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Article

Effect of Sesame Oil Addition during Olive Oil Deep-frying

Evangelia T. Ioannou [1] and Constantinos A. Georgiou [1,2,*]

[1] Chemistry Laboratory, Department of Food Science and Human Nutrition, Agricultural University of Athens, 75 Iera Odos, 118 55 Athens, Greece
[2] FoodOmics.GR Research Infrastructure, Greece
*Corresponding author: cag@aua.gr

Abstract: Fresh potatoes were deep-fried in olive oil (OO) & extra virgin olive oil (EVOO) and their blends with 5%, 10% and 20% v/v sesame oil (SO). This is the first report on the use of sesame oil as natural source of antioxidant for olive oil deep-frying. Oil was evaluated for Peroxide Value (PV), Free Fatty Acids (FFA), K_{232}, K_{270}, Trolox Equivalent Antioxidant Capacity (TEAC) and Total Phenols (TP) until Total Polar Compounds (TPCs) reached 25%. Sesame lignan transformations were monitored through Reverse-phase HPLC. While TPCs in olive oils increased at a steady rate, the addition of 5%, 10% and 20% v/v SO created a time window lasting 1, 2 and 3 hours, respectively, where TPCs were constant. SO addition to OO increased the total frying time. Furthermore, the addition of SO reduced the peroxides formation rate for both OO and EVOO. EVOO was more resistant to oxidation than OO as measured by TPCs and TEAC, while frying time raised from 21.5 to 25.25 h when EVOO replaced OO. The increase in frying time for olive oil but not for extra virgin olive oil, after SO addition, is pointing out a niche market for extra virgin olive oil in deep-frying.

Keywords: deep frying; olive oil; sesame oil; sesame lignans; Total Polar Compounds

1. Introduction

Deep frying is one of the world's most popular culinary processes, both for industrial and domestic food preparation purposes. During deep frying food is immersed in hot oil at temperatures of 150 to 190^{o}C and in the presence of air many complex reactions take place such as oxidation, hydrolysis and polymerization [1,2,3]. These reactions influence the quality of the final product such as flavor, texture, shelf life of the oil and nutrient composition, with potential adverse effects on human health [4]. The type of frying oil, its chemical composition and its physical and physicochemical properties are major parameters that influence the chemical reactions and determine the performance of the frying oil against oxidation and decomposition [5].

Over the ages, Olive Oil has been widely produced and consumed in Mediterranean countries, while it is also considered to be the main lipid source in the Mediterranean diet. Its beneficial properties are associated with fatty acid composition, phenolic antioxidants and other minor compounds that make olive oil a very interesting option among heating oils and fats [6,7]. Extra Virgin Olive Oil exhibits high resistance to oxidation, in comparison with other vegetable oils, and it is well known for its very good sensory and health properties [8,9]. Olive oil is resistant to degradation under domestic frying conditions, independently of its category label [10]. Olive oils naturally occurring antioxidants play a significant role in the thermal stability during deep frying [11]. Synthetic and natural antioxidants can be added to prevent or minimize the oxi-dative deterioration of the oil. The most commonly used antioxidants are butylated hydroxy anisole (BHA), butylated hydroxytoluene (BHT), propyl gallate (PG) and tert-butyl hydro-quinone (TBHQ). However, the use of synthetic antioxidant additives is regulated in most countries because of the concerns regarding their long-term health effects [3]. Natural components in foods with radical scavenging or antioxidant activ-ity have attracted interest as alternatives to synthetic antioxidants. Addition of sources of natural antioxidants can possibly be used for

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the improvement of olive oils resistance to the formation of primary and secondary oxidation products without mak-ing considerable changes in their natural composition [12]. Sesame oil demonstrates higher oxidative stability than other vegetable oils [2,13]. The study of this superior oxidative stability has mainly been focused on sesame lignans, which are present in small amounts in sesame oil. Sesamin and sesamolin are the major lignans found in sesame seeds [14,15]. When sesame seeds are roasted at high temperature, sesamolin degrades into sesamol [16]. Sesamol is reported to process higher radical scavenging activity compared to sesamin and sesamolin [17]. The significant stability of sesame oil could be related with the continuous generation of sesamol from the degradation of sesamolin during thermal oxidation rather than the initial antioxidant content [18].

The addition of sesame lignans in oils during frying increased sesamol and decreased sesamolin, while sesamin was rather resistant to heat. Sesame lignans could have ap-plication as natural antioxidants in the edible oil and food industry [19]. Other studies suggest that lignan compounds in sesame oil are effective antioxidants in deep-fat frying due to their high stability and efficacy [2,20]. The addition of roasted sesame oil, as a natural source of antioxidants, prolonged the oils heat stability and shelf life. Moreover, when the roasted sesame oil concentration increased, the antioxidant capacity of frying oils increased [21].

Consumers' perception of the health benefits of olive oil creates a niche marked for olive oil for deep frying despite its high price. Consumers nowadays may have a different perception of extra virgin olive oil, recognizing its superiority over olive oil. This creates the need to further study these two olive oil products during deep frying and assess the benefits of adding antioxidants as blends with natural products that will not deteriorate consumers' perception of the health benefits of olive oil. Although olive oil deep frying characteristics have been studied, we were not able to find any study with sesame oil admixtures. In this study we describe the effect of adding virgin sesame oil as a source of natural antioxidants at different levels in extra virgin olive oil and olive oil during domestic deep frying. Sesame lignans changes in oil samples during deep frying and the correlation between the sesame oil antioxidant activity and its efficacy in retarding the oxidative deterioration of olive oil and extra virgin olive oil were in-vestigated. In order to evaluate the oxidation progress, we monitored total polar com-pounds, peroxide value, free acidity, K_{232}, K_{268}, total phenols and antioxidant activity.

2. Materials and Methods

2.1. Reagents.

All reagents were of analytical, chromatographic or spectroscopic grade and were supplied by Merck, Sigma–Aldrich or Honeywell Fluka.

2.2. Oil Samples.

A commercial extra virgin olive oil (EVOO), a commercial blend of refined olive oil with virgin olive oil labeled as olive oil (OO) purchased from a major local manufacturer, and commercial virgin sesame oil (SO), purchased from another major local manufacturer, were used for the frying experiments. The oils were purchased from local stores in Athens, Greece in sealed and marked commercial containers.

2.3. Frying process.

A domestic deep-fat electric fryer (KENWOOD DF520) with 2.5 L capacity was used for frying where temperature was regulated at 170 ± 5 °C. Fresh potato slices (7 cm x 0.5cm x 0.5 cm) of spunda variety, cultivated in Greece, were deep fried in 70 g batches at constant frying temperature throughout all frying sections. The batches were fried at 9 min intervals for 12 h per day for two consecutive days, without oil replenishment, until oil was discarded. The end of frying assay and oils rejection point were determined by the value of total polar compounds (max. 25%), according to the regu-lation of frying fats and oils in most European Countries [22].

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2.4. Deep frying oil sampling.

After each frying session, lasting 15 min, TPCs were accessed directly on the hot oil. Peroxide Value (PV), Extinction Coefficients (K_{232}, K_{270}), Antioxidant Capacity (AC), Free Fatty Acids (FFA) and Total Phenols (TP) were accessed by removing a 6 ml sam-ple a) before frying potatoes, right after thermal equilibration of the oil to 170±5 °C, b) at 12h and c) at the oil rejection point, when TPC reached 25%. Samples were placed in screw caped glass vials and were immediately stored in freezer until analysis. To monitor sesame lignan transformations, 2 ml oil samples were removed before frying, after 1 h, 2 h and 4 h of frying.

2.5. Total polar compounds.

TPCs estimation was based on the dielectric constant changes, measured directly on the hot oil, with a Testo 270 sensor (Testo, Germany). About 5 min were allowed after removing the fried potatoes until there were no more bubbles rising before measuring. The sensor took about 1 min to get a stable reading. TPCs % content along with the temperature were displayed on the screen of the sensor. Sensor calibration was performed through oil, supplied by the Testo 270 manufacturer, right before the analysis. The sensor was cleaned with warm water and neutral detergent and dried well between measurements.

2.6. HPLC analysis.

Sesamin and sesamolin were isolated and crystallized from sesame oil, as described by Hemalatha and Ghafoorunissa, 2004 [23]. Sesamin and sesamolin were characterized by mass spectrometry and 1H- NMR and 13C- NMR spectrometry. Sesamol was purchased from Sigma Chemical Co. (St. Louis, MO, USA). HPLC analysis was carried out on an Agilent technologies 1100 series with Diode array detector. Sesame lignans were analyzed as described by Wen-Huey Wu ,2007 [24], using HPLC equipped with a Supelco Analytical HPLC column (25 cm x 4.6 mm i.d., 5 lm film, HS C18). The mobile phase was a mixture of methanol deionized water (70:30, v/v) at a flow rate of 0.8 ml/min. Absorption at 290 nm was monitored. Twenty microliter aliquots of oils, dissolved in chloroform (0.5 mg/ml), were injected for analysis. The retention times for sesamol, sesamin, and sesamolin were 4.6, 15.6 and 20.8 min, respectively.

2.7. Analytical methods.

Peroxide Value (PV), extinction coefficients (K_{232} and K_{270}) and free acidity of the oils were determined according to analytical methods described in European Commission Regulations [25]. Total antioxidant capacity was determined through DPPH radical. Oil sample 65 mg was added to 4 ml of 1.3×10^{-4} M solution of DPPH in ethyl acetate. Then, the mixture was shaken vigorously and left in darkness for 1 hour. Finally, the absorbance of the mixture was measured against ethyl acetate (blank) at 515 nm by a spectrophotometer (Cary 60 Scan UV–visible Spectrophotometer). Total antioxidant capacity was expressed as Trolox equivalent antioxidant capacity (TEAC) defined as the mmol Trolox/kg of oil [26]. Total phenols in the methanolic extract of the oils were determined colorimetrically at 765 nm with Folin-Ciocalteau reagent according to Capannesi et al., 2000 [27]. Gallic acid standard solutions were used for calibration (r=0.9998).

3. Results and Discussion

3.1. Total Polar Compounds.

Formation of polar compounds indicates oil deterioration and is strongly related to primary and secondary oxidation during frying [28,29]. Frying oil is discarded when TPCs reach 25 % [22]. TPCs increased linearly, as shown in Figure 1 for OO and Figure 2 for EVOO.

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OO frying time increased from 21.5 to 23, 25 and 24 h upon addition of 5, 10 and 20 % SO, respectively. During this procedure, the amount of TPCs was constant for a certain time window. The window was proportional to SO concentration being 1, 2, 3 h for 5, 10 and 20 % SO, respectively. Then, TPCs increased at a steady rate of 1% per hour for all three blends that was lower than 1.5% for OO.

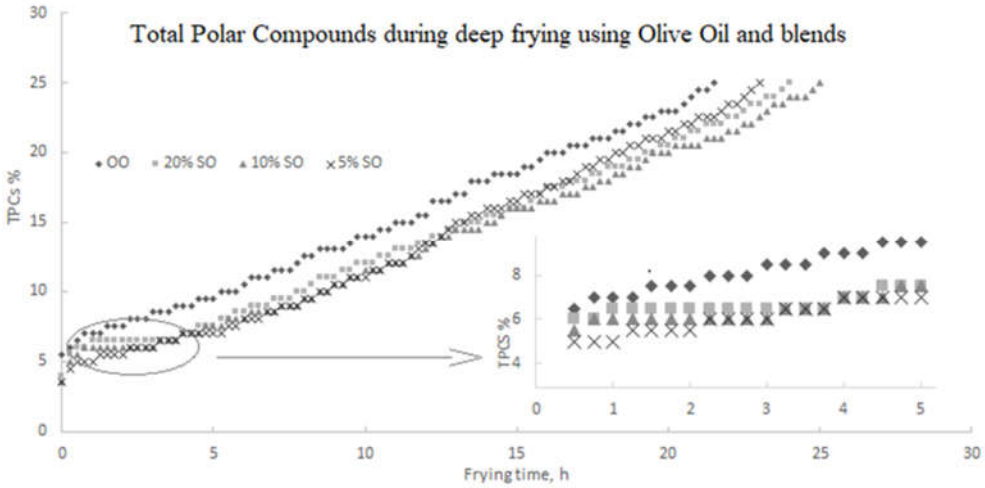

Figure 1. Changes in Total Polar Compounds during deep frying using Olive Oil and blends with Sesame Oil.

EVOO frying time was not affected by SO addition and was approximately the same (25-26 h) for all blends. This can be attributed to the higher antioxidant potential of EVVO, indicating the higher nutritional value and health benefits of EVOO in comparison to OO. TPC formation rate for EVOO did not decrease upon addition of SO, it was always 1% per hour. The time window where the TPCs were constant, was also detected in EVOO for 10 and 20 but not for 5% SO addition. After the time window, TPCs increased at the steady rate of 1% per hour (Figure 2).

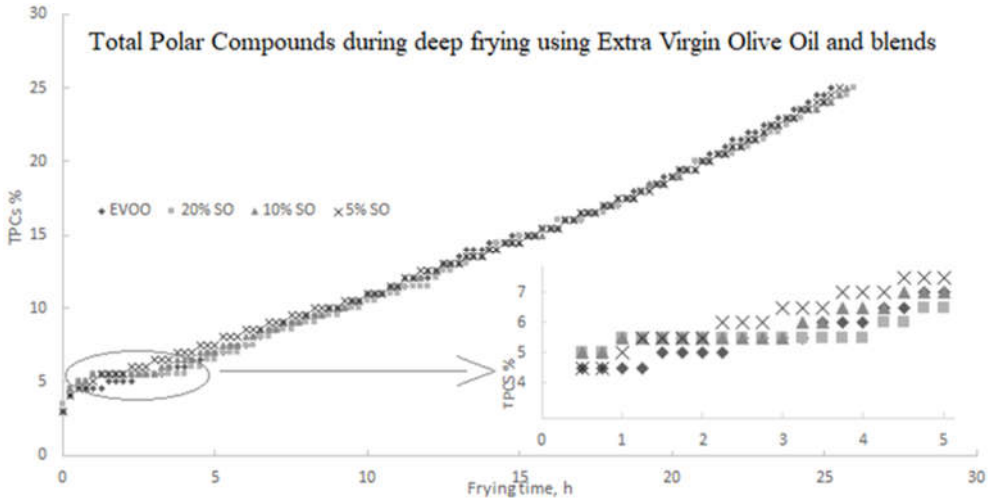

Figure 2. Changes in Total Polar Compounds during deep frying using Extra Virgin Olive Oil and blends with Sesame Oil.

3.2. Lignans.

Large amounts of sesamol are produced from sesamolin during frying contributing to frying oil stability [16,30]. Chatzos and Georgiou [26] reported that radical scavenging activity increases upon virgin sesame oil heating in contrast to all other seed oils in their study. This paradox is attributed to the fact that sesamol has higher antioxidant activity

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than its precursor, sesamolin [17]. We have followed the decomposition of sesamolin to sesamol during domestic deep-frying of potatoes in admixtures with olive oil through reverse phase HPLC. Initially, only sesamine and sesa-molin were detected (Figure 3). After 1h of frying sesamol was detected and sesamolin decreased by 18%. After 2 h of deep frying both sesamol and sesamolin decreased by 5% and 38%, respectively. After 4 hours sesamol and sesamolin decreased more by 60% and 70%, respectively. Similar study on sesame oil by Hemalatha and Ghafoorunissa, is in accordance to our study reporting maximum concertation of sesamol after 1 h followed by gradual decrease for one more hour [19].

As said in Section 3.1, after one hour of frying increase of TPCs stops at the same time when sesamol concentration peaks. Then after four hours, when sesamol is depleted, TPCs start increasing while all blends exhibit similar behavior. This is the beneficial role of sesame oil addition. The window size is related to the depletion of sesamolin: Higher concentrations result in a longer window of constant TPCs. This find-ing comes in agreement with previous research where antioxidant capacity of sesamol may be influenced by the concentration [32].

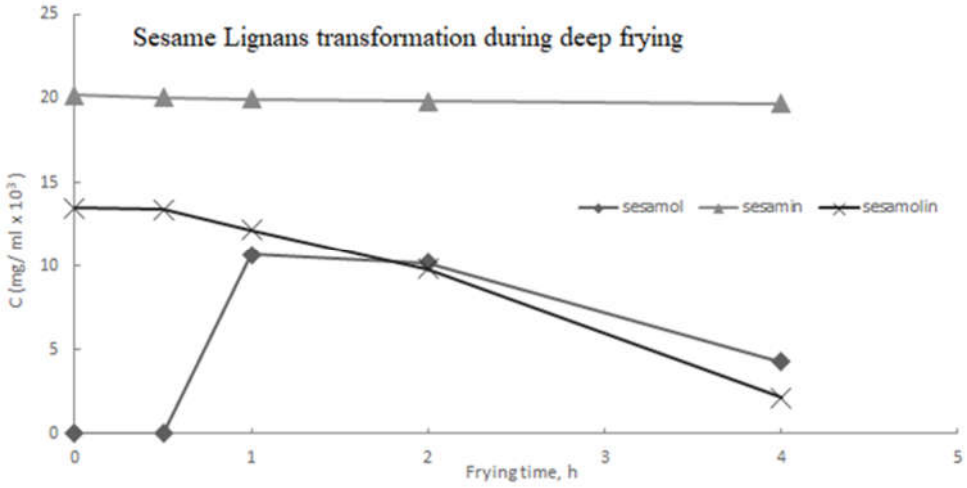

Figure 3. Sesame Lignans transformation during deep frying using 20% v/v Sesame Oil in Olive Oil.

3.3. Changes in Peroxide Value.

Deep frying induces oxidation. Primary oxidation products are peroxides that are unstable and decompose to carbonyl and aldehydic compounds in the presence of heat, air and light [33].

Peroxide values for OO and blends with SO are shown in Table 1. Peroxide formation rate was reduced by the addition of sesame oil from 54.4% to 22.7%, 31.7% and 35.9% for the 5%, 10% and 20% v/v blend, respectively. At 12 h, peroxides reach the maximum to decline afterwards. This decrease is similar for all blends.

Peroxide values for EVOO and blends with SO are shown in Table 2. Peroxide formation in EVOO during the first 12h was also reduced by the addition of sesame oil from 74.83% to 39.7%, 49,7% and 59,1% for the 5%, 10% and 20% v/v blend, respectively. At 12 h, peroxides reach the maximum to decline afterwards. This decrease is similar for all blends. EVOO rates were higher than OO but PV values were lower in EVOO and all blends with SO.

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Table 1. Olive Oil and blends with Sesame Oil, changes in PV, FFA, extinction coefficients and TEAC value.

Frying time (h)	PV	FFA	K_{232}	K_{270}	TEAC (mmol/Kg)
OO					
0	10.76	0.71	1.55	0,20	0.911
12	16.61	1.44	3.91	1.77	0.148
21.5	14.01	1.91	3.99	1.86	0.257
5% SO					
0	11.46	0.69	1.19	0.21	1.100
12	14.07	1.52	3.91	1.08	0.019
23	12.85	2.34	3.90	1.21	0.598
10% SO					
0	10.29	0.58	1.43	0.26	1.290
12	13.55	1.47	2.24	1.29	0.099
25	11.58	2.81	4.15	1.74	0.534
20% SO					
0	10.11	0.41	2.09	0.36	1.367
12	13.74	1.59	2.16	1.61	0.183
24	11.82	3.03	4.13	2.27	0.915

OO: olive oil, SO: sesame oil

3.4. Free Fatty Acids.

Hydrolysis in fats and oils results in the formation of free fatty acids, mono- and di-glycerides and glycerol inducing oxidative degradation contributing to shelflife reduction [2]. Free fatty acid content increases during frying [31].

The FFA content in OO sample was 0.71%, that is within the legal accepted limit of 1%. After 12h of frying, the FFA content in OO and the three blends increased to 1.44, 1.52, 1.47 and 1.59 respectively as shown in Table 1. Total frying time was 21.5, 23, 25, 24 h for OO and the three blends. The end FFA values were 1.9, 2.3, 2.8 and 3.0 % respectively. Although frying time increased by SO addition it was accompanied by higher FFA content that could have a negative organoleptic impact or even surpass the maximum value set in specific countries (2.5%) [22].

The FFA content in EVOO was 0.67 %, that is within the legal accepted limit of 0.8%, established by Commission Delegated Regulation (EU 2015/1830). After 12h of deep frying, the FFA content in EVOO and the three blends increased to 1.29, 1.30, 1.45 and 1.41%, respectively, as shown in Table 2. Total frying time was 25, 25.25, 25.5, 26 h for EVOO and the three blends. The end FFA values were 2.2, 1.8, 2.4, 2.4 %, respec-tively. Frying time was accompanied by higher FFA content without exceeding the maximum value set in specific countries (2.5%) [22].

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Table 2. Extra Virgin Olive Oil and blends with Sesame Oil, Changes in PV, FFA, extinction coefficients and TEAC value.

Frying time (h)	PV	FFA	K_{232}	K_{270}	TEAC (mmol/Kg)
EVOO					
0	7.64	0.67	1.22	0.13	1.905
12	13.357	1.29	3.63	0.89	0.323
25	11.85	2.21	2.94	0.79	0.494
5% SO					
0h	8.8	0.67	2.13	0.25	1.631
12h	12.29	1.30	3.39	1.21	0.343
25.25 h	11.21	1.79	3.47	1.27	0.443
10% SO					
0h	7.54	0.63	2.06	0.19	2.098
12h	11.29	1.45	3.11	1.31	0.457
25.5 h	9.87	2.44	2.35	1.41	0.674
20% SO					
0h	7.83	0.63	2.03	0.36	2.025
12h	12.46	1.41	2.77	1.55	0.558
26h	10.35	2.42	4.06	1.92	0.939

EVOO: olive oil, SO: sesame oil

3.5. Extinction coefficients, K_{232} and K_{270}.

When polyunsaturated fatty acids are oxidized ultraviolet absorption increases. The changes in ultraviolet absorption at 232 nm are associated with the formation of conjugated dienes of poly unsaturated fatty acids, while changes at 270 nm are associated with the formation of conjugated trienes, and carbonyl compounds. OO and EVOOs values of K_{232} and K_{270} are shown in Tables 1 and 2, respectively. The values for OO, EVOO and their blends with SO increase during frying. The increase was not related to the amount of SO. Moreover, no difference between OO and EVOO was recorded.

3.6. Changes in Antioxidant Capacity.

Lipid oxidation is a free radical reaction that is strongly modulated by synthetic and natural antioxidant compounds. During deep frying antioxidant compounds are consumed resulting in lower score in antioxidant capacity tests. Antioxidant capacity is measured by the radical scavenging ability while reacting with a relatively stable radical such as 2,2-diphenyl-1-picrylhydrazyl (DPPH) [26,34].

In our study, total antioxidant capacity was expressed as trolox equivalent antioxidant capacity (TEAC), defined as the mmol Trolox/kg of oil. OO blends with SO initially have higher TEAC value than OO, that is proportional to the percentage of SO in the blend (Table 1). After 12h of deep frying, all oils reach extremely low TEAC values. This comes in accordance with Kalantzakis et al. (2006), where olive oil samples practically lost their radical scavenging activity after 5 h of heating at 180 oC [35]. Although, oil samples are not expected to present any radical scavenging activity at the end of the frying experiment, the TEAC values are elevated for all samples (Figure S1a). This is an artifact explained by the reaction of DPPH with aldehydic compounds that are end products of lipid oxidation and are produced in high concentrations dur-ing the late stage of deep frying [36].

The antioxidant capacity of EVOO is not increased that much by the addition of SO as with OO (Tables 2 and 1, zero frying time). The antioxidant capacity of EVOO after 12 h of deep frying decreased by 84%. This improves to 72% upon 20% SO addition (Table 2). In a similar way as said for OO above, the TEAC values increase after 25 h of deep frying (Figure S1b).

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3.7. Changes in total phenols.

Phenolics are the major health promoting compounds in olive oil and the Mediterranean diet [37]. It is therefore important to follow the evolution of phenolics during deep frying to assess potential health benefits using olive oil.

The initial phenolic content of OO was 55.96 mg/Kg. Total phenols suffer a significant decrease for OO and all blends with SO after 12 h deep frying, going almost to zero (Figure S2).

The initial amount of phenolics in EVOO was 127.0 mg/Kg. After 12 h of deep frying EVOO and all the blends with SO retained 30% of their phenolic content that was zero after 25 h (Figure S3).

5. Conclusion

Both olive oil and extra virgin olive oil resist to oxidation under continuous domestic deep frying of potatoes, with extra virgin olive oil being more resistant. Addition of sesame oil to olive oil prolongs the frying cycles in proportion to the amount of sesame oil added for up to 4 hours. For extra virgin olive oil, that contains more natural antioxidant compounds, sesame oil is not beneficial. In summary, addition of sesame oil is useful in olive oil but not in extra virgin olive oil.

Peroxides that are created during deep frying could have detrimental effects on human health. Sesame oil addition decreases the amount of peroxides for both olive oil and extra virgin olive oil.

We report for the very first time that total polar compounds do not increase while sesame oils antioxidants are not depleted. This implies that further research should aim at the extension of this time window.

Olive oil is very much appreciated in the Mediterranean and Greek diets mostly because of the phenolic antioxidants. Sesame oil addition didn't have any effect on protecting olive oil phenolics during frying beyond the 20% sesame oil blend with extra virgin olive oil where 10% more phenolics were detected after 12 h. Our study showed that olive oil lost all phenolics after 12 h while extra virgin olive oil retained around 30%. This result could evolve after further research to a guideline on the use of extra virgin olive oil for deep frying so that the beneficial phenolics are not depleted. Such guideline could help branding of extra virgin olive oil as a health promoting frying oil. We envisage that deep frying with extra virgin olive oil should not continue till total polar compounds reach 25 % but stop much earlier to spare phenolic antioxidants.

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