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ORIGINAL PAPER

Changes in Total Polar Compounds, Peroxide Value, Total Phenols and Antioxidant Activity of Various Oils Used in Deep Fat Frying

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Abstract In this study, the effect of deep fat frying on oil degradation, total phenols (TP) and total antioxidant activity (TAA) of hazelnut, corn, soybean and olive oils were investigated. Oil degradation and oxidation were monitored by measuring the total polar compounds (TPC) and the peroxide value (PV). The amount of TPC in corn, soybean and olive oils increased significantly with the time increment ($p < 0.05$). The PV of the oils did not exceed the maximum acceptable limit of 10 mequiv O_2/kg after 125 min frying except for hazelnut oil (10.64 mequiv O_2/kg). Deep-fat frying did not cause any significant change in the TP of corn oil, soybean oil and olive oil ($p < 0.05$). A significant decrease in the antioxidant activity was observed after 50 min frying using hazelnut oil and corn oil ($p < 0.05$). However, the antioxidant activity of soybean oil and olive oil significantly decreased after 75 and 25 min frying, respectively.

Keywords Deep fat frying · Peroxide value · Total antioxidant activity · Total phenols · Total polar compounds

Introduction

Frying of foods has become the most common food preparation technology during the last six decades. Deep fat frying is the most common frying method used in the food service industry [1]. Deep fat frying can be defined as the cooking process in which foods are immersed in an edible oil or fat maintained at a temperature of about 150–200 °C [2]. In deep fat frying, the layer of the frying oil is about 20–200 mm or greater and frying oil is reused several times [3, 4]. The quality of the products cooked by deep fat frying depends not only on the frying conditions such as temperature of the heated oil, frying time, food weight and frying oil volume, but also on the types of oil and the kind of food used [5].

Tocopherols and phenolic compounds are of great importance as natural antioxidants of vegetable oils and are also added to oils for improving their stability against oxidation. Crude vegetable oils also contain different components such as sterols, carotenoids, phospholipids, which increase their stability during the frying process [6]. During the frying process via a series of complex physical and chemical reactions, oils are subjected to thermal oxidation, polymerization, and hydrolysis. These reactions lead to a decrease in tocopherols and total phenols (TP), an increase in the peroxide value (PV) and formation of decomposition products with high molecular weights such as polar compounds and polymeric triacylglycerides [3–7]. Formation of polar compounds is strongly related with the primary and secondary oxidation that takes place during frying and it comprise an established quality index for frying oils with 20–25% limits for rejection or replenishment of the cooking oil due to negative effects on the quality of frying oil and the flavor and nutritional value of the fried food [8, 9]. Some of these compounds may also be harmful to human health [4].

Different types of oils such as corn oil, soybean oil, sunflower oil, palm oil and canola oil can be used for frying. The chemical composition of the frying oil and its physical and physicochemical properties has an influence both on the frying process and on the stability characteristic of oil against oxidation and decomposition. Therefore, the

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importance of the correct selection of the oil for frying is one of the most considerable issues [6]. Effects of domestic deep-frying of potatoes on the oxidation process of different types of oils including soybean oil [10], virgin olive oil [11], sunflower oil [12] and corn oil [13] have been studied. In the study of Andrikopoulos et al. [11] potatoes were deep-fried in virgin olive oil at 170 °C. The oil was used ten times for a total frying time of 120 min. At the end of the deep-frying process, it was found that total polar compounds did not exceed the 25–27% limit. In general the oil must be able to withstand high temperatures and have high enough stability to be reusable. Furthermore, the oil needs to maintain a high oxidative stability during the life of the product and it should be originated a low emission of potentially toxic volatile organic compounds [14].

The aim of this study was to determine the changes in total polar compounds (TPC), peroxide values (PV), total phenols (TP) and total antioxidant activities (TAA) of hazelnut oil, corn oil, soybean oil and olive oil used under domestic household frying conditions.

Materials and Methods

In the present study, four oils were used to fry potato chips over two replicates of 125 min trials. The oils were hazelnut oil, corn oil, soybean oil and Riviera type olive oil (mixture of virgin plus refined olive oil). Potato chips were prepared by slicing fresh potatoes into 8 mm × 8 mm dimensions using a potato slicer. Oils (3 l) were placed in the fryer (SEB de Luxe 8225 T). The oil was heated to 190 ± 2 °C. Potato chips (75 g) were fried in each oil for 8 min. After each frying process, the oils were allowed to cool for 7 min and then frying and cooling steps were repeated 15 times. To minimize other effects on oil quality, the oils were not filtered and only those floating objects on the top of the oil were removed during the frying process. Samples were taken every 25 min (25, 50, 75, 100 and 125 minutes) and analyzed or stored at −40 °C until being analyzed.

Chemicals

Folin–Ciocalteu's phenol reagent (F 9252) and DPPH (2,2 diphenyl-1-picryl-hydrazyl, D 9132) were purchased from Sigma-Aldrich (Germany), Gallic acid (48630) was obtained from Fluka. All other chemicals used were of analytical grade.

Spectrophotometric Measurements

Oil samples were placed in a standard disposable cuvette and were scanned from 470 to 500 nm wavelength using a UV–visible spectrophotometer (Cary 50 Scan UV–visible Spectrophotometer, Victoria, Australia). The spectrophotometer absorbance was zeroed against air. At first the oils were scanned from 350 to 650 nm wavelengths and then individual absorbance values of each frying oils were obtained at 470, 480, 490, and 500 nm where systematic changes were most evident.

Total Polar Compounds

Total Polar Compounds (TPC) of the oils were evaluated using spectrophotometric method proposed by Xu [3]. This method is simply based on the correlation between total polar compound contents (mainly resulting from the free fatty acids, dimers, polymers and other decomposition products) in oils and spectrophotometric absorbances of the oils. The equation used for conversion of the spectrophotometric absorbance to TPC content was $y = -2.7865x^2 + 23.782x + 1.039$, where y is the TPC of oil samples and x is the absorbance of oil samples at 490 nm after each frying period [3].

Peroxide Value

The Peroxide Value (PV) of the oils was determined by AOCS Official Method Cd 8b-90 [15] except that 0.01 N $Na_2S_2O_3$, instead of 0.1 N $Na_2S_2O_3$, was used as the titrant.

Total Phenol

Total Phenol (TP) of the oils were determined according to Folin–Ciocalteu's method [16]. Gallic acid was used as the standard and the results were given as gallic acid equivalents (GAE). Although Folin–Ciocalteau reagent can react with reducing substances in the extract, it detects all phenolic groups found in the extracts, including phenolics in extractable proteins; however, this assay has been commonly used to determine total phenolics [17]. Briefly for the extraction of phenolic compounds, 10 g of oil was mixed with hexane (50 ml). This mixture was placed into a separation funnel and 20 ml of methanol:water (60:40, w:w) was added. The mixture was shaken for 2 min and the separated water phase was removed. This procedure was applied three times and all the water phases were combined and used for TP analysis. For TP analysis, the mixture of the sample solution (50 µl), distilled water (3 ml), 250 µl Folin–Ciocalteu's reagent solution, and 7% Na_2CO_3 (750 µl) was vortexed and incubated for 8 min at room temperature. Then 950 µl of distilled water was added. The mixture was allowed to stand for 2 h at room temperature. The absorbance was measured at 765 nm against distilled water as blank [18].

DPPH Radical Scavenging Activity

The total antioxidant activity (TAA) of the oils were determined by the free radical scavenging activity using 2,2-diphenyl-1-picrylhydrazyl radical (DPPH) according to the method proposed by Brand-Williams et al. [19]. Briefly, 50 µl of oil was added to 950 µl of 0.030 mg/ml methanol solution of DPPH. Then, the mixture was shaken vigorously and left in darkness for 5 min. Finally, the absorbance of the mixture was measured against methanol (blank) at 515 nm by a spectrophotometer (Cary 50 Scan UV–visible Spectrophotometer). The DPPH scavenging activity was expressed as the inhibition of free radical DPPH.

$$\text{Inhibition } (\%) = \left(1 - \frac{A_{\text{sample}}}{A_{\text{Blank}}}\right) \times 100$$

Where, A_{sample}:absorbance of the sample, A_{blank}:absorbance of the DPPH.

Statistical Analysis

The data were statistically analyzed using one-way ANOVA and correlation test using the statistical software SPSS 13.0.

Results and Discussion

The absorbance values of each oil measured at 470, 480, 490 and 500 nm, respectively, all increased significantly during frying (Fig. 1) and were all significantly correlated with frying time ($p < 0.05$). Xu [3] reported that systematic changes in spectrophotometric absorbance for each frying oil (high oleic-canola oils, palm olein and two blends of palm olein and canola oil) were obtained most evidently between the wavelengths of 470–500 nm. The correlation values (r^2) between frying time and absorbance changes at 470, 480, 490 and 500 nm for the oils analyzed were given in Table 1.

The r^2 values obtained for absorbance changes at 470 nm were greater than those obtained from other wavelengths (Table 1). Xu [3], indicating that absorbance values obtained at 490 nm had the highest r^2 value ($\geq$0.992) for most of the oils tested. In the present study, r^2 values decreased in the order of 470 > 480 > 490 > 500 nm for the oils tested except for hazelnut oil. The lowest r^2 value was obtained at 490 nm for hazelnut oil. However, the absorbance readings at 490 nm were chosen for the TPCs determinations because, Xu [3] determined a strong correlation between absorbance readings at 490 nm and TPCs. In our study we used the

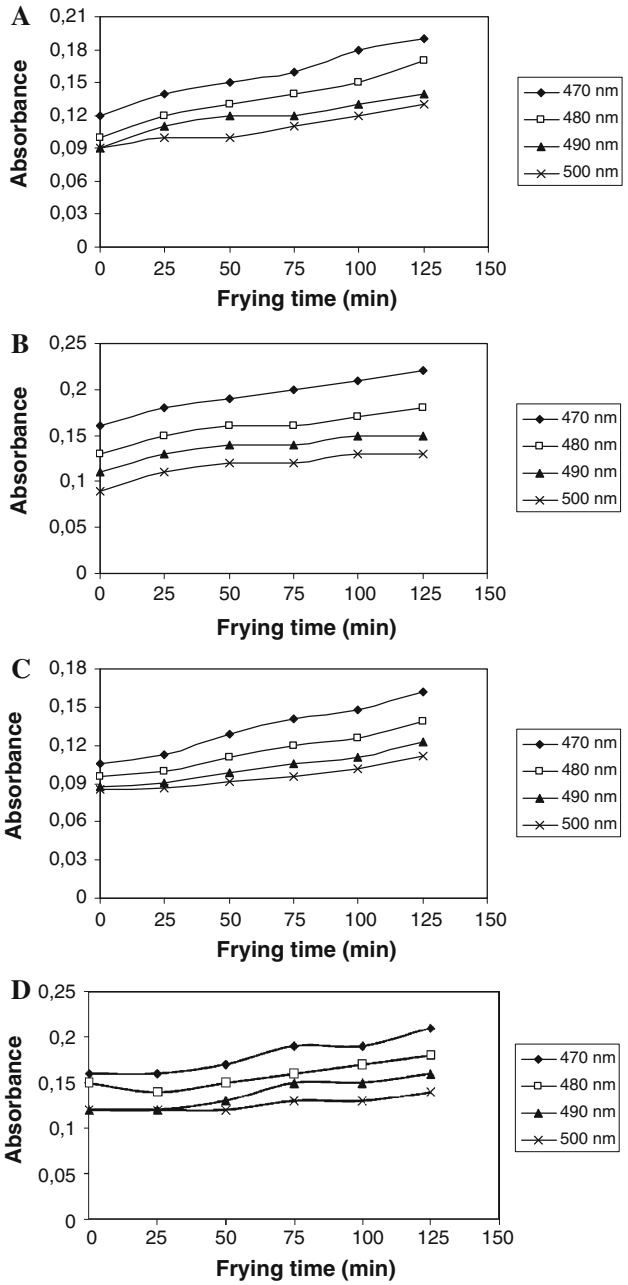

Fig. 1 Changes in absorbances during frying. Hazelnut oil (**a**), corn oil (**b**), soybean oil (**c**), olive oil (**d**)

Table 1 r^2 values obtained for frying time versus absorbance changes measured at 470, 480, 490 and 500 nm

Frying oils	r^2 values (nm)			
	470	480	490	500
Hazelnut oil	0.987	0.965	0.926	0.961
Corn oil	0.980	0.926	0.852	0.852
Soybean oil	0.991	0.987	0.971	0.934
Olive oil	0.926	0.824	0.844	0.840

equation that Xu [3] found for absorbance readings at 490 nm and TPCs.

Formation of polar compounds, which indicates oil deterioration, is strongly related with the primary and secondary oxidation that takes place during frying. When the amounts of TPCs reach 24% levels, oil is considered to be thermally degraded and should be replaced with fresh oil [3, 10]. TPCs formed during deep fat frying process are given in Table 2. The amount of TPCs in corn oil, soybean oil and olive oil increased with the time increment ($p < 0.05$). Although the amount of TPCs in hazelnut oil increased during frying, this increase was not found to be statistically significant. At the end of 125 min frying time, TPCs in all oils analyzed were found to be lower than 5% level. This result indicated that all of the oils analyzed could be used for frying of potato chips up to 125 min frying period. Gil et al. [20] reported that the amount of polar compounds in fresh oils namely palm oil, soybean oil, shortening and beef tallow were 8% or less and reached 30% after 80 h frying except soybean oil. At the end of 80 h frying, TPCs in soybean oil reached about the 15% level. Benedito et al. [21] applied frying with extra virgin olive oil for 16 h at 200 °C. At the end of the incubation period, they observed that the TPC content of virgin olive oil had reached about 45.7%, whereas in fresh oil it was 6.2%. Sanchez-Gimeno et al. [5] also studied the deterioration of extra virgin olive oil and high oleic sunflower oil during different frying cycles. The amount of polar compounds increased linearly with the frying cycle, however, the increase was faster in high oleic sunflower oil. After 60 frying cycles, the amount of polar compounds for both oils reached approximately 20–23%.

The PV is useful in monitoring the initial stage of oxidation, where the primary oxidation products are measured. According to Turkish Codex Standards, the the PVs of Riviera type olive oil and vegetable oils should not exceed 15 and 10 mequiv O_2/kg, respectively [22]. The PVs of frying oils obtained at 25, 50, 75, 100 and 125 min frying are shown in Table 2. PVs of frying oils analyzed decreased after 125 min frying except corn oil. This decrease can be explained by the formation of secondary oxidation products such as hydrocarbons, alcohols, ketones and aldehydes from very unstable primary oxidation products, i.e., hydroperoxides. As reported elsewhere, the PV decreases as oxidation proceeds due to rapid decomposition of hydroperoxides [23]. The PV of corn oil increased following 125 min frying ($p < 0.05$). This result can be explained as that primary oxidation continues during 125 min frying. However, in general, according to the results of the study, PVs of the oils analyzed did not exceed the maximum acceptable level of 10 mequiv O_2/kg at the end of the 125-min frying period except hazelnut oil. The PV of hazelnut oil after 125 min frying was 10.64 mequiv O_2/kg. Naz et al. [24] reported that deep frying of French fries at 180 °C caused an increase in PVs of the oils. The PVs with respect to the oils, increased as soybean > corn > olive.

Table 2 Total polar compounds and peroxide values of frying oils

Frying oils	Frying time (min)	Total polar compounds (%)[a]	Peroxide value (mequiv/kg)[a]
Hazelnut	0	2.77 ± 0.00^{a}	10.17 ± 0.83^{a}
	25	3.10 ± 0.47^{a}	11.96 ± 0.06^{ab}
	50	3.32 ± 0.47^{a}	14.32 ± 1.09^{c}
	75	3.54 ± 0.47^{a}	11.34 ± 1.07^{ab}
	100	3.76 ± 0.77^{a}	12.38 ± 0.33^{b}
	125	3.98 ± 0.77^{a}	10.64 ± 0.96^{ab}
Corn	0	2.99 ± 0.00^{a}	3.02 ± 0.08^{a}
	25	3.43 ± 0.00^{b}	3.05 ± 0.20^{a}
	50	3.65 ± 0.00^{b}	3.92 ± 1.04^{a}
	75	3.76 ± 0.16^{bc}	4.23 ± 0.64^{a}
	100	4.10 ± 0.00^{c}	4.29 ± 0.42^{ab}
	125	4.20 ± 0.15^{cd}	5.58 ± 0.63^{b}
Soybean	0	2.72 ± 0.00^{a}	8.15 ± 1.69^{a}
	25	2.77 ± 0.00^{b}	7.91 ± 2.03^{ab}
	50	2.95 ± 0.00^{c}	6.12 ± 2.22^{ab}
	75	3.11 ± 0.00^{d}	4.41 ± 1.37^{ab}
	100	3.23 ± 0.00^{e}	4.30 ± 1.26^{ab}
	125	3.48 ± 0.00^{f}	4.07 ± 1.55^{b}
Olive	0	3.98 ± 0.15^{ac}	8.85 ± 0.50^{a}
	25	3.54 ± 0.15^{b}	5.65 ± 0.01^{b}
	50	3.76 ± 0.16^{ab}	6.53 ± 0.33^{b}
	75	4.09 ± 0.00^{ac}	5.74 ± 0.91^{b}
	100	4.10 ± 0.00^{ac}	4.80 ± 0.14^{b}
	125	4.31 ± 0.00^{c}	5.85 ± 0.00^{b}

Different letters in the same column indicate significant differences among frying times in the same oil ($p < 0.05$)

[a] Means ± standard deviation

The TP contents of 1,280, 1,240, 1,030, and 1,190 mg GAE/kg oil in hazelnut, corn, soybean, and olive oils, respectively, were not significantly ($p > 0.05$) different (Table 3). Frying did not cause significant change in the TP content of corn oil, soybean oil and olive oil. The TP content was expected to decrease with frying time due to the thermal degradation of phenolic compounds; however, none of the oils had significant reductions in TP content. The frying times may not have been long enough to promote degradation of phenols and thus producing the non-significant reduction in TP content. It is well known that potatoes and other plant foods accumulate different kinds of secondary metabolites including phenolic compounds. The major phenol in potato has been reported as chlorogenic acid [25]. Quiles et al. [26], reported that frying time, type of oils and interaction between time and oil had effects on the TP content of the oils. In the study of

Table 3 Changes in total phenol content of the oils during frying

Frying time (min)	Total phenols (GAE)[c] (ppm)			
	Hazelnut oil	Corn oil	Soybean oil	Olive oil
0	1,280 ± 220[a]	1,240 ± 115[a]	1,030 ± 2[a]	1,190 ± 80[a]
25	1,130 ± 210[ab]	1,180 ± 220[a]	1,040 ± 25[a]	1,040 ± 85[a]
50	990 ± 5[b]	1,415 ± 205[a]	1,050 ± 40[a]	1,140 ± 85[a]
75	1,000 ± 30[ab]	1,235 ± 115[a]	1,055 ± 1[a]	1,095 ± 40[a]
100	980 ± 35[b]	1,120 ± 50[a]	1,110 ± 60[a]	1,095 ± 2[a]
125	1,145 ± 70[ab]	1,155 ± 65[a]	1,115 ± 60[a]	1,095 ± 1[a]

Different letters in the same column indicate statistically significance ($p < 0.05$)

[c] Gallic acid equivalent (means ± standard deviation)

Gómez-Alanzo et al. [27], the concentration of the dihydroxyphenol components of virgin olive oil was found to be reduced to 50–60% of the original value at the end of the first frying and after six frying process only about 10% of the initial components remained. However, tyrosol and its derivatives were reported to be much more stable during 12 frying operations. Kalogeropoulos et al. [25], reported that shallow frying of potatoes, zucchinis, green peppers and eggplant resulted in partial loss of all the antioxidants namely α-tocopherol, polyphenols and hydroxyl pentacyclic triterpene acids and enrichment of fried vegetables with olive oil antioxidants.

Changes in TAA during the frying process are shown in Table 4. A significant decrease in TAA was observed after 50 min of frying with hazelnut oil (from 67 to 47%) and corn oil (from 87 to 75%) ($p < 0.05$). However, the TAA of soybean oil (from 86 to 75%) and olive oil (from 22 to 18%) significantly decreased after 75 min frying and 25 min frying, respectively ($p < 0.05$). Olive oil had the weakest TAA, which was likely due to the type of olive oil, i.e., the Riviera type that contains refined oil. It is well known that the refining process can cause a decrease or loss in tocopherols, which were not analyzed in this study. Quiles et al. [26], reported that virgin olive oil and sunflower oil had approximately two times more TAA than olive oil before and after different frying times. They also indicated that the determination of same antioxidant response for virgin olive oil and sunflower oil with a different fatty acid compositions. Quiles et al. [26] postulated that the tocopherols were being consumed in the sunflower oil resulting in constant antioxidant activity as measure by electron spin resonance. There was a significant correlation between TPC and TAA for all of the oils analyzed (Table 5). Similarly, Quiles et al. [26] reported that antioxidant capacity of the edible oils was mainly correlated with the amount of antioxidants present in the oils, with polar compounds and ultraviolet indices. A correlation was found between TP and TAA ($r = 0.559$) only for olive oil which represents an increase in TAA positively related with the increase in TP content ($p < 0.05$). A correlation between TPC and TP was obtained for hazelnut oil and soybean oil. Although there was a negative relationship between TPC and TP of hazelnut oil, a positive relationship was determined between TPC and TP of soybean oil.

Table 4 Changes in total antioxidant activity of the oils during frying

Frying time (min)	Total antioxidant activity[c] (inhibition %)			
	Hazelnut oil	Corn oil	Soybean oil	Olive oil
0	67 ± 1[a]	87 ± 4[a]	86 ± 2[a]	22 ± 4[a]
25	60 ± 6[a]	87 ± 1[a]	80 ± 2[a]	18 ± 2[b]
50	47 ± 4[b]	75 ± 2[b]	82 ± 3[a]	15 ± 2[b]
75	41 ± 2[b]	73 ± 6[b]	75 ± 4[b]	16 ± 1[b]
100	35 ± 2[b]	68 ± 2[b]	72 ± 3[b]	15 ± 2[b]
125	36 ± 2[b]	68 ± 1[b]	64 ± 3[b]	12 ± 1[b]

Different letters in the same column indicate statistically significance ($p < 0.05$)

[c] Means ± standard deviation

Table 5 Correlation coefficient for the TPC and AA, TP and AA, TPC and TP

Oils	Correlation coefficients (r)		
	TPC × AA	TP × AA	TPC × TP
Hazelnut	−0.897[a]	0.267	−0.506[b]
Corn	−0.903[a]	0.287	−0.282
Soybean	−0.908[a]	−0.443	0.674[a]
Olive	−0.483[b]	0.559[b]	−0.091

[a] Correlation is significant at the 0.01 level ($p < 0.01$)

[b] Correlation is significant at the 0.05 level ($p < 0.05$)

Conclusion

TPCs are accepted as being good markers to determine changes in oil composition after short time frying. The

TPCs in all oils analyzed were found to be lower than 5%, indicating that the oils were suitable for frying up to 125 min of use. Only hazelnut oil had a PV (10.64 mequiv O_2/kg) above the acceptable limit after 125 min of frying. No significant loss of phenolic compounds was detected. However, significant reductions in TAA were observed indicating that phenolics may not have be the active compounds and that tocopherols may be responsible for slowing oxidation of the oils. Based on the results, all oils except hazelnut can be used to fry potato chips up to 125 min before being discarded.

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