

Influence of the Essential Oil of *Foeniculum vulgare* Mill. on Sunflower Oil during the Deep-frying Process of Chinese *Maye*

Haohao Wu, Yunlong Zhao, Haoduo Yang, Dongying Wang*, and Xuede Wang*

College of Food Science and Engineering, Henan University of Technology, Zhengzhou 450001, CHINA

Abstract: Sunflower oil (SFO) is faced with serious oxidation problems during the deep-frying of Chinese *Maye*, and the search for natural antioxidants has become a focus of scientific research due to the potential toxicity of synthetic antioxidants. In the present study, the *Foeniculum vulgare* Mill. essential oil (FVEO), *tert*-butylhydroquinone (TBHQ) were added to SFO for a 30 h deep frying experiment and the results showed that FVEO added to sunflower oil at 1 g/kg was similar to that of TBHQ-0.01 g/kg, and FVEO-1.5 g/kg would promote the oxidation of SFO. FVEO to sunflower oil also prominently restrained the decrease of the sensory properties of the fried product, Chinese *Maye*, including appearance, taste, flavor and overall acceptance by 24.2%, 20.2%, 46.1% and 56.0% ($p < 0.01$ or $p < 0.05$), respectively. The results indicated that FVEO could be used as a natural antioxidant to replace TBHQ in the deep-frying process of SFO, but further research is needed on the key antioxidant constituent of FVEO.

Key words: *Foeniculum vulgare* Mill. essential oil, sunflower oil, oxidative stability, sensorial attributes

1 Introduction

With the rapid progress of modern lifestyle, deep-frying procedure has become an extremely common method for food preparation. The fried products are quite popular because of its unique flavor and tempting taste^{1,2}. Meanwhile, the concerns about the safety of vegetable oils employed in deep-frying procedure have increased³. In the deep-frying procedure, certain secondary products of vegetable oils such as esters, ketones, alcohols, lactones, aldehydes and hydrocarbons are produced by the oxidative degradation. Both of the nutritional contents and sensorial properties for the fried products are affected by these secondary products, although the frying vegetable oils are carefully monitored, including frying temperature, time and the storage of frying oil after use, they be oxidized after repeated heating is inevitable⁴. Synthetic antioxidants have been added to foods as additives to avoid lipid oxidation in oils for more than 70 years⁵. The use of synthetic antioxidants including butylated hydroxyanisole (BHA), butylated hydroxytoluene (BHT) and *tert*-butylhydroquinone (TBHQ) have been found to be effective in preventing oxidation and changes in sensory properties of foods¹. Nevertheless, the usages of synthetic antioxidants have been limited because of concerns about their possible

toxicity and carcinogenic effects on humans and animals. For instance, TBHQ is no longer be allowed in the food industry in many nations⁶. In recent years, various natural antioxidants have been investigated. Spices and herbs have received a great deal of scientific attention for their potential medicinal and therapeutic value. The essential oils, bioactive substances and phenolic components of spices and herbs have demonstrated significant antioxidant and antibacterial activities⁷. It has been found that essential oils derived from herbs and spices not only provide a pleasant flavour, but also improve the oxidative stability and organoleptic properties of vegetable oils⁸. For example, the addition of chili pepper seed oil and sweet pepper seed oil to sunflower oil could increase its oxidative stability during the deep-frying process⁹. The volatile oils of flowers of *Tripleurospermum conoclinium* had significant oxygenated sesquiterpene content and showed exceptional anti-inflammatory and antidiabetic properties¹⁰. Therefore, it is very meaningful to search for natural antioxidants from spices and herbs⁶.

As one of the widespread medicinal plants, *Foeniculum vulgare* Mill. belongs to Apiacea (Umbelliferae) family¹¹. It is an annual or perennial herb native to the Mediterranean region. *Foeniculum vulgare* Mill. has been introduced in

*Correspondence to: Dongying Wang, College of Food Science and Engineering, Henan University of Technology, Zhengzhou 450001, CHINA. Xuede Wang, College of Food Science and Engineering, Henan University of Technology, Zhengzhou 450001, CHINA
E-mail: dywang@haut.edu.cn (DW), wangxuede1962@126.com (XW)

Accepted July 19, 2023 (received for review May 23, 2023)

Journal of Oleo Science ISSN 1345-8957 print / ISSN 1347-3352 online

<http://www.jstage.jst.go.jp/browse/jos/> <http://mc.manuscriptcentral.com/jjocs>



China for more than 1000 years. Extracts and essential oils from *Foeniculum vulgare* Mill. were found to possess antimicrobial¹²⁾, antioxidant, antidiabetic, and anti-inflammatory activities^{13–15)}. In these studies, Wild *Foeniculum vulgare* Mill. has been shown to be a potential source of natural antioxidants and antibacterial agents¹⁶⁾. *Foeniculum vulgare* Mill. has also been shown to have hepatoprotective, antioxidant, antithrombotic, anti-inflammatory, antidiabetic, antitumor, acaricidal, antifungal and antibacterial activities^{17, 18)}. Other studies have found that (*E*)-anethole, the main component of the essential oil of *Foeniculum vulgare* Mill. from Florence has antibacterial activity, the essential oil and hexane extract of Florence fennel can be used as a food preservative¹⁹⁾. The essential oils from different spices or herbs and their applications in frying oils have been reported widely, but there are no study on the effects of *Foeniculum vulgare* Mill. essential oil on fried sunflower oil.

Therefore, the purpose of this study was to investigate the influence of the essential oil of *Foeniculum vulgare* Mill. on the oxidative stability of sunflower oil during the deep-frying process and the sensory properties of the fried product, Chinese *Maye*, which is an economical, crispy, fried product made from pure flour, and revealed the essential compounds by GC-MS analysis. This will be the first research about the effects of *Foeniculum vulgare* Mill. essential oil (FVEO) on frying sunflower oil (SFO) and its frying products. The study will also provide the theoretical basis for the application of *Foeniculum vulgare* Mill. in the food and other cosmeceutical industries.

2 Materials and Methods

2.1 Materials and chemicals

Foeniculum vulgare Mill. (4 × 10 kg) was purchased from Zhang Zhongjing Pharmacy, Zhengzhou, China. Cold-pressed sunflower oil (without added antioxidants) was purchased from COFCO Group Co., Ltd. Hemp leaf embryo was purchased from Yonghui supermarket, Zhengzhou, China. TBHQ was obtained from applyhem, Darmstadt, Germany. In addition, all analytical/HPLC-grade chemicals were provided by Zhengzhou Senbo Biotechnology Co., Ltd., China.

2.2 Steam distillation of FVEO

The steam distillation of FVEO was carried out according to the procedure described previously²⁰⁾. The yield of essential oil was the ratio between the volume of essential oil obtained and the weight of the *Foeniculum vulgare* Mill. seeds used²¹⁾. The water was removed from the distilled essential oil with anhydrous sodium sulfate and stored in a vial protected from light at –20°C before analysis.

2.3 Chemical analysis of FVEO

After being diluted 1:50 (m/v) with chromatogen-grade *n*-hexane, the FVEO was filtered through a 0.22 µm microporous filtration membrane into gas phase vials and subsequently kept at –20°C. The volatile oil composition was analyzed and determined by gas chromatography/mass spectrometry based on the previous study with slight modifications¹⁰⁾. Briefly, the chemical analysis was performed with an Agilent 6890-5973 N system operated at 60 eV using an HP-5MS column (30 m × 0.25 mm i.d., 0.25 mm f.t., Agilent, USA). For the detector and the injector, the temperatures were set to 290°C and 250°C, respectively. At a split ratio of 1:20, the carrier gas helium was supplied at 1 mL/min. The temperature of the system oven was programmed as follows: Initial temperature 50°C, increased at 10°C/min to 70°C and kept for 2 min, increased at 1°C/min to 125°C and kept for 2 min, and then, increased at 4°C/min to 210°C and kept for 2 min, at last, increased at 20°C/min to 280°C and kept for 5 min. The mass spectra were saved at 70 eV with the mass range *m/z* 30 to 500. Mass spectrometry retrieval standard library: NIST2017 database (<http://webbook.nist.gov>) was chosen since it matches more than 90% of the compounds, and the area normalization method was used to determine the contents of each FVEO component. Mass spectrometry retrieval standard library: NIST2017 database (<http://webbook.nist.gov>), selected matches more than 90% of the substances and the content of each component of FVEO was calculated by area normalization method.

2.4 Antioxidant test of FVEO

The antiradical activity of FVEO was evaluated by 1,1-diphenyl-2-picrylhydrazyl (DPPH) radical, 2-ethyl-benzothiazoline sulphonate acid-6 (ABTS) radical (ABTS^{•+}) and hydroxyl radical (HO[•]) assays, in contrast, the reducing power of FVEO was evaluated by the ferric ion reducing antioxidant power (FRAP), following the methods reported in previous studies and with slight modifications^{19, 22–24)}.

2.5 Deep-frying process

According to our previous studies on this topic, SFO samples FVEO-1, FVEO-1.5 and TBHQ-0.01 were obtained by adding FVEO and TBHQ to the SFO. SFO samples without antioxidants (Control) also need to be prepared. All of these SFO samples were stored in a refrigerator at 4°C. The deep-frying procedure was started once the temperature of the SFO samples reached 180°C, and the whole frying process was continued for 30 h, each batch of Chinese *Maye* could be obtained when it stayed in sunflower oil for about 3 min, and there was 616 *Maye* batch were obtained, while the sunflower oil was heated over for 30 h. During the whole deep-frying process, the color, viscosity, acid value, malonaldehyde (MDA), total polar compounds and conjugated diatriene of the SFO samples were

measured every 6 hours according to Chinese National Standards GB5009.229–2016, GB/T 22235–2008, GB 5009.229-2016, GB5009.181-2016, GB5009.202-2016 and GB/T22500-2008, respectively.

2.6 Determination of fatty acid composition

The fatty acids of the frying oil samples were determined according to the normalization method in CNS GB 5009.168-2016 Determination of fatty acids in foods. Gas chromatographic conditions: HP-88 capillary column (100 m × 0.25 mm × 0.2 μm); inlet and detector temperatures of 250°C and 280°C, respectively; carrier gas: high purity nitrogen at a flow rate of 1 mL/min; hydrogen generator: flow rate of 30 mL/min. The column temperature was initially 170°C, then increased to 220°C at 4°C/min without holding, and then increased to 240°C at 1°C/min; injection volume: 1 μL.

2.7 Sensory analysis of Chinese *Maye* samples

At the end of the frying process, the different sensory attributes of *Maye* were evaluated including appearance, taste, flavor and overall acceptability. A consumer-based hedonic test was performed by 25 males and 25 females (18–40 years old) on a nine-point hedonic scale (1: dislike, 5: neutral, 9: extremely like) *Maye* fried in the same SFO was evaluated as a large group, with samples of *Maye* placed randomly at different times. Each large group of samples was tasted at 15-minute intervals and each sample was tasted at 1-minute intervals. We explained the meaning of each attribute to the group members to reduce misunderstanding and asked them to read the instructions on the sensory sheet before performing the sensory analysis. Most importantly, they will have plenty of time to familiarize themselves with the sensory assessment process if they were unsure of the details of the process.

2.8 Statistical analysis

Unless otherwise stated, data are presented as means and data in tables and figures are indicated as mean ± standard deviation (n = 10). For the statistical analysis, GraphPad Prism 8 (GraphPad Software, San Diego, USA) analysed the data by one-way ANOVA, where $p < 0.05$ and $p < 0.01$ were determined to be statistically significant and highly significant, respectively.

3 Results and Discussion

3.1 Extraction of FVEO

The FVEO yield was 1.53% (w/w). At room temperature, FVEO is one yellowish clear liquid oil with a distinctive anise aroma and sweetness. It is important to note that the results of extraction yield are close to the previous studies²⁵⁾.

3.2 Chemical composition of FVEO

The use of plants in medicine and cosmetics has increased dramatically with the rapid growth of physical therapy²⁶⁾. Natural ingredients derived from plants are getting a lot of attention in the cosmetic industry²⁷⁾. In the present investigation, as reported in Table 1, 28 chemical constituents were identified in FVEO, which represent 99.42% of total essential oil with the main chemical constituents anethole (79.53%), fenchone (8.49%), estragole (2.95%), limonene (2.56%), α-pinene (1.47%), and γ-terpinene (1.14%). Napoli *et al.* also found that the main components of wild Sicilian fennel were fenchone and estragole, with growth location having an impact on the content²⁸⁾. Shahat *et al.* (2011)²⁹⁾ found that the main components of the *Foeniculum vulgare* Mill. essential oil from Egyptian were estragole (51.04%), limonene (11.45%), l-fenchone (8.19%). These research's slightly differing findings could be explained by variations in the genetic makeup of the populations, geographic and environmental characteristics, and biological and abiotic pressures³⁰⁾.

3.3 FVEO antioxidant activity

DPPH radical and ABTS radical scavenging is commonly used in antioxidant tests, where antioxidants bind by donating hydrogen or electrons to free radicals to scavenge them³¹⁾. The result demonstrated a dose-dependent scavenging capacity against DPPH (Fig. 1A) and ABTS⁺ (Fig. 1B). The free radical scavenging percentages, with the concentration of FVEO at 10, 20, 30, 40 and 50 mg/mL, were 13.0%, 25.5%, 31.2%, 38.21% and 46.1% against DPPH, while the concentration of FVEO at 10, 15, 20, 25 and 30 mg/mL, were 19.3%, 30.1%, 40.8%, 49.9% and 56.1% against ABTS⁺. The OH radical is generated when H₂O₂ encounters Fe²⁺. Wang *et al.* studied the Fe²⁺ chelating ability of *Ocimum basilicum* essential oil and found that the Fe²⁺ chelating ability of *Ocimum basilicum* essential oil was mainly due to the presence of *Artemisia absinthium*³²⁾. The ability of the essential oil to scavenge OH radicals can thus be seen in a direct scavenging manner, as well as in a manner that chelates Fe²⁺ before the onset of the reaction and thus prevents the production of free radicals. As can be seen in Fig. 1C, the OH radical scavenging rate of FVEO increased from 36.45% to 51.67% as the concentration increased from 10 mg/mL to 30 mg/mL, which is close to the scavenging rate of 59.98% for BHT. The increase in the OH radical scavenging rate of the essential oil was slower and the scavenging capacity of BHT was not as good as the rest of the indicators, this phenomenon may be caused by the indicator being a water-based indicator. Meanwhile, FRAP (Fig. 1D) was determined to find the FVEO reducing power. The FRAP values increased from 0.24 to 0.44 as the concentration increased from 10 mg/mL to 50 mg/mL. The results indicate that FVEO had the antioxidant capacity to be investigated with

Table 1 Volatile components of FVEO.

No.	Compound name	Molecular formula	CAS number	w/w (%)
1	α -Pinene	C ₁₀ H ₁₆	80-56-8	1.47%
2	Camphene	C ₁₀ H ₁₆	79-92-5	0.06%
3	Sabinene	C ₁₀ H ₁₆	3387-41-5	0.29%
4	β -Pinene	C ₁₀ H ₁₆	18172-67-3	0.12%
5	β -Myrcene	C ₁₀ H ₁₆	123-35-3	0.41%
6	α -Phellandrene	C ₁₀ H ₁₆	99-83-2	0.21%
7	α -Terpinene	C ₁₀ H ₁₆	99-86-5	0.02%
8	<i>p</i> -Cymene	C ₁₀ H ₁₄	99-87-6	0.75%
9	D-Limonene	C ₁₀ H ₁₆	5989-27-5	2.56%
10	Eucalyptol	C ₁₀ H ₁₈ O	470-82-6	0.17%
11	trans- β -Ocimene	C ₁₀ H ₁₆	3779-61-1	0.39%
12	β -Ocimene	C ₁₀ H ₁₆	13877-91-3	0.04%
13	γ -Terpinene	C ₁₀ H ₁₆	99-85-4	1.14%
14	L-Fenchone	C ₁₀ H ₁₆ O	7787-20-4	8.49%
15	Butanoic acid, 2-methyl-, 3-methyl butyl ester	C ₁₀ H ₂₀ O ₂	27625-35-0	0.01%
16	Isopentyl valerate	C ₁₀ H ₂₀ O ₂	2050-9-1	0.01%
17	1,3-Cyclohexadiene, 1,3,5,5-tetramethyl-	C ₁₀ H ₁₆	4724-89-4	0.01%
18	(-)-Camphor	C ₁₀ H ₁₆ O	464-48-2	0.09%
19	(+)-2-Bornanone	C ₁₀ H ₁₆ O	464-49-3	0.05%
20	Terpinen-4-ol	C ₁₀ H ₁₈ O	562-74-3	0.05%
21	Estragole	C ₁₀ H ₁₂ O	140-67-0	2.95%
22	α -Fenchyl acetate	C ₁₀ H ₂₀ O ₂	1000429-50-4	0.02%
23	Carvone	C ₁₀ H ₁₄ O	99-49-0	0.25%
24	Benzaldehyde, 4-methoxy-	C ₈ H ₈ O ₂	123-11-5	0.12%
25	Benzaldehyde, 3-methoxy-	C ₈ H ₈ O ₂	591-31-1	0.20%
26	Anethole	C ₁₀ H ₁₂ O	104-46-1	79.53%
27	Pentadecanal-	C ₁₅ H ₃₀ O	2765-11-9	0.01%
28	5-Heptenal, 2,6-dimethyl-	C ₉ H ₁₆ O	106-72-9	0.01%
Total				99.42%

further research as a natural antioxidant³³⁾.

3.4 Antioxidant effect of FVEO in the deep-frying of Chinese *Maye*

Polar compounds are produced by the thermal, hydrolytic, and oxidative alteration during the frying process of vegetable oils and for which the level of TPC is often considered to be an important indicator of oil deterioration³⁴⁾. Malondialdehyde (MDA) is the main secondary product of oxidative deterioration of vegetable oils³⁵⁾. In addition, conjugated dienes and conjugated trienes are formed, which can give rise to absorption peaks at 232 nm and 268 nm, respectively. Therefore, their content could be assessed

using absorbance at 232 nm and 268 nm^{36–38)}. In the present study (Fig. 2), the values of MDA, TPC, conjugated dienes and conjugated trienes of all of the SFO samples were dramatically rose as the amount of frying time increased. For the MDA value (Fig. 2A), the SFO sample TBHQ-0.01 showed the slowest growth rate, followed by the control sample and FVEO group samples. However, for TPC (Fig. 2B), conjugated dienes (Fig. 2E) and conjugated trienes (Fig. 2F) values, the control samples were the slowest, followed by TBHQ and FVEO group samples.

The changes in MDA value (Fig. 2A) of the SFO sample of FVEO were not significantly different from the sample of TBHQ-0.01 and the control samples, and the inhibitory

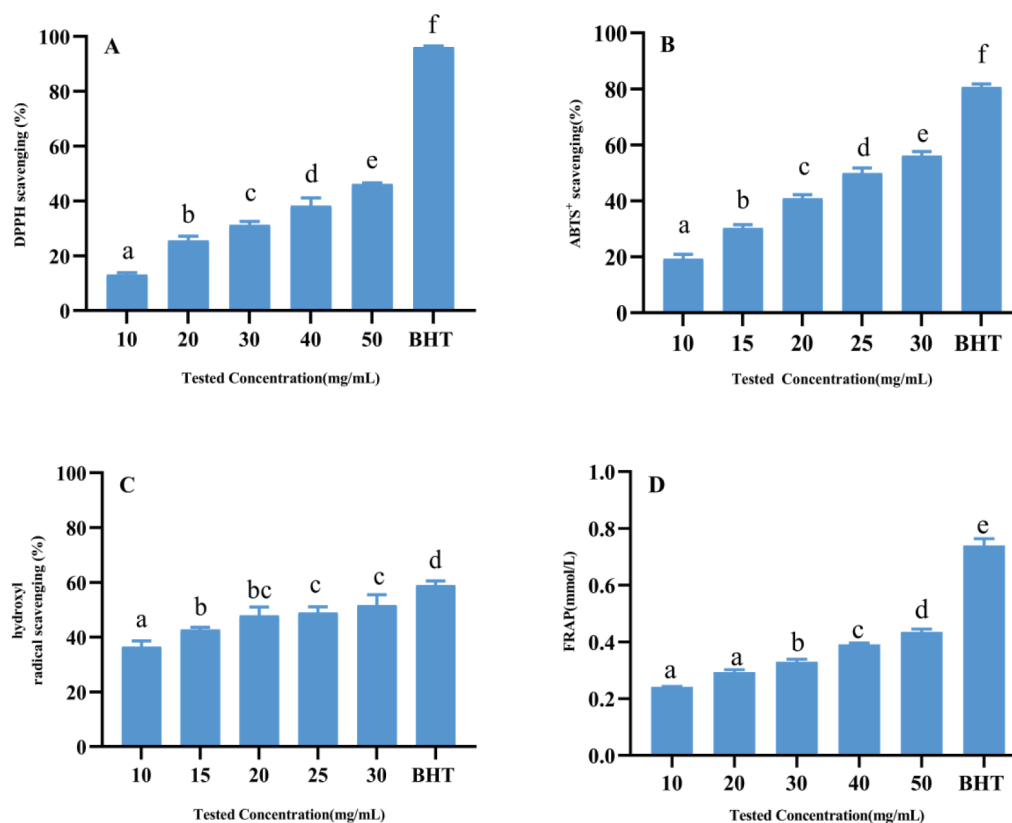


Fig. 1 Antioxidant effects of FVEO and positive BHT *in vitro*. Values are expressed as means $\pm$ SD. (A) DPPH[•] scavenging activity of FVEO compared with BHT (0.1 mg/mL). (B) ABTS⁺ scavenging activity of FVEO compared with BHT (0.1 mg/mL). (C) OH[•] scavenging activity of FVEO compared with BHT (0.1 mg/mL). (D) Ferric-reducing antioxidant power (FRAP) of FVEO compared with BHT (0.1 mg/mL). Different letters (a, b, c, d) indicate that the difference is significant ($p < 0.05$).

effect of FVEO on MDA content was $1.5 \text{ g/kg} > 1 \text{ g/kg}$. After 18 h, the MDA values (Fig. 2A) of SFO samples from the FVEO group showed a significant increase ($p < 0.01$). It can be seen that FVEO was weaker than TBHQ in terms of antioxidant capacity and a pro-oxidation phenomenon was observed at the late stage of frying. For conjugated dienes (Fig. 2E) and conjugated trienes (Fig. 2F), the growth rate of the FVEO group showed a significant increase in the late frying period ($p < 0.01$). The SFO sample of FVEO-1.5 g/kg showed a significantly faster increase than 1 g/kg; meanwhile, the CD value (Fig. 2E) of the sample FVEO-1.5 g/kg reached 23.835 at 30 h. The CT value (Fig. 2F) showed that the sample FVEO-1.5 g/kg showed a significantly higher level than the rest of the SFO samples at 30 h of frying ($p < 0.05$), while the FVEO-1 g/kg showed no significant difference with TBHQ. For the total polar compounds, the changes in content were affected by several factors and were similar to those for the conjugated diatrienes. It is observed from this study that high concentrations of FVEO during frying led to an increase in malondialdehyde, polar components and conjugated diatrienes in SFO, whereas low concentrations of FVEO samples had a similar effect on

SFO as TBHQ.

The oxidative polymerization reactions during frying produce functional groups with a high degree of conjugated double bonding which leads to a deepening of the colour of the frying oil. The viscosity change of the oil is mainly influenced by the increase of polymer, and antioxidants, as well as the generation of FVEO in the late stage of frying can affect the viscosity of sunflower oil. As shown in Fig. 2, the values of yellow (Fig. 2G), red (Fig. 2H) and viscosity (Fig. 2C) of the four SFO samples increased significantly ($p < 0.05$). During the frying process, the control group samples showed the slowest increase in color and viscosity values, followed by the TBHQ-0.01 samples and the FVEO group samples. As observed by the changes in the values of yellow and viscosity, the values of yellow (Fig. 2G) and viscosity (Fig. 2C) of the sample FVEO-1.5 g/kg grew higher than the sample FVEO-1 g/kg. There was no significant difference in the change in viscosity values between the SFO sample FVEO-1 g/kg and TBHQ samples and the control samples before frying for 18 hours. After 18 h, the values of yellow and viscosity from the FVEO group showed significant differences ($p < 0.05$) with control as well as the

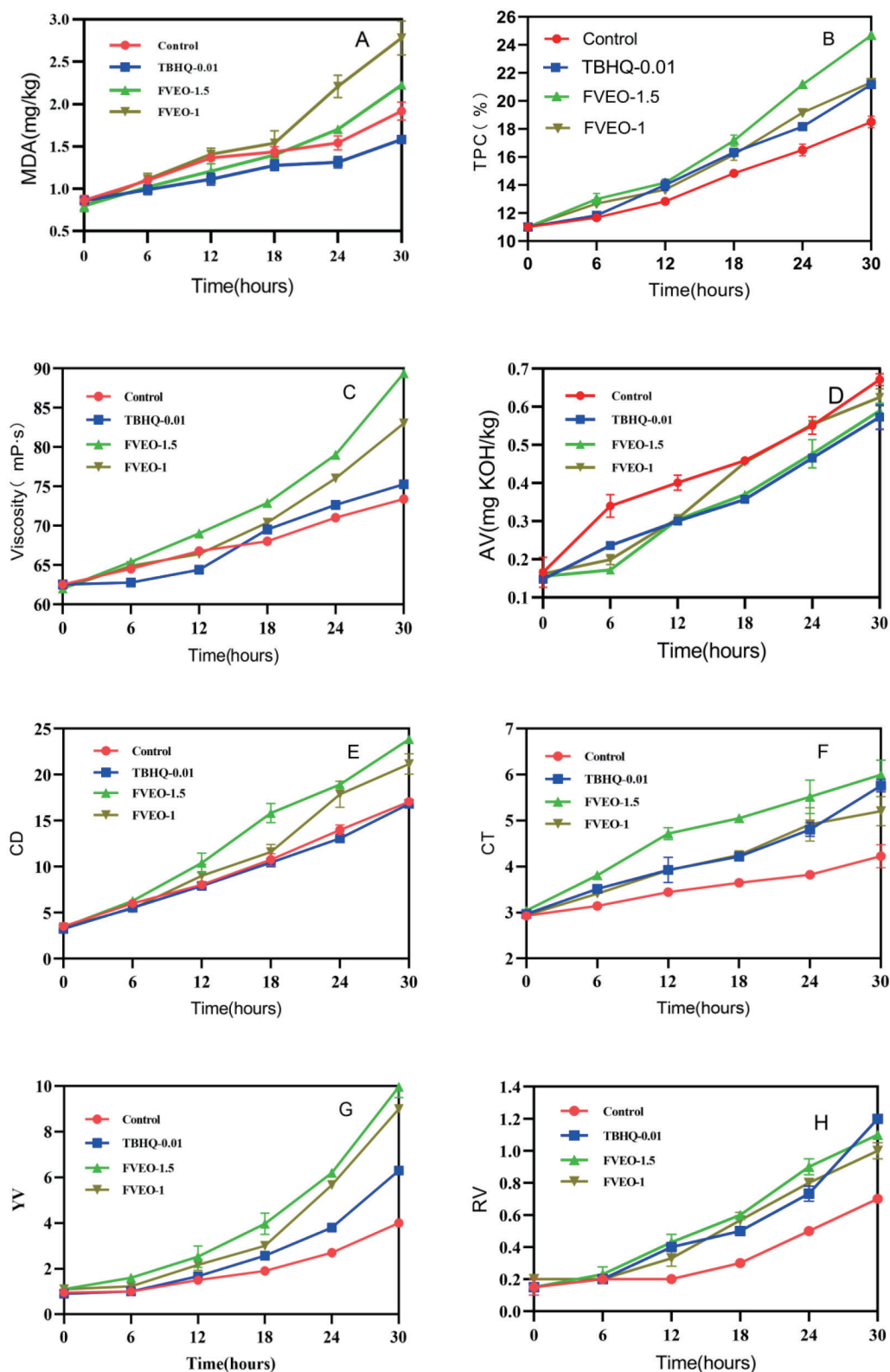


Fig. 2 The values for the viscosity, malonaldehyde (MDA), total polar compounds (TPC), acid value (AV), conjugated dienes (CD), conjugated triteness (CT), yellow value and red value of sunflower oil added by nothing, FVEO (1.5 g/kg), FVEO (1 g/kg) TBHQ (0.01 g/kg) during the deep-frying of Chinese *Maye* for 30 h. Values are expressed as means $\pm$ SD (n=10).

Table 2 Fatty acid composition of frying oil.

Group	Hour	C14:0 ^c	C16:0	C16:1	C18:0	C18:1	C18:2	C20:0	C18:3	C22:0
Control	0	0.077 ± 0.001	6.292 ± 0.059	0.079 ± 0.007	3.744 ± 0.001	26.989 ± 0.004	61.573 ± 0.051	0.242 ± 0.002	0.259 ± 0.001	0.746 ± 0.002
	6	0.075 ± 0.001	6.323 ± 0.007	0.079 ± 0.001	3.771 ± 0.001	27.123 ± 0.008	61.396 ± 0.004	0.240 ± 0.002	0.244 ± 0.005	0.751 ± 0.002
	12	0.077 ± 0.001	6.496 ± 0.002	0.075 ± 0.001	3.827 ± 0.013 ^a	27.285 ± 0.026 ^a	61.002 ± 0.006 ^a	0.241 ± 0.005	0.234 ± 0.001 ^a	0.765 ± 0.001
	18	0.077 ± 0.004	6.571 ± 0.005	0.075 ± 0.001	3.844 ± 0.001 ^a	27.573 ± 0.039 ^a	60.635 ± 0.046 ^a	0.241 ± 0.002	0.217 ± 0.004 ^a	0.770 ± 0.011
	24	0.078 ± 0.001	6.739 ± 0.289	0.075 ± 0.001	3.855 ± 0.006 ^a	27.766 ± 0.091 ^a	60.255 ± 0.214 ^a	0.243 ± 0.001	0.208 ± 0.004 ^a	0.779 ± 0.014
	30	0.079 ± 0.001 ^a	6.893 ± 0.134 ^a	0.076 ± 0.002	3.933 ± 0.021 ^a	28.108 ± 0.027 ^a	59.668 ± 0.101 ^a	0.248 ± 0.004	0.203 ± 0.001 ^a	0.794 ± 0.014 ^a
TBHQ	0	0.076 ± 0.001	6.405 ± 0.099	0.077 ± 0.004	3.723 ± 0.004	26.931 ± 0.068	61.534 ± 0.025	0.251 ± 0.002	0.254 ± 0.002	0.751 ± 0.003
	6	0.076 ± 0.002	6.380 ± 0.270	0.075 ± 0.002	3.740 ± 0.029	27.189 ± 0.168	61.355 ± 0.060 ^a	0.244 ± 0.001	0.251 ± 0.005	0.754 ± 0.005
	12	0.077 ± 0.001	6.386 ± 0.002	0.072 ± 0.004	3.792 ± 0.014 ^a	27.208 ± 0.044	61.118 ± 0.041 ^a	0.242 ± 0.002	0.239 ± 0.006	0.762 ± 0.002
	18	0.077 ± 0.001	6.524 ± 0.001	0.070 ± 0.003	3.825 ± 0.011 ^a	27.446 ± 0.014 ^a	60.809 ± 0.018 ^{ab}	0.247 ± 0.001 ^b	0.231 ± 0.009 ^{ab}	0.773 ± 0.008 ^a
	24	0.078 ± 0.001	6.693 ± 0.006	0.077 ± 0.001	3.980 ± 0.002 ^{ab}	27.780 ± 0.003 ^a	60.168 ± 0.011 ^a	0.245 ± 0.001	0.215 ± 0.002 ^{ab}	0.763 ± 0.001 ^a
	30	0.078 ± 0.001	6.722 ± 0.001	0.080 ± 0.014	3.938 ± 0.006 ^a	28.034 ± 0.049 ^a	59.924 ± 0.030 ^a	0.249 ± 0.003	0.204 ± 0.002 ^a	0.774 ± 0.001 ^a
FVEO (1 g/kg)	0	0.069 ± 0.001	6.471 ± 0.015	0.074 ± 0.001	3.731 ± 0.196	26.802 ± 0.078	61.577 ± 0.109	0.252 ± 0.006	0.248 ± 0.001	0.787 ± 0.001 ^b
	6	0.071 ± 0.001	6.355 ± 0.002 ^a	0.076 ± 0.009	3.890 ± 0.001 ^b	27.045 ± 0.004 ^a	61.279 ± 0.005 ^a	0.245 ± 0.006	0.248 ± 0.002	0.796 ± 0.001 ^b
	12	0.073 ± 0.001 ^a	6.486 ± 0.001 ^a	0.078 ± 0.001	3.905 ± 0.134	27.434 ± 0.094 ^a	60.835 ± 0.030 ^{ab}	0.253 ± 0.001 ^b	0.245 ± 0.008	0.811 ± 0.002 ^b
	18	0.075 ± 0.001 ^a	6.544 ± 0.001 ^a	0.079 ± 0.001	3.949 ± 0.001 ^b	27.546 ± 0.030 ^a	60.528 ± 0.008 ^a	0.255 ± 0.002 ^b	0.219 ± 0.001 ^a	0.828 ± 0.024 ^{ab}
	24	0.076 ± 0.001 ^a	6.768 ± 0.002 ^a	0.080 ± 0.002	4.086 ± 0.001 ^b	28.336 ± 0.001 ^{ab}	59.404 ± 0.003 ^{ab}	0.271 ± 0.003 ^{ab}	0.209 ± 0.001 ^a	0.871 ± 0.001 ^{ab}
	30	0.081 ± 0.001 ^a	7.092 ± 0.004 ^a	0.086 ± 0.004 ^a	4.222 ± 0.002 ^{ab}	29.455 ± 0.057 ^{ab}	57.719 ± 0.038 ^{ab}	0.283 ± 0.006 ^{ab}	0.204 ± 0.001 ^a	0.862 ± 0.017 ^{ab}
FVEO (1.5 g/kg)	0	0.077 ± 0.003	6.517 ± 0.053	0.076 ± 0.003	3.744 ± 0.004	26.865 ± 0.019	61.540 ± 0.019	0.251 ± 0.009	0.252 ± 0.001	0.778 ± 0.001 ^b
	6	0.080 ± 0.001	6.938 ± 0.074 ^a	0.076 ± 0.001	3.839 ± 0.004 ^{ab}	27.315 ± 0.089 ^a	60.481 ± 0.084 ^{ab}	0.251 ± 0.001 ^b	0.245 ± 0.002 ^a	0.776 ± 0.004 ^b
	12	0.079 ± 0.001	6.907 ± 0.028 ^{ab}	0.079 ± 0.001	3.901 ± 0.006 ^a	27.816 ± 0.009 ^{ab}	60.052 ± 0.026 ^{ab}	0.261 ± 0.005 ^{ab}	0.218 ± 0.001 ^{ab}	0.790 ± 0.002 ^{ab}
	18	0.079 ± 0.003	6.987 ± 0.043 ^{ab}	0.076 ± 0.001	3.931 ± 0.028 ^{ab}	28.229 ± 0.154 ^{ab}	59.539 ± 0.083 ^{ab}	0.260 ± 0.001 ^{ab}	0.208 ± 0.001 ^a	0.794 ± 0.003 ^a
	24	0.078 ± 0.001	7.114 ± 0.002 ^{ab}	0.077 ± 0.001	3.973 ± 0.004 ^{ab}	28.629 ± 0.004 ^{ab}	58.938 ± 0.006 ^{ab}	0.265 ± 0.001 ^{ab}	0.202 ± 0.004 ^a	0.806 ± 0.001 ^a
	30	0.081 ± 0.001	7.528 ± 0.215 ^{ab}	0.078 ± 0.001	4.109 ± 0.030 ^{ab}	29.467 ± 0.056 ^{ab}	57.410 ± 0.125 ^{ab}	0.270 ± 0.003 ^{ab}	0.194 ± 0.001 ^{ab}	0.836 ± 0.001 ^a

^a represents a significant difference from the same set of data on day 0; ^b represents a significant difference from the data for the same frying time compared to blank; ^c C14:0, myristic acid; C16:0, palmitic acid; C16:1, palmitoleic acid; C18:0, stearic acid; C18:1n9, oleic acid; C18:2n6, linoleic acid; C20:0, arachidic acid; C18:3n3, α -linolenic acid; C22:0, behenic acid.

sample TBHQ-0.01, and the difference became more significant with increasing FVEO concentration. The change in the values of red and viscosity shows that the change in the sample of FVEO is similar to that of TBHQ-0.01. Most essential oils contain olefin double bonds and aromatic rings, which are capable of showing a certain color and can have an effect on their color when reacting with each other and frying oil. After the addition of FVEO, the components in the FVEO at high temperatures increase the polymers in the sunflower oil to some extent, which leads to an increase in its viscosity. Babiker *et al.* found that sunflower oil with rosemary essential oil and extract added separately during frying showed an increase in red and yellow values, while the samples with essential oil added increased more slowly than those with additive extracts³⁹⁾. It can be seen that the essential oil showed more advantages than the extracts when frying with sunflower oil.

3.5 Fatty acid changes in SFO

During the deep-frying process of SFO, the fatty acid composition would be affected by the oxidation reaction in sunflower oil⁴⁰⁾. The fatty acids on the triglycerides are constantly oxidized and changed. The changes in fatty acid

composition can be detected by methyl esterification, which reflects the changes in triglycerides in the oil⁴¹⁾. Table 2 shows that the fatty acid composition of all four groups of oil samples changed to a different level as the frying time increased. The fatty acids that changed significantly ($p < 0.05$) between the control and TBHQ samples were: stearic acid, oleic acid, linoleic acid and α -linolenic acid. As essential fatty acids, the changes in linoleic acid and α -linolenic acid have important research value. Based on the data in Table 2, a visual graph of the changes in linoleic and α -linolenic acids was obtained. It can be seen in Fig. 3 that the changes in linoleic and linolenic acids between the 1 g/kg oil group and the blank oil sample start to show significant differences ($p < 0.05$) at 18 hours and beyond, with a significant decrease in the 1.5 g/kg oil group. The complex and variable composition of the essential oil may be due to some of the heat intolerant components of the oil, which produce more oxidation products in the later stages of frying.

3.6 Sensory analysis of Chinese *Maye*

The aromatic compounds in the FVEO with some special fragrance will be transferred to the *Maye* during the frying

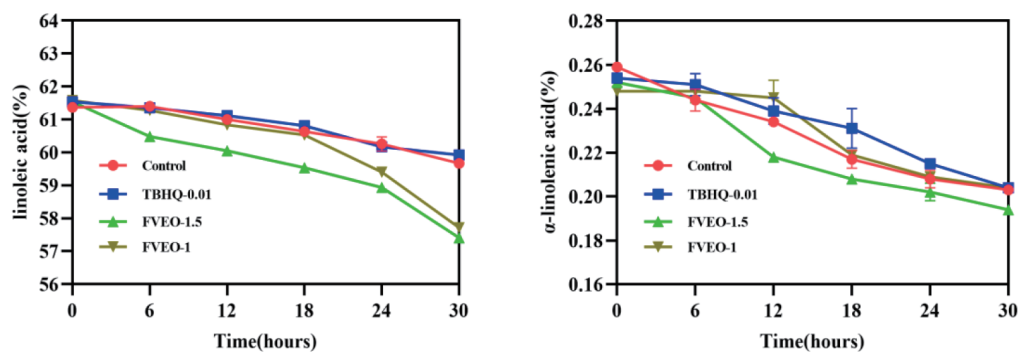


Fig. 3 The values for the linoleic acid, α -linolenic acid. Values are expressed as means $\pm$ SD ($n = 10$).

Table 3 Sensory evaluation score of Chinese *Maye*.

	Hour	Control	TBHQ	FVEO
Appearance	0	8.71 $\pm$ 0.82	8.71 $\pm$ 0.82	8.71 $\pm$ 0.82
	6	8.03 $\pm$ 0.74	8.27 $\pm$ 0.91	8.33 $\pm$ 0.73
	12	7.22 $\pm$ 0.68	7.43 $\pm$ 1.48	7.86 $\pm$ 0.94
	18	6.37 $\pm$ 0.84	6.12 $\pm$ 0.86	7.20 $\pm$ 1.02
	24	5.65 $\pm$ 0.75	5.80 $\pm$ 0.82	6.61 $\pm$ 0.69 ^b
	30	4.78 $\pm$ 0.77	5.54 $\pm$ 0.57 ^b	5.97 $\pm$ 0.96 ^b
Taste	0	8.85 $\pm$ 1.40	8.85 $\pm$ 1.40	8.85 $\pm$ 1.40
	6	8.13 $\pm$ 0.93	8.43 $\pm$ 0.91	8.45 $\pm$ 0.83
	12	7.25 $\pm$ 1.54	7.78 $\pm$ 0.84	8.23 $\pm$ 0.65
	18	6.47 $\pm$ 1.34	6.50 $\pm$ 0.76	7.84 $\pm$ 0.71 ^b
	24	5.49 $\pm$ 0.95	5.64 $\pm$ 0.53	6.43 $\pm$ 0.79 ^b
	30	4.84 $\pm$ 0.78	5.05 $\pm$ 1.11	5.82 $\pm$ 0.66 ^b
Flavor	0	8.64 $\pm$ 1.21	8.64 $\pm$ 1.21	8.64 $\pm$ 1.21
	6	8.15 $\pm$ 0.84	8.45 $\pm$ 0.65	8.29 $\pm$ 0.75
	12	7.22 $\pm$ 0.95	7.50 $\pm$ 0.83	7.60 $\pm$ 1.02
	18	6.26 $\pm$ 1.53	6.32 $\pm$ 0.66	7.24 $\pm$ 0.56
	24	5.43 $\pm$ 0.67	5.65 $\pm$ 1.21	6.61 $\pm$ 0.81 ^b
	30	4.21 $\pm$ 0.83	4.97 $\pm$ 0.65	6.15 $\pm$ 0.97 ^c
Overall acceptance	0	8.91 $\pm$ 1.04	8.91 $\pm$ 1.04	8.91 $\pm$ 1.04
	6	7.75 $\pm$ 0.96	7.85 $\pm$ 0.76	8.41 $\pm$ 0.90
	12	6.87 $\pm$ 0.89	6.46 $\pm$ 1.41	7.86 $\pm$ 0.68 ^b
	18	5.28 $\pm$ 0.90	5.78 $\pm$ 0.66	7.42 $\pm$ 1.04 ^c
	24	4.09 $\pm$ 1.22	4.63 $\pm$ 0.70	6.81 $\pm$ 0.63 ^c
	30	3.30 $\pm$ 0.83	4.11 $\pm$ 0.46	6.09 $\pm$ 0.82 ^c

^b represents a significant difference level of $p < 0.05$ from the same group of data on day 0

^c represents a significant difference level of $p < 0.01$

time, thus enhancing the sensory qualities of Chinese *Maye*. The most critical thing to evaluate whether a fried product is suitable for the market is the sensory evaluation of the fried product. If the product does not appeal to con-

sumers, it will be rejected⁴²⁾. As shown in Table 3, the appearance, taste, flavor and overall acceptance of Chinese *Maye* in the SFO sample gradually decreased with increasing frying time. Both TBHQ and FVEO inhibited the de-

crease of sensory quality of Chinese *Maye*. As the frying time increased, the sensory scores of the SFO samples of FVEO were higher than that of the SFO sample of TBHQ. There were significant differences between the FVEO group and the control group in taste and acceptance scores at the late frying stage ($p < 0.01$). At the same time, there was no significant difference in these attributes between the SFO sample of TBHQ and the control sample. The results indicate that the direct addition of FVEO to vegetable oils was considered to be an efficient way to improve the sensory properties of deep-fried products, which is similar to the results of previous studies⁹⁾.

4 Conclusion

In the current study, the antioxidant effect of FVEO was investigated by adding FVEO to SFO during high-temperature frying of Chinese *Maye*, and sensory evaluation of Chinese *Maye* after frying was conducted. The results showed that the antioxidant effect of FVEO-1 g/kg on SFO was similar to that of TBHQ-0.01 g/kg, and FVEO-1.5 g/kg would promote the oxidation of SFO. The addition of FVEO also significantly improved various sensory attributes of Chinese *Maye*, such as appearance, taste, flavor and overall acceptability. The main antioxidant components in FVEO are currently being investigated in our laboratory and can be developed as a natural antioxidant once the main antioxidant components in FVEO are identified and their safety is established through toxicological tests.

Acknowledgments

This work was financially supported by National Natural Science Foundation of China (32001744).

Data Availability Statement

Research data are not shared.

Conflicts of Interest

The authors indicate no conflict of interest.

References

- 1) Yu, K.S.; Cho, H.; Hwang, K.T. Physicochemical properties and oxidative stability of frying oils during repeated frying of potato chips. *Food Sci. Biotechnol.* **27**, 651-659 (2018).
- 2) Romanić, R.; Lužaić, T.; Grgić, K. Examining the possibility of improving the properties of sunflower oil in order to obtain a better medium for the process of frying food. *The 1st International Electronic Conference on Food Science and Functional Foods* (2020).
- 3) Morales, G.; Jimenez, M.; Garcia, O.; Mendoza, M.R.; Beristain, C.I. Effect of natural extracts on the formation of acrylamide in fried potatoes. *LWT - Food Sci. Technol.* **58**, 587-593 (2014).
- 4) Gupta, M.K.J.J.W.; Sons Inc. N.J. Frying Oils, Bailey's Industrial Oil and Fat Products. Kaivan Bahmani; Ali Izadi Darbandi; Hossein Ali Ramshini; Narges Moradi; Akbari, A. Agro-morphological and phytochemical diversity of various Iranian fennel landraces. *Ind. Crops Prod.* **77**, 282-294 (2015).
- 5) Man, Y.B.C.; Jaswir, I. Effect of rosemary and sage extracts on frying performance of refined, bleached and deodorized (RBD) palm olein during deep-fat frying. *Food Chem.* **69**, 301-307 (2000).
- 6) Wang, D.; Chen, X.; Wang, Q.; Meng, Y.; Wang, D.; Wang, X. Influence of the essential oil of *Mentha spicata* cv. Henanshixiang on sunflower oil during the deep-frying of Chinese *Maye*. *LWT - Food Sci. Technol.* **122**, 109020 (2020).
- 7) Majhenić, L.; Škerget, M.; Knez, Ž. Antioxidant and antimicrobial activity of guarana seed extracts. *Food Chem.* **104**, 1258-1268 (2007).
- 8) Caponio, F.; Durante, V.; Varva, G.; Silletti, R.; Previtali, M.A. *et al.* Effect of infusion of spices into the oil vs. combined malaxation of olive paste and spices on quality of naturally flavoured virgin olive oils. *Food Chem.* **202**, 221-228 (2016).
- 9) Cocan, I.; Negrea, M.; Cozma, A.; Alexa, E.; Poiana, M.-A. *et al.* Chili and sweet pepper seed oil used as a natural antioxidant to improve the thermo-oxidative stability of sunflower oil. *Agronomy* **11**, 2579 (2021).
- 10) Servi, H.; Sen, A.; Dogan, A. Chemical composition and biological activities of endemic *Tripleurospermum conoclinium* (Boiss. & Balansa) Hayek essential oils. *Flavour Frag. J.* **35**, 713-721 (2020).
- 11) Servî-Ali ŞEn-Esra Yildirim Servî, H. Chemical composition and biological activities of essential oils of *Foeniculum vulgare* Mill. and *Daucus carota* L. growing wild in Turkey. *J. Res. Pharm.* **25**, 142-152 (2021).
- 12) Gulfraz, M.; Mehmood, S.; Minhas, N.; Jabeen, N.; Kausar, R. *et al.* Composition and antimicrobial properties of essential oil of *Foeniculum vulgare*. *Afr. J. Biotechnol.* **7**, 4364-4368 (2008).
- 13) Abou El-Soud, N.; El-Laithy, N.; El-Saeed, G.; Wahby, M.; Khalil, M. *et al.* Antidiabetic activities of *Foeniculum vulgare* Mill. essential oil in streptozotocin-induced diabetic rats. *Maced. J. Med. Sci.* **4**, 139-146 (2011).

- 14) Chang, S.; Mohammadi Nafchi, A.; Karim, A.A. Chemical composition, antioxidant activity and antimicrobial properties of three selected varieties of Iranian fennel seeds. *J. Essent. Oil Res.* **28**, 357-363 (2016).
- 15) Choi, E.-M.; Hwang, J.-K. Antiinflammatory, analgesic and antioxidant activities of the fruit of *Foeniculum vulgare*. *Fitoterapia* **75**, 557-565 (2004).
- 16) Khammassi, M.; Loupassaki, S.; Tazarki, H.; Mezni, F.; Slama, A. *et al.* Variation in essential oil composition and biological activities of *Foeniculum vulgare* Mill. populations growing widely in Tunisia. *J. Food Biochem.* **42**, 12532 (2018).
- 17) Diao, W.-R.; Hu, Q.-P.; Zhang, H.; Xu, J.-G. Chemical composition, antibacterial activity and mechanism of action of essential oil from seeds of fennel (*Foeniculum vulgare* Mill.). *Food Control* **35**, 109-116 (2014).
- 18) Rather, M.A.; Dar, B.A.; Sofi, S.N.; Bhat, B.A.; Qurishi, M.A. *Foeniculum vulgare*: A comprehensive review of its traditional use, phytochemistry, pharmacology, and safety. *Arab. J. Chem.* **9**, 1574-1583 (2016).
- 19) Cetin, B.; Ozer, H.; Cakir, A.; Polat, T.; Dursun, A. *et al.* Antimicrobial activities of essential oil and hexane extract of florence fennel [*Foeniculum vulgare* var. *azoricum* (Mill.) Thell.] against foodborne microorganisms. *J. Med. Food* **13**, 196-204 (2010).
- 20) Bammou, M.; Bouhlali, E.D.T.; Sellam, K.; Ramchoun, M.; McDonald, A.G. *et al.* Investigation on chemical composition, antioxidant and antimicrobial potential of *Pulicaria mauritanica* essential oil applied by direct addition or vapor contact. *Am. J. Essent. Oil.* **7**, 07-13 (2019).
- 21) Abdellaoui, M.; Bouhlali, E.d.T.; Derouich, M.; El-Rhaf-fari, L. Essential oil and chemical composition of wild and cultivated fennel (*Foeniculum vulgare* Mill.): A comparative study. *S. Afr. J. Bot.* **135**, 93-100 (2020).
- 22) Karik, Ü.; Demirbolat, I. Chemical composition, antioxidant and antimicrobial activities of *Pimpinella en-guezekensis*: A novel species from Anatolia, Turkey-fruit essential oil. *J. Essent. Oil-Bear. Plants* **23**, 356-362 (2020).
- 23) Xu, B.J.; Chang, S.K. A comparative study on phenolic profiles and antioxidant activities of legumes as affected by extraction solvents. *J. Food Sci.* **72**, 159-166 (2007).
- 24) Xu, J.G.; Hu, Q.P.; Liu, Y. Antioxidant and DNA-protective activities of chlorogenic acid isomers. *J. Agric. Food Chem.* **60**, 11625-11630 (2012).
- 25) Abdellaoui, M.; Bouhlali, E.d.T.; Derouich, M.; El-Rhaf-fari, L. Essential oil and chemical composition of wild and cultivated fennel (*Foeniculum vulgare* Mill.): A comparative study. *S. Afr. J. Bot.* **135**, 93-100 (2020).
- 26) Yuan, R.; Zhang, D.; Yang, J.; Wu, Z.; Luo, C. *et al.* Review of aromatherapy essential oils and their mechanism of action against migraines. *J. Ethnopharmacol.* **265**, 113326 (2021).
- 27) Aburjai, T.; Natsheh, F.M. Plants used in cosmetics. *Phytother. Res.* **17**, 987-1000 (2003).
- 28) Napoli, E.M.; Curcuruto, G.; Ruberto, G. Screening the essential oil composition of wild Sicilian fennel. *Biochem. Syst. Ecol.* **38**, 213-223 (2010).
- 29) Shahat, A.A.; Ibrahim, A.Y.; Hendawy, S.F.; Omer, E.A.; Hammouda, F.M. *et al.* Chemical composition, antimicrobial and antioxidant activities of essential oils from organically cultivated fennel cultivars. *Molecules* **16**, 1366-1377 (2011).
- 30) Afkhami, F.; Zare, N. Phytochemical composition and cytotoxic effects of essential Oil of Inflorescences of *Achillea santolina* L. Growing in Iran. *J. Essent. Oil-Bear. Plants* **24**, 1291-1299 (2021).
- 31) Karik, U.; Demirbolat, I.; Toluk, Ö.; Kartal, M. Comparative study on yields, chemical compositions, antioxidant and antimicrobial activities of cumin (*Cuminum cyminum* L.) seed essential oils from different geographic origins. *J. Essent. Oil-Bear. Plants* **24**, 724-735 (2021).
- 32) Wang, H.V.; Pickett, L.J.; Faraone, N. Repellent and acaricidal activities of basil (*Ocimum basilicum*) essential oils and rock dust against *Ixodes scapularis* and *Dermacentor variabilis* ticks. *Exp. Appl. Acarol.* **86**, 583-598 (2022).
- 33) Zhang, X.; Yu, X.; Shi, Y.; Zhao, X.; Xing, M. *et al.* *Chrysanthemum morifolium* cv. Hang-ju leaves: An abundant source of preservatives for food industry. *Eur. Food Res. Technol.* **246**, 939-946 (2020).
- 34) Li, X.; Li, J.; Wang, Y.; Cao, P.; Liu, Y. Effects of frying oils' fatty acids profile on the formation of polar lipids components and their retention in French fries over deep-frying process. *Food Chem.* **237**, 98-105 (2017).
- 35) Angelo, P.M.; Jorge, N. Antioxidant evaluation of coriander extract and ascorbyl palmitate in sunflower oil under thermoxidation. *J. Am. Oil Chem. Soc.* **85**, 1045-1049 (2008).
- 36) Blasi, F.; Rocchetti, G.; Montesano, D.; Lucini, L.; Chi-odelli, G. *et al.* Changes in extra-virgin olive oil added with *Lycium barbarum* L. carotenoids during frying: Chemical analyses and metabolomic approach. *Food Res. Int.* **105**, 507-516 (2018).
- 37) Keramat, M.; Golmakani, M.-T.; Aminlari, M.; Shekarforoush, S. Oxidative stability of virgin olive oil supplemented with *Zataria multiflora* Boiss. and *Rosmarinus officinalis* L. essential oils during accelerated storage. *J. Food Process. Preserv.* **41**, e12951 (2017).
- 38) Montesano, D.; Rocchetti, G.; Cossignani, L.; Senizza, B.; Pollini, L. *et al.* Untargeted metabolomics to evaluate the stability of extra-virgin olive oil with Added *Lycium barbarum* Carotenoids during Storage. *Foods* **8**, 179 (2019).
- 39) Babiker, E.E.; Al-Juhaimi, F.Y.; Tannriverdi, E.S.; Özcan,

- M.M.; Ahmed, I.A.M.; Ghafoor, K. Effect of rosemary extracts on stability of sunflower oil used in frying. *J. Oleo Sci.* **69**, 985-992 (2020).
- 40) Wang, D.; Fan, W.; Guan, Y.; Huang, H.; Yi, T.; Ji, J. Oxidative stability of sunflower oil flavored by essential oil from *Coriandrum sativum* L. during accelerated storage. *LWT - Food Sci. Technol.* **98**, 268-275 (2018).
- 41) Rodriguez, G.; Squeo, G.; Estivi, L.; Quezada Berru, S.; Buleje, D. *et al.* Changes in stability, tocopherols, fatty acids and antioxidant capacity of sachu inchi (*Plukenetia volubilis*) oil during French fries deep-frying. *Food Chem.* **340**, 127942 (2021).
- 42) Taha, E.; Abouelhawa, S.; El-Geddawy, M.; Sorour, M.; Aladedunye, F.; Matthäus, B. Stabilization of refined rapeseed oil during deep-fat frying by selected herbs. *Eur. J. Lipid Sci. Technol.* **116**, 771-779 (2014).

CC BY 4.0 (Attribution 4.0 International). This license allows users to share and adapt an article, even commercially, as long as appropriate credit is given. That is, this license lets others copy, distribute, remix, and build upon the Article, even commercially, provided the original source and Authors are credited.

