

Comparison of the Frying Performance of Olive Oil and Palm Superolein

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Abstract: Deep-fat frying is an important method of food preparation in which foods are immersed in hot oil. Repeated use of frying oils is a common practice, and in the presence of atmospheric oxygen it produces various undesirable reactions in used oils. Stable frying oils usually require low linolenic acid (LnA < 3%), increased oleic acid (OA > 40%), and decreased linoleic acid (LA < 50%). The aim of this study was to establish the behavior of palm superolein (PSO) (OA 45%; LA 12.5%; LnA 0.2%) and olive oil (OO) during repeated, discontinuous deep frying of French fries. The behavior of the oils under controlled heating conditions was also studied by maintaining all of the process variables the same as those in deep frying, except that there was no food in the oil. The PSO selected to be tested in this study may represent an alternative to OO as a frying medium. Although PSO presented a faster increase in some oxidation indices, such as free fatty acid and total polar compounds, for other indicators, PSO showed better behavior than OO (less formation of C8:0 and lower peroxide value).

Keywords: alka-2,4-dienals, frozen French fries, head space analysis, high-resolution gas chromatography, intermittent deep fat frying, total polar compounds, volatile organic compounds

Practical Application: The super palm olein selected for use in this study can be suggested as a suitable replacement for olive oil for frying and cooking purposes because it provides higher oxidative stability besides the beneficial effects of olive oil on human health. In fact, several studies have indicated that palm oil exhibits similar frying performance to *high-oleic* oils, with the advantages of greater availability in the market and a lower price.

Introduction

Frying is a process that involves immersing food in hot oil with contact among oil, air, and food at a high temperature (150 to 190 °C). The simultaneous heating and mass transfer of oil, food, and air during deep-fat frying produces the desirable and unique quality of fried foods. Frying oil acts as a heat transfer medium and contributes to the texture and flavor of fried food. Its success comes from its versatility and flexibility at industrial and household scales. Additionally, it offers several advantages that are difficult to reproduce with alternative technologies: very efficient heat transfer, the crispy texture associated with rapid drying, and good-tasting fried products (Raoult-Wack and others 2000; Pedreshi and Moyano 2005). However, frying has several limitations, including those associated with (i) the difficulty of orienting independently heat- and water-mediated phenomena that occur simultaneously during frying and (ii) increased consumer demand for low-fat products.

The hydrolysis, oxidation, and polymerization of oil are common chemical reactions in frying oil and produce volatile and nonvolatile compounds. A few chemical changes are necessary so that food will have a golden-brown color and deep-fried flavor. The frying temperature and time, frying oil, antioxidants present, and type of fryer used affect the hydrolysis, oxidation, and poly-

merization of the oil during frying. In particular, frying time increases the content of free fatty acids (FFAs) (Mazza and Qi 1992), polar compounds, such as triacylglycerol dimers and oxidized triacylglycerols (Romero and others 1998; Xu and others 1999), dimers (Gordon and Kourimska 1995), and polymers (Tompkins and Perkins 2000).

A high frying temperature accelerates the thermal oxidation and polymerization of oils (Fedeli 1988; Blumenthal 1991; Tyagi and Vasishta 1996). Additionally, a high frying temperature decreases the formation of polymers with peroxide linkages and increases polymers with ether linkages or carbon-to-carbon linkages. The intermittent heating and cooling of oils causes greater deterioration of oils than continuous heating because of increased oxygen solubility in the oil when the oil cools down from the frying temperature (Clark and Serbia 1991).

Regarding the quality of frying oil, FFAs increase the thermal oxidation of oils, and their unsaturation, rather than chain length, leads to significant effects on thermooxidative degeneration.

Stevenson and others (1984) and Warner and others (1994) have reported that the oxidation rate of oil increases as the content of unsaturated fatty acids in frying oil increases. In particular, the content of linolenic acid is critical for frying performance, the stability of oil, and the flavor quality of fried food (Liu and White 1992; Xu and others 1999).

The type of fryer used affects frying oil deterioration. A rapid heat transfer to oil can prevent the formation of hot spots and oil scorching. Polymerized fat deposited in the fryer causes gum formation, formation of foam, darkening of oil color, and further deterioration of frying oil. A small surface-to-volume ratio of a

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fryer to achieve minimum contact of oil with air is recommended for deep-fat frying (Negishi and others 2003).

Because of these modifications, deep-fat frying produces desirable or undesirable flavor compounds and changes the flavor stability and quality, color, and texture of fried foods as well as their nutritional quality.

Oil that has just reached frying temperature will not produce the color and flavor typical of oil that has been conditioned or heated for several hours before frying. During deep-fat frying, oils decompose to form volatile products and nonvolatile monomeric and polymeric compounds. The increases and decreases in these compounds and variations in the physical properties of oil during frying differ depending on oil composition and frying conditions. Most volatile compounds evaporate into the atmosphere with steam, and the remaining volatile compounds in oil undergo further chemical reactions or are absorbed in fried foods. Non-volatile compounds in the oil change the physical and chemical properties of oil and fried foods. Nonvolatile compounds affect the flavor stability, quality, and texture of fried foods during storage. Understanding how flavors are developed in oils during the frying process is important to determine how to enhance positive flavors and inhibit negative flavors. Briefly, flavors in frying oils can come from one or more of the following: naturally occurring flavors (as from peanut oil and olive oil [OO]); flavors imparted from processing procedures (such as hydrogenation); and degradation of fatty acids at the high temperatures used in frying. Flavor development due to the decomposition of fatty acids is the most significant of these 3 effects.

The amount and type of degradation products formed in frying oils are primarily dependent on the fatty acid composition of the frying oil, so it is important to keep this in mind when selecting frying oils. Even though linolenic acid is a nutritionally important fatty acid, it is also very oxidizable. Therefore, reducing linolenic acid to <3% of the total content of fatty acids is necessary to achieve good oil stability and to limit the development of undesirable flavors. In addition, linoleic acid should be present in the oil in greater amounts than linolenic acid not only to provide good deep-fried flavor but also to help mask off flavors from linolenic acid degradation. Even though linoleic acid oxidizes in the fryer, some is needed for developing a deep-fried flavor. Therefore, the content of linoleic acid in frying oil should be in the range of 20% to 30% (Warner and others 1997). The precise level can be adjusted to meet the fry-life requirements of the oil and the shelf-life needed for any stored fried food. In general, most modified-composition oils have the following characteristics. Low linolenic acid-containing oils provide only moderate frying stability, but they are associated with a high deep-fried flavor intensity because their linoleic acid content can still be as high as 50% to 55% as in soybean oil. High oleic oils provide good frying stability and a high waxy/plastic flavor intensity because of oleic acid decomposition, but they are low in linoleic acid, so they result in a low deep-fried flavor intensity. Therefore, in this study, OO and palm superolein (PSO) were used, with the 1st oil exhibiting 71.09% oleic acid and 12.00% linoleic acid and the 2nd 45.20% oleic acid and 12.50% linoleic acid.

The objective of this study was to evaluate the effects of discontinuous and prolonged heat treatment on these 2 oils, which are characterized by different ratios of unsaturated/saturated fatty acids (UFA/SFA; 6.05 for OO and 1.41 for PSO), measuring different indexes that can provide information on the thermal oxidation stability of the oily matrix. In addition, we attempted to identify possible markers of heat treatment correlated with

total polar compound (TPC) in assessing the quality of used frying oils. TPCs include all oxidized and dimerized triglycerides, FFAs, monoglycerides and diglycerides, sterols, carotenoids, antioxidants, antifoamers, and other materials soluble in, emulsified in, or suspended particulates in frying oil. The routine and well-recognized determination of TPC in heated fats is carried out by eluting the sample through a silica column (adsorption chromatography). This official method is time consuming, requires too much solvent, and in some cases it may be misleading to use TPC in judging oil quality. In fact, Berger (2005) reported that the TPC test was associated with problems in interpreting the quality of oils with high diacylglycerols (DG) content, such as palm oil and palm olein. For this reason, an objective of this study was to identify new possible markers to establish the degree of alteration of frying oils.

Materials and Methods

Materials

Refined PSO and OO were supplied by a local market in Naples. A Supelco 37 fatty acid methyl ester (FAME) mix standard was used to identify the chromatographic peaks of the samples (about fatty acid composition). Frozen French fries were bought in the local market and conditioned with a final temperature at 15 ± 1 °C. The thermal process was conducted in a thermostatically temperature-controlled fryer (Tefal, Milan, Italy) at approximately 180 ± 5 °C. All reagents were of analytical or spectroscopic grade and were supplied by Sigma Aldrich (St. Louis, Mo., U.S.A.).

Methods

Our experiments were conducted using OO and PSO. These oils were subjected to a thermo-oxidation process and to a frying process with frozen pre-fried potatoes. Both thermal treatments were prolonged and discontinuous and were conducted over a period of 8 h per day for 6 d. Every 25 min, 200 g of frozen pre-fried potatoes (McCain) were deep-fried for 4 min (based on preliminary trials), for a total of 8 h per day. The frying cycles (8 h per day) were repeated for 6 d. The batch volume of the deep fat fryer was 3.0 L. Fresh oil was added every 8 h after oil samples were collected to maintain a constant level in the fryer. Both experiments were performed under the same frying conditions. The frying oil used to fry the frozen potatoes, oil extracted from French fries, and oil heated in the fryer without potatoes were sampled every 8 h. The oil samples (30 mL) were stored at a temperature of -20 ± 2 °C prior to the analysis.

Analytical methods

Fat adsorbed during the frying process was extracted from the French fries. Then, the following parameters were determined for all of the samples (thermo-oxidized oils, frying oils, and fat extracted from French fries): FFAs; peroxide values (PVs); fatty acid (FA) composition; TPCs; and volatile organic compounds (VOCs), using a dynamic head space system (DHS) coupled with gas chromatography/mass spectrometry (GC/MS).

Extraction of lipids from French fries

A mixture of 200 mL of diethyl ether and petroleum ether (1:2 v/v) was used for fat extraction from 20 g of French fries.

The fat content extracted from French fries was quantified on the basis of the weight of the fat removed through semicontinuous Soxhlet solvent extraction according to AOAC procedure (AOAC 1990). The method described by Soxhlet is a gravimetric method,

namely, a method for determining the mass of the analyte. According to the Soxhlet procedure, oils and fats are extracted from solid samples by repeated washing (leaching) with an organic solvent, in a special glass.

In the semicontinuous solvent extraction (Soxtec), the solvent accumulates in the extraction chamber (sample is held in a filter paper thimble) for 5 to 10 min and then siphons back to the boiling flasks. After a certain period of time the solvent layer is recovered, and the dissolved fat is isolated by evaporating the organic solvent.

FFA

FFA levels were determined according to the European official methods of analysis (EECR 2568/91). FFA content is given as a percent FFA (% FFA), calculated as oleic acid.

PV

The PV is the classical method for determining the extent of oil oxidation (Rossell 1983; Lawson 1985) and measures the formation of intermediate hydroperoxides in milliequivalents of active oxygen per kilogram of sample according to the Reg. CE 1989/03.

TPC

TPCs were determined by preparative column chromatography as described by Dobarganes and others (2000).

FA composition

An FA composition analysis was performed by GC following derivatization to FAMES with 2 N KOH in methanol according to the IUPAC standard method (IUPAC 2.301). FAMES, including methyl octanoate, were analyzed on a GC Perkin Elmer AutoSystem XL (PerkinElmer, Mass., U.S.A.) equipped with a programmed temperature vaporizer, a flame ionization detector (FID), and a capillary column of 100 m × 0.25 mm ID and a film thickness of 0.20 μm using a stationary phase of 50% cyanopropyl methyl silicone (Supelco, Bellefonte, Pa., U.S.A.). The carrier gas, helium, was introduced at a flow rate of 20 cm/s. The oven temperature program was as follows: 120 °C for 5 min, 5 °C/min ramp-up to 165 °C for 5 min, and then 10 °C/min ramp-up to 240 °C for 20 min. The split ratio was 1/60, and the FID temperature was 260 °C. Identification of peaks was performed using an external standard (Supelco™ 37 component FAME MIX). The sample concentrations were calculated through a comparison with the pure standard retention time and were based on response factors to convert peak areas into weight percentages.

VOCs

VOCs were isolated from frying oil using the purge-and-trap DHS concentrator from Teledyne Tekmar Instruments (Manchester, UK) equipped with a Tenax trap. Each sample (2 mL) was spiked with 100 ppb of undecane as an internal standard (IS) contained in a deodorized oil solution. A purge vessel containing the sample was connected to the purge-and-trap unit and was subjected to a specific program. A VOC analysis was performed using the Agilent 6890N gas-chromatograph equipped with an Agilent 5973 N mass spectrometer and a capillary column with 5% phenylmethylpolysiloxane (30 m × 0.25 mm id × 0.25 μm) HP-5 MS (Agilent J&W, Santa Clara, Calif., U.S.A.). Helium was used as the carrier gas at a flow rate of 1.2 mL/min. The oven temperature program was as follows: 45 °C for 3 min, a 10 °C/min ramp-up to 240 °C for 1 min, and a 15 °C/min ramp-up to 270 °C for

1 min. Mass spectra were generated at 70 eV in the range of 35 to 400 UMA. VOC identification was achieved by comparing the obtained mass spectra and GC retention times with those of the available pure standard compounds and the data system library of the GC/MS equipment (NIST 02 and WILEY 275). Estimations of the concentrations of the detected compounds were performed by comparing the obtained peak areas with the area of the known amount of undecane as IS.

Statistical analysis

All determinations and experiments were performed in triplicate, and the results presented are the average values of 3 determinations. The coefficient of determination (R^2) of linearity for the data was statistically analyzed using the Microsoft Excel 2000 program (Microsoft Corporation, Redmond, Wash., U.S.A.). An analysis of variance was conducted using the software XLSTAT 2006, version 2006.6 (Addinsoft, Paris, France) to compare the data obtained for different frying times and different oils. Differences were considered significant at $P \leq 0.05$.

Results and Discussion

Yield

The procedures of lipid extraction from French fries include several steps: (a) size reduction of the sample; (b) homogenization of the tissue in the presence of a solvent; (c) separation of liquid (organic and aqueous) and solid phases; and (e) removal of solvent and drying of the extract.

The extraction efficiency of lipids from a sample also depends on the size of the particles. Therefore, particle size reduction increases surface area, allowing more intimate contact of the solvent, and enhances lipid extraction. For this reason, the French fries are appropriately milled before being subjected to solvent extraction.

The most important characteristic of the ideal solvent for lipid extraction is the high solubility of lipids coupled with low or no solubility of proteins, amino acids, and carbohydrates. The extracting solvent may also prevent enzymatic hydrolysis of lipids, thus ensuring the absence of side reactions. The solvent should readily penetrate sample particles and should have a relatively low boiling point to evaporate readily without leaving any residues when recovering lipids.

The extraction yields corresponding to the different sampling times for French fries in the 2 oils are given in Table 1. As can be seen, in both oils, there was an increase in the amount of oil absorbed during the heat treatment ranging from 4% (time 0 h) to 13% (time 48 h). At the end of treatment, the absorption of oil was of 225% higher respected to frying sample at time 0. This phenomena depends heavily on the quality of the oil, or its resistance to degradation during frying. With increasing frying time, there is an increase of degradation products that cause the polarity of the oil to rise (Dobarganes and others 2000).

The consequences of this are an increase in viscosity due to the formation of polar compounds and polymers, which increases the amount of oil on the food surface (Alim and Morton 1974), and a reduction in the interfacial tension of oil/food which facilitates oil absorption (Dobarganes and others 2000). The viscosity of natural oils is highly dependent on their degree of unsaturation, and it changes during frying and even during simple heating. The increase in the viscosity of PSO was greater than that of OO (Kalogianni and others 2011), which could explain the higher extraction yield obtained from potatoes fried in the bi-fractionated palm oil.

Table 1—FFA, PV, TPC, and yield (% of fat extracted from French fries) trends in palm superolein and olive oil at different frying time.

Time (h)	FFA (% oleic acid)						PV (meq O ₂ /kg oil)						TPC (µg/100 g)						Yield (%)	
	T	FF	PSO	F	OO	T	T	FF	PSO	F	OO	T	T	FF	PSO	F	OO	T	FF	OO
0	0.16 ^a ±0.02	0.16 ^a ±0.03	0.32 ^a ±0.07	0.42 ^a ±0.08	0.42 ^a ±0.05	0.32 ^a ±0.01	4.99 ^a ±0.25	4.99 ^a ±0.21	4.99 ^a ±0.25	4.99 ^a ±0.25	4.99 ^a ±0.25	4.99 ^a ±0.25	5.30 ^a ±0.09	5.30 ^a ±0.09	5.30 ^a ±0.09	5.30 ^a ±0.09	5.30 ^a ±0.09	5.30 ^a ±0.09	3.92 ^a ±0.15	3.92 ^a ±0.18
8	0.39 ^b ±0.11	0.38 ^b ±0.12	0.70 ^b ±0.10	0.79 ^b ±0.12	0.40 ^b ±0.10	0.87 ^b ±0.09	2.75 ^b ±0.10	4.54 ^b ±0.24	2.75 ^b ±0.10	2.75 ^b ±0.10	2.75 ^b ±0.10	2.75 ^b ±0.10	10.43 ^b ±0.10	10.43 ^b ±0.10	10.43 ^b ±0.10	10.43 ^b ±0.10	10.43 ^b ±0.10	10.43 ^b ±0.10	8.97 ^b ±0.21	8.61 ^b ±0.26
16	0.54 ^c ±0.16	0.65 ^c ±0.25	0.70 ^b ±0.14	0.84 ^c ±0.17	0.53 ^b ±0.09	0.73 ^b ±0.18	2.90 ^b ±0.15	5.39 ^b ±0.31	2.90 ^b ±0.15	2.90 ^b ±0.15	2.90 ^b ±0.15	2.90 ^b ±0.15	8.84 ^b ±0.36	8.84 ^b ±0.36	8.84 ^b ±0.36	8.84 ^b ±0.36	8.84 ^b ±0.36	8.84 ^b ±0.36	17.52 ^c ±0.13	11.67 ^b ±0.33
24	0.78 ^c ±0.23	0.90 ^c ±0.16	0.97 ^c ±0.21	0.99 ^c ±0.26	0.66 ^b ±0.13	0.84 ^c ±0.21	2.80 ^b ±0.21	3.13 ^b ±0.12	2.80 ^b ±0.21	2.80 ^b ±0.21	2.80 ^b ±0.21	2.80 ^b ±0.21	13.10 ^c ±0.21	13.10 ^c ±0.21	13.10 ^c ±0.21	13.10 ^c ±0.21	13.10 ^c ±0.21	13.10 ^c ±0.21	18.20 ^c ±0.41	16.42 ^c ±0.13
32	0.96 ^c ±0.19	1.20 ^c ±0.05	1.12 ^c ±0.19	1.12 ^c ±0.08	0.78 ^b ±0.21	0.87 ^c ±0.11	3.09 ^b ±0.29	2.36 ^b ±0.13	3.09 ^b ±0.29	3.09 ^b ±0.29	3.09 ^b ±0.29	3.09 ^b ±0.29	13.80 ^c ±0.29	13.80 ^c ±0.29	13.80 ^c ±0.29	13.80 ^c ±0.29	13.80 ^c ±0.29	13.80 ^c ±0.29	32.72 ^d ±0.51	17.19 ^c ±0.21
40	0.95 ^c ±0.09	1.33 ^c ±0.18	1.24 ^c ±0.29	1.02 ^c ±0.09	0.91 ^b ±0.29	0.94 ^c ±0.09	2.85 ^b ±0.18	5.27 ^c ±0.81	2.85 ^b ±0.18	2.85 ^b ±0.18	2.85 ^b ±0.18	2.85 ^b ±0.18	12.11 ^c ±0.13	12.11 ^c ±0.13	12.11 ^c ±0.13	12.11 ^c ±0.13	12.11 ^c ±0.13	12.11 ^c ±0.13	34.30 ^d ±0.67	25.33 ^c ±0.67
48	1.09 ^c ±0.13	1.83 ^c ±0.21	1.67 ^c ±0.31	1.17 ^c ±0.14	1.03 ^b ±0.17	1.13 ^c ±0.13	2.75 ^b ±0.16	4.82 ^c ±0.65	2.75 ^b ±0.16	2.75 ^b ±0.16	2.75 ^b ±0.16	2.75 ^b ±0.16	9.27 ^b ±1.10	4.58 ^b ±0.13	8.10 ^b ±1.03	7.68 ^b ±0.88	30.20 ^d ±1.21	28.50 ^d ±1.13	37.80 ^d ±0.99	24.60 ^d ±0.79

FFA = free fatty acid; PV = peroxide value; TPC = total polar compounds; PSO = palm superolein; OO = olive oil; T = thermus-oxidized oil; F = frying oil; FF = French fries fat. a–f: Different letters indicate significant differences between treatment times for the same oil for $p \leq 0.05$.

FFA

FFA content is the most frequently used quality test. Determination of FFA content appears to be the method favored for quality-control evaluation of frying oils. This acidity is mainly formed through the hydrolysis of triglycerides, which is promoted by food moisture, and by oxidation or through oil reacting with moisture formed during other deterioration reactions (Al-Harbi and Al-Kabtani 1993).

The methods used for FFA determination do not permit differentiation between FFAs formed by hydrolysis and those formed by oxidation. Therefore, use of FFA alone cannot indicate when frying oil should be discarded. The FFA values for the 2 oils tested are given in Table 1. It can be seen that the FFA values increased with increasing heat treatment times for both oils. For PSO, the FFA values were higher in the frying samples than in the thermo-oxidized samples: at 48 h of heat treatment, the FFA value of the thermo-oxidized oil was 1.09%, while the FFA value of the frying oil was 1.83%. The highest level of FFAs in the frying oil is due to the release of water from the food matrix immersed in the oil bath. The prefried potatoes used in our experiments contain a high concentration of frozen water, which contributes to the increased hydrolysis of triglycerides and, thus, to a higher acidity level (Frankel 2005). The FFA content of the fat extracted from French fries was higher than the FFA percentage of the thermo-oxidized samples. This result is possible because during frying there is an exchange between the fats in food and bath oil, and more than 90% of the final lipid content in the resulting food comes from the bath oil (Dobarganes and others 2000). In OO, both the thermo-oxidized oil and the fat extracted from French fries exhibited higher FFA levels than the frying samples.

PV

The primary products of lipid oxidation are hydroperoxides, which are generally referred to as *peroxides*. Peroxides are unstable organic compounds formed from triglycerides. The PV serves as an indicator of oil quality. Although it does not distinguish between the various unsaturated FAs that undergo oxidation and does not supply information about the secondary oxidative products formed by hydroperoxide decomposition, it can generally be stated that the PV is an indicator of the primary level of oil oxidation (Frankel 2005).

The PV for the 2 oils tested are given in Table 1. The peroxides showed an irregular trend: during both the thermo-oxidation and frying treatments, continuous formation and decomposition of primary products of oxidation occurred in both oils. Hydroperoxides are unstable at high frying temperatures and decompose rapidly in a wide range of volatiles (aldehydes, ketones, alcohols, some esters, and some acids) and nonvolatile compounds (dimers and polymers, polar and nonpolar compounds). Additionally, during frying, the formation of peroxides is inhibited by reduced availability of oxygen because its solubility in oil is reduced at high temperatures and because the water released from food creates a protective cloak of steam. Unfortunately, intermittent heat treatment is more destructive, compared with continuous treatment, because new peroxides form during cooling, and in the subsequent heating phase, these hydroperoxides undergo hydrolysis (Frankel 2005). In general, the PV in PSO was always lower than in OO. This difference can be explained considering the different FA compositions of the 2 oils: PSO exhibits a lower content of unsaturated FAs than OO and is less susceptible to free radical chain reactions leading to the formation of hydroperoxides.

TPC

According to Billek and others (1978) and Paradis and Nawar (1981), the presence of polar compounds indicates degradation of oils and the breakdown of triglycerides, mainly resulting in the formation of dimer triglycerides with a molecular weight of approximately 1800 and monoglycerides. Polar compounds include all oxidized and dimerized triglycerides, FFAs, monoglycerides and diglycerides, sterols, carotenoids, antioxidants, antifoamers, crystal inhibitors, bleaching earth, filteraid, hydrogenation catalyst residues, soaps, residues of chlorophyll, and phospholipids (Blumenthal 1996), and other materials that are soluble, emulsified, or present as suspended particulates in the frying oil. Many researchers consider measurement of polar materials to be the single and most important test for assessing degrading oil. Fritch (1981) reported that an analysis of the percentage of polar compounds is considered to be one of the most reliable indicators of the state of oil deterioration, which is also supported by other researchers (Gere 1982). As indicated by Lalas and Dourtoglou (2003) and Romero and others (1999), several European countries have passed specific laws and regulations concerning culinary oil used in frying. Many countries have set a maximum level of polar compounds of 25%, while others have established a polar compound cutoff point between 20% and 27%. Billek and others (1978) have stated that any heated oil with $\geq 27\%$ polar compounds should be discarded.

The obtained TPC values are reported in Table 1. For PSO, the thermo-oxidized and frying samples exceeded the maximum limit of 25% at 32 h of treatment, whereas the fat extracted from French fries did not exceed this legal limit. However, it must be considered that the TPC value in fresh PSO is higher compared with that of fresh OO and to other oils reported in the literature (Matthäus 2006). The major TPC fractions were PTG (polymeric triglycerides), OTG (oxidized triacylglycerols), and DG. Small fractions of monoglycerides, FFAs, and polar unsaponifiable compounds were also observed (Bansal and others 2010). The limit of detection of the analysis method used for TPC fractions was 0.01%. On the basis of the quantification of these fractions, the high initial TPC content in fresh PSO could be attributed to the large proportion of DG in this oil. Similar results have been published in the literature. Berger (2005) reported that the TPC test was associated with problems in interpreting the quality of oils with high DG content, such as palm oil and palm olein. Typically, 6% to 8% DG are found in palm oil and palm olein, whereas other oils contain only 2% to 3% or less. As DG are measured as a fraction of TPC, it may be misleading to use TPC in judging oil quality.

In OO, TPC increased with the progress of heat treatment. For the thermo-oxidized samples, a fairly rapid increase occurred, with a TPC value that exceeds the legal limit being detected between 24 and 32 h of heating, reaching a value of 37.8% at 48 h. The TPC values of frying oil and fat extracted from French fries were always lower than those of the thermo-oxidized samples. This result could be attributed to the protective effect of the water released from the food during frying. The removal of water from food in the form of steam leads to a distillation process associated with removal of oxygen dissolved in the oil, volatile compounds, and probably also free radicals formed during frying (Dana and others 2003). The frying oil was just above the legal TPC limit at the 40th h, after which it showed a slight reduction at 48 h. The fat extracted from French fries did not reach the legal limit during the heat treatment. It has been reported (Xu and others 1999) that the formation of TPC during a repeated frying process increases with an increasing degree of unsaturation of the oil itself. Considering this finding,

we would have expected higher TPC values for OO than PSO; instead, in the samples from both frying oil and the fat extracted from potatoes, OO appeared to be associated with a lower TPC content than PSO, which is less unsaturated. This result is in agreement with the findings of a previous scientific study (Tabee and others 2009) and can be explained by considering that OO, although rich in monounsaturated FAs, is actually very stable with respect to thermal degradation. In addition, the TPC value for the bi-fractionated palm oil could be overestimated because of the high content (11.7%) of TPC in the fresh oil.

Fatty acid composition

In Table 2 and 3, the FA compositions of the frying oil, the thermo-oxidized oil, and the fat extracted from French fries after different treatment times are reported for PSO and OO, respectively.

The composition of the major FAs present in PSO in its fresh state was as follows: palmitic acid (C16:0), 36.17 g/100 g; oleic acid (C18:1), 45.20 g/100 g; stearic acid (C18:0), 3.40 g/100 g; linoleic acid (C18:2), 12.50 g/100 g; and linolenic acid (C18:3), 0.19 g/100 g. With respect to FA composition, the short-chain FA C8:0 may be used to indicate the degree of oxidative deterioration of frying oils. In oils and fat, oxidation occurs at the level of double bonds, so C8:0 and other short-chain FAs are attached to the glyceridic skeleton forming the nonvolatile fraction. It showed a steady increase in PSO samples, resulting in good linear correlations with TPC values ($R^2 = 0.9247$, $R^2 = 0.893$, and $R^2 = 0.9372$ for the thermo-oxidized oil, frying oil, and French fry oil samples, respectively) (Figure 1). Furthermore, the C8:0 content in PSO is lower than that of OO, which demonstrates the greater stability of PSO. The content of trans fatty acids (TFAs) and the C18:2 n6/C16:0 ratio may also be considered as oil degradation indicators for PSO. In particular, TFAs were represented by the trans isomers of oleic and linoleic acid; their sums showed good correlations with TPC values ($R^2 = 0.942$, $R^2 = 0.949$, and $R^2 = 0.936$ for the thermo-oxidized oil, frying oil, and French fry oil samples, respectively).

The composition of the major FAs present in OO in its fresh state was as follows: palmitic acid (C16:0), 11.36 g/100 g; oleic acid (C18:1), 71.09 g/100 g; stearic acid (C18:0), 2.62 g/100 g; linoleic acid (C18:2): 12.00 g/100 g; and linolenic acid (C18:3), 0.60 g/100 g. The C18:2 n6/C16:0 and UFA/SFA ratios were considered as oil degradation indicators. The latter ratio could be considered a good frying indicator for OO and showed a good correlation with frying TPC values ($R^2 = 0.9264$) (Figure 1). The C18:2n6/C16:0 ratio clearly decreased with the progress of the thermo-oxidation treatment for both oils, showing good correlations with TPC values ($R^2 = 0.9568$ for PSO and $R^2 = 0.9509$ for OO) as reported in Figure 2.

VOCs

Many of the volatile decomposition products formed during frying volatilize and/or further decompose, so it is difficult to obtain an accurate measure of oil deterioration through instrumental and chemical analyses of these compounds. The PV is not a good measure of deterioration in frying oils because peroxides are unstable at frying temperatures. Peroxides decompose very easily into secondary oxidation products, so the analysis of the peroxides after random oil use times provides little information regarding the overall quality of the oil. In our experiment conditions, the oil was heated for 8 h to day and so remains until the next day. In this period, the oil is in contact with the oxygen present in head space

Table 2—Fatty acid composition (%) of palm superolein (thermo-oxidized, frying, and fat extracted from French fries) at different treatment times.

FAME (%)	Thermo-oxidized					Frying oil					French fries fat					
	0	8	24	40	48	8	24	32	40	48	0	8	24	32	40	48
C8:0	0.02 ± 0.00	0.04 ^{ab} ± 0.00	0.16 ^{cd} ± 0.01	0.16 ^{cd} ± 0.01	0.20 ^{de} ± 0.01	0.05 ^a ± 0.01	0.12 ^b ± 0.02	0.18 ^c ± 0.03	0.16 ^{bc} ± 0.01	0.15 ^b ± 0.00	—	0.06 ^a ± 0.00	0.12 ^b ± 0.00	0.15 ^b ± 0.00	0.15 ^b ± 0.01	0.16 ^b ± 0.00
C10:0	0.02 ± 0.00	0.02 ± 0.00	0.02 ± 0.00	0.02 ± 0.00	0.03 ± 0.01	0.02 ± 0.00	0.03 ± 0.01	0.03 ± 0.01	0.02 ± 0.00	0.02 ± 0.00	—	0.03 ± 0.01	0.02 ± 0.00	0.02 ± 0.00	0.02 ± 0.00	0.03 ± 0.01
C12:0	0.28 ± 0.08	0.24 ± 0.00	0.26 ± 0.01	0.26 ± 0.01	0.25 ± 0.01	0.24 ± 0.01	0.25 ± 0.01	0.30 ± 0.08	0.24 ± 0.01	0.24 ± 0.00	0.19 ± 0.06	0.23 ± 0.00	0.27 ± 0.01	0.26 ± 0.00	0.27 ± 0.02	0.25 ± 0.00
C14:0	1.15 ± 0.08	1.16 ± 0.00	1.23 ± 0.02	1.23 ± 0.02	1.25 ± 0.03	1.13 ± 0.02	1.16 ± 0.02	1.36 ± 0.28	1.18 ± 0.05	1.16 ± 0.01	0.97 ± 0.16	1.10 ± 0.01	1.22 ± 0.02	1.23 ± 0.07	1.18 ± 0.06	0.58 ± 0.40
C15:0	0.04 ± 0.01	0.04 ± 0.00	0.04 ± 0.00	0.04 ± 0.00	0.05 ± 0.01	0.04 ± 0.00	0.05 ± 0.01	0.05 ± 0.02	0.04 ± 0.00	0.04 ± 0.00	—	0.04 ± 0.00	0.04 ± 0.00	0.05 ± 0.01	0.04 ± 0.00	0.05 ± 0.02
C16:0	36.13 ± 0.98	36.28 ^{ab} ± 0.13	38.19 ^{bc} ± 0.14	38.70 ^{bc} ± 0.14	39.29 ^{cd} ± 0.14	35.83 ± 0.31	36.78 ± 0.41	39.99 ± 2.19	37.62 ± 1.40	37.03 ± 0.02	19.89 ± 0.17	35.05 ^{ab} ± 0.11	37.20 ± 0.18	37.32 ^a ± 0.87	36.84 ^a ± 0.61	36.18 ^a ± 0.01
C16:1	0.23 ± 0.01	0.21 ± 0.00	0.21 ± 0.00	0.21 ± 0.00	0.21 ± 0.00	0.21 ± 0.00	0.21 ± 0.00	0.21 ± 0.05	0.20 ± 0.02	0.20 ± 0.00	0.11 ± 0.19	0.23 ± 0.03	0.21 ± 0.00	0.20 ± 0.02	0.21 ± 0.00	0.20 ± 0.00
C17:0	0.09 ± 0.01	0.09 ± 0.00	0.09 ± 0.00	0.09 ± 0.00	0.10 ± 0.00	0.09 ± 0.00	0.09 ± 0.00	0.09 ± 0.01	0.09 ± 0.03	0.10 ± 0.01	—	0.08 ± 0.00	0.10 ± 0.00	0.09 ± 0.01	0.10 ± 0.00	0.10 ± 0.00
C17:1	0.03 ± 0.00	0.03 ± 0.00	0.02 ± 0.00	0.02 ± 0.00	0.03 ± 0.00	0.05 ± 0.02	0.03 ± 0.00	0.04 ± 0.03	0.03 ± 0.01	0.02 ± 0.00	—	0.03 ± 0.00	0.01 ± 0.00	0.01 ± 0.00	0.02 ± 0.00	0.02 ± 0.01
C18:0	3.40 ± 0.12	3.61 ^{ab} ± 0.01	3.83 ^{bc} ± 0.01	3.89 ^{bc} ± 0.05	3.94 ^c ± 0.01	3.62 ^{ab} ± 0.02	3.76 ^{ab} ± 0.07	3.47 ^{ab} ± 0.16	3.76 ^{ab} ± 0.24	3.90 ^{ab} ± 0.00	3.83 ^a ± 0.18	3.61 ^b ± 0.01	3.64 ^{ab} ± 0.00	3.64 ^{ab} ± 0.16	3.80 ^{ab} ± 0.07	3.96 ^a ± 0.10
C18:1n9t	0.06 ± 0.00	0.16 ^a ± 0.01	0.29 ^b ± 0.01	0.47 ^c ± 0.01	0.52 ^c ± 0.01	0.13 ± 0.00	0.26 ^a ± 0.01	0.27 ^a ± 0.00	0.33 ^{ab} ± 0.00	0.40 ^a ± 0.00	—	0.10 ^a ± 0.01	0.24 ^b ± 0.01	0.26 ^b ± 0.03	0.34 ^{cd} ± 0.03	0.32 ^a ± 0.01
C18:1n9c	45.20 ± 0.86	45.60 ± 0.09	45.11 ± 0.40	45.33 ± 0.26	45.16 ± 0.06	45.62 ± 0.12	44.96 ± 0.26	42.44 ± 2.10	44.27 ± 0.83	44.43 ± 0.05	36.45 ^a ± 0.08	43.74 ^b ± 0.00	42.76 ^b ± 0.16	42.61 ^b ± 0.44	42.60 ^b ± 0.43	43.29 ^b ± 0.57
C18:2n6t	—	0.01 ± 0.00	0.03 ^{bc} ± 0.01	0.03 ^{bc} ± 0.00	0.04 ^c ± 0.00	0.02 ± 0.01	0.03 ± 0.02	0.02 ± 0.00	0.05 ± 0.04	0.03 ± 0.01	—	0.03 ± 0.00	0.04 ± 0.00	0.05 ± 0.01	0.06 ± 0.05	0.05 ± 0.02
C18:2n6c	12.50 ± 0.18	11.64 ^b ± 0.01	9.93 ^a ± 0.06	9.25 ^a ± 0.04	8.67 ^{ab} ± 0.01	12.07 ± 0.02	11.29 ± 0.02	10.75 ± 0.38	11.17 ^{bc} ± 0.26	11.36 ^{bc} ± 0.03	37.90 ^{cd} ± 0.11	14.73 ^d ± 0.13	13.29 ^d ± 0.05	13.30 ^d ± 0.14	13.50 ^d ± 0.08	13.90 ^{cd} ± 0.04
C20:0	0.28 ± 0.04	0.32 ^{ab} ± 0.01	0.31 ^{ab} ± 0.03	0.34 ^{ab} ± 0.00	0.36 ± 0.01	0.33 ± 0.01	0.34 ± 0.02	0.30 ± 0.08	0.31 ± 0.05	0.34 ± 0.00	0.41 ± 0.07	0.31 ± 0.01	0.29 ± 0.00	0.28 ± 0.05	0.30 ± 0.03	0.34 ± 0.03
C18:3n6	0.05 ± 0.00	0.06 ± 0.00	0.03 ^a ± 0.00	0.03 ^a ± 0.00	0.02 ^a ± 0.00	0.04 ^a ± 0.00	0.03 ^a ± 0.00	0.03 ^a ± 0.00	0.03 ^a ± 0.00	0.03 ^a ± 0.00	—	0.04 ^a ± 0.00	0.03 ^{ab} ± 0.00	0.03 ^{ab} ± 0.00	0.03 ^{ab} ± 0.00	0.03 ^{ab} ± 0.01
C20:1	0.19 ± 0.02	0.17 ± 0.00	0.17 ± 0.00	0.18 ± 0.01	0.17 ± 0.00	0.26 ± 0.12	0.33 ± 0.01	0.14 ± 0.01	0.16 ± 0.02	0.17 ± 0.00	—	0.16 ± 0.00	0.15 ± 0.01	0.14 ± 0.02	0.15 ± 0.02	0.17 ± 0.01
C18:3n3	0.05 ± 0.01	0.14 ± 0.00	0.08 ^{ab} ± 0.00	0.07 ± 0.00	0.06 ± 0.00	0.01 ± 0.00	0.02 ± 0.00	0.10 ^a ± 0.02	0.09 ^a ± 0.00	0.09 ^a ± 0.00	—	0.18 ± 0.01	0.13 ± 0.00	0.13 ± 0.01	0.12 ± 0.00	0.13 ± 0.01
C22:0	0.05 ± 0.02	0.01 ± 0.00	0.05 ± 0.02	0.02 ± 0.00	0.02 ± 0.00	0.07 ^{ab} ± 0.00	0.05 ± 0.01	0.04 ± 0.02	0.05 ± 0.01	0.02 ± 0.00	0.27 ± 0.07	0.05 ± 0.02	0.09 ± 0.00	0.09 ± 0.02	0.11 ± 0.02	0.11 ± 0.02
C23:0	—	0.01 ± 0.00	0.02 ± 0.00	0.02 ± 0.00	0.02 ± 0.00	0.03 ± 0.01	0.03 ± 0.01	0.03 ± 0.01	0.03 ± 0.01	0.02 ± 0.00	—	0.01 ± 0.01	0.02 ± 0.00	0.04 ± 0.02	0.02 ± 0.01	0.02 ± 0.00
C24:0	0.04 ± 0.00	0.05 ± 0.00	0.05 ± 0.02	0.06 ± 0.00	0.08 ± 0.00	0.06 ± 0.00	0.07 ± 0.01	0.05 ± 0.00	0.06 ± 0.02	0.08 ± 0.00	—	0.06 ± 0.00	0.06 ± 0.00	0.06 ± 0.01	0.07 ± 0.01	0.08 ± 0.01
C24:1	—	—	—	—	—	0.02 ± 0.00	0.01 ± 0.00	0.01 ± 0.00	0.02 ± 0.00	—	—	0.02 ± 0.00	—	—	0.01 ± 0.00	—
C22:1n3	—	—	—	—	—	0.01 ± 0.00	0.03 ± 0.00	0.03 ± 0.01	0.05 ± 0.01	0.06 ± 0.00	—	0.05 ± 0.01	0.03 ± 0.00	0.03 ± 0.00	0.04 ± 0.00	0.04 ± 0.00
SFA	41.51 ± 0.99	41.94 ± 0.11	44.06 ^{bc} ± 0.15	44.93 ^{bc} ± 0.26	45.63 ^{cd} ± 0.10	41.52 ± 0.28	42.76 ^{bc} ± 0.28	45.91 ^d ± 2.37	43.61 ^{ab} ± 1.11	43.20 ^{ab} ± 0.03	25.28 ± 0.23	40.68 ^a ± 0.09	43.09 ^b ± 0.20	43.20 ^b ± 0.67	42.89 ^b ± 0.55	41.84 ^{ab} ± 0.59
MUFA	45.71 ± 0.83	46.19 ± 0.09	45.82 ± 0.41	46.30 ± 0.10	46.22 ± 0.27	46.28 ± 0.26	45.80 ± 0.26	43.11 ± 2.03	44.97 ± 0.87	45.22 ± 0.05	36.56 ± 0.11	44.29 ^b ± 0.15	43.37 ^b ± 0.15	43.22 ^b ± 0.50	43.34 ^b ± 0.42	43.99 ^b ± 0.55
PUFA	12.79 ± 0.16	11.87 ^{ab} ± 0.01	10.13 ^a ± 0.08	9.44 ^{ab} ± 0.04	8.85 ^a ± 0.03	12.20 ^{ab} ± 0.28	11.4 ^a ± 0.02	10.99 ^a ± 0.33	11.42 ^{cd} ± 0.24	11.58 ^{cd} ± 0.02	38.16 ± 0.15	15.03 ^b ± 0.11	13.55 ^b ± 0.05	13.58 ^b ± 0.16	13.77 ^{cd} ± 0.14	14.17 ^{cd} ± 0.04
UFA	58.49 ± 0.99	58.06 ± 0.11	55.94 ^{ab} ± 0.15	55.07 ^{ab} ± 0.26	54.37 ^{ab} ± 0.10	58.48 ± 0.28	57.24 ^{ab} ± 0.28	54.09 ^a ± 2.37	56.39 ^{ab} ± 1.11	56.80 ^{ab} ± 0.03	74.72 ± 0.23	59.32 ^b ± 0.09	56.91 ^b ± 0.20	56.80 ^b ± 0.67	57.11 ^b ± 0.55	58.18 ^b ± 0.59
TRANS	0.06 ± 0.00	0.17 ^a ± 0.01	0.32 ^b ± 0.01	0.50 ^{bc} ± 0.01	0.57 ^{cd} ± 0.01	0.14 ± 0.01	0.29 ± 0.02	0.30 ± 0.00	0.38 ± 0.04	0.43 ^a ± 0.01	—	0.14 ± 0.01	0.28 ± 0.01	0.31 ± 0.03	0.40 ^d ± 0.01	0.36 ± 0.00
MUFA/SFA	1.41 ± 0.06	1.38 ± 0.01	1.27 ^{bc} ± 0.03	1.23 ^{bc} ± 0.01	1.19 ^{bc} ± 0.01	1.41 ± 0.01	1.34 ^{ab} ± 0.02	1.18 ± 0.11	1.29 ^{ab} ± 0.06	1.32 ^{ab} ± 0.00	2.96 ^{cd} ± 0.04	1.46 ^{cd} ± 0.01	1.32 ^{cd} ± 0.01	1.31 ^{cd} ± 0.04	1.33 ^{cd} ± 0.03	1.30 ^{cd} ± 0.03
MUFA/SFA	1.10 ± 0.05	1.10 ± 0.01	1.04 ^{ab} ± 0.02	1.03 ^{ab} ± 0.01	1.01 ^{ab} ± 0.00	1.11 ± 0.01	1.07 ± 0.01	1.04 ± 0.09	1.03 ± 0.05	1.05 ± 0.00	1.45 ^{cd} ± 0.02	1.09 ^{cd} ± 0.00	1.01 ± 0.01	1.00 ± 0.03	1.01 ± 0.02	1.02 ^{ab} ± 0.03
PUFA/SFA	0.31 ± 0.01	0.28 ± 0.00	0.23 ^{ab} ± 0.00	0.20 ^{ab} ± 0.00	0.18 ± 0.00	0.29 ^{ab} ± 0.00	0.27 ^{ab} ± 0.00	0.24 ± 0.02	0.26 ^{ab} ± 0.01	0.22 ^{ab} ± 0.00	1.31 ± 0.02	0.37 ^{cd} ± 0.00	0.31 ± 0.00	0.31 ± 0.01	0.32 ± 0.01	0.34 ^{cd} ± 0.01
18:2/16:0	0.35 ± 0.01	0.32 ± 0.00	0.24 ^{ab} ± 0.00	0.22 ^{ab} ± 0.00	0.21 ^{ab} ± 0.00	0.34 ± 0.00	0.31 ± 0.00	0.27 ± 0.02	0.30 ^{ab} ± 0.02	0.31 ^{ab} ± 0.00	1.91 ± 0.02	0.42 ^{cd} ± 0.00	0.36 ± 0.00	0.36 ± 0.01	0.37 ± 0.01	0.38 ^{cd} ± 0.00

a-c: Different letters indicate significant differences between treatment times for the same oil for P ≤ 0.05.

—: Under detection limit (<0.01%).

Table 3—Fatty acid composition (%) of olive oil (thermo-oxidized, frying, and fat extracted from French fries) at different treatment times.

FAME (%)	Thermo-oxidized oil					Olive oil					French fries fat				
	0	8	24	32	40	8	24	32	40	48	8	24	32	40	48
C8:0	-	0.10 ^a ± 0.08	0.24 ^b ± 0.08	0.29 ^b ± 0.00	0.29 ^b ± 0.00	0.43 ^c ± 0.00	0.14 ^a ± 0.14	0.14 ^a ± 0.00	0.48 ^c ± 0.44	0.62 ^c ± 0.22	-	0.05 ± 0.08	-	-	-
C10:0	-	-	-	-	-	0.08 ± 0.07	-	-	-	-	-	0.08 ± 0.13	-	-	-
C11:0	-	-	-	-	-	-	-	-	-	-	-	0.08 ± 0.13	-	-	-
C12:0	-	0.07 ± 0.06	0.07 ± 0.06	0.04 ± 0.06	-	0.15 ± 0.06	0.13 ± 0.06	0.04 ± 0.06	0.24 ^b ± 0.06	0.20 ^b ± 0.00	0.19 ± 0.06	0.18 ± 0.13	0.17 ^b ± 0.06	0.04 ± 0.06	0.04 ± 0.06
C14:0	0.10 ^a ± 0.00	0.10 ^a ± 0.00	0.10 ^a ± 0.00	0.10 ^a ± 0.00	0.10 ^a ± 0.00	0.41 ^b ± 0.20	0.13 ^a ± 0.06	0.17 ^b ± 0.06	-	0.07 ± 0.06	0.97 ^b ± 0.16	0.72 ± 0.20	0.20 ^b ± 0.00	0.20 ^b ± 0.00	0.20 ^b ± 0.00
C15:0	-	-	-	-	-	0.04 ± 0.06	-	-	-	-	-	0.18 ± 0.22	-	-	-
C16:0	11.36 ^a ± 0.50	12.49 ^b ± 0.07	13.33 ^c ± 0.23	13.90 ^d ± 0.09	14.06 ^d ± 0.04	15.05 ^e ± 0.39	12.16 ^b ± 0.28	14.18 ^c ± 0.47	14.28 ^c ± 0.12	14.52 ^c ± 0.39	19.89 ^b ± 0.17	13.86 ^b ± 0.09	13.77 ^b ± 0.16	14.34 ^b ± 0.52	15.35 ^b ± 2.04
C16:1	1.41 ^a ± 0.06	1.69 ^b ± 0.18	1.70 ^b ± 0.00	1.68 ^b ± 0.06	1.68 ^b ± 0.06	1.71 ^b ± 0.00	1.48 ± 0.11	1.43 ± 0.07	1.35 ± 0.07	1.35 ± 0.06	0.11 ^a ± 0.19	1.28 ^a ± 0.00	1.24 ^a ± 0.06	1.21 ^a ± 0.06	1.24 ^a ± 0.23
C17:0	0.11 ± 0.00	0.11 ± 0.00	0.11 ± 0.00	0.11 ± 0.00	0.11 ± 0.00	0.11 ± 0.00	0.11 ± 0.00	0.11 ± 0.00	0.07 ± 0.06	0.11 ± 0.00	0.11 ^a ± 0.19	0.11 ± 0.00	0.04 ^b ± 0.06	0.11 ^a ± 0.00	0.11 ^a ± 0.00
C17:1	0.11 ± 0.00	0.11 ± 0.00	0.11 ± 0.00	0.11 ± 0.00	0.11 ± 0.00	0.11 ± 0.00	0.15 ± 0.06	0.19 ± 0.13	0.11 ± 0.00	0.15 ± 0.06	-	0.11 ± 0.00	0.11 ± 0.00	0.07 ± 0.06	0.07 ± 0.06
C18:0	2.62 ± 0.00	2.25 ± 0.05	2.50 ± 0.06	2.68 ^b ± 0.06	2.72 ± 0.05	3.09 ^c ± 0.16	2.73 ^b ± 0.01	3.31 ^b ± 0.06	3.20 ^b ± 0.05	3.21 ^b ± 0.04	3.83 ^b ± 0.18	3.18 ^b ± 0.06	3.01 ^b ± 0.06	3.45 ^b ± 0.01	3.26 ± 0.08
C18:1 n9c	71.09 ^{ab} ± 0.53	70.28 ^b ± 0.58	71.48 ^{ab} ± 0.21	71.57 ^{ab} ± 0.12	71.97 ^b ± 0.17	71.02 ^b ± 0.81	69.85 ^b ± 0.44	67.93 ^b ± 0.31	67.03 ^b ± 0.38	66.07 ^b ± 0.42	36.45 ^b ± 0.08	66.57 ^b ± 1.11	66.12 ^b ± 0.39	64.88 ^b ± 0.55	63.31 ^b ± 1.91
C18:2 n6c	12.00 ^a ± 0.04	11.73 ^a ± 0.19	11.59 ^a ± 0.06	11.11 ± 0.00	11.11 ± 0.00	10.11 ± 0.00	11.11 ± 0.00	11.11 ± 0.00	11.11 ± 0.00	11.11 ± 0.00	37.90 ^a ± 0.11	13.86 ^b ± 0.09	14.67 ^b ± 0.01	14.86 ^b ± 0.09	15.33 ^b ± 0.46
C18:3 n3	0.60 ^a ± 0.00	0.48 ^a ± 0.01	0.32 ^a ± 0.07	0.28 ^a ± 0.00	0.28 ^a ± 0.00	0.20 ^a ± 0.00	0.36 ^a ± 0.00	0.36 ^a ± 0.00	0.32 ^a ± 0.07	0.24 ^a ± 0.21	0.41 ± 0.07	0.49 ± 0.12	0.44 ± 0.14	0.36 ± 0.00	0.36 ± 0.00
C20:1	0.48 ^a ± 0.00	0.16 ^a ± 0.06	0.19 ± 0.00	0.23 ^a ± 0.06	0.20 ^a ± 0.00	0.19 ± 0.00	0.32 ^a ± 0.11	0.16 ^a ± 0.06	0.13 ^a ± 0.06	0.19 ^a ± 0.00	-	0.07 ± 0.11	0.10 ± 0.00	0.10 ± 0.00	0.13 ± 0.06
C21:0	-	0.28 ^a ± 0.00	0.14 ^a ± 0.00	0.05 ^a ± 0.08	0.14 ^a ± 0.00	-	-	-	-	-	-	-	-	-	-
C20:2	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
C20:4 n6	0.11 ± 0.00	-	-	-	-	-	0.11 ± 0.00	0.11 ± 0.00	0.11 ± 0.00	0.11 ± 0.00	0.27 ^a ± 0.07	0.08 ^b ± 0.13	0.11 ± 0.00	0.07 ± 0.06	0.11 ± 0.00
C22:0	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
C20:3 n6	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
C22:1 n9	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
SFA	14.19 ^a ± 0.50	15.40 ^b ± 0.20	16.49 ^{bc} ± 0.31	17.21 ^c ± 0.09	17.53 ^c ± 0.06	19.35 ^d ± 0.92	15.27 ^b ± 0.23	17.61 ^b ± 0.16	18.27 ^b ± 0.42	18.74 ^b ± 0.37	24.87 ^b ± 0.26	16.82 ^b ± 0.93	17.35 ^b ± 0.08	18.15 ^b ± 0.58	19.02 ^b ± 2.14
MUFA	73.09 ^{ab} ± 0.46	72.24 ^{ab} ± 0.49	73.48 ^{ab} ± 0.21	73.59 ^{ab} ± 0.09	73.93 ^{ab} ± 0.11	73.04 ^{ab} ± 0.81	71.81 ^a ± 0.27	69.65 ^b ± 0.21	68.63 ^{bc} ± 0.39	67.76 ^c ± 0.34	36.56 ^c ± 0.11	68.68 ^b ± 0.86	67.13 ^{bc} ± 0.14	66.30 ^{bc} ± 0.49	64.75 ^d ± 1.69
PUFA	12.71 ^b ± 0.04	12.36 ^b ± 0.29	10.03 ^b ± 0.10	9.24 ^b ± 0.07	8.63 ^b ± 0.04	7.61 ^b ± 0.11	12.92 ^a ± 0.10	12.74 ^a ± 0.05	13.10 ^{ab} ± 0.04	13.50 ^{ab} ± 0.04	38.57 ^a ± 0.20	14.42 ^a ± 0.12	15.51 ^a ± 0.07	15.35 ^a ± 0.24	15.55 ^a ± 0.19
UFA	85.81 ^b ± 0.50	84.60 ^b ± 0.20	83.51 ^{bc} ± 0.31	82.83 ^c ± 0.09	82.58 ^c ± 0.09	80.65 ^d ± 0.92	84.73 ^b ± 0.23	82.39 ^b ± 0.16	81.73 ^{bc} ± 0.42	81.26 ^{bc} ± 0.37	75.13 ^c ± 0.26	83.10 ^b ± 0.98	82.65 ^b ± 0.28	81.89 ^b ± 0.58	80.98 ^b ± 2.14
TRANS	6.05 ^a ± 0.24	5.49 ^a ± 0.09	5.06 ± 0.11	4.81 ^a ± 0.03	4.71 ^a ± 0.02	4.17 ^a ± 0.02	5.55 ^a ± 0.10	4.68 ^a ± 0.05	4.47 ^a ± 0.14	4.47 ^a ± 0.13	3.02 ^a ± 0.04	4.95 ^b ± 0.34	4.76 ^b ± 0.34	4.87 ^b ± 0.12	4.30 ^b ± 0.56
UFA/SFA	5.15 ^a ± 0.21	4.69 ^a ± 0.09	4.46 ^a ± 0.10	4.28 ^a ± 0.03	4.22 ^a ± 0.02	3.78 ^a ± 0.22	4.70 ^a ± 0.09	3.94 ^a ± 0.11	3.76 ^a ± 0.11	3.62 ^a ± 0.09	1.47 ^a ± 0.02	4.09 ^a ± 0.29	3.87 ^a ± 0.02	3.97 ^a ± 0.10	3.44 ^a ± 0.45
PUFA/SFA	0.90 ^a ± 0.03	0.80 ^a ± 0.01	0.61 ^a ± 0.02	0.54 ^a ± 0.00	0.49 ^a ± 0.00	0.39 ^a ± 0.02	0.85 ^a ± 0.01	0.72 ^a ± 0.00	0.72 ^a ± 0.02	0.72 ^a ± 0.02	1.35 ^a ± 0.02	0.86 ^a ± 0.06	0.89 ^a ± 0.00	0.90 ^a ± 0.02	0.86 ^a ± 0.11
18:2/16:0	1.06 ^a ± 0.05	0.94 ^a ± 0.01	0.72 ^a ± 0.01	0.64 ^a ± 0.00	0.59 ^a ± 0.00	0.48 ^a ± 0.00	1.00 ^a ± 0.02	0.87 ^a ± 0.03	0.88 ^a ± 0.01	0.89 ^a ± 0.02	1.91 ^a ± 0.02	1.06 ^a ± 0.01	1.06 ^a ± 0.01	1.06 ^a ± 0.14	1.03 ^a ± 0.16

a-c: Different letters indicate significant differences between treatment times for the same oil for p ≤ 0.05. - : Under detection limit (<0.01%).

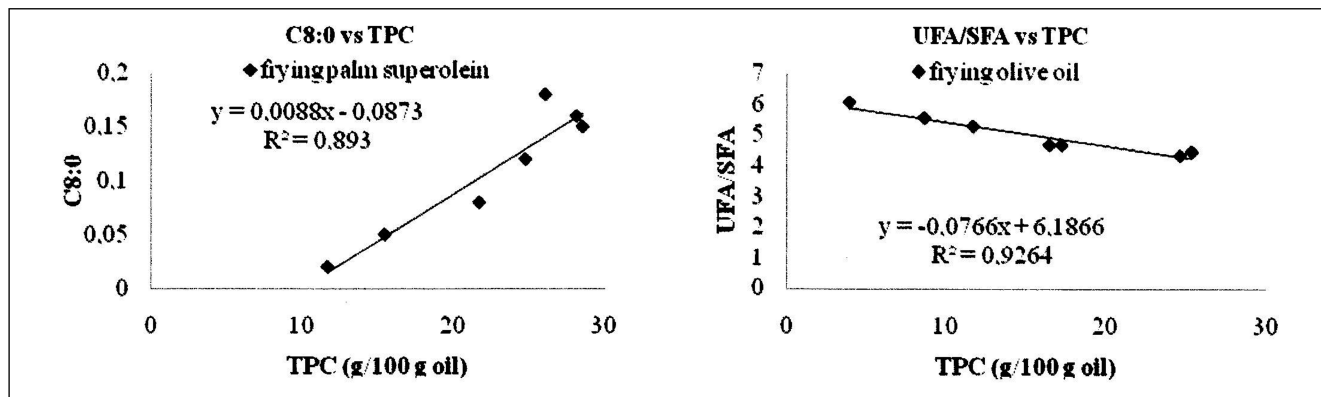


Figure 1—Correlations between C8:0 and UFA/SFA ratio versus TPC in frying PSO and OO, respectively.

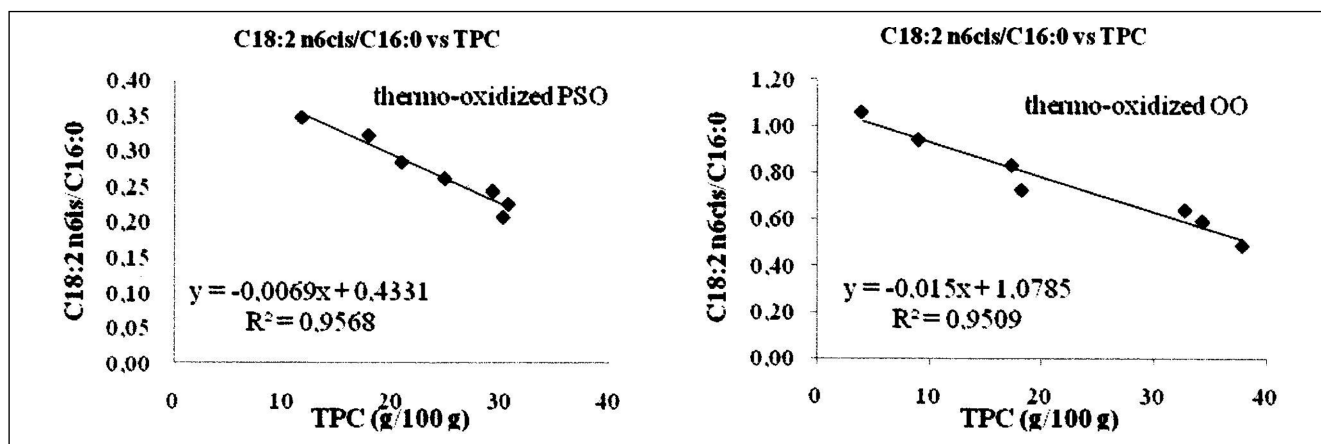


Figure 2—Correlation between C18:2 n6cis/C16:0 ratio versus TPC in thermo-oxidized PSO and OO.

of fryer, and so undergoes to the oxidation process. In addition, the replenishment with fresh oil provides fat that can undergo to oxidation and lead to the formation of new hydroperoxides. For these reasons the PVs show a random trend and cannot be used to establish the oil quality. The gas chromatographic volatile compound analysis measures compounds that are directly related to the flavor of fried food. Identifying volatile compounds in fried food is important because knowledge of these compounds assists in understanding the chemical reactions that occur during frying and because the flavor of deep-fried food is caused by these volatile compounds. Although the volatile compounds in frying oil change continually, measuring these compounds can provide some indication of oil deterioration, but care should be taken in interpreting data on volatile compounds in used frying oil because of the fluctuations in the formation and degradation of these compounds at frying temperatures. In our experiments, a purge-and-trap device coupled with GC/MS was used to identify volatile compounds in frying oils, such as hydrocarbons, aldehydes, alcohols, furans, esters, ethers, acids, and lactones (Table 4 and 5).

Under our experimental conditions, the VOCs identified in the frying oils and French fries included products of the β -scission of oleic and linoleic acids (heptane, octanal, nonanal, 2-E-decenal, 2-undecenal, hexanal, 2,4-E,E-decadienal), aromatic hydrocarbons (ethylbenzene, o-xylene, decahydro naphthalene, 2-pentylfuran), and products of the Maillard reaction (2,5-dimethylpyrazine) and Strecker degradation (disulfide-bis-1-methylethyl-2-methyl-butanoic acid). All of these compounds ex-

hibited random evolution with the progress of thermal treatment. The results showed that the thermo-oxidative reactions induced under the heating conditions were faster than those under the frying conditions because of the protective role of the water released by food during frying. Considering the 2 oils at time zero, the main classes of volatile compounds identified were represented by alkanes and alkanals. In fresh PSO, alkanals represented approximately 80% of the volatile components, while alkanes constituted the remaining 20%. In contrast, in fresh OO, alkanes are the main fractions, representing approximately 69% of the total amount of volatile components present, while the alkanals represent approximately 25%; there are also small amounts of alcohols, alkenals, ketones, acids, and aromatic heterocyclic compounds. In both oils, after only 8 h of thermal treatment, an enrichment of the pattern of VOCs was observed, most likely associated with the formation of new compound classes (alkadienals, alkenes, acids, alcohols, ketones) due to further decomposition reactions of polyunsaturated aldehydes.

Relative to hydroperoxide decomposition products, trans,trans-2,4-decadienal was found and quantified in all frying oils (PSO and OO), but this aldehyde only showed a good correlation with TPC in thermo-oxidized OO ($R^2 = 0.8349$). Additionally, 2,4-E,E-nonadienal and Σ acids (hexanoic and octanoic acids) showed good correlations with TPC ($R^2 = 0.9161$ and 0.8985 , respectively) as reported in Figure 3.

In the samples fried in OO, alkylbenzenes were detected, and a possible mechanism of their formation is proposed.

Table 4—VOCs (ppb) of palm superolein (thermo-oxidized, frying, and fat extracted from French fries) at different treatment times.

Alkanes	Palm superolein				Frying oil				French fries fat			
	0	8	24	48	0	8	24	48	0	8	24	48
Heptane	—	—	—	144.21 ± 10.73	23.43 ± 1.58	71.97 ^b ± 5.35	42.16 ^c ± 2.84	—	—	—	—	—
Methyl-cyclohexane	0.93 ± 0.07	—	—	2.17 ^c ± 0.17	0.39 ^a ± 0.03	0.51 ^b ± 0.03	0.37 ^a ± 0.03	—	—	—	—	—
Propyl-cyclohexane	—	—	1.46 ^b ± 0.11	5.36 ^c ± 0.39	1.10 ^b ± 0.08	2.05 ^b ± 0.16	1.06 ^a ± 0.07	—	—	—	—	—
Buryl-cyclopentane	—	—	4.07 ^b ± 0.30	1.60 ± 0.12	—	—	—	—	—	—	—	—
Propyl-cyclopentane	—	—	1.49 ± 0.11	1.37 ± 0.85	—	—	—	—	—	—	—	—
Decane	—	—	—	—	—	—	—	—	—	—	—	—
Dodecane	—	—	—	—	—	—	—	—	—	—	—	—
2,6-dimethyl-undecane	—	—	—	—	—	—	—	—	—	—	—	—
Eicosane	—	—	—	—	—	—	—	—	—	—	—	—
Total	0.93	4.56	7.02	10.5	24.92	74.53	43.59	0.99	2.52	1.18	2.51	
1-octene	—	—	—	6.88 ± 0.51	1.60 ^b ± 0.12	3.05 ^b ± 0.23	2.53 ^b ± 0.17	—	—	—	—	—
2-E-octene	—	0.76 ± 0.05	—	—	—	—	—	—	—	—	—	
Total	—	0.76	—	6.88	—	—	—	—	—	—	—	
1-pentanol	—	—	—	—	3.16 ^c ± 0.21	7.51 ^b ± 0.56	3.57 ^a ± 0.24	—	—	—	—	—
4-methyl-1-pentanol	—	—	—	—	1.08 ^a ± 0.08	1.45 ^b ± 0.11	0.67 ^c ± 0.04	—	—	—	—	—
1-hexanol	—	3.54 ^b ± 0.26	4.51 ^b ± 0.33	1.77 ^c ± 0.13	0.42 ^c ± 0.03	0.79 ^b ± 0.06	0.60 ^c ± 0.50	—	—	—	—	
1-heptanol	—	1.37 ^a ± 0.10	1.95 ^a ± 0.14	2.28 ^a ± 0.17	0.72 ^c ± 0.05	1.23 ^b ± 0.09	0.76 ^c ± 0.05	—	—	—	—	
1-octen-3-ol	—	3.39 ^a ± 0.25	2.14 ^b ± 0.16	1.89 ^b ± 0.14	0.25 ^a ± 0.01	0.56 ^b ± 0.04	0.39 ^a ± 0.03	—	—	—	—	
1-octanol	—	1.20 ^a ± 0.09	1.18 ^a ± 0.09	1.39 ^a ± 0.11	5.63	11.54	5.99	—	—	—	—	
Total	—	9.50	9.78	7.33	48.52 ^a ± 3.27	95.75 ^b ± 7.13	65.49 ^c ± 4.41	4.44 ± 0.33	3.95 ± 0.27	—	—	
Hexanal	2.47 ^b ± 0.17	109.87 ^b ± 8.18	111.81 ^b ± 7.53	179.33 ^b ± 13.35	64.99	123.78	85.57	6.56	6.54	3.29	7.44	
Heptanal	—	11.62 ^a ± 0.86	13.83 ^a ± 1.03	12.47 ^a ± 1.77	0.74 ^c ± 0.05	2.00 ^b ± 0.15	1.24 ^c ± 0.09	—	—	—	—	
n-octanal	0.24 ^a ± 0.01	6.00 ^b ± 0.45	8.51 ^b ± 0.64	23.86 ^b ± 0.93	3.74 ^c ± 0.25	8.19 ^b ± 0.61	5.94 ^c ± 0.41	1.97 ^a ± 0.08	2.44 ^{ab} ± 0.16	2.92 ^{ab} ± 0.22	6.37 ^a ± 0.47	
Nonanal	0.86 ^b ± 0.06	16.43 ^b ± 1.22	17.99 ^b ± 1.33	21.75 ^b ± 1.62	2.18 ^a ± 0.15	5.16 ^a ± 0.25	4.15 ^a ± 0.28	0.15 ^a ± 0.01	0.15 ^a ± 0.01	0.08 ^a ± 0.01	0.26 ^a ± 0.02	
Decanal	—	1.13 ^{ab} ± 0.08	0.96 ^{ab} ± 0.07	1.21 ^b ± 0.09	0.35 ^a ± 0.02	0.56 ^b ± 0.04	0.46 ^b ± 0.03	—	—	—	—	
Dodecanal	—	0.11 ^a ± 0.00	0.10 ^a ± 0.01	—	—	—	—	—	—	—	—	
Total	3.57	147.16	153.2	238.62	64.99	123.78	85.57	6.56	6.54	3.29	7.44	
2-E-hexenal	—	2.95 ^a ± 0.22	2.13 ^b ± 0.14	3.94 ^a ± 0.29	0.74 ^c ± 0.05	2.00 ^b ± 0.15	1.24 ^c ± 0.09	—	—	—	—	
2-Z-heptenal	—	16.42 ^{ab} ± 1.22	15.28 ^a ± 1.14	20.04 ^b ± 1.49	6.18 ^b ± 0.42	12.10 ^b ± 0.90	7.61 ^{bc} ± 0.52	—	—	—	—	
2-E-octenal	—	4.77 ^{ab} ± 0.36	4.81 ^{ab} ± 0.36	5.02 ± 0.38	2.07 ^a ± 0.14	2.80 ^b ± 0.21	2.13 ^c ± 0.14	—	—	—	—	
2-E-nonenal	—	1.37 ^a ± 0.10	2.29 ^{bc} ± 0.17	2.49 ^a ± 0.18	0.53 ^a ± 0.03	1.03 ^b ± 0.08	1.04 ^b ± 0.07	0.33 ^a ± 0.02	0.49 ^b ± 0.03	0.43 ^b ± 0.03	0.78 ^a ± 0.06	
2-E-decenal	—	8.53 ^a ± 0.63	5.88 ^b ± 0.44	6.23 ^b ± 0.47	2.44 ^a ± 0.16	4.71 ^b ± 0.35	3.27 ^{bc} ± 0.22	0.88 ^a ± 0.06	1.57 ^b ± 0.11	1.99 ^b ± 0.15	4.68 ^a ± 0.35	
2-undecenal	—	5.19 ± 0.39	2.92 ± 1.72	4.31 ± 0.32	1.72 ^c ± 0.12	3.72 ^b ± 0.27	2.71 ^c ± 0.19	0.74 ^a ± 0.05	1.35 ^a ± 0.10	2.40 ^a ± 0.16	4.56 ^a ± 0.34	
Total	—	39.23	33.31	42.03	13.68	26.36	18.00	1.95	3.41	4.82	10.02	
2,4-E, E-heptadienal	—	1.40 ^a ± 0.10	1.12 ^b ± 0.08	—	0.58 ± 0.04	—	—	—	—	—	—	
2,4-E, E-nonadienal	—	0.20 ^a ± 0.02	0.21 ^a ± 0.02	—	5.13 ^a ± 0.38	4.84 ^a ± 0.36	3.27 ^b ± 0.22	1.42 ^a ± 0.13	2.47 ^{ab} ± 0.19	1.92 ^{bc} ± 0.14	3.23 ^b ± 0.24	
2,4-E, E-decadienal	—	5.34 ^a ± 0.40	3.80 ^b ± 0.26	2.62 ± 0.20	5.71	4.84	3.27	1.42	2.47	1.92	3.51	
Total	—	6.94	5.13	3.14	0.97 ^a ± 0.06	2.12 ^a ± 0.16	1.51 ^c ± 0.10	—	—	—	—	
2-heptanone	—	4.25 ^a ± 0.32	3.56 ^{ab} ± 0.26	5.67 ^a ± 0.43	—	—	—	—	—	—	—	
Cyclohexanone-2-methyl-5-(1-methylethyl), trans	—	—	—	—	—	—	—	—	—	—	—	
2-methyl butanoic acid	—	—	—	—	—	—	—	—	—	—	—	
Hexanoic acid	—	2.30 ± 0.17	—	—	—	—	—	—	—	—	—	
Octanoic acid	—	10.82 ± 0.81	—	—	—	—	—	—	—	—	—	
Butanedioic acid methyl bis (1-methylpropyl)ester	—	0.33 ^a ± 0.02	0.33 ^a ± 0.02	0.19 ± 0.01	0.32 ^a ± 0.02	0.62 ^b ± 0.04	0.46 ^c ± 0.03	0.35 ^a ± 0.03	0.34 ^a ± 0.03	0.72 ^b ± 0.06	0.41 ^a ± 0.03	
Total	—	13.45	0.33	0.19	0.32	0.62	0.46	1.09	5.97	14.08	4.51	
2,5-dimethyl-pyrazine	—	—	—	—	—	—	—	—	—	—	—	
2-pentyl furan	—	9.40 ^a ± 0.70	13.51 ^b ± 1.00	17.72 ^c ± 1.31	3.40 ^a ± 0.23	4.77 ^b ± 0.35	3.69 ^{bc} ± 0.24	0.58 ^a ± 0.06	0.58 ^a ± 0.06	0.55 ^a ± 0.04	0.55 ^a ± 0.04	
2n-heptyl furan	—	0.47 ^a ± 0.04	0.77 ^b ± 0.05	0.84 ^b ± 0.06	—	—	—	—	—	—	—	
2n-octyl furan	—	—	1.68 ^a ± 0.13	1.49 ^a ± 0.11	—	—	—	—	—	—	—	
Total	—	9.87	15.96	20.05	3.40	4.77	3.69	0.58	1.05	0.55	0.55	
o-xylene	—	—	—	—	—	—	—	—	—	—	—	
Styrene	—	—	—	—	—	—	—	—	—	—	—	
Total	—	—	—	—	—	—	—	—	—	—	—	
D-limonene	—	—	—	—	—	—	—	—	—	—	—	
Hexyl pentyl ether	—	—	0.45 ^a ± 0.04	0.39 ^{ab} ± 0.03	—	—	—	—	—	—	—	
Total	—	—	0.45 ^a ± 0.04	0.39 ^{ab} ± 0.03	—	—	—	—	—	—	—	
2,45 ^{ab} ± 0.16	2.62 ^{ab} ± 0.19	3.31 ^a ± 0.22	1.69 ^b ± 0.11	3.82	5.01	5.01	5.01	3.50 ^a ± 0.42	4.19 ^{ab} ± 0.31	4.94 ^{ab} ± 0.37	5.01 ^{bc} ± 0.34	

^{a-d} The reliability of the identification or structural proposal is indicated by the following: (0) —, not detected; (1) —, consistent with those of an authentic standard; (2) —, structural proposals are given on the basis of mass spectral data (NIST 02, WILEY 275) with a similar index (SD) > 90%. a–d. Different letters indicate significant differences between treatment times for the same oil for *P* ≤ 0.05.

Table 5—VOCs (ppb) of olive oil (thermo-oxidized, frying, and fat extracted from French fries) at different treatment times.

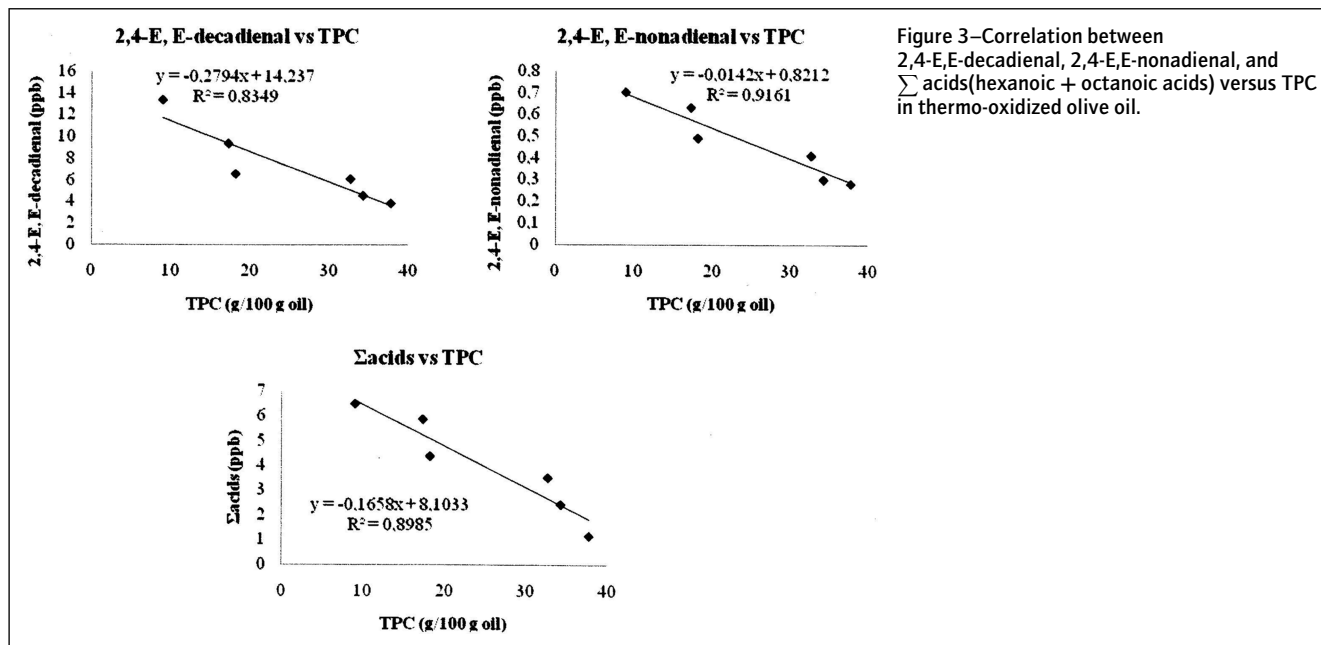
Compound (ppb)	Thermo-oxidized				Frying				French fries fat			
	0	8	24	48	0	8	24	48	0	8	24	48
Alkanes												
Heptane	23.15 ^a ± 0.68	114.62 ^b ± 24.57	150.72 ^{bc} ± 10.39	147.33 ^b ± 4.31	76.04 ^a ± 5.00	61.98 ^{ab} ± 7.12	26.01 ^c ± 6.40	0.38 ^a ± 0.05	3.24 ^b ± 0.21	0.46 ^c ± 0.04	1.28 ^d ± 0.54	
Octane	—	73.82 ^a ± 17.62	57.40 ^b ± 4.50	59.81 ^b ± 0.14	0.34 ^a ± 0.05	0.23 ^a ± 0.00	—	—	1.81 ^a ± 0.06	0.18 ^b ± 0.05	—	
Propyl-cyclohexane	—	1.32 ^a ± 0.17	1.18 ^a ± 0.11	1.04 ^a ± 0.03	0.94 ^a ± 0.02	0.93 ^a ± 0.10	0.15 ^b ± 0.03	0.81 ^a ± 0.06	5.83 ^b ± 0.42	5.14 ^b ± 0.16	2.33 ^c ± 0.36	
Butyl-cyclopentane	—	4.23 ^a ± 0.64	4.19 ^a ± 0.03	3.66 ^b ± 0.47	—	—	—	—	1.86 ^b ± 0.03	1.63 ^b ± 0.06	0.77 ^b ± 0.20	
Nonane	—	—	—	—	—	—	—	—	2.31 ^{bc} ± 0.21	2.34 ^{bc} ± 0.33	1.03 ^{bc} ± 0.13	
Decane	—	—	—	—	—	—	—	—	0.93 ^a ± 0.07	0.80 ^{ab} ± 0.22	0.32 ^a ± 0.04	
Dodecane	—	—	—	—	—	—	—	—	0.48 ^a ± 0.00	0.32 ^{ab} ± 0.04	0.15 ^b ± 0.01	
Hexylcyclohexane	—	—	—	—	—	—	—	—	2.31 ^{bc} ± 0.21	2.34 ^{bc} ± 0.33	1.03 ^{bc} ± 0.13	
2,6-dimethyl undecane	—	—	—	—	—	—	—	—	1.48 ^a ± 0.10	1.23 ^a ± 0.11	—	
Cis-decanyl-2syn-methyl	—	—	—	—	—	—	—	—	—	—	—	
Decanyl-syn-1-methyl-cis	—	—	—	—	—	—	—	—	—	—	—	
2,6-dimethyl undecane	—	—	—	—	—	—	—	—	—	—	—	
Trans, trans-1,6-dimethylspiro(4,5)decane	—	—	—	—	—	—	—	—	—	—	—	
Z,Z-3ethylbicyclo(4,4,0)decane	—	—	—	—	—	—	—	—	1.19 ^a ± 0.09	1.06 ^b ± 0.03	0.50 ^b ± 0.06	
Cyclododecane	—	—	—	—	—	—	—	—	—	0.44 ^a ± 0.48	2.44 ^b ± 1.80	
10-methyl nonadecane	—	—	—	—	—	—	—	—	—	2.09 ± 0.18	—	
1,1-dimethyl cyclohexane	—	—	—	—	—	—	—	—	0.81 ^a ± 0.03	0.75 ^a ± 0.19	0.38 ^b ± 0.10	
4-methyl dodecane	—	—	—	—	—	—	—	—	1.50 ^a ± 0.07	1.03 ^a ± 0.18	—	
10-methyl nonadecane	—	—	—	—	—	—	—	—	—	2.09 ± 0.18	—	
Tridecane	—	—	—	—	—	—	—	—	6.32 ^a ± 0.25	6.11 ^a ± 0.57	3.17 ^b ± 0.09	
1,1-dimethyl cyclohexane	—	—	—	—	—	—	—	—	0.81 ^a ± 0.03	0.75 ^a ± 0.19	0.38 ^b ± 0.10	
1,1-bicyclohexyl	—	—	—	—	—	—	—	—	0.74 ^a ± 0.01	0.65 ^a ± 0.18	0.36 ^b ± 0.08	
Heptylcyclohexane	—	—	—	—	—	—	—	—	1.76 ^{ab} ± 0.04	1.10 ^{ab} ± 0.06	2.10 ^b ± 0.89	
5-methyltridecane	—	—	—	—	—	—	—	—	0.59 ^a ± 0.06	0.58 ^a ± 0.07	0.73 ^a ± 0.42	
4-methyltridecane	—	—	—	—	—	—	—	—	0.63 ^a ± 0.08	0.75 ^a ± 0.06	0.59 ^a ± 0.06	
2-methyl tridecane	—	—	—	—	—	—	—	—	—	0.72 ± 0.15	—	
Tetradecane	—	—	—	—	—	—	—	—	3.48 ^a ± 0.41	3.17 ^a ± 0.29	2.22 ^b ± 0.27	
Pentadecane	—	—	—	—	—	—	—	—	2.52 ^a ± 0.11	1.97 ^a ± 0.26	1.73 ^{ab} ± 0.09	
Hexadecane	—	—	—	—	—	—	—	—	0.92 ^a ± 0.04	0.37 ^a ± 0.20	—	
9-methyl nonadecane	—	—	—	—	—	—	—	—	1.56 ^a ± 0.09	1.54 ^a ± 0.11	—	
2,6,10-trimethyl dodecane	—	—	—	—	—	—	—	—	—	0.70 ^a ± 0.01	0.78 ^a ± 0.09	
Alkenes												
Total	23.15	193.99	213.49	211.84	77.32	63.14	26.16	1.37	36.96	35.13	20.88	
Cyclohexene	—	1.32 ^a ± 0.23	0.80 ^b ± 0.00	0.45 ^b ± 0.01	—	—	—	—	—	—	—	
1-methyl-4-(1methyl)ethyl	—	—	—	—	—	—	—	—	—	—	—	
2-methyl-3-ethyl	—	1.01 ^a ± 0.28	0.31 ^b ± 0.03	—	—	—	—	—	—	—	—	
1,3-hexadiene	—	—	—	—	—	—	—	—	—	—	—	
1-hexene	—	—	—	—	—	—	—	—	—	—	—	
1-decen	—	—	—	—	—	—	—	—	—	—	—	
2,4,4-trimethyl-1-pentene	—	—	—	—	—	—	—	—	—	—	—	
Alcohols												
Total	—	2.33	1.11	0.45	—	—	—	—	—	—	—	
1-pentanol	—	10.56 ^{ab} ± 1.58	13.86 ^{ac} ± 0.71	7.59 ^b ± 0.31	—	—	—	—	—	—	—	
1-hexanol	—	1.41 ^a ± 0.20	1.51 ^a ± 0.09	1.58 ^a ± 0.05	—	—	—	—	—	—	—	
Heptanol	—	2.40 ^a ± 0.13	3.06 ^b ± 0.16	2.62 ^a ± 0.07	—	—	—	—	—	—	—	
n-octen-3-ol	—	4.69 ^{ab} ± 0.60	4.10 ^{bc} ± 0.26	2.43 ^a ± 0.19	0.46 ^a ± 0.01	0.48 ^a ± 0.05	0.30 ^b ± 0.01	—	—	—	—	
1-octanol	0.07 ^a ± 0.01	2.23 ^b ± 0.44	2.47 ^b ± 0.02	2.24 ^b ± 0.18	0.41 ^{ab} ± 0.03	0.35 ^b ± 0.07	0.52 ^a ± 0.01	—	—	—	—	
1-heptadecanol	—	—	—	—	—	—	—	—	—	—	—	
2-hexyl-1-decanol	—	—	—	—	—	—	—	—	—	—	—	
Alkanals												
Total	0.07	21.29	25.00	16.46	1.32	1.72	1.11	—	2.09 ± 0.30	0.82 ± 0.04	0.61 ± 0.06	
Hexanal	4.88 ^a ± 1.02	60.44 ^b ± 12.28	63.79 ^b ± 4.88	53.07 ^b ± 0.95	48.69 ^{ab} ± 2.63	57.72 ^a ± 6.66	38.67 ^b ± 1.14	4.44 ^a ± 0.33	195.20 ^b ± 1.41	8.24 ^a ± 3.46	71.88 ^a ± 13.89	
Heptanal	0.45 ^a ± 0.16	11.51 ^b ± 1.92	12.85 ^{bc} ± 0.75	11.64 ^b ± 0.63	2.88 ^a ± 0.15	3.63 ^b ± 0.29	2.81 ^a ± 0.00	—	—	—	—	
n-octanal	0.65 ^a ± 0.14	6.53 ^b ± 0.85	8.28 ^b ± 0.29	8.41 ^c ± 0.11	2.26 ^a ± 0.08	2.55 ^a ± 0.46	2.11 ^a ± 0.16	0.19 ^a ± 0.01	0.24 ^a ± 0.01	0.47 ^{ab} ± 0.21	1.35 ^a ± 0.88	
Nonanal	2.34 ^a ± 0.61	23.63 ^b ± 5.71	23.21 ^b ± 2.74	19.39 ^{bc} ± 1.32	8.30 ^a ± 0.09	7.18 ^a ± 0.88	6.65 ^a ± 1.31	1.97 ^a ± 0.01	2.84 ^{ab} ± 0.05	1.96 ^b ± 0.00	5.23 ^a ± 2.31	
Decanal	0.09 ^a ± 0.00	0.74 ^b ± 0.14	0.82 ^b ± 0.14	0.66 ^b ± 0.05	0.18 ^a ± 0.07	0.21 ^a ± 0.07	0.22 ^a ± 0.11	—	—	—	—	
4-oxononanal	—	—	—	0.81 ± 0.01	—	—	—	—	—	—	—	
Dodecanal	—	0.19 ± 0.04	—	—	—	—	—	—	—	—	—	
Total	8.41	103.04	108.95	93.98	6.60	198.28	10.67	6.60	198.28	10.67	78.46	

(Continued)

Table 5-Continued.

Compound (ppb)	Olive oil											
	Thermo-oxidized					Frying						
	0	8	24	48	8	24	48	0	8	24	48	
Alkenals												
2-E-hexenal	—	4.14 ^{ab} ± 0.52	3.49 ^b ± 0.22	2.40 ^c ± 0.12	1.45 ^c ± 0.04	1.29 ^{ab} ± 0.13	0.68 ^c ± 0.08	0.45 ± 0.08	—	—	—	—
2-Z-heptenal	0.38 ^a ± 0.00	21.81 ^{bc} ± 3.64	17.87 ^c ± 0.21	12.06 ^d ± 0.34	6.10 ^a ± 0.02	5.50 ^{ab} ± 0.23	3.57 ^c ± 0.12	—	—	—	—	—
2-E-octenal	—	6.28 ^{ab} ± 1.47	5.33 ^{ab} ± 0.41	4.11 ^{bc} ± 0.07	1.12 ^c ± 0.00	1.08 ^c ± 0.18	1.03 ^c ± 0.01	—	—	—	—	—
2-E-nonanal	—	2.63 ^a ± 0.66	3.21 ^b ± 0.03	3.17 ^b ± 0.45	0.62 ^c ± 0.00	0.50 ^{ab} ± 0.04	0.53 ^{ab} ± 0.00	—	—	—	—	—
2-E-decenal	0.50 ^a ± 0.05	10.79 ^b ± 1.88	12.90 ^b ± 0.41	11.38 ^b ± 1.21	3.26 ^{ab} ± 0.05	2.65 ^b ± 0.19	2.10 ^b ± 0.54	—	—	—	—	—
2-undecenal	—	7.50 ^a ± 1.11	10.38 ^a ± 1.83	10.20 ^a ± 1.89	2.66 ^a ± 0.16	2.36 ^a ± 0.14	1.81 ^b ± 0.57	—	—	—	—	—
Total	0.88	53.15	53.18	43.32	15.21	13.38	9.70	0.45	0.73	1.88	5.14	
2,4-E-heptadienal	—	2.81 ^b ± 0.36	1.73 ^b ± 0.01	0.86 ^c ± 0.09	1.22 ^a ± 0.08	0.31 ^b ± 0.03	0.23 ^b ± 0.07	—	—	—	—	
2,4-E-E-nonadienal	—	0.70 ^a ± 0.22	0.40 ^a ± 0.12	0.28 ^b ± 0.03	—	—	—	—	—	—	—	
2,4-E-E-dodecadienal	—	4.21 ^a ± 1.16	2.78 ^{ab} ± 0.15	—	—	—	—	—	—	—	—	
2,4-E-E-decadienal	—	13.36 ^a ± 3.41	6.56 ^b ± 0.91	3.81 ^{bc} ± 0.90	3.07 ^a ± 0.15	2.52 ^{ab} ± 0.01	1.38 ^a ± 0.43	1.42 ^a ± 0.13	0.78 ^a ± 0.03	0.68 ^a ± 0.08	1.11 ^a ± 0.21	
Total	—	21.08	11.56	4.95	4.29	2.83	1.61	1.42	2.98	1.98	4.13	
Ketones	0.21 ^a ± 0.03	2.24 ^b ± 0.36	3.79 ^c ± 0.19	3.27 ^c ± 0.08	0.45 ^{ab} ± 0.04	0.66 ^b ± 0.04	0.55 ^b ± 0.10	—	—	—	—	
3-octanone	—	—	0.53 ^a ± 0.01	0.39 ^b ± 0.14	—	—	—	—	—	—	—	
2-propylcyclopentanone	—	1.00 ^a ± 0.25	0.65 ^a ± 0.35	—	—	—	—	—	—	—	—	
Total	0.21	3.24	4.97	3.66	0.45	0.66	0.55	—	—	—	—	
Acids	0.35 ^a ± 0.02	4.89 ^b ± 1.37	2.28 ^{bc} ± 0.13	—	1.01 ^a ± 1.09	0.71 ^{ab} ± 0.25	1.07 ^a ± 1.09	0.74 ^a ± 0.09	1.48 ^{ab} ± 0.25	0.49 ^a ± 0.08	2.57 ^{bc} ± 1.53	
Pentanoic acid	—	—	—	—	—	—	—	—	—	—	—	
Hexanoic acid	—	—	—	—	—	—	—	—	—	—	—	
Octanoic acid	—	1.11 ^a ± 0.30	1.60 ^a ± 0.11	—	—	—	—	—	—	—	—	
Decanedioic acid-didecyl ester	0.24 ^a ± 0.07	0.49 ^b ± 0.03	0.50 ^b ± 0.04	1.16 ^{bc} ± 0.09	0.31 ^{ab} ± 0.01	0.13 ^b ± 0.18	0.18 ^b ± 0.25	—	—	—	—	
Butanedioic acid-methyl-bis-(1-methylpropyl)ester	—	—	—	—	—	—	—	—	—	—	—	
Total	0.59	6.49	4.38	1.16	1.32	0.84	1.25	0.74	1.96	1.60	3.01	
Heterocyclic aromatic hydrocarbons	0.43 ^a ± 0.00	5.87 ^b ± 0.95	7.65 ^c ± 0.0	6.50 ^{bc} ± 0.87	0.69 ^a ± 0.03	1.04 ^a ± 0.12	1.26 ^a ± 0.12	—	—	—	—	
2-pentyl furan	—	—	—	—	—	—	—	—	—	—	—	
Hexylfuran	—	—	0.64 ^a ± 0.03	0.78 ^a ± 0.34	—	—	—	—	—	—	—	
2n-heptyl furan	—	—	0.86 ^a ± 0.01	1.00 ^a ± 0.06	—	—	—	—	—	—	—	
Total	0.43	5.87	9.15	8.28	0.69	1.04	1.26	3.50 ^a ± 0.42	1.42 ^b ± 0.13	1.15 ^b ± 0.30	0.29 ^c ± 0.09	
Monoterpenes cyclic Sulfide compounds	—	0.24 ^a ± 0.00	0.92 ^b ± 0.06	0.16 ^a ± 0.02	—	—	—	—	—	—	—	
D-limonene	—	—	—	—	—	—	—	—	—	—	—	
Disulfide-bis-1-methylethyl	—	—	—	—	—	—	—	—	—	—	—	
Ethylbenzene	—	—	—	—	—	—	—	—	—	—	—	
o-xylene	—	—	—	—	—	—	—	—	—	—	—	
Naphthalene-decahydro-2,3-dimethyl	—	—	—	—	—	—	—	—	—	—	—	
Total	—	0.24	0.92	0.16	—	—	—	—	—	—	—	
Heterocyclic aromatic hydrocarbons	—	—	—	—	—	—	—	—	—	—	—	
2,5-dimethyl pyrazine	—	—	—	—	—	—	—	—	—	—	—	
2-pentyl furan	—	—	—	—	—	—	—	—	—	—	—	
Total	—	—	—	—	—	—	—	—	—	—	—	
Total	0.58 ^a ± 0.06	0.81 ^{ab} ± 0.05	0.17 ^{bc} ± 0.02	0.55 ^c ± 0.20	0.58	0.81	0.42	0.58	0.81	0.42	0.55	

*The reliability of the identification or structural proposal is indicated by the following: (1) mass spectrum and retention time consistent with those of an authentic standard; (2) structural proposals are given on the basis of mass spectral data (NIST 02, WILEY 275) with a similar index; (8) > 90%. - Under detection limit (0.010 ppb). a-c Different letters indicate significant differences (P ≤ 0.001 and P ≤ 0.05 for hexanal, octanal, 1-pentanol, and E-2-octenol) between treatment times for the same oil.



FAs could be oxidized to form hydroperoxides and then undergo an intramolecular reaction to result in the formation of a cyclic compound, such as cyclohexene. This degradation product may be further oxidized to form benzene, which in turn reacts with a C4 compound to form naphthalene and other polycyclic aromatic hydrocarbons (Pitts 1983).

o-Xylene (1,2-dimethylbenzene) was identified in fat extracted from French fries produced in both OO and PSO. In potatoes fried in OO, 2 other derivatives of benzene were identified: ethylbenzene and decahydro-naphthalene.

In PSO, styrene was detected and quantified. The main source of public exposure to oral doses of styrene has been estimated to be migration from polymer packaging materials (Tang and others 2000). The amount of styrene migrated increases with the fat content of food because of the high lipid solubility of styrene (Franz and others 1994; Linssen and Reitsma 1995; Tawfik and Huyghebaert 1998). The total styrene intake has been estimated to be about 9 μg per day in the United States (Lickly and others 1995). The maximum value of styrene found in this study was 7 ppb in fat extracted from French fries produced in PSO at the 40th thermal treatment hour. However, there is also a possible mechanism for the formation of styrene from trans, trans-2,4-decadienal. This mechanism provides an explanation for the presence of styrene in samples fried in PSO and its absence in all other samples. It is known that 2,4-decadienal decomposes to either a 2,3-epoxy or 4,5-epoxy derivative, which further decomposes to 2-octenal and acetaldehyde or to 2-octene and glyoxal (Andrikopoulos and others 2003). Methyl glyoxal interacts with L-phenylalanine and leads to the formation of styrene through the formation of 2 intermediate products. Ascoli-Trascrizione fonetica These intermediates, 1-phenylethylaminopropan-2-one and 2-phenylethylamine, play a role in the formation of styrene, with the latter being of more importance in high-moisture systems, while the former favors the release of styrene in low-moisture systems.

In both oils, we also detected etherocyclic aromatic hydrocarbons, such as alkylfurans, particularly 2-pentyl furan.

Because there are many variables that affect oil degradation, a specific method for its detection may be ideal for one frying oil but completely useless for another.

Conclusion

The results obtained from our analytical evaluation of frying oils lead us to conclude that the PSO tested in this study may represent an alternative to OO as a frying medium. Although this particular PSO showed a faster increase than OO in some oxidation indices, such as FFA and TPC, the PSO showed better behavior than OO for other indicators (less formation of C8:0 and lower PV). Several studies (Naghshineh and others 2010) have indicated that palm oil exhibits similar frying performance to *high oleic* oils, with the advantage of greater availability on the market and a lower price. In addition, about VOCs the OO showed the presence of sulfide compounds (disulfide bis, 1-methylethyl) and of 2,5-dimethyl pyrazine. The alkylsulfide are VOCs produced from food-oil interaction. In particular, from the reaction between glucose and methionine is formed methionale, a classical Strecker aldehydes, which can degrade to methanethiol which in turn produces n-alkyl sulfides. It has also been shown that oxidized lipids in the presence of methionine are easily oxidized to methionine sulfoxide, from which are formed alkyl sulfides. On the other hand pyrazines are formed by reaction between α -dicarbonyl compounds formed during frying with amino-acids and proteins. In PSO samples these compounds did not appear. The present study indicated that the PSO oil provided higher chemical stability compared to OO. This could be explained by the fact that the presence of a high content of saturated FAs (that is, C16:0 and C18:0) resulted in an increase in the chemical stability of PSO oil frying.

Most of the chemical and physical degradation indicators addressed in this study suggested that degradation, mainly thermal degradation, was faster in the heated oil samples compared with the frying samples. Our results lead to the conclusion that the rate of the chemical reactions involved during heating conditions is different from those occurring during the frying of food. These findings must be confirmed by extending this research to a larger number of samples, and further information could be provided by an analysis of other compounds. With respect to the question of when frying oil should be discarded, because there are many variables that affect oil degradation, a specific method to determine this may be ideal for one operation but completely useless

for another, and no single parameter can be used to judge heating life adequately in all situations.

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