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Influence of distillation time and distillation apparatus on the chemical composition and quality of *Lavandula angustifolia* Mill. essential oil

Aneta Wesołowska[1*], Dorota Jadczak[2], Karolina Zyburtowicz[1]

[1]West Pomeranian University of Technology, Szczecin, Faculty of Chemical Technology and Engineering, Department of Organic and Physical Chemistry, Piastov Ave. 42, 71-065 Szczecin, Poland
[2]West Pomeranian University of Technology, Szczecin, Faculty of Environmental Management and Agriculture, Department of Horticulture, Słowackiego 17, 71-434 Szczecin, Poland
[*]*Corresponding author: e-mail:* aneta.wesolowska@zut.edu.pl

In the study, the influence of distillation time as well as distillation apparatus on the chemical composition and quality of lavender (*Lavandula angustifolia* Mill.) essential oil were investigated. Two different types of distillation apparatuses: Deryng (popular in Poland) and Clevenger-type (recommended by European Pharmacopoeia) were used for the isolation of the essential oil from dried lavender flowers (*Lavandulae flos*). Moreover, different distillation times (2, 3 and 4 hours) were also applied. The chemical composition of the isolated oils, determined by gas chromatography coupled with mass spectrometry (GC-MS), revealed the dominance of linalool (11.55–17.19%) and linalyl acetate (12.84–16.78%) in the all analyzed samples. Other important constituents were: caryophyllene oxide (5.66–7.35%), lavandulyl acetate (4.64–5.40%) and borneol (4.62–5.51%). On the basis of the obtained data it was proved that the distillation time and distillation apparatus affect the amounts of some constituents in the lavender oil.

Keywords: Lavandula angustifolia essential oil, distillation time, distillation apparatus, GC-MS, quality of lavender essential oil.

INTRODUCTION

Lavandula angustifolia Mill. (true lavender), a perennial shrub belonging to the Lamiaceae family, is cultivated all over the world due to its huge incorporation in perfumes, cosmetics, food and pharmaceutical industries[1, 2]. Products derived from lavender are distributed globally in boutiques, grocesry stores, home stores and in online stores. The essential oil isolated from the flower heads exhibit antibacterial, antioxidant, antifungal, anti-depresive, anti-inflammatory, anticonvulsive and antiviral activity. Because of its characteristic and pleasant aroma, it is widely used in aromatherapy or massage[3, 4]. Lavender oils and aqeous extracts can be found as an active ingredients in a variety of bath and body products as well as in a variety of household products. In addition to these applications, lavender oil is also used in the food industry as natural flavoring in baked goods, alcoholic and nonalcoholic beverages, puddings, chewing gum, candy and ice cream[5, 6].

The quality and commercial significance of the essential oil isolated from *L. angustifolia* depends on the levels of linalool, linalyl acetate and camphor in it. The oil with high content of linalool and linalyl acetate and low content of camphor is the most apreciated for the perfume and cosmetic industries, while this richer in camphor is mainly used in aromatherapy[7]. On the other hand, the percentage of these components in lavender oil can vary significantly depending on the cultivar, growing conditions, stage of plant development, storage conditions and even distillation conditions[8].

Steam distillation is the most preferable technique for isolation of lavender oil on a commercial scale, hovewer, during this process, molecular rearrangements, hydratation of double bonds and hydrolysis of esters may occur. Moreover, there is no agreement in the literature as to the optimal distillation time for lavender oil extraction. If the distillation time is too long, then the essential oil may have an unpleasant smell. In turn, too short distillation time may result in the lack of the higher boiling components in the essential oil[6, 9–10].

Hydrodistillation is a traditional method employed in the extraction of essential oils from aromatic plants on a laboratory scale[11]. This process may be carried out in a glass Clevenger-type or Deryng apparatus. Both devices are recommended by Polish Pharmacopoeia for determination of essential oil content in the plant material[12–13].

Our previous research indicated that the steam distillation time had a significant effect on the amounts of linalool, linalyl acetate and α-terpineol in lavender oil[14]. Therefore, we decided to investigate the influence of the type of distillation apparatus and duration of hydrodistillation on the chemical composition and quality of essential oil isolated from dried *L. angustifolia* flowers.

MATERIAL AND METHODS

Essential oils isolation

The plant material (*Lavandulae flos*) used for essential oils preparation was originated from Poland (Wielkopolska), Herb Factory 'Kawon-Hurt' (Gostyń). According to the producer, 1 g of the product contains 1g *Lavandula angustifolia* Mill. In brief, 20 g of dried lavender flowers in a 1000 mL round bottom flask together with 400 mL of distilled water was subjected to hydrodistillation using Deryng apparatus recommended by Polish Pharmacopoeia VII[12]. The hydrodistillation process was also performed with use of Clevenger-type apparatus, which is recommended by both, Polish Pharmacopoeia VII[13] as well as European Pharmacopieia[15]. In order to determine the infuence of the duration of distillation on essential oil content and its composition, three different distillation times (2, 3 and 4 hours) were applied. The obtained lavender oils were separated from water, dried over anhydrous sodium sulphate, filtered and stored in refrigerator prior to GC-MS analysis. Each distillation was repeated three times. The yield of the essential oil,

expressed as (% v/w) in Table 1, was calculated using the following equation[16]:

$$Y_{EO}\ (\%\ v/w) = \frac{volume\ of\ extracted\ essential\ oil\ (mL)}{mass\ of\ dry\ plant\ material\ (g)} \times 100$$

Gas chromatography-mass spectrometry (GC-MS) analysis

The analyses of the chemical composition of lavender essential oil samples were carried out using an Hewlett Packard 6890 gas chromatograph with a Hewlett Packard 5973 Mass Selective Detector and 6890 Series Injector. The GC was equipped with HP-5MS capillary column (30 x 0.25 mm i.d., film thickness 0.25 μm). Helium was used as a carrier gas at a flow rate of 1.2 mL/min. The injector temperature was 250 °C, the transfer line temperature was 280 °C, and the ion source temperature was 230 °C. The GC oven temperature was programmed: initial temperature of the column was 45 °C, then it was increased to 200 °C at a rate of 5 °C/min (kept constant for 10 min), and then heated up to a final temperature of 250 °C at a rate of 5 °C/min (kept constant for 20 min). The injection volumes were 1 μL (20 mg of essential oil dissolved in 1.5 mL of dichloromethane). The split injection was conducted with a split ratio of 5:1. The mass spectra were recorded at 70 eV (EI) and were scanned (2.94 scans/s) in the range 50–550 m/z. The total running time for a single sample was 71 min.

Lavender oils constituents were identified by comparison of their mass spectra with those stored in the MS databases (Wiley, NIST 2002), as well as by comparison of their mass spectra with authentic compounds (β-pinene, p-cymene, limonene, camphor, menthone, carvone, carvacrol, thymol) available in our laboratory (Fluka, Aldrich). The identity of compounds was also confirmed by comparison of their calculated retention indices (relative to n-alkanes: C_7-C_{30}, Supelco, Bellefonte, PA USA; on a HP-5MS column) with those reported in the NIST Chemistry WebBook (https://webbook.nist.gov/chemistry/) and the literature[1, 17].

Relative percentage amounts of the essential oil constituents were evaluated from the total peak area (TIC) by apparatus software (MSD ChemStation).

Statistical analysis

The analysis of variance (ANOVA) was applied for the comparison of means, while significant differences were calculated according to the post-hoc Tuckey's HSD (honestly significant difference) test at a significance level of p = 0.05, using the Statistica 13 (TIBCO Software INC. StatSoft, Poland). The statistical analysis of the results given in Table 4 was conducted for constituents of content greather than 1% of the lavender essential oil.

RESULTS AND DISCUSSION

The essential oils obtained from dried lavender flowers using two types of distillation apparatuses at different distillation times, were found to be yellow liquids with characteristic floral-herbal smell. The results presented in Table 1 indicate that the time of essential oil distillation of *L. angustifolia*, both in the Deryng and Clevenger-type apparatus, had no significant effect of its content. Hovewer, the amounts of lavender oil obtained in the Deryng apparatus was higher as compared with the Clevenger-type apparatus (1.17% and 0.86%, respectively).

The content of essential oil in *Lavandulae flos* generally depends on cultivar, geographical location, altitude, soil as well as climate conditions, propagation, morphology and ranges from 0.5 to 9.62%[7]. Our results are in agreement with those described in the literature. Although, the pharmaceutical requirements (European Pharmacopoeia 7.0) of minimum 1.3% essential oil in lavender flowers was not met in case of studied plant material.

The identified components of *L. angustifolia* oils, their retention indices (RI) and relative percentage concentrations are listed in Table 2 and 3, in order of their elution form a HP-5MS column.

A total of 121 different constituents were identified in the lavender essential oils isolated by hydrodistillation in the Deryng apparatus (Table 2). GC-MS analysis revealed that the major constituents of the oils were linalyl acetate (12.84–15.04%), linalool (11.55–13.14%), caryophyllene oxide (6.42–7.35%) and lavandulyl acetate (4.76–5.40%). Other predominant components were borneol (4.62–4.93%) and τ-cadinol (3.53–3.93%). Interestingly, small amounts of α-citral (0.09%), α-selinene (0.05%), β-bisabolene (0.04%) and α-eudesmol (0.31%) were detected only in the oil obtained by hydrodistillation conducted for three hours.

The essential oils obtained by hydrodistillation in the Clevenger-type apparatus consisted of 128 compounds in total (Table 3). Similarly, linalool (14.64–17.19%), linalyl actetae (14.50–16.78%), caryophyllene oxide (5.66–6.07%) and lavandulyl acetate (4.64–5.35%) were the major components. The isolated oils were also rich in borneol (4.88–5.51%), β-caryophyllene (3.14–3.64%) and eucalyptol (2.16–3.09%).

Generally, the major constituents of all of the tested lavender oil samples were the same. However, compounds such as: β-thujene, α-terpinolene, lavandulol, nerol, geraniol, citronellyl formate, thymol, undecanal, hexyl hexanoate, α-cedrene, isospathulenol, β-sinensal, isopropyl myristate and methyl stearate were present only in the oils isolated in the Clevenger-type apparatus. Whereas, longifolene, geranylacetone, alloaromadendrene, germacrene D, α-selinene, β-bisabolene, tridecanal, humulene epoxide epi-α-muurolol, hexyl salicylate and (2E, 6E)-farnesol were detected only in the oils obtained in the Deryng apparatus.

The statistical analysis of the results presented in Table 4 shows that the type of distillation apparatus affected the percentage content of some lavender oil constituents. The highest concentrations of linalool (17.19%)

Table 1. Essential oil content in lavender flowers in dependence on distillation time and distillation apparatus

Essential oil content % (v/w)										
Deryng apparatus			Mean value	Clevenger apparatus			Mean value	Distillation time mean value		
Distillation time				Distillation time						
2 h	3 h	4 h		2 h	3 h	4 h		2 h	3 h	4 h
1.17a	1.12a	1.21a	1.17a	0.85b	0.82b	0.92b	0.86b	1.01ab	0.97b	1.06a

Means followed by the same letter are not significantlydifferent at p = 0.05

Table 2. Percentage composition of lavender oil in dependence on distillation time in the Deryng apparatus

No.	Compound	Rt [min]	$RI_{Exp.}$	$RI_{Lit.}$	Distillation time		
					2h	3h	4h
1.	α-Pinene	6.58	934	934	0.13	0.17	0.19
2.	Camphene	6.94	949	949	0.19	0.23	0.25
3.	β-Pinene	7.66	977	978	0.09	0.10	0.11
4.	1-Octen-3-ol	7.71	979	979	0.15	0.15	0.17
5.	3-Octanone	7.90	987	988	0.35	0.33	0.37
6.	β-Myrcene	7.94	991	992	0.51	0.64	0.76
7.	3-Octanol	8.15	996	996	–*	0.14	0.14
8.	3-Carene	8.47	1008	1009	–	0.10	0.11
9.	Hexyl ethanoate	8.62	1014	1015	0.29	0.30	0.32
10.	α-Terpinene	8.71	1017	1017	0.21	0.14	0.19
11.	m-Cymene	8.86	1022	1023	0.09	0.09	0.10
12.	p-Cymene	8.93	1025	1026	0.41	0.45	0.52
13.	Limonene	9.05	1029	1030	0.33	0.33	0.37
14.	Eucalyptol (1,8-Cineole)	9.11	1031	1031	1.75	1.79	1.90
15.	(Z)-β-Ocimene	9.30	1038	1038	0.35	0.36	0.40
16.	(E)-β-Ocimene	9.59	1048	1048	0.41	0.49	0.51
17.	γ-Terpinene	9.89	1059	1060	0.12	0.14	0.15
18.	*cis*-Linalool oxide	10.28	1073	1073	2.86	2.75	2.94
19.	*trans*-Linalool oxide	10.72	1089	1089	2.24	2.17	2.36
20.	Linalool	11.08	1102	1103	13.14	11.55	11.85
21.	Nonanal	11.17	1105	1105	0.92	1.07	1.39
22.	1-Octen-3-yl acetate	11.37	1112	1112	1.26	1.10	1.23
23.	(Z)-p-Menth-2-en-1-ol	11.66	1122	1123	0.04	0.21	0.24
24.	α-Campholenal	11.70	1124	1124	0.29	0.16	0.18
25.	allo-Ocimene	11.90	1131	1132	0.21	0.46	0.56
26.	(E)-Pinocarveol	12.17	1140	1140	0.23	0.19	0.21
27.	*cis*-Sabinol	12.24	1143	1143	0.14	0.14	0.14
28.	Camphor	12.32	1146	1146	0.99	0.92	0.95
29.	Camphene hydrate	12.40	1148	1148	–	0.11	0.11
30.	Menthone	12.51	1152	1153	0.12	0.12	0.12
31.	Nerol oxide	12.59	1155	1155	0.40	0.41	0.43
32.	Pinocarvone	12.85	1164	1164	0.04	0.05	0.08
33.	Borneol	12.93	1167	1168	4.93	4.62	4.74
34.	1-Nonanol	13.14	1175	1175	0.29	0.26	0.29
35.	Terpinen-4-ol	13.25	1178	1178	2.29	2.06	2.08
36.	p-Cymen-8-ol	13.37	1183	1183	0.40	0.39	0.40
37.	Cryptone	13.50	1187	1187	1.58	1.52	1.55
38.	α-Terpineol	13.62	1192	1192	2.84	2.57	2.59
39.	Myrtenal	13.80	1198	1197	0.30	0.32	0.34
40.	*trans*-Dihydrocarvone	13.88	1201	1201	0.30	0.05	–
41.	Verbenone	14.14	1210	1211	0.80	0.48	0.46
42.	(E)-Carveol	14.42	1220	1220	0.23	0.24	0.23
43.	(Z)-Carveol	14.56	1229	1229	1.01	0.97	1.01
44.	Pulegone	14.87	1237	1237	0.11	0.11	0.10
45.	Cuminal	15.00	1242	1242	0.71	0.71	0.69
46.	Carvone	15.10	1245	1246	0.30	0.30	0.28
47.	Linalyl acetate	15.42	1257	1258	15.04	13.31	12.84
48.	α-Citral	15.84	1272	1273	–	–	0.09
49.	Phellandral	15.90	1277	1276	0.16	0.30	0.34
50.	Bornyl acetate	16.25	1287	1287	0.69	0.69	0.66
51.	Lavandulyl acetate	16.34	1291	1292	5.40	4.91	4.76
52.	Carvacrol	16.66	1302	1302	0.36	0.42	0.40
53.	Bicycloelemene	17.41	1331	1330	0.12	0.12	0.13
54.	δ-Elemene	17.57	1337	1339	–	0.06	0.06
55.	Piperitenone	17.66	1341	1343	0.29	0.22	0.26
56.	α-Cubebene	17.96	1352	1351	0.23	0.10	0.11
57.	Thymol acetate	18.04	1355	1357	0.13	0.06	0.05
58.	Neryl acetate	18.28	1364	1365	1.18	1.10	1.08
59.	Decanoic acid	18.56	1375	1374	0.16	0.15	0.17
60.	3-Methyltridecane	18.65	1378	1377	–	0.10	0.11
61.	Geranyl acetate	18.69	1384	1385	2.19	2.10	2.03
62.	β-Bourbonene	18.97	1391	1391	–	–	0.09
63.	β-Elemene	19.16	1398	1397	0.18	0.14	0.10
64.	Longifolene	19.37	1406	1407	-	0.03	–
65.	*cis*-α-Bergamotene	19.61	1416	1417	0.16	0.18	0.21
66.	β-Caryophyllene	19.78	1423	1423	2.92	3.16	3.37
67.	*trans*-α-Bergamotene	20.15	1438	1438	0.17	0.23	0.27
68.	Aromadendrene	20.40	1448	1449	–	0.07	0.08
69.	Geranylacetone	20.45	1450	1452	–	–	0.05
70.	(E)-β-Farnesene	20.63	1457	1458	0.91	0.96	0.99

No.	Compound	Rt [min]	$RI_{Exp.}$	$RI_{Lit.}$	Distillation time		
					2h	3h	4h
71.	Alloaromadendrene	20.72	1461	1462	0.06	0.07	0.08
72.	Germacrene D	21.18	1479	1480	–	0.11	0.10
73.	γ-Muurolene	21.31	1484	1485	0.32	–	–
74.	β-Selinene	21.38	1487	1488	0.10	0.12	0.12
75.	α-Selinene	21.62	1497	1498	–	–	0.05
76.	β-Bisabolene	21.93	1510	1511	–	–	0.04
77.	Tridecanal	22.02	1514	1513	–	0.04	0.05
78.	γ-Cadinene	22.10	1517	1517	0.85	0.97	1.02
79.	β-Sesquiphellandrene	22.20	1521	1523	0.89	0.88	0.80
80.	δ-Cadinene	22.31	1526	1528	0.08	0.27	0.27
81.	Cadina-1,4-diene	22.53	1535	1536	0.18	0.20	0.20
82.	α-Cadinene	22.69	1542	1544	–	0.04	0.09
83.	α-Calacorene	22.79	1546	1546	0.31	0.34	0.31
84.	Germacrene B	23.04	1557	1558	0.64	0.56	0.50
85.	(E)-Nerolidol	23.26	1566	1565	0.24	0.26	0.22
86.	Dendrasaline	23.53	1578	1579	0.21	0.22	0.20
87.	Spathulenol	23.63	1582	1582	0.24	0.25	0.22
88.	Caryophyllene oxide	23.78	1588	1589	7.35	7.18	6.42
89.	Humulene epoxide	24.17	1605	1606	–	0.14	0.13
90.	Tetradecanal	24.36	1613	1614	0.40	0.40	0.39
91.	Humulene epoxide II	24.48	1619	1619	0.49	0.57	0.53
92.	γ-Eudesmol	24.66	1627	1627	0.35	0.43	0.46
93.	epi-α-Cadinol	24.89	1637	1638	0.18	0.33	0.33
94.	epi-α-Muurolol	24.97	1641	1642	0.36	0.60	0.60
95.	τ-Cadinol	25.05	1644	1644	3.53	3.93	3.57
96.	α-Muurolol	25.27	1654	1654	0.30	0.36	0.34
97.	Valerianol	25.38	1659	1659	0.22	–	0.08
98.	α-Cadinol	25.43	1661	1663	0.79	1.53	1.37
99.	α-Eudesmol	25.53	1666	1667	–	–	0.31
100.	Cadalene	25.73	1675	1675	1.56	1.91	1.74
101.	1-Tetradecanol	25.83	1679	1679	0.32	0.42	0.38
102.	Hexyl salicylate	25.91	1683	1683	0.08	0.22	0.19
103.	α-Bisabolol	26.01	1687	1687	0.50	0.63	0.56
104.	epi-α-Bisabolol	26.13	1692	1692	1.01	1.23	1.13
105.	Heptadecane	26.34	1702	1700	0.17	0.27	0.25
106.	5-Ethyl-5-Methylpentadecane	26.46	1707	1710	0.17	0.23	0.21
107.	Pentadecanal	26.58	1713	1714	0.39	0.59	0.63
108.	(E,E)-Farnesal	26.70	1719	1719	0.36	0.50	0.47
109.	Oplopanone	26.97	1731	1735	–	0.18	0.15
110.	(2E,6E)-Farnesol	27.21	1743	1743	–	0.02	0.02
111.	α-Sinensal	27.33	1748	1752	0.71	0.88	0.79
112.	Myristic acid	27.82	1771	1771	0.19	0.28	0.25
113.	(E)-α-Atlantone	27.94	1777	1777	0.08	0.16	0.16
114.	Phenanthrene	28.13	1786	1784	–	0.11	0.13
115.	Octadecane	28.47	1801	1800	0.15	0.24	0.20
116.	Hexadecanal	28.60	1808	1811	0.11	0.21	0.20
117.	Hexahydrofarnesyl acetone	29.30	1842	1843	0.45	0.60	0.59
118.	1-Nonadecene	30.39	1896	1894	0.05	0.19	0.18
119.	Palmitoleic acid	31.57	1952	1953	0.28	0.39	0.35
120.	Palmitic acid	31.71	1959	1960	–	–	0.02
121.	1-Octadecanol	34.99	2089	2090	0.12	0.13	0.16
	Total identified [%]				99.43	99.53	99.92

R_t – retention time
$RI_{Exp.}$ – retention indices relative to n-alkanes (C_7-C_{30}) on a HP-5 MS capillary column
$RI_{Lit.}$ – literature retention indices
–* – not detected

and linalyl acetate (16.78%) were noticed in the lavender oil obtained by 2-hour distillation in the Clevenger-type apparatus. However, the percentage content of caryophyllene oxide was higher in the lavender oil obtained in the Deryng apparatus. Moreover, essential oil derived from lavender flowers distilled in the Deryng apparatus was richer in *cis*- and *trans*-linalool oxide, neryl acetate, α-cadinol, cadalene and epi-α-bisabolol. For lavandulyl acetate the most effective was a 2 hours distillation time, less effective was 3 hours and the least effective was 4 hours, both in the Deryng and Clevenger-type apparatus. A slightly higher percentage of camphor was recorded in the oil isolated from lavender flowers by 2-hour distillation in the Clevenger-type apparatus. Interestingly, the content of this compound in the lavender oil isolated in the Deryng apparatus was almost equal, regardless of the duration of distillation.

According to our analysis, a shorter distillation time (2 hours) of lavender flowers may result in an essential oil richer in linalool, 1-octen-3-yl acetate, terpinen-4-ol, linalyl acetate, lavandulyl acetate and geranyl acetate. A higher content of γ-cadinene, τ-cadinol, α-cadinol and cadalene was noted in the essential oil obtained by two- and three-hour distillation. Hovewer, the concentrations of these constituents varied irregulary depending on the duration of distillation. In case of such constituents as eucalyptol, *cis*- and *trans*-linalool oxide, borneol, cryptone, α-terpineol, neryl acetate, β-caryophyllene, (E)-β-

Table 3. Percentage composition of lavender oil in dependence on distillation time in the Clevenger-type apparatus

No.	Compound	Rt [min]	$RI_{Exp.}$	$RI_{Lit.}$	Distillation Time		
					2 h	3 h	4 h
1.	(E)-2-Hexen-1-ol	4.95	864	864	–*	–	0.05
2.	α-Thujene	6.32	928	928	0.03	–	–
3.	α-Pinene	6.58	934	934	0.30	0.25	0.24
4.	Camphene	6.94	949	949	0.47	0.32	0.28
5.	β-Thujene	7.43	970	971	0.06	–	–
6.	β-Pinene	7.66	977	978	0.34	0.41	0.41
7.	3-Octanone	7.90	987	988	0.68	0.49	0.48
8.	β-Myrcene	7.94	991	992	0.84	0.44	0.39
9.	6-Methyl-5-hepten-2-ol	7.99	992	993	–	0.08	0.13
10.	3-Octanol	8.15	996	996	0.13	0.17	0.17
11.	3-Carene	8.47	1008	1009	0.15	0.13	0.13
12.	Hexyl ethanoate	8.62	1014	1015	0.56	0.46	0.41
13.	α-Terpinene	8.71	1017	1017	0.24	0.23	0.14
14.	m-Cymene	8.86	1022	1023	0.18	0.15	0.13
15.	p-Cymene	8.93	1025	1026	0.77	0.60	0.58
16.	Limonene	9.05	1029	1030	0.55	0.41	0.38
17.	Eucalyptol (1,8-Cineole)	9.11	1031	1031	3.09	2.25	2.16
18.	(Z)-β-Ocimene	9.30	1038	1038	0.47	0.37	0.36
19.	(E)-β-Ocimene	9.59	1048	1048	0.60	0.42	0.40
20.	γ-Terpinene	9.89	1059	1060	0.23	0.19	0.19
21.	*cis*-Sabinene hydrate	10.11	1069	1070	0.08	0.04	0.09
22.	*cis*-Linalool oxide	10.28	1073	1073	1.88	1.86	1.73
23.	*trans*-Linalool oxide	10.72	1089	1089	1.40	1.41	1.32
24.	α-Terpinolene	10.77	1093	1093	0.13	–	–
25.	Linalool	11.08	1102	1103	17.19	14.65	14.64
26.	Nonanal	11.17	1105	1105	1.12	1.13	1.31
27.	1-Octen-3-yl acetate	11.37	1112	1112	1.93	1.44	1.38
28.	(Z)-p-Menth-2-en-1-ol	11.66	1122	1123	0.18	0.27	0.30
29.	α-Campholenal	11.70	1124	1124	0.23	0.18	0.17
30.	allo-Ocimene	11.90	1131	1132	0.42	0.22	0.39
31.	(E)-Pinocarveol	12.17	1140	1140	0.06	0.37	0.31
32.	*cis*-Sabinol	12.24	1143	1143	0.16	0.09	0.12
33.	Camphor	12.32	1146	1146	1.43	1.25	1.21
34.	Camphene hydrate	12.40	1148	1148	0.15	0.13	0.11
35.	Menthone	12.51	1152	1153	0.10	0.05	0.09
36.	Nerol oxide	12.59	1155	1155	0.62	0.52	0.56
37.	Pinocarvone	12.85	1164	1164	0.20	0.12	0.12
38.	Borneol	12.93	1167	1168	4.88	5.34	5.51
39.	Lavandulol	12.96	1171	1171	–	0.09	0.12
40.	1-Nonanol	13.14	1175	1175	0.05	0.25	0.23
41.	Terpinen-4-ol	13.25	1178	1178	2.46	2.09	2.15
42.	p-Cymen-8-ol	13.37	1183	1183	0.37	0.39	0.34
43.	Cryptone	13.50	1187	1187	1.70	1.59	1.54
44.	α-Terpineol	13.62	1192	1192	2.56	2.49	2.46
45.	Myrtenal	13.80	1198	1197	0.36	0.36	0.34
46.	*trans*-Dihydrocarvone	13.88	1201	1201	0.19	0.31	0.23
47.	Verbenone	14.14	1210	1211	0.77	0.93	0.62
48.	(E)-Carveol	14.42	1220	1220	0.05	0.33	0.31
49.	(Z)-Carveol	14.56	1229	1229	0.17	1.10	1.13
50.	Nerol	14.59	1230	1231	0.43	–	–
51.	Pulegone	14.87	1237	1237	0.16	0.11	0.13
52.	Cuminal	15.00	1242	1242	0.99	0.82	0.85
53.	Carvone	15.10	1245	1246	0.36	0.27	0.28
54.	Geraniol	15.16	1251	1252	0.11	–	0.03
55.	Linalyl acetate	15.42	1257	1258	16.78	14.78	14.50
56.	α-Citral	15.84	1272	1273	–	–	0.13
57.	Phellandral	15.90	1277	1276	0.19	0.27	0.29
58.	Citronellyl formate	15.95	1279	1282	0.14	0.07	0.10
59.	Bornyl acetate	16.25	1287	1287	0.97	0.86	0.82
60.	Lavandulyl acetate	16.34	1291	1292	5.35	4.80	4.64
61.	Thymol	16.38	1295	1296	–	0.13	0.12
62.	Carvacrol	16.66	1302	1302	0.03	0.26	0.52
63.	Undecanal	16.76	1310	1309	0.07	0.37	0.08
64.	Bicycloelemene	17.41	1331	1330	0.13	0.17	0.17
65.	δ-Elemene	17.57	1337	1337	0.09	0.09	0.08
66.	Piperitenone	17.66	1341	1343	0.24	0.31	0.29
67.	α-Cubebene	17.96	1352	1351	0.17	0.10	0.10
68.	Thymol acetate	18.04	1355	1357	0.13	0.06	0.06
69.	Neryl acetate	18.28	1364	1365	0.94	0.84	0.84
70.	Decanoic acid	18.56	1375	1374	–	0.19	0.18
71.	3-Methyltridecane	18.65	1378	1377	–	0.11	0.12

No.	Compound	Rt [min]	$RI_{Exp.}$	$RI_{Lit.}$	Distillation Time		
					2 h	3 h	4 h
72.	Geranyl acetate	18.69	1384	1385	2.01	1.60	1.52
73.	Hexyl hexanoate	18.74	1386	1386	–	0.05	0.11
74.	β-Bourbonene	18.97	1391	1391	0.02	–	–
75.	β-Elemene	19.16	1398	1397	0.14	0.15	0.14
76.	α-Cedrene	19.48	1415	1413	0.09	0.14	0.15
77.	*cis*-α-Bergamotene	19.61	1416	1417	0.10	0.03	0.10
78.	β-Caryophyllene	19.78	1423	1423	3.14	3.31	3.64
79.	*trans*-α-Bergamotene	20.15	1438	1438	0.28	0.28	0.27
80.	Aromadendrene	20.40	1448	1449	0.08	0.10	0.06
81.	(E)-β-Farnesene	20.63	1457	1458	0.79	0.98	1.01
82.	γ-Muurolene	21.31	1484	1485	0.32	0.39	0.15
83.	β-Selinene	21.38	1487	1488	0.11	0.12	0.13
84.	γ-Cadinene	22.10	1517	1517	0.93	1.04	1.12
85.	β-Sesquiphellandrene	22.20	1521	1523	–	0.80	0.78
86.	δ-Cadinene	22.31	1526	1528	0.14	0.12	0.22
87.	Cadina-1,4-diene	22.53	1535	1536	0.24	0.22	0.16
88.	α-Cadinene	22.69	1542	1544	0.17	–	–
89.	α-Calacorene	22.79	1546	1546	0.19	0.25	0.27
90.	Germacrene B	23.04	1557	1558	0.49	0.57	0.53
91.	(E)-Nerolidol	23.26	1566	1565	0.15	0.18	0.17
92.	Dendrasaline	23.53	1578	1579	0.19	0.20	0.19
93.	Spathulenol	23.63	1582	1582	0.11	0.26	0.25
94.	Caryophyllene oxide	23.78	1588	1589	6.07	5.90	5.66
95.	Hexadecane	23.96	1601	1600	0.20	–	–
96.	Tetradecanal	24.36	1613	1614	0.26	0.36	0.31
97.	Humulene epoxide II	24.48	1619	1619	0.19	0.46	0.42
98.	γ-Eudesmol	24.66	1627	1627	0.21	0.25	0.24
99.	Isospathulenol	24.77	1632	1633	–	0.13	0.20
100.	epi-α-Cadinol	24.89	1637	1638	–	0.44	0.44
101.	τ-Cadinol	25.05	1644	1644	1.25	3.03	3.05
102.	α-Muurolol	25.27	1654	1654	0.51	0.26	0.26
103.	Valerianol	25.38	1659	1659	0.52	–	0.15
104.	α-Cadinol	25.43	1661	1663	0.05	1.02	0.90
105.	α-Eudesmol	25.53	1666	1667	–	0.24	0.15
106.	Cadalene	25.73	1675	1675	–	1.40	1.47
107.	1-Tetradecanol	25.83	1679	1679	0.25	0.26	0.27
108.	α-Bisabolol	26.01	1687	1687	0.06	0.32	0.33
109.	epi-α-Bisabolol	26.13	1692	1692	0.27	0.14	0.13
110.	β-Sinensal	26.15	1693	1694	0.75	0.73	0.71
111.	Heptadecane	26.34	1702	1700	0.03	0.04	0.12
112.	5-Ethyl-5-Methylpentadecane	26.46	1707	1710	0.05	0.18	0.17
113.	Pentadecanal	26.58	1713	1714	–	0.20	0.41
114.	(E,E)-Farnesal	26.70	1719	1719	0.14	0.36	0.36
115.	Oplopanone	26.97	1731	1735	–	–	0.10
116.	α-Sinensal	27.33	1748	1752	0.59	0.58	0.59
117.	2-Methylheptadecane	27.59	1767	1764	0.04	–	–
118.	Myristic acid	27.82	1771	1771	–	0.12	0.10
119.	(E)-α-Atlantone	27.94	1777	1777	0.05	–	–
120.	Octadecane	28.47	1801	1800	–	0.13	0.14
121.	Hexadecanal	28.60	1808	1811	–	0.16	0.19
122.	Isopropyl myristyate	28.93	1832	1831	0.07	–	–
123.	Hexahydrofarnesyl acetone	29.30	1842	1843	0.27	0.42	0.50
124.	1-Nonadecene	30.39	1896	1894	–	0.03	0.05
125.	Palmitoleic acid	31.57	1952	1953	0.15	0.37	0.48
126.	Palmitic acid	31.71	1959	1960	–	0.21	0.26
127.	1-Octadecanol	34.99	2089	2090	0.18	0.21	0.22
128.	Methyl stearate	36.13	2131	2131	–	–	0.07
	Total identified [%]				99.25	99.72	99.61

Explanations see Table 2.

farnesene, caryophyllene oxide and epi-α-bisabolol, time of distillation had no significant effect on their content in lavender oil.

Our results are in line with those published by Baj et al.[18], who stated that the differences in percentage composition of the essential oils obtained using Deryng and Clevenger-type apparatus can be explained by the differences in the construction of both devices.

Literature survey shows that both the content and composition of the essential oils of *Rosa damascena*, *Mentha piperita*, *Juniperus scopulorum*, *Acorus calamus, Lavandula angustifolia* Mill., *Foeniculum vulgare* Mill., *Origanum vulgare* L. and *Origanum minutiflorum* were affected by the duration of distillation[10, 19-22]. The results obtained in the current study are similar to those reported previously by other researchers.

The quality of lavender oil is determined by two factors: a pleasant aroma and a desired content of some constituents[23]. Moreover, the ratio of linalyl acetate to linalool should be greather than 1 in a good quality oil[14]. According to ISO requirements, the accetable ranges for the main components of *L. angustifolia* oil are: linalyl acetate (25–47%), linalool (20–45%), *cis*-β-ocimene (1–10%), *trans*-β-ocimene (0.5–6%), 3-octanone

Table 4. Content of main essential oil constituents in lavender flowers in dependence on distillation time in the Deryng and Clevenger apparatus

Essential oil constituent	Deryng apparatus			Mean value	Clavenger apparatus			Mean value	Distillation time mean value		
	Distillation time				Distillation time						
	2 h	3 h	4 h		2 h	3 h	4 h		2 h	3 h	4 h
Eucalyptol	1.75 b	1.79 b	1.90 b	1.81 b	3.09 a	2.25 b	2.16 b	2.50 a	2.42 a	2.02 a	2.03 a
cis-Linalool oxide	2.86 a	2.75 a	2.94 a	2.85 a	1.88 b	1.86 b	1.73 b	1.82 b	2.37 a	2.30 a	2.34 a
trans-Linalool oxide	2.24 a	2.17 a	2.36 a	2.26 a	1.40 b	1.41b	1.32 b	1.38 b	1.82 a	1.79 a	1.84 a
Linalool	13.14 bc	11.55 c	11.85 c	12.18 b	17.19 a	14.65 b	14.64 b	15.49 a	15.17 a	13.10 b	13.25 b
Nonanal	0.92 c	1.07 bc	1.39 a	1.13 b	1.12 bc	1.13 bc	1.31 ab	1.19 a	1.02 b	1.10 b	1.35 a
1-Octen-3-yl acetate	1.26 bc	1.10 c	1.23 bc	1.20 b	1.93 a	1.44 b	1.38 bc	1.59 a	1.60 a	1.27 b	1.31 b
Camphor	0.99 c	0.92 c	0.95 c	0.96 b	1.43 a	1.25 ab	1.21 b	1.30 a	1.21 a	1.09 b	1.08 b
Borneol	4.93 ab	4.62 b	4.74 ab	4.76 b	4.88 ab	5.34 ab	5.51 a	5.24 a	4.91 a	4.98 a	5.13 a
Terpinen-4-ol	2.29 ab	2.06 b	2.08 b	2.14 a	2.46 a	2.09 b	2.15 ab	2.24 a	2.38 a	2.08 b	2.12 b
Cryptone	1.58 a	1.52 a	1.55 a	1.55 a	1.70 a	1.59 a	1.54 a	1.61 a	1.64 a	1.56 a	1.54 a
α-Terpineol	2.84 a	2.57 a	2.59 a	2.67 a	2.56 a	2.49 a	2.46 a	2.50 a	2.70 a	2.53 a	2.53 a
(Z)-Carveol	1.01 a	0.97 a	1.01 a	1.00 a	0.17 b	1.10 a	1.13 a	0.80 b	0.59 b	1.04 a	1.07 a
Linalyl acetate	15.04 b	13.31 cd	12.84 d	13.73 b	16.78 a	14.78 bc	14.50 bcd	15.35 a	15.91 a	14.04 b	13.67 b
Lavandulyl acetate	5.40 a	4.91 ab	4.76 b	5.03 a	5.35 a	4.80 b	4.64 b	4.93 a	5.38 a	4.86 b	4.70 b
Neryl acetate	1.18 a	1.10 ab	1.08 ab	1.12 a	0.94 bc	0.84 c	0.84 c	0.87 b	1.06 a	0.97 a	0.96 a
Geranyl acetate	2.19 a	2.10 a	2.03 a	2.11 a	2.01 a	1.60 b	1.52 b	1.71 b	2.10 a	1.85 b	1.78 b
β-Caryophyllene	2.92 a	3.16 a	3.37 a	3.15 a	3.14 a	3.31 a	3.64 a	3.36 a	3.03 a	3.23 a	3.51 a
(E)-β-Farnesene	0.91 a	0.96 a	0.99 a	0.95 a	0.79 a	0.98 a	1.01 a	0.93 a	0.85 a	0.97 a	1.00 a
γ-Cadinene	0.85 a	0.97 a	1.02 a	0.95 a	0.93 a	1.04 a	1.12 a	1.03 a	0.89 b	1.01 ab	1.07 a
Caryophyllene oxide	7.35 a	7.18 ab	6.42 abc	6.98 a	6.07 bc	5.90 c	5.66 c	5.88 b	6.71 a	6.54 a	6.04 a
τ-Cadinol	3.53 a	3.93 a	3.57 a	3.68 a	1.25 b	3.03 a	3.05 a	2.45 b	2.39 b	3.48 a	3.31 a
α-Cadinol	0.79 c	1.53 a	1.37 ab	1.23 a	0.05 d	1.02 abc	0.90 bc	0.66 b	0.42 b	1.28 a	1.13 a
Cadalene	1.56 ab	1.91 a	1.74 ab	1.74 a	–*	1.40 b	1.47 b	0.96 b	0.78 b	1.66 a	1.61 a
epi-α-Bisabolol	1.01 a	1.23 a	1.13 a	1.12 a	0.27 b	0.14 b	0.13 b	0.18 b	0.64 a	0.68 a	0.63 a

Means followed by the same letter are not significantly different at p = 0.05; –* – not detected

(0–5%), camphor (0–1.5%), 1,8-cineole (0–3%), limonene (0–1%), terpinen-4-ol (0–8%), lavandulyl actetae (0–8%), lavandulol (0–3%) and α-terpineol (0–2%)[23–24].

All the studied lavender oil samples contained less linalyl acetate, linalool, *cis*- and *trans*-β-ocimene than the range called for by ISO standard. They also contained higher level of α-terpineol (2.46–2.84%), than called for in the specification (0–2%). Although, the levels of 1,8-cineole (eucalyptol), limonene, 3-octanone, camphor, terpinen-4-ol and lavandulyl acetate were in the regulated ranges in the all investigated samples.

Based on the results presented in Table 5 it can be concluded that the higher quality lavender oils were obtained by hydrodistillation in the Deryng apparatus. The linalyl acetate to linalool ratio in the essential oils distilled in the Clevenger-type apparatus was slightly lower than that in the Deryng apparatus and only in case of 3-hour distillation was higher than 1. Moreover, the content of camphor (Table 4) in the essential oils obtained in the Deryng apparatus did not exceed 1%, which also confirms their better quality.

The aspect related to the aroma quality of lavender oil is very important from the industrial point of view. Linalyl acetate is responsible for the floral-woody scent of the oil, while 1,8-cineole and camphor give it a sharper note. The presence of camphor in an amount exceeding 1.2% reduces the quality of the aroma, giving it a fresher accent[25].

The chemical composition of the essential oil isolated by hydrodistillation from dried flowers of *L. angustifolia* cultivated in Wielkopolska was previously assessed by Śmigielski et al.[26] Linalool (30.6%), linalyl acetate (14.2%), geraniol (5.3%), β-caryophyllene (4.7%) and lavandulyl acetate (4.4%) were the major constituents

Table 5. Comparison of the linalyl acetate to linalool ratio for lavender oils isolated in the Deryng and Clevenger-type apparatus

Distillation time	Linalool (%)	Linalyl acetate (%)	Ratio (linalyl acetate/linalol)
Deryng apparatus			
2 h	13.14	15.04	1.14
3 h	11.55	13.31	1.15
4 h	11.85	12.84	1.08
Clevenger apparatus			
2 h	17.19	16.78	0.98
3 h	14.65	14.78	1.01
4 h	14.64	14.50	0.99

of oil. Other compounds, in a lesser amount were terpinen-4-ol (3.4%), α-terpineol (2.7%), 1,8-cineole (2%) and lavandulol (1.6%). Moreover, these authors stated that only *cis*-β-ocimene, linalyl acetate and α-terpineol in Polish lavender oil are out of ranges of the ISO requirements. In addition, the study by Śmigielski and Prusinowska[27] has shown that linalool (27.3–34.7%), linalyl acetate (19.7–22.4%), lavandulyl acetate (4.5–5.7%), ocimene (1.9–2.9%), terpinen-4-ol (1.1–2.0%), lavandulol (0.6–0.8%), camphor (0.2–0.3%) and cineol (0.2–0.5%) characterized the essential oils isolated from *L. angustifolia* cultivated in Poland.

Our results are partly in line with the findings of Śmigielski et al.[26] Interestingly, the content of linalool and linalyl acetate found in our oils was also lower than that reported by Śmigielski and Prusinowska[27] in Polish lavender. Such differences in the chemical composition of lavender oils are most likely due to different weather conditions during lavender growing or fertilization.

CONCLUSIONS

In this study, the influence of the type of distillation apparatus and duration of hydrodistillation on essential oil content and composition of *L. angustifolia* were investigated. It was observed that increasing distillation time did not result in higher essential oil content, both in the Deryng and Clevenger-type apparatus. It was revealed that linalool, linalyl acetate, caryophyllene oxide, lavandulyl acetate and borneol dominated in the all analysed oil samples. The obtained oils did not conform to the requirements of ISO for the chemical composition of *L. angustifolia* Mill. The main reason for such nonconformity was the percentage of linalool, linalyl acetate and ocimenes. Additionally, it was proved that in order to obtain the essential oil with the highest content of linalool and linalyl acetate, the process of hydrodistillation should by carried out by 2 hours in the Clevenger-type apparatus. If a low-camphor oil (below 1.2%) is desirable, lavender flowers need to be distilled for 2 hours in the Deryng apparatus.

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