

The degradation of some volatile compounds in evening primrose oil under UV/TiO₂ process

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Submitted: 25 September 2022; Accepted: 16 November 2022; Published online: 10 October 2023

SUMMARY: Off-flavor is one of the limiting factors in the quality and commercial acceptability of evening primrose oil (EPO). The results of this study demonstrated that ultraviolet light irradiated with titanium dioxide (UV/TiO₂) was able to effectively reduce odorous aldehyde concentrations, which would produce undesired flavors. Specifically, reductions in the E-2-Decenal, 1-octen-3-ol and hexanoic acid in EPO reached 50, 75.2 and 61.4% after a UV/TiO₂ process of 5 min, respectively. The odor active values (OAV) and hierarchical cluster analysis (HCA) showed that the result of the 5 min group was similar to that of the original oil. In addition, the physicochemical characteristics of EPO after processing did not change significantly. The result of the aroma profile analysis was consistent with the OAV and HCA results. Therefore, it has been concluded that 5 min UV/TiO₂ treatment could degrade some volatile compounds and provide a potential deodorization method for industry.

KEYWORDS: Evening primrose oil; Fatty acid composition; Volatile components; UV/TiO₂.

RESUMEN: Degradación de algunos compuestos volátiles de aceites de onagra bajo un proceso UV/TiO₂. El mal sabor es uno de los factores limitantes de la calidad y aceptabilidad comercial del aceite de onagra (EPO). Los resultados de este estudio demostraron que la luz ultravioleta irradiada con dióxido de titanio (UV/TiO₂) fue capaz de reducir eficazmente las concentraciones de aldehídos volátiles, que darían olores no deseados. En concreto, la reducción del E-2-Decenal, 1-octen-3-ol y el ácido hexanoico en EPO alcanzó el 50%, 75,2% y 61,4% respectivamente tras un proceso UV/TiO₂ de 5 min. Los valores de olor activo (OAV) y el análisis de conglomerados jerárquicos (HCA) mostraron que el resultado del grupo de 5 min fue similar al del aceite original. Además, las características fisicoquímicas del EPO después del procesamiento no cambiaron significativamente. El resultado del análisis del perfil de aroma fue consistente con nuestro resultado de OAV y HCA. Por lo tanto, se ha llegado a la conclusión de que el tratamiento con UV/TiO₂ durante 5 min podría degradar algunos compuestos volátiles y proporcionar un método de desodorización potencial para la industria.

PALABRAS CLAVE: Aceite de onagra; Componentes volátiles; Composición de ácidos grasos; Ultravioleta/TiO₂.

Citation/Cómo citar este artículo: Pan FG, Chen XM, Yang EQ, Liu BQ. 2023. The degradation of some volatile compounds in evening primrose oil under UV/TiO₂ process. *Grasas Aceites* 74 (3), e514. <https://doi.org/10.3989/gya.0992221>

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1. INTRODUCTION

Evening Primrose Oil (EPO) is obtained from the seeds of evening primrose by the cold-pressed process. Due to its high content in unsaturated fatty acids, especially γ -linolenic acid, it is widely consumed and used to relieve the effects of dermatitis, psoriasis, premenstrual and menopausal syndromes (de Santana *et al.*, 2019). However, these unsaturated fatty acids are also easily oxidated and some oxidation products with low molecular weight are formed. Among the complex production, some compounds could directly influence the overall flavor of EPO and contribute to off-flavor. As a consequence, there is a decline in commercial acceptability.

TiO₂ photocatalysis is considered a very promising technology due to its environmental protection, high antibacterial activity, low toxicity, and low cost. Hence, its application has been steadily growing in the food industry (Rashid *et al.*, 2021). One of these technologies is TiO₂ photocatalysis under UV, which is always used for the decomposition of organic compounds (Fonseca *et al.*, 2021). Zhang *et al.* (2016) have established a nanocomposite based on TiO₂, which showed excellent ability to degrade acetone in air. Similarly, the combination of UV and H₂O₂ successfully removed organic chemicals from waste water (Miklos *et al.*, 2018). However, the oxidation of fatty acids should be taken into consideration when applying UV to oil compared to the aqueous system (Sun *et al.*, 2019). As early as 2006, Luna *et al.* (2006) discovered that UV irradiation would cause virgin olive oil oxidation, and the concentration of volatile compounds responsible for the rancid

sensory increased. However, a similar result was not found in essential oil. Li *et al.* (2016) demonstrated some aldehydes in citrus essential oil were degraded after UV irradiation. In addition, UV/TiO₂ was used to remove aflatoxins which were harmful to humans from oil. Researchers used UV radiation combined with TiO₂ to remove aflatoxins from peanut oil and corn oil. They found that the effect was satisfactory, and the physicochemical characteristics of the oil were not significantly affected (Magzoub *et al.*, 2019; Sun *et al.*, 2019). Therefore, it is necessary to explore the changes in the volatile compounds in EPO due to the UV/TiO₂ process and to evaluate the impact on oil quality.

In recent years, headspace solid-phase microextraction-mass spectrometry (HS-SPME-GC-MS) was widely developed in the analysis of volatile compounds. It was useful to detect the composition and concentration of volatile compounds in the sample (Song *et al.*, 2022). However, the intensity of the fragrance is not only related to the concentration, but also the threshold. Thus, the odor activity value (OAV) was calculated to indicate the importance of the odor in the overall flavor. Generally speaking, the larger the OAV, the greater the effect it has. Fang *et al.* (2022) evaluated the effect of the deodorization of oil after the nanofiltration membrane process by OAV analysis. In addition to the methods described above, a cluster heat map was also used as an important tool for the analysis of large amounts of data.

A study from our previous investigation has established the volatile compounds of original evening promise oil (Pan *et al.*, 2022). As shown in Figure 1,

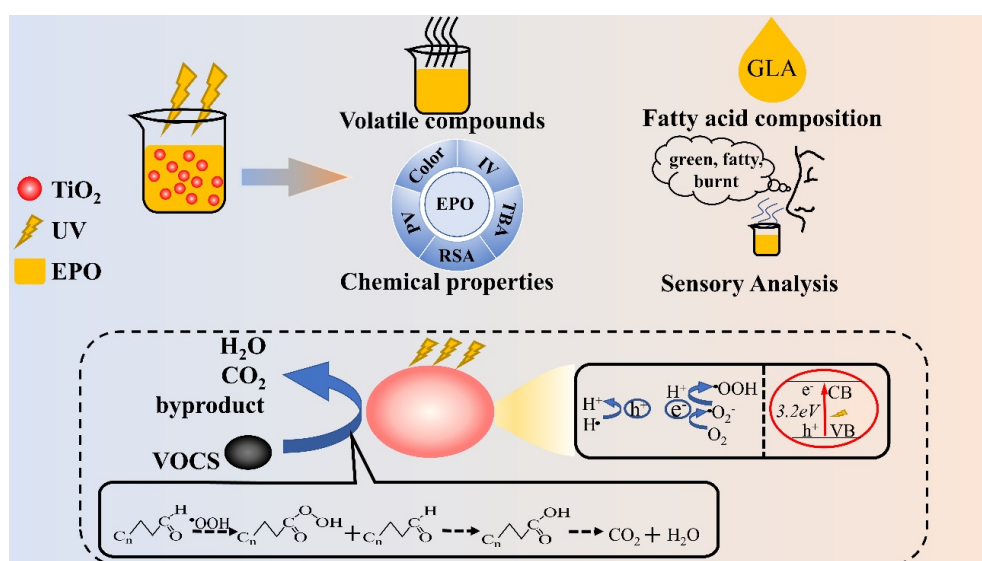


FIGURE 1. Schematic diagram of the study on the influence of UV/TiO₂ process in EPO.

this study aimed to explore the volatile compounds of EPO subjected to UV/TiO₂ irradiated at different times (0, 5, 15, 30, 45, 60 min), separately. A sensory evaluation was conducted to compare the aroma characteristics of EPO before and after treatment. The relationship between the volatile compounds and the irradiation time was also revealed. In addition, the chemical properties of EPO were discussed.

2. MATERIALS AND METHODS

2.1. Oil sample preparation

Evening primrose oil came from the Changchun Baili biotech company (Changchun, Jilin province, China). TiO₂ nanopowder (P25) was commercially obtained from the Benze Reagent company, manufactured in Germany. All chemical reagents, including solvents, were of analytical grade and purchased from local suppliers.

Before irradiation, EPO (10 g) and TiO₂ (10 mg) were stirred in the dark for 30 min to achieve equilibrium. The samples (10 g EPO stirred with 10 mg TiO₂) were placed under ultraviolet light (254 nm) for 5, 15, 30, 45, and 60 minutes, and stored at 4 °C.

2.2. The identification of volatile compounds

The extraction of volatile compounds in EPO was carried out as described by Wang *et al.* (2022) with slight modifications. The volatile compounds of the irradiated and control samples were extracted with 1 cm of Divinylbenzene / Carboxen / Polydimethylsiloxane (DVB / CAR / PDMS) SPME fiber (50/30 μm), (grey, Supelco, USA), and then analyzed by GC-MS (GC-MS-QP2020, Shimadzu, Kyoto, Japan). According to the manufacturer's suggestion, the fiber was heated before use at 250 °C in the gas chromatographic injection port for 30 minutes. The sample (2.5 g) was evenly distributed in a 15-mL headspace vial, and then 1 μL of 2-methyl-3-Heptanone (14 μg/μL in n-hexane) was added as internal standard. The sample was equilibrated at 50 °C for 10 minutes, then adsorbed by fiber at 50 °C for 30 minutes, and finally thermally adsorbed into the GC-MS at 250 °C for 5 minutes.

2.3. Qualitative and quantitative analysis of volatile compounds

The relative concentration of volatile compounds in the evening primrose oil samples was quantita-

tively calculated based on internal standards. The detected compounds were identified by comparing the mass spectra obtained from the NIST17 library.

OAV is equal to the ratio of compound concentration (ci) to the odor threshold (oti). Threshold intensity in the oil was found in the book and the compounds with OAV ≥ 1 were generally considered to contribute greatly to aromatic volatile compounds (Fang *et al.*, 2022).

2.4. Determination of the fatty acid composition in oil

The fatty acid composition of the samples was determined by GC-MS, and area normalization was used to quantify the fatty acid. The results were expressed as a percentage of the relative peak area. The method was based on our previous study with slight modifications (Pan *et al.*, 2020).

2.5. Radical scavenging activity

The radical scavenging capacity (RSA) of EPO was measured according to the methods applied in the previous study with slight modifications (Szydłowska-Czerniak and Łaszewska, 2015). Briefly, the sample was extracted with methanol, and the absorbance of the obtained solutions was measured using a UV-Vis spectrophotometer and automated microplate reader (Bio TEK, USA). The radical scavenging activity was estimated by the following equations:

$$\text{Radical scavenging capacity (\%)} = \frac{A_c - A_s}{A_c} \times 100\%$$

where A_s and A_c represent the absorbance of the oil samples and control group, respectively.

2.6. Chemical properties

The peroxide value (PV) and iodine value (IV) of EPO were determined by the procedure of the AOCS (2003) Official Method. Thiobarbituric acid (TBA) value was evaluated according to Baghdadi *et al.* (2019). The color parameters (L, a* and b* values) of EPO were determined using a Chroma Meter (CR-400, Konica Minolta, Japan) at 25 °C.

2.7. Sensory analysis

Trained panelists (5 females and 5 males between the ages of 20–35 years) with experience in sensory analysis of oil were selected from Jilin Universi-

ty to evaluate the intensity of odor attributes in the samples. Prior to analysis, several aroma qualities were selected by a descriptive test. The intensities of selected odor attributes (green, fruity, fatty, vinegar-like, burnt) were rated using a linear scale from 0 (not perceivable) to 5 (strong, high intensity) on a five-point scale in steps of 0.5. Each sample was analyzed three times by the panelist, and the data was expressed as the mean. Adequate time was provided between the different samples to avoid tiredness on the part of the panelists. The test was carried out at a comfortable temperature (25 °C) and the samples were poured into the glasses for further analysis.

2.8. Data analysis

All experiments were carried out in triplicate and the results are given as mean \pm standard deviation. Variance analysis was executed using SPSS Statistics software v.21.0 (SPSS Inc., Chicago, IL, USA) with a p value < 0.05 .

3. RESULTS AND DISCUSSION

3.1. Analysis of volatile compounds in evening primrose oil

The previous report confirmed that the increase in temperature has a negative effect on oil quality, which will lead to the autoxidation and the decomposition of hydroperoxides, and generate undesired flavors (Bao *et al.*, 2022). To avoid raising the temperature during irradiation, an ice bath was used to make the experiment work at a lower temperature. As the results in Table 1 show, a total of 56 dominant odorants (identified by adding 2-methyl-3-heptanone as internal standard) were extracted from our samples by fiber and identified by the machine. They can be classified into seven chemical categories according to different properties, including 7 alcohols, 18 aldehydes, 4 ketones, 8 acids, 4 esters, 14 hydrocarbons and 1 other compound. Among them, hydrocarbons were the most abundant volatile compound in all samples, followed by aldehydes and alcohols. However, due to the high odor thresholds (Zhang *et al.*, 2021), hydrocarbons were considered inadequate aroma contributors for our samples despite having the highest content determined in the EPOs. The alcohols and aldehydes were formed by the decomposition of fatty acids. The analysis of our study confirms again that the aldehydes, which are

considered to have contributed to the overall odor in a large quantity of edible oil, such as Camellia oleifera Seed Oil, virgin olive oil, cold-pressed peanut oil (Angerosa *et al.*, 2004), are also major compounds in EPO. It is worth noting that the aldehydes, which provide a typical grassy and fatty odor in oil, may be harmful to the sensory quality of the product. Esters were produced by the esterification of alcohols with free small molecular fatty acids, generally described as fruit and flower aromas (Zhang *et al.*, 2021). They were also detected in our sample and contributed to a positive odor in EPO. Besides the above four kinds of components, the remaining substances also play a role in the overall flavor. They interact with and influence each other and constitute the complicated aroma profile of EPO.

The evolution of key VOCs mainly affected by the irradiation of the EPO is shown in Figure 2. E-2-Decenal, 1-octen-3-ol and hexanoic acid showed very similar trends with diverse amounts. They rapidly decreased under 5 min irradiation and then reached their maximum concentration after 60 min irradiation. (E, E)-2,4-decadienal and 3-methyl-1-pentanol showed similar behavior in that they initially decreased and then increased with the increase in UV/TiO₂ pretreatment time, and reached the lowest concentration at 30 min, which means that the decomposition capacity of UV/TiO₂ was different for each molecule. In addition, the concentration of E-2-octenal, (E, E)-2,4-Nonadienal and nonanal decreased after the UV/TiO₂ irradiation. Therefore, UV/TiO₂ treatment for a short time, less than 30 min, was considered beneficial for EPO flavor. In fact, nonanal was an important odorant in oil, including olive oil and soybean oil, derived from the autoxidation of oleic acid, and was previously considered to be a suitable index of the degree of oxidation (Vichi *et al.*, 2003). It showed very low amounts, approximately 0.05 mg/kg in EPO with 60 min UV irradiation. As already pointed out in the literature, (E)-2-Octenal was detected in the deep-frying process and suggested to be responsible for the rancid defect (Multari *et al.*, 2019). In our study, the concentration of (E)-2-Octenal in the sample of 60 min decreased by approximately 5 times compared to the original sample. Hexanal showed a similar amount in EPO at 0, 5, and 15 min and a significant decrease over time. Heptanal exhibited a different behavior from hexanal. It increased significantly after 30, 45,

TABLE 1. Identification of volatile compounds extracted by HS-SPME

Nr	Compounds	Identification method	Concentration (mg/kg)					
			0 min	5 min	15 min	30 min	45 min	60 min
Alcohol								
1	(E)-2-Octenol	MS	0.13±0.04 ^a	0.02±0.00 ^a	0.06±0.03 ^a	0.04±0.01 ^a	0.03±0.00 ^a	0.06±0.04 ^a
2	(Z, Z)-9,12-Octadecadien-1-ol	MS	ND	0.04±0.00 ^a	0.02±0.01 ^c	0.01±0.01 ^c	0.02±0.01 ^c	0.03±0.01 ^b
3	2-ethyl-1-Hexanol	MS	0.31±0.06 ^b	0.28±0.10 ^b	0.32±0.07 ^b	0.24±0.05 ^b	0.25±0.07 ^b	0.47±0.08 ^a
4	2-Propanol	MS	0.03±0.01 ^a	0.02±0.01 ^{ab}	0.01±0.01 ^{ab}	ND	0.00±0.01 ^b	0.03±0.01 ^a
5	1-Octen-3-ol	MS	1.25±0.03 ^a	0.31±0.04 ^c	0.48±0.06 ^d	0.77±0.04 ^c	0.82±0.08 ^c	1.00±0.08 ^b
6	3-methyl-1-Pentanol	MS	0.26±0.04 ^a	0.22±0.05 ^{ab}	0.16±0.10 ^{ab}	0.14±0.04 ^b	0.22±0.05 ^{ab}	0.21±0.03 ^{ab}
7	1-butanol	MS	2.98±0.61 ^a	2.73±0.37 ^a	2.56±0.24 ^a	2.67±0.13 ^a	2.81±0.18 ^a	2.92±0.14 ^a
Aldehydes								
8	(E)-2-Heptenal	MS	0.20±0.07 ^a	0.25±0.12 ^a	0.16±0.05 ^a	0.17±0.03 ^a	0.19±0.03 ^a	0.29±0.04 ^a
9	Nonanal	MS	0.81±0.06 ^a	0.35±0.11 ^b	0.21±0.05 ^c	0.26±0.05 ^{bc}	0.09±0.06 ^d	0.05±0.01 ^d
10	Hexanal	MS	0.82±0.26 ^{ab}	0.75±0.03 ^{ab}	0.89±0.14 ^a	0.76±0.04 ^{ab}	0.60±0.08 ^b	0.59±0.10 ^b
11	(E)-2-Nonenal	MS	0.11±0.02 ^{ab}	0.05±0.01 ^{dc}	0.15±0.05 ^a	0.05±0.04 ^{bc}	0.04±0.01 ^c	0.10±0.04 ^{ab}
12	Decanal	MS	ND	ND	0.01±0.01 ^a	ND	ND	0.01±0.01 ^a
13	(E, E)-2,4-Dodecadienal	MS	0.11±0.04 ^b	0.19±0.04 ^a	0.11±0.02 ^b	0.15±0.03 ^{ab}	0.13±0.03 ^{ab}	0.13±0.03 ^{ab}
14	(E, E)-2,4-Decadienal	MS	0.65±0.02 ^a	0.33±0.09 ^{bc}	0.26±0.05 ^{bc}	0.21±0.02 ^c	0.24±0.04 ^{bc}	0.42±0.01 ^b
15	(E)-2-Octenal	MS	1.24±0.10 ^a	0.41±0.18 ^b	0.22±0.05 ^{bc}	0.28±0.07 ^{bc}	0.17±0.02 ^c	0.24±0.11 ^{bc}
16	2,5-Dihydroxybenzaldehyde	MS	ND	0.42±0.09 ^a	0.02±0.01 ^b	0.02±0.01 ^b	0.01±0.00 ^b	0.02±0.01 ^b
17	Octanal	MS	1.93±0.15 ^a	1.61±0.33 ^a	1.65±0.26 ^a	1.72±0.31 ^a	1.63±0.11 ^a	1.78±0.28 ^a
18	(Z)-9,17-Octadecadienal	MS	1.35±0.37 ^{ab}	1.76±0.23 ^a	0.98±0.13 ^b	1.23±0.65 ^{ab}	1.59±0.39 ^{ab}	1.22±0.22 ^{ab}
19	Dodecanal	MS	0.70±0.05 ^b	0.81±0.18 ^b	1.14±0.36 ^b	1.71±0.37 ^a	1.61±0.26 ^a	1.64±0.28 ^a
20	(E)-4-Nonenal	MS	ND	ND	0.01±0.00 ^b	0.01±0.01 ^a	ND	ND
21	Pentanal	MS	0.6±0.15 ^a	0.89±0.16 ^a	0.65±0.13 ^a	0.63±0.12 ^a	0.60±0.29 ^a	0.76±0.16 ^a
22	Heptanal	MS	0.03±0.03 ^b	0.02±0.00 ^b	0.02±0.00 ^b	0.07±0.02 ^a	0.05±0.00 ^{ab}	0.05±0.01 ^{ab}
23	(E, E)-2,4-Nonadienal	MS	0.07±0.05 ^a	0.03±0.02 ^{ab}	0.01±0.01 ^b	0.03±0.01 ^{ab}	0.02±0.01 ^{ab}	0.04±0.01 ^{ab}
24	(E)-2-Decenal	MS	0.06±0.03 ^{bc}	0.03±0.01 ^c	0.11±0.04 ^{bc}	0.15±0.06 ^{ab}	0.12±0.02 ^{ab}	0.20±0.06 ^a
25	(E, Z)-2,4-Decadienal	MS	0.31±0.07 ^d	0.35±0.10 ^d	0.52±0.11 ^{cd}	0.63±0.12 ^{bc}	0.77±0.12 ^b	1.02±0.23 ^a
Ketone								
26	5-Tridecanone	MS	0.16±0.03 ^a	0.15±0.04 ^a	0.14±0.06 ^a	0.15±0.03 ^a	0.18±0.03 ^a	0.21±0.03 ^a
27	Elsholtzia ketone	MS	ND	0.04±0.03 ^b	0.02±0.02 ^a	0.03±0.01 ^b	0.04±0.01 ^b	0.06±0.01 ^a
28	3-Octen-2-one	MS	0.05±0.02 ^c	0.27±0.11 ^{ab}	0.34±0.10 ^a	0.28±0.05 ^{ab}	0.14±0.14 ^{bc}	0.29±0.09 ^{ab}
29	1-Nonen-3-one	MS	0.12±0.07 ^{ab}	0.22±0.09 ^a	0.08±0.05 ^b	0.15±0.02 ^a	0.16±0.03 ^{ab}	0.20±0.01 ^a
Acid								
30	2-Hexenoic acid	MS	0.06±0.06 ^a	0.03±0.05 ^a	0.03±0.04 ^a	0.04±0.01 ^a	0.19±0.25 ^a	0.03±0.01 ^a
31	2-Octynoic acid	MS	0.34±0.28 ^a	0.40±0.05 ^a	0.30±0.06 ^a	0.32±0.09 ^a	0.38±0.08 ^a	0.36±0.05 ^a
32	Acetic acid	MS	0.03±0.03 ^a	ND	ND	ND	ND	ND
33	Heptanoic acid	MS	0.13±0.07 ^a	0.17±0.09 ^a	0.22±0.04 ^a	0.13±0.01 ^a	0.12±0.06 ^a	0.16±0.02 ^a
34	Hexanoic acid	MS	1.27±0.48 ^a	0.49±0.20 ^b	0.59±0.15 ^{ab}	0.62±0.09 ^{ab}	0.78±0.13 ^{ab}	0.95±0.11 ^{ab}
35	(Z, Z)-9,12-Octadecadienoic acid	MS	ND	0.07±0.02 ^b	0.02±0.02 ^c	0.06±0.01 ^b	0.08±0.03 ^b	0.12±0.01 ^a
36	Octadecanoic acid	MS	0.03±0.04 ^a	ND	ND	ND	ND	ND
37	(E)-2-Hexenoic acid	MS	ND	0.05±0.03 ^a	0.03±0.01 ^a	0.05±0.01 ^b	0.09±0.06 ^a	0.05±0.02 ^a
Ester								
38	Docosanoic acid, docosyl ester	MS	0.40±0.06 ^{ab}	0.43±0.13 ^a	0.28±0.10 ^{ab}	0.31±0.04 ^{ab}	0.17±0.04 ^b	0.18±0.08 ^b
39	9,12-Octadecadienoic acid (Z, Z)-methyl ester	MS	1.23±0.24 ^{ab}	1.49±0.49 ^a	0.46±0.12 ^c	0.82±0.30 ^{bc}	1.30±0.27 ^{ab}	1.45±0.42 ^{ab}
40	Hexadecanoic acid, methyl ester	MS	0.60±0.27 ^a	0.25±0.11 ^{bc}	0.58±0.17 ^{ab}	0.29±0.08 ^{bc}	0.18±0.15 ^c	0.28±0.10 ^{bc}
41	Octadecanoic acid, 9-octadecenyl ester, (Z)-hydrocarbon	MS	0.04±0.02 ^a	0.01±0.01 ^a	0.02±0.01 ^a	0.03±0.01 ^a	0.02±0.03 ^a	ND
42	4,6-dimethyl-Dodecane	MS	3.85±0.53 ^a	3.41±1.62 ^a	3.64±0.69 ^a	3.94±0.28 ^a	3.34±1.18 ^a	4.65±0.47 ^a
43	2,6,10-Trimethyltridecane	MS	0.15±0.05 ^a	0.15±0.08 ^a	0.15±0.08 ^a	0.13±0.03 ^a	0.16±0.02 ^a	0.22±0.04 ^a
44	3-Octadecene, (E)-	MS	0.93±0.07 ^b	1.24±0.39 ^{ab}	1.59±0.37 ^a	0.99±0.27 ^b	1.01±0.08 ^b	1.24±0.30 ^{ab}
45	Cyclononasiloxane, octadecamethyl-	MS	0.86±0.18 ^a	0.40±0.09 ^b	0.39±0.14 ^b	0.40±0.09 ^b	0.38±0.09 ^b	0.43±0.08 ^b
46	Decane, 3,7-dimethyl-	MS	0.11±0.06 ^{ab}	0.15±0.05 ^a	0.09±0.01 ^{ab}	0.06±0.02 ^b	0.09±0.00 ^{ab}	0.14±0.03 ^a
47	Dodecane	MS	0.10±0.06 ^a	0.13±0.03 ^a	0.11±0.04 ^a	0.10±0.04 ^a	0.08±0.07 ^a	0.09±0.02 ^a
48	Heptadecane	MS	0.40±0.05 ^a	0.33±0.06 ^a	0.12±0.04 ^a	0.07±0.05 ^a	0.36±0.45 ^a	0.41±0.08 ^a
49	Hexadecane	MS	ND	0.14±0.09 ^a	0.14±0.04 ^a	0.08±0.02 ^{ab}	0.05±0.05 ^{bc}	0.07±0.03 ^{ab}
50	Octadecane	MS	0.07±0.05 ^{ab}	0.01±0.01 ^b	0.01±0.01 ^b	0.01±0.00 ^b	0.05±0.05 ^b	0.12±0.03 ^a
51	Pentadecane	MS	ND	0.03±0.04 ^a	0.04±0.01 ^a	0.03±0.01 ^a	0.03±0.03 ^a	0.04±0.04 ^a
52	Pentane	MS	1.13±0.09 ^a	0.85±0.10 ^b	0.56±0.07 ^c	0.52±0.09 ^c	0.49±0.02 ^c	1.24±0.20 ^a
53	Tetradecane, 5-methyl-	MS	1.92±0.60 ^a	1.84±0.54 ^a	1.72±0.23 ^a	1.67±0.20 ^{ab}	1.63±0.15 ^{ab}	0.75±0.93 ^b
54	Tridecane	ND	1.26±0.32 ^a	0.72±0.14 ^{bc}	0.66±0.07 ^c	0.80±0.14 ^{bc}	0.77±0.05 ^{bc}	1.17±0.41 ^{ab}
55	Undecane	ND	0.45±0.16 ^a	0.56±0.12 ^a	0.54±0.09 ^a	0.62±0.11 ^a	0.55±0.04 ^a	0.44±0.10 ^a
Other								
56	Ethylene glycol - Adipate - Diethylene glycol	MS	ND	0.02±0.01 ^a	0.02±0.01 ^a	0.03±0.01 ^a	0.01±0.00 ^a	0.02±0.00 ^a
TOTAL			34.92±5.04^a	28.03±2.75^{bc}	26.71±1.68^c	28.02±2.00^{bc}	28.74±1.53^{bc}	33.38±2.65^{ab}

Each value in the table represents the mean ± SD (n=3). Valued labelled with the same letter did not differ significantly ($p \leq 0.05$) according to Duncan's test. ND: not detected in the sample, HS-SPME: headspace solid-phase microextraction, MS: mass spectrometry.

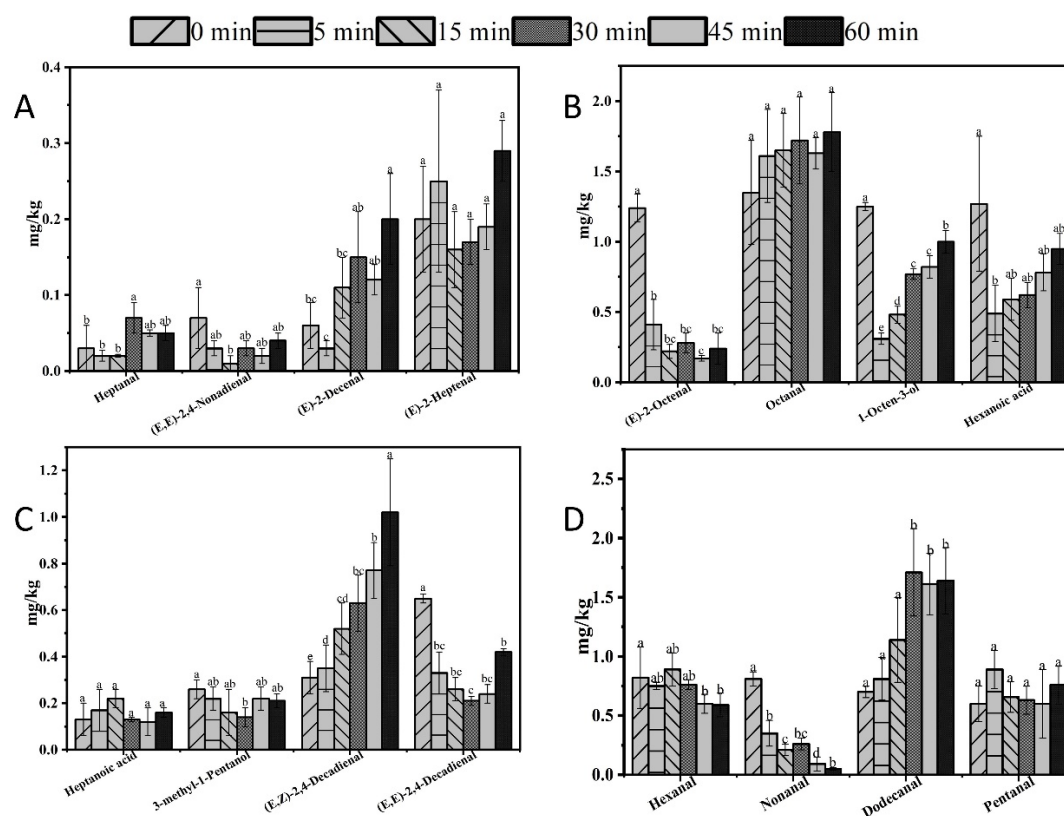


FIGURE 2. Evolution of the content of some volatile compounds subjected to different irradiation times. Each value represents the mean \pm SD (n=3). Values labelled with the same letter did not differ significantly ($p \leq 0.05$) according to Duncan's test.

60 min irradiation because of the oxidation of free fatty acids. Heptanoic acid, (E)-2-Heptenal and pentanal displayed an insignificant change in all samples, which suggested that they were stable after a short time of irradiation. Apart from the compounds mentioned above, octanal, (E, Z)-2,4-decadienal and dodecanal showed an increasing trend. More specifically, (E, Z)-2,4-decadienal showed significant (and in some cases very strong) growth with the increase in processing time. The concentration of octanal was higher than original EPO, although there was no significant difference.

In our experiment, the concentration of some aldehydes was reduced. The change in volatile substances appears to be different between a short time and continuous irradiation for a long time of more than 1 day. According to the previous study, the decomposition of the organic compound was related to the oxidation reaction (Jamil *et al.*, 2017; Jo and Dietrich, 2009; Tsang *et al.*, 2019). Therefore, the possible photocatalytic mechanism of TiO_2 on reduction of volatile compounds was discussed. Comprehensively, the electron (e^-) would be removed

and a positive hole (h^+) would be generated, when the TiO_2 was exposed to light with energy greater than the band gap energy (higher than 3.2 eV). They would migrate to the surface of TiO_2 . Then the combination of oxygen and e^- would produce active superoxide radical ion ($\bullet\text{O}_2^-$) and hydroperoxyl radical ($\bullet\text{OOH}$), respectively. The volatile substances such as E-2-Decenal, 1-octen-3-ol and hexanoic acid would be oxidized, and degraded, leading to a decrease in their contents.

3.2. The analysis of the OAVs of the aroma compounds in EPO

The composition and content of the volatile substances are discussed above. However, the analysis does not fully represent the aroma characteristics of EPO. The threshold of the volatile compounds also influences their contribution to the aroma of EPO. The OAV, calculated by the ratio of its concentration to its odor threshold, was a key indicator in flavor analysis. As shown in table 2, 16 key aroma compounds were selected, including 12 aldehydes, 2 alcohol, and 2 acids. Among them, (E)-2-Hept-

TABLE 2. Identification of volatile flavor compounds by OAV analysis

Compounds	Odor description ^a	Thresholds (mg/kg) ^a	OAV					
			0 min	5 min	15 min	30 min	45 min	60 min
(E)-2-Heptenal	Intense green, fatty, oily	0.01	15.38	19.23	12.31	13.08	14.62	22.31
Hexanal	Green, fatty, leafy	0.08	10.93	10.00	11.87	10.13	8.00	7.87
Nonanal	Strong, floral	0.15	5.40	2.33	1.40	1.73	0.60	0.33
(E, E)-2,4-Decadienal	Fatty, chicken, fried,	0.14	4.81	2.44	1.93	1.56	1.78	3.11
(E)-2-Octenal	green, pungent, spicy,	0.04	31.00	10.25	5.50	7.00	4.25	6.00
(E, Z)-2,4-Decadienal	fried fatty, green, waxy	0.18	1.72	1.94	2.89	3.50	4.28	5.67
Octanal	fat, soap, lemon, green	0.90	2.14	1.79	1.83	1.91	1.81	1.98
Dodecanal	Soapy, waxy, citrus	0.90	0.78	0.90	1.27	1.90	1.79	1.82
Pentanal	almond, malt, pungent	0.15	4.00	5.93	4.33	4.20	4.00	5.07
Heptanal	fat, citrus, rancid	0.05	0.60	0.40	0.40	1.40	1.00	1.00
(E, E)-2,4-Nonadienal	geranium, pungent	0.46	0.15	0.07	0.02	0.07	0.04	0.09
(E)-2-Decenal	green, must	0.15	0.40	0.20	0.73	1.00	0.80	1.33
3-methyl-1-Pentanol	pungent	0.47	0.55	0.47	0.34	0.30	0.47	0.45
1-Octen-3-ol	green, oily	0.04	34.72	8.61	13.33	21.39	22.78	27.78
Heptanoic acid	Cheesy, waxy, sweaty	0.10	1.30	1.70	2.20	1.30	1.20	1.60
Hexanoic acid	mild, fatty	2.50	0.51	0.20	0.24	0.25	0.31	0.38

^aThe Odor description and Thresholds of volatile compounds were referenced from www.thegoodscentscompany.com, <http://www.flavor-net.org/flavornet.html>. OAV: odor active values

nal, 1-octen-3-ol, (E, E)-2,4-Decadienal, Nonanal, (E)-2-Octenal, (E, Z)-2,4-Decadienal, octanal, Pentanal and Heptanoic acid have a major impact on the overall flavor. By comprehensively considering the OAV, it seems that the EPO is more acceptable after 5 min of treatment.

The volatile compound with the maximum OAV in the original oil was 1-Octen-3-ol, followed by E-2-Heptenal, Hexanal and Nonanal. E-2-Heptenal was reported as the production generated by the linoleic acid, known to have a low odor threshold value, and it is usually described as oxidized, green, grassy, and pungent (Cecchi *et al.*, 2019; Tsitlakidou *et al.*, 2019). Although the concentration of E-2-Heptenal was only 0.2 mg/kg, the OAV was 15.13. 1-Octen-3-ol is generally considered to be mushroom-like and described as a secondary oxidation product of linoleic acid in the previous study (Choe and Min, 2006). It has an important contribution to the overall flavor profile because the OAV is 34.72, larger than 1. In the processed sample, the OAV was changed due to the change in concentration. The lowest OAV of E-2-Heptenal was 12.30 after 15 min treatment, followed by the OAV of 13.08 after 30 min. The OAV of

1-octen-3-ol decreased to 8.61, 13.33, 21.39, 22.78, 27.78, respectively. (E, Z)-2,4-Decadienal was described as deep-fried, and green, which is unexpected because it would give a negative flavor to the EPO.

To make a clearer comparison among the samples, the volatile compounds were investigated using a clustering heatmap based on the OAV in table 2. A total of 16 compounds were input and the plot is shown in Figure 3. The heatmap provided the profile of the input value by the colored box, and HCA was used for classifying the group of samples displayed on the left of the plot. The intensity of the color represents the relative contents of the value. The darker the yellow, the larger the contribution to the overall odor. As Figure 3 shows, the samples irradiated for 5 minutes and other periods are grouped into a cluster and distinguished from other EPOs. Considering a comparatively comprehensive and systematic study of their sensory description, we can state again that the EPO with 5 min irradiation was more acceptable. The figure also shows that 1-octen-3-ol, (E)-2-octenal and (E)-heptenal have a significant effect on all samples. On the top of the graph, the compounds were classified by HAC as well.

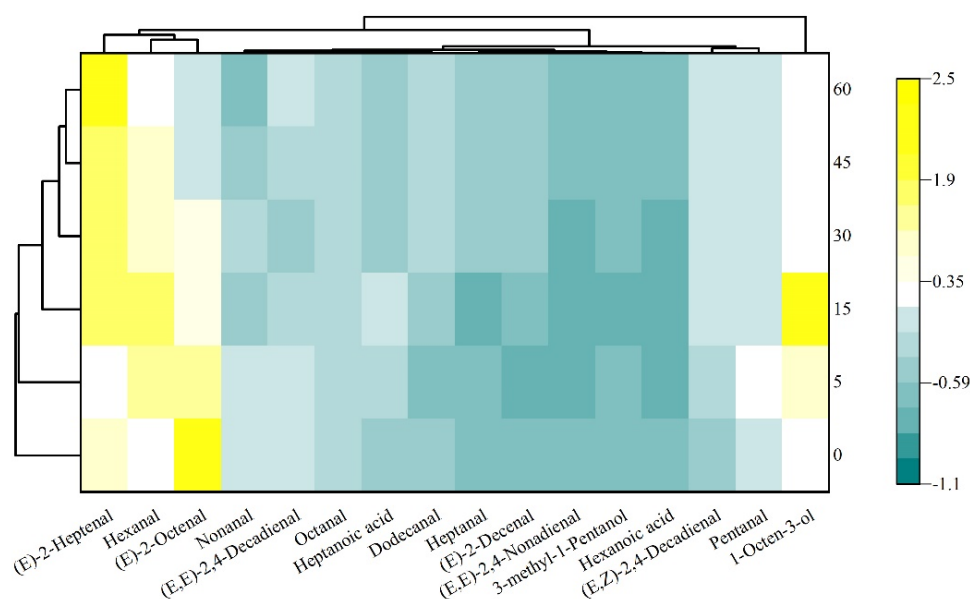


FIGURE 3. HCA of volatile compounds in EPO after different irradiation times.

3.3. Fatty acid composition of the EPO

We hope that UV irradiation has no remarkable negative effect on the quality of the oil, especially on the fatty acids. Previous reports have determined that fatty acids play an essential role in human organisms. They provide energy for our body, are beneficial against some diseases and have a distinctive sensory performance. The composition and percentage of fatty acids have an important impact on the quality of the oil. So, it is necessary to determine the fatty acids in EPO. The fatty acid composition was determined by converting the oil into fatty acid methyl esters (FAME) and detecting it using GC-MS. The results are presented in table 3. From the table, we can see that linoleic acid (C18:2) showed the highest percentage in EPO, followed by γ -linolenic (C18:3n6), oleic (C18:1), palmitic (C16:0), and stearic (C18:0) acids. The result showed that after irradiation the unsaturated acids remained relatively stable. Although a slight reduction existed in some fatty acids, there were no statistically significant differences. γ -linolenic, which is believed to have benefits for human health, is one of the most prominent fatty acids in evening primrose oil. The content of γ -linolenic was 12.15 in the original EPO sample, which is consistent with our previous study (Pan *et al.*, 2020). As the time increased, the content decreased to 11.99, 11.91, 12.01, 11.93 and 11.86, respectively. Luna *et al.* (2006) monitored the content of UFA in virgin

olive oil. They found there is no significant reduction during the first 5 days of irradiation (Luna *et al.*, 2006). Our result showed the same trend. The composition and content of fatty acids in EPO were steady. It seemed 30 min irradiation had a minimal impact on EPO. Sun *et al.* (2021) also report that magnetic graphene oxide/TiO₂ nanocomposite can reduce aflatoxin B1, but the treatment does not affect the quality of corn oil. During storage, the main fatty acid contents in corn oil showed no significant change ($P \geq 0.05$). Magzoub *et al.* (2019) reported a similar result for Sudanese peanut oil. They found after irradiation, the fatty acid profiles were similar in the untreated and treated peanut oil samples, but the aflatoxins were reduced by immobilized titanium dioxide. This demonstrates the use of UV/TiO₂ an application which does not cause any unexpected alterations to the nutritional qualities of EPO, especially when it irradiates under moderate conditions.

3.4. Free radical scavenging capacity

The free radical scavenging capacities of EPO were determined by DPPH and ABTS. The results are shown in Figure 4. The results showed that the antioxidant capacity of EPO with UV irradiation was weaker than the original sample, especially the sample with longer UV pretreatment time (45 and 60 min), which was attributed to the UV irradiation. The DPPH scavenging abilities of EPO with 0, 5,

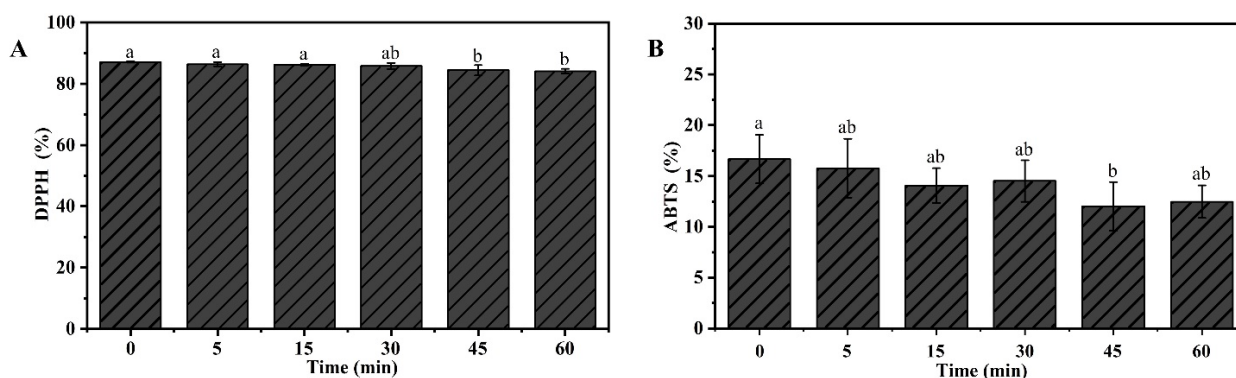


FIGURE 4. DPPH (A) and ABTS scavenging capacity (B) of different samples. Each value in the table represents the mean \pm SD (n=3). Values labelled with the same letter did not differ significantly ($p \leq 0.05$) according to Duncan's test.

TABLE 3. Fatty acid composition and content of EPO subjected to UV/TiO₂ irradiation

Fatty acid	0 min	5 min	15 min	30 min	45 min	60 min
C14:0	0.05 \pm 0.01 ^a	0.05 \pm 0.01 ^a	0.05 \pm 0.01 ^a	0.05 \pm 0.01 ^a	0.05 \pm 0.00 ^a	0.05 \pm 0.01 ^a
C16:0	8.19 \pm 0.10 ^a	8.06 \pm 0.23 ^a	8.00 \pm 0.41 ^a	8.36 \pm 0.52 ^a	8.05 \pm 0.20 ^a	8.00 \pm 0.30 ^a
C16:1	0.04 \pm 0.00 ^b	0.04 \pm 0.01 ^b	0.03 \pm 0.00 ^b	0.04 \pm 0.01 ^b	0.03 \pm 0.01 ^b	0.05 \pm 0.01 ^a
C17:0	0.09 \pm 0.01 ^a	0.09 \pm 0.01 ^a	0.09 \pm 0.01 ^a	0.09 \pm 0.00 ^a	0.10 \pm 0.01 ^a	0.09 \pm 0.02 ^a
C18:0	2.85 \pm 0.09 ^a	2.93 \pm 0.23 ^a	2.90 \pm 0.07 ^a	2.91 \pm 0.16 ^a	2.91 \pm 0.05 ^a	2.88 \pm 0.11 ^a
C18:1	5.47 \pm 0.11 ^a	5.65 \pm 0.04 ^a	5.65 \pm 0.16 ^a	5.62 \pm 0.34 ^a	5.67 \pm 0.28 ^a	5.67 \pm 0.14 ^a
C18:2	62.91 \pm 0.48 ^a	62.88 \pm 1.90 ^a	63.07 \pm 2.17 ^a	62.87 \pm 0.40 ^a	62.94 \pm 1.27 ^a	63.12 \pm 2.57 ^a
C18:3n3	0.79 \pm 0.12 ^a	0.80 \pm 0.01 ^a	0.80 \pm 0.01 ^a	0.80 \pm 0.10 ^a	0.80 \pm 0.06 ^a	0.79 \pm 0.14 ^a
C18:3n6	12.15 \pm 0.44 ^a	11.99 \pm 0.18 ^a	11.91 \pm 0.96 ^a	12.01 \pm 0.13 ^a	11.93 \pm 0.30 ^a	11.86 \pm 0.65 ^a
C20:0	0.50 \pm 0.04 ^a	0.50 \pm 0.01 ^a	0.51 \pm 0.07 ^a	0.51 \pm 0.01 ^a	0.52 \pm 0.08 ^a	0.51 \pm 0.08 ^a
C20:1	0.08 \pm 0.01 ^a	0.08 \pm 0.01 ^a	0.09 \pm 0.02 ^a	0.08 \pm 0.01 ^a	0.08 \pm 0.00 ^a	0.08 \pm 0.00 ^a
C20:2	0.29 \pm 0.02 ^a	0.25 \pm 0.05 ^a	0.30 \pm 0.01 ^a	0.30 \pm 0.02 ^a	0.25 \pm 0.03 ^a	0.25 \pm 0.07 ^a
SFA	11.68 \pm 0.19 ^a	11.62 \pm 0.45 ^a	11.54 \pm 0.48 ^a	11.92 \pm 0.61 ^a	11.63 \pm 0.08 ^a	11.53 \pm 0.26 ^a
UFA	81.73 \pm 0.56 ^a	81.69 \pm 2.00 ^a	81.85 \pm 2.12 ^a	81.72 \pm 0.41 ^a	81.70 \pm 1.36 ^a	81.82 \pm 1.32 ^a
MUFA	5.59 \pm 0.11 ^a	5.76 \pm 0.40 ^a	5.77 \pm 0.16 ^a	5.73 \pm 0.33 ^a	5.79 \pm 0.28 ^a	5.80 \pm 0.14 ^a
PUFA	76.14 \pm 0.58 ^a	75.93 \pm 1.98 ^a	76.08 \pm 2.17 ^a	75.98 \pm 0.58 ^a	75.91 \pm 1.22 ^a	76.01 \pm 1.19 ^a

Each value in the table represents the mean \pm SD (n=3). Values labelled with the same letter did not differ significantly ($p \leq 0.05$) according to Duncan's test. EPO: evening primrose oil, UV/TiO₂: ultraviolet light irradiation with titanium dioxide. C14:0: Myristic acid, C15:0: Pentadecanoic acid, C16:0: Palmitic acid, C16:1: Palmitoleic acid, C17:0: Margaric acid, C18:0: Stearic acid, C18:1: Oleic acid, C18:2: Linoleic acid, C18:3n3: α -Linolenic acid, C18:3n6: γ -Linolenic acid, C20:0: Arachidic acid, C20:1: Eicosenoic acid, C20:2: Eicosadienoic Acid, SFA: saturated fatty acids, UFA: unsaturated fatty acids, MUFA: monounsaturated fatty acids, PUFA: polyunsaturated fatty acids.

15, 30, 45, 60 min were 87.12, 86.38, 86.25, 85.79, 84.40 and 84.12%, respectively, which were higher than the value obtained with ABTS methods. This might be related to the machine used in these methods. In general, the antioxidant capacity of the original EPO is better than that of the irradiated sample, which suggests again that a period of less than 5 min is more suitable for EPO.

3.5. Quality parameters of PSO

In our experiment, we explored the effect of UV/TiO₂ processing on the quality of EPO. The results are shown in table 4. These results indicate that a short irradiation time is appropriate, and the EPO remained relatively stable under such treatment. Overall, these findings are in accordance with the findings reported

TABLE 4. Quality characteristics of EPO subjected to UV/TiO₂ irradiation

	0 min	5 min	15 min	30 min	45 min	60 min
TBA (mg/kg)	0.03±0.00 ^a	0.03±0.00 ^a	0.04±0.00 ^b	0.04±0.00 ^b	0.04±0.00 ^b	0.04±0.00 ^b
POV (meq/kg)	0.86±0.05 ^a	0.91±0.09 ^a	0.91±0.12 ^a	0.95±0.05 ^a	0.99±0.06 ^a	0.97±0.07 ^a
IV (gI ₂ /100g)	184.30±1.48 ^a	176.48±3.60 ^{ab}	183.16±2.27 ^{ab}	174.95±3.10 ^{ab}	173.10±5.03 ^b	174.83±6.33 ^{ab}
L	27.82±0.52 ^a	27.44±0.42 ^a	27.56±0.59 ^a	27.55±0.69 ^a	26.77±0.79 ^a	27.42±0.60 ^a
a*	0.55±0.08 ^a	0.57±0.03 ^a	0.54±0.03 ^a	0.53±0.02 ^a	0.53±0.03 ^a	0.54±0.02 ^a
b*	5.44±0.25 ^a	5.53±0.32 ^a	5.29±0.06 ^a	5.33±0.10 ^a	5.31±0.11 ^a	5.31±0.20 ^a

Each value in the table represents the mean ± SD (n=3). Values labelled with the same letter did not differ significantly ($p \leq 0.05$) according to Duncan's test. EPO: evening primrose oil, UV/TiO₂: ultraviolet light irradiation with titanium dioxide. TBA: thiobarbituric acid values, POV: Peroxide value, IV: Iodine value.

by (Sun *et al.*, 2021), who also found that the quality of corn oil subjected to less than 30 min irradiation was acceptable. Table 4 demonstrates how POV undergoes a significant change due to a short irradiation time, but remained lower than the national limits ($POV \leq 19.7$ mmol/kg). TBA and IV also showed a statistically significant change in EPO. The values for TBA and IV varied from 0.03 to 0.04 and 184.30 to 174.83. The deterioration was due to the treatment of UV/TiO₂. UV irradiation has an impact on the oxidation of EPO, although the deterioration is negligible. Color is one of the essential factors which determines the selection during a consumer's purchase. As shown in table 4, the changes in L, a* and b* values in EPO were not significant, which means UV/TiO₂ treatment does little damage to consumer perception.

3.6. Sensory analysis

To further obtain the differences in the overall flavor between the control group and treatment group, a flavor profile analysis was performed. The flavor attributes, including green, fruity, fatty, vinegar-like, and burnt were used to describe the EPO. The results are shown in Figure 5. Compared to the EPO with 5 min irradiation, the control group showed high scores green and fatty flavors, and lower intensities in fruity, vinegar-like and burnt. This suggests that undesired flavors are strongly perceived in EPO after 5 min of UV exposure as the intensities of green flavor decreased. This is consistent with what found in the previous experiment, a distinct slip in some volatile compounds (E-2-Decenal, 1-octen-3-ol) existed, which led to a reduction in the scores for green and fatty flavors. The sample treated for 5 min showed a better evaluation and higher sensory quality than other treatment-time groups.

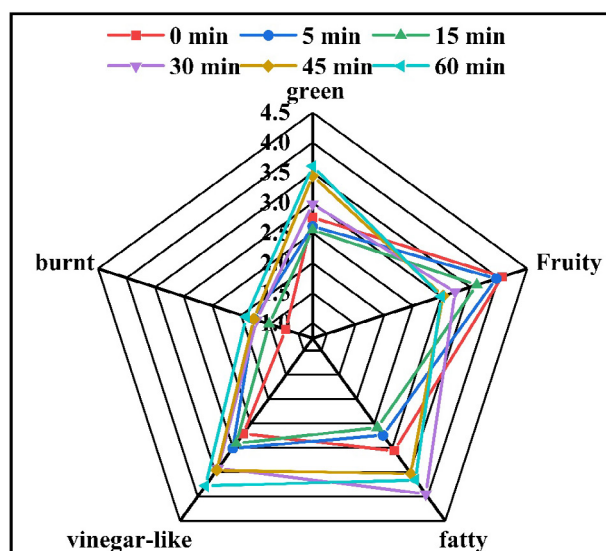


FIGURE 5. Aroma profile of EPO under different treatment conditions

4. CONCLUSIONS

In conclusion, the UV/TiO₂ process was able to effectively reduce odorous compound concentrations, such as E-2-Decenal, 1-octen-3-ol and hexanoic acid, which reached 50, 75.2 and 61.4% reduction after the UV/TiO₂ process of 5 min, respectively. It may be due to the positive hole (h^+) active superoxide radical ion ($\bullet O_2^-$) generated by the UV radiation, which exerts a strong oxidative capacity and promotes the decomposition of these volatile substances. The HCA result and sensory analysis show that the sample with a 5 min process was similar to the control group. The results also indicate a reduction in some volatile compounds due to the UV/TiO₂ treatment, which does not have a significant impact on the quality of corn oil. These results have demonstrated that 5 min UV/TiO₂ processing is appropriate and for a large-scale application for deodorization in

the EPO industry based on ease of operation and low cost,

ACKNOWLEDGMENTS

The authors acknowledge the financial support provided by the Jilin Province Science and Technology Development Project (NO.20220202074NC).

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