



CAN ESSENTIAL OILS STABILIZE FRYING OIL?! INSIGHTS TO THE EFFECT OF ESSENTIAL OILS FROM *FERULAGO ANGULATA*, *MENTHA PULEGIUM*, AND *CUMINUM CYMINUM* ON FRYING OIL DURING DEEP-FRYING OF POTATO SLICES

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ABSTRACT

The effect of essential oils (EOs) from *Ferulago angulata* (F), *Mentha pulegium* (M), and *Cuminum cyminum* (C) was considered on oxidative stability of frying oil during frying of potato slices. The EOs were applied in concentrations of 200 and 400 ppm and a mixture sample (140 ppm of each essential oil). Tertiary butyl hydroquinone (TBHQ) was used as a standard at 100 ppm. The efficacy of EOs was evaluated by 1,1-diphenyl-2-picrylhydrazyl (DPPH) radical scavenging, and parameters of free fatty acid content (FFA), peroxide value (PV), p-anisidine value (P-AnV), total polar content (TPC), and sensory evaluation during three cycles frying. The scavenging activity of TBHQ was higher followed by M-400, F-400, M-200, C-400, C-200, Mixture, and F-200, respectively. The obtained results from chemical parameters were in agreement with each other and except acidity, the content of PV, P-AnV, and TPC almost in all samples containing EOs were higher than the control during cycles of frying. Sensory evaluation data also showed the superiority of synthetic antioxidant followed by control and EOs. According to the results, EOs had a weak antioxidant effect on frying oil due to their volatility and sensitivity to high temperatures during frying.

1. Introduction

Deep-fat frying is an old and popular process that produces desirable color, flavor and crispy texture in fried foods. It includes immersing of food in hot oil at temperatures of 150-190°C. Simultaneous mass and heat transfer of oil, air and food during this process causes the unique quality of fried materials. In addition to desirable properties, deep-fat frying poses undesirable characteristics including off flavor, rancid odor and discoloration due to

producing of volatile and nonvolatile components as a result of oxidation, hydrolysis and polymerization reactions that affect the sensory, functional and nutritional value of oils. Frying oil, time and temperature of frying, type of fryer and antioxidants affect these reactions (Choe and Min, 2007).

Today, due to increasing awareness of people and scientists from harms of synthetic additives including antioxidants, there was a

considerable tendency for substitution of these materials with natural ones.

Spices and herbs due to their flavor, perfume, and preservative properties have been used in different products and cosmetic and medical industries from ancient times (Bakkali *et al.*, 2008). Essential oils are a mixture of volatile organic components that obtain from non-woody parts of the plant by hydro-distillation and steam (Batish *et al.*, 2008). *Ferulago* is a member of the *Apiaceae* (*Umbelliferae*) family. It includes two subspecies: subsp. *Carduchorum* (Boiss and Hausskn) and subsp. *Angulata* (Schlecht). *F.angulata* (Schlecht) Boiss as title Chavir in Iran, grows in the east of Turkey, and north of Iran and Iraq. It has been used as an additive to animal oil for many years due to its preservative effect in Kermanshah province (Sadeghi *et al.*, 2016 a). Antioxidant and antimicrobial effect of this plant has been proved in several studies (Taran *et al.*, 2010; Sadeghi *et al.*, 2016 a; Sadeghi *et al.*, 2017). *Mentha* spices are in *Lamiaceae* family. *Mentha pulegium* L. (Pennyroyal) is one of these spices that are founded in damp and humid areas including north Africa, south Eroup and near east countries. In folk medicine, it is used as carminative, antispasmodic, diaphoretic, stimulant, sedative, antiseptic bronchitis, diuretic, and for skin diseases (Lawrence, 2007). The essential oil of this plant has antioxidant, antibacterial, antifungal and insecticidal activity (Teixeria *et al.*, 2012; Zekri *et al.*, 2013). *Cuminum cyminum* L. (Cumin) belongs to the *Apiaceae* family. It is cultivated in China, Iran, India, Japan, Morocco, Indonesia, South Russia, Turkey, and Algeria. Cumin seed is a popular spice used for the treatment of dyspepsia, epilepsy, diarrhea, jaundice, and toothache in folk medicine and as a flavoring agent. Antioxidant effect of cumin has proved in some studies (Bettaieb *et al.*, 2010).

Although, the mentioned essential oils have shown antioxidant effect in various studies, the effect of them in high temperatures such as frying has not considered. Therefore, in this

study, we considered the efficacy of Chavir, Cumin, and Pennyroyal essential oils on the stability of frying oil in the frying process of potato slices for a response to this question: if essential oils can stabilize frying oil?

2. Materials and methods

2.1. Materials

Frying oil without antioxidant including 50% palm-olein, 30% soybean and 20% sunflower oils were prepared from Agro-industry Complex & Vegetable Oil of Mahidasht, Kermanshah, Iran. *F.angulata* and *M.pulegium* were supplied from Dalahoo Mountains, Kermanshah, Iran, and Sari mountains, Mazandaran, Iran, respectively. *C.cyminum* seeds as dried were collected from the local market in Kerman province, Iran. The mentioned plants were identified by the Institute of Medicinal Plants, Tehran, Iran. 1,1-diphenyl-2-picrylhydrazyl (DPPH) was purchased from Sigma-Aldrich. Other chemical materials with analytical grade were prepared from Merck. The potato was supplied from the local supermarket.

2.2. Essential oils extraction and identification

Essential oils (EOs) were extracted by Clevenger apparatus and were analyzed by GC-MS in prior studies (Sadeghi *et al.*, 2016 a; Sadeghi *et al.*, 2016 b; Sadeghi *et al.*, 2013).

2.3. Sample preparation and frying process

Each of EOs was added to frying oil in concentrations of 200 and 400 ppm (F-200, F-400, M-200, M-400, C-200, and C-400 for *F.angulata*, *M.pulegume*, and *C.cyminum*, respectively). Also, a mixture of these EOs (140 ppm of each EOs) was used. TBHQ was applied as standard at 100 ppm and a control sample was prepared without antioxidant. Potatoes sliced in the dimension of 8 × 0.8 × 0.8cm. The slices washed with water and dehydrated by specific cloth before frying. Various treatments of EOs in frying oil in the volume of 1L were poured in the domestic fryer and after reaching of temperature to 180°C,

slices (200 gr) were added to oil and frying was carried out for 5 min in 3 batches with intervals of 1hr. Samples from frying oil and fried potatoes were taken after each time, cooled to room temperature and stored in -20°C for analysis.

2.4. Chemical parameters

Official methods of AOCS were used for measurement of peroxide value (cd 8-53), free fatty acid content (cd 3a-63), P-anisidine value (cd 18-90), and polar compounds (cd 20-91) (AOCS, 1990).

2.5. DPPH radical scavenging assay

Efficacy of EOs for scavenging of DPPH free radical was assayed according to Gyamfi *et al.* (1999). Thus, a methanol solution of EOs at mentioned concentrations was prepared and 50 µl of them was added to 5 ml DPPH methanol solution (0.004%). After vortex (15s), solutions were incubated at 25°C in dark for completion of reaction for 30 min. The absorbance of samples and control (DPPH methanolic solution) was read at 517 nm by UV-Visible spectrophotometer. Percent of the radical scavenging calculated as follow:

$$\% \text{ RSA} = 100 \times (A_c - A_s) / A_c \quad (1)$$

Where A_c and A_s are absorbance of the control and sample, respectively.

2.6. Sensory evaluation

Samples analyzed by 7 semi-trained panelists. Sensory evaluation was carried

according to 9 points hedonic scale for flavor, aroma, color and total acceptability of fried potatoes with score 9 for extraordinary well and 1 for unacceptable (Sadeghi *et al.*, 2017).

2.7. Statistical analysis

The data were analyzed by SAS software and Duncan's multiple range tests were applied for the assay of significant differences ($p < 0.05$).

3. Results and discussions

3.1. Essential oils extraction and identification

The main components of *F.angulata*, *M.pulegium*, and *C.cyminum* essential oils are shown in Table 1 adapted from previous works. The antioxidant materials in plants are including various mixtures of polyphenolics, tocopherols, ascorbic acid or terpenoids (Grabmann, 2005). They inhibit oxidation with different mechanisms such as autoxidative chain reaction breaking (primary antioxidants) and chelating of prooxidant metals, decomposition of hydroperoxides, donating of hydrogen to primary antioxidants, quenching of singlet oxygen, or absorption of ultraviolet ray (secondary antioxidants). Terpenoids specifically monoterpenes and sesquiterpenes are predominant compounds of essential oils (Akoh and Min, 2008). Antioxidant properties of the mentioned EOs are attributed to the identified ingredients in their essential oils that mostly are monoterpenes and sesquiterpenes.

Table 1. The main components of essential oils from *Ferulago angulata*, *Mentha pulegium*, and *Cuminum cyminum*

<i>F.angulata</i>	Percentage	<i>M. pulegium</i>	Percentage	<i>C. cyminum</i>	Percentage
Cis-Ocimene	30.17	Pulegone	36.68	Cuminaldehyde	29.02
α-Pinene	15.4	Piperitenone	16.88	α-Terpinene-7-al	20.70
Trans-β- Ocimene	5.7	1,8 Cineole	14.58	γ-Terpinene	12.94
γ-Terpinene	5.57	α- terpineol	5.98	γ-Terpinene-7-al	8.9
Germacrene-D	5.03	Menthone	4.72	ρ-Cymene	8.55
Limonene	4.88	Cis- Salvene	3.56	β-Pinene	7.72
Bornyl Acetate	4.57	Piperitenone oxide	3.27	Cis-Dihydrocarvone	4.45

Myrcene	3.62	δ -terpineol	3.19	Myrcene	1.1
Camphene	2.41	Endo borneol	3.04		
Noe-Allo-Ocimene	1.87	β -Caryophyllene	1.79		
β -Phellandrene	1.84	Caryophyllene oxide	1.57		
α -Terpinolene	1.7	Carvacrol	1.34		
Bicyclogermacrene	1.29	Limonene	1.26		
δ -Cadinene	1.18				

3.2. Chemical parameters

3.2.1. Free fatty acid content (FFA)

When food is heated in frying oil; oxygen, water, and steam initiate some reactions in food and oil. Water and steam cause hydrolysis of oil and production of mono and diacylglycerols, FFAs, and glycerol. Thus, the content of free fatty acids increases during frying (Chang *et al.*, 2004). FFAs were significantly increased after the first cycle of frying for all samples and gradually enhanced during the process (Figure 1). These outcomes were in accordance with other studies (Alizadeh *et al.*, 2016; Naz *et al.*, 2005; Sayyad, 2017). In end of 3 cycles frying, control sample had the highest FFA (0.16%) followed by Mixture (0.153%), F-200

(0.134%), M-400 (0.125%), M-200 (0.124%), C-400 (0.123%), C-200 (0.121%), F-400 (0.12%), and TBHQ (0.12%), respectively. According to obtained results, all of the antioxidant samples had lower acidity than control but there was a slight difference among them during cycles of frying (see supplementary, Table S1). Inanc and Maskan (2014) obtained an equivalent result for palm oil treated with Carvacrol during potato frying. Also, the concentration of essential oils had no effect on the decrease of FFAs. However, FFA content is not a very authoritative factor for evaluation of frying oil degradation, because it is hard to distinguish FFA produced by hydrolysis or oxidation (Ramadan *et al.*, 2006).

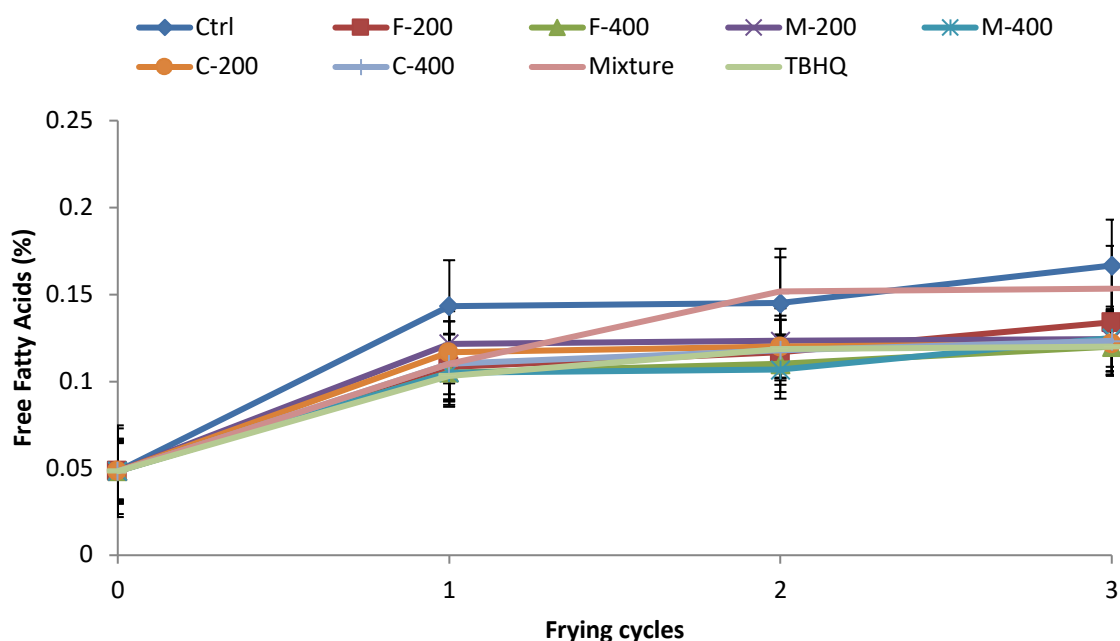


Figure 1. The effect of essential oils on free fatty acid content of frying oil during frying cycles

3.2.2. Peroxide value (PV)

Oil oxidation causes the formation of peroxides as primary products that are a good index of oil quality under normal condition. For control, PV increased with frying cycles (Figure 2). In samples of F-200 and F-400, PV during cycle 2 decreased and then increased at cycle 3. M-200, M-400, and C-200 showed enhancement in peroxide during frying. The content of peroxides of C-400, Mixture, and TBHQ rose to cycle 2 frying and then reduced at cycle 3. Acquired results showed all of the essential oils in a dose of 400 ppm had a lower PV than 200 ppm samples. DU and Li (2008) observed similar results with consideration antioxidant effect Cassia essential oil on deep frying of beef in soybean, peanut, rapeseed, sunflower, and palm oils. At the end of 3 cycles frying, M-400 had the lowest PV followed by

C-400, TBHQ, Control, Mixture, F-400, F-200, C-200, and M-200, respectively. This show EOs exceptionally M-400 and C-400 had PV higher than control that is not in accordance to Bensmira *et al.* (2007) study on the effect of Lavender and Thyme essential oil in sunflower oil. They declared peroxide value of lower in oil with antioxidant rather than untreated samples. Moreover, TBHQ and control samples had no significant difference at the end of the process. Totally, there were significant differences between antioxidants and frying cycles (Table S1). Abatement in PV at cycles 2 and 3 frying is related to the decomposition of peroxides because these products are unstable and readily break at frying temperatures. Therefore PV cannot be a very safe indicator for prediction of frying oil quality.

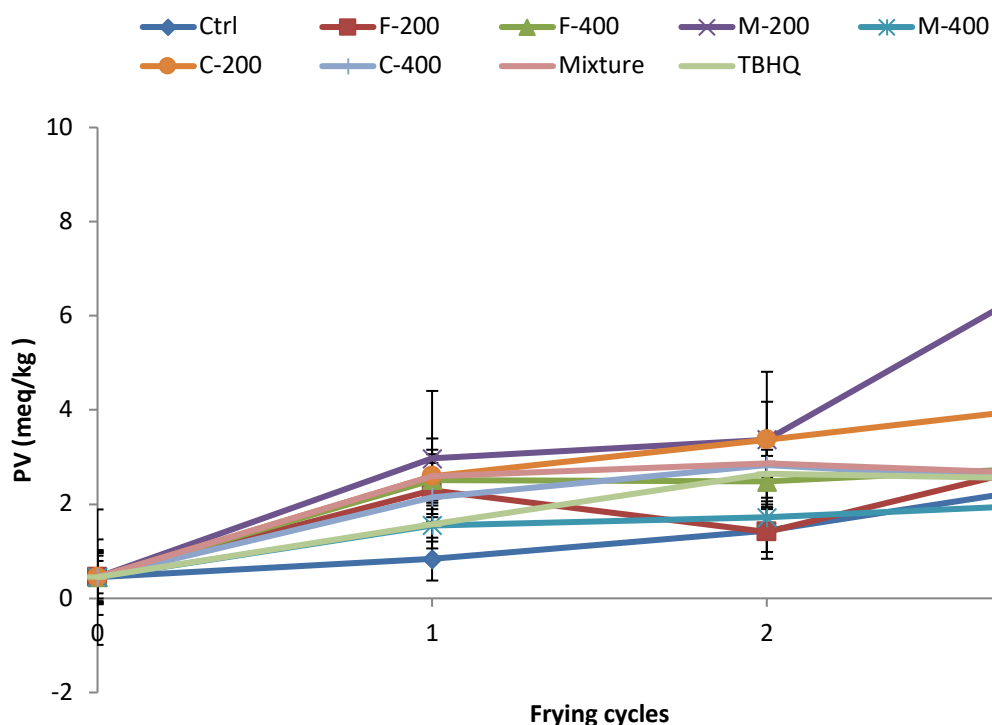


Figure 2. The effect of essential oils on peroxide value of frying oil during frying cycles

3.2.3. P-Anisidine value (P-AnV)

As previously mentioned; decomposition of peroxides at frying high temperatures leads to the formation of oxidation secondary products. P-AnV measures compounds such as α and β -

alkenals and other components that react with P-anisidine reagent (Guillen and Cabo, 2002). The P-AnV content of samples is presented in Figure 3. In all samples, anisidine value increased with frying cycles that are in

agreement to results of Alizadeh *et al.* (2016), Sayyad (2017), and Naz *et al.* (2008). Also, enhancement of AnV during the first frying cycle was severer than the next cycles. Researchers declared that degradation of carbonyl components and their absorption to fried food can reduce the increment rate of them during deep frying (Aladedunye and Przybylski, 2009). Moreover, AnV with increasing dose of EOs decreased that is in accordance with PV results. The lowest value was related to TBHQ (5.41) followed by M-400 (6.58), F-400 (6.62), Ctrl (7.21), F-200 (7.23), Mixture (7.5), M-200 (8.37), C-400 (9.29), and C-200 (9.87), respectively in end of 3 cycles frying. These consequences are representative of this fact that essential oils except M-400 and F-400 had AnV higher than control and also TBHQ was the best antioxidant. This is in agreement to observations of Zhang *et al.* (2010) by evaluation oxidative stability sunflower oil treated with Carnosic acid in comparison to synthetic antioxidants BHA, BHT, and TBHQ. The mixture sample was better than M-200, C-400, and C-200, alone. Perhaps, this event is due to the synergistic

effect of EOs. Similar results were obtained in PV content and prior study (Sadeghi *et al.*, 2016 a).

The higher anisidine value in some of the EOs rather than control is due to decomposition and evaporation of them at frying high temperatures because EOs are volatile. As previously stated, terpenoids are the main ingredients of EOs. They generally are susceptible to rearrangement process at elevated heats. McGraw *et al.* (1999) graded thermal decomposition of terpenes to four various oxidative reactions including epoxidation, breaking of double bands, allylic oxidation to aldehydes, ketones, and alcohols, and dehydrogenation to aromatic products. The lower content of active components in the essential oils is another reason for their low activity. Inanc and Maskan (2013) with the investigation of the effect of seven EOs (Sage, Cinnamon, Turmeric, Rosemary, Thyme, Clove, and Oregano) and BHT on corn oil at 150°C and 180°C observed essential oils except oregano were not effective antioxidants for corn oil.

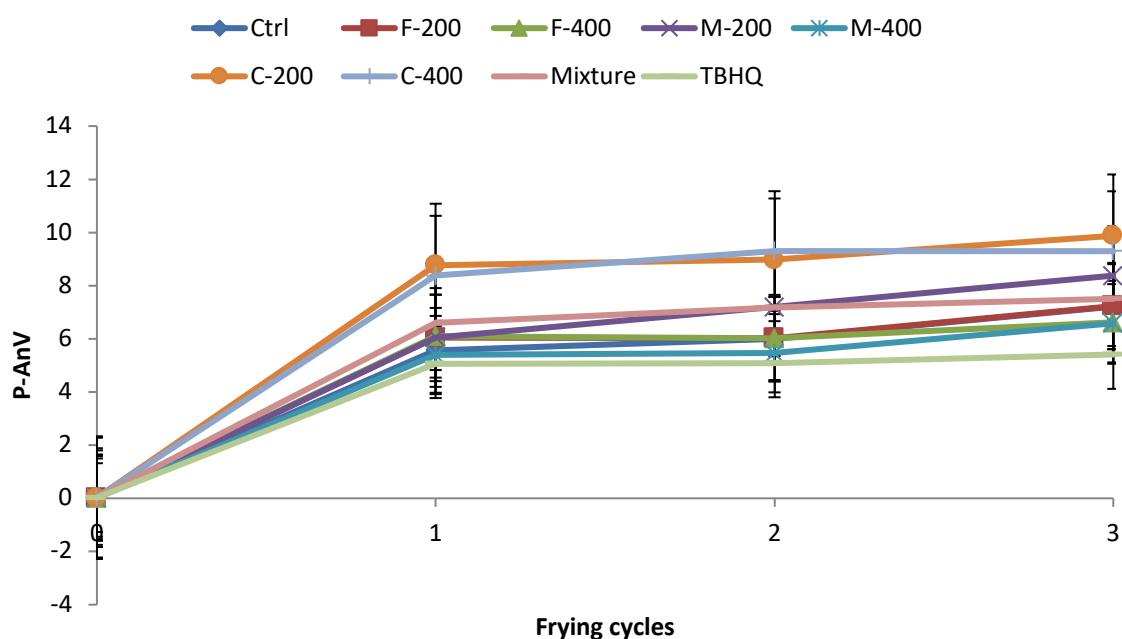


Figure 3. The effect of essential oils on P-Anisidine value of frying oil during frying cycles

3.2.4. Total polar compounds (TPC)

Polar compounds measurement is one of the best indicators for the evaluation of deterioration in frying oil that provide information about newly formed components with a higher polarity rather than triglycerides. The TPC is determined by solving of oil in fairly nonpolar solvents (such as benzene or toluene) and loading of it on silica gel column that absorbs polar materials. Then these compounds eluted directly from a column by diethyl ether or a mixture of methanol and chloroform or indirectly with solvent evaporation and weighting nonpolar fat and calculation of its weight difference with the primary sample. The content of the TPC is shown in Figure 4. It was 8.43% before frying. After the first cycle, the TPC was increased for all samples significantly that was in agreement with other parameters (see supplementary, Table S1). At the second cycle, the quantity of polar compounds in F-200, F-400, M-200, and M-400 samples slightly raised but for Control, C-200, C-400, mixture, and TBHQ slightly decreased. The small decrease in polar compounds perhaps is due to the exiting of

volatile ones during frying. In the third cycle, this value enhanced for all samples except F-400. Similar results obtained by Farhoosh and Tavassoli-Kafrani (2010), and Tabee et al. (2009). Also, the TPC for all samples with a nominal difference was in the same range at 3 cycles frying process and ever there was no difference between TBHQ and control at cycle 3. Moreover, at the end of 3 frying cycles F-400 (10.11), C-200 (10.01), and mixture (10.12) had lower polar components content than the control (10.15). Nothing change in polar compounds maybe is due to this fact that sufficient time for increasing production of polar compounds had not given. In other words, probably during 3 cycles frying deterioration of oil does not happen significantly. Similarly, Casarotti and Jorge (2014) declared there was no significant difference among treats of Soybean oil, TBHQ, Rosemary extract and a mixture of TBHQ+ Rosemary extract during primary cycles of frying. Thus, frying time has a purposeful effect in the decomposition of frying oil and formation of polar mixtures (Corsini et al., 2009; Abdel-Razek et al., 2012).

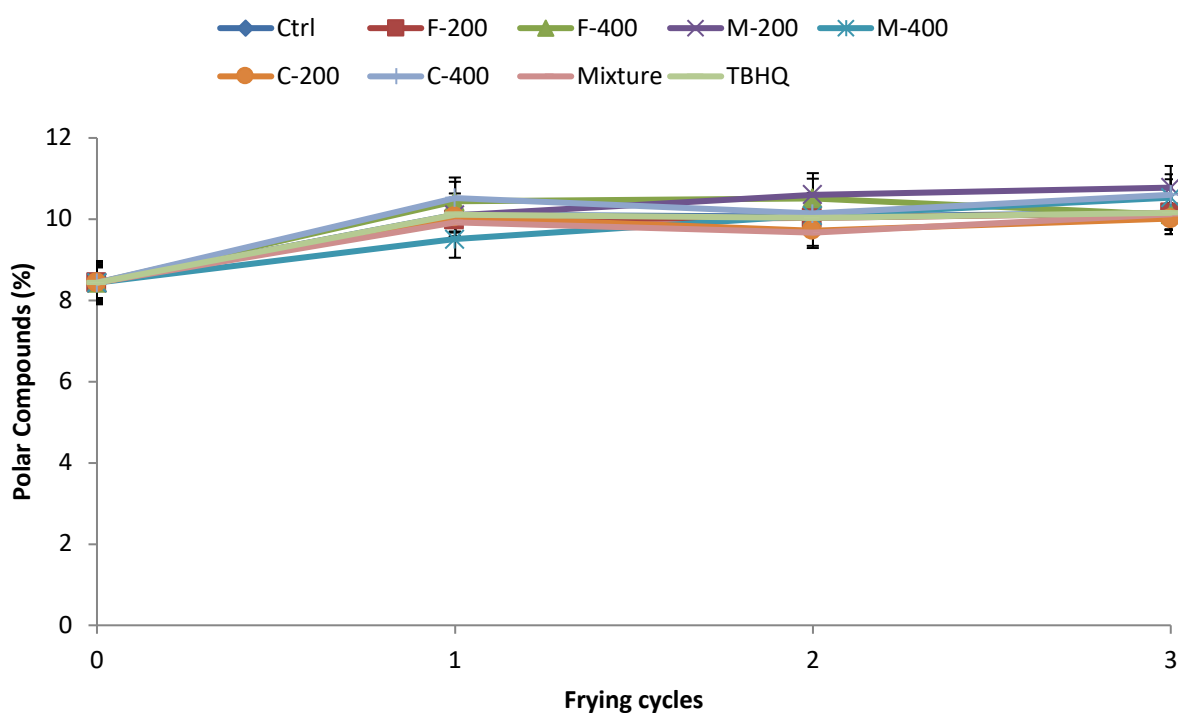


Figure 4. The effect of essential oils on content of polar compounds of frying oil during frying cycles

3.3. DPPH radical scavenging assay

This test evaluates the ability of essential oils for scavenging of radical DPPH by donating hydrogen to free radical and is a good indicator for investigation antioxidant activity (Chung et al., 2006). Antioxidant activity of EOs is presented in Table 2. According to the table, the highest activity was related to TBHQ followed by M-400, F-400, M-200, C-400, C-200, Mixture, and F200, respectively. Also, all EOs in concentration 400 ppm were more effective than concentration 200 ppm that was

agreed with PV and P-AnV parameters but not for mixture sample. These observations showed that essential oils had low scavenging activity. Hosseini *et al.* (2012), Ghasemi Pirbalouti *et al.* (2016), Einafshar *et al.* (2012), and Bettaieb *et al.* (2010) acquired analogous results with studying of antioxidant activity *Ferulago*, *Mentha* and *Cuminum* essential oils. Lower scavenging activity in essential oils possibly is due to the lower content of active components in them (Inanc and Maskan, 2013).

Table 2. The DPPH radical scavenging activity of the essential oils from *Ferulago angulata*, *Mentha pulegium*, and *Cuminum cyminum* at different concentrations beside the synthetic antioxidant

Samples	F-200	F-400	M-200	M-400	C-200	C-400	Mixture	TBHQ
% RSA	1.54	2.12	2.11	2.46	1.93	2.06	1.69	8.54

RSA: Radical scavenging activity

3.4. Sensory evaluation

Results of the sensory evaluation are shown in Figure 5. The scores of aroma for TBHQ and control did not have a significant difference in all cycles of frying. Also, they had higher scores rather than other samples. Moreover, a significant difference was not observed among essential oil treats (see supplementary, Table S2). For flavor, the highest score was belonged to TBHQ, followed by control and essential oils. From the point of this characteristic, the difference among samples was meaningful. The obtained results for color were similar to aroma and flavor results and almost all samples had scores higher than 7. In fact, potatoes color for all treats was similar with well quality. Finally, the total acceptability of treats was too in agreement with stated properties. Concerning observed results for all investigated properties,

the synthetic antioxidant had the highest score. Since it retards induction period of oxidation and prevents the formation of secondary products responsible for rancid aroma and flavor. After TBHQ, the control was the best treatment because the aroma and flavor of fried potatoes were changed by essential oils and this was effective on the acceptability of panelists. Furthermore, in no one of treats there were significant differences among cycles of frying perhaps because after 3 cycles process yet significant oxidation has not happened in the oil that was in agreement with chemical parameters (TPC & P-AnV, FFA) outcomes and issues of Alizadeh et al. (2016) and Lalas and Dourtoglou (2003) that notice significant difference among treatments after third and fourth cycles of frying, respectively.

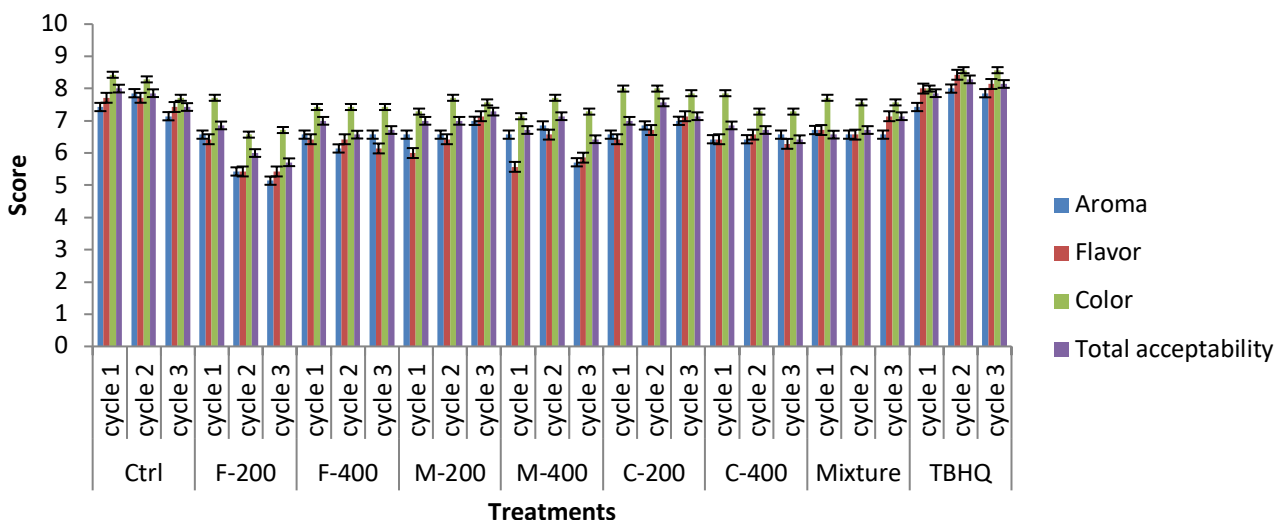


Figure 5. The effect of essential oils on sensory properties of the fried potatoes during frying cycles

4. Conclusions

Totally, we can say although M-400 was more effective than the other essential oils in PV and P-AnV and scavenging of DPPH, most of the EOs were not effective antioxidants during 3 cycles frying basically due to sensitivity natural components to high temperatures and volatility of them. Also, it is predicted that with increasing frying cycles, they cannot be effective antioxidants for the stability of frying oil in reason to the decomposition of these natural compounds. Thus, it is recommended EOs be used for storage stability of oils instead of frying processes.

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