Comparison of aroma profiles of essential oils extracted by hydro-distillation from orange peel waste dried by various methods

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Summary

Effects of oven-drying at 45 °C (OD45), oven-drying at 55 °C (OD55) and microwave-drying at 400 W, 560 W and 700 W (MW400, MW560, MW700) on the content of essential oil extracted using hydro-distillation were studied. To get the highest quality and quantity of essential oil, it is necessary to know the suitable methods for drying. The highest concentration of phenolic compounds was determined by UV-spectrophotometryfor OD45, MW400 and MW560, respectively. The highest essential oil yield that was obtained from OD55 samples and the lowest from MW700. Thirty-two components were identified by gas chromatography-mass spectrometry in essential oil samples, mostly oxygenated monoterpenes and hydrocarbon monoterpenes. The main components were limonene (87.4–90.7 %), β-linalool (2.0–3.7 %), β-pinene (1.7–1.8 %), *n*-octanal (1.0–1.2 %), α-terpineol (0.4–0.7 %) and *n*-decanal (0.5–0.7 %). The most important changes were remarkable in MW400. It was observed that some components such as 1-nonanol, β-citral, *E*-2-decanol or *o*-hydroxybiphenyl, which were negligible in oils obtained from fresh peels, showed a significant increase in oils obtained from MW400.

Keywords

essential oil; hydrodistillation; microwave drying; orange

Oranges are consumed fresh or processed to beverages, purées or jam. Orange juice is one of the most widely consumed beverages today all over the world [1]. A large portion of orange peel wastes is thrown out, without recognizing their possible nutritional values and bioactive compounds. It is very important to process these wastes into new products with high added value. At this point, the most valuable commercial form in which orange peel can be processed is essential oil. However, there is a critical challenge for evaluating essential oil from orange peels because orange peels have a high moisture content that causes a very short storage life. It is important to understand the change in essential oil quality with decreasing the water content. Drying methods with various temperatures and humidity conditions are generally used to preserve the important compounds in plant materials. Besides, they are also important to prevent the growth of microorganisms and deteriorative reactions [2]. Recently, traditional drying methods were combined with some modern methods. Oven drying removes moisture from food at steady flow and temperature. However, oven drying has a limited rate of water diffusion from the centre to the surface [3]. Microwave drying is a relatively modern method characterized by shorter drying times and heating in the volume. Nevertheless, microwave drying could lead to high regional temperature that may encourage destruction of sensitive compounds [3]. The other important parameters, namely, energy consumption, cost and availability of the selected technique should be considered, especially for larger scale applications.

Orange oils are reported to be rich in bioactive compounds such as flavonoids, carotenes, terpenes or linalool. It is widely known that the biological activities are strongly related with its specific chemical composition [4]. The drying method affects orange essential oils and major differences are also observed for essential oil yield. Low

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essential oil yield is obtained from fresh orange peel due to its high moisture and big particle size. Furthermore, studies concluded that drying plant materials under various conditions can have a significant effect on the chemical profile and biological attributes of the essential oils derived. Besides, major differences are also observed for aroma compounds of essential oils [4–6].

Orange oils are mixtures that contain over a hundred compounds such as monoterpene hydrocarbons, oxygenated compounds and non-volatile compounds. Some differences between the main component of monoterpene hydrocarbons (limonene) and some other constituents occur during drying [4]. Each compound is affected differently by drying, depending on its chemical properties. Drying method and drying conditions determine the temperature in the inner parts of the orange peel and affect the level of undesirable oxidative reaction. Besides, molecular weight and polarity affect the ease of aroma compounds extraction from the plant material. They are responsible for oxidation reactions at prolonged drying times.

Not much literature data are available on the effects of drying methods on components of orange essential oils. Therefore, the present research aimed to study the effect of drying methods of orange peels on the main components of these volatile oils.

MATERIALS AND METHODS

Materials

Fresh oranges (*Citrus sinensis* L. Osbeck, *Rutaceae*) were obtained from a local market in Kirklareli (Turkey). Only the fruits lacking defects, insect damage, disease, impaired surface colour or other defects were selected for experiments. The fresh oranges were peeled and the peels were manually cut into small pieces with surface of 1 cm^2. Essential oil extraction was performed with both fresh and dried peels.

Oven drying

Drying experiments were conducted in a laboratory-scale hot air oven Atacama Pro F77000 (TRE Spade, Torino, Italia) equipped with automatic temperature and air flow rate control devices. Samples were placed on three shelves with 12 columns and 20 lines per treatment. Drying temperatures were 45 °C and 55 °C, and the airflow rate was 1 $m \cdot s^{-1}$ [7]. The drying times for all samples were determined on the basis of humidity reaching a value of less than 5 %. The drying process was completed in 120 min at 45 °C and in 60 min at 55 °C.

Microwave drying

Microwave drying experiments were performed in a domestic microwave oven Altus ALMD-17BY 20L (Arcelik, Istanbul, Turkey) with a maximum output of 700 W. Three output powers (400 W, 560 W and 700 W) were used in drying experiments. The microwave power was applied until the weight of the sample was reduced to a level corresponding to the moisture content of less than 5%. The drying process was completed in 10 min at 400 W or 560 W, and in 7 min at 700 W.

Hydro-distillation

Before extraction, the dry matter content was determined as a basis for calculation of the essential oil yield from fresh and dried orange peels. Fresh and dried orange peels were ground in a stainless steel grinder Prg-277 (Premier, Istanbul, Turkey) with a glass bowl to allow a better extraction of the oil contained in plant cells. This grinding was immediately followed by extraction so that essential oil evaporated with more volatile compounds and also with higher quality and yield. Essential oils of all treatments were extracted by hydro-distillation using a Clevenger-type apparatus Weightlab Insturument WF-BA 1000, 350W (Sentez Optical Electronic, Atasehir, Turkey) for 4 h. For this, 60 g of the fresh or dried orange peel samples were weighed into a flask and then 600 ml of distilled water was added. The mixture was brought to the boil and the steam isolated essential oils. The steam containing essential oil was condensed and separated directly into a 50 ml graduated burette, in which the amount of oil produced was determined. The oily phase, which was supernatant, was recovered with a Pasteur pipette. The essential oil content was calculated as a relative percentage (w/w). The essential oil was collected, dried with anhydrous sodium sulfate, and stored at 4 °C until use for a maximum 1 day.

Calculation of oil yield

The yield of the extracted oils using each of the six different treatments of drying before extraction was calculated using Eq. 1 [8].

$$Y = \frac{m_E}{m_S} \times 100 \quad (1)$$

where Y is yield of extracted oil (in percent), m_E is the weight of oil extract and m_S is the weight of sample.

Phenolic compounds extraction procedure

Each essential oil sample (1 g) was weighed into a test tube and then 3 ml of solvent (methanol-water, 80 : 20, v/v) was added. The test tube was mixed by vortexing and then centrifuged at 4 000 ×*g* for 5 min and the supernatant was collected. This procedure was repeated two more times. Products of all three extractions were combined and the final volume was brought to 10 ml with the extraction solvent. The resulting solution was then kept in the dark until further analyses for 24 h at 4 °C [9].

Determination of total phenolic compounds

Diluted essential oil samples were filtered through a 0.45 μm polyvinylidene fluoride (PVDF) filter (Millipore, Bedford, Massachusetts, USA), 0.2 ml of Folin-Ciocalteu reagent that was freshly prepared in the laboratory was added to 40 μl of the filtered samples and mixed by vortexing. Then, 0.6 ml of saturated Na_2CO_3 and 0.76 ml of pure water was added, and then mixed by vortexing. After 2 h of reaction at ambient temperature, absorbance at 765 nm was measured using a spectrophotometer UV-2550 (Shimadzu, Kyoto, Japan). Phenolics content in oils was calculated using gallic acid as a standard. The gallic acid standard curve was prepared with 100, 200, 300, 400, 500, 600, 700, 800, 900 and 1000 mg·l^{-1} gallic acid. The total phenolics content (TPC) of the samples was calculated as grams of gallic acid equivalents per kilogram of the sample [9]. Measurements were performed in triplicate.

Determination of aroma compounds

Aroma compounds in essential oils of fresh and dried orange peels were analysed by gas chromatography combined with a triple quadrupole mass spectrometry system (GC-MS/MS) Scion TQ 456 (Bruker, Billerica, Massachusetts, USA) coupled with a triple quadrupole (TQ) mass spectrometer Scion TQ MS/MS (Bruker Daltonics, Billerica, Massachusetts, USA) equipped with a DB-1MS column (30 m × 0.25 mm internal diameter × 0.25 μm film thickness; Agilent Technologies, Santa Clara, California, USA). The GC–MS/MS electron impact source was operated in multiple reaction monitoring (MRM) mode with the MS source temperature of 250 °C, the manifold temperature of 40 °C, transfer line temperature of 280 °C and collision-induced dissociation on argon as collision cell gas with pressure 267 Pa. The injector temperature was maintained at 250 °C with a constant flow rate of 1.0 ml min^{-1} of helium. Injection was splitless with a hold of 1 min. The electron energy was –70 eV. The oven temperature program consisted of a 3 min hold at 50 °C, followed by a 8 °C·min^{-1} ascent to 100 °C and a 30 min hold at 250 °C. The samples were diluted with methylene chloride (Sigma-Aldrich, St. Louis, Missouri, USA). The concentrated extract was immediately injected injected to the GC device.

Statistical analysis

Analysis of variance of data for each attribute were carried out using SPSS 17.0 (SPSS, Chicago, Illinois, USA). Significant differences ($p < 0.05$) among means were determined by Duncan's multiple range test. A probability value of $p < 0.05$ was considered to denote a statistically significant difference between the mean values.

RESULTS AND DISCUSSION

Oil yield

Data on essential oil contents in orange peels are presented in Tab. 1. The percentages of essential oils in orange peel were affected by drying and orange peel form. The yields of essential oils from orange peels were significantly ($p < 0.05$) affected by drying treatments. Hydro-distillation yielded essential oil in a range from a minimum of 0.4 % to a maximum of 1.1 % (Tab. 1). The highest

Tab. 1. Yield and total phenolics content of essential orange peel oils.

Sample	Drying method	Drying conditions	Yield [%]	*TPC* [g·kg^{-1}]
Fresh	none	–	0.4 ± 0.1 ^{f}	5.76 ± 0.50 ^{c}
MW400	Microwave-dried	400 W	1.0 ± 0.2 ^{b}	6.96 ± 0.22 ^{a}
MW560	Microwave-dried	560 W	0.7 ± 0.1 ^{d}	6.88 ± 0.37 ^{a}
MW700	Microwave-dried	700 W	0.5 ± 0.2 ^{e}	5.83 ± 0.27 ^{c}
OD45	Oven-dried	45 °C	0.7 ± 0.2 ^{c}	6.99 ± 0.49 ^{a}
OD55	Oven-dried	55 °C	1.1 ± 0.2 ^{a}	6.57 ± 0.19 ^{b}

Values represent mean ± standard deviation ($n = 3$). Values with different letters in superscript within the same column differ significantly at $p < 0.05$.
TPC – total phenolic content (expressed as grams of gallic acid equivalents per kilogram of sample).

amount of essential oil (1.1 %) was obtained from samples oven-dried at 55 °C, while the minimum percentage was recorded at microwave drying at 700 W (0.5 %). The following highest yield of orange essential oil (1.0 %) was achieved using microwave drying at 400 W. Oven drying at 45 °C and 55 °C as well as microwave drying at 400 W had higher essential oil yield compared with microwave drying at 560 W or 700 W, which were the lowest effective among the drying treatments. Thus, it could maintain more EOs in the dried samples with microwave power and they preserve aromatic compounds from diffusion into the atmosphere [10]. At the beginning of the drying process, moisture is transferred by diffusion from inner parts to surface, carrying the essential oil with it. Accordingly, since diffusion is more noticeable at higher microwave powers, the recommended mechanism describes the reason for the significant "loss" of the essential oil. The results of our present study of the effects of drying on essential oil yield from peel are in agreement with the findings of studies investigating the essential oil contents of oven-dried *Mentha longifolia* (L.) Hudson in comparison with and microwave-dried samples, in which high yield from oven-dried samples was determined [11]. Some other reports in literature also put forth remarkable effects of drying on the yield and characteristics of the essential oils [12–14].

Aroma compounds

A total of 32 compounds were identified in the volatile fraction of orange peel oils according to the drying method of the orange peel (Fig. 1). As shown in Tab. 2, different drying methods had notable effects on all major components identified in the essential oils. Among the dominant aroma groups, monoterpene hydrocarbons were the most important volatile compounds. Although the highest content of monoterpene hydrocarbons was obtained by OD55 (93.6 %), concentration of various compounds differed when using other drying methods. The highest percentage of limonene was found in samples dried by OD55 (90.7 %) and MW560 (90.4 %). According to the values presented in Tab. 2, the drying processes caused an increase in the content of limonene in all treatments except MW400. The increase was 1.3 % (MW560), 0.9 % (MW700), 1.1 % (OD45) and 1.6 % (OD55) and the decrease was 1.7 % for MW400.

The other monoterpene hydrocarbons were β-pinene, α-pinene, β-phellandrene, 3-carene, terpinolene and γ-terpinene. Overall, the total content of monoterpene hydrocarbons in all dried samples, except MW400, was higher than in the fresh sample (Tab. 2). It is known that phenolic compounds in essential oils have low polarity [15]. Besides, monoterpene hydrocarbons are categorized as non-polar compounds and it seems that these compounds have low affinity to the water fraction of fruit peels. Thereby, they would not be evaporated along a with water during the hydro-distillation process. As a result, they could not be evaporated with water via the hydro-distillation process [16]. Another monoterpene hydrocarbon is β-pinene, which is also a major component of essential oils from orange peel. β-Pinene and camphor were initially found to be present at less than 1% in the orange peel. β-Pinene was also determined at remarkable levels (1.7–1.8 %). Monoterpene hydrocarbons are a group of compounds affected by the drying process, in addition to hydro-distillation [17].

Different drying methods are known to remarkably change the content of volatile compounds or cause the formation of new components in essential oils [3]. Although the highest content of monoterpene hydrocarbons was achieved by OD55, several components of this class showed different changes in other drying treatments (Tab. 2). The highest content of limonene (90.7 %) was achieved by OD55. Both MW560 and OD55 were the best in the preservation of monoterpene hydrocarbons, probably thanks to the lower temperature in the inner parts of orange peel, which allowed drying at lower oxygen access that prevented undesirable oxidative reactions of these compounds [3]. MW400 provided the lowest content of monoterpene hydrocarbons (90.5 %), however, samples thus prepared contained significantly more oxygenated monoterpenes than any other treatments (by 6.7 % higher than fresh samples or samples dried in other way). Some oxygenated components like β-citral were absent from the fresh sample, yet they were observed in dried samples. This may be associated with the formation of new compounds by oxidation, esterification, glycosylation, hydrolysis or other processes [3], which could occur due to longer exposure of orange peel to the air during OD45, OD55 and MW400 processes.

The alterations in the contents of oxygenated monoterpenes (specifically, β-linalool, α-terpineol, β-citronellol and α-citral as the major oxygenated monoterpenes) are presented in Tab. 2. The highest content of β-linalool (3.7 %) was obtained by MW400. β-Linalool is recognized as being very important to the good flowery aroma and it is important for flavour character [18]. Citrus fruit contains enzymes for the synthesis of linalool and its cyclization to 2,8-menthadien-1-ol, α-terpineol and D-limonene. Linalool may be a key interme-

diate in the terpenoid metabolism of citrus [18]. As displayed in Tab. 2, not only the drying processes cause a change in some oxygenated monoterpenes and sesquiterpenes, but they also lead to formation of other volatile compounds that were not present in fresh peel essential oil, such as β-citral, 1-nonanol, (*E*)-2-decenol or *o*-hydroxybiphenyl. The content of the other oxygenated compounds, especially of octanal and decanal, is generally considered one of the standards for characterization of orange peel oil [18, 19].

The total content of sesquiterpenes was significantly influenced by the drying treatments (Fig. 1). MW400 led to higher degradation of sesquiterpenes than other drying procedures. As shown in Tab. 2, the lowest contents of valencene (0.2 %) and β-cadinene (< 0.1 %) were determined in samples dried by MW400. Between oven-drying treatments, higher decrease was found to occur in OD45 than OD55 (Tab. 2). The sesquiterpenes have higher molecular weight than monoterpenes and likewise they are less volatile and it is more difficult to extract them from the plant material. They are heat-sensitive compounds and their retention depends on the drying temperature. Besides, they are responsive to oxidation reactions and the prolonged drying times would reduce the sesquiterpene contents [2, 13]. That could explain

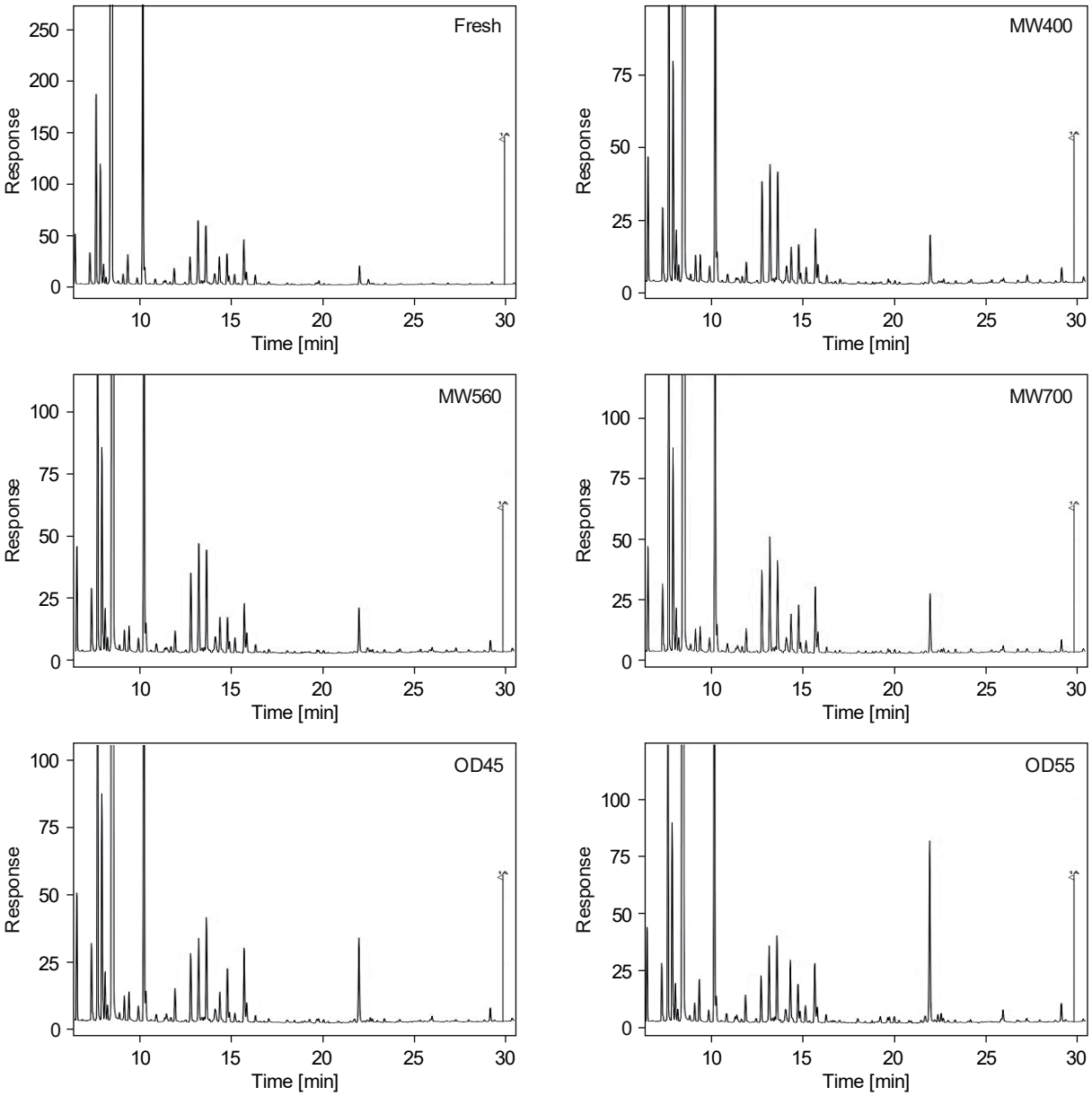

Fig. 1. GC-MS chromatograms of orange peel essential oils.

Tab. 2. Aroma compounds of the essential oils from fresh and dried orange peel.

RT [min]	Compound	Yield [%]						*p*
		Fresh	MW400	MW560	MW700	OD45	OD55	
Monoterpene hydrocarbons								
6.561	α-Pinene	0.5	0.5	0.5	0.5	0.5	0.5	–
7.370	β-Phellandrene	0.3	0.3	0.3	0.3	0.3	0.3	–
7.705	β-Pinene	1.7	1.8	1.8	1.8	1.7	1.7	–
9.174	γ-Terpinene	0.1	0.1	0.1	0.1	0.1	0.1	–
8.529	Limonene	89.1 ^{e}	87.4 ^{f}	90.4 ^{b}	90.0 ^{d}	90.2 ^{c}	90.7 ^{a}	***
8.247	Terpinolene	0.1	0.2	0.2	0.2	0.1	0.1	–
8.904	(*E*)-β-Ocimene	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	–
8.113	3-Carene	0.2	0.2	0.2	0.2	0.2	0.2	–
Total		92.0	90.5	93.5	93.1	93.1	93.6	
Oxygenated monoterpenes								
10.25	β-Linalool	2.4 ^{b}	3.7 ^{a}	2.2 ^{c}	2.4 ^{b}	2.2 ^{c}	2.0 ^{d}	***
10.92	*trans-p*-Mentha-2,8-dien-1-ol	0.1	0.1	0.1	0.1	0.1	0.1	–
11.392	*cis-p*-Mentha-2,8-dien-1-ol	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	–
11.473	Limonene oxide	0.1	0.1	0.0	0.0	0.1	0.1	–
11.954	β-Citronellal	0.2	0.2	0.1	0.1	0.1	0.2	–
12.542	1-Nonanol	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	–
12.814	1-Terpinen-4-ol	0.3	0.3	0.5	0.4	0.5	0.3	–
13.252	α-Terpineol	0.5 ^{bc}	0.7 ^{a}	0.6 ^{ab}	0.6 ^{ab}	0.6 ^{ab}	0.4 ^{c}	*
14.155	Carveol	0.1	0.2	0.1	0.1	0.1	0.1	–
14.413	β-Citronellol	0.4 ^{a}	0.4 ^{a}	0.2 ^{b}	0.2 ^{b}	0.2 ^{b}	0.2 ^{b}	*
14.825	β-Citral	< 0.1 ^{b}	0.3 ^{a}	0.2 ^{a}	0.2 ^{a}	0.3 ^{a}	0.3 ^{a}	*
15.237	Geraniol	0.1	0.1	0.1	0.1	0.1	0.1	–
15.746	α-Citral	0.4	0.5	0.3	0.3	0.4	0.4	–
14.936	Carvone	0.1	0.1	0.1	0.1	0.1	0.1	–
Total		4.7	6.7	4.5	4.6	4.8	4.3	
Sesquiterpene hydrocarbons								
22.039	Valencene	1.1 ^{a}	0.2 ^{c}	0.3 ^{bc}	0.3 ^{bc}	0.3 ^{bc}	0.4 ^{b}	***
22.782	β-Cadinene	< 0.1	< 0.1	0.0	< 0.1	< 0.1	0.0	–
Total		1.1	0.2	0.3	0.3	0.3	0.4	
Other oxygenated compounds								
7.940	*n*-Octanal	1.1	1.2	1.0	1.0	1.0	1.0	–
9.427	1-Octanol	0.2 ^{ab}	0.3 ^{a}	0.1 ^{b}	0.1 ^{b}	0.1 ^{b}	0.1 ^{b}	–
10.349	*n*-Nonanal	0.1 ^{ab}	0.2 ^{a}	0.2 ^{a}	< 0.1 ^{b}	0.1 ^{ab}	< 0.1 ^{b}	*
13.676	*n*-Decanal	0.5	0.7	0.5	0.5	0.5	0.5	–
15.454	(*E*)-2-Decenol	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	–
15.882	Perillal	0.1	0.2	0.1	0.1	0.1	0.1	–
22.524	*o*-Hydroxybiphenyl	< 0.1	0.1	0.0	< 0.1	< 0.1	< 0.1	–
29.248	Nootkatone	0.1	< 0.1	0.1	0.1	0.1	0.1	–
Total		2.1	2.7	2.0	1.8	1.9	1.8	

Values with different letters in superscript within the same column differ significantly (* – $p < 0.05$; ** – $p < 0.01$, *** – $p < 0.001$). *RT* – retention time, MW400 – samples after microwave drying at 400 W, MW560 – samples after microwave drying at 560 W, MW700– samples after microwave drying at 700 W, OD45 – samples after oven drying at 45 °C, OD55 – samples after oven drying at 55 °C.

the most losses of these compounds by MW400 and OD45, which have the longest drying time [18, 20]. To conclude, the worst effects of drying on essential oils and volatile compounds could be attributed to longer processing at lower temperatures compared to the shorter times required at higher temperatures.

CONCLUSIONS

The essential oils of orange peel were rich in limonene and β-linalool. Besides, the chemical composition varied with the drying method used, and so with duration of drying. According to the results, the essential oil of microwave-dried peel at 400 W had the highest quality. These samples had lower content of linalool than that obtained from fresh samples and from samples dried in other way, but it contained substantially more aldehydes, in particular octanal and decanal, which generally belong to standards characterizing the orange flavour.

REFERENCES

1. Gavahian, M. – Chen, Y. M. – Khaneghah, A. M. – Barba, F. J. – Yang, B. B.: In-pack sonication technique for edible emulsions: Understanding the impact of acacia gum and lecithin emulsifiers and ultrasound homogenization on salad dressing emulsions stability. Food Hydrocolloids, *83*, 2008, pp. 79–87. DOI: 10.1016/j.foodhyd.2018.04.039.
2. Farahmandfar, R. – Tirgarian, B. – Dehghan, B. – Nemati, A.: Changes in chemical composition and biological activity of essential oil from Thomson navel orange (*Citrus sinensis* L. Osbeck) peel under freezing, convective, vacuum, and microwave drying methods. Food Science and Nutrition, *8*, 2020, pp. 124–138. DOI: 10.1002/fsn3.1279.
3. Farahmandfar, R. – Tirgarian, B. – Dehghan, B. – Nemati, B.: Comparison of diferent drying methods on bitter orange (*Citrus aurantium* L.) peel waste: changes in physical (density and color) and essential oil (yield, composition, antioxidant and antibacterial) properties of powders. Journal of Food Measurement and Characterization, *14*, 2020, pp. 862–875. DOI: 10.1007/s11694-019-00334-x.
4. Ibrahim, M. E. – El-Sawi, S. A.: Quality and quantity of volatile oil resulting from the recycling of different forms of orange peel using drying methods. Journal of Materials and Environmental Science, *10*, 2019, pp. 598–603. ISSN: 2028-2508. <https://www.jmaterenvironsci.com/Document/vol10/vol10_N7/63-JMES-Ibrahim%202-2019.pdf>
5. Omidbaigi, R. – Kabudani, M. – Tabibzadeh, Z.: Effect of drying methods on the essential oil content and composition of *Tanacetum parthenium* (L.) Schultz Bip cv. Zardband. Journal of Essential Oil Bearing Plants, *10*, 2007, pp. 26–30. DOI: 10.1080/0972060X.2007.10643514.
6. Shalaby, A. S. – El-Gengaihi, S. – Khattab, M.: Oil of *Melissa officinalis* L., as affected by storage and herb drying. Journal of Essential Oil Research, *7*, 1995, pp. 667–669. DOI: 10.1080/10412905.1995.9700525.
7. M'hiri, N. – Ioannou, I. – Boudhrioua, N. M. – Ghoul, M.: Effect of different operating conditions on the extraction of phenolic compounds in orange peel. Food and Bioproducts Processing, *96*, 2015, pp. 161–170. DOI: 10.1016/j.fbp.2015.07.010.
8. Giwa, S. O. – Muhammad, M. – Giwa, A.: Utilizing orange peels for essential oil production. Journal of Engineering and Applied Sciences, *13*, 2018, pp. 17–27. ISSN: 1819-6608. <http://www.arpnjournals.org/jeas/research_papers/rp_2018/jeas_0118_6632.pdf>
9. Parry, J. – Su, L. – Luther, M. – Zhou, K. – Yurawecz, M. P. – Whittaker, P. – Yu, L.: Fatty acid composition and antioxidant properties of cold-pressed marionberry, boysenberry, red raspberry, and blueberry seed oils. Journal of Agricultural and Food Chemistry, *53*, 2005, pp. 566–573. DOI: 10.1021/jf048615t.
10. Rahimmalek, M. – Goli, S. A. H.: Evaluation of six drying treatments with respect to essential oil yield, composition and color characteristics of *Thymy sdaenensis* subsp. *daenensis*. Celak Leaves. Industrial Crops and Products, *42*, 2013, pp. 613–619. DOI: 10.1016/j.indcrop.2012.06.012.
11. Saeidi, K. – Shirmaridi, H. A. – Bastan, M. R.: Essential oil content and composition of *Achillea kellalensis* Boiss, an endemic plant in Iran. Journal of Essential Oil Bearing Plants, *19*, 2016, pp. 516–518. DOI: 10.1080/0972060X.2014.929047.
12. Kamal, G. M. – Anwar, F. – Hussain, A. L. – Sarri, N. – Ashraf, M. Y.: Yield and chemical composition of *Citrus* essential oils as affected by drying pretreatment of peels. International Food Research Journal, *18*, 2011, pp. 1275–1282. ISSN: 2231-7546. <http://www.ifrj.upm.edu.my/18%20(04)%202011/(10)IFRJ-2011-062.pdf>
13. Chong, K. – Lim, Y.: Effects of drying on the antioxidant properties of herbal tea from selected *Vitex* species. Journal of Food Quality, *35*, 2012, pp. 51–59. DOI: 10.1111/j.1745-4557.2011.00422.x.
14. Chua, L. Y. – Chong, C. H. – Chua, B. L. – Figiel, A.: Influence of drying methods on the antibacterial, antioxidant and essential oil volatile composition of herbs: A review. Food and Bioprocess Technology, *12*, 2019, pp. 450–476. DOI: 10.1007/s11947-018-2227-x.
15. Marmouzi, I. – Serragui, S. – Saidi, N. – Bouksaim, M. – Cherrah, Y. – Faouzi, M. E. A.: Effect of drying, polarity and molecular weight on oat chemical content and bioactivity. Journal of Biologically Active Products from Nature, *7*, 2017, pp. 331–340. DOI: 10.1080/22311866.2017.1357500.
16. Hazrati, S. – Farnia, P. – Habibzadeh, F. – Mollaei, S.: Effect of different drying techniques on qualitative and quantitative properties of *Stachys schtsche-*

gleevii essential oil. Journal of Food Processing and Preservation, *42*, 2018, e13686. DOI: 10.1111/jfpp.13686.
17. Babazadeh Darjazi, B. – Rustaiyan, A. – Talaei, A. – Khalighi, A. – Golein, B. – Hayatbakhsh, E. – Taghizad, R. : The effects of rootstock on the volatile flavour components of page mandarin [(*C. Reticulata* var *dancy* × *C. Paradisi* var *dancan*) × *C. clemantina*] juice and peel. Iranian Journal of Chemistry and Chemical Engineering, *28*, 2009, pp. 99–111. DOI: 10.30492/IJCCE.2009.13398.
18. Bourgou, S. – Rahali, F. Z. – Ourghemmi, I. – Tounsi, M. S.: Changes of peel essential oil composition of four Tunisian citrus during fruit maturation. Scientific World Journal, *2012*, 2012, article ID 528593. DOI: 10.1100/2012/528593.
19. Qiao, Y. – Xie, B. – Zhang, Y. – Zhang, Y. – Fan, G. – Yao, X. – Pan, S.: Characterization of aroma active compounds in fruit juice and peel oil of jinchen sweet orange fruit (*Citrus sinensis* (L.) Osbeck) by GC-MS and GC-O. Molecules, *13*, 2018, pp. 1333–1344. DOI: 10.3390/molecules13061333.
20. Nisperos-Carriedo, M. O. – Shaw, P. E. – Baldwin, E. A.: Changes in volatile flavor components of pineapple orange juice as influenced by the application of lipid and composite films. Journal of Agricultural and Food Chemistry, *38*, 1990, pp. 1382–1387. DOI: 10.1021/jf00096a018.

Received 2 March 2021; 1st revised 11 May 2021; 2nd revised 25 June; 3rd revised 10 August 2021; accepted 19 August 2021; published online 16 September 2021.