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Enhancement of sunflower oil stability during deep-frying using extracts from olive oil by-products and soy lecithin

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Abstract

The potential application of olive mill wastewater (OMWW) and olive pomace (OP) extracts with lecithin (L) as antioxidants to enhance the stability of refined sunflower oil (SFO) during deep-frying (at 180°C) was investigated. Changes in SFO during deep-frying conditions were evaluated according to some physical and chemical parameters. *p*-anisidine value (*p*-AV), conjugated dienes (CD), tocopherols, total polar compounds, free fatty acids (FFA), color (*L**, *a**, *b**), viscosity and fatty acid composition were determined. According to the results, the addition of OMWW and OP extracts combined with lecithin inhibited lipid deterioration and retarded lipid oxidation. The oil containing extracts and/or lecithin showed a slight darkening as compared to the control. Profiles in the fatty acids were similar during deep-frying. The enrichment of SFO with OMWW and OP extracts is a beneficial application in the deep-frying oils and these extracts could be used with lecithin to retard lipid oxidation of oils during deep-frying.

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Introduction

Deep-frying is one of the most commonly used practices in the preparation and manufacture of foods around the world (Ramadan, 2015; Kim *et al.*, 2018). Fried foods are preferred worldwide due to their unique sensory traits, including texture, flavour and appearance. The demand for consumption of these foods increases due to the desirable sensory properties (Jaswir and Che Man, 1999; Dana and Saguy, 2006). Vegetable oils are used in both pan-frying and deep-frying process, wherein high temperature (generally between 170°C and 190°C) causes the loss of some oil's quality (Goburdhun and Jhurree, 1995). Despite many desirable features, some potentially toxic compounds could be induced in deep-frying of oils by chemical reactions such as hydrolysis, oxidation and polymerisation (Choe and Min, 2007).

Antioxidants are important substances that protect oils against lipid oxidation during deep-frying. Synthetic antioxidants such as butylated hydroxytoluene (BHT), butylated hydroxyanisole (BHA) and tertiary butylhydroquinone (TBHQ) are widely used in the oil industry. However, several studies emphasised that synthetic antioxidants could cause several health problems; therefore there is a growing interest to replace it with natural antioxidants (Wojcik *et al.*, 2010; Carocho and Ferreira, 2013). The effect of using natural antioxidants depends on its chemical structure. The most effective natural antioxidants contain high levels of phenolic compounds that have strong H-donating activity (Brewer, 2011). In recent years, the extraction of phenolics from food wastes or by-products and their use as natural antioxidants have been focussed (Moure *et al.*, 2001; Iqbal *et al.*, 2007; Sultana *et al.*, 2007; Yangui and Abderrabba, 2018; Di Nunzio *et al.*, 2018; Sousa *et al.*, 2019).

Olive mill wastewaters (OMWW) and olive pomace (OP) are the main by-products of olive (*Olea europaea*) oil (OO) processing.

These by-products are rich in phenolic compounds such as hydroxytyrosol and 3,4-dihydroxyphenyl acetic acid with strong antioxidant activity (Yangui and Abderrabba, 2018; Albahari *et al.*, 2018; Di Nunzio *et al.*, 2018; Sousa *et al.*, 2019). Hydroxytyrosol and 3,4-dihydroxyphenyl acetic acid were purified from OMWW and used as a natural antioxidant alternative

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to synthetic antioxidant in refined oils (Fki *et al.*, 2005). Besides, extracts from OMWW and/or OP were used in thermal oxidation experiments of some oils such as lard (De Leonardis *et al.*, 2007), extra virgin olive oil and refined olive kernel oil (Galanakis *et al.*, 2018), and refined sunflower oil (SFO) (Günal and Turan, 2018).

There have been few reports on deep-frying experiments using extracts from olive oil by-products as natural antioxidants in edible oils. Orozco-Solano *et al.* (2011) studied the variation in α-tocopherol and some individual phenolics (i.e., tyrosol, hydroxytyrosol, secoiridoid derivatives, vanillic acid, *p*-coumaric acid, hydroxytyrosol acetate, ferulic acid, luteolin and apigenin) in sunflower oil (SFO enriched with OP extracts during deep-frying. Ethanol extracts from olive waste cake were mixed with SFO at 100 - 600 ppm, and pan-frying experiment carried out at 180°C (Abd-ElGhany *et al.*, 2010). Phospholipids and in particular lecithin are used as an emulsifier in the food industry and have antioxidant activities. Lecithin exhibited a good synergistic effect with tocopherols and phenolics (Judde *et al.*, 2003, Ramadan, 2008; 2012).

The aim of the present work was to evaluate the implementation of methanol extracts from OMWW and OP with lecithin for the prevention of rancidity in SFO during deep-frying.

Materials and methods

Materials

OMWW and OP were obtained from a local commercial olive mill (Taylieli Laleli Olive and Olive Oil Plant, Balikesir, Turkey) and were stored at -18°C. The refined SFO was purchased from a local market (Balikesir, Turkey). All chemicals and reagents were of analytical grade. Lecithin (soy lecithin, type II-S, containing 14 - 23% phosphatidyl choline) was purchased from Sigma-Aldrich (St. Louis, MO, USA). *p*-anisidine, 2,2′-bipyridine (99%) and ferric chloride hexahydrate were purchased from Acros Organics (New Jersey, USA). Other chemicals and reagents were purchased from Merck (Darmstadt, Germany).

Preparation of extracts and SFO samples
Preparation of extracts

Extracts were prepared according to Günal and Turan (2018). Briefly, 100 g OMWW and 20 g OP were weighed in a flask. Next, 100 mL methanol was added. Flasks were shaken at 150 rpm using a shaking water bath for 60 min. After waiting overnight at 20 ± 2°C, the extracts were filtered through a filter paper. The residue was extracted with 100 mL solvent as described above, and the filtrates were pooled. In order to remove lipids that might be present in filtrates, each filtrate was stirred on a magnetic stirrer for 20 min after the addition of *n*-hexane. Methanol:water and hexane phases were separated with a separation funnel. Methanol:water phase was filtered through Whatman 1 filter paper and evaporated under vacuum using rotary evaporator at 40°C. Extracts were transferred into a colored bottle, and nitrogen gas was given for 20 min in order to remove the alcohol, and then dried using a freeze-dryer. Lyophilised extracts were stored at -18°C.

Phenolic profile of extracts

Phenolic profile of the extracts was determined according to Cioffi *et al.* (2010) with some modifications. Lyophilised extracts were dissolved in methanol and after filtration through nylon membrane filter (5 µm), 10 µL filtrate was injected into UFLC Shimadzu HPLC (Shimadzu, Japan) equipped with DAD detector. The chromatography column was COL-Analytical C_{18} column, (5 µm 250 × 4.6 mm, Perkin Elmer). The column temperature was set at 30°C and the flow rate of the mobile phase was 0.8 mL/min. The following gradient was used during analysis: [solvent A: acidified water (0.1%), solvent B: methanol]: 0 min, 100% A; 2 min, 95% A; 8 min 75% A; 10 min 60% A; 10 - 30 min 100% B, 30 - 35 min 40% B; and 35 - 45 min 5% B. The wavelength was set at 278 nm. The identification of tyrosol, hydroxytyrosol and oleuropein were carried out by injection of standards.

Preparation of SFO samples

Methanol extracts of OMWW and OP were added to SFO at concentrations of 1 mg/g after dissolving in propanediol. Lecithin (5 mg/g) was added into some samples. All samples were vortexed thoroughly and kept at 40°C for 20 min in an ultrasonic water bath to increase the amount of dissolved extract. SFO samples were used for deep-frying experiment.

Deep-frying experiment

A kitchen-type fryer with a volume of 1 L was used in the deep frying experiments. Frying experiments were carried out according to Che Man and Jaswir (2000) with some modifications. Nitrogen was given into 100 g of each SFO sample before the experiment, and all these samples was named zero and stored at -18°C. The rest of the SFO samples was heated at 180 ± 5°C for 10 min. Frozen French fries used in the present work were 0.9 × 0.9 × 6/8 cm in

dimension. Frozen French fries were weighed (100 ± 1 g), stored at -18°C and removed from the freezer 10 min before deep-frying. And they were fried for 3 min at 180 ± 5°C. Deep-frying experiments were done ten times a day for six days. At the end of the each day, the deep-frying oil was allowed to cool to 60°C, and 100 mL oil was transferred to a brown bottle after filtration and nitrogen gas was applied and stored at -18°C until analysis. Fresh oil was added daily instead of the sample taken and absorbed by the potatoes. The quantity of oil absorbed by each sample was completed because the fryer had 1 L capacity at least.

Collected samples were analysed to determine the free fatty acid-p0ojhgynlj (FFA) content, conjugated diene (CD) content, *p*-anisidine value (*p*-AV), changes in polar material content, tocopherol content, viscosity and colour changes. In addition, the fatty acid composition of oil samples taken at the beginning of deep-frying and at the end of sixth day were also determined.

Conjugated diene (CD) content was determined at 232 nm by using a spectrophotometer (Shimadzu, Japan) according to AOCS (1998) Ti 1a-64. *p*-anisidine value (*p*-AV) was determined at 350 nm by using a spectrophotometer (Shimadzu, Japan) according to AOCS (1998) Cd 18-90. Tocopherol analysis was measured spectrophotometrically according to Wong *et al.* (1988). For calibration, absorbance values of solutions containing α-tocopherol at different concentrations (25 - 200 µg/5 mL) were read under the same analysis conditions. The tocopherol content of samples was calculated as mg/kg (ppm). The free fatty acid (FFA) content was measured according to Foglia *et al.* (1993) as % oleic acid equivalent.

Miroil Optifry (Miroil Division of Oil Process Systems, Inc., ABD) were used to determine the changes in polar material content. This is a system that calculates the polar components due to the dielectric constant. Fresh refined SFO was used in the calibration of the device. The polar material content of SFO was assumed zero during the measurement and the change in the amount of polar material was determined during deep-frying.

The change in colour of SFO during deep-frying was determined by the CIE- *L**, *a**, *b** color system using the Hunter Lab Colorflex colorimetric device (Hunterlab, USA). The device was calibrated using white and black plates prior to use.

The viscosity was determined using an SV-10 Vibro Viscometer (Malvern Instruments Ltd., UK) at 30°C. Oil samples were stored at 30°C for 8 h before measurement. It was possible to keep the temperature constant during the analysis by passing the water at 30 ± 1°C through the inner wall of the cell.

The fatty acid composition of the oils was determined by gas chromatography (GC) as fatty acid methyl esters (FAMEs). FAMEs were prepared by using AOCS method Ce 2-66 (AOCS, 1997). The GC-MS operating parameters were: capillary chromatography column, TR-CN 100 (Teknokroma, Spain) 60 m × 0.25 mm × 0.2 µm; carrier gas, Nitrogen; column temperature, 90°C held for 5 min, heated up to 240°C at 4°C/min, then held for 10 min; split injection volume, 1 µL; split ratio, 1:50; and injection temperature, 250°C. Fatty acids were identified by comparing retention times with standard methyl ester mix (37 FAMEs mix, Sigma-Aldrich, St Louis, USA). The amounts of fatty acids were expressed as percentages using peak areas. C18:2/C16:0 and C18:1/C16:0 ratios of samples were calculated using fatty acid composition results.

Statistical analysis

The statistical analysis was performed with the SPSS package software, version 18.0 (SPSS Inc., Chicago, IL). Results were presented as means ± standard deviation of the two replicates of each experiment. Analysis of variance (ANOVA) was performed. Significant differences among the means ($p < 0.05$) were determined by Duncan's multiple tests.

Table 1. Main phenolic compounds of OMWW and OP extracts (mg/kg)

Extract	Hydroxytyrozol	Tyrozol	Oleorupein
Wastewater	2,772.8 ± 118.0	3,465.8 ± 10.6	1,094.4 ± 36.5
Pomace	1,287.6 ± 7.6	2,798.1 ± 528.4	537.0 ± 182.2

Results and discussion

Phenolic profile of OMWW and OP extracts

The main phenolics detected in OMWW and OP extracts are presented in Table 1. It was found that OMWW extract had higher phenolics content as compared to OP extract. Tyrosol was the main phenolic compound in extracts. OMWW extract contained 3,465.8 mg/kg tyrosol, while OP extract contained 2,798.1 mg/kg. Hydroxytyrosol and oleuropein were detected in levels lower than tyrosol. El-Abbassi *et al.* (2012) reported that hydroxytyrosol was the most abundant phenolic compound in OMWW extract. They reported the hydroxytyrosol content of two OMWW extracts as 3.766 and 2.127 g/L, wherein tyrosol contents were reported to be 2.491 and 0.246 g/L. DeJong and Lanari (2009)

Table 2. Conjugated diene content, p-anisidine value and tocopherol content of SFO samples during frying.

Day	SFO	SFO + L	SFO + OMWW	SFO + OMWW + L	SFO + OP	SFO + OP + L
Conjugated diene content (%)						
Zero	0.23 ± 0.01d^{AB}	0.23 ± 0.00^{eAB}	0.20 ± 0.02^{dB}	0.23 ± 0.01^{gAB}	0.23 ± 0.01^{gAB}	0.24 ± 0.00^{eA}
1	0.96 ± 0.02^{cA}	0.81 ± 0.00^{dBC}	0.93 ± 0.04^{cA}	0.65 ± 0.00^{fD}	0.86 ± 0.03^{fB}	0.75 ± 0.04^{dC}
2	1.64 ± 0.30^{bA}	1.48 ± 0.32^{cAB}	1.47 ± 0.16b^{AB}	1.05 ± 0.03^{eB}	1.30 ± 0.02^{eAB}	1.34 ± 0.03^{cAB}
3	1.73 ± 0.02^{bA}	1.60 ± 0.03^{bcAB}	1.55 ± 0.13^{bB}	1.37 ± 0.04^{dC}	1.54 ± 0.03^{dBC}	1.52 ± 0.06^{bBC}
4	2.11 ± 0.02^{aA}	1.64 ± 0.01^{bcC}	2.04 ± 0.01^{aB}	1.61 ± 0.02^{cCD}	2.00 ± 0.01^{cB}	1.58 ± 0.05^{bD}
5	2.16 ± 0.07^{aA}	1.85 ± 0.01^{abB}	2.10 ± 0.03^{aA}	1.82 ± 0.07^{bB}	2.07 ± 0.00^{bA}	1.83 ± 0.04^{aB}
6	2.32 ± 0.15^{aA}	1.99 ± 0.14^{aBC}	2.23 ± 0.00^{aA}	1.92 ± 0.08^{aC}	2.21 ± 0.01^{aAB}	1.92 ± 0.08^{aC}
p-anisidine value (mmol/kg)						
Zero	6.67 ± 0.07^{fC}	6.30 ± 0.07^{fD}	6.31 ± 0.01^{gD}	8.68 ± 0.07^{fA}	6.72 ± 0.00^{gC}	7.52 ± 0.04^{fB}
1	95.64 ± 0.89^{eA}	80.87 ± 2.34^{eC}	91.45 ± 0.18^{fB}	72.88 ± 2.04^{eE}	76.99 ± 0.02^{fD}	84.07 ± 0.61^{eC}
2	135.51 ± 1.34^{dB}	116.41 ± 0.03^{dF}	132.87 ± 0.13^{eC}	120.39 ± 0.98^{dE}	122.38 ± 0.27^{eD}	139.92 ± 0.83^{cA}
3	147.28 ± 1.23^{cA}	142.72 ± 1.51^{cB}	150.07 ± 1.01^{dA}	140.80 ± 0.88^{cB}	147.31 ± 0.55^{dA}	142.86 ± 1.83^{cB}
4	157.59 ± 1.15^{bB}	145.23 ± 0.46^{cC}	167.96 ± 0.42^{bA}	143.61 ± 1.42^{cC}	159.92 ± 1.43^{cB}	128.79 ± 0.41^{dD}
5	170.84 ± 2.40^{aB}	152.77 ± 2.57^{bE}	195.56 ± 0.03^{aA}	161.03 ± 1.70^{bCD}	165.35 ± 0.73^{bBC}	159.08 ± 4.26^{bD}
6	173.06 ± 1.09^{aB}	157.44 ± 2.64^{aD}	183.69 ± 1.65^{bA}	174.45 ± 2.43^{aB}	180.98 ± 1.03^{aA}	167.88 ± 1.02^{aC}
Tocopherol content (ppm)						
Zero	558.05 ± 3.2^{aD}	533.94 ± 0.51^{aE}	582.81 ± 5.53^{aBC}	594.96 ± 0.00^{aA}	578.70 ± 4.02^{bC}	588.18 ± 0.00^{aAB}
1	388.11 ± 5.29^{bD}	466.25 ± 0.84^{bB}	388.07 ± 8.42^{bD}	486.38 ± 5.02^{bA}	415.10 ± 0.49^{bC}	457.46 ± 1.84^{bB}
2	302.35 ± 2.89^{cD}	359.41 ± 7.46^{cB}	305.90 ± 2.09^{cD}	389.64 ± 4.43^{cA}	328.25 ± 2.59^{cC}	366.58 ± 3.11^{cB}
3	216.18 ± 1.36^{dE}	269.97 ± 3.84^{dC}	217.41 ± 5.57^{dE}	314.83 ± 0.88^{dB}	236.82 ± 0.33^{dD}	331.09 ± 1.32^{dA}
4	146.26 ± 1.16^{fD}	212.81 ± 1.79^{eB}	139.19 ± 0.03^{eE}	240.96 ± 3.31^{eA}	165.46 ± 3.19^{eC}	242.65 ± 3.13^{eA}
5	120.76 ± 2.77^{gD}	188.64 ± 4.35^{fB}	141.54 ± 7.56^{eC}	208.08 ± 1.54^{fA}	128.60 ± 2.14f^{CD}	214.16 ± 11.09^{fA}
6	153.39 ± 0.54^{eD}	178.16 ± 4.58^{gB}	133.48 ± 0.09^{eE}	149.60 ± 0.16^{gD}	160.58 ± 0.11^{eC}	203.62 ± 0.21^{fA}

Analyses were done in duplicate and results are given as mean ± standard deviation. SFO, Sunflower oil; L, Lecithin, OMWW, Wastewater methanol extract; OP, Pomace methanol ; OP-M, Pomace methanol extract. Small letter superscripts show the variation between days ($p < 0.05$). Capital letter superscripts show the variation between SFO samples on the same day ($p < 0.05$).

reported wastewaters of olive oil pomace contained hydroxytyrosol (70.6%), tyrosol (17.5%), caffeic acid (9.5%), p-coumaric acid (1.9%) and vanillic acid (0.3%).

Conjugated diene content, p-anisidine value and tocopherol content of oils during deep-frying

The results for CD, *p*-AV and tocopherol content are shown in Table 2. In the deep-frying experiment, the CD value in SFO rapidly increased from 0.23% to 2.32%. However, oil containing extracts or/and lecithin had significantly lower CD values than those without additives ($p \leq 0.05$). Furthermore, the best antioxidative effect was obtained when the combination of extracts from OO by-products and lecithin (SFO + OMW + L and SFO + OP + L) was applied, for which the values of CD were lower at the end of deep-frying.

p-AVs of oils enriched with only OMWW and OP extracts were higher than the control sample. Besides, samples mixed with lecithin and a combination of extracts with lecithin showed similarity and lower values for *p*-AV as compared to control sample.

With regards to tocopherol, lecithin addition to extracts decreased the loss of tocopherols in SFO samples during deep-frying. The major decrease for tocopherols was observed in oil enriched with OMWW. However, OP extract or/and lecithin exhibited a protective effect for tocopherols in SFO. These results of OP extract showed similarity with Orozco-Solano *et al.* (2011), who reported that α-tocopherol content of sample decreased by 50% at the end of eighth and 80% at the end of the sixteenth heating period.

Free fatty acid content, polar material content and viscosity of oils during deep-frying

Table 3 shows the FFA content, polar material content and viscosity of oil samples during 6-day deep-frying. The FFA content of all treatments gradually increased from day zero to day six of deep-frying. The FFA content of the control sample increased from 0.35% to 0.87% during deep-frying. Results showed that only extracts of OMWW and OP significantly ($p < 0.05$) reduced the FFA contents of oil samples during deep-frying. Results are supported

Table 3. FFA content, polar material content and viscosity of SFO samples during frying.

Day	SFO	SFO + L	SFO + OMWW	SFO + OMWW + L	SFO + OP	SFO + OP + L
Free fatty acid (% oleic acid)						
Zero	0.35 ± 0.05^{eA}	0.33 ± 0.08^{eA}	0.30 ± 0.04^{cA}	0.36 ± 0.04^{dA}	0.28 ± 0.00^{dA}	0.37 ± 0.04^{cA}
1	0.43 ± 0.00^{eCD}	0.63 ± 0.04^{dA}	0.34 ± 0.00^{cD}	0.47 ± 0.04^{dBC}	0.34 ± 0.00^{dD}	0.59 ± 0.12^{bAB}
2	0.44 ± 0.00^{deD}	0.78 ± 0.00^{cA}	0.34 ± 0.00^{cE}	0.53 ± 0.04^{cdC}	0.41 ± 0.04^{dD}	0.64 ± 0.04^{bB}
3	0.53 ± 0.05^{cdCD}	0.84 ± 0.08^{bA}	0.42 ± 0.04^{cD}	0.61 ± 0.08^{bcBC}	0.50 ± 0.01^{bcCD}	0.70 ± 0.04^{bB}
4	0.59 ± 0.04^{cB}	0.92 ± 0.03^{abA}	0.56 ± 0.08^{bB}	0.62 ± 0.04^{bcB}	0.59 ± 0.12^{bB}	0.70 ± 0.04^{bB}
5	0.75 ± 0.04^{bB}	1.01 ± 0.08^{aA}	0.61 ± 0.08^{bC}	0.67 ± 0.01^{bBC}	0.65 ± 0.04^{BC}	0.89 ± 0.01^{aA}
6	0.87 ± 0.04^{aB}	1.07 ± 0.00^{aA}	0.70 ± 0.03^{aC}	0.95 ± 0.00^{aAB}	0.73 ± 0.08^{aC}	0.97 ± 0.05^{aAB}
Changes in polar material content (%)						
Zero	0.00 ± 0.00^{gE}	2.18 ± 0.15^{gB}	0.90 ± 0.00^{gD}	3.78 ± 0.45^{gA}	1.65 ± 0.30^{gC}	2.48 ± 0.15^{gB}
1	9.03 ± 0.34^{fC}	10.28 ± 0.29^{fA}	9.53 ± 0.15^{fB}	9.75 ± 0.17^{fB}	7.33 ± 0.15^{fD}	9.45 ± 0.17^{fB}
2	13.68 ± 0.75^{eC}	16.10 ± 0.00^{eA}	13.53 ± 0.15^{eC}	13.60 ± 0.00^{eC}	11.18 ± 0.15^{eD}	15.50 ± 0.00^{eB}
3	18.95 ± 0.71^{dB}	21.68 ± 0.29^{dA}	17.08 ± 0.15^{dD}	16.10 ± 0.00^{dE}	17.75 ± 0.17^{dC}	21.50 ± 0.23^{dA}
4	26.68 ± 0.29^{cA}	26.60 ± 0.24^{cA}	24.40 ± 0.00^{cC}	20.33 ± 0.15^{cD}	25.18 ± 0.15^{cB}	25.18 ± 0.15^{cB}
5	32.43 ± 0.51^{bC}	36.13 ± 0.15^{bA}	30.95 ± 0.40^{bD}	29.03 ± 0.15^{bE}	32.68 ± 0.25^{bC}	33.80 ± 0.20^{bB}
6	42.55 ± 0.17^{aA}	39.75 ± 0.17^{aC}	40.88 ± 0.90^{aB}	34.70 ± 0.00^{aE}	42.25 ± 0.17^{aA}	38.40 ± 0.00^{aD}
Viscosity (mPa.s)						
Zero	42.62 ± 0.11^{gD}	44.23 ± 0.12^{gA}	43.67 ± 0.10^{gC}	44.00 ± 0.08^{gB}	44.01 ± 0.08^{gB}	42.50 ± 0.04^{gD}
1	52.43 ± 0.09^{fA}	51.77 ± 0.03^{fC}	51.06 ± 0.09^{fD}	47.35 ± 0.09^{fF}	50.31 ± 0.06^{fE}	52.28 ± 0.13^{fB}
2	57.09 ± 0.12^{eC}	63.16 ± 0.09^{eA}	61.09 ± 0.11^{eB}	55.19 ± 0.12^{eF}	55.83 ± 0.23^{eE}	56.77 ± 0.08^{eD}
3	66.26 ± 0.10^{dB}	68.46 ± 0.09^{dA}	64.25 ± 0.06^{dD}	62.46 ± 0.08^{dE}	65.92 ± 0.12^{dC}	66.05 ± 0.11^{dC}
4	73.79 ± 0.13^{cB}	76.85 ± 0.03^{cA}	72.76 ± 0.10^{cC}	66.27 ± 0.07^{cF}	71.60 ± 0.13^{cE}	71.87 ± 0.12^{cD}
5	91.94 ± 0.07^{bA}	87.91 ± 0.10^{bB}	79.54 ± 0.12^{bE}	80.20 ± 0.08^{bD}	84.47 ± 0.02^{bC}	78.66 ± 0.16^{bF}
6	108.87 ± 0.16^{aB}	100.52 ± 0.16^{aE}	105.07 ± 0.25^{aB}	92.04 ± 0.12^{aF}	113.06 ± 0.19^{aA}	102.42 ± 0.21^{aC}

Analyses were done in duplicate and results are given as mean ± standard deviation. SFO, Sunflower oil; L, Lecithin, OMWW, Wastewater methanol extract; OP, Pomace methanol extract. Small letter superscripts show the variation between days ($p < 0.05$). Capital letter superscripts show the variation between SFO samples on the same day ($p < 0.05$).

by findings of Abd-ElGhany *et al.* (2010) who reported the antioxidant effect of extracts from the olive waste in decreasing FFA of SFO during heating at 180°C. However, the FFA in the lecithin enriched treatments increased up to 0.95% (SFO + OMWW + L) and 0.97 (SFO + OP + L). Similar results were mentioned by Koprivnjak *et al.* (2008) who reported that the addition of lecithin slightly increased the FFA content of virgin olive oil.

There was a significant ($p < 0.05$) effect on the polar material content of SFO enriched with the extracts with lecithin. After six days of deep-frying, the polar material content for control increased up to 42.55%, while for OMWW and OP extract with lecithin treatments, the polar material content increased up to 34.70% and 38.40%, respectively. The other treatments showed almost similarity for polar material content as compared to control. The similar results were observed by Abd-ElGhany *et al.* (2010), who reported that olive waste extracts inhibited the increase in the polar material content of SFO during heating at 180°C. During deep-frying, the protective effect of extracts or/and lecithin in polar material content could be originated from tocopherols and phenolic compounds (Andrikopoulos *et al.*, 2002).

At elevated temperatures in a fryer, the levels of polymers increased which caused an increase in oil viscosity (Gertz, 2000). The initial value of viscosity in the control sample was 42.62 mPa.s, while at the end of the deep-frying period (sixth day), the value increased up to 108.87 mPa.s. The deep-frying process changed the viscosity of the deep-frying oil samples and this result is in agreement with that reported by Santos *et al.* (2005), who observed an increase in viscosity of some vegetable oils during deep-frying probably due to the formation of undesirable compounds occurred by oxidation and further polymerisation reactions. At day 6, the lowest viscosity values observed in oils enriched with extracts and lecithin, as well as in oils enriched only with lecithin compared to control sample.

Table 4. L*, a*, b* values of SFO samples during frying.

Days	SFO	SFO + L	SFO + OMWW	SFO + OMWW + L	SFO + OP	SFO + OP + L
*L**						
Zero	65.31 ± 0.00^{aB}	65.54 ± 0.00aA	64.34 ± 0.01^{aC}	60.24 ± 0.01^{aF}	64.20 ± 0.00^{aD}	61.92 ± 0.01^{aE}
1	62.97 ± 0.01^{bA}	27.16 ± 0.06^{gD}	61.89 ± 0.04^{bC}	22.76 ± 0.00^{gF}	62.01 ± 0.01^{bB}	25.21 ± 0.21^{gE}
2	59.95 ± 0.01^{cA}	29.17 ± 0.02^{fE}	58.81 ± 0.01^{cB}	23.37 ± 0.08^{fF}	56.96 ± 0.02^{cC}	30.22 ± 0.03^{fD}
3	55.40 ± 0.01^{dA}	31.80 ± 0.05^{dE}	52.74 ± 0.03^{dB}	26.31 ± 0.13^{eF}	52.54 ± 0.02^{dC}	34.74 ± 0.02^{eD}
4	50.16 ± 0.01^{eA}	30.43 ± 0.09^{eF}	47.83 ± 0.04^{eB}	34.83 ± 0.04^{cE}	46.54 ± 0.01^{eC}	36.42 ± 0.01^{dD}
5	46.45 ± 0.01^{fA}	32.48 ± 0.01^{cF}	43.43 ± 0.05^{fB}	41.19 ± 0.02^{bD}	42.65 ± 0.01^{fC}	37.59 ± 0.04^{cE}
6	42.99 ± 0.02^{gA}	37.94 ± 0.16^{bD}	41.01 ± 0.05^{gB}	34.44 ± 0.01^{cF}	40.99 ± 0.04^{gC}	37.67 ± 0.01^{bE}
*a**						
Zero	-3.59 ± 0.01^{C}	-3.94 ± 0.01^{dF}	-3.65 ± 0.01^{gD}	-1.54 ± 0.01^{gA}	-0.37 ± 0.01^{gE}	-2.44 ± 0.01^{gB}
1	-4.10 ± 0.03^{fF}	20.15 ± 0.05^{cA}	-3.44 ± 0.01^{fE}	18.68 ± 0.03^{fC}	-3.21 ± 0.02^{fD}	19.70 ± 0.02^{fB}
2	-1.90 ± 0.01^{eF}	22.62 ± 0.04^{bA}	0.10 ± 0.02^{eE}	21.65 ± 0.05^{dC}	1.93 ± 0.01^{eD}	22.32 ± 0.02^{eB}
3	5.29 ± 0.02^{dE}	23.85 ± 0.03^{bA}	8.69 ± 0.04^{dD}	23.15 ± 0.03^{bB}	9.50 ± 0.01^{dC}	23.13 ± 0.04^{dB}
4	13.99 ± 0.02^{cF}	26.59 ± 0.12^{aA}	16.65 ± 0.02^{cE}	22.96 ± 0.05^{cC}	18.38 ± 0.01^{cD}	23.96 ± 0.03^{cB}
5	19.13 ± 0.01^{bF}	26.34 ± 0.05^{aA}	22.09 ± 0.04^{bD}	21.43 ± 0.02^{eE}	23.56 ± 0.04^{bC}	24.65 ± 0.09^{bB}
6	24.11 ± 0.01^{aB}	27.30 ± 2.19^{aA}	26.10 ± 0.04^{aAB}	27.16 ± 0.00^{aA}	26.96 ± 0.08^{aA}	25.54 ± 0.03a^{AB}
*b**						
Zero	15.73 ± 0.02^{gF}	26.31 ± 0.01^{fC}	19.76 ± 0.03^{fE}	35.44 ± 0.05^{eA}	21.35 ± 0.01^{fD}	32.82 ± 0.00^{eB}
1	34.28 ± 0.06^{fC}	37.73 ± 0.45eA	34.28 ± 0.06^{eC}	29.78 ± 0.05^{gD}	35.24 ± 0.06^{eB}	35.41 ± 0.36^{dB}
2	45.06 ± 0.00^{eD}	42.84 ± 0.45^{dE}	50.28 ± 0.00^{dB}	33.69 ± 0.40^{fF}	52.73 ± 0.02^{dA}	46.09 ± 0.21^{cC}
3	61.02 ± 0.13^{dB}	49.08 ± 0.01^{bD}	64.67 ± 0.25^{bA}	38.54 ± 0.14^{dE}	64.42 ± 0.24^{bA}	51.79 ± 0.13^{bC}
4	66.55 ± 0.13^{cC}	46.58 ± 0.16^{cE}	67.70 ± 0.92^{aB}	50.85 ± 0.34^{bD}	70.34 ± 0.08^{aA}	51.83 ± 0.11^{bD}
5	68.13 ± 0.23^{aA}	42.76 ± 0.11^{dE}	65.04 ± 0.35^{bB}	57.95 ± 0.08^{aC}	64.91 ± 0.33^{bB}	53.32 ± 0.08^{aD}
6	67.62 ± 0.12^{bA}	57.48 ± 0.26^{aC}	59.55 ± 0.31^{cB}	49.33 ± 0.27^{cE}	59.51 ± 0.42^{cB}	51.71 ± 0.00^{bD}

Analyses were done in duplicate and results are given as mean ± standard deviation. SFO, Sunflower oil; L, Lecithin, OMWW, Wastewater methanol extract; OP, Pomace methanol extract. Small letter superscripts show the variation between days ($p < 0.05$). Capital letter superscripts show the variation between SFO samples on the same day ($p < 0.05$).

Colour

The changes in *L**, *a** and *b** colour values of oils are presented in Table 4. There was a decrease in the *L** (lightness-darkness) value of the oil, and an increase in the values of *a** (red-green) and *b** (yellow-blue) due to the induced coloured compounds as a result of oxidation, polymerisation and other chemical changes during frying (Maskan, 2003). When compared with control, *L** values showed lower values in oils mixed with extracts and/or lecithin at the end of deep-frying. A higher *L** values indicate a lighter colour, which is desirable in frying oils, so a decrease in *L** showed that the oil becomes darker (Troncoso *et al.*, 2009).

The *a** value indicates red-green color. This value in oils increased during frying. At the end of deep-frying, similar *a** values were observed in both control and SFO samples enriched with extracts and/or lecithin. The *b** values, which express the yellow-blue color, increased as the deep-frying period increased. This indicated that the intensity of the yellow color increased. SFO showed the highest increase for *b** value and increased up to 67.62, while SFO samples enriched with extracts and/or lecithin showed the lowest increase at the end of sixth day of deep-frying with the value below 60. Results also showed that the *b** values of SFO samples enriched with extracts and lecithin were lower than SFO samples enriched with extracts alone. Similar results were obtained by Abdulkarim *et al.* (2007) and Maskan and Horuz (2017). Darkening of oils (decrease in *L** value) could be caused be oxidised products and chemical reactions of these oxidised products with Maillard reaction products (Bansal *et al.*, 2010, Maskan and Horuz, 2017). Besides, higher concentrations of both red and yellow colours in oil samples could be related to the pigments present and colour induced from Maillard reactions (Gharachorloo *et al.*, 2010).

Fatty acid composition

The change in the fatty acid composition of SFO samples at initial and on the sixth days of deep-frying is shown in Table 5. The major fatty acid, linoleic acid, dramatically decreased, while other main fatty

Table 5. Fatty acid composition of SFO samples at the initial and on the sixth day.

	SFO		SFO + L		SFO + OMWW		SFO + OMWW + L		SFO + OP		SFO + OP + L	
Fatty Acid (%) / Days	Zero	6	Zero	6	Zero	6	Zero	6	Zero	6	Zero	6
Myristic acid (C14:0)	0.05	0.35	0.05	0.28	0.05	0.34	0.07	0.32	0.07	0.32	0.05	0.31
Palmitic acid (C16:0)	5.94	18.43	5.87	15.76	5.97	17.97	5.97	17.34	5.97	17.37	5.90	17.79
Palmitoleic acid (C16:1)	0.10	0.12	0.10	0.12	0.10	0.12	0.10	0.12	0.10	0.12	0.10	0.12
Heptadecanoic acid (C17:0)	0.03	0.05	0.03	0.04	0.03	0.05	0.04	0.07	0.04	0.07	0.03	0.04
cis-10-heptadecanoic acid (C17:1)	0.02	0.03	0.02	0.02	0.02	0.03	0.02	0.02	0.02	0.02	0.02	0.02
Stearic acid (C18:0)	2.71	3.70	2.65	3.41	2.70	3.63	2.66	3.50	2.66	3.51	2.65	3.54
Elaidic acid (C18:1 n9t)	0.00	0.02	0.00	0.01	0.00	0.02	0.00	0.01	0.00	0.01	0.00	0.02
Oleic acid (C18:1 n9c)	35.29	40.12	35.87	40.21	35.31	40.21	35.45	39.19	35.46	39.25	35.36	39.55
Linolelaidic acid (C18:2 n6t)	0.22	0.41	0.22	0.35	0.22	0.36	0.22	0.34	0.22	0.34	0.22	0.38
Linoleic acid (C18:2 n6c)	54.62	35.73	54.19	38.77	54.65	36.23	54.54	38.04	54.56	38.10	54.74	37.20
Arachidic acid (C20:0)	0.20	0.28	0.20	0.26	0.20	0.27	0.20	0.27	0.20	0.27	0.19	0.27
cis-11-eicosenoic acid (C20:1)	0.15	0.17	0.15	0.16	0.00	0.16	0.00	0.16	0.00	0.16	0.00	0.16
Linolenic acid (C18:3 n6c)	0.07	0.05	0.08	0.06	0.15	0.05	0.15	0.06	0.15	0.06	0.15	0.06
Behenic acid (C22:0)	0.58	0.52	0.55	0.51	0.57	0.51	0.54	0.50	0.54	0.50	0.56	0.50
Tricosanoic acid (C23:0)	0.03	0.03	0.02	0.02	0.03	0.02	0.02	0.02	0.02	0.02	0.02	0.02
Others	0.00	0.01	0.00	0.01	0.00	0.02	0.01	0.02	0.01	0.02	0.00	0.01
C18:1/C16:0	5.90	2.20	6.10	2.60	5.90	2.20	5.90	2.30	5.90	2.30	6.00	2.20
C18:2/C16:0	9.20	1.90	9.20	2.50	9.20	2.00	9.10	2.20	9.10	2.20	9.30	2.10
Total trans fatty acid	0.23	0.43	0.22	0.37	0.23	0.39	0.21	0.36	0.21	0.36	0.23	0.40

Analyses were done in duplicate and results are given as mean ± standard deviation. SFO, Sunflower oil; L, Lecithin, OMWW, Wastewater methanol extract; OP, Pomace methanol extract.

acids in SFO, oleic and palmitic acids increased during deep-frying. During deep-frying of SFO, a reduction in linoleic acid and an increase in oleic and palmitic acids were reported by Aydınkaptan and Mazı (2017). C18:1/C16:0 and C18:2/C16:0 ratios were used to determine deep-frying deterioration. During deep-frying, C18:1/C16:0 ratios decreased from 5.9 to 2.2 and the ratio C18:2/C16:0 decreased from 9.2 to 1.9 during the deep-frying process of the control sample. Moreover, trans fatty acid content increased from 0.23% to 0.43%. Similar declining trend for C18:1/C16:0 and C18:2/C16:0 ratios and increase in trans fatty acids were observed in the studies of Abdulkarim and Ghazali (2012) and Tohma and Turan (2015).

Conclusion

The present work demonstrated that OMWW and OP extracts from olive oil by-products, inhibited SFO deterioration during deep-frying. The addition of soy lecithin with these extracts showed better antioxidant activity than individual extracts. In terms of chemical and physical degradation parameters including CD, *p*-anisidine, total polar material content, FFA and colour, the enrichment of SFO samples with extracts and lecithin improved the oxidative stability of SFO during deep-frying, while slight activity was observed when the extracts were individually applied without the lecithin addition. As for fatty acids, slight differences were noted between control SFO and SFO samples enriched with extracts and/or lecithin. In view of these findings, it could be concluded that extracts with lecithin might be applied in the deep-frying of SFO and other vegetable oils as antioxidants.

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