9 Changes in oil color during frying at different temperatures. *Error bars* indicate significant differences ($P < 0.05$) in polar compounds between temperatures

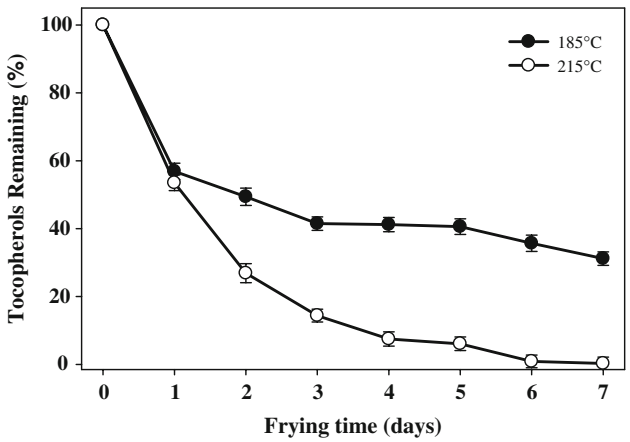

Fig. 10 Total tocopherols remaining over frying time at different temperatures. *Error bars* indicate significant differences ($P < 0.05$) in polar compounds between temperatures

of the seventh day of the frying period, approximately 31% of the total tocopherols remained after frying at 185 °C (Fig. 10). For frying at 215 °C the entire amount of tocopherols was gone at the end of sixth day of frying. The calculated half life of tocopherols for oil heated at 185 °C was 8 h while for frying at 215 °C was 5.3 h. Consistent with previously published results [35], a strong inverse relationship was observed between TPC formation and the reduction of tocopherol at both frying temperatures. In this study, γ-tocopherol degraded at a faster rate than α-tocopherol at the lower frying temperature, but the order was reversed during frying at 215 °C (data not shown).

Correlation Between Assessment Parameters

Although TPC remains the best assessment parameter for evaluating frying oil performance and oxidative stability, a faster and yet objective alternative is desirable. Assessment methods that correlate well with TPC may provide the much needed alternative. In this study, a low correlation was found between AV and TPC, and AV and color at both frying temperatures (Table 2). However, a good correlation was observed between color and TPC at both frying temperatures (Table 2). Lopez-Varela et al. [35] reported a correlation coefficient of 0.885 between color and TPC for sunflower oil used in 75 successive fryings of potatoes. In summary, the effect of temperature and frying time on performance of canola oil as measured by TPC, AV, fatty acid composition, tocopherols amount and color was significant.

Conclusions

The rate of the thermal and oxidative degradation of PUFA was drastically higher at the elevated temperature tested, forming larger amounts of components with potential detrimental health effects. This study showed that increasing frying temperature above 195 °C can cause intensive isomerization of PUFA and the amount of *trans* isomers may increase above the threshold level described by "zero trans definition" annulling the *trans* fat free claim for fried product.

Table 2 Correlation coefficient for some assessment parameters at frying temperatures

	TPC 185 °C	AV 185 °C	Polymers 185 °C	$C_{18:2}$ 185°C	$C_{18:3}$ 185 °C	TPC 215 °C	AV 215 °C	Polymers 215 °C	$C_{18:2}$ 215 °C	$C_{18:3}$ 215 °C
Color	0.9302	0.6281	0.9688			0.9650	0.8350	0.9818		
AV	0.8609		0.7226			0.5554		0.5719		
Tocopherol reduction	0.9540		0.8728			0.9313		0.9600		
C_8				0.9388	0.9928				0.9596	0.9987

For correlation coefficients calculation statistical significance at $P < 0.05$ was applied

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