



Scientific Research

Optimization of distillation time in medicinal and industrial crops using chromatographic-spectrometric conditions and based on the chemical profile of compounds

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ARTICLE INFO	ABSTRACT
Article History: Received:2025/2/10 Accepted:2025/5/4 Keywords: Bioactive compounds, Essential oils, Extraction, Food-pharmaceutical DOI: 10.22034/FSCT.22.166.213. *Corresponding Author E- reza.shahhoseini@gmail.com r-shahhoseini@araku.ac.ir	Despite the progress and innovation of new methods in the field of extracting and processing pharmaceutically active compounds (PhACs), the distillation technique which has a long history combined with scientific progress and evolution, is still considered an innovative technological approach. Productivity, quality, and sustainability are its main indicators. This study was conducted to determine the optimal extraction time and draw the biochemical profile of compounds using gas chromatography-mass spectrometry analysis at different extraction times. <i>Melissa officinalis</i> was selected as a widely used medicinal species in the food and pharmaceutical industries. Data analysis revealed a significant difference in essential oil content at various extraction times. The maximum (0.33% v/w) and minimum (0.25% v/w) extraction efficiency was observed at 180 and 240 minutes, respectively. In total, 62 chemical compounds were isolated in different treatments, with 58 compounds identified. Geranial, neral and citronellal were the main indicators of effectiveness, comprising total of 77.1%, with the highest levels found in the 180-minute treatment. In terms of compound class, oxygenated monoterpenes decreased as extraction time increased. After observing and analyzing the results, it can be determined that the ideal distillation time for this and similar species is 180 minutes. This timeframe not only ensures maximum metabolite yield and high-quality products, but also helps prevent the unnecessary waste of consumable inputs and energy during the extraction process. This finding can serve as a valuable reference point for the production of food and pharmaceutical items.

1. Introduction

Achieving maximum quality products is always a priority for production goals and a distinguishing indicator of research and development departments in the food and pharmaceutical industries. In this regard, choosing the appropriate process for extracting active ingredients is one of the important parameters, which itself depends on various technical and industrial components such as the selection of the appropriate method, solvent and time. In optimizing the extraction process of active metabolites, an unjustified increase in the extraction time will have consequences such as destructive reactions and the presence of unwanted or harmful compounds such as waxes, sterol esters, waste of energy and manpower, reduced therapeutic effects, reduced quality and marketability, and ultimately economic decline [1, 2, 3, 4].

According to the European Pharmacopoeia (Ph. Eur.), essential oils are valuable volatile and aromatic metabolites containing a variety of chemical compounds. These compounds are extracted by various methods and obtained from different organs of medicinal species [5]. Essential oils have been considered and used in health, food and aromatherapy for a long time. They possess various therapeutic effects and biological properties, such as anti-cancer, anti-inflammatory, anti-diabetic, antiviral, antibacterial, antifungal, analgesic, sedative, immune and nervous system protection and antioxidant effects. Essential oils are utilized in the treatment of various disorders and diseases and are widely used in medicine, aromatherapy, pharmaceuticals, food, beverages, cosmetics, hygiene products, detergents, perfumes, colognes, teas and spices. Consequently, they have been increasingly considered and used around the world. The largest consumers of essential oils include the United States, France, and other European countries [6, 7, 8, 9]. So far, more

than 3,000 types of essential oils have been identified with precise determination of their compounds. About one-tenth of these oils are used commercially. Due to their high commercial value, the demand for essential oils in the global market is increasing. The trade value of essential oils is estimated to be around \$9 billion, with a growth rate of 10% over the past 5 years. These active and hydrophobic compounds are insoluble in water but soluble in solvents such as alcohol, ether, and fixed soluble oils. Essential oils belong to a large class of terpenoids, which are a homogeneous and complex mixture. They include a diverse range of fractions from the most volatile to the heaviest components, ranging from monoterpenes to hydrocarbon and oxygenated sesquiterpenes, as well as oxygenated derivatives such as phenylpropanoids [4, 10, 11]. Due to their low levels in medicinal species, essential oils require specialized extraction methods. Utilizing the correct extraction technology can lead to an increase in their added value [12].

The selection of methods for isolating metabolites mainly depends on the yield, output quality, and chemical profile of the compounds in the desired method. Other factors such as ease of access, cost, medicinal species, organ, complex of co-compounds, industries and target markets also affect the selection of isolation methods. Essential oils are extracted using techniques such as distillation, solvent extraction, fixed oil extraction, ultrasonic, microwave, enfleurage, or a combination of the above methods [13, 14]. Despite the invention of extraction methods and their modification and combination, distillation remains the oldest and most common technique. Hydrodistillation is one of the primary and conventional methods for extraction of essential oils [4, 15].

Distillation itself includes various modern methods such as solar energy-assisted hydrodistillation, which can provide maximum yield in the shortest time by optimizing the extraction process. This

method is highly efficient for extracting essential oils, especially for important and industrially used medicinal species like clove, cumin, mint, ginger, dill, lemon balm, and basil [16]. The hydrodistillation has a relative advantage in terms of both quantity and quality of the compounds extracted [17], with the maximum presence of key phytochemical compounds compared to other distillation methods further justifying its use [18, 19].

Essential oils are compounds that are sensitive to light, temperature, oxygen, and moisture [13]. Their constituent compounds have different boiling points and are transferred to the condenser at different times during the distillation process. Therefore, if the distillation apparatus is turned off at an inappropriate time and the extraction time is increased, there is a possibility of significant removal or reduction of the beneficial compounds in the essential oil or negative changes in the color, taste and odor of the essential oil [12, 20].

The medicinal plant lemon balm (*Melissa officinalis*), known by the Persian names Meliss and Warangboo, is one of the most valuable medicinal species. It has its origins in Iran and has been consumed by humans as a food-medicine since ancient times. The active ingredients in lemon balm, especially its essential oil, have various medicinal effects and are used in the cosmetic and health industries. Additionally, lemon balm is widely used in the food industry for flavoring (ice cream, sweets), decorating and producing beverages, fixed oils, soups, meat products and sauces. Its essential oil is important in the complex of food and biological systems due to its antioxidant and antimicrobial effects. The essential oil of lemon balm is colorless or light yellow liquid, relatively pale in color, and has a very pleasant, fresh lemon aroma [9, 13, 21, 22]. Lemon balm contains various active

phytochemicals including volatile oils or aromatic bioactive compounds, triterpenes, flavonoids and phenolic compounds, with the most important being the volatile oils. The German Commission E has approved the use of lemon balm for improving nervous disorders, sleep disorders, depression and digestive disorders [22]. It has high antibacterial and antifungal activity [13], as well as memory enhancement, pain relief, sedative, anti-anxiety, antidepressant and anti-inflammatory properties [9, 22, 23, 24]. Its anti-flatulent effects have been reported in the European Pharmacopoeia [25]. Lemon balm also has antitumor, antiviral (herpes simplex virus and influenza virus) effects [12]. Despite its significant therapeutic value and widespread use in the pharmaceutical and food industries, the essential oil content of lemon balm is relatively low, at less than 0.5%, compared to most medicinal species of the Lamiaceae family. However, it still holds high value in global markets. Therefore, there is a need to study and invest in its production and processing techniques to enhance biomass productivity, as well as the quantity, and quality of essential oil [9].

Despite the high diversity in industrial, widely used, native and endemic medicinal species and consequently the differences in their organs, tissues and metabolite storage channels, the author's detailed studies have shown that the quantitative and qualitative effects of hydrodistillation extraction time have only been evaluated in a limited number of medicinal species. These species include juniper¹ [26], coriander² [27], eucalyptus³ [17], rosemary⁴ [28], artemisia⁵ [29], bay laurel⁶ [30], dill⁷ [31], thyme⁸ [32], marjoram⁹, juniper¹⁰ [6, 33, 34],

1. *Juniperus communis*
 2. *Coriandrum sativum*
 3. *Eucalyptus globulus*
 4. *Rosmarinus officinalis*
 5. *Artemisia turcomanica*

6. *Laurus nobili*
 7. *Anethum graveolens*
 8. *Thymus serpyllum*
 9. *Origanum spp.*
 10. *Juniperus scopulorum*

fennel¹¹, lavender¹², anise¹³, artemisia¹⁴ [35, 36, 37, 38], myrtle¹⁵ [39], lemon verbena¹⁶ [3], agastache¹⁷ [40] and apearmint¹⁸ [4]. In most of the relevant studies, the appropriate extraction time was not determined through prior optimization or a specific reference, leading to variations in the quantity and quality of metabolites, even within a specific species. To address this issue, the present study was conducted to investigate metabolites fluctuations at different extraction times using lemon balm, an important and widely used medicinal plant in the pharmaceutical and food industries.

2- Materials and methods

2.1. Production and preparation

This study was conducted in 2023 and 2024 in the laboratories of the Medicinal Plants Department at Arak University. The seeds of lemon balm, a medicinal plant, were obtained from the Medicinal Plants and Drugs Research Institute, Shahid Beheshti University. After testing and ensuring the germination and health of the seeds, they were immersed in a 1% sodium hypochlorite solution for seven minutes for disinfection. Subsequently, they were thoroughly washed with distilled water and placed in special trays containing cocopeat and perlite. With continued care and development over three months, healthy, uniform, pest, and disease-free seedlings were transferred to the main growth medium. No chemical pesticides were used during this period, and the control of invasive species was carried out by physical removal. After the emergence and development of reproductive meristems (REM) and reaching the full-flower phase, sampling was conducted. The samples were then dried in shade and room temperature conditions, away from direct light for one week, before extracting their essential oil through distillation.

2.2. Distillation process

Essential oil extraction was performed using the hydrodistillation technique with a Clevenger

apparatus (European Pharmacopoeia). Four repetitions were designed for each extraction time, with 50g of leaf samples crushed and placed in the flask for each repetition. 600 ml of water was added to the flask (1:12 ratio) before the extraction process began. The extraction time was accurately monitored from the moment the flask contents started boiling. After distillation was completed and the device cooled, the amount of essential oil was measured, and its efficiency was calculated as a volume-weight percentage (v/w%). To remove any possible water and prevent undesirable reactions, the resulting essential oil was dehydrated with Sodium sulfate anhydrous (Na₂SO₄) and stored in a refrigerator at 4°C until further analysis.

2.3. Gas chromatography–mass spectrometry (GC–MS) analysis

The GC-MS analyses were performed using an Agilent 5975 apparatus with a HP-5ms column (30 m x 0.25 mm i.d., 0.25 µm film thickness) interfaced with a quadruple mass detector and a computer equipped with Wiley 7n.l library; oven temperature 50°C (5min), 50°C-250°C (5°C /min), 250°C (10 min); injector temperature 250°C; volume injection, 1 µL; split ration, 1:50; carrier gas Helium at 1.0ml/min; ionization potential, 70 eV; ionization current, 150 µA; ion source temperature, 280°C; mass range, 35-465 m/z.

2.4. Identification of Compounds

The compounds were identified by calculation of their retention indices under temperature programmed conditions for n-alkanes (C8-C20). The identification of chemical compounds was conducted using factors such as retention time, retention indices, and Kovats retention indices. This involved examining the mass spectra of the sample and comparing them with the mass spectra

¹¹. *Foeniculum Vulgare*

¹². *Lavandula angustifolia*

¹³. *Pimpinella anisum*

¹⁴. *Artemisia annua*

¹⁵. *Myrtus communis*

¹⁶. *Lippia citriodora*

¹⁷. *Agastache astromontana*

¹⁸. *Mentha spicata*

and Kovats retention indices of standard compounds in sources, as well as information from the library (Wiley-7 and Terpenoids) and the NIST web library. Quantification of the relative amount of the individual components was performed as a percentage of the area under the peak and through internal normalization with the condition of the same mass response factor for all compounds [41, 42].

2.5. Statistical analysis

Statistical analysis of the data was performed in a completely randomized design with four replications. The comparison of means was estimated using Duncan's test at a 99% probability level ($p < 0.01$) and SAS version 9 software (SAS Institute Inc., Cary, NC, USA). R software was utilized for cluster analysis (v3.4.3, <https://www.rproject.org>).

3- Results and discussion

In this study, after reviewing previous studies and research experiences, the hydrodistillation technique [43] and different time intervals were designed and considered for extracting the essential oil. Previous studies have used hydrodistillation methods and extraction times of 90 min [44], 180 min [13, 21, 24, 25, 45, 46, 47], 240 min [23, 48], and 300 min [49] to extract compounds of this species. Various factors affect the yield and quality of metabolites, with genetics, environment, and processing being the main types. Among the processing factors, extraction time and determining its optimal limit are crucial [50]. From an operational standpoint, extraction time is essential. When considered according to guidelines and standards, it plays a key role in yield, quality, competitiveness, and sustainable production. Proper implementation of extraction time enables higher efficiency, better quality, lower energy and water consumption, and improved capacity management [20].

In terms of organoleptic properties, the extracted essential oils were all light yellow in color and had a penetrating odor similar to but stronger than that of fresh lemon. In

this respect, no perceptible difference was observed among the samples extracted from different distillation times. These properties have also been reported in other studies [9, 13, 45, 46]. Regarding the importance of the compounds, this indicates that in such experiments, it is not possible to rely solely on organoleptic components. The effectiveness and efficiency must be specifically explored and tracked through measurements and analysis such as metabolites yield and phytochemical profile of the compounds.

In the extraction yield section, the results showed that the essential oil yield in different treatments differed significantly. The maximum amount of essential oil was obtained in the 120-minute (0.33%), and the minimum in the 240-minute (0.25%). The 60- and 180-minute treatments also accounted for 0.29% and 0.31%, respectively (Fig. 1). The essential oil content in other studies has been reported as 0.5% [23, 44], 0.47% [13], 0.37% [46], 0.24% [47], 0.17% [49], and 0.16% [21]. These differences are not simply related to the extraction time but are affected by various components. The content, diversity, and essential oil compounds in a specific medicinal species can vary in different reports due to growth conditions, post-growth development, and processing factors. The complexity of environmental and ecological components that affect growth and synthesis of metabolites, and their interactions under different conditions, can make tracking and control challenging. However, factors related to the processing sector have a high ability to control, generalize, and assimilate these variables. The method and timing of extraction are considered to be some of the most important processing indicators. They are seen as separate, specific and distinct factors. Their implementation in various laboratory conditions can be easily adapted and repeated, making them useful as a fixed model in different studies, as well as in laboratory and industrial scales, with greater certainty.

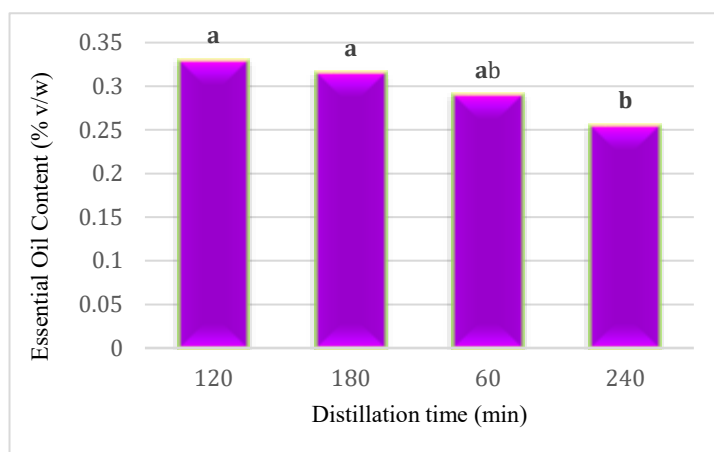


Fig 1. Extraction efficiency in different treatments (% v/w)

Based on the results of GC-MS analysis and interpretation of the compounds, in the present study and various treatments, a total of 62 chemical compounds were detected and 58 compounds were identified in the lemon balm essential oil. At 60 min, 49 compounds were detected and 47 compounds were identified, accounting for a total of 99.70% of the total components. At 120 min, 32 compounds were detected and 31 compounds were identified, accounting for a total of 99.99% of the total components. At 180 min, 41 compounds were detected and 39 compounds were

identified, accounting for a total of 99.70% of the total components. This trend continued for the 240 min, with the detection of 48 compounds and the identification of 46 compounds, accounting for 99.50% of the total components. The types and amounts of compounds varied in different treatments, with differences visible from low-abundance compounds to major compounds (Table 1).

Table 1. Chemical composition analysis of lemon balm (*Melissa officinalis* L.) essential oil

No.	Compositions	Molecular formula	RI ¹⁹	Ex Time ₆₀	Ex Time ₁₂₀	Ex Time ₁₈₀	Ex Time ₂₄₀
1	2E-hexenal	C ₆ H ₁₀ O	843	1.4	1.4	0.7	0.6
2	3Z-hexenol	C ₆ H ₁₂ O	846	0.1	0.1	0.1	0.1
3	α-pinene	C ₁₀ H ₁₆	938	<i>t</i> ²⁰	-	-	-
4	sabinene	C ₁₀ H ₁₆	977	<i>t</i>	-	-	-
5	β-pinene	C ₁₀ H ₁₆	979	<i>t</i>	-	-	-
6	1-octen-3-ol	C ₈ H ₁₆ O	987	0.7	0.7	0.5	0.4
7	6-methyl-5-hepten-2-one	C ₈ H ₁₄ O	993	1.5	1.6	1.1	1.0
8	myrcene	C ₁₀ H ₁₆	994	0.1	0.2	0.1	0.1
9	3-octanol	C ₈ H ₁₈ O	998	<i>t</i>	-	-	-
10	2E,4E-heptadienal	C ₇ H ₁₀ O	1013	<i>t</i>	-	-	-
11	limonene	C ₁₀ H ₁₄	1032	0.1	-	<i>t</i>	<i>t</i>
12	1,8-cineol	C ₁₀ H ₁₈ O	1035	0.3	0.3	0.2	0.1
13	Z-β-ocimene	C ₁₀ H ₁₆	1043	<i>t</i>	-	-	-
14	benzenacetaldehyde	C ₈ H ₈ O	1048	<i>t</i>	-	-	0.1
15	E-β-ocimene	C ₁₀ H ₁₆	1054	0.1	0.1	0.1	0.1

. The Kovats retention indices relative to C₈-C₂₀ 19 n-alkanes were determined on HP-5 ms capillary column

2. Trace<0.05

16	bergamol	C ₁₂ H ₂₀ O ₂	1059	0.1	-	0.1	0.1
17	cis-linalool oxide	C ₁₀ H ₁₈ O	1076	-	-	-	<i>t</i>
18	unknown	-	1098	-	-	-	0.3
19	rosefurane	C ₁₀ H ₁₄ O	1099	0.2	0.2	0.2	-
20	linalool	C ₁₀ H ₁₈ O	1102	0.2	0.3	0.3	0.5
21	n-nonanal	C ₉ H ₁₈ O	1104	0.4	0.3	0.3	0.3
22	cis-rose oxide	C ₁₀ H ₁₈ O	1113	0.2	0.2	0.2	0.2
23	trans-rose oxide	C ₁₀ H ₁₈ O	1131	0.1	0.1	0.1	0.1
24	epi-Photocitral	C ₁₀ H ₁₆ O	1144	0.5	0.4	0.4	0.4
25	exo-isocitral	C ₁₀ H ₁₆ O	1151	0.4	0.5	0.4	0.4
26	photocitral A	C ₁₀ H ₁₆ O	1155	1.0	0.9	0.9	0.8
27	citronellal	C ₁₀ H ₁₈ O	1166	8.3	7.7	7.3	5.5
28	Z-isocitral	C ₁₀ H ₁₆ O	1173	1.6	1.3	1.2	1.2
29	menthol	C ₁₀ H ₂₀ O	1178	-	-	<i>t</i>	-
30	rosefuran epoxide	C ₁₀ H ₁₄ O ₂	1181	0.5	0.5	0.4	0.5
31	E-isocitral	C ₁₀ H ₁₆ O	1185	2.3	1.6	1.7	1.9
32	α-terpineol	C ₁₀ H ₁₈ O	1195	-	-	-	<i>t</i>
33	unknown	-	1199	0.2	0.1	0.2	0.2
34	4-methylen-isophorene	C ₁₀ H ₁₄ O	1222	-	-	0.1	0.1
35	nerol	C ₁₀ H ₁₈ O	1241	-	0.5	-	-
36	pulegone	C ₁₀ H ₁₆ O	1248	3.2	4.3	3.6	-
37	neral (Z-citral)	C ₁₀ H ₁₆ O	1271	32.6	31.1	31.9	33.4
38	piperitone	C ₁₀ H ₁₆ O	1272	-	1.1	-	-
39	methyl citronellate	C ₁₁ H ₂₀ O ₂	1274	0.7	1.6	0.7	0.6
40	geranial (E-citral)	C ₁₀ H ₁₆ O	1290	35.4	37.5	37.9	36.6
41	neric acid	C ₁₀ H ₁₆ O ₂	1311	0.1	0.6	-	0.1
42	methyl geranate	C ₁₁ H ₁₈ O ₂	1337	0.8	-	0.8	0.8
43	piperitenone	C ₁₀ H ₁₄ O	1356	0.3	0.1	0.2	0.2
44	unknown	-	1371	-	-	0.2	-
45	neryl acetate	C ₁₂ H ₂₀ O ₂	1372	0.1	-	-	0.1
46	piperitone oxide	C ₁₀ H ₁₆ O ₂	1376	<i>t</i>	-	-	0.1
47	α-copaene	C ₁₅ H ₂₄	1383	-	-	-	0.1
48	geranyl acetate	C ₁₂ H ₂₀ O ₂	1392	2.2	1.3	1.9	2.4
49	unknown	-	1402	0.1	-	-	-
50	E-caryophyllene	C ₁₅ H ₂₄	1427	2.4	2.1	3.4	5.5
51	α-humulene	C ₁₅ H ₂₄	1453	0.1	0.1	0.2	0.3
52	germacrene-D	C ₁₅ H ₂₄	1475	0.1	-	0.1	0.2
53	E-β-ionene	C ₁₃ H ₂₀ O	1477	<i>t</i>	-	<i>t</i>	0.1
54	E,E-α-farnesene	C ₁₅ H ₂₄	1511	-	-	-	<i>t</i>
55	δ-cadinene	C ₁₅ H ₂₄	1530	-	-	-	0.1
56	germacrene-D-4-ol	C ₁₅ H ₂₆ O	1586	-	-	-	0.1
57	caryophyllene oxide	C ₁₅ H ₂₄ O	1594	1.6	1.1	2.5	4.2
58	caryophylla-4(12),8(13)-dien-5.α-ol	C ₁₅ H ₂₄ O	1641	<i>t</i>	-	<i>t</i>	0.1
59	caryophylla-4(12),8(13)-dien-5.β-ol	C ₁₅ H ₂₄ O	1645	0.1	-	0.1	0.2
60	epi-α-muurolol	C ₁₅ H ₂₆ O	1649	-	-	-	0.1
61	α-cadinol	C ₁₅ H ₂₆ O	1662	<i>t</i>	-	<i>t</i>	0.1
62	14-hydroxy-9-epi-(E)-caryophyllene	C ₁₅ H ₂₄ O	1679	0.1	-	0.2	-

According to the results of chromatography analysis, the dominant and major components of the essential oil are geranial, nerol, and citronellal. Following those, compounds such as caryophyllene, caryophyllene oxide, pulegon, isocitral and geranyl acetate

accounted for a smaller share of compounds. Other studies have also identified main and significant components of the essential oil including geranial, neral [43]; geranial, neral, citronellal [9, 13, 45, 51]; nerol, geranial, neral, isopulgol [23]; geranial, neral, citronellal, β-caryophyllene

21. *trans* citral; α-citral; citral A; *E*-isomer22. *cis* citral; β-citral; citral B; *Z*-isomer

23. Citronellal; Rhodinal; 3,7-Dimethyl-6-octenal

[47]; geranial, neral, citronellal, citronellol [44]; geranial, neral, β -caryophyllene, germacene D [21]; geranial, neral, citronellal, β -caryophyllene, germacene-D, δ -3-carene [25]; geranial, neral, geranyl acetate, caryophyllene oxide, β -caryophyllene [46] and caryophyllene oxide, β -caryophyllene, geranial, neral [48]. Studies have shown that extraction time does not significantly affect the type of these main compounds (Table 1).

Due to the high presence of neral and geranial isomers in essential oils, the main biologically active effects are related to these isomers and are considered one of the important factors determining the quality of essential oils [20]. Neral and geranial are considered the most important components of essential oils known as citral²⁴. Citral is a biological molecule with significant biological activities. It is a linear oxygenated monoterpene with the molecular formula $C_{10}H_{16}O$ and has *cis* and *trans* forms. In addition to numerous biological effects, it is widely used as a flavoring agent in medicine, food and cosmetic products, as well as in the preparation of perfumes and colognes [52]. When taken orally, it is completely digested in the digestive tract. In the food industry, it is one of the most important natural flavoring compounds with an intense lemon aroma and taste, and it is widely used as an additive in foods and beverages [53]. Citral and citronellal are members of the aldehyde group and have antiviral, antibacterial, anti-inflammatory, anticancer, antipyretic, antispasmodic, antioxidant, vasodilator, hypotensive, and sedative effects [52, 54]. The total amounts of the main compounds (geranial, neral, and citronellal) in 60, 120,

180, and 240 min treatments were 76.3%, 76.3%, 77.1%, and 75.5%, respectively. Despite a slight decrease in the relative amount of some main compounds as the distillation process progresses and in the 120 min, the amount of decrease is insignificant. The total of these compounds, as the basis of effectiveness in the 180 min treatment, is still at the maximum level (77.1%) (Table 1 – Fig. 2), and its active and biological effects do not change. Therefore, considering the efficiency of the essential oil, which has a significant and significant difference compared to the 120 min, the 180 min treatment can be chosen as a priority selection. By increasing the time to 240 min, along with the decrease in the level of the main compounds, the efficiency of the essential oil also experienced a significant reverse trend. Taken into account other influential factors, particularly energy consumption, would not be justified even if the increase in some minor and low-amount compounds such as caryophyllene, caryophyllene oxide and geranyl acetate were considered. This conclusion is supported even when introducing a larger number of low-amount compounds like pulegone, isocitral, methyl citronellate, piperitone, etc in the analysis. Consequently, the 240 min treatment stands out distinctly and with a greater separation compared to the other treatments (Fig. 2). In fact, eliminating the 240 min treatment allows the producer to extract the maximum amount of essential oil with a higher content of target compounds such as geranial, neral, and citronellal in the desired product. This shorter distillation time also results in lower energy consumption, significantly reducing production costs.

24. 3,7-dimethyl-2,6-octadienal

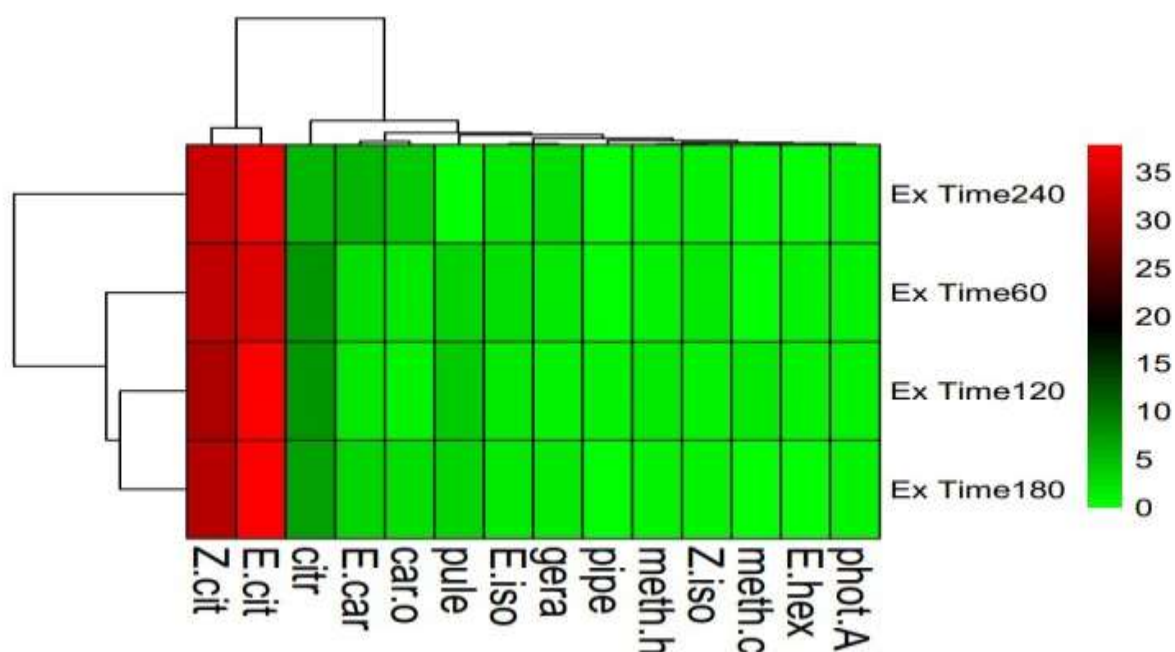


Fig 1. Presence of major and minor compounds in different extraction treatments

When looking at the profile of main and minor compounds, it is noted that the highest levels of lighter monoterpene compounds, such as limonene and 1,8-cineole are present in the essential oil within the initial 1 to 2 h of extraction. These levels then decrease as extraction time increases, sometimes dropping by more than 100 percent by the end of the process or becoming negligible altogether [33, 55]. Although the aforementioned compounds are of low importance in this experiment, it should be noted that if the main focus is on compounds from this group, this issue should be examined and taken into account. Additionally, as distillation time increases, there is a possibility of oxidation of compounds like menthol and their reduction, leading to an increase in certain compounds such as menthone [4]. In the current experiment, this phenomenon is observed with the pulegone compound, as after an extraction time of 120 min, the amount of pulegone decreased and was completely eliminated by 240 min.

In the class of compounds, five main groups were distinguished: monoterpene

hydrocarbons, oxygenated monoterpenes, sesquiterpene hydrocarbons, oxygenated sesquiterpenes, and miscellaneous compounds. The distribution of these compounds varied in different treatment durations: 0.8%, 90.1%, 2.6%, 1.8% and 4.4% in the 60 min treatment; 0.3%, 89.3%, 2.3%, 1.1%, 6.8% in the 120 min treatment; 0.2%, 87.8%, 3.7%, 2.9%, 5.2% in the 180 min treatment; and 0.2%, 83.5%, 6.2%, 4.7%, 4.9% in the 240 min treatment (Table 2). As can be seen, despite the decreasing trend with increasing extraction time, oxygenated monoterpenes occupied the highest values within class of compounds in all treatments. As distillation time increased, the amount of monoterpenes and sesquiterpenes without oxygen in the essential oil relatively increased, while the amount of oxygenated monoterpenes decreased relatively [30]. A similar trend is observed in the present analysis, particularly in relation to oxygen-free sesquiterpenes and oxygenated monoterpenes. In fact, with increasing distillation time, the amount of oxygenated compounds decreased while the amount of hydrocarbon compounds increased.

Table 2. Classification of chemical compounds of essential oils (*Melissa officinalis* L.)

Major Grouped Compounds	Ex Time ₆₀	Ex Time ₁₂₀	Ex Time ₁₈₀	Ex Time ₂₄₀
Monoterpene hydrocarbones	0.8	0.3	0.2	0.2
Oxygenated monoterpenes	90.1	89.3	87.7	83.5
Sesquiterpene hydrocarbones	2.6	2.3	3.7	6.2
Oxygenated sesquiterpenes	1.8	1.1	2.9	4.7
Misellaneous	4.4	6.8	5.2	4.9
Total Identified	99.70	99.90	99.70	99.50

In other studies, it was found that in the *fennel*²⁵, increasing the extraction time from 15 to 150 min resulted in a higher the yield of essential oil. The maximum amount of essential oil, hydrocarbon compounds, and the minimum amount of oxygenated compounds were recorded at 150 min [18]. In the medicinal species *piné*²⁶, the highest yield of essential oil was achieved after 160 min of extraction. The two main compounds, α -pinene and β -pinene, reached their peak levels at the beginning of the extraction process (min 5 to 20) and decreased over time. On the other hand, low-abundance compounds like germacrene-D, limonene, myrcene, and γ -cadinene increased until around 2 h, after which they either stabilized or decreased [56]. In the medicinal plant Damask rose²⁷, the amount of essential oil continued to increase up to 3 h of extraction, with no significant increase observed beyond that point. For oxygenated monoterpene compounds like citronellol, neral and geranial, it is recommended to extract for about 3 h, while for compounds like nerol and geraniol, 90 min is suggested as the optimal time [1, 57, 58]. In the medicinal species Denaei thyme²⁸ and Shirazi thyme²⁹, an extraction time of 2 h was chosen based on the quantity and quality of the compounds present, as well as the

presence of the main phenolic monoterpenes such as thymol and carvacrol [59]. In the medicinal species cumin, extending the extraction time resulted in higher content and yield of the essential oil. However, after 2 h, the amount of the main compound cuminaldehyde decreased, along with its antimalarial efficacy [60]. In the medicinal species cajuput³⁰, the essential oil yield increased with the extraction time increasing to 8 h, but the maximum amount of the dominant and main compound, nerolidol was achieved in 5 h [61]. In different varieties of the medicinal plant *hemp*, increasing the extraction time from 2 to 4 h resulted in some varieties showing an increase in the yield of essential oil, while in others there was no difference in the yield of essential oil. The compounds β -myrcene and terpinolene accounted for larger amounts at 2 h, and their amounts decreased over time. This trend was reverse in most cases with the compounds β -caryophyllene and humulene [62]. During the extraction of essential oil from the rhizome of the medicinal species sweet flag³¹ using distillation, it was observed that with a fourfold increase in extraction time, the yield of essential oil also increased fourfold. As the extraction time continued to pass, the compound bornyl acetate decreased significantly and eventually

25. *Foeniculum Vulgare*26. *Pinus ponderosa*27. *Rosa damascena*28. *Thymus daenensis*29. *Zataria multiflora*30. *Melaleuca leucadendra*31. *Acorus calamus*

reached zero [63]. Some studies have not yielded any specific findings regarding the impact of extraction time, attributing this to the variability of pharmaceutical raw materials and fluctuating environmental conditions [64]. It is worth noting that there may be differences in the efficiency, type, and quality of the resulting compounds among the various distillation methods [water, steam, water and steam, microwave], and the results are not necessarily similar or close. In addition to the availability of equipment, the choice of distillation type mainly depends on the organ used, the size or grinding of the particles, and even the presence and amount of other accompanying compounds such as saponins, waxes, and resins [15, 24]. The differences are sometimes so great that they affect the type and amount of the dominant and main compounds among the distillation methods and generally change the chemical profile, properties, and effects of the essential oil [65].

As can be seen, the results of other studies, despite some differences in findings, can mainly be due to variations in species or cultivars, the ducts and tissues secreting and storing essential oil, and the level of grinding. However, especially in relation to the aerial organs of medicinal species, they consistently show a time interval of about 120 to 180 min for optimal quantity and quality of compounds. In the present evaluation, overall and considering the quantity and quality of essential oil, particularly the presence of main compounds like geranial, neral, and citronellal, which are key indicators of effectiveness and quality in the food and pharmaceutical industries, an extraction time of 180 min can be considered an appropriate treatment. This aligns well with previous studies. By examining and comparing studies conducted in this field, it can be concluded that distillation time serves multiple purposes. It not only influences the quantity and maximum output of the extraction process but also helps prevent resource depletion and

energy waste. Additionally, distillation time can be utilized as a technique to obtain essential oils with specific targeted compositions. Based on the operating conditions outlined in this study, extending the distillation time beyond 180 min does not result in qualitative or functional superiority. As a result, there is no significant industrial or economic advantage to doing so. It is found within 180 min, essential oils can be obtained with good yield, suitable properties and low operating costs.

4- Conclusion

Based on the findings of the present study, it is important to highlight the significance of the compounds and the savings in resources and energy consumption that can be achieved with the 180 minute treatment in order to obtain an appropriate output of essential oil and its valuable compounds. It is recommended that further research be conducted on this issue using other industrial extraction methods. The findings of this study, when considered alongside other studies, suggest that an indiscriminate increase in extraction time for metabolites can lead to a waste of energy and result in some destructive and undesirable hydrolysis reactions. This, in turn, may diminish the biological and therapeutic effects of essential oils, particularly in the food and pharmaceutical industries. The extraction yield, types of compounds, and their amounts will vary at different extraction times. Therefore, in such studies, determining the purpose of extraction is of primary importance. To achieve optimal efficiency of metabolites at the right time, as well as quality, one must consider related parameters such as energy consumption during extraction and the economic value of the essential oil. Since the yield of the active ingredients and the profile of their compounds can be altered in this process, each manufacturing unit can optimize the extraction time and other processing parameters in their field based on factors such as heating and cooling energy consumption systems, equipment used,

capacity and depreciation. In fact, if the goal is to extract essential oils with specific amounts of desired compounds and specific biological and medicinal effects, a targeted and sustainable process can be achieved by adjusting the distillation time.

5-References

- [1] Mirzaei, M., Ahmadi, N., Sefidkon, F., Shojaeiyan, A., & Mazaheri, A. (2015). Evaluation of phytochemical profiling of damask rose (*Rosa damascena* Mill.) at various post-harvest incubation conditions and determination of the best hydro-distillation time. *Iranian Journal of Medicinal and Aromatic Plants*, 31(4), 732-742. <https://doi.org/10.22092/ijmapr.2015.102689>
- [2] Zheljaskov, V.D., Gawde, A., Cantrell, C.L., Astatkie, T., & Schlegel, V. (2015). Distillation time as tool for improved antimalarial activity and differential oil composition of cumin seed oil. *PLoS One*, 10(12), 1-12. <https://doi.org/10.1371/journal.pone.0144120>
- [3] Ghorbanpour, M., & Shahhoseini, R. (2017). Influence of distillation time on the content and constituent of essential oils isolated from lemon verbena (*Lippia citriodora* Kunth). *Journal of Essential Oil Bearing Plants*, 20(4), 1083-1089. <https://doi.org/10.1080/0972060X.2017.1345648>
- [4] Marques, S.M., Pinheiro, R.O., Nascimento, R.D., Andrade, E.A., & Faria, L.D. (2023). Effects of Harvest Time and Hydrodistillation Time on Yield, Composition, and Antioxidant Activity of Mint Essential Oil. *Molecules*, 28(22), 1-13. <https://doi.org/10.3390/molecules28227583>
- [5] Kant, R., & Kumar, A. (2022). Review on essential oil extraction from aromatic and medicinal plants: Techniques, performance and economic analysis. *Sustainable Chemistry and Pharmacy*, 30, 100829. <https://doi.org/10.1016/j.scp.2022.100829>
- [6] Toker, R., Golukcu, M., & Tokgoz, H. (2017). Effects of distillation times on essential oil compositions of *Origanum minutiflorum* O. Schwarz Et. and PH Davis. *Journal of Essential Oil Research*, 29(4), 330-335. <http://dx.doi.org/10.1080/10412905.2016.1276026>
- [7] Aljaafari, M.N., AlAli, A.O., Baqais, L., Alqubaisy, M., AlAli, M., Molouki, A., & Lim, S.E. (2021). An overview of the potential therapeutic applications of essential oils. *Molecules*, 26(3), 1-27. <https://doi.org/10.3390/molecules26030628>
- [8] Vora, L.K., Gholap, A.D., Hatvate, N.T., Naren, P., Khan, S., Chavda, V.P., & Khatri, D.K. (2024). Essential oils for clinical aromatherapy: a comprehensive review. *Journal of Ethnopharmacology*, 330, 1-21. <https://doi.org/10.1016/j.jep.2024.118180>
- [9] Medeiros, A.R., Leite, J.F., de Assis, R.A., Rocha, J.M., Bertolucci, S.V., & Pinto, J.P. (2024). Application of natural elicitors to promote growth, photosynthetic pigments, and the content and composition of essential oil in *Melissa officinalis* L. *Industrial Crops and Products*, 208, 117885. <https://doi.org/10.1016/j.indcrop.2024.118244>
- [10] Dodos, T., Rajcevic, N., Janackovic, P., Vujisic, L., & Marin, P.D. (2019). Essential oil profile in relation to geographic origin and plant organ of *Satureja kitaibelii* Wierzb. ex Heuff. *Industrial Crops and Products*, 139, 1-8. <https://doi.org/10.1016/j.indcrop.2019.111549>
- [11] Yammine, J., Chihib, N.E., Gharsallaoui, A., Ismail, A., & Karam, L. (2024). Advances in essential oils encapsulation: Development, characterization and release mechanisms. *Polymer Bulletin*, 81(5), 3837-3882. <https://doi.org/10.1007/s00289-023-04916-0>
- [12] Ye, R., Tian, K., Hu, H., Li, P., & Tian, X. (2023). Extraction process optimization of essential oil from *Melissa officinalis* L. using a new ultrasound-microwave hybrid-assisted Clevenger hydrodistillation. *Industrial Crops and Products*, 203, 117165. <https://doi.org/10.1016/j.indcrop.2023.117165>
- [13] Abdellatif, F., Akram, M., Begaa, S., Messaoudi, M., Benarfa, A., Egbuna, C., & Simalgandara, J. (2021). Minerals, essential oils, and biological properties of *Melissa officinalis* L. *Plants*, 10(6), 3-16. <https://doi.org/10.3390/plants10061066>
- [14] Perovic, A.B., Karabegovic, I.T., Krstic, M.S., Veličkovic, A.V., Avramovic, J.M., Danilovic, B.R., & Veljkovic, V.B. (2024). Novel hydrodistillation and steam distillation methods of essential oil recovery from lavender: A comprehensive review. *Industrial Crops and Products*, 211, 118244. <https://doi.org/10.1016/j.indcrop.2024.118244>
- [15] Pheko-Ofithile, T., & Makhzoum, A. (2024). Impact of hydrodistillation and steam distillation on the yield and chemical composition of essential oils and their comparison with modern isolation techniques. *Journal of Essential Oil Research*, 36(2), 105-115. <https://doi.org/10.1080/10412905.2024.2320350>
- [16] Katekar, V.P., Rao, A.B., & Sardeshpande, V.R. 2023. A hydrodistillation-based essential oils extraction: A quest for the most effective and cleaner technology. *Sustainable Chemistry and Pharmacy*, 36, 101270. <https://doi.org/10.1016/j.scp.2023.101270>
- [17] Barazandeh, M.M. (2005). The effect of method and time of distillation on the essential oil yield and composition of *Eucalyptus globulus*. *Iranian Journal of Medicinal and Aromatic Plants Research*, 21(1), 75-93. <https://doi.org/10.22092/ijmapr.2005.115209>
- [18] Jamshidi, A.H., Shams Ardakani, M.R., Hadjiakhondi, A., & Abdi, K. (2004). The influence of distillation conditions on the essential oil composition of fennel (*Foeniculum vulgare* Mill.).

- Journal of Medicinal Plants*, 3(11), 68-72. [20.1001.1.2717204.2004.3.11.9.9](https://doi.org/10.1001.1.2717204.2004.3.11.9.9)
- [19] Hekmatsorush, I., Milani Kalkhorani, N., Rezaee, M.B., Hero Abadi, F., & Hamisi, M. (2014). Phytochemical analysis of essential oil of *Tanacetum parthenium* L. with hydro-distillation and steam distillation. *Journal of Medicinal plants and By-Products*, 3(1), 53-57. doi: [10.22092/jmpb.2014.108604](https://doi.org/10.22092/jmpb.2014.108604)
- [20] Machado, C.T., Hodel, K.S., Lepikson, H.A., & Machado, B.S. (2024). Distillation of essential oils: An innovative technological approach focused on productivity, quality and sustainability. *Plos one*, 19(2), 214-220. <https://doi.org/10.1371/journal.pone.0299502>
- [21] Souihi, M., Amri, I., Souissi, A., Hosni, K., Brahim, N.B., & Annabi, M. (2020). Essential oil and fatty acid composition of *Melissa officinalis* L. *Progress in Nutrition*, 22, 253-258. <https://doi.org/10.23751/pn.v22i1.7758>
- [22] Castro, R., Boczkaj, G., & Cabezas, R. (2023). A perspective on missing aspects in ongoing purification research towards *Melissa officinalis*. *Foods*, 12(9), 1-15. <https://doi.org/10.3390/foods12091916>
- [23] Bounihi, A., Hajjaj, G., Alnamer, R., Cherrah, Y., & Zellou, A. (2013). *In vivo* potential anti-inflammatory activity of *Melissa officinalis* L. essential oil. *Advances in Pharmacological and Pharmaceutical Sciences*, 2013(1), 1-8. <https://doi.org/10.1155/2013/101759>
- [24] Alvarado, P.A., Soto, M.R., Gomez, F.I., Rodriguez, N.G., de Guzman, Y.R., Jara-Aguilar, D.R., & Alfaro, I.M. (2023). Effects of *Melissa officinalis* essential oil on state and trait anxiety. *Pharmacognosy Journal*, 15(3), 456-460. <https://doi.org/10.5530/pj.2023.15.101>
- [25] Latief, R., Bhat, K.A., Khuroo, M.A., Shawl, A.S., & Chandra, S. (2017). Comparative analysis of the aroma chemicals of *Melissa officinalis* using hydrodistillation and HS-SPME techniques. *Arabian Journal of Chemistry*, 10, 2485-2490. <https://doi.org/10.1016/j.arabjc.2013.09.015>
- [26] Chatzopoulou, P.S., & Katsiotis, S.T. (1995). Procedures influencing the yield and the quality of the essential oil from *Juniperus communis* L. berries. *Pharmaceutica Acta Helvetiae*, 70(3), 247-253. [https://doi.org/10.1016/0031-6865\(95\)00026-6](https://doi.org/10.1016/0031-6865(95)00026-6)
- [27] Smallfield, B.M., Klink, J.W., Perry, N.B., & Dodds, K.G. (2001). Coriander spice oil: effects of fruit crushing and distillation time on yield and composition. *Journal of Agricultural and Food Chemistry*, 49(1), 118-123. <https://doi.org/10.1021/jf001024s>
- [28] Khorshidi, J., Mohammadi, R., Fakhr, M.T., & Nourbakhsh, H. (2009). Influence of drying methods, extraction time, and organ type on essential oil content of rosemary (*Rosmarinus officinalis* L.). *Natural Science*, 7(11), 42-44. <https://www.researchgate.net/publication/284222289>
- [29] Biniyaz, T., Habibi, Z., & Yousefi, M. (2011). Comparison of chemical composition of the oils from aerial parts of *Artemisia turcomanica* Gand. at different distillation times. *Iranian Journal of Medicinal and Aromatic Plants Research*, 26(4), 574-582. <https://doi.org/10.22092/ijmapr.2011.6673>
- [30] Naderi, M., Sefidkon, F., Azizi, A., & Pourheravi, M.R. (2011). The influence of different distillation times on essential oil content and composition of *Laurus nobilis* L. *Iranian Journal of Medicinal and Aromatic Plants Research*, 27(2), 249-260. <https://doi.org/10.22092/ijmapr.2011.6401>
- [31] Khalid, K.A. (2012). Influence of hydro-distillation time on the yield and quality of dill volatile constituents. *Medicinal and Aromatic Plant Science and Biotechnology*, 6(1), 46-49.
- [32] Wesolowska, A., Jadczyk, D.A., & Grzeszczuk, M. (2012). Influence of distillation time on the content and composition of essential oil isolated from wild thyme (*Thymus serpyllum* L.). *Herba Polonica*, 58(4), 40-50.
- [33] Zheljazkov, V.D., Astatkie, T., Jeliaskova, E.A., & Schlegel, V. (2012). Distillation time alters essential oil yield, composition, and antioxidant activity of male *Juniperus scopulorum* trees. *Journal of Oleo Science*, 61(10), 537-546. <https://doi.org/10.5650/jos.61.537>
- [34] Zheljazkov, V.D., Astatkie, T., & Schlegel, V. (2012). Distillation time changes oregano essential oil yields and composition but not the antioxidant or antimicrobial activities. *HortScience*, 47(6), 777-784. <https://doi.org/10.21273/HORTSCI.47.6.777>
- [35] Zheljazkov, V.D., Horgan, T., Astatkie, T., & Schlegel, V. (2013). Distillation time modifies essential oil yield, composition, and antioxidant capacity of fennel (*Foeniculum vulgare* Mill). *Journal of Oleo Science*, 62(9), 665-672. <https://doi.org/10.5650/jos.62.665>
- [36] Zheljazkov, V.D., Cantrell, C.L., Astatkie, T., & Jeliaskova, E. (2013). Distillation time effect on lavender essential oil yield and composition. *Journal of Oleo Science*, 62(4), 195-199. <https://doi.org/10.5650/jos.62.195>
- [37] Zheljazkov, V.D., Astatkie, T., O'Brocki, B., & Jeliaskova, E. (2013). Essential oil composition and yield of anise from different distillation times. *HortScience*, 48(11), 1393-1396. <https://doi.org/10.21273/HORTSCI.48.11.1393>
- [38] Zheljazkov, V.D., Astatkie, T., Horgan, T., Schlegel, V., & Simonnet, X. (2013). Distillation time effect on essential oil yield, composition, and antioxidant capacity of sweet sage (*Artemisia annua* L.) oil. *HortScience*, 48(10), 1288-1292. <https://doi.org/10.21273/HORTSCI.48.10.1288>
- [39] Henna, A., Miguel, M.G., Brada, M., Nemmiche, S., & Figueiredo, A.C. (2016).

Composition, chemical variability and effect of distillation time on leaf and fruits essential oils of *Myrtus communis* from north western Algeria. *Journal of Essential Oil Research*, 28(2), 146-156. <http://dx.doi.org/10.1080/10412905.2015.1090936>

[40] Wesolowska, A. (2019). Influence of distillation time on the content and composition of essential oils isolated from different parts of *Agastache astromontana* 'Pink Pop'. *Journal of Essential Oil Bearing Plants*, 22(2), 311-323. <https://doi.org/10.1080/0972060X.2019.1618205>

[41] Adams RP. (2007). Identification of essential oil components by gas chromatography/quadrupole mass spectroscopy: Allured Publishing Corporation, Carol Stream, Illinois USA.

[42] Babushok, V.I., & Zenkevich, I.G. (2009). Retention indices for most frequently reported essential oil compounds in GC. *Chromatographia*, 69, 257-269. <https://doi.org/10.1365/s10337-008-0872-3>

[43] Almeida, H.H., Crujeira, P.J., Amaral, J.S., Rodrigues, A.E., & Barreiro, M.F. (2024). Disclosing the potential of *Cupressus leylandii* AB Jacks & Dallim, *Eucalyptus globulus* Labill., *Aloysia citrodora* Palau, and *Melissa officinalis* L. hydrosols as eco-friendly antimicrobial agents. *Natural Products and Bioprospecting*, 14(1), 1-12. <https://doi.org/10.1007/s13659-023-00417-9>

[44] Alizadeh, B., & Shahidi, F. (2019). *Melissa officinalis* essential oil: Chemical compositions, antioxidant potential, total phenolic content and antimicrobial activity. *Nutrition and Food Sciences Research*, 6(1), 17-25. <https://doi.org/10.29252/nfsr.6.1.17>

[45] Nurzynska, R., Bogucka-Kocka, A., & Szymczak, G. (2014). Volatile constituents of *Melissa officinalis* leaves determined by plant age. *Natural Product Communications*, 9(5), 1-4. <https://doi.org/10.1177/1934578X140090053>

[46] Nouri, A., Mirabzadeh, M., Safari, N., & Ebadi, M.T. (2020). Evaluation of essential oil composition and rosmarinic acid content in lemon balm (*Melissa officinalis* L.) cultivated in south of Iran. *Journal of Medicinal plants and By-Products*, 9(2), 159-166. <https://doi.org/10.22092/jmpb.2020.123119>

[47] Abdellatif, F., & Hassani, A. (2015). Chemical composition of the essential oils from leaves of *Melissa officinalis* extracted by hydrodistillation, steam distillation, organic solvent and microwave hydrodistillation. *Journal of Materials and Environmental Science*, 6(1), 207-213.

[48] Bogdanovic, A., Tadic, V., Arsic, I., Milovanovic, S., Petrovic, S., & Skala, D. (2016). Supercritical and high pressure subcritical fluid extraction from lemon balm (*Melissa officinalis* L., Lamiaceae). *The Journal of Supercritical Fluids*, 107, 234-242. <https://doi.org/10.1016/j.supflu.2015.09.008>

[49] Virchea, L.I., Gligor, F.G., Frum, A., Mironescu, M., Myachikova, N.I., & Georgescu, C.

(2021). Phytochemical analysis and antioxidant assay of *Melissa officinalis* L. (lemon balm). In *BIO Web of Conferences* (Vol. 40, p. 02004). EDP Sciences. <https://doi.org/10.1051/bioconf/20214002004>

[50] Cannon, J.B., Cantrell, C.L., Astatkie, T., & Zheljazkov, V.D. (2013). Modification of yield and composition of essential oils by distillation time. *Industrial Crops and Products*, 41, 214-220. <https://doi.org/10.1016/j.indcrop.2012.04.021>

[51] Medeiros, A.R., Assis, R.A., dos Santos Boeira, L., Leite, J.F., Rocha, J.M., Diotto, A.V., & Pinto, J.P. (2023). Magnetically treated water affects *Melissa officinalis* L. growth, nutritional status and essential oil compounds. *Journal of Cleaner Production*, 428, 139311. <https://doi.org/10.1016/j.jclepro.2023.139311>

[52] Sharma, S., Habib, S., Sahu, D., & Gupta, J. (2021). Chemical properties and therapeutic potential of citral, a monoterpene isolated from lemongrass. *Medicinal Chemistry*, 17(1), 2-12. <https://doi.org/10.2174/1573406416666191227111106>

[53] Maswal, M., & Dar, A.A. (2014). Formulation challenges in encapsulation and delivery of citral for improved food quality. *Food Hydrocolloids*, 37, 182-195. <https://doi.org/10.1016/j.foodhyd.2013.10.035>

[54] Djilani, A., & Dicko, A. (2012). The therapeutic benefits of essential oils. *Nutrition, Well-Being and Health*, 7, 155-179. <https://doi.org/10.5772/25344>

[55] Moudachirou, M., Gbenou, J.D., Chalchat, J.C., Chabard, J.L., & Lartigue, C. (1999). Chemical composition of essential oils of *Eucalyptus* from Benin: *Eucalyptus citriodora* and *E. camaldulensis*. Influence of location, harvest time, storage of plants and time of steam distillation. *Journal of Essential Oil Research*, 11(1), 109-118. <https://doi.org/10.1080/10412905.1999.9701085>

[56] Zheljazkov, V.D., Astatkie, T., & Schlegel, V. (2012). Effects of distillation time on the *Pinus ponderosa* essential oil yield, composition, and antioxidant activity. *HortScience*, 47(6), 785-789. <https://doi.org/10.21273/HORTSCI.47.6.785>

[57] Baydar, H., Schulz, H., Kruger, H., Erbas, S., & Kineci, S. (2008). Influences of fermentation time, hydro-distillation time and fractions on essential oil composition of Damask Rose (*Rosa damascena* Mill.). *Journal of Essential Oil Bearing Plants*, 11(3), 224-232. <https://doi.org/10.1080/0972060X.2008.10643624>

[58] Mirza, M., & Najafpour Navaei, M.D. (2016). The effect of extraction time on essential oil composition of 4 genotype of *Rosa damascene* Mill. *Eco-phytochemical Journal of Medicinal Plants*, 4(2), 23-32. [20.1001.1.23223235.1395.4.2.3.7](https://doi.org/10.1001.1.23223235.1395.4.2.3.7)

[59] Najafian, S.H., Shahcheraghi, M.A., & Rowshan, V. (2016). Investigation of the effect of extraction duration on the essential oil content and

composition of *Zataria multiflora* and *Thymus daenensis* under flowering stages. *IJHS*, 17(2), 181-192. URL: <http://journal-irshs.ir/article-1-155-en.html>

[60] Zheljazkov, V.D., Astatkie, T., Shiwakoti, S., Poudyal, S., Horgan, T., Kovatcheva, N., & Dobrev, A. (2014). Essential oil yield and composition of garden sage as a function of different steam distillation times. *HortScience*, 49(6), 785-790. <https://doi.org/10.21273/HORTSCI.49.6.785>

[61] Kholiya, S., Bhatt, G., Chauhan, A., Kumar, D., Venkatesha, K.T., Upadhyay, R.K., & Padalia, R.C. (2023). Effect of seasons, storage and distillation times on essential oil composition of *Melaleuca leucadendra* (L.). *Indian Journal of Natural Products and Resources (IJNPR)* [Formerly *Natural Product Radiance (NPR)*], 14(4), 611-616. <https://doi.org/10.56042/ijnpr.v14i4.5449>

[62] Palmieri, S., Maggio, F., Pellegrini, M., Ricci, A., Serio, A., Paparella, A., & Lo Sterzo, C. (2021). Effect of the distillation time on the chemical composition, antioxidant potential and antimicrobial activity of essential oils from different *Cannabis sativa* L. cultivars. *Molecules*, 26(16), 1-13.

[63] Kumar, R., Sharma, S., Sharma, S., & Kumar, N. (2016). Drying methods and distillation time affects essential oil content and chemical compositions of *Acorus calamus* L. in the western Himalayas. *Journal of Applied Research on Medicinal and Aromatic Plants*, 3(3), 136-141. <http://dx.doi.org/10.1016/j.jarmap.2016.06.001>

[64] Bozovic, M., Navarra, A., Garzoli, S., Pepi, F., & Ragno, R. (2017). Essential oils extraction: A 24-hour steam distillation systematic methodology. *Natural Product Research*, 31(20), 2387-2396. <https://doi.org/10.1080/14786419.2017.1309534>

[65] Elhawary, E.A., Nilofar, N., Zengin, G., & Eldahshan, O.A. (2024). Variation of the essential oil components of *Citrus aurantium* leaves upon using different distillation techniques and evaluation of their antioxidant, antidiabetic, and neuroprotective effect against Alzheimer's disease. *BMC Complementary Medicine and Therapies*, 24(1), 4-10. <https://doi.org/10.1186/s12906-024-04380-x>



بهینه‌سازی زمان استحصال تقطیری در محصولات دارویی و صنعتی تحت شرایط کروماتوگرافی-

طیف‌سنجی و مبنی بر پروفایل شیمیایی ترکیبات

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اطلاعات مقاله	چکیده
تاریخ های مقاله : تاریخ دریافت: ۱۴۰۳/۱۱/۲۲ تاریخ پذیرش: ۱۴۰۴/۲/۱۴ کلمات کلیدی: مواد مؤثره، روغن های ضروری، استخراج، غذا-دارو. DOI: 10.22034/FSCT.22.166.213. * مسئول مکاتبات: reza.shahhoseini@gmail.com r-shahhoseini@araku.ac.ir	علیرغم پیشرفت و ابداع روش های نوین در حوزه استحصال و فرآوری ترکیبات مؤثره دارویی، استحصال با تکنیک تقطیر با داشتن پیشینه متمادی توأم با پیشرفت و تکامل علمی، همچنان یک رویکرد فناوری نوآورانه محسوب می گردد که بهره‌وری، کیفیت و پایداری از شاخص های اصلی آن می باشد. این پژوهش به منظور تعیین زمان بهینه استخراج و ترسیم پروفایل بیوشیمیایی ترکیبات بر اساس آنالیز کروماتوگرافی گازی- طیف‌سنجی جرمی و در زمان های مختلف تقطیر با انتخاب بادرنجبویه (<i>Melissa officinalis</i> L. به عنوان یک گونه دارویی پرمصرف در صنایع غذایی و دارویی صورت گرفت. تجزیه و تحلیل داده ها، وجود اختلاف معنی دار در محتوی اسانس را در زمان های مختلف استخراج تبیین نمود؛ به نحوی که در تیمارهای ۱۸۰ و ۲۴۰ دقیقه به ترتیب حداکثر (۰/۳۳ درصد، v/w) و حداقل (۰/۲۵ درصد، v/w) راندمان استخراجی ثبت گردید. به طور کلی در تیمارهای مختلف تعداد ۶۲ ترکیب شیمیایی جداسازی و تعداد ۵۸ ترکیب شناسایی گردید که ژرانیال، نرال و سیترونال به عنوان شاخص اصلی اثربخشی با مجموع ۷۷/۱ درصد، سطح حداکثری را در تیمار ۱۸۰ دقیقه به خود اختصاص دادند. در کلاس ترکیبات به موازات افزایش زمان استخراج، مونوترپن های اکسیژنه کاهش یافت. بر اساس مشاهدات و تجزیه و تحلیل نتایج، زمان استحصال تقطیری مناسب برای این گونه و گونه های مشابه را می توان ۱۸۰ دقیقه ثبت نمود که علاوه بر دستیابی به حداکثر عملکرد متابولتی و در پی آن فرآورده های با کمیت و کیفیت بهینه، می توان از هدررفت نهاده های مصرفی و انرژی در فرآیند استحصال ممانعت کرد و این مسئله را به عنوان یک مرجع مهم در تولید فرآورده های غذایی و دارویی لحاظ نمود.